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EXTENDED ABSTRACTS

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Institute of Metals and Technology, Ljubljana, Slovenia

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HEAT TREATMENT

POSSIBILITIES OF HEAT TRANSFER CONTROL DURING QUENCHING

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EXTENDED ABSTRACT

Quenching is a nonstationary thermodynamic process the aim of which is to attain the required level of superficial hardness,as well as adequate hardness distribution on the cross-section of a hardened workpiece, with minimum deformation and size change. These two requirements are opposite to each other. A higher quenching intensity to achieve greater depth of hardening increases deformation and size change.Therefore it is necessary to control *the dynamics of heat extraction* from the workpiece i.e. to optimize the quenching parameters and control the quenching intensity during the whole quenching process.

From the first moment when a workpiece is immersed in a quenchant, three different processes start and develop simultaneously:the thermodynamical one (heat extraction), the metallurgical one (microstructure transformation) and the mechanical one (stress and strain development).The transformation of the microstructure does not start on the whole cross-section simultaneously,but gradually from the surface to the core only then when a particular point attains the temperature A_1. This fact makes it possible (at least for bigger cross-sections) to *intentionally change the heat extraction dynamics* i.e. to control the quenching process.

For numerical simulation of every quenching process it is necessary to know the temperature dependent heat transfer coefficient (HTC), which depends on many influential factors: geometrical, fluid-dynamical, material's dependent and surface condition's dependent. There is a fundamental difference between the HTC for quenching in liquid quenchants and for gas quenching.The usual way to calculate the HTC is to measure the temperature at a point below surface of a specimen or probe and by applying *the inverse heat conduction method* to calculate the heat flux on the surface and the surface temperature. For estimation of the HTC one needs to know: the cooling curve at the location where the temperature is measured, thermophysical properties of the workpiece's material and the temperature of the quenchant.It is important to distinguish between the HTC calculated for the small laboratory test specimen and for real workpieces. The HTC calculated for small laboratory test specimen cannot be used for quenching simulation of real workpieces in practice because of the following reasons: real workpieces have bigger mass (in case of cylindrical parts have bigger diameters); their cooling times are much longer, and some parameters which occur in practice (bath temperature,agitation rate and direction, position and loading arrangement in a batch) cannot be taken into account at laboratory tests. Therefore for calculation of HTC in case of real cylindrical workpieces the Liscic/ Nanmac quench probe was used. It is a cylinder of 50 mm diameter and 200 mm length instrumented with three thermocouples (TC) the outher of which of special design (U.S.Patent No.2,829.185) measures the temperature at the very surface.By applying the *Temperature Gradient Method* the heat flux density and the HTC can easily be calculated. To calculate realistic values of HTC for workpieces in practice the quenching probe should be of the same shape as the workpiece (cylinder; plate; ring) and of similar dimensions (volume to surface ratio). The TC should have the smalles possible diameter and its position should be as close to the surface as possible, in order to minimize the damping effect, the time lag and the TC response time.

Changing *intentionally* the heat extraction dynamics *during quenching* in liquid quenchants, especially when workpieces of thin cross-sections are involved, is practically impossible or limited to special quenching techniques. Two of such examples are: *Intensive Quenching* and *Delayed Quenching.*At **Intensive Quenching** usually plain water is used but its flow velocity is extremely high, so that(at the IQ-3 variant) from the beginning of quenching eliminates the film as well as the boiling phases.As a consequence suddenly a hardened crust around the workpiece is formed in which high compressive stresses exist. The most important feature of this

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technology is the interruption of cooling at the moment when the compressive stresses attain its maximum. As the results from practice prove, the distortion of parts is less and the fatigue-strength is increased so that the working life of workpieces is substantially increased, even if lower alloyed steels are used. At **Delayed Quenching** the first part is slow cooling (usually in air) followed by immersing the workpiece in a liquid quenchant. The cooling rate within the surface region is low, but after immersion in a liquid quenchant it becomes high during the period when the structure transformation in the core takes place.The characteristic feature of this technology is *the discontinuous change of cooling rate.* From the earlier works of Shimizu and Tamura it is known that the pearlite transformation in case of discontinuous change of cooling rate differs from what it should be according to the CCT diagram, and depends on the consumed incubation before the discontinuous change of cooling rate occured. They have also given explanation why at delayed quenching an *inverse hardness distribution* on the workpiece's cross-section (i.e.higher hardness in the core than at the surface) can occur. Recent investigation by Liscic and Totten has shown that Polyalkilen-Glykol (PAG) solution of high concentration can be used for a preprogrammed and reproducible delayed quenching. The results of this investigation enable two important conclusions.The first is: the dynamic of heat extraction at quenching is responsible for the hardness distribution achieved. The second one is: a controlled delay quenching has a much greater potential to achieve through-hardening, compared to conventional quenching process.

Gas Quenching. High Pressure Gas Quenching (HPGQ) in vacuum furnaces is a modern and promising technology with many advantages compared to liquid quenchants. Cooling takes longer time, hence some quenching parameters can be intentionally changed *during* quenching. Because cooling follows the Newton's law there is no uncontrollable changes in heat transfer. The main shortcoming of HPGQ, especially at workpieces of big cross-section made of steel having low hardenability, is the inability to attain the required hardness in the core, due to insufficient quenching intensity. According to the Newton's law there are two possibilities to remedy this situation. The first one is to increase the heat transfer by increasing the gas pressure and its velocity, which is usually applied but is limited because of economical reasons (furnace design and blower's power required). The other one is to increase the temperature difference between the workpieces' surface and the circulating gas by *transient spraying with liquid nitrogen* which suddenly increases the heat flux, because of the very low temperature of evaporated nitrogen. By using both possibilities simultaneously i.e.a combination of main gas stream (of adequate pressure and velocity) and transient spraying of liquid nitrogen, new *Controllable Heat Extraction Technology* (*CHE*) can be developed. It would enable a broad spectrum of quenching intensities and eliminate the above described shortcoming of the HPGQ. While the change of gas pressure or of its velocity requires a certain period of time, spraying of liquid nitrogen can start and be interrupted momentarily, which enables a very flexible control of the quenching intensity. Moreover providing that the furnace is equipped with necessary control system and software programme, fully automatic control of the heat extraction during quenching is possible.

THE INFLUENCE OF COOLING RATE AND AUSTENITIZATION TEMPERATURE ON THE MICROSTRUCTURE AND PROPERTIES OF A MEDIUM CARBON MICROALLOY FORGING STEEL

Morteza Abed*, Ahad Zabett

ABSTRACT

A practice was carried out to evaluate the influence of austenitization temperature and cooling rate on the microstructure and mechanical properties of medium carbon microalloyed steel. After austenitization at two temperature of 1100 °C and 1300 °C the microalloyed steel specimens were cooled to various schedules in order to study the influence of cooling rate and austenitization temperature on the characteristic of microstructure and some mechanical properties. The yield and tensile strength and hardness values were determined. The volume fraction of ferrite and pearlite as the function of cooling rate and austenitization temperature was followed by optical microscopy using MIP technique. The results signify that by increasing the cooling rate both strength and hardness increase. Also the volume fraction of ferrite is increased by increasing the cooling rate. Increasing the austenitization temperature seems to raise the both strength and hardness and also volume fraction of ferrite.

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THE USE OF NEW TYPES OF LARGE AND MIDDLE SIZE VACUUM BATCH FURNACE FOR THE HEAT TREATMENT OF MOULDS AND DIES

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ABSTRACT

Vacuum heat treating of tools and dies under demands of optimized parts properties and less distortion is one of the most important goals in vacuum furnace technology actually. Due to optimized cooling conditions grain boundary precipitation leading to shorter die life can be avoided.

This presentation describes the vacuum heat treating of moulds and dies in the Ipsen newly developed Turbo˛Treater M and SuperTurbo™ vacuum furnace under different nitrogen gas quenching conditions.

Keywords: vacuum heat treatment, high pressure gas quenching, less distortion, GMPT 16" H13-standards

1 INTRODUCTION

With the increased use of high-pressure gas quenching and faster quench rates for moulds and dies, the quality of heat treatment has improved significantly over the last decade.

The demands are focused on distortion control, especially on large die inserts, as minimizing distortion saves money on post machining. The disadvantage is that low distortion is mostly realized through a very slow gas quench, which consequently results in precipitation of grain boundary carbides and a shorter die life due to reduced impact toughness (**Figure 1**).

For many applications GM POWERTRAIN Standard Specification DC-9999-1 is the key criterion for the performance of vacuum furnaces. Its definition is as follows:

“DC-9999-1 is the standard procedure and specification that ensures the quality of all H-13 and other hot work tool materials and their heat treatment as required for the GM tooling projects.”

Beside the requirements relative to the material in terms of chemical analysis and quality, the test piece and the heat treating cycle parameters and the testing procedure are specified very clearly there. In That way approved material suppliers, heat treating companies and testing laboratories are acceptable to GM only.

2 EXPERIMENTS

The test piece consists in a cube of premium grade H-13 steel (Mat. No. 1.2344 = X 40 Cr Mo V 51), size 16"x16"x16" (approx. 400mm edge length) and weighing 1173 lbs (approx. 532 kg).

The Heat treatment of that bloc is described very precisely and is shown in (**Figure 2**).

As to GMPT DC 9999-1 the heat treatment process has to comply with various requirements, like location of the load thermocouples, preheating procedure, Austenitizing

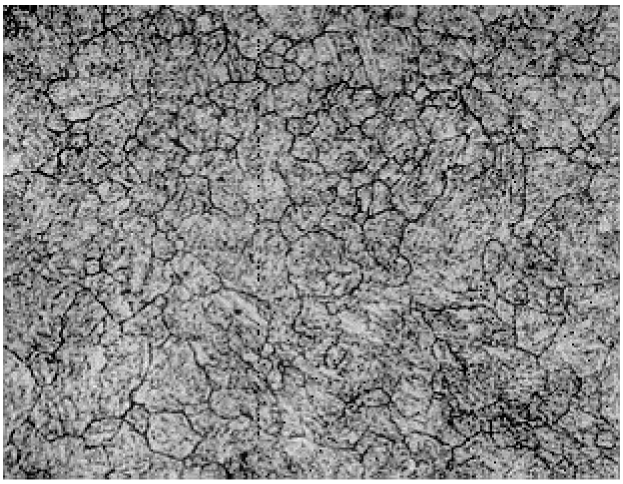

Figure 1: Inacceptable dies microstructure

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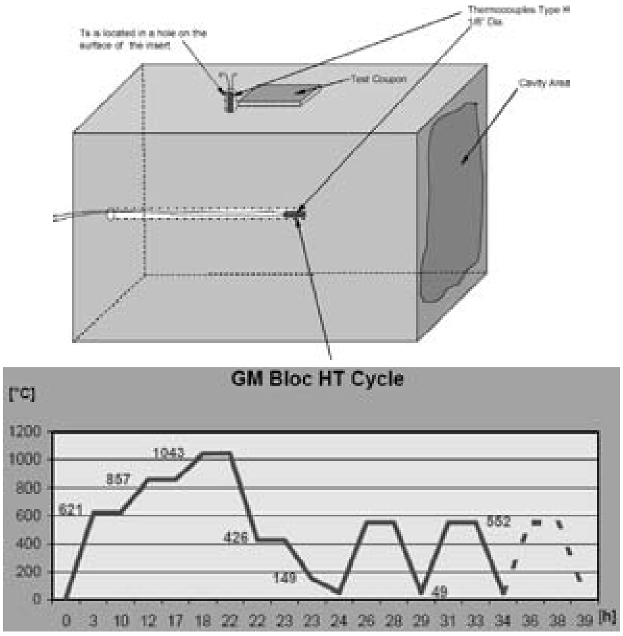

Figure 2: Test piece and Heat treating cycle as per GM POWER-TRAIN Standard Specification DC 9999-1

temperature, quenching parameters with isothermal hold, if necessary, tempering parameters (number of tempering cycles depending on the achieved hardness, temperatures and holding times).

Additionally to the trials with the GM block trials with other parts are described in the presentation:

One large 5,6 t diecasting die (1.2343) and two 230 kg forging dies (1.2343).

The equipment specified by GMPT DC 9999-1 has to meet various requirements:

Vacuum furnace with min. 10 bar nitrogen pressure backfill, minimum quench rate 39°C/min, programmable process control with load thermocouples, digital data recording of the whole cycle, furnace certification according to MIL-H-6875.

Based on those requirements two Ipsen Vacuum furnaces of different sizes have been used for the trials:

- SuperTurbo™ 77100 (useful dimensions WxLxH = 1700x3000x1400 mm, max. gross weight of load = 5000kg)
- Turbo^{2}Treater M (600x900x600 mm, 800 kg).

Figure 3: IPSEN SuperTurbo™ 77100

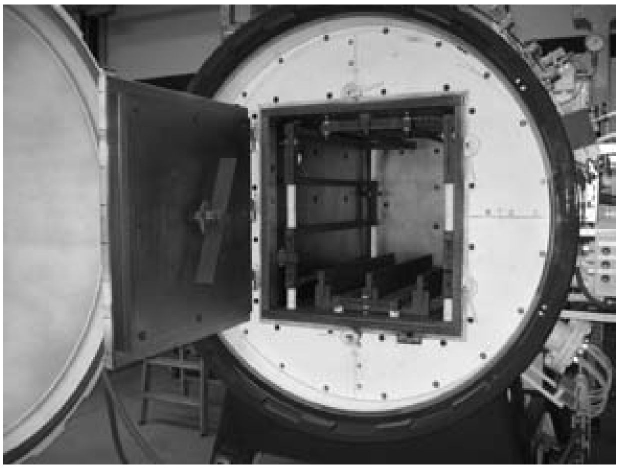

Figure 4: IPSEN Turbo^{2}Treater M

3 RESULTS

The requirement stated in the GMPT Standard Specification DC 9999-1 in terms of furnace performances and in terms of metallurgical could be achieved in both furnaces IPSEN SuperTurbo™ 77100 and in Turbo^{2}Treater M.

The diecasting die and the 230 kg forging dies showed excellent results in hardness and microstructure.

	IPSEN SuperTurbo™ 77100		Turbo^{2}Treater M	
	Diecasting die	GM 16" bloc	Forging die	GM 16" bloc
Material	1.2343	H13 (1.2344)	1.2343	H13 (1.2344)
Weight	5600 kg	532 kg	460 kg	532 kg
Austenized at	~1020 °C	1043 °C	1.000 °C	1043 °C
Gas quenching at	>9 bar	>9 bar	7 bar	12 bar
Tempering	~595 °C	–	585°C	–
Hardness	47 HRC	–	49 HRC	–

TWO-COMPONENT DIFFUSIVE STEEL SATURATION

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ABSTRACT

The present work is devoted to the research on diffusive saturation of steel with boron and copper, boron and nickel, and boron and cobalt. A subject of analysis have been the thickness of the obtained boride layers, their structure and composition, the level of cohesion of the layers, as well as the quantity of the alloying elements in them.

Keywords: steel, boriding, saturation, boride layer, thickness

1 INTRODUCTION

Boriding is a method for diffusive saturation of the surface of steel products with boron. As a result, one-phase (Fe_2B) or two-phase ($FeB+Fe_2B$) boride layer is formed. This layer is distinguished for the high level of solidity, stability to corrosion and high temperature, but also for the great fragility. When alloying elements (Cu, Ni, Co, etc.) are put into the boride layer, the pace of its forming changes significantly. Its composition, structure, cohesion to the metal base, and other properties change, too [1, 2].

The aim of the present work aims the systemization of our research on diffusive saturation of steel with boron and copper [3], boron and nickel [4], and boron and cobalt [5].

2 EXPERIMENTAL SETUP

Medium-carbon steel and alloyed instrumental steel have been borided in a powder boriding mixture with major composition [97%(12% amorphous B+88% Al_2O_3)+ 3%$Na_2O.4BF_3$] [6], alloyed separately with Cu_2O, Ni and Co_3O_4. The quantity of the alloying supplement is in the range of 0,0 to 5,0 weight %. The boriding is carried in stainless steel container [7] out at 1080 K (810 °C) and lasts 5 hours.

The borided samples are subjected to microstructural analysis, roentgenostructural analysis (RSA), and roentgenofluorescent analysis (RFA). The microstructural analysis and the measurement of the boride layers thickness (δ) are carried out with a Neophot 2 microscope after the black and white development (5% solution of HNO_3 in C_2H_5OH) of metalographic grinds. The cohesion rate (s) of the layer to the steel base is determined as a positive square root of the deviations of the measured values of layer thickness from its average values [8]. The RSA is carried out on DRON-2 with a cobalt x-ray pipe with K_α-rays, 30kV operating pressure, 20 μA current, and 1/2°/min ray speed. The contents of Cu, Ni, and Co on the surface of the boride layers is determined through RFA. For this purpose samples are prepared by pressing powder mixtures of Fe and Cu; Fe and Ni; Fe and Co. As excitement source is used 241 Am, radiating ă-rays on a silver padding. Carbon steel borided samples are submitted to a layer RFA, after layers as thick as 10-30 μm are taken mechanically.

3 RESULTS AND DISCUSSIONS

The amount of Cu_2O in the starting mixture is increased, the boride layer becomes smoothly thinner. There is an exception when samples are borided in a mixture with 2 weight % Cu_2O. The introduction of the same amounts of Ni or Co_3O_4 in the starting mixture leads to reducing the thickness of the boride layer to a greater extent.

The thickness of the boride layers in carbon steel samples in any case remains superior to the thickness of the samples of alloyed steel. This is probably due to the tormenting effect of the redistribution of chrome, carbon, molybdenum, etc., contained in the steel, on the boron diffusion.

Cu alloying improves the cohesion rate which reaches its highest level at 2 weight % Cu_2O. Ni alloying leads to a

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significant cohesion rate (18.8 μm) at 1% Ni in the starting mixture, then the cohesion rate varies from 9 to 13 μm. Alloying the boride layer with Co does not improve the cohesion rate.

Figure 1 shows microstructures of boride layers formed on samples of carbon steel in powder mixtures with no alloying supplement (a), with supplement of 2.0%Cu_2O (b), 3.0%Ni (c), and 2.0%Co_3O_4 (d). It's obvious that the prickly structure of the layer does not change, and the layer becomes thick. For samples of alloyed steel the layer is much thinner, the prickly structure is broken, and the front of growth of the layer is smoothed.

The results from RSA indicate that the obtained boride layers are two-phase (FeB+Fe_2B) with prevailing low-boron (FeB) phase. No borides of Ni and Co have been found (Cu does not form borides). The alloying elements are dissolved in the ferrous borides.

The results from the RFA analysis shows that the amount of the introduced elements Cu, Ni, and Co is higher in the boride layers formed on samples of the alloyed steel.

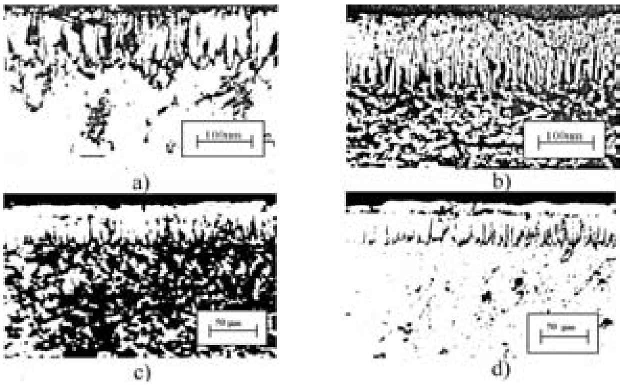

Figure 1: Microstructures (x100) of boride layers formed on carbon steel in powder mixtures: a) with no alloying supplement; b) with supplement of 2.0%Cu_2O; c) 3.0%Ni; d) 2.0%Co_3O_4.

4 CONCLUSIONS

The boride layer obtained on the two types of steel is two-phase (FeB+Fe_2B), alloyed with Cu, Ni, or Co. The layer obtained on carbon steel keeps its prickly structure and becomes thicker. In the layer obtained on alloyed steel, the prickly structure is broken and the front of growth is smoothed.

In conclusion, in order to raise the saturating ability of the starting boriding mixture and to improve the cohesion rate of the obtained boride layer to the steel base, it should be recommended to be introduced in the mixture up to 2.0% Cu_2O as an alloying supplement. Supplements containy Ni and Co for such purposed is not advisable.

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HEAT TREATMENT AND WELDING EFFECTS ON MECHANICAL PROPERTIES AND MICROSTRUCTURE EVOLUTION OF 2024 AND 7075 ALUMINIUM ALLOYS

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ABSTRACT

Aluminium alloys are sensitive to hot cracking during the welding operation both liquidation cracking in the heat affected zone (HAZ) and solidification cracking in the weld can occur.

The GTAWelding results of aluminium alloys 2024 T3 and 7075 T6 for 2 mm plate thickness with different parameters of welding are presented in this paper.

Before welding, different alloys was heat treated at different temperature to follow the evolution of microstructure and mechanicals properties.

After welding, the strength of the materials in heat affected zone (HAZ) is reduced. This reduction on properties is due to the different phenomenon that occurs during welding.

Keywords: heat treatment, aluminium alloys welding, hardness, precipitation, tensile strength, yield strength, microstructures

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Topic: A – Heat Treatment

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AFFECT OF VIBRATORY WELD CONDITIONING ON IMPACT TOUGHNESS OF WELD

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ABSTRACT

Vibratory weld conditioning is a proceeding to achieve enhancement of weld metal microstructure. It could also affect mechanical properties, level of residual stresses and deformation. In presented research work affect on impact toughness of weld metal was performed. Four different types of single welds were made. Vibrated, tempered and combination of both conditions were compared to as-welded condition. Results were compared and confirmed with multipass weld impact toughness measurements. Vibration during welding benefits energy absorbed in impact toughness test of weld metal and improves type of fracture.

1 SCOPE AND RESULTS

Purpose of research work in this article has been comparison of impact toughness of different conditions of welds. As welded condition was primary compared to weld subjected to vibration during welding (vibratory weld conditioning – VWC). To notice an influence of heat of next pass when multipass welding is performed, we made two more samples. One of them was tempered to 500 °C for 20 min after welding; the other was subjected to VWC and tempered at 500 °C with vibration after welding (vibratory stress relieving – VSR).

Material researched was steel Niomol 490, welding process was SAW with flux cored wire. Single pass V welds were made to machined slots. Nugget of weld was examined to impact toughness with non-standard Charpy samples. Some welds of different conditions were metallographic and fractographic examined.

Results express differences in impact toughness. Best results were achieved with VWC samples. Influence of heat impairs results, which could deter values achieved with VWC in multipass welds. (**Figure 1**)

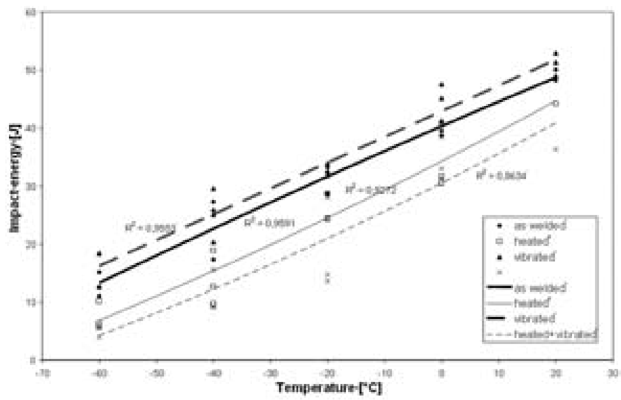

Figure 1: Impact toughness of welds

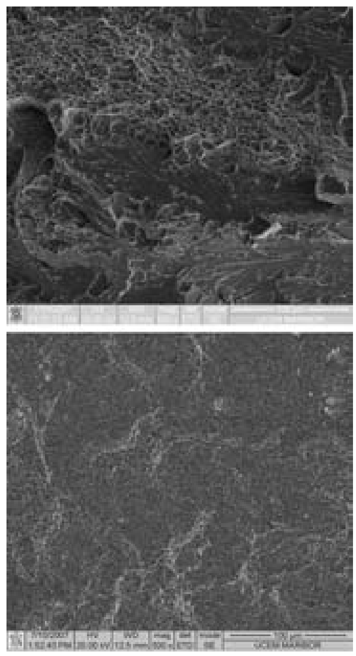

Figure 2: Fracture surface of unvibrated and vibrated specimen

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Topic: A – Heat Treatment
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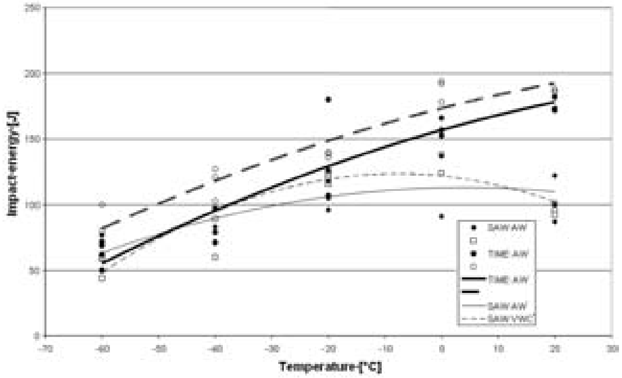

Figure 3: Impact toughness of multipass welds

According to values and shape of instrumented Charpy diagrams, vibration during welding benefits impact toughness. Fractographic examination of fractures, visually and with scanning electron microscope (SEM), reveals differences in microstructure and therefore differences in type of fracure (**Figure 2**).

To clear up exclusive results of influence of vibration and heat, we made two multipass welds with same base and filler material. As-welded condition was compared to VWC condition (**Figure 3**). Vibrated welds express higher impact toughness values.

2 CONCLUSIONS

According to results of impact testing, metallographic and fractographic research, there is positive influence of vibration on welds. Vibration affects microstructure formation, type and energy of fracture. Although tempered single pass samples express lower toughness, multipass welds also have higher impact energies when using VWC.

THE EFFECT OF AGING PARAMETERS ON PROPERTIES OF MARAGING STEEL

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ABSTRACT

In the paper the investigation of the aging parameters (temperature and time) effects on mechanical properties and dimensional changes of Maraging 15 10 5 steel is presented. Aging testings were carried out in dilatometer accompanied by simultaneous registration of forthcoming alternations of size. The obtained results indicate to significant influence of aging temperature and aging time duration on achieved hardness values, as well as on dimensional changes, which is caused to diffusive character (diffusion of alloyed elements atoms from Ni-martensite matrix) of aging process.
On the basis of carried out testing optimal aging temperature and aging time duration of Maraging 15 10 5 steel are determined.

Keywords: maraging steel, aging, mechanical properties, dimensional changes

1 INTRODUCTION

Maraging steels are delivered in solution annealed condition and have relatively soft and ductile qualities what makes them suitable for exposure to work treatment. The required operational properties of these steels are obtained by precipitation hardening (aging), with result of almost doubling their hardness and tensile strength values (compared to solution annealed condition).[1, 2]
Besides high tensile and yield strength other advantages and benefits of this sort of steel are to be emphasized: good plastic properties, good weld ability, resistance to brittle fractures and stability at low and increased temperatures. The advantages are also displayed in their convenient heat treatment qualities. Due to the above specified advantages and their wide application these steels were submitted to further improvements and development. [2, 3]

2 EXPERIMENTAL WORK

The study included carrying out of experiments with the purpose to investigate optimal parameters (temperature and time) for aging of maraging steel with the following composition: 0,010% C, 0,19% Cr, 14,33% Ni, 10,347% Co, 4,83% Mo, 0,006% V, 0,080% Al, 0,141% Cu, 0,224% Ti, 0,004% Si and 0,008% Mn, which corresponds to designation of Maraging 15 10 5. The entire samples were solution annealed and aged in Netszsch electronic dilatometer 402 EP. The use of dilatometer enables observation of ongoing dilatometric changes. The experiment was organised according to factorial experiment 3^2 as a variation of two factors (factor A – aging temperature, factor B – aging time) on three levels (ϑ_1=400 °C, ϑ_2=500 °C, ϑ_3=600 °C, t_1=1h, t_2=4h, t_3=10h). With factorial experiment 3^2, the influence of individual factors and their interactions on mechanical properties and dimensional changes of Maraging 15 10 5 steel was determined statistically. Besides variations of all factor levels the three repetitions for the same combination of factor level were conducted.
After the completion of the heat treatment, each sample was tested for hardness value by Vickers method with the load of 9,81N (HV1), five measurements each, and after that the average value was calculated.

2.1 Determination of the aging parameters effects on Maraging 15 10 5 steel

The results of variance analysis of measured hardness values HV1 are presented in **table 1**. In **table 2** the results of variance analysis of measured dimensional changes (contraction) are presented. Analysis of variance was carried out by the computer programme Statistica.
The analysis of variance (**table 1** and **table 2**) indicates the significant influence of factor A (aging temperature) on hardness values HV1 and dimensional changes after aging. Factor B (aging time) and interaction between factor A and factor B have lower influence on hardness values and dimensional changes.

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Table 1: Analysis of variance of HV1 values

Source of variation	Degr. of freedom	Sum of squares	Mean of square	Variance ratio v_o	Fisher criterion for P=95%
A	2	48185	24093	8340	3,554
B	2	2763	1381	478	3,554
AB	4	19908	4977	1723	2,928
Error	18	52	3		

Table 2: Analysis of variance of dimensional changes

Source of variation	Degr. of freedom	Sum of squares	Mean of square	Variance ratio v_o	Fisher criterion for P=95%
A	2	15801,56	7900,78	599,22	3,554
B	2	2214,89	1107,44	84	3,554
AB	4	2228,22	557,06	42,25	2,928
Error	18	237,33	13,19		

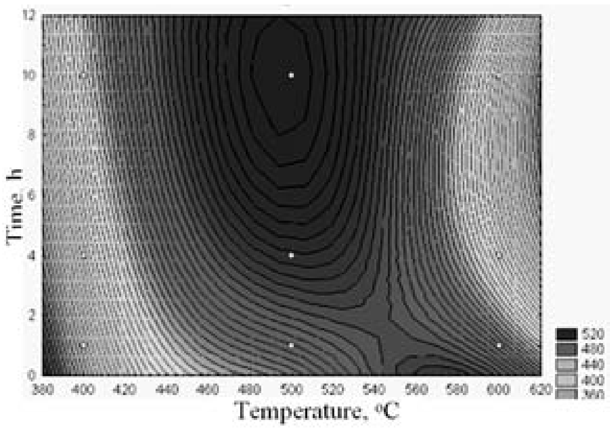

Figure 1: Distribution of hardness values HV1 of Maraging 15 10 5 steel depending on factors levels

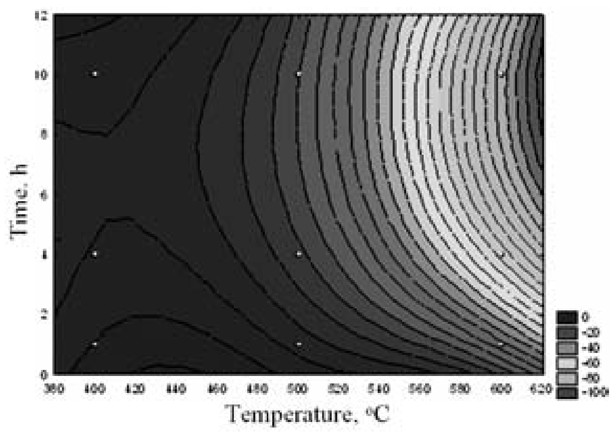

Figure 2: Distribution of dimensional changes of Maraging 15 10 5 steel depending on factors levels

Figure 1 presents distribution of hardness values HV1 and **figure 2** presents distribution of dimensional changes (contraction) of maraging 15 10 5 steel depending on factors levels.

The analysis of **figure 1** and **figure 2** indicates the next:

- Hardness values HV1 and dimensional changes of Maraging 15 10 5 steel are changed by variation of aging temperature and aging time.
- The highest hardness values are achieved by aging temperature of 500 °C.
- The increase of aging temperature above 500 °C and aging time above 4 hours significantly contributes the contraction of maraging 15 10 5 steel.

3 CONCLUSION

On the basis of performed testing of optimal aging temperature and aging time duration for Maraging 15 10 5 steel the following conclusions can be made:

- The obtained results indicate to essentially significant influence of aging temperature when compared with aging time duration on achieved hardness values, as well as on shortening of steel samples, which may be contributed to diffusive character of process of aging.
- The aging of Maraging 15 10 5 steel at 500 °C during 4 hours has a result optimal hardness value and dimensional change (contraction).
- During aging by above mentioned parameters hardness value of 510 HV1 and contraction of only 0,05 % have been achieved.
- The obtained, relatively high hardness value with accompanying dimensional changes (contraction) give argument to conclude that this steel can be successfully used up to maximal temperature of 500 °C.

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AUSTEMPERING HEAT TREATMENT EFFECT ON MECHANICAL PROPERTIES OF AISI O1 STEEL

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ABSTRACT

The paper describes results obtained with respect to mechanical properties, impact resistance of an AISI O1 steel alloy, after heat treatment by istothermal austempering, as well as comparison with the conventional quenching and tempering process. This study was conducted to understand the microstructural transformation kinetics with respect to time in the bainitic transformation region to verify the gradual increase of the bainite fraction until the total transformation. Microhardness and impact characterization was performed on the austempered and the quenched and tempered samples to evaluate the interaction between microstructure and impact toughness at the same hardness range for both processes. The results of this work will be discussed in this paper.

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COOLING ASPECTS OF VACUUM FURNACES

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ABSTRACT

The vacuum heat treatment with overpressure gas quenching is a fully accepted kind of heat treatment and firmly established due to its versatile use. Due to the very different furnace versions the corresponding tools and work pieces can be heat treated according to the customers' requirements. Even large forming tools can be successfully quenched and tempered in vacuum furnaces according to the internationally well-known standards like NADCA. The accomplishing and measuring or the cooling process are shown in details.

Keywords: vacuum furnace, hot work steel, die casting dies, gas quenching, NADCA specification, martempering

1 TEMPERATURE DIFFERENCE IN THE COMPONENT – REASON OF DISTORTION

On principle the cycle of each heat treatment consists of the sections heating up, holding and cooling. When heating up as well as during cooling temperature differences occur in the edges and in the core of the component. These temperature differences can not be avoided and are a reason for component stress which results into distortion. In principle this component distortion can be reduced considerably by slowly heating up and cooling. However, the microstructure, grain growth, hardenability of steel (quenching speed) and the economy demand a fast run of the processes.

1.1 Heating up

Heating up in the vacuum chamber furnace is effected by convection and radiation. In the lower temperature range the fast convective heating is made for a high temperature uniformity in the load. In the upper temperature ranges the heat transfer is dominated by radiation. One requirement for the heating is the lowest temperature difference possible within each component as well as within the whole load.

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Topic: A – Heat Treatment
Presentation type: Oral

1.2 Cooling down

The cooling process of the heat treatment must fulfil the requirements like hardenability of steel, "quenching as fast as necessary and as slowly as possible", uniform cooling of the load and keeping the temperature difference in the component as low as possible.
The realisation of these requirements leads to the main target: fully martensitic hardness structure with lowest distortion. The quenching speed influences the measurement strength considerably. An ideal situation would be a cooling medium with exactly that speed which reaches the sufficient hardness value. Because that would mean the lowest risk for distortion and cracks. Quenching pressure, quenching gas, cooling gas circulation speed, a.s.o. are parameters which make it possible to select the cooling speed in steps and to achieve absolutely reproducable cycles and results. The constructive design of the furnace influences an uniform cooling decisively.
The gas guidance and gas stream are important factors regarding low distortion values. From all furnace concepts the principle of through streaming with direction reversal has succeeded at the market. The programmable cooling enables a defined, either-way vertical or horizontal cooling of the load.

1.3 Martempering

In order to minimise the thermal tension between component surface and -core a "marquenching simulation" ("isothermal quenching") at a temperature higher than martensitic-start can be effected. The "marquenching simulation" lowers the distortion especially for big, geometrically complicated formed parts. For this "marquenching simulation" two thermocouples are fixed at one part of the load, one surface thermocouple and one core thermocouple.
The load is cooled down from austenisation temperature to "marquenching temperature", for example 400 °C.

The surface temperature goes down to this "marquenching temperature", but the core temperature is considerably higher at this moment, for example at 650 °C. To achieve that the surface temperature is not going down on a deeper temperature, the cooling is interrupted and the heating is switched on. The core temperature is adapted slowly to the "marquenching / surface temperature". As soon as the surface- / core adaptation has taken place cooling down to unloading temperature can be continued.

2 VACUUM-HEAT-TREATMENT OF HOT-WORK STEEL ACCORDING TO THE STANDARD OF THE AMERICAN AUTOMOTIVE INDUSTRY

For the heat treatment of die casting dies of hot-work steel material 1.2343 – X38CrMoV51 – H11the American automotive industry defines in their standards (NADCA-specification) the heat treatment process. Among other requirements the minimum quenching speed is measured from austenisation temperature to 538°C in a depth of the workpiece (Ts) of 16 mm which must be of 30 °C/min. After that a martempering process of 425°C is demanded where the surface (Ts) must be kept between the close temperature range of 440°C and 415°C until the difference between surface (Ts) and core (Tc) is smaller than 90°C. The proof must be delivered with a test component of 400 x 400 x 400 mm. At different companies the complete heat-treatment processes in a vacuum furnace with the useful space of 1000 x 1500 x 1000 mm, payload 2500 kg, maximum quenching pressure 13 bar was realised.

2.1 quenching speed and marquenching at the specimen 400 x 400 x 400 mm

At the specimen with the dimension 400 x 400 x 400 mm, single weight 566 kgs the cooling rate from austenisation temperature 1000°C to 538°C at the depth of component of 16 mm measured in the middle of the side surface (Ts) with an overpressure gas quenching of 13 bar nitrogen was more than 30°C/min. The demanded temperature between surface (Ts) and core (Tc) during the martempering phase could be realised.

3 VACUUM FURNACE WITH SEPARATE QUENCHING CHAMBER

The conventional vacuum furnace with 10 bar overpressure has limits regarding the cooling intensity. Traditionally the demanded cooling speed of low alloyed materials is reached with oil- or salt bath processes. One of the many disadvantages of these techniques are the bad distortion results.

The vacuum furnace with separate quenching chamber system *2 PLUS* offers an increase of quenching speeds compared to the conventional vacuum furnace. The principle of this system is the spatial separation of the heating- and cooling process. The processes run in single chambers which are separated by a closing mechanism serving as thermal barrier. A fully automatic loading car transports the load from one chamber to the other. By separating the heating- and cooling mechanism the cooling performance could be doubled compared to the conventional vacuum furnace.

The specimen 400 x 400 x 400 mm was tested (according to NADCA-specification) regarding the maximum achievable quenching speed in the vacuum furnace with the system *2 PLUS* with the useful space of 600 x 900 x 600 mm. At this process with 10 bar nitrogen overpressure quenching a maximum cooling speed from austenisation temperature to 538°C of more than 60°C/min in the surface (at a depth of 16 mm) was achieved.

4 SUMMARY

The modern furnace technology enables to do a low distortion vacuum heat treatment of several components and steels.

Bigger components can be hardened with low distortion and a high profitability. The advantages of new developments in vacuum heat-treatment with overpressure gas quenching can also be transferred to lower alloyed steels.

IN-SITU MONITORING OF VACUUM CARBURIZING

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ABSTRACT

The present paper describes development and testing of a new measurement method that enables non-destructive and in-situ monitoring of vacuum carburizing process. The principle of the method is based on monitoring the carbon diffusion during vacuum carburizing by the in-situ measurements of electrical resistance changes in the carburizing sample. The results of experiments that were performed in a laboratory vacuum furnace under a low pressure acetylene atmosphere (5 mbar) and at a temperature of 950°C are also depicted.

Keywords: vacuum carburizing, electrical resistance measurements, pure iron, acetylene

1. INTRODUCTION

Vacuum carburizing is a modern, very efficient and environmentally friendly process for case hardening of steels. The advantages of this technology compared to conventional gas carburizing process are a high reproducibility and uniformity of heat treated samples, clean sample surface after treatment, the absence of internal and grain boundary oxidation, smaller shape change of treated parts after quenching, easy carburizing of complex shapes such as thin or blind holes, low gas consumption and gentle working environment for people and earth [1, 2]. The carburizing is performed in vacuum furnaces under low pressure hydrocarbon gas atmosphere – usually pure acetylene or mixtures with other gases [1-3]. The desired case depth and corresponding concentration profile of carbon are obtained by diffusion of carbon atoms from the furnace atmosphere into the steel matrix actuated by cyclical changing the saturated (active) and the diffusion (passive) stage of the process [1-3]. At the constant carburizing temperature the diffusion of carbon can be supervised by controlling the duration of these two stages, where the optimal process parameters are usually pre-determined with vacuum carburizing simulation programs [2, 3]. However, this approach has many shortcomings needed to be overcome. For instance, the diffusion process during vacuum carburizing is very complex and therefore the desired depth and morphology of the carburized zone throughout the steel matrix is not easy to control. To avoid these problems and to take all advantages of this technology it is necessary to monitor both stages of the process in-situ and continuously. The present paper describes one of the possible ways to overcome the above problems and to fulfill the requirements of in-situ monitoring.

2 EXPERIMENTAL SETUP

We developed a nondestructive measurement method that enables identification and characterization of phenomena during vacuum carburizing of steels. The principle of the method is based on monitoring the carbon diffusion during vacuum carburizing by in-situ measurements of electrical resistance changes in the carburizing sample. For this purpose the unique laboratory device was set up (**Figure 1a**). It is based on a special measurement cell that enables in-situ electrical resistance measurements by four probe technique at high temperatures (up to 1100 °C) and in reactive atmosphere (**Figure 1b**). Moreover, it is also feasible to transform electrical resistance changes into an instantaneous microstructure of carburized zone by a mathematical-physical algorithm which will be presented. This algorithm was based on the assumption that the process of vacuum carburizing can be treated as a sequence of the partial reactions presented in the algorithm as parallel and/or serial time-dependent resistors in the electrical circuit.

3 RESULTS AND DISCUSSION

The vacuum carburizing experiments were performed in a laboratory vacuum furnace under a low pressure acety-

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lene atmosphere (5 mbar) at a temperature of 950 °C. With use of this measurement method the electrical resistance changes during vacuum carburizing of pure iron were acquired and analyzed (**Figure 2**). The results of monitoring the kinetics of carburized zones obtained by the novel measurement method were compared with kinetics obtained by metallographic analysis of carburized samples (**Figure 3**). The comparison shows that our measurement method gives enough qualitative and quantitative information about the proceeding of the reactions that take place during vacuum carburizing in the material, to monitor and to control the process online and non-destructive. Additionally, it is possible to: (i) distinguish between saturated and diffusion stage of the process, (ii) optimize the duration of each stage and the thereby time-ratio between this two stages and (iii) predict the progress of case depth into the matrix.

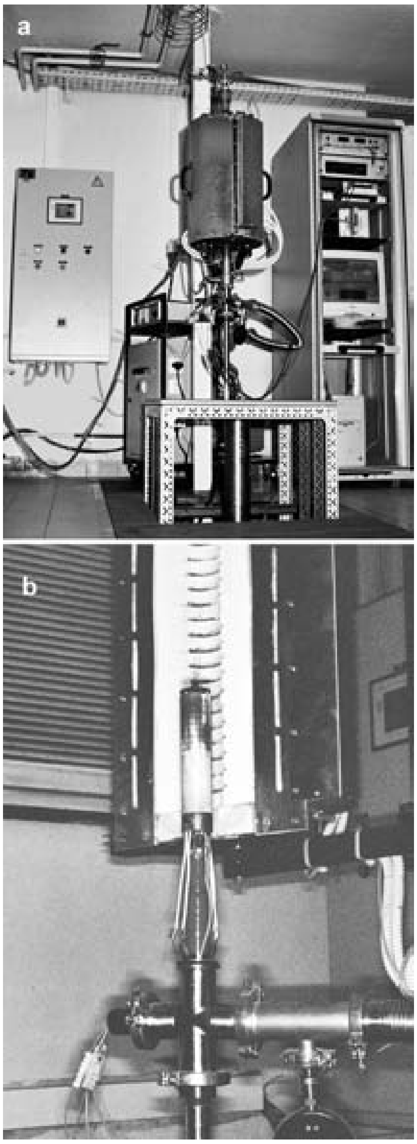

Figure 1: Laboratory device (**a**) and measurement cell (**b**) for "in-situ" electrical resistance measurements

4 CONCLUSIONS

In this research a measurement method enabling in-situ monitoring of electrical resistance on the basis of the four probe technique during vacuum carburizing under a low pressure acetylene atmosphere was designed and constructed. The design of the measurement method forms the foundation for the effective "in-situ" monitor-

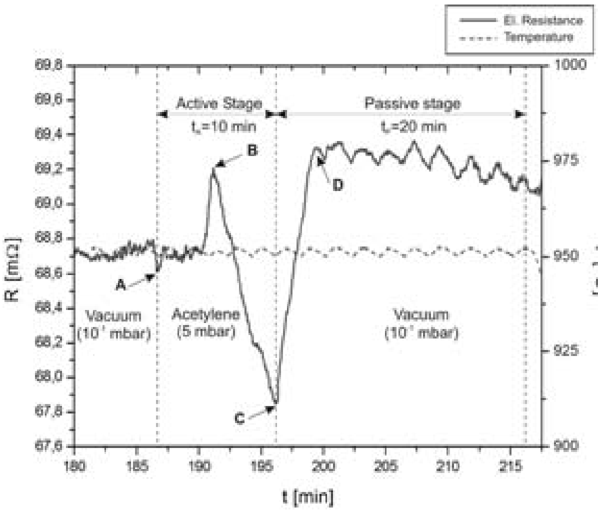

Figure 2: In-situ measurement of electrical resistance changes during vacuum carburizing of the pure iron at T=950°C

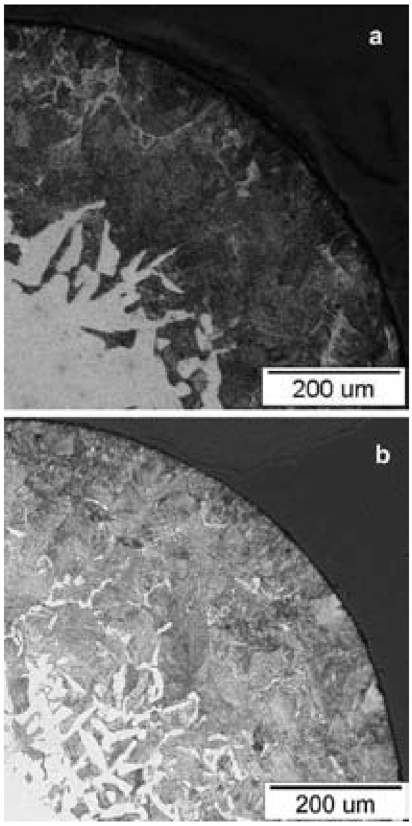

Figure 3: The progress and the morphology of carburized zone after active (**a**) and passive stage (**b**) of the process

ing and characterization of the phenomena during vacuum carburizing of steels.

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HEAT TREATMENT OF CORROSION RESISTANT TOOL STEELS FOR PLASTIC MOULDING

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1 INTRODUCTION

Steels for the processing of synthetic materials are the fastest growing market within the tool steel sector. Of these, corrosion resistant tool steels are used for those applications with the highest requirements not only on corrosion resistance, but also on surface quality and in many cases on toughness and wear resistance. Heat treatment parameters play a vital role in the adjustment of these properties in relation to each other and have to be selected carefully with regard to specific requirements.

2 MATERIALS OF INTEREST AND IMPORTANT HEAT TREATMENT PARAMETERS

Typical steels in use belong either to the group of 13%-Cr or to the 17%-Cr steels and usually contain C or C+N contents sufficient to achieve hardness levels between 50 and 60 HRC. Some examples are the standard grades X42Cr13, X36CrMo17 and X90CrMoV18 or the newer nitrogen alloyed grades M333 (~X30CrN14), M303 (~X30CrMoN14-1) and M340 (~X55CrMoN17-1) from Böhler Edelstahl.

As these steels are usually heat treated in vacuum-furnaces with gas quenching, there are three important parameters to be considered:

– austenitising parameters (mainly temperature, but for large workpieces also holding time)
– quenching rate (mainly based on quenching pressure and gas velocity)
– tempering temperatures (which have the largest impact on the steel's properties)

3 REQUIRED AUSTENITIZING PARAMETERS

Austenitising is the necessary precondition for any quenching process, but in the case of corrosion resistant tool steels, it requires special attention. Not only is the transformation of the ferritic bcc lattice structure into fcc austenite of importance here, but also the formation of a solid solution containing C (and N), whereby a minimum content of 13% Cr also has to be dissolved and evenly distributed within the austenite to achieve full corrosion resistance. This is especially important for the high carbon grades which require a minimum austenitising temperature significantly above A_{c1}. Too high austenitising temperatures on the other side can cause different types of heat treatment errors. Steels with lower carbon contents can suffer undesired grain growth, an effect that is more strongly pronounced in the highest quality electro-slag-remelted (ESR) or vacuum-arc-remelted (VAR) and nitrogen alloyed steel grades, which have the finest and most homogeneous carbide distribution in the soft annealed state. This aspect has to be taken into special account for large workpieces which require long holding times at austenitising temperature for an even temperature distribution in the whole workpiece.

As for the high C and C+N alloyed grades, too high austenitising temperatures lead to strongly increased amounts of retained austenite and go hand in hand with higher distortion and higher internal stresses. The usual process window for achieving optimum properties for these types of steels is between 980 and 1030°C, whereas the ideal austenitising temperature is strongly dependent on the size of the tool. Especially for the C+N alloyed grades the austenitising temperature should be decreased with increasing wall thickness to avoid the formation of grain boundary precipitations caused by the lower cooling rates achieved during quenching.

An additional aspect for this group of steels when used in protective gas and vacuum furnaces is the nitrogen pressure applied during austenitisation. The main reason for this topic is the increased N solubility in the austenite due to the high Cr content. While there exists the possi-

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Topic: A – Heat Treatment
Presentation type: Oral

bility to enrich the surface with nitrogen for a nitrogen casehardening by operating the vacuum furnace under a controlled (low) nitrogen pressure, no evidence of de-nitrogenation for nitrogen alloyed steels with 0,1 – 0,2 % N has been found.
Finally, it should not be overlooked that heating to austenitising temperature should be performed with one or two pre-heating steps around the phase transformation temperature to avoid internal stresses and corresponding distortion.

4 OPTIMUM QUENCHING RATE

Traditional oil or salt-bath quenching usually provided a very rapid quenching rate, especially in the temperature range of 800 and 500 °C. With the introduction of gas quenching in vacuum furnaces, the main concern initially was focused on the maximum workpiece dimensions that could be hardened to the core due to the lower quenching rate. With the steady increase of maximum quenching pressures and optimised gas flow today nearly all workpiece dimensions of these steels can be gas quenched, changing the focus towards internal stresses and distortion during quenching.
However, it is widely regarded that those quenching rates which produce the lowest internal stresses and distortion just by achieving full hardness in the centre regions are indeed the optimum. In doing so, quenching pressures (or velocities) are reduced to low levels (far) below the capabilities of the furnace. Unfortunately such slow cooling rates do not correspond with the highest toughness properties of the material, resulting in a workpiece that is rather brittle. Therefore cooling rates higher than the absolute minimum required to achieve full hardness are highly recommended in order to achieve the best material properties. Distortion can than be reduced by using helium as a quenching media, providing a more uniform quenching.

5 SELECTION OF TEMPERING PARAMETERS

The largest effect on the material properties can be achieved by the selection of tempering parameters. Material properties such as hardness, toughness and corrosion resistance, but also residual stresses depend widely on the tempering temperature, and therefore application specific combinations of these properties can be adjusted.
No tempering or very low (< 150 °C) tempering temperatures are uncommon as they result in high internal stresses with high material brittleness. With increased temperatures toughness properties can be improved significantly with only limited loss in hardness (still above 50 HRC) and almost no loss in corrosion resistance. When looking at toughness not as fracture toughness, but as impact toughness, which includes the energy necessary to create a crack, the degree of improvement, including the toughness level in general, highly depends on the microstructure (fine and uniform or coarse) and segregations, as well as on the cleanliness level. These factors strongly depend on the steel's composition (with advantages for low C and N alloyed grades) and on the production method (standard ingot casting versus premium quality electro-slag or vacuum-arc-remelted steel grades). Depending on the grade, typical tempering temperatures for this combination of properties vary between 200 and 400°C. Still it should be kept in mind, that this combination of properties does not only yield good toughness properties, but still bears high residual stresses and can therefore not be recommended for complicated workpieces, especially those with small radiuses and drastic changes of large and small dimensions. Another option, often used for pre-hardened and tempered material, is the use of tempering temperatures above 550°C, leading to high toughness in combination with low residual stresses. Unfortunately, hardness values are only in the range of typically 30 – 40 HRC. Corrosion properties strongly depend on the steel composition, with clear advantages shown for 17%-Cr, low C and for N alloyed steels.
In special cases the temperature gap between 400 and 550°C but especially between 510 and 550°C, is used for tempering operations. Sometimes tempering in this region is inevitable because of additional eroding or coating processes applied to the workpiece afterwards, sometimes this is performed to achieve a better compromise between deteriorating corrosion and toughness properties and reduced internal stresses for more complicated workpieces at high hardness levels. Thereby N alloyed low-C grades exhibit a significantly better performance, as the loss in corrosion resistance is less pronounced and shifted towards higher temperatures.

6 CONCLUSIONS

To achieve the best properties for tools and dies made out of martensitic corrosion resistant steels, several aspects of heat treatment should be taken into careful consideration:
- Austenitising temperatures should be kept within a very small range (usually 980-1030 °C) for the whole workpiece.
- The ideal austenitising temperature is strongly depending on the size of the tool. Especially for the C+N alloyed grades the austenitising temperature should be decreased with increasing wall thickness to avoid the formation of grain boundary precipitations caused by the lower cooling rates achieved during quenching.
- Quenching in vacuum furnaces should be mostly uniform and preferably at higher quenching rates to avoid the precipitation of grain-boundary carbides, especially for larger workpieces.

– The selection of the tempering temperature highly depends on the desired properties, and thereby it should be kept in mind that good toughness properties at medium temperatures (200-400 °C) usually correspond with still high residual stresses, especially in complicated workpieces. High Cr-low C and N alloyed steels offer the possibility of increasing tempering temperatures up to 510-550 °C while still keeping acceptable levels of corrosion and toughness properties.
– High quality electro-slag or vacuum-arc-remelted and N alloyed steels offer the potential for better properties, but only if the heat treatment is performed with the optimum combination of parameters.

HEAT TREATMENT OF HOT WORK TOOL STEELS – SIZE MATTERS!

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ABSTRACT

This article is intended to draw attention to the problems as well as some solutions manufactures face when using hot work tool steels. Remarkable advances have been made in the performance of these steels in the last decade. Material properties have been improved with increasing cleanliness and homogeneity and a fuller understanding of heat treatment and service conditions. These advances have led to greater loading as well as larger and larger tools and dies. This increase in size in particular brings with it significant challenges to steel making, heat treatment and tool making. The normal material properties of hot work tool steels are discussed along with a description of typical heat treatment parameters. Attention is focused on the very narrow range of treatment parameters that gives desirable material properties.

1 MATERIAL SELECTION AND IMPORTANT HEAT TREATMENT PARAMETERS

Typical hot work tool steels belong to the group of 5%-Cr- steels and usually contain a C-content between 0,3 to 0,5 %C. These steels are additionally alloyed with varying contents of Mo, V and in some cases with Co or W to achieve a secondary hardening effect for the necessary hot hardness. Depending on the application, the working hardness for these steels lies between 40 to 48 HRC for aluminium die casting and 50 – 54 HRC for some forging tools, squeeze casting and aluminium extrusion dies.

The most common heat treatment for hot work tool steels today is vacuum hardening with high pressure gas quenching. The parameters of the hardening process for an optimum of properties need to be kept in a very close range.

The main austenitising temperature range for this group of steels lies generally between 1000 to 1050 °C, but depending on the exact alloying content and additionally on the method of production – conventionally melted, ESR (electro-slag-remelting) or even VAR (vacuum arc remelting) – the optimal temperature range for the chosen steel grade may vary only within such a close range as 20 °C.

The tempering cycle also influences all other mechanical properties, most importantly toughness, yield strength and ductility. The tempering range lies well above 500 °C, but the optimal combination of properties – again depending of the exact alloying content – are often achieved only in a small range of ±20 °C for certain applications. The content of retained austenite of these steels requires a minimum of two separate tempering steps, better is a 3- or more step tempering.

2 CHALLENGES AND SOLUTIONS

The first challenge that has to be dealt with is the steel quality being used. The excellent properties now available from grades developed in the last decade can be achieved when heat treating pieces with a small cross section. Large components require more thought, however. Naturally large components need large ingots and it is elementary to steel making that large ingots contain more undesired segregations, resulting in microstructural differences from core to surface. These segregations can noticeably reduce mechanical properties such as toughness. High quality grades produced by ESR or VAR are required to achieve even the minimum of the specified properties.

Heating thick cross sections to austenitising temperature is limited by the steel's thermal conductivity; so the total heat treatment cycle (austenitising, quenching and tempering) can take 50 hours or more of furnace time. This can mean that the core requires hours to reach tempera-

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ture while the surface and thin sections suffer undesired grain growth in the mean time. Controlled heating steps are necessary to ensure that core and surface are exposed to the right temperatures for the right length of time.

The next issue to be dealt with is the problem posed by increasing cross sections on the next step in heat treatment; quenching. Normal practice is to keep quenching as gentle as possible to reduce the risk of cracking and keep distortion to a minimum.

When the part dimensions are 50mm or less, most hot work tool steels have sufficient hardenability that complete transformation to martensite is possible with mild quenching rates. The toughness of hot work tool steels in particular is markedly reduced if this transformation does not take place fully. As cross sections increase to 200 mm and beyond even oil quenching is insufficient for complete transformation in the core. To achieve high toughness, quenching must then be done as quickly as possible. The importance of this can be seen when comparing the results of Charpy impact testing performed on samples quenched at different rates and media. Increasing the nitrogen pressure from 3 bar to 4.9 bar increased toughness by 13%. When the pressure was increased further and the nitrogen replaced with helium, the results were 30% higher than the 3 bar N_2 quench. Surprisingly in this case the increase in toughness was also coupled with an increase in hardness by up to 2 HRC.

The need for rapid quenching does come with a price. Distortion will, of course, be more severe as will the risk of quench cracks. Step quenching, controlled by the same accurate thermal measurements needed for heating, can help control the temperature difference between surface and core thus reduce these risks. Tool design and pre-machining also needs to be considered in this regard. Redesign and careful pre-machining of stress raising areas will further reduce the potential for cracking.

Another consequence from the extra distortion is the fact that machining tolerances for hard machining must be raised for large tools and dies. Distortion is a volume effect after all. Another unavoidable point is the need for more hard-machining. Investment in equipment and greater understanding of hard-machining processes is necessary unless new steels with better hardenability become available on the market in the future.

The tempering of a hardened piece is of course a critical step in any heat treatment cycle. As with austenitising, the properties of the tool are sensitive to both time and temperature. The same temperature control for austenitising is also required for tempering. It must also be remembered that the maximum hardness available for these steels corresponds to the lowest toughness. This lack in toughness is further worsened when combined with the loss from incomplete hardening.

Although care will ensure the best practical results, there is no possibility to avoid differences in hardness completely. The standard specifications for an acceptable hardness range must be increased accordingly or results should only be taken from critical areas.

3 CONCLUSIONS

This paper concludes with suggestions and recommendations on how to solve some of the problems faced. These include:

- Very homogeneous grades of steel are required for large tools and dies made from hot work tool steel.
- Heat treatment equipment, including tempering furnaces, with high accuracy in the temperature distribution – while fully loaded – and the possibility of temperature control by thermocouple should be used.
- Holding steps during the preheating and close to the austenitisation temperature should be used and adjusted to the actual tool or die.
- Quenching should be performed uniformly with the highest possible quenching rate.
- Machining allowances need to be increased as a result of increased distortion.
- Special care must be taken with pre-heat treatment machining to avoid stress raisers and thus reduce the risk of cracking.
- Tolerances for hardness values must be adjusted due to the lower hardenability of larger sections compared to smaller sections.

DIRECT METHOD OF TRACING OF OXIDATION IN METALS AND ALLOYS

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ABSTRACT

The controlled process of high temperature oxidation can be used for creation of protective coatings or for dispersion strengthening of metallic matrices. Because of complexity and aggressive environment in which the high – temperature oxidation occurs there are the problems of non-destructive and in-situ monitoring of the process in both, the laboratory and in industrial practice. In this paper the device and the measurement technique that enable non-destructive monitoring of high – temperature oxidation of metals and alloys on the basis of "in-situ" measurements of electrical resistance changes are presented.

Keywords: High-temperature oxidation, internal oxidation, non-destructive monitoring, electrical resistance measurement

1 INTRODUCTION

Practically all metals and alloys are thermodynamically unstable and oxidize at high temperatures in oxygen containing atmosphere. The products of this reaction can grow as external oxide layers on surfaces, or as discrete oxide particles which precipitate in the interior of metal matrices. In the first case, we talk about the well known external oxidation and the other case presents the so-called internal oxidation. Both processes are very important, because they determine properties and applicability of metallic materials. In general, external and internal oxidations are undesired, because they cause deterioration of mechanical properties and decomposition of metallic material. On the other side, the controlled process of external oxidation can result in creation of protective coatings and internal oxidation can be used for dispersion strengthening of metallic materials. In order to monitor and control the course of high-temperature oxidation several destructive and non-destructive methods can be applied [1]. In some cases, monitoring of the process is discontinuous and is performed by microstructural examinations of oxidized samples which include measuring of thicknesses of external scales and internally oxidized zones. However, the high temperature oxidation is more often monitored continuously by controlling the gas consumption in the annealing atmosphere or by monitoring the changes of some physical properties of the samples by different methods of thermal analysis.

The microstructural changes during high-temperature oxidation in the metallic materials have also significant influences on their electrical resistivity. Formation of external oxide scale causes the increase of electrical resistivity due to thinning of the conducting part of the metallic sample. On the other hand, the internal oxidation of alloys decreases resistivity due to removal of the solute atoms from the matrix crystal lattice with precipitation of oxides [2]. The "in-situ" monitoring of high-temperature oxidation of metallic materials with electrical resistance measurements was also the main topic of this research. With the novel measurement method we monitored the high-temperature oxidation of Ag-Sn (2 at. % Sn), Cu-Al (1.25 at % Al), Ag (99.99%) and Cu (99.99%).

2 EXPERIMENTAL SETUP

The high-temperature oxidation experiment with "in-situ" electrical resistance measurement is based on the four probe method. The measurements were performed in the laboratory device consisting of a three-zone tubular electro-resistance furnace, vacuum system and the measurement cell placed in the furnace inside the retort. The cell consists of the ceramic cylinder where the four prismatic contacts made from pure platinum are fixed. The outer contacts (current contacts) are

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designed for charging the sample with current and the inner contacts (voltage contacts) enable the measurement of the voltage change in the sample. In addition, the temperature changes were measured at the central part of the sample with a Ni-NiCr thermocouple with an outer diameter of 1 mm and accuracy ± 1.5 °C. The experiments of high-temperature oxidation were performed at 800°C and different oxidation times. The thickness of the external scale as well as of the subscale (internal oxidation zone) were examined on the transversal cross-section of metallographic samples with an optical microscope, Nikon Epiphot 300, equipped with a system for digital quantitative image analysis (Olympus DB12 and software program Analysis).

3 RESULTS

The results of monitoring of high temperature oxidation of selected alloys and metals (Ag-Sn, Cu-Al, Ag and Cu) can be divided, as a consequence of proceeding of the reactions during high temperature oxidation into the following characteristic groups: (i) monitoring of dissolution of oxygen in the pure Ag matrix (ii) monitoring of the internal oxidation in the Ag-Sn alloy, (iii) monitoring of the external oxidation of the pure Cu metal, and (iv) monitoring of the simultaneous external and internal oxidation of Cu-Al alloy. The results of "in-situ" electrical resistance measurements during high temperature oxidation of Cu and Cu-Al alloy in air atmosphere (10^5 Pa) and at 800°C are presented in Figure 1. The resistance curves, *R(t)*, show the electrical resistance changes of the samples, and the temperature curves, *T(t),* indicate the actual temperature during experiments. The curves can be divided into two regions. The first region (left from the point A) presents the stage of heating of the sample in the vacuum up to the desired isothermal annealing temperature. The second region (right from the point A) comprises the proceeding of the reactions during process of high temperature oxidation of selected metals or alloying systems.

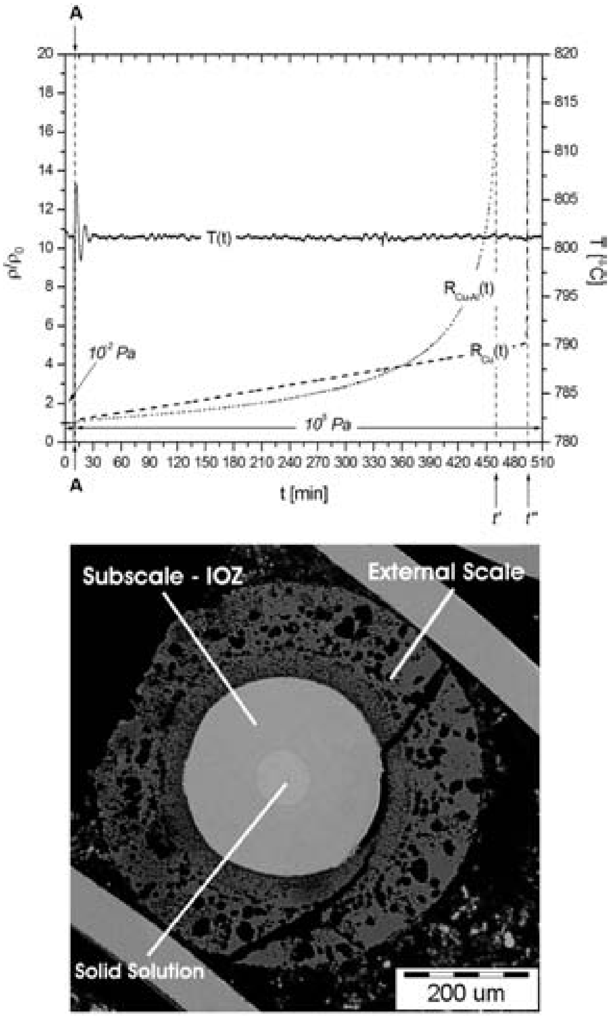

Figure 1: Change of electrical resistance during annealing of Cu and Cu-Al alloy at 800°C and Microstructure of partially high – temperature oxidized cylindrical sample of Cu-Al (1.25 at.% Al) alloy at T=800°C and t=240 min.

4 CONCLUSIONS

In this paper the measurement method enabling "in-situ" measurements of electrical resistance on the basis of four probe method during annealing of metals and alloys in oxidizing atmosphere was described. The developed measurement method has the following advantages:

1. It enables accurate, reliable and repeated measurements up to 1100°C even after long duration of annealing experiments in vacuum, inert or reactive atmosphere.
2. It monitors continuously and nondestructive changes of the electrical resistance in the whole volume of the sample with possibility to identify the start and the end of the process.
3. Its resolution and response give us also an opportunity to detect the distinctions between fundamental reactions that proceed during high – temperature oxidation of metallic materials as for instance (i) growth of the external scale, (ii) growth of subscale – internal oxidation and (iii) dissolution of oxygen in the metal matrix.

The use of developed measurement method for "in-situ" monitoring and characterization of high – temperature oxidation presents one of possible tools that will contribute to a better fundamental understanding of the phenomena that occur during high – temperature oxidation of metallic materials.

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HEAT TREATMENT OF TOOL STEELS

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Heat treatment represents a fundamental step in the production process of tool steels. The possibility to obtain very drastic changes in the final properties by modifying few parameters evidences the crucial role of hardening and tempering to which these specific class of steels are subjected. This role becomes even more important in as-cast products (e.g. high speed steels and white cast irons for hot rolls) where no thermomechanical working (forging and/or rolling) is provided. The panorama of heat treatment is considerably wider considering surface engineering as a natural extension of heat treatment. Nitriding, PVD coating and their combination in duplex systems are actually employed in a wide range of applications including forming, cutting tools and dies. In the same way, cold and deep cryogenic treatments (DCT) can be considered as an implementation of conventional treatments. The production of tools with improved performance is strictly correlated to the knowledge of relationships between steel metallurgy/microstructure and hardness, mechanical properties, toughness and wear resistance. From this viewpoint the intense research activity in Europe and in the world was decisive to promote the selection of the best heat treatment solution for the specific application considered. On the other side, the development of new technologies allowed to produce components with higher reliability, often associated with a reduction in the environmental impact.

The aim of this paper is to review the principal results obtained in the last years in the laboratory of metallurgy of the University of Trento. Many efforts have been spent to evaluate the correlations between heat treatment parameters, microstructure and properties of cold work, hot work and high speed steels in a wide range of applications. Studies have been devoted to the comprehension of the basic damage mechanisms of tools and dies. The thermal cracking (heat checking) occurring in hot work tool steels for casting/forging dies but even in high speed steel work rolls has been studied by means of a customary thermal fatigue test developed in our laboratory. The corrosion by molten metal in die casting dies has been studied by immersion in liquid aluminium. The tribological properties of dies during aluminium hot extrusion was considered by means of specific simulation test. The results will be presented paying specific attention to the influence of heat treatment and surface engineering to the cited properties.

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CRYOGENIC TECHNOLOGY

THE EFFECT OF SOME HEAT TREATMENT PARAMETERS ON THE PROPERTIES OF AISI D2

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ABSTRACT

AISI D2 is usually vacuum hardened then tempered several times. Deep cold treatment between the hardening and tempering processes could improve the final properties. Hardened blocks of AISI D2 were then subjected to various combinations of single and multiple tempering steps and deep cold treatments. The results showed that for all austenitising temperatures a single deep cold treatment followed by a single tempering cycle was sufficient to reduce the retained austenite to below 1%. The colder the cold treatment temperature and the longer the treatment time, the higher the hardness values that could be achieved. The deep cold treatment did not affect the fracture toughness or distortion.

Keywords: deep cold, AISI D2, vacuum hardening, properties

1 INTRODUCTION

It has long been recognised that deep cold treatment can reduce or eliminate retained austenite in many steels and thus minimise the need for tempering [1]. However, there is some uncertainty about its effects on the final properties. This paper reports on a project to optimise the heat treatment of AISI D2 and determine the effects of deep cold treatment on the final properties of the steel.

2 HARDENING

The first step in the heat treatment of AISI D2 is hardening. The samples were austenitised at 1020, 1050 or 1070 °C. At the austenitising temperature the sample temperature was largely homogeneous, but some small differences in temperature between positions at the center and on the edge were detected during the cycle. As would be expected, the retained austenite increased with increasing austenitising temperature, as shown in **Table 1**. There was also a significant positional dependence of the retained austenite for a given austenitising temperature. Dilatometery studies suggested that this was caused by position-dependent martensite start temperatures.

3 TEMPERING AND CRYOGENIC TREATMENT

Various combinations of cryogenic (-90, -120, 150 °C for 25 minutes and -90, 150 °C for 24 hours), tempering (520 and 540 °C) and austenitising (1030 and 1070 °C) treatments were investigated. The effects of the various cryogenic treatments are summarised in **Figures 1 and 2**.

All the cryogenic treatments reduced the retained austenite present in the sample. At the higher austenitising temperature (1070 °C) this advantage could be retained by single tempering, so that after any cryogenic treatment followed by a single temper samples retained less than 1% austenite. A triple treatment produced no further improvement in retained austenite. Examination through a light microscope revealed no microstructural differences between the cryogenically and non -cryogenically treated samples after tempering.

Charpy impact testing at room temperature showed that there was no significant difference in fracture toughness between the cryogenically and non-cryogenically treated samples. The possibility that cryogenic treatment might cause distortion was also investigated. Careful measurement of the samples in all three planes showed that although there were some differences in the distortion produced during the individual treatment steps, once the retained austenite levels were equalised the distortion was also equalised. Tempering alone and -90 °C cryo-

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Topic: B – Cryogenic Technology
Presentation type: Oral

Table 1: Retained austenite and martensite start temperature variation with position

Austenitising temperature	1020 ºC		1050 ºC		1070 °	
Position	RA (%)	M_s (ºC)	RA (%)	M_s (ºC)	RA (%)	M_s (ºC)
Core	7	250	9	240	15	230
Surface					16	220
Edge					17	216

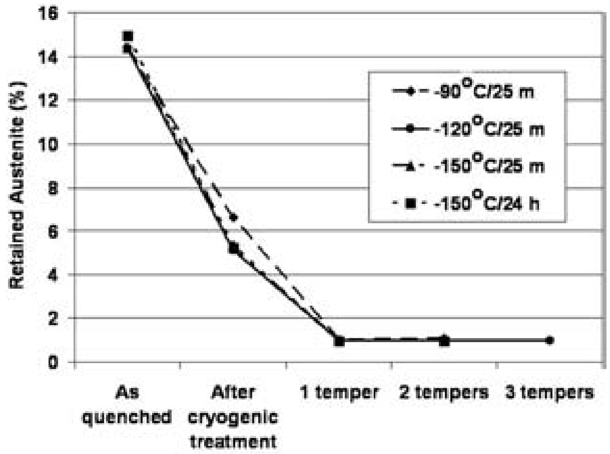

Figure 1: The effect of deep cold treatment on retained austenite

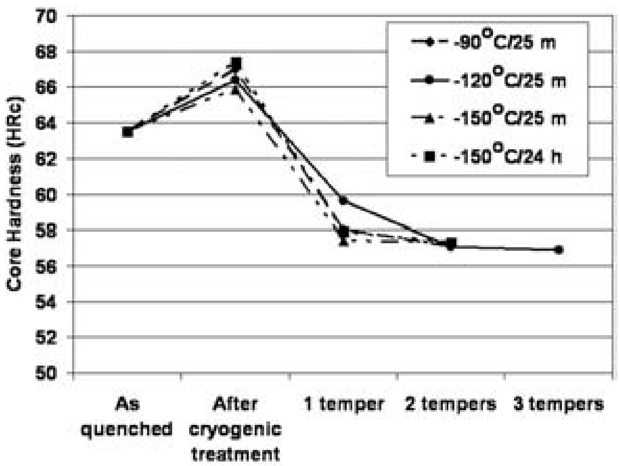

Figure 2: The effect of deep cold treatment on hardness

genic treatment required a total of three steps to reach this condition, but cryogenic treatment at -120 °C and below required only one additional step. This testing also showed that a 24 hour treatment at -150 °C gave exactly the same final result in terms of distortion as a 25 minute cryogenic treatment.

4 CONCLUSIONS

In AISI D2 the amount of retained austenite present can vary according to position. This may be due to local variations in the martensite start temperature that may, in turn, be caused by variations in thermal history.

Deep cold treatment at -120 °C and below allows the processor to reduce post hardening treatment to a short cryogenic treatment followed by a single temper.

The shortened cycle does not affect microstructure, fracture toughness or distortion and may slightly increase the final hardness for high austenitising temperatures.

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EFFECT OF DEEP-CRYOGENIC TREATMENT ON HIGH SPEED STEEL PROPERTIES

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ABSTRACT

Advantages of deep-cryogenic treatment over standard heat treatment of high speed steels for the purpose of obtaining better exploitation properties are quoted in an increasing number of scientific papers to be found in literature. This paper deals with the most important improvements of high speed steel properties achieved by using deep-cryogenic treatment. The effect of deep-cryogenic treatment on impact and fracture toughness, on erosion wear resistance and on the material microstructure has been tested on test pieces made of PM S390 MC high speed steel. A set of test pieces was heat treated by conventional methods (hardened and three times high temperature tempered), and the other set was deep-cryogenic treated. From obtained results it can be concluded that the application of deep-cryogenic treatment results in significantly higher wear resistance of high speed steels, but no significant improvements in toughness have been noticed.

Keywords: high speed steel, deep-cryogenic treatment, toughness, fracture toughness, wear resistance

1 INTRODUCTION

Standard (conventional) heat treatment of high speed steel tools consists of austenization in a protective medium, of quenching and of obligatory multiple high temperature tempering. During the tempering process, conditioning of highly alloyed retained austenite takes place, together with the precipitation of carbides and the creation of secondary martensite. With deep-cryogenic treatment, immediately after the tool quenching has been completed, practically all highly alloyed retained austenite is eliminated, and potential spots for the nucleation of fine ç-carbides (in dimensions less than 0.006 μm) are created in martensite. According to literature[1-3], these carbides contribute to the increase in toughness and wear resistance. These facts, together with some hypothesis made in this paper, have been investigated by carrying out heat treatment by hardening and tempering with and without deep-cryogenic treatment on test samples made of PM S390 MC high speed steel.

2 DESCRIPTION OF TESTS AND DISCUSSION OF RESULTS

Test samples made of PM S390 MC high speed steel are heat treated with varying temperatures of austenization (1300, 1180 and 1230 °C). After quenching, a number of test samples was tempered (twice at 540 °C/2 hours and once at 510 °C/2 hours), while other samples were submitted to deep-cryogenic treatment at a temperature of -196 °C/25 hours, and then tempered at 540 °C/2 hours), **Figure 1**.

Micrographic testing of heat treated test samples was carried out using a light microscope and a scanning electron microscope together with hardness tests, dilatometric tests, toughness test, X-ray tests and wear resistance tests. The share of present phases was determined by a quantitative analysis, and fracture toughness was calculated on the basis of the following expression [2]:

$$K_{Ic} = 1.363 \cdot \left(\frac{HRC}{HRC-53}\right) \cdot \left[\sqrt{Ed_p}\,(f_{carb})^{-(1/6)} \cdot (1+f_{aust})\right] \ [\text{MPa m}^{1/2}]$$

Impact toughness was tested on a special instrument for brittle materials. While impact toughness was tested at all three temperatures of austenization, fracture toughness was tested only at austenization temperature of

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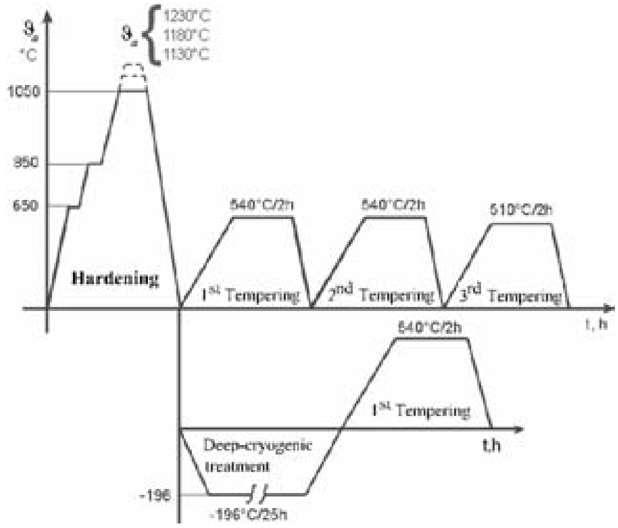

Figure 1. Graphical presentation of heat treatment of PM S390 MC high speed steel test samples

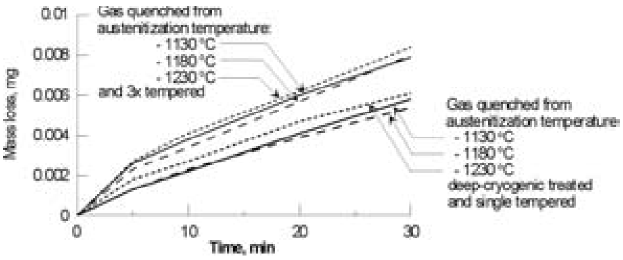

Figure 2. Mass loss by erosion wear of PM S390 MC high speed steel test samples after standard heat treatment and deep-cryogenic treatment

1230 °C. Some test results of impact and fracture toughness are presented in **Table 1**. The application of deep-cryogenic treatment resulted in reduced impact toughness values compared to the standard heat treatment (for austenization temperature of 1230 °C), and fracture toughness remained practically unchanged. Obtained results indicate that there is no possible correlation between impact and fracture toughness.

Table 1: Comparison between calculated fracture toughness and tested impact toughness for PM S390 MC high speed steel test samples submitted to different heat treatments.

Austenized at temperature of 1230°C/2min + ...	Impact energy, J	K_{Ic} (MPa$^{1/2}$)
+quenched in N_2 + tempered 2x (540 °C/2h) + tempered 510 °C/2h	0.2032	8.00
+ quenched in N_2 + deep-cryogenic treated -196 °C/25 h + tempered 540 °C/2h	0.1434	8.05

Figure 2 presents test results of PM S390 MC high speed steel erosion wear resistance. These results show undoubtedly that the application of deep-cryogenic treatment results in higher erosion wear resistance, which is in accordance with literature [1,3]. This result is a consequence of significantly higher amount of carbides in the tested steel microstructure in the case when deep-cryogenic treatment is applied, which was determined by X-ray diffraction.

3 CONCLUSION

This research has determined a positive effect of deep-cryogenic treatment on the increase in erosion wear resistance of PM S390 MC high speed steel. This is a consequence of significantly higher amount of carbides in the tested steel microstructure in the case when deep-cryogenic treatment is applied than in the case of standard heat treatment without deep-cryogenic treatment. As for other properties, no significant improvements have been obtained. At the same austenization temperature, the application of deep-cryogenic treatment has resulted in the same value of fracture toughness, and impact toughness has been reduced, which is probably a consequence of higher amount of carbides in the steel microstructure.

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DEEP SUB ZERO PROCESSING OF METALS AND ALLOYS – PART I

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ABSTRACT

Deep Sub Zero Processing of metals and alloys (DSZ) is a deep stress relieving technology. This paper in two parts will enlist the basic process of Deep Sub Zero processing as adapted by us in this research work and also discuss the results of the process on various materials. Whenever any metal or alloy is subject to any manufacturing process, be it machining, forming, stamping, hardening, grinding, polishing, wire EDM, etc., they are subject stress. This stress manifests itself in the nature of defects in the crystal structure of the materials. The most commonly observed defects are in the form of vacancies, dislocations, stacking faults, etc. As the level of stress increases, the density of these defects increases, leading to an increase in the inter atomic spacing. When the distance between atoms exceeds a certain critical distance, cracks develop and failure takes place.

The third law of thermodynamics states that Entropy is zero at Absolute Zero temperature. DSZ processing uses this principle to relieve stresses in the material. The materials are subject to extremely low temperatures for prolonged periods of time leading to the development of equilibrium conditions. This leads to ironing out of defects in the material and also the attainment of a minimum entropy state. Grain shape and size gets refined and made uniform. Defect elimination takes place and inter atomic distances is reduced. When the material is brought back to room temperature, the defect level reflects an equilibrium concentration. Compaction of the crystal structure, leads to much superior resistance to abrasive, adhesive and erosive wear and enhances fatigue strength and resilience.

1 DSZ PROCESSING

DSZ Processing is carried out at temperatures of around –185 to –190 °C, which is close to the liquefaction temperature of nitrogen. Liquid nitrogen is used as a refrigerant and cooling medium and the processing is carried out in a controlled manner. In hardened steels, the DSZ processing is carried out prior to the grinding and after the complete hardening and full tempering is completed, and there is no restriction on the time between finish tempering and DSZ Processing. For non-ferrous materials the DSZ Processing is carried out on the finished products. In the case of Age hardening and PH hardening materials the DSZ Processing is usually carried out after the solutionising is completed.

Deep Sub Zero Processing entails subjecting the materials to the low temperatures in a controlled manner. The metals and alloys are cooled from Room temperature to the low processing temperature in a very slow controlled manner at a cooling rate of between 15 to 45 °C/ hr. Once at that temperature, it is soaked for periods upto 24 hrs. Once the soaking is complete, the material temperature is slowly raised back to room temperature over a period of around 24 hrs. Once at the room temperature the materials are subject to elevated temperatures of around 150 °C in a controlled manner and for different soaking times.

2 RESULTS & DISCUSSIONS

Two main benefits accrue as fallout of DSZ Processing in Ferrous alloys. Firstly, retained austenite is converted to martensite. DSZ Processing merely continues the conversion initiated by heat treatment, whereby almost all the retained austenite is converted to martensite. Secondly, fine eta (?) carbides are precipitated. These fine particles act as fillers, and along with the larger secondary carbides, they form a denser, more coherent and tougher matrix in the steel. This leads to a more wear re-

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sistant material with a uniform hardness. A fallout of this processing in a better surface finish devoid of grinding marks and which is better resistant to premature failure. The surface energy of martensite is higher than that of austenite due to their different atomic structures. In potential adhesive wear situation, the martensite is less likely to tear out as compared to austenite. The probability of wear particles forming in steels in which austenite has been transformed to martensite is less than for a steel containing excess retained austenite. The adhesive wear coefficient is reduced and the wear rate is reduced. In the case of DSZ treated steel, some of the cold weld junctions, which would break off and form a wear particle if the steel were untreated, simply shear at the junction interface. There is a reduction in the stress concentration levels in the material and the relaxation of stresses results in the enhancement of mechanical properties of the various metals and alloys.

This paper discusses the X-ray diffraction results of the effects of deep sub zero processing on the stress levels in various metals and alloys, pin on disc wear results on various materials as well as microstructural changes observed in various metals and alloys. Part II of the paper deals with specifics observed in the two different steel grades when subject to this processing operation.

DEEP SUB ZERO PROCESSING OF METALS AND ALLOYS – PART II

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ABSTRACT

In this paper, the results of the tests carried out on two different grades of high speed steels which were subject to the deep sub zero processing as described in the earlier part. The two steels were conventionally heat treated in a salt bath furnace to get the basic hardness as recommended for the two steel grades. The steel grades chosen were AISI M2 and T42 grades.

The M2 steel samples, 10mm dia and 25 mm long were Austenitised at 1210 °C and tempered thrice at 560 °C to achieve a hardness of 64 – 65 HRC. The T42 steel pieces, 12 mm x 12mm x 25 mm were Austenitised at 1240 °C and tempered thrice at 560 °C to achieve a hardness of 66 – 68 HRC. The heat treated pieces were then subject to DSZ Processing using the procedure described in Part I of this paper. The soaking time for the DSZ process was varied between 8 and 24 hrs to observe the effect of the additional soaking time on the microstructure.

1 TESTING PROCEDURE

The samples were subject to optical, SEM, TEM and x-ray micrography to see the results and also to quantitatively note the changes that are taking place in the steels during the DSZ processing. Analysis of the micrographs shows that there is a lot of relaxation of stresses taking place in the microstructure due to the DSZ processing. The coarser carbides tend to get rounded and also smaller unstable carbide particles are absorbed and agglomerated in to form larger more stable carbides, which can resist the abrasive forces better.

2 RESULTS & DISCUSSIONS

There is quantitative evidence of the transformation of Retained Austenite into Martensite and this is exhibited in the displacement of the carbide particles relative to each other. The particulate distances are increased and this is evidence to the increase in volume that accompanies the phase transformation. In addition there is a distinct change in the dispersion of carbide and also in the density of carbide suggesting a dissolution of carbide into the matrix during the DSZ Processing.

In addition, there is a precipitation of tertiary carbides taking place during the DSZ processing. These carbides are spherical in nature and there is a pronounced high concentration of Fe in these carbides and they have a mixed chemistry indicating complex carbides, which are not normally associated with High speed steels.

The results are compared with and without post tempering on the DSZ processed samples and the results show a distinct difference in the microstructure with and without the low temperature tempering. The results are also evidenced in the wear results and the wear patterns show a distinct optimization, which takes place in the mirostructure and this is borne out in the results based on the soaking period with a minimum soaking period being necessary for the irreversible results to take place.

The results in the M2 grades also show that there is a fragmentation of the inclusion particles taking place during the deep sub zero processing and the formation of smaller particles also suggests a lowering in the relative stresses in the material as smaller inclusion sizes lead to a better wear resistance. This is also borne out in the wear studies on these particles and the results of all the experiments are discussed herein.

Microstructural evidence to describe the various changes, x-ray analysis of microconstituents in the matrix and also results of wear analysis and possible mechanism to explain the observed changes will be discussed in the paper.

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SURFACE ENGINEERING

CORRELATION BETWEEN SPUTTERING CONDITIONS AND GROWING PROPERTIES OF (TiAl)N/AlN MULTILAYER COATINGS

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ABSTRACT

Multi-layered coatings indicates better thermo/mechanical properties than mono layered coatings in cutting tool applications. In this study, TiAlN/AlN multilayer coatings were produced by diffirent magnetron process parameters. Partilally, magnetron discharge power, bias voltages and N_2 flow rate changes growing morphology and surface quality. The microstructure and growing morphologies correlated to sputtering conditions in a range of TiAlN/AlN multi-layer coatings with nanoscale bi-layer period thickness investigated carefully in order to identify the excellent properties of the coatings. The crystal structure of multilayer films was determined by XRD analysis. Surface and fracture surfaces were characterized by scanning electron microscopy (SEM).

Keywords: cutting tool caotings, magnetron sputtering PVD, multi layer thin films, SEM, XRD

1 INTRODUCTION

Hard coatings deposited by physical vapor deposition (PVD) techniques have been developed from monolayer coatings (e.g., TiN, TiAlN, etc.) to multi-layered coatings (e.g, TiAlN/ TiN, TiAlN/ CrN, TiN/CrN, CrN/NbN, TiN/ZrN, etc.) for improving performance in the field of cutting tools and molds. Multi-layered coatings indicates better mechanical and thermomechanical properties than mono layered coatings. Magnetron sputtering methods have been widely used for control of process parameters and growing morphologies of multilayer microstructures. Titanium aluminum nitride (TiAlN) which produced by PVD was developed in the late 1980s as alternative to TiN films for dry and high-speed operations [1,2]. TiAlN exhibits superior oxidation resistance and abrasion resistance as compared to TiN films [3,4]. It has been reported that the oxidation behavior of TiAlN is mainly controlled by Al content [3]. The properties of the TiAlN coating make it suitable for high temperature cutting operations with minimum use of lubricant or dry machining. TiAlN is used successfully to machine titanium, aluminium and nickel alloys, stainless steels, alloy steels, Co-Cr-Mo and cast irons. TiAlN is also used to protect dies and moulds that are required to operate at high temperatures such as those in medium and hot forging and extrusion industries. However, newly growth TiAlN/AlN multi layered coatings are alternative to TiAlN monolayered coatings.

2 EXPERIMENTAL DETAILS

In this study, TiAlN/AlN multilayer coatings were produced by diffirent magnetron process parameters. Partilally, magnetron discharge power, bias voltages and N_2 flow rate changes growing morphology and surface quality TiAlN and TiAlN/AlN multilayer coatings were deposited by a pulse dc reactive magnetron sputtering ion plating system (Tip L 560 Leybold). High speed steels (AISI HSS) and hardmetal (K13) were used as substrate materials. Two magnetron targets with TiAl (20% Al) and pure Al targets were used together for the production of multilayer coatings. When the substrate placed on the rotating holder comes in front of the TiAl target, a TiAlN layer deposits on the surface, and when the samples comes in front of the pure Al target, then AlN deposits on TiAlN layer. The crystal structure of multilayer films was determined by grazing incidence x-ray diffraction (XRD) analysis. Surface and fracture surfaces were characterized by scanning electron microscopy (SEM).

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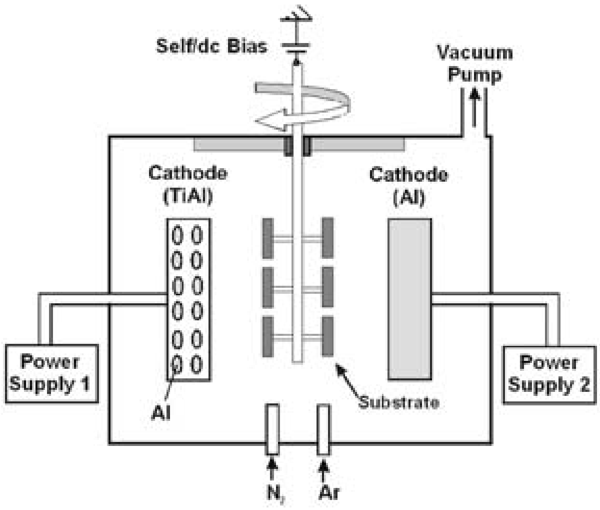

Figure 1: Schematic representation of magnetron sputtering system

Table 1: Process parameters and affects on deposition temperature, thickness

DC Bias	N2	Power	Deposition temperature	Summe Thickness	Thickness (TiAlN/AlN)
Self bias	25	500	350	2,5	105
		1000	350	4,0	168
	50	500	312	1,9	80
		1000	327	2,1	88
-30 V	25	500	500	3,3	138
		100	Not meas.	4,8	206
	50	500	435	1,9	74
		1000	620	2,3	94
-60 V	25	500	394	3,2	133
		100	352	4,4	188
	50	500	308	1,1	38
		1000	377	2,4	97

3 RESULTS AND CONCLUSIONS

Al content has significant effects on the mechanical properties, oxidation resistance and microstructure of the coatings. If Al content excess %26, layer structure includes TiAlN (AlN) hexagonal crystals. Hexagonal phases harmful effects on mechanical properties of coating [5,6]. If Al content less than %26, cubic (like TiN) structure was formed. This result showed that Al atoms replaced with Ti atoms in TiN lattice, for this reason lattice parameter decreased and film hardness and adhesion resistance were increased. The microstructure and growing morphologies correlated to sputtering conditions in a range of TiAlN/AlN multi-layer coatings with nanoscale bi-layer period thickness investigated carefully in order to identify the excellent properties of the coatings.

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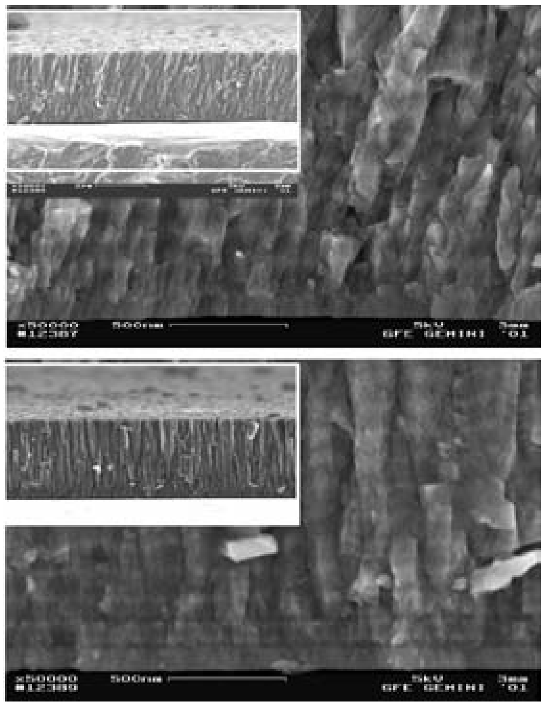

Figure 2. SEM micrographs of coatings, increasing of sputtering power versus thickness **a.** 500W **b.** 1000W

EXAMINATION ON SURFACE PROPERTIES OF MODIFIED AISI 1090 STEEL BY PULSE PLASMA TECHNIQUE

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A new way of modification of steels by pulse plasma technology is characterized by low consumption of alloying elements and electric power and high productivity. This paper presents the results of the modification of AISI1090 steel by used pulse plasma technique. To modify the surface different battery capacities (500 and 600 μF) were chosen and different distances between nozzle and sample (20mm and 30mm) and different number of pulse (10, 30 and 50) were determined. Detailed estimation of the quality of the modified metal surface was performed by additional testing of chemical compositions by EDS and X-ray diffraction, scanning electron microscope (SEM), microstructure and micro-hardness. The micro-hardness of the samples surfaces increased with decreasing battery capacities and with increasing distance between nozzle and sample and number of pulse. Possible mechanisms of modification are discussed.

Key Words: pulse plasma technique (PPT), modification, micro-hardness

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IMPACT WEAR RESISTANCE OF LASER-CLAD VALVE SEATS WITH STELLITE 6 ALLOY

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ABSTRACT

A cobalt-based alloy, Stellite 6, was deposited on the valve seat of control valves by a coaxial laser cladding process. For comparison with conventional hardfacing techniques, the plasma transferred arc (PTA) welding process was also performed. The laser-clad layers, which consisted of refined solidification structures, were characterized by increased hardness and impact toughness, as compared to the PTA overlays. The laser-clad valve seats exhibited superior impact wear resistance, i.e., the resistance to impact cracking at high loads, relative to the PTA-clad valve seats.

Keywords: impact wear, stellite 6 alloy, laser cladding, PTA welding, microstructure

1 INTRODUCTION

Cobalt-based alloys are often used in wear-related applications, in which Stellite 6 is the most versatile material for a wide range of hardfacing applications [1,2]. It has been reported that an increased hardness and a refined structure of Stellite 6 coatings could be obtained by laser cladding [3]. The purpose of this investigation was focused on the impact wear resistance of valve seat faces, which were deposited with Stellite 6 by laser and PTA cladding processes. Impact wear tests were conducted on a self-built apparatus at room temperature to evaluate the resistance to impact cracking under simulated conditions. In addition, similar tests were performed on a limited number of clad specimens at 540 °C.

2 EXPERIMENTAL SETUP

The materials employed in this study were a mild steel and a Cr-Mo-V steel, with the chemical composition (wt%) of Fe-0.5 Mn- 0.2 Si (for specimens tested at 25°C) and Fe- 8.44 Cr- 0.966 Mo- 0.193 V- 0.102 Ni- 0.416 Mn- 0.347 Si- 0.11C (for specimens tested at 540 °C), respectively. These steels were utilized to manufacture a reduced size of control valves. The valve seat was hardfaced with Stellite 6 powder (53 to 180 μm) by laser cladding at a preheat temperature of 250 °C. For comparison, parallel experiments were also conducted on similar specimens using the PTA welding process.

Laser cladding was performed using a Rofin-Sinar 850 CO_2 laser equipped with a coaxial powder feed nozzle. **Table 1** lists the laser processing parameters used for hardfacing the seat face of control valves, in which the overlap between laser tracks was 33%. After cladding, Cr-Mo-V steel specimens were tempered at 650 °C for 1 h for stress-relief.

Figure 1 shows the schematic diagram of the impact wear test, in which a nitrided 422 SS valve disk was connected to a hydraulic piston rod and struck on the seat face of a cylindrical specimen (control valve body) repeatedly. The detailed dimension of the valve seat is also shown to the right of the figure. The piston was driven by a load-controlled hydraulic system that generated impact loads of 78 and 98 kN on the control valve in the simulation experiment. Tests were conducted to identify the number of impacts (in the unit of 1,000 impacts) necessary to observe marcocracks on the clad layer at a given load. If no such cracks could be found, the specimens were subjected to another cycle of 1000 impacts.

3 RESULTS AND DISCUSSION

The as-deposited structures of laser- and PTA-clad layers comprise of a hypoeutectic structure with eutectics at interdendritic regions as depicted in **Figure 2**, with the

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Presentation type: Poster

Table 1: Laser cladding parameters used in the investigation

Laser power (kW)	Travel speed (mm/min)	Powder flow (g/min)	Carry gas (l/min)	Central gas (l/min)	Shroud gas (l/min)	Stand-off dist. (mm)	Number of layers
3.2	600	13.2	Ar: 2.5	He: 10	Ar: 15	15	3

hardness values indicated. Owing to the low heat input of the laser cladding process, the microstructure of the laser-clad layer is much finer than the PTA-clad layer. The refined structure of Stellite 6 layers were harder and had better impact toughness. The impact values of un-notched specimens (sub-size 2.5 mm thick) were 7.7 J and 4.7 J for laser- and PTA-clad specimens, respectively.

Table 2 is the results of impact wear tests under various loading conditions at room temperature. Apparently, the laser-clad specimens were superior to the PTA-clad specimens. For instance, a laser-clad specimen could survive after 12,000 impacts without forming a macrocrack at 98 kN, while the PTA-clad specimen exhibited a surface crack of about 1 mm after 5,000 impacts. The presence of macrocracks on the seat face could be identified easily with the aid of an 8X magnifier. However, SEM was needed to observe microcracks, which were formed mainly along the interfaces between the eutectic and cobalt-rich γ-phase on the impact wear surfaces. Note that the laser-clad specimen also developed fewer and smaller microcracks than the PTA-clad specimen after impact wear tests. For such tests at 540 °C, no visible cracks were observed on the seat face after 12,000 impacts at 98 kN for different processed specimens. This clearly indicated that the presence of oxides, possibly Cr_2O_3 and CoO, was beneficial for the impact wear resistance of the valve seat.

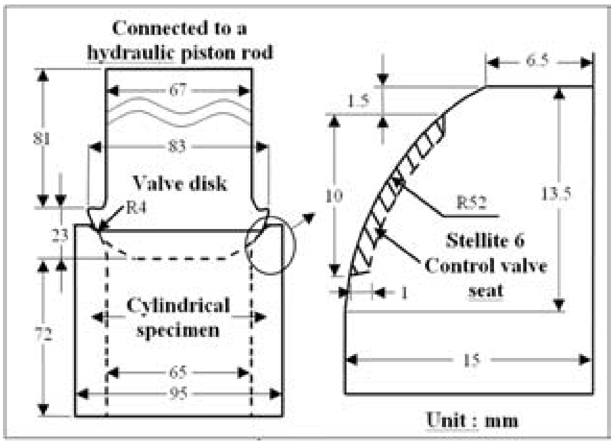

Figure 1: Schematic diagram of the impact wear test

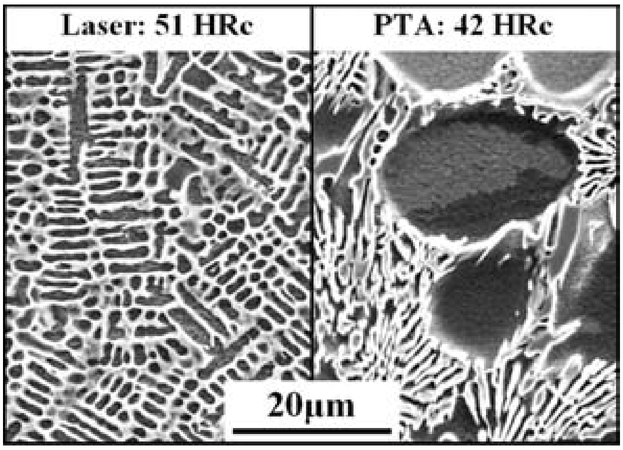

Figure 2: Microstructure of the Stellite 6 layers

Table 2: The results of impact wear tests at room temperature

Impact force (kN)	Laser-clad specimens	PTA-clad specimens
78	>12000*	8000**
78	>12000*	6000**
98	>12000*	5000**

* No visible cracks (macrocracks) under a magnification of 8X after 12000 impacts

** The observation of at least one macrocrack after the indicated number of impacts

4 CONCLUSIONS

In this study, the advantages of laser cladding of Stellite 6 alloy on the seat face of control valves have been demonstrated. The laser-clad layers exhibited finer microstructure, higher hardness and impact toughness relative to conventional PTA overlays. The impact wear tests of simulated valve specimens at room temperature confirmed the superior resistance to impact cracking of the laser-clad specimens. No macrocracks were found for such specimens after 12,000 repetitive impacts at high loads (78 and 98 kN), while macrocracks were observed for the PTA-clad specimens after 8,000 impacts or less. Nevertheless, microcracks along the interfaces of eutectics were observed, but to a lesser extent for the laser-clad specimen. For impact wear tests at 540°C, no macrocracks were observed on the seat faces of the laser- and PTA-clad specimens after 12,000 impacts at 98 kN. The presence of surface oxides on Stellite 6 deposits improved significantly the impact wear performance of valve seats at elevated temperatures.

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OBSERVATION MULLITE STRUCTURE DEPENDING ON SPRAYING PARAMETERS

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ABSTRACT

In this study, mullite coating deposited on various substrates by APS using 3MB spray gun. We investigated effects of coating parameters (gaseous pressure, spraying distance etc.) on mullite microstructure whether it is amorphous or crystalline. We observed all coatings' rate of amorphous phase and crystalline phase. The crystallization temperature was determined by DTA. After all, we study to obtain an optimum coating parameter. The coatings were characterized by XRD, SEM, DTA and DSC.

Keywords: EBC, TBC, mullite coating, plasma spray

1 INTRODUCTION

The higher efficiency requirement, in gas turbines aero planes, turned the attention toward the usage of Si based ceramics, which could obtain higher combustion temperature, as structure material. However Si based ceramics have some limitations because of Si losses at high temperature. Therefore EBCs have been started to study to prevent silica losses from structure material and for usage thermal isolator. Mullite has excellent corrosion, creep resistant and correlation thermal expansion coefficient between SiC. These properties had mullite become candidate material for EBCs. In Gleen Labrotory mullite had deposited by conventional thermal spray techniques on Si based ceramics by NASA Glenn group. But it had showed amorphous structure that is not able to use for EBCs. Subsequently, a modified plasma spraying process was developed at NASA Glenn, which successfully eliminated most of the amorphous mullite from the coating [1]. In this case, we investigate how mullit's structure is affected by the coating parameters. We obtained coated mullite crystallization temperature.

2 EXPERIMENTAL SETUP

The coatings were produced by atmospheric plasma spray system that use 3MB spray gun on various cylindrical substrate. The coating powder %2 SiO_2 impurity. The substrates were heated 500, 800 °C. The sample stereo microscope figures are shown at **Figure 1**. Substrate temperature recorded along the coating process. DTA was used for all coating to obtain crystallization temperature and XRD was used to calculate rate of phases.

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Figure 1: The mullite coating stereo microscope figure at 16x

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CHARACTERISTICS OF ELECTROCODEPOSITED Ni–Al_2O_3 NANO PARTICLE REINFORCED METAL MATRIX COMPOSITE (MMC) COATINGS

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ABSTRACT

Electrocodeposited composite coatings consist of a metal or alloy matrix containing dispersion of second phase particles. These particles can be hard oxides or carbide particles such as Al2O3, TiO2, SiO2, SiC, WC or diamond or a solid lubricant such as PTFE, graphite or even liquid containing microcapsules that improve wear resistance and reduce wear friction. The well-dispersed nano-sized ceramic particles in a Ni matrix can not only enhance the mechanical properties, but also would be a necessity for the use as the composite materials in microdevices. In the present work, Ni/Al_2O_3 metal matrix composite (MMC) coatings were prepared from a modified Watt's type electrolyte containing nano-Al_2O_3 particles by direct current (DC) plating method. It was aimed to increase the surface hardness and wear resistance of the electrodeposited Ni by introducing nano sized Al_2O_3 particles. It was also aimed to obtain high volume of the ceramic nano particles on the deposited Ni layers without agglomeration of nano Al_2O_3 particles. For these purposes the bath composition, the effect of surfactant quantity and deposition parameters suc as, current density stirring speed and temperature were investigated to optimize to obtain high quality coatings. Since Ni coatings have low surface hardness, this application is thought to provide new materials for dies, tools and different parts for automobiles and microdevices. Al_2O_3 nano powders with average particle size of 80 nm was co-deposited with nickel matrix on the steel substrates. The deposition of the nano Al2O3 particle reinforced Ni matrix composites were carried out with different conditions such as, different current densities, surfactants, bath pH etc. The depositions were controlled to obtain specific thickness (between 50 – 200 μm) and particle volume fraction in the matrix (between 0.03 – 0.2). The characterization of the coatings was investigated by scanning electron microscopy (SEM) and X-ray diffraction (XRD) facilities. The hardness of the resultant coatings was also measured and found to be 300 – 500 Hv depending on the particle volume in the Ni matrix. The effects of the surfactant on the zeta potential, co-deposition and distribution of Al_2O_3 particles in nickel matrix, and tribological properties of composite coatings were investigated. The tribological behaviors of the electrodeposited nanocomposite coatings sliding against M50 steel ball (ϕ 10mm) were examined on a CSM Instruments Tribometer designed according to DIN 50 324 ve ASTM G 99-95a in a ball-on-disk configuration. The sliding was performed at normal loads and sliding speeds. All the friction and wear tests were performed under unlubricated condition at room temperature and in ambient air (relative humidity 55–65 %). The results showed that the wear resistance of the nano composites was approximately 2-3 times increased compared with unreinforced Ni deposited material.

Keywords: electrocodeposition, nano Al_2O_3, MMC, microstructure, friction, wear

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Title: Characteristics of Electrocodeposited Ni–Al_2O_3 Nano Particle Reinforced Metal Matrix Composite (MMC) Coatings
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Topic: C – Surface Engineering
Presentation type: Oral

FABRICATION AND CHARACTERIZATION OF Ni–SiC METAL MATRIX COMPOSITE (MMC) NANO-COATINGS BY ELECTRODEPOSITION

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ABSTRACT

Composite coating has been well developed for the last two decades. Its first application was the SiC/Ni coating used as the wear-resistant improvement in the Wankel engine by Metzger et al. [2] in 1970, and the SiC/Ni composite coating is still successfully used for the wear parts in the automotive industry. The advantages of this composite coating are clear. The simultaneous deposition of the non-metal inclusions and metal phases to form composite layers brings a significant improvement on several physical and mechanical properties of the coating. Recently electrolytic metal matrix composites reinforced with nano-sized particles have scientific and technological interest due to the enhanced properties. Ni–SiC composites have gained widespread application for the protection of friction parts in the automobile industry.

In this study, the plating electrolyte was a nickel sulphate bath contained size of 100–200 nanometers SiC particles was used. The aim of this work to obtain hard and wear resistance nano particle reinforced Ni-SiC MMCs on steel surfaces for anti wear applications such as dies, tools and working parts for automobiles and vehicles. In this respect optimization of the deposition parameters for specific applications was also thought to investigate. The deposition was performed on steel substrates. The influence of variables like SiC content, current density, stirring speed, pH and surfactant quantity on particle distribution, microhardness and wear resistance of nano-composite coatings has been studied. The effect of the concentration of SiC particle on the co-deposition of SiC in the Ni layer was also investigated. The nickel films were characterized by scanning electron microscopy (SEM) and X-ray diffraction (XRD) analysis. The depositions were controlled to obtain specific thickness (between 50 – 200 μm) and particle volume fraction in the matrix (between 0.03 – 0.15). The characterization of the coatings was investigated by scanning electron microscopy (SEM) and X-ray diffraction (XRD) facilities. The hardness of the resultant coatings was also measured and found to be 350 – 650 Hv depending on the particle volume in the Ni matrix. The effects of the surfactant on the zeta potential, co-deposition and distribution of SiC particles in nickel matrix, and tribological properties of composite coatings were investigated. The tribological behaviors of the electrodeposited SiC nanocomposite coatings sliding against M50 steel ball (ϕ 10mm) were examined on a CSM Instruments Tribometer designed according to DIN 50 324 ve ASTM G 99-95a in a ball-on-disk configuration. The sliding was performed at normal loads and sliding speeds. All the friction and wear tests were performed under unlubricated condition at room temperature and in ambient air (relative humidity 55–65 %). The results showed that the wear resistance of the nano composites was approximately 2-4 times increased compared with unreinforced Ni deposited material.

Keywords: electrocodeposition, nano SiC, MMC, microstructure, friction, wear resistance

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SURFACE MODIFICATION OF LOW AND MEDIUM CARBON STEEL BY USING ELECTROLYTIC PLASMA THERMOCYCLIC TREATMENT

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In this work, using the electrolytic plasma thermocyclic treatment (EPTT) techniques, surface modifications of low and medium carbon steel were carried out. The EPTT system was set as industrial scale. Na_2CO_3 (soda ash, sodium carbonate) was used as electrolyte in water. The approximately ratio of sodium carbonate and water was 15, 85 respectively. Various voltage and ampere values were performed to obtain good surface properties. Moreover different impulse ratio and cooling rates were performed. Then modified surface of steel samples were cleaned by stirring with hot water after that the standard metallographic studies were carried out by using optic microscope. Also micro hardness and Rockwell hardness tests were performed. Furthermore, the SEM – EDS and XRD studies were examined in order to good understanding of hardened and modified surfaces. The reliable microstructure and hardness values were obtained on surface of steel samples.

Keywords: electrolyte, electrolytic plasma thermocyclic treatment techniques (EPTT), low carbon steel, medium carbon steel

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MAGNETIC-ASSISTANCE IN CYLINDER-SURFACES FINISH

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ABSTRACT

A new finish mode uses a magnetic-assistance with high efficiency to assist discharging dregs out of the electrodes' gap during the electrochemical finishing on the cylinder-surfaces is investigated in the current study. The factors affecting the effects of the magnetic-assistance in electrochemical finishing are primarily discussed. The mainly experimental parameters are magnetic strength, distance between the two magnets, current density, on/off period of pulsed-current, and width of tool-electrode. Providing large magnetic field intensity or using a small distance between the two magnets produces a larger magnetic force and discharge ability, and a better finish. Small width of tool-electrode provides sufficient discharge space, which is advantageous for magnetic-assistance electrochemical finishing. A higher current density with magnetic-assistance reduces the finish time and avoids the difficulty of dreg discharge. Pulsed direct current can slightly promote the effect of electrochemical finishing, but the current density needs to be increased. The magnetic-assistance during the electrochemical finishing process makes a great contribution in a short time by making the surface of the workpiece smooth and bright.

Keywords: magnetic-assistance, magnetic force, cylinder surfaces, finish processes, electrochemical finishing

1 INTRODUCTION

Electromagnetic metal formation (EMF) is an example of a high-speed formation process that is determined by the dynamics of a coupled electromagnetic-mechanical system. The "magnetic" pressure provides the formation energy. The energy is usually supplied to the workpiece in the form of kinetic energy. [1]. A magnetic deburring method for a drilled hole on the inside of the tubing is proposed. It was proved that this magnetic deburring method was effective for the internal deburring of long tubing [2]. Electropolishing is a very effective technique for approaching mirror-like surfaces on many metals. For many applications, a smooth and bright surface is essential and electropolishing is the best technique for this. Additionally, it is recognized that highly polished surfaces are easier to maintain in a high state of cleanliness [3]. The electropolishing process is still under-utilized because of a lack of understanding of the metal removal mechanism and inefficient tool design methodology being used. Even for simple cases, it is impossible to predict work profiles accurately [4, 5]. The potential for the design of magnetic assistance during the surface finish is yet to be explored. The current study discusses the design features of the performance assessment on discharging dregs using magnetic force. The cylindrical surface uses a magnetic system and an effective partial mate-form electrode supplied with continuous and pulsed direct current during electrochemical finishing. An effective evaluation on the process both features and sets up the complete processing parameters data, which is expected to apply propagation for freeform surface finishing in the future.

2 EXPERIMENTAL SETUP

The experimental set-up of a finish process using the magnetic-assistance in electrochemical finishing is illustrated in **Figure 1**, and includes a magnetic system, DC power supply, pulse generator, pump, flow meter, electrolytic tank, and filter. The schematics and configuration of the magnetic-assistance system, electrodes and workpiece are shown in **Figure 2**. The workpiece material uses SKD1. The chemical compositions are shown in **Table 1**. The amount of reduction in the workpiece's surface after electrochemical finishing is 20 µm. The mag-

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Table 1 Chemical Composition of Workpiece

(Wt %)	Fe	C	Si	Mn	P	S	Cr	W	Mo	V	Co	Ni
SKD1	REM.	2.10	0.25	0.45	0.023	0.01	13.50	/	/	/	/	/

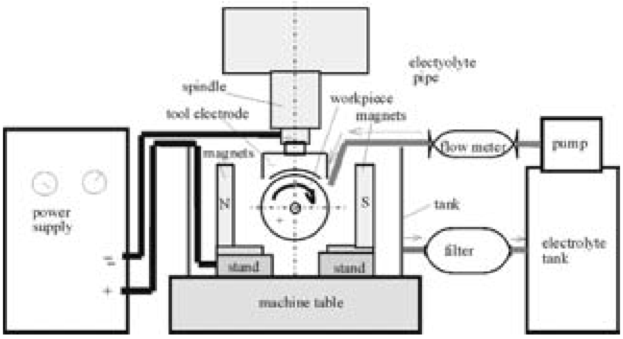

Figure 1: Experimental set-up

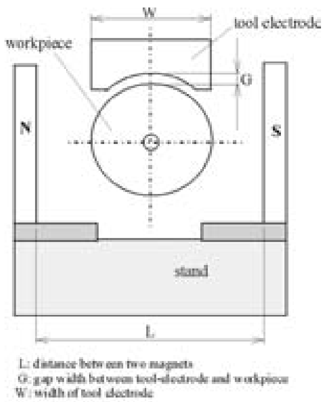

Figure 2: Schematics and configuration of the magnetic-assistance system, tool electrode and workpiece

netic strength (magnetic field intensity) of the magnet is 1500, 2000, 2500, and 3000 Gauss. The distance between the two magnets is 80, 90, 100, 110, and 120 mm. The electrolyte is $NaNO_3$ of 20 %wt. The temperature of the electrolyte is maintained at 35±5 °C. The flow rate of the electrolyte is 15 L/min. The current density is 10, 15, 20, 25, 30, and 35 A/cm^2. The gap width between the electrode and workpiece is 0.3, 0.4, 0.5, and 0.6mm. The width of tool-electrode is 20, 25, 30, 35, and 40 mm. The rotational speed of the cylindrical eorkpiece is 200 to 1200 rpm. The pulsed period (on/off time) is 100 ms/100ms, 100ms/200ms, 100ms/300ms, 100ms/400 ms and 100ms/500ms. The different features for the finishing process include electrochemical finishing (EF), pulsed-electrochemical finishing (PEF), magnetic-assistance electrochemical finishing (MEF), and magnetic-assistance pulsed-electrochemical finishing (MPEF). After different finishing processes, all workpieces are measured by the surface roughness measurement (Hommel T500, the accuracy is within ±5 % after standard correction). The surface roughness is characterized by Ra, where the length of cut-off is 0.8 mm, and the measuring direction is perpendicular to the tooth mark. The measuring data is chosen from at least two different locations.

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STRUCTURAL CHARACTERISTICS OF PLASMA NITRIDED 32CrMoV33 HOT WORKING DIE STEEL

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ABSTRACT

In this study, nitriding behavior of 32CrMoV33 hot working die steel was investigated by plasma process. Nitriding process was carried out at temperature of 450 °C, 500 °C and 550 °C for treatment time in the range of 2-32 h. The thickness of compound layer, measured by means of a digital thickness instrument attached to optical microscope, ranged from 2 µm to 14 µm depending on nitriding temperature and treatment time. The presence of γ-Fe_4N, ε-$Fe_{2\text{-}3}N$, Cr_2N, α-Fe and Mo_3N_2 phases formed in the compound layer was confirmed by XRD analysis. Using cross sectional samples and a micro hardness indenter, hardness depth profiles were also obtained. In kinetic studies, effective diffusion coefficient and activation energy was calculated from the time and temperature dependences of the hardness profiles, and increasing nitriding temperature increased effective diffusion coefficient.

Keywords: plasma nitriding, micro hardness, compound layer, die steel

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THE EFFECT OF BIAS VOLTAGE ON OXIDATION BEHAVIOR OF MONOLAYER TiAlN AND MULTILAYER TiAlN/AlN COATINGS

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ABSTRACT

TiAlN coatings have been known to be superior to other coatings such as TiN and TiCN in protecting tools which may be damaged by high thermal load. It is well known that multi-layered coatings indicates better thermo/mechanical properties than mono layered coatings. Monolayer TiAlN coating have less oxidation resistance than multilayer TiAlN/ AlN coatings. In this study, TiAlN/AlN multi layer coatings were deposited at different bias (self bias, –30V, –60V) voltages by the method of physical vapor deposition (d.c. magnetron sputtering) on steel substrates. Experimentally all coatings are oxidized as isothermals for 1 hour at 800 °C to inspect oxidation of the films. Then oxidised surfaces were characterized by optic microscopy, electron micsroscopy (SEM) and EPMA. Crystal structure were analyzed using XRD. It was found that bias voltage increase to the coating density and oxidation resistance.

Keywords: magnetron sputtering PVD, multi layer thin films, bias voltage, oxidation resistance, SEM, XRD

1 INTRODUCTION

During the cutting operation, degradation of the coatings occurs by a mixture of wear, oxidation and diffusion. TiAlN coatings have been developed as an alternative coating to TiN coatings for cutting tools and moulds. Metastabil TiAlN has excellent oxidation resistance and it has good cutting performance at high speeds. During the oxidation, Al_2O_3 film formed on the TiAlN coating and provide protection to furthermore diffusion. Effect of substrate bias voltage significantly important on oxidation behaviour of coatings.

2 EXPERIMENTAL DETAILS

TiAlN and TiAlN/AlN multilayer coatings were deposited by a pulse dc reactive magnetron sputtering ion plating system (Tip L 560 Leybold). High speed steels (AISI HSS) and hardmetal (K13) were used as substrate material. Bias voltages are repectively: self bias, –30 and –60V. Prepared samples were inserted into the sputtering system by clamping them on to the substrate holders. Before the sputtering, the chamber was evacuated to less than $2 \cdot 10^{-6}$ mbar. The chamber was then back –filled with argon to $6 \cdot 10^{-3}$ mbar, and the substrates were sputter cleaned for 30 min using 200 W RF discharge (DC –120V). Two magnetron targets with TiAl (20% Al) and pure Al targets were used together for the production of multilayer coatings. When the substrate placed on the rotating holder comes in front of the TiAl target, a TiAlN layer deposits on the surface, and when the samples comes in front of the pure Al target, then AlN deposits on TiAlN layer. SiC-tube furnace is used for oxidation. Coatings heated 1 hour at 800 °C. Surface oxidation

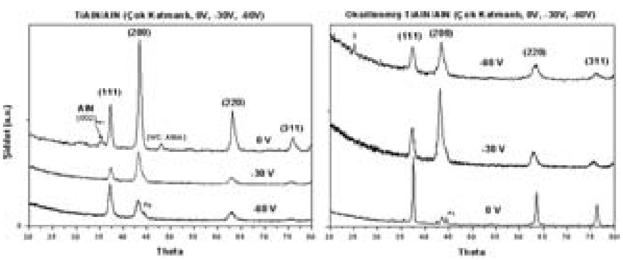

Figure 1: Multilayer TiAlN coatings XRD analysis versus diffirent bias voltage: original (left), oxidised (right)

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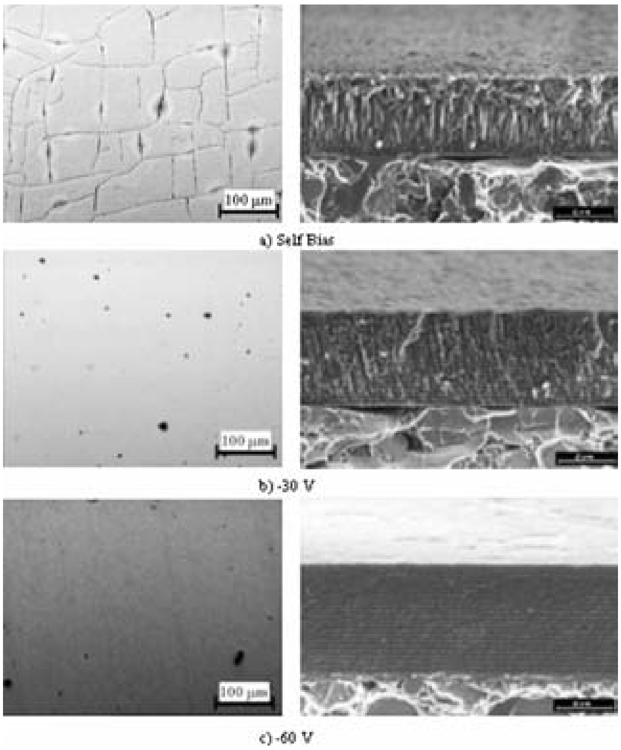

Figure 2: SEM images of coating by using diffirent bias voltage: surface (left) cross section (right)

occured at the coating surface. The oxidation behaviour of coatings in air was studied using X-ray diffraction (XRD), scanning electron microscopy and EPMA analysis.

3 RESULTS AND CONCLUSIONS

Result show that, multi layer coatings improve the resistance of the oxidation. Bias voltage effected film growing morphology.

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PROPERTIES OF HARD Ni-P-Al_2O_3 AND Ni-P-SiC COATINGS ON Al-BASED CASTING ALLOYS

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ABSTRACT

In the presented work, electroless Ni-P-Al_2O_3 and Ni-P-SiC composite coatings on an aluminium-silicon casting alloy are studied. A suspension containing nickel lactate, nickel hypophosphite, lactic acid, water and dispersed ceramic particles was used for the electroless deposition. The structural study, hardness and abrasion tests reveal that properties of the coatings strongly depend on the composition of deposition bath and on the heat-treatment. The composite coatings heat treated at 400°C/1 h showing an outstanding abrasion resistance are promising candidates for the surface protection of light components.

Keywords: electroless coating, aluminium, ceramics, casting, abrasion resistance

1 INTRODUCTION

Al-based alloys show several attractive properties, such as low weight, high specific strength, high thermal conductivity and relatively good corrosion resistance in air. For these reasons, they became popular construction materials in automotive and aerospace industry. In some applications, however, they suffer from insufficient wear resistance and several approaches have been adopted to prolong their life, including reinforcement with particles or fibers, increase of Si content, hard PVD or electrodeposited Cr coatings [1,2]. Unfortunately, problems often arise when components of complex shapes are coated. These problems are in part avoided when using electroless Ni-P coatings. The electroless Ni-P coatings are produced by a redox reaction in which Ni^{2+} cations are reduced by hypophosphite ($H_2PO_2^-$) anions on the substrate surface [3]:

$$2Ni^{2+} + 8H_2PO_2^- + 2H_2O \rightarrow \\ \rightarrow 2Ni + 6H_2PO_3^- + 2P + 3H_2 + 2H^+ \qquad (1)$$

It has been shown in a number of studies that hardness of the as-deposited coatings ranges from 500 to more than 1000 HV, depending on phosphorus content and heat treatment procedure [4,5]. Additional improvement of the wear resistance can be achieved by the co-deposition of hard particles (silicon carbide, diamond, aluminium oxide etc.) [6,7]. A lot of studies have been devoted to the electroless composite coatings on steel substrates but little work has been reported on the surface protection of Al-based alloys. The important difference between steels and aluminium alloys is that aluminium easily forms the stable passive oxide layer, reducing the adhesion of the coating. Hence, a chemical pre-treatment of aluminium substrate, removing the oxide, is necessary. The presented work aimes to describe properties of electroless Ni-P-Al_2O_3 and Ni-P-SiC composite coatings on the commercial aluminium-silicon casting alloy.

2 EXPERIMENTAL PROCEDURE

The commercial Al-12wt.%Si alloy provided by an industrial supplier was used as substrate for the electroless deposition. Pre-treatment of the substrate surface included following steps: 1. grinding with SiC papers, 2. degreasing with acetone, 3. double deoxidizing and 4. rinsing. The electroless deposition was carried out in a suspension consisting of ceramic particles (Al_2O_3 or SiC, irregular particles of 5 μm in size, amount in the bath between 5-15 g/l) dispersed in a water solution of nickel lactate, nickel hypophosphite and lactic acid. After deposition, some of the coated samples were heat treated at 400 °C/1 h [5]. Structure of both as-deposited and as-heat treated coatings was examined by light microscopy (LM), scanning electron microscopy (SEM), energy dispersive x-ray spectrometry (EDS) and x-ray diffraction analysis (XRD). Vickers hardness of the Ni-P

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phase was mesured using a loading of 5 g. Wear resistance was determined by using the modified "pin-on-disc" method (P4000 SiC paper, sliding distance of 330 m, normal force of 5.8 N).

3 RESULTS AND DISCUSSION

Structural investigation reveals that the ceramic particles are uniformly distributed in the coatings. Their volume fraction increases with increasing amount in the bath to a maximum of about 30-40 %. The coating thickness varies between 15 and 25 μm, giving an average deposition rate of about 8-13 μm/h. The average surface roughness also depends on the particle content in the bath. The smoothest surface is characteristic for pure Ni layer, while the co-deposited particles increase roughness up to about 5-7 μm. It is also observed that the coatings show a good adherence to the Al-Si substrate, suggesting a positive role of silicon in the alloy. Chemical composition of the Ni-P phase in the coatings determined by EDS is Ni-10wt.%P. This value does not depend either on particle type or on particle volume fraction. XRD patterns of the as-deposited composite coatings contain single broad amorphous peaks of Ni-P phase and sharp well separated peaks of ceramic phase, Al and Si. The broad peak is commonly regarded as corresponding to a nanocrystalline phase composed of extremely fine nanometer scaled crystalline regions (in our case about 2 nm) surrounded by amorphous grain boundaries.

Hardness of the Ni-P phase in both types of composite coatings lies around 550 HV0.005. In spite of some random variations, no dependence of hardness on ceramics content and type is seen. Hardness of the Ni-P phase mainly depends on the P content. The higher the P content, the lower the hardness results. It is generally explained in terms of the nanocrystalline grain refinement due to phosphorus. The grain refinement leads to a softening due to the inverse Hall-Petch behaviour commonly observed for phases having grains less than about 10 nm in size [8]. The heat treatment at 400 °C/1 h causes considerable hardness increase of the Ni-P phase from initial 550 HV0.005 to about 900 HV0.005. XRD reveals that the nanocrystalline Ni-P phase decomposes to crystalline Ni and Ni_3P phases. Therefore, the observed hardening can be attributed to precipitation of very fine phosphide particles acting as effective obstacles for dislocation slip.

Results of abrasion tests show that the Ni-P coating free of ceramic particles reduces abrasion rate by about 50 percent as compared to the Al-Si substrate. Incorporation of hard ceramic particles leads to further significant improvement of the abrasion resistance. The highest abrasion resistance is a charasteristic of the composite coatings which are heat treated at 400 °C/1 h. The thickness loss of the heat treated Ni-P-SiC composite coating after 330 m abrasion is about 20 times lower than that of the uncoated alloy.

4 CONCLUSIONS

It is shown in the presented paper that hard Ni-P-Al_2O_3 and Ni-P-SiC composite coatings can be succesfully prepared on the pre-treated Al-Si casting alloy by co-deposition of ceramic particles from a stirred bath containing nickel lactate, nickel hypophosphite and lactic acid. Due to silicon particles in the structure of the Al-Si substrate, adhesion of the composite coatings is good. Composite coatings can be used to significantly improve abrasion resistance of Al-Si castings, particularly, if they are subsequently heat treated at 400 °C/1 h.

Acknowledgements

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INVESTIGATION OF SPUTTER CRATERS AFTER GDOES ANALYSIS OF TiCN COATINGS

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ABSTRACT

The investigated TiCN coatings were deposited on a chromium-mollybdenium-vanadium alloyed tool steel Sleipner of Uddeholm Company by cathode arc evaporation technique (BAI 1200) in Balzers Hard Coating Center (Kapfenberg, Austria). In this work the depth profiling method glow discharge optical emission spectrometry (GDOES) is used for determining the thickness of TiCN coating after determining the sputtering rate. GDOES is a rapid depth profiling technique, this analysis is destructive and there is a formation of sputter craters on the sample surface. The obtained data were compared with the thickness values obtained from the SEM fracture cross-sections. Structure of sputter craters after GDOES experiment at different coating depths as well as the coating surface were evaluated by the scanning electron microscopy (SEM), 3D profilometer and atomic force microscopy (AFM). The chemical composition of the coating was determined by the EDS method.

Keywords: PVD, TiCN coatings, sputter craters

1 INTRODUCTION

Tool steels were developed to resist wear at temperatures of forming and cutting applications. They are grouped into six categories: cold work, shock resisting, hot work, high speed, mold and special-purpose tool steels. Cold work tool steels are used for many types of tools and dies and other applications where high wear resistance and low cost are demanded. Surface engineering processes such as surface heat treatment, coating and surface modification are employed to increase the surface hardness and minimise adhesion (reduce friction) and improve wear resistance of tool steel substrates. PVD hard coatings are well known for providing engineering surfaces with high hardness and high tribological properties [1-4].

In this paper, the depth qualitative composition profiles, sputtering rate and thickness of TiCN coating were determined by the glow discharge optical emission spectrometry (GDOES) method. Primarily, depth profiling methods using ion sputtering deliver a sputter (measuring) time scale, and not a depth scale. Converting the time scale into the true depth scale is not easy, because many different sputter effects may occur and, particularly at interfaces with changing concentration gradients, stable and well defined conditions are not reached [5].

2 EXPERIMENTAL SETUP

TiCN coatings were prepared in Balzers BAI 1200 deposition system by cathode arc evaporation technique (coating commercial name: Balinit B). The coatings were deposited onto the Sleipner tool steel substrates. The steel sample was finely grinded, ultrasonically cleaned and sputter cleaned prior to coating deposition in order to obtain optimum adhesion between coating and substrate. The Rockwell C hardness of the Sleipner tool after hardening and tempering was 61 HRC. The thickness of hard coatings was determined by the GDOES depth profiles by means of spectrometer GDS 750 (Leco) as well as from SEM fracture cross-sections. The depth of sputter craters obtained from GDOES analysis is evaluated after determining the sputtering rate. Sputtering rate (SR) was determined by measuring depth of crater at different times (30, 50, 70, 90 and 150s) of GDOES analysis. Structure of each sputter crater and thickness of TiCN coating were evaluated by scanning

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electron microscopy, 3D profilometer and atomic force microscopy (AFM).
The chemical composition of each sputter crater was evaluated by means of SEM analysis with EDS detector. Sputtering rate of each sputter crater was determined according to the following equation:

SR = depth of crater / sputtering time.

After determination of average sputtering rate, the thickness of the investigated TiAlN coating was determined by means of GDOES analysis.

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PREVIOUS OR SUBSEQUENT ELECTRON BEAM HARDENING OF THERMOCHEMICAL TREATED AND PVD HARD COATED STEELS FOR TOOLS AND COMPONENTS

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ABSTRACT

The improvement of the tribological properties of tools and components by wear resistant coatings deposited by thermochemical processes (nitriding, nitrocarburizing) and physical vapour deposition (PVD) technologies respectively is state of the art. The distinct properties gradient between the very hard but brittle layer and the soft, ductile base material can be significantly improved by previous or subsequent electron beam hardening (EBH). Surface hardness is increased, which results in a better load support of the base materials. The dependence of the tempering stability of the steels and the chemical and structural stability of the coatings on the chosen sequence of treatments is discussed.

Keywords: duplex technology, electron beam (EB), nitriding, nitrocarburizing, boronizing, PVD, hard coating

1 INTRODUCTION

With regard to complex load conditions for most tools and components, especially close to the surface, the properties attainable by single treatments (mechanical, thermal, thermochemical and coating methods), in particular, often are insufficient. Therefore, combined processes (duplex or hybrid processes) came into the focus of investigations. It has been shown that in the case of optimized process parameters the advantages of combined treatment (for example hard coating and electron beam hardening) complement each other and the disadvantages of the single processes cancel each other out at least partially. In certain cases novel effects arise which are not attainable by any single process.

Hard coatings basing on titanium, aluminium or chromium carbides are successfully applied as hard wear resistant layers for tools and components. These hard but also brittle coatings are often unable to bring their excellent properties fully to bear on relatively soft base materials. Therefore the base materials are usually subjected to additional heat treatment before or after hard coating. Surface treatment technology using a high energy beam, especially the electron beam (EB) acting under vacuum, is a forward-looking economical and ecologically friendly alternative to the mostly used bulk heat treatments.

Because of a precisely definable energy deposition an exact adaptation of the surface heat treatment to the locally occurring load conditions is achieved. Furthermore, it is possible to limit the heat treatment to the highest loaded areas and up to the depth where a martensitic transformation is necessary. The thermal loading of the overall component is minimized. With regard to a subsequent heat treatment of hard coated steels it permits to prevent undesirable changes of composition, structure and properties of the hard coating because of a very short interaction time and the process-related treatment in vacuum. Moreover, the electron beam hardening technology is well known to cause small changes in size and shape, which means that distortion is reduced in that way also.

2 EXPERIMENTAL SETUP

Base materials used for these investigations were different steels (unalloyed steel C45, low alloyed nitriding steel 31CrMoV9 low alloyed tool steels 50CrV4 and

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100Cr6 and high alloyed tool steels X153CrMoV12, X220CrVMo13-4) in the normalised (N) or annealed (A) and hardened and tempered (H+T) pre-heat-treated state. EBH was carried out on an electron beam facility (type S20) with an acceleration voltage (Ua) of 60 kV and an output power of 12 kW. For energy transfer, high speed beam deflection was used. This results in a track-type hardening with a track width of 20 mm, whereas the neighbouring areas remain nearly unaffected. Transformation depths of 0.2 – 1.0 mm have been achieved.
The specimens were coated either before or after surface heat treatment. The hard coatings (TiN, TiCN, TiAlN, CrN, DLC) are commercially produced. Depending on the processing conditions (time and temperature) they differ in composition, layer thickness (1 – 4 µm) and layer configuration (monolayer, multilayer, gradient layer). The nitriding (N), nitrocarburizing (NC) process was carried out as gaseous nitriding. The compound layer varies between nitriding without a compound layer and a layer thickness of 20 µm, and diffusion layer thickness was smaller than 0.2 mm. Furthermore, powder-pack boronizing was used for boronizing (B).
The results have been characterised with regard to effects of EBH on the chemical and structural composition of the hard coatings and improvements of the mechanical properties (hardness, critical loads in scratch test) of the layer-matrix-compounds.

3 MAIN RESULTS

The sequence of combined surface treatment EBH + HC and the combination of EBH+N is only useful in cases where the layer deposition is carried out at "low" temperatures, e.g. when the hard coating is a PVD process. The level of the processing temperature in relation to the tempering stability of the bulk material determines the success of this treatment combination. The better the tempering stability of the steel, the smaller the hardness reduction in the previously produced EBH layer as a result of the hard coating process.
A subsequent EB heat treatment after HC and 400 boronizing has no significant influences on the visual appearance of the coatings on the combined treated specimens. In case of nitriding/nitrocarburizing the compound layer is partially transformed. The achievable hardening depth profile (**Figure 1**, **Figure 2**) depends on the chemical composition and the pre-heat-treated state of the base material and on the beam hardening conditions.
A martensitic layer beneath the hard coating or the compound layer is produced, resulting in a significant improvement of the base material's load support for the hard coatings. Therefore high surface hardness and high critical loads measured by scratch tests are obtained. Additionally, the properties gradient is improved distinctly.

4 CONCLUSIONS

The combination of the surface technologies of hard coating with EBH prior or subsequent to layer deposition opens up new structure/property relations of the layer/matrix compounds. The sequence of treatment has a considerable influence on treatment results.
Possible applications for a combination of hard coatings or a thermochemical treatment with EBH are cold forming tools with locally high loaded areas (e.g. tools for deep drawing, cold extrusion, hydroforming or solid forming) and components subjected to local high load in the form of high pressure intensity combined with sliding wear such as automotive components.

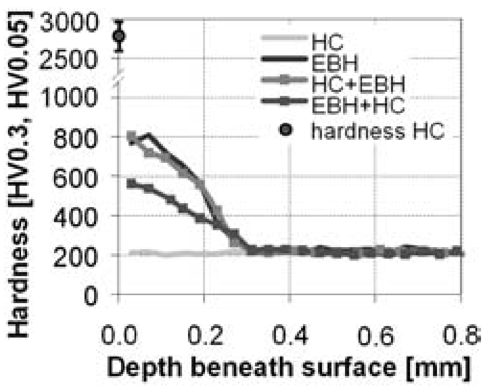

Figure 1: Hardness depth profiles of the combination of HC and EBH

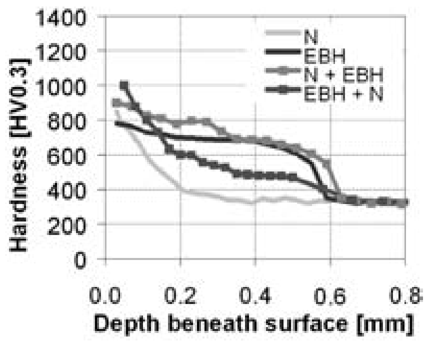

Figure 2: Hardness depth profiles of the combination of N and EBH

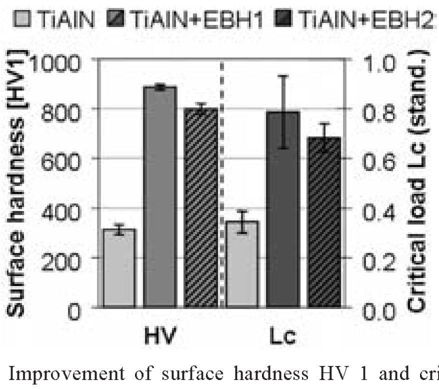

Figure 3: Improvement of surface hardness HV 1 and critical load (standardized values) of TiAlN coatings on C45 (H+T) basic material by subsequent EB hardening

MICROSTRUCTURAL AND TRIBOLOGICAL INVESTIGATION OF AlTiN-COATED CONVENTIONAL AND POWDER METALLURGY COLD WORK TOOL STEEL SUBSTRATES

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ABSTRACT

In this work microstructural and tribological investigations of AlTiN-coated conventional and powder metallurgy cold work tool steel substrates will be presented. For preparation of AlTiN we used deposition system CC800 (CemeCon) equipped with four unballanced magnetron sputter sources. Special emphasis will be on characterization of surface topography of uncoated and coated substrates.

Keywords: PVD, AlTiN hard coatings, surface topography, powder metallurgy cold work tool steel

1 INTRODUCTION

The demands of tool performance for sheet metal forming are constantly increasing especially for forming process of new high-strength sheet steels. The methods used for forming such steel sheet are basically the same as those used for forming of conventional carbon steel. However, there are some important differences due to their special deformation and surface properties. The forming process typically requires greater forming forces which consecutively leads to greater tool stresses. Some of the new materials (e.g. stainless steel) also have a greater tendency to gall. To solve these problems, high-performance tool steel materials and lubricants as well as different types of hard protective coatings are commonly used. Beside the surface chemical composition of PVD hard coating its surface topography also plays an important role.

It is well known that PVD coatings show surface irregularities such as microdroplets and dimples in the as-deposited condition [1-4]. In order to keep the friction coefficient at a low and stable value it is of outmost importance that the forming tool has a smooth surface before coating deposition and that the coating process does not significatly affect the surface topography. In such a way we can reduce the tendency of material pick-up and subsequent problems with galling.

The roughness of uncoated and coated substrate surface exists simultaneously on several size scales. While the macrofeatures (scratches, long macrowaves and pin-holes) are mostly an inheritance from earlier grinding and polishing procedures, the morphology on micro- and submicroscale is a consequence of ion etching and deposition. Some of macrofeatures also originate from deposition process. In this work we will present the influence of tool steel materials and its pretreatment on topographical features of coated substrates, surface roughness and adhesion.

2 EXPERIMENTAL SETUP

Sputtering system CC800 (CemeCon) with four 8 kW unballanced magnetron sources was used for deposition of app. 3 μm thick AlTiN hard coating. The substrate temperature was about 450 °C. The substrates undergo three-axis planetary rotation during ion bombardment as well as during deposition. Conventional and powder metallurgy cold work tool steels were used as substrates. The steel samples were finally polished with diamond paste to Ra=10-14 nm, ultrasonically cleaned and

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Topic: C – Surface Engineering
Presentation type: Poster

degreased in bath of hot alkali solution, de-ionized water and then dried in hot air before mounting on the substrate holders. They were in situ cleaned by ion etching prior to coating deposition. During substrate cleaning an RF bias was applied (maximum RF power was 2 kW), while the etching time was 85 min.

The field emission scanning electron microscope Zeiss Supra 35 VP was used for study of substrate surface morphology. The topography of uncoated and coated substrates as well as surface roughness was characterized by atomic force microscopy (AFM) and 3D-stylus profilometer (Taylor Hobson). Vickers microhardness HV and indentation modulus were determined by microindentation (Fischerscope H100C). The coating adhesion was evaluated with a scratch tester (CSEM REVETEST).

3 RESULTS AND DISCUSSION

SEM and AFM micrographs of tool steel substrates show small hillocks with a typical height in the range of 4 to 8 nm after standard polishing with diamond paste (with grain size of 1/4 μm). The lateral dimensions of such hillocks depend on type of tool steel and are in the range of 1–10 μm. This is comparable with lateral size of carbides in selected tool steel. The hardness of carbides is much higher than hardness of the matrix (50–65 HRC). Therefore during polishing, the matrix was removed at a more rapid rate than the carbides. During etching, the topography changed preferentially on carbide grains, where pits or hillocks appeared as a consequence of different sputtering rates. Namely, the sputtering rate of certain of carbides (e.g. chromium carbides, vanadium carbides) is lower than for the matrix, while it is higher for other (e.g. (Mo,W)C). Therefore the surface roughness increases. The roughness increase also arises from differences in sputtering yield from grains of different orientation. These are the reasons why the tool steel material has a large influence on topographical changes of the substrate. After deposition this topography was mostly retained. We found that the carbides with structural properties close to hard coating materials (e.g. VC) and with suitable orientation can act as nuclei for epitaxial growth, which also influences coating morphology and adhesion. We also found that the micro- and submicroparticles which flake from vacuum chamber components during deposition were incorporated in hard coating forming cone-like defects on the surface. A correlation between roughness and frequency of arcs was observed.

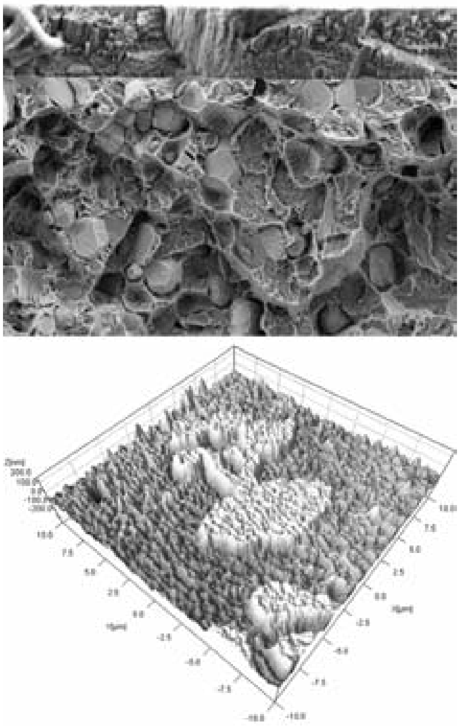

Figure 1: Fractured cross-section of AlTiN coating on powder metallurgy cold work tool steel substrates ASP30 (left) and surface topography of D2 cold work tool steel after deposition of AlTiN hard coating (right).

4 CONCLUSIONS

We found a correlation between microstructural features of the tool steel material and topography of polished substrates after deposition of PVD hard coatings. The changes are related to non-uniform hardness of substrates as well as to different sputtering rates of carbide inclusions in comparison to the matrix. The performance test on the powder metallurgy cold tool steel, which consists of 1–2 μm primary carbides evenly dispersed in a matrix, was much better in comparison to the conventional cold work steel.

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2nd INTERNATIONAL CONFERENCE ON HEAT TREATMENT AND SURFACE ENGINEERING OF TOOLS AND DIES
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PLASMA NITROCARBURIZING OF AISI H-13 STEEL FOR IMPROVED ABRASION RESISTANCE

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ABSTRACT

AISI H-13 tool steel was plasma nitrocarburized at different treatment temperatures and pulse frequencies to obtain the optimal wear-resistant layer. The gas mixture use for all treatments was 2% CH_4 / 78% H_2 / 20% N_2. The nitrocarburized layers were characterized by microhardness tests, optical microscopy (OM), scanning electron microscopy (SEM), with energy-dispersive x-ray spectrometry (EDS) and x-ray diffraction (XRD) analyses. Wear resistance was evaluated using a pin-on-disk abrasive test. All of the test specimens exhibited a substantial increase in abrasive wear resistance relative to test specimens that were not nitrocarburized. The direct current treatment temperature of 550 °C exhibited the best abrasion resistance. The formation of compound layers and the presence of the diffusion layers with high hardness and thickness indicates the potential of the process for surface hardening of the H-13 steel and application for parts and dies fabricated with this steel.

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CONTEMPORARY INDUSTRIAL APPLICATION OF NITRIDING AND ITS MODIFICATIONS

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ABSTRACT

Paper presents industrial application of gas nitriding and its modifications. Results of industrial nitriding technologies, developed in Institute of Precision Mechanics are presented. The following nitriding technologies are described: anticorrosion, hot forging tools, heavy-loaded elements, steel with high chromium content and tools from high-speed steels.

Keywords: industrial gas nitriding, anticorrosion nitriding, nitriding of heavy-loaded parts, nitriding of tools, nitriding of steel with high chromium content

1 ANTICORROSION NITRIDING

The paper presents examples of anticorrosion nitriding with subsequent oxidation and inhibitor impregnation, as applied to hydraulic actuator rods and some mass-produced automotive parts. The risks are presented of the formation of an unfavorable structure of the nitride layer, characterized by local areas of insufficient thickness of the compact zone[4,5,7]. Anticorrosion nitriding was applied to rods with the diameter of 45 to 130mm (mostly 50 -60mm) and lengths from 450 to 1050 mm. The hydraulic rods were nitrided in batches from 5 to 25 pieces, the corresponding surface area was from 0.8 to 3.8 m^2 and the mass from 75 to 485 kg (Figure1). The treatment took place in a pit furnace with retort dimensions ϕ600 x 900 mm, provided with automatic nitriding potential control. The process parameters, temperature, time and atmosphere composition were adjusted so that in agreement with earlier investigations the total compound layer thickness would stay within the limits min. 15 do 20 µm, with the compact zone not thinner than 7.5 µm (**Figure 1**).

Figure 1: View of hydraulics rods oxidation and impregnation after nitriding and microstructure of nitrided 40HM steel (AISI4140)

2 NITRIDING WITH CRYOGENIC TREATMENT OF DIESES

The nitriding of hot forging tools is presented by process for a pair of forging dies made of tool steel – WNL (**Figure 2**). After nitriding was carried out cryogenic treat-

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Topic: C – Surface Engineering (A – Heat Treatment)
Presentation type: Poster

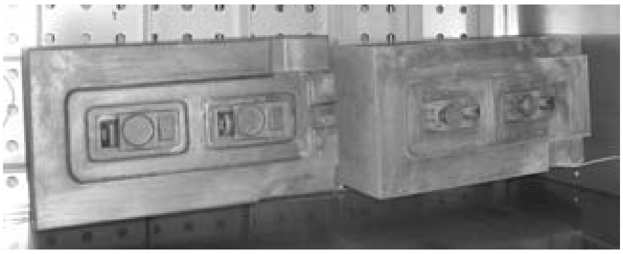

Figure 2: A pair of forging dies made of WNL steel processed with nitriding and next cryogenic treatment

ment. The service lifetime increases to 20-30% after duplex processes.

3 NITROOXIDATION AG PROCESS OF TOOLS FROM HIGH-SPEED STEELS

The nitriding of tools from high-speed steels is presented by process with oxidation (nitrooxidation process). After nitroxidation is obtained on surface of high-speed steel oxide layer and diffusion nitrided layer. After process the elements – screw-taps – have a better wear and corrosion resistance. The structure and screw-taps after nitrooxidation shown in **Figure 3**.

4 THE NITRIDING OF HEAVY-LOADED ELEMENTS

The nitriding of heavy-loaded elements is very specifics process. Nitrided layers for aviation and automotive parts content on surface controlled white layers and thick diffusion zone. This type layer has high hardness and good tough. In this type layer is possibly behavior of dimensional tolerance.

In nitriding process is very important the heating-up stage. Usually is use intensive atmosphere – 100%NH_3. At this time is nucleation of white layer and increase of nitrided layer. If we use to intensive atmosphere (to strong) the white layer is porous and thick. The nitriding atmosphere should be appropriately controlled during the nitriding process, including the stage when the treated material is heated to the process temperature [3,6].

Usually the nitriding process has two stages [1,2]. First stage is in intensive atmosphere and next stage in less intensive atmosphere. The paper presents industrial nitriding process for shafts with control white layer.

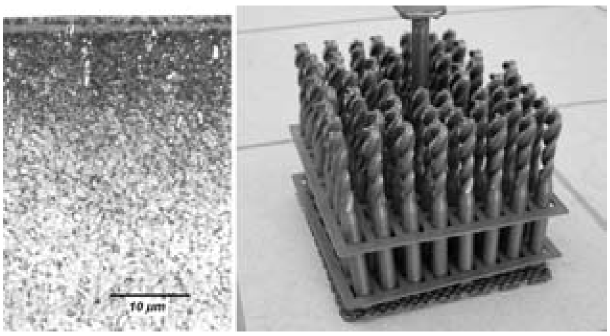

Figure 3: Microstructure and view of screw-taps in furnace equipment after nitrooxidation AG made of SW7M steel

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INFLUENCE OF ION ETCHING IN LOW PRESSURE ARC DISCHARGE IN PLASMA ON DUPLEX COAT ADHESION PRODUCED BY GAS NITRIDING AND PA PVD-ARC PROCESSES

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ABSTRACT

The aim of this study was duplex process parameters optimalization where the main attention connect ion etching process of nitriding layer. The range of our works: technological experimenthals contain with ion etching generated by using Cr, Ti, V and TiAl electodes, optymalization of gas nitriding and PA PVD-Arc processes, SEM investigation of nitrided surface after ion etching, hardness and tribological investigation and adhesion and corrosion investigation. Duplex layer obtain by gas nitriding and PAPVD-Arc processes, may be used for improve mechanical, tribological and anticorrosion properties of tools, dies and machine parts. Destination of that technology is for large and middle enterprises which produce tools and special machine elements.

Keywords: gas nitriding, PA PVD-Arc, ion etching, duplex layer, CrN, TiN

1 INTRODUCTION

The one stage of PVD process is ion etching. The works practiced in Institute of Precision Mechanics are concentrated at TiN, TiC and CrN thin coats synthesis onto tool steels surface after heat treatment and chemical heat treatment. The ion etching was extended in Ti and Cr ion stream. The effect of realized investigation shows that there is possibility to obtain coats with high adhesion onto nitriding surface of X37CrMoV5-1 steel during the ion etching with Cr ions. A interested results, caused contain a problem about coat synthesis in areological systems such like nitriding layer – TiN coat. The optymalization of technological parameters of Duplex process with particular consideration an ion etching process were the aim of this work. An areological system was composed of PAPVD-Arc coat and nitriding coats and surface substrate. The X37CrMoV5-1 steel was used like basic surface substrate in a large majority of synthesis process. Process depositions were also made onto constructional steel and carbon steel.

2 EXPERIMENTAL

In our investigation we carry out a lot of gas nitriding process of alloy steel, X37CrMoV5-1 steel, constructional steel and carbon steel. A lot of processes PA PVD-Arc were made during our researches. In PA PVD-Arc process, it ion etching stage we deposited a Cr, Ti, V and TiAl thin coats. In other process of PAPVD-Arc were obtaining a CrN, TiN, TiAlN and VN coats onto nitrited steels.

3 RESULTS

The structure of nitriding layer has a huge influence on the nitriding layer PAPVD-Arc coat composite properties. In the subsurface area of nitridig layer has produced a thin mix layer of iron nitrides żelaza ε-$Fe_{2\text{-}3}N$ and γ'-Fe_4N or there is no nitrides on the surface, than in this case there is only diffusion area. The ion etching process in a PAPVD-Arc synthesis process of thin coats is unfaboravle because of appearance of white layer in subsurface area. Ion etching could caused layer decomposition which is very unfavorable phenomenon, the is a denitriding surface which arrived to obtain a black area composed only pour iron. In many authors opinion [1] the necessary condition to receive a good adhesion of

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Title: INFLUENCE OF ION ETCHING IN LOW PRESSURE ARC DISCHARGE IN PLASMA ON DUPLEX COAT ADHESION PRODUCED BY GAS NITRIDING AND PA PVD-ARC PROCESSES
Authors: M. Betiuk, J. Michalski, K. Burdynski, P. Wach, A. Nakonieczny
Topic: C – Surface Engineering
Presentation type: Poster

PAPVD coats to nitriding surface is caused by appearance of surface only Fe(N) phase.
The micromechanical properties and adhesion of deposited coats in our areological system were realized onto WCL steel. Adhesion of these areological systems was executed by root furrow observation. The CrN and TiN coates obtained onto nitrided steel surface (polished before PVD process) have the highest quality (**Figure 2**).

4 CONCLUSION

- The Cr ion etching of nitrited coats lead to produce a sublayer. A sublayer caused a good connection between deposited coat and surface.
- There is no influence of ion etching by Cr ions of denitrided layers.
- The hardnes of areological system X37CrMoV5-1 steel /nitriding layer/pvd coat has a almost twice higher hardness than X37CrMoV5-1 steel after quenching and tempering.

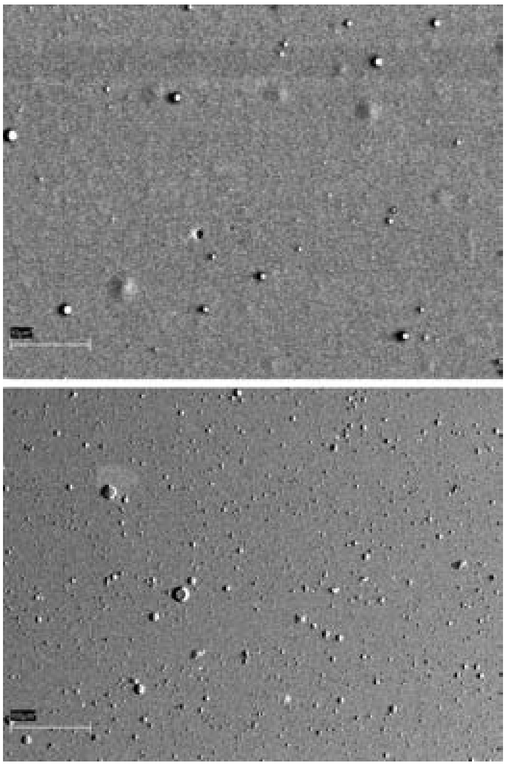

Figure 1: The structure of polishing and nitriding X37CrMoV5-1 steel after ion etching: a) Cr ions (marker – 10 μm), b) Ti ions (marker – 10 μm)

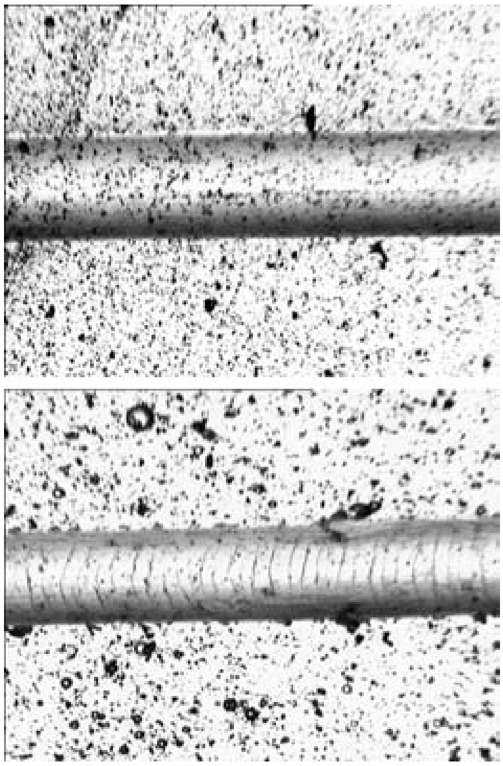

Figure 2: Scratch test on the PVD coat obtain on the nitrided steel: a) CrN coat, b) TiN coat

- The CrN coat deposition caused increase of wear resistance steals compared with basic state and state after nitiding.
- The result of our investigation is that a CrN coat has a higher plastic than TiN coat because CrN coat hasn't have in structure no cohesion cracks.

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STATE OF THE ART DEPOSITION TECHNOLOGIES AND COATINGS FOR TOOL AND DIE APPLICATIONS

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ABSTRACT

In order to meeting the increasingly stringent demands of the tool and die market, new coating materials and deposition techniques must be developed. Specifically, coatings which prevent adhesive and abrasive wear and which increase thermal fatigue resistance must be developed to increase die life time. PVD aluminum oxide coatings, which can be deposited at temperatures between 350-600°C, are now available to fulfill these needs. For tooling applications, such as punching and forming, coatings such as CrN and DLC can be applied to resist cold welding and to improve part finish. A summary of available coatings and applications will be presented along with cutting and die cast test data. All coatings have been deposited with hybrid technology in Hauzer Flexicoat 1000/1200 machines.

Keywords: PVD coatings, die casting, oxides

1 INTRODUCTION

Traditionally, cathodic arc based coatings, such as CrN, AlTiN and TiN have been used to prolong tool life and to increase productivity in the tool and die industry. These coatings are industrially viable and widely used. However, in order to further increase productivity, combined deposition technologies must be employed to develop new coatings for specific applications. The coating combination AlTiN + Al_2O_3 has been shown to reduce wear and to improve thermal fatigue resistance in both cutting tool and metal die casting applications. In this process AlTiN is deposited by cathodic arc deposition and the Al_2O_3 coating is produced by reactive sputtering. DLC is another combination coating, consisting of a metal based adhesion layer and an intermediate transition layer, which is suitable for forming and punching applications. This materials system combines the toughness of CrN with the low coefficient of friction/low chemical reactivity of DLC. In this case, the CrN layer can be deposited by either cathodic arc or sputtering, the WC:h layer is deposited by sputtering, and the DLC layer can be deposited by either PACVD or sputtering. An emerging technology, High Impulse Magnetron Sputtering, is now under investigation. With this technology, new coatings, such as amorphous TiN coatings will be explored on an industrial production scale.

The material properties for new coatings and several results from application tests will be presented. The possible deposition techniques and system configurations will also be discussed.

2 EXPERIMENTAL DETAILS

All coatings have been produced on a Hauzer Flexicoat 1000/1200 machines. The system configurations are listed below:

AlTiN+ Al_2O_3
- 3 x 4 CARC cathodes
- 1 x UBM sputter cathode with t-mode control for high deposition rate of oxides
- Plasma etching source
- Coating volume – 650mm diameter x 650mm height

CrN+WC:h+DLC
- 4 x UBM sputter cathodes
- Plasma etching source
- Coating volume – 650mm diameter x 850mm height

For all processes, the parts are put under vacuum, heated and plasma etched. For AlTiN+Al2O3 coatings, AlTiN is applied via cathodic arc deposition and the Al_2O_3 is deposited via reactive magnetron sputtering deposition.

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For CrN+WC:h+DLC coatings, CrN is deposited via cathodic arc or reactive magnetron sputtering, WC:h is deposited via reactive magnetron sputtering and DLC is deposited via PACVD.

3 RESULTS, APPLICATIONS AND DISCUSSION

A particular application of interest for Al_2O_3 coatings is aluminum die casting. The major problems associated with this application are the adhesion of the cast aluminum material to the die, changes in die dimensionality due to abrasion and thermal cracking in the die material itself. It was found that the Al_2O_3 coating could prevent or reduce the occurrence of all three problems.
CrN+WC:h+DLC coatings lend themselves well to applications where operating temperatures are somewhat lower (under 300C) and where adhesion of the workpiece material is a problem. Tool life as well as part finish can be significantly improved with such coatings.

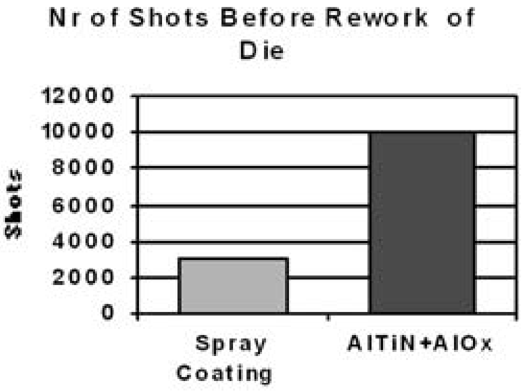

Figure 2: Aluminum die casting results /Diesel Injection pump housing

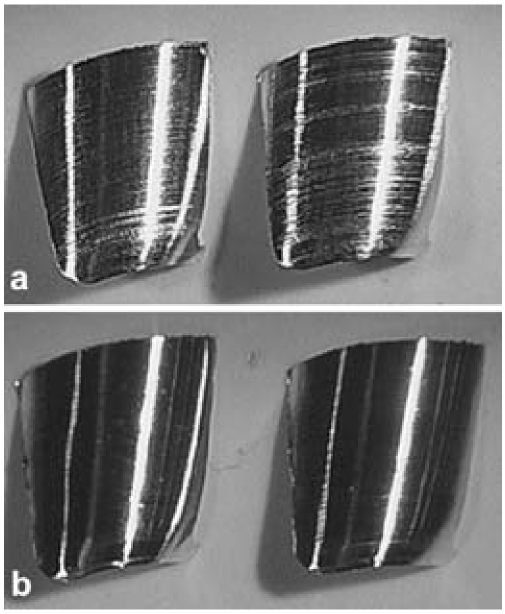

Figure 3: Cutting chips from dry Al machining (a – uncoated tool, b – Cr+WC:h+DLC coated tool)

4 CONCLUSION

The need for new coatings deposited via hybrid technologies such as cathodic arc, sputtering and PACVD is evident in the market. Two of these new coatings which are industrially available, AlTiN+ Al_2O_3 and CrN+WC:h+DLC, have shown much potential for the tool and die industries. In addition to these, other novel oxide, nitride and carbon based coatings are being developed. New technologies which allow for the development of new crystal phase structures within the existing coating base are also under investigation. These new coatings will have increasingly better corrosion resistance as well as unique mechanical properties.

DEVELOPMENT OF OPTIMAL PVD NANO-COMPOSITE COATINGS FOR ALUMINIUM ALLOY DIE CASTING APPLICATIONS

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ABSTRACT

In aluminium die casting, tools are exposed to erosion, corrosion and soldering due to the frequent contact of the tool surface to casting alloy, to heat checking and gross cracking due to thermal fatigue and to oxidation due to high pouring temperatures.

In the present paper a set of CrAlSiN coatings with increasing Al contents were deposited on a typical hot working tool steel to evaluate the influence of the coating composition on the hardness and the specific resistance to cyclic immersion in molten aluminium alloy. In such environment a three times lifetime was achieved if compared with uncoated steel.

Keywords: PVD nanocomposite, aluminum die casting, thermal fatigue resistance, aluminum soldering

1 INTRODUCTION

The durability of material, including both metals and ceramics, exposed to molten aluminium alloy is an important concern in many engineering applications since molten aluminium is one of the most aggressive metals to a number of materials. Important fields examples are die casting, containment of liquid aluminium and semi solid processing. Molten aluminium can cause considerable corrosion of handling, container materials and die castings tools [1-2]. In particular, the gradual destruction of die surfaces during service decreases casting piece quality and limits die lifetime [1-4].

To prevent the failed causes of die casting tool there are many protection proposals. The proposals going from bulk thermal treatments to surface treatments such as nitriding, CVD and PVD coatings by sputtering or cathodic arc deposition [5-8]. As far as the corrosion protection is concerned the major part of coating studies focus on CrN, TiAlN and TiN with a great increasing interest for coatings such as TiAlSiN and CrAlSiN. If thermal stability and tribological properties are to be maximized different systems such as W_2N/ Si_3N_4, nc-VN/a-Si_3N_4, ZrN/Ni, TiAlYN and TiAlN/VN have to be preferentially considered [5-7]. Further to the chemical composition of coatings the multilayer arrangement permit to create a coatings with a mix of layers exhibiting different properties so as to prevent at the same time different damage sources [7-8].

2 EXPERIMENTAL SETUP

An AISI H11 (1.2343) tool steel was used as substrate material. Specimens of different shapes and dimensions were gross machined in the annealed state, vacuum heat treated (hardening at 1000 °C, 5 bar quenching in N_2, double tempering at 550 and 595°C, 45-46 HRC) and mechanically finished. Then cathodic arc physical vapour deposition (PVD) process was applied to deposit multilayer coatings. A two cathodes Platit PL-55 prototype unit equipped with the innovative Lateral Arc Rotating Cathodes (LARC®). The multilayered structure was selected due to the crack deflection inherent features of such structure. Actually in Al-alloy die casting tool service thermal fatigue cracking (both heat checking and edge gross cracking) is highly likely to occur and as consequence severe surface damaging is also very frequent. The deposited coatings are nanocomposite belonging to the $(Cr, Al)_{1-x}N_x/Si_3N_4$ system, which is an evolution of

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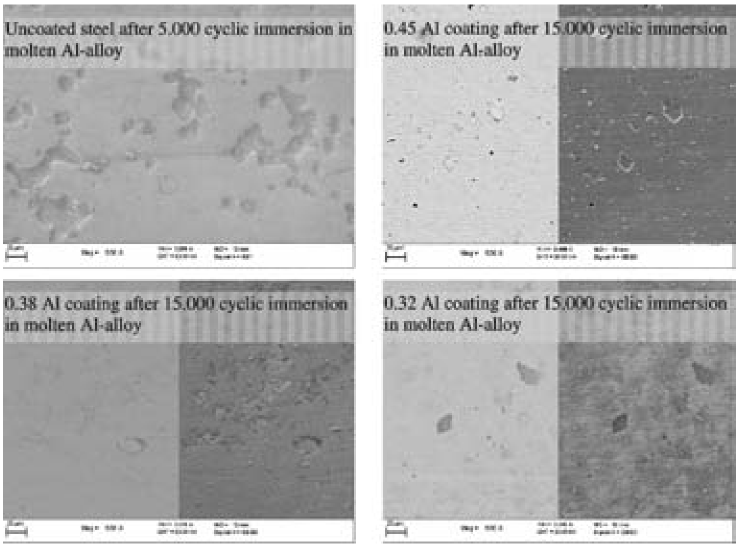

Figure 1: SEM observation of uncoated and coated surface subjected to cyclic immersion in molten alumiunium alloy.

the well known (Ti, Al)$_{1-x}$N$_x$/Si$_3$N$_4$ system. A multilayer structure, common to all deposited coatings, was created by alternating two layers enriched in Al or Cr respectively. On the top of such structure an external layer, differing basically on Cr to Al ratio, was deposited for the different coatings.

X-ray diffraction patterns were recorded for the different coatings identifying constitutive phases and measuring both the average crystallite dimension and the lattice parameters. Coating hardness of the different coatings were recorded performing microhardness indentation with increasing load (and penetration) and applying a work of indentation model. Finally a test of cyclic immersion in molten aluminium alloy was performed to evaluate the performances of different coated systems in simulated melt repeated contact. Internally cooled specimens were used for this test so as to enhance the thermal gradients through the wall. SEM analysis of top coated surface after increasing immersion cycles were recorded and compared with uncoated AISI H11 surfaces subjected to the same test.

3 MAIN ACHIEVEMENTS

In the deposited top coatings the atomic fraction of Al ranged from 0.15 to 0.45. The experimental plan explained that: the highest was the aluminum content in the top layer the highest was the lattice deformation achieved and the superficial hardness. Both these features continuously increases with the Al content.

The cyclic immersions in molten aluminium provided a very fast (just after 2.500-5.000 immersion cycles) and evident surface damaging of the uncoated steel. As far as the coated systems are concerned the poorly Al rich coatings (0.15-0.2 Al fraction) didn't give any particular gain in resistance to such environment. On the contrary the most Al-enriched coatings presented a clearly increased lifetime if compared to the uncoated reference steel (**Figure 1**). Actually the optimally coated specimens exhibited detachments-free surfaces for lifetimes ranging from 1.3 to 3 times (15.000 immersion cycles) the average lifetime of uncoated steel specimens.

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ENHANCEMENTS OF THERMAL FATIGUE RESISTANCE OF HOT WORKING TOOLING: THE ROLE OF MATERIALS, HEAT TREATMENTS AND COATINGS

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Wear and failure of hot working tooling involve a complex interaction between various mechanisms. In particular wear and heat checking cause failure due to different phenomena, for example to the so-called washout damages on working surfaces of high pressure casting dies, being attributed to erosion, corrosion and soldering caused by Al based alloys, or high stressing wear phenomena in forging processes; however thermal fatigue remains the most important failure mode in hot working applications. The suitable protection of working surfaces against thermal alternance effects will results in longer tooling life with consequent materials and energy saving with important environmental and economical benefits.

Different tooling materials and suitable heat treatments are available for the manufacturing of high performance dies and tools, both play an important role to contrast the thermal fatigue damages. The paper discuss the most important features related to the optimal choice among the available hot working alloys and the optimisation of their heat treatments. Moreover, further improvements of tooling performances can be attained thank to suitable coatings. In particular, the use of wear resistant thermal barriers applied by advanced thermal spray technologies can give a strong contribution to the increase of die life and performances. Different type of thermal sprayed coatings for the protection of hot working tools are here presented.

Experimental samples have been obtained by plasma and HVOF processes to test hardness properties, wear and heat checking resistances. The obtained results and the main microstructural features are here presented and discussed.

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NOVELITY IN DIFFUSION COATING TECHNOLOGY

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ABSTRACT

Diffusion forming of hard carbide layer is applied for many years, mostly in Japan (TDR- Toyota Diffusion Process) and in USA (TRD- Thermo Reactive Deposition) but not so much in Europe. Carbide layers on constructive parts and tools formed in diffusion process, especial vanadium carbide layer, have excellent tribological properties and some technical and economical advantages in comparison with carbide layers formed by CVD and PVD processes. Diffusion formed layers are thicker with very strong bond to steel substrate and are produced using significantly chipper equipment as for deposition coating. Origin process has one disadvantage: partial decarburization of steel surface beneath the carbide layer reducing hardness and loading capacity of parts surface. These disadvantage are eliminate by developed and patented duplex process introducing previous carburising to prevent decarburization and to form case hardened under layer beneath the carbide layer.

Keywords: diffusion, carbide layer, vanadising, duplex process, tool steels, wear resistance

1 INTRODUCTION

The quality and life time of tools and dies is an important factor in production, particularly in large batch and sophisticated productions. For a large number of years, considerable efforts have been put into attempts to increase wear resistance and service life by using different technological procedures. Attempts have been made in the direction of the application of higher quality materials and adequate heat treatment, and in the direction of the application of different procedures of modification and coating of surfaces in order to increase durability. Surface layers differ from the treated base material with respect to the chemical composition, microstructure, crystal lattice and other physical and chemical properties which result in different properties in exploitation. Among the surface modification and coating procedures, the treatment of chromium plating and the thermo-chemical treatment like nitriding are rare applied, and lately, it has been the case with the physical vapour deposition–PVD (PACVD) procedures (TiN, TiCN, TiAIN, CrN). In addition to one-layer coating (e.g. TiN) and multi-layer coating (TiCN, Al_2O_3, TiN), duplex processes, such as ion nitriding + PVD TiN, are also applied. Using PVD processes, thin and hard layers are formed on high precision tools since the preceding heat treatment has been carried out at the temperatures which are higher than the temperatures at which the PVD processes are carried out. For some time now, concurrently with the PVD processes, diffusion coating processes have also been applied. These processes have their origin in the Toyota Diffusion Process [1] and in several patents [2,3] and they have some technical and economic advantages over the PVD processes, but also some limitations in application. The advantages of diffusion coating processes are that they generate layers of greater thickness and greater hardness, as well as of superior tribological properties, higher resistance to abrasion and tribo corrosion. A drawback or a limiting factor of this process is the application on parts with close dimensional tolerances. As the process is performed at high temperatures causing a change in the microstructure of the substrate, the consequence is that dimensions are changed and a layer is generated on the machine parts surface. Thus, dimensional changes are higher than with the application of PVD processes. Diffusion formation of carbide layers is typically used in the same applications as the PVD (PACVD) coatings, but there are some applications where diffusion formation of carbide layers has proven to be superior: aluminium and zinc die casting, components (moulds, cores, etc.), hot forging dies and tools for stamping and forming stainless steels. Generally, a layer with the

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thickness in the range from 1 to 20 μm and the hardness from 3200 to 3800 HV is produced.

2 DIFFUSION FORMATION AND PROPERTIES OF CARBIDE LAYERS

Diffusion formation of a carbide layer on the surface of steel has a growing application in industry. This is due to the fact that relatively low investment costs for equipment are needed in order to enable the carrying out of the process and to obtain carbide layers of high hardness (from 3200 to 3800 HV) and of high abrasion and tribo corrosion resistance. Vanadium carbide layer has high thermal stability, more than 800 °C. The process is most often carried out in a salt bath of an appropriate composition, with the addition of one of carbide-forming elements (V, Cr, Nb, W, Ti). A carbide-forming element (e.g. vanadium, V) reacts on the surface of steel with carbon (C), which is at high temperatures of the process (» 1000 °C) atomically dissolved in austenite, thus forming a very stable V_8C_7 carbide or some other carbide (NbC, Cr_7C_3, WC, TiC) [4,5]. The carbide layer grows on the surface in the diffusion process of carbon from the surface layer of steel towards the carbide layer, and through it to a newly forming surface where it reacts with the carbide-forming element, a smaller amount of the carbide-forming element diffuses through the forming carbide layer into the surface layer of steel. In addition to high temperature, the rate of carbide layer formation is greatly affected by the chemical composition of steel. Experimental results obtained at the same time confirmed the developed mathematical models of the pattern, and a computer program, enabling the determination of technological parameters of the vanadising process in order to achieve a required thickness of the layer for a chosen steel type [6], was also developed.

3 APPLICATIONS OF VANADISING PROCESS

Vanadium carbide, V_8C_7 has a cubic lattice of high hardness (from 3200 to 3800 HV) with very good tribological properties. Abrasion resistance is very high because the vanadium carbide layer is harder that most hard mineral materials. Tribocorrosive adhesion resistance is very high as the layer is very stable and chemically resistant to aggressive agents (NaOH, HCl, etc.). High hardness of the carbide layer indicates that the layer is brittle and therefore the resistance to the surface fatigue is reduced. A strong metallurgical bound between the carbide layer and the base material (steel substrate) gives a good adhesibility of the surface layer to the substrate. Even a considerably greater thickness of hard surface layers of 5-20 μm, in comparison with the thickness of PVD layers of 1-3 μm, can be achieved. The industrial application of diffusion vanadising on a larger scale started in Japan (Toyota Diffusion Process) and then has spread to other industrially developed countries due to excellent tribological properties of vanadium carbide surface layers. The application of vanadising on structural elements and tools in different branches of engineering contributes to a significant increase in their durability in exploitation. The application of vanadising on the manufacture of tools has produced excellent results with numerous tool types which are subjected to the adhesive and tribochemical wear mechanism in their exploitation. For the abrasive wear mechanism, vanadising is the right choice for treating the parts of tools that are exposed to wear by hard abrasive particles. Surface layers of vanadium carbide extend the tool life by several times. Typical examples are tool parts for powder compacting and extruder worms. Regarding the adhesive wear mechanism, vanadising is applied to the tools for metal forming processes such as extrusion, forging and drawing. As far as the tribocorrosive wear mechanism is concerned, the vanadium carbide surface layer efficiently protects the tools used in very aggressive media, even in HCl and HNO_3. The carbide layer is particularly useful in reactive soldering for the tool parts for aluminium die casting. The process has a broad range of applications, from feeding attachments to the forming of punches and dies, and provides a considerable performance improvement. The largest industrial application is punches for piercing and extrusion. The application on various die steels is also evident. However, vanadium carbide on cemented carbide dies is also utilized with a notable improvement of die life. The ability to change from cemented carbide to cold work die steel or high speed steel, and from high speed steel to cold work die steel, has been realized in various punches and dies. The process is also successfully utilized in the forging of stainless steel, ball bearing steel and non-ferrous metals. Dies used in the warm forging of stainless steel are also treated by this process. The vanadium carbide coating is continuously applied even for hot forging dies. The life time increases of several times due to the elimination of galling problems. As a result, a great saving on die consumption has been achieved in industry. Additional advantages, other than savings on die consumption, were in some cases far greater.

4 CONCLUSION

Diffusion formation of hard carbide layers offers a new possibility in tool processing which increases wear resistance considerably, thus enabling a longer life in exploitation and consequently a more economic reason. Different thermo chemical processes produce different surface layers with adequate properties for specific applications. On the basis of the knowledge of the basic physical process pattern, an adequate thermo chemical treatment process has to be chosen for adequate exploitation conditions. With diffusion formation of hard carbide layers, the choice of substrate, i.e. steel, is of major importance. In addition, due to high temperatures of the process, dimensions are changed because of subsequent hardening and additionally due to formation of a carbide layer. In the cases of close dimensional tolerances, a trial treatment needs to be performed in order to correct dimensions in the way as to compensate for the changes of dimensions during the forming of hard carbide layers. Processes of diffusion formation of carbide layers cannot replace the PVD process when it comes to cutting tools, milling cutters and reamers, which require close dimensional tolerances. The results obtained in the previous research and technological improvements of the duplex process, together with the developed computer programs, have contributed to the improvement of the original process of carbide layer formation.

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THERMAL STABILITY AND AGE HARDENING OF METASTABLE HARD COATINGS

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ABSTRACT

Metastable phases have attracted increasing interest in modern development of hard coatings as they offer a broad range of unique properties or property-combinations which allow for increased life times and application fields of e.g., tools and components. Here, the model systems Ti–Al–N and Cr–Al–N are ideal candidates for systematic investigation of their chemically determined phase stability, structure, elastic constants, and thermal stability. Especially plasma-assisted vapor deposition techniques with their extremely high cooling rates enable the preparation of coatings containing phases beyond their thermodynamic stability range. During thermal treatments above the deposition temperature (e.g., during operation) these supersaturated phases undergo decomposition processes to reach equilibrium. While for Ti–Al–N the formation of the stable hexagonal (h) AlN occurs via c-AlN this intermediate step can not be observed during decomposition of Cr–Al–N, see **Figure 1**.

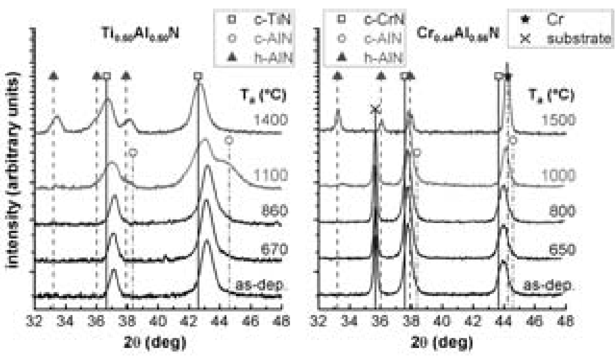

Figure 1: X-ray diffraction patterns of $Ti_{0.50}Al_{0.50}N$ and $Cr_{0.44}Al_{0.56}N$ after annealing at temperatures T_a

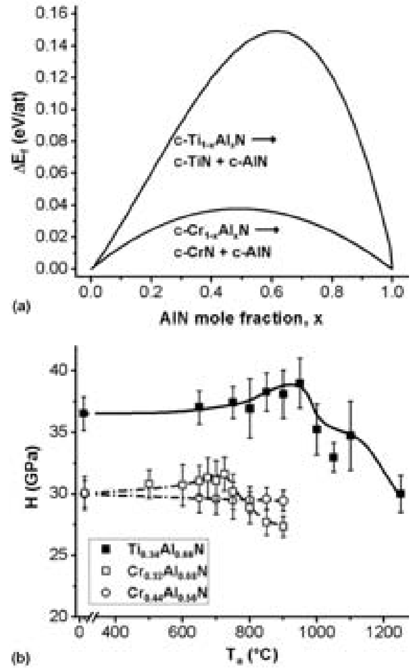

Figure 2: **(a)** Energy of formation difference (ΔE_f) for the separation of c-$Ti_{1-x}Al_xN$ into c-TiN and c-AlN and the separation of c-$Cr_{1-x}Al_xN$ into c-CrN and c-AlN as a function of AlN mole fraction. **(b)** Hardness (H) of $Ti_{0.34}Al_{0.66}N$, $Cr_{0.32}Al_{0.68}N$ and $Cr_{0.44}Al_{0.56}N$ as a function of annealing temperature T_a.

Based on differential scanning calorimetrical studies and a combination of *ab initio* and continuum mechanical based calculations the differences for both coating systems can be understood. The change of formation energy (ΔE_f) for decomposition of supersaturated c-$Ti_{1-x}Al_xN$ into c-TiN and c-AlN has a maximum of ~0.146 eV/at (28.18 kJ/mol) at x ~0.61 which is about four times larger than the maximum of the enthalpy change of ~0.037 eV/at at x ~0.47 for the decomposition of c-$Cr_{1-x}Al_xN$ into c-CrN and c-AlN, see **Figure 2a**.
This huge difference in enthalpy change explains that c-AlN forms as a metastable phase during decomposition of c-$Ti_{1-x}Al_xN$ before phase transforming into the stable constituents c-TiN and h-AlN, whereas no metastable

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Presentation type: Oral

c-AlN forms during decomposition of c-$Cr_{1-x}Al_xN$. As the thereby formed domains and phases act as additional obstacles for dislocation movement hardening processes are observed during thermal treatment. Based on the formation of coherent cubic nanometer-size domains prior to the precipitation of h-AlN these hardening processes are more pronounced for Ti–Al–N as compared to Cr–Al–N, see **Figure 2b**.

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HARD COATINGS FOR DIES AND MOULDS

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Hard Coatings are in place in present manufacturing technologies. Particularly for metal cutting applications they captured a wide market. But also in forming uses hard coatings provides considerable results. Anyhow, the spreading of HC in this field is still smaller, due to the increased complexity of the application area.

Alone the demands to the surface structure of plastic moulds covers the range from high polished surfaces up to structures "destroyed" by EDM or chemical processing. To support the microstructure of these surfaces after coating needs a plenty of know how and technology.

The same applies accordingly for sheet metal forming such as stamping, folding or deep drawing. Like the processing of moulds a lot of different machining technologies where in use, in particular EDM.

Die casting Aluminum is also a really complex operation. Besides the accepted needs die casting Aluminum askes for enhanced chemical stability and the increased resistance against dynamic fatique of the interface.

One of the most important factors for successful coating are surfaces adapted to the expected loads. EDM machined surfaces of plastic moulds for example can be coated with only minor modification to preserve the desired microstructure. To prevent punctual overload in stamping and folding operations however it is necessary to flatten surface features, as deformations caused by peak overloads result in cracks in the coating and subsequently in chipping. The choice of the base material, the structure of the tool surface and mechanical and chemical properties of the layer have to be considered together to arrive at an optimal solution for a specific application. Disregarding this mutual interdependence leads to less than satisfying results.

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POSSIBILITIES OF STRENGTHENING OF DIFFUSION NITRIDED LAYERS BY SHOT-PEENING

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ABSTRACT

The paper presents the results of examinations of microstructure and mechanical properties of the surface layers produced by duplex technique combining controlled gas nitriding with shot-peening.

Keywords: shot-peening, nitrided layers, hardness, residual stresses

1 INTRODUCTION

Dynamic surface deformation by shot-peening is a technology widely used in industrial practice in order to improve the mechanical properties of machine components [1,2]. In the case of materials with enhanced hardness, such as nitrided layers, the process of mechanical strengthening is not well known and complicated. However, increasing demands related to mechanical properties of machine parts make a modification of actuel technologies, necessary.

The aim of this project was to test out the possibility of strengthening of nitrided layers by dynamic surface deformation, i.e. shot-peening, from the point of view of enhancing their service properties.

2 EXPERIMENTAL

Investigations were carried out on specimens made from 38HMJ (EN: 41CrAlMo7) grade steel. The specimens were quenched and tempered to a hardness of 45 HRC.

Controlled gas nitriding process was conducted at a temperature of 470°C in NH_3/NH_{3diss} atmosphere for 16h. Diffusion layers were obtained without external layer of iron nitrides. The surface hardness was approximately 1150 HV0.5.

Shot peening was carried out with the aid of a prototype stand, developed and built at the Institute of Precision Mechanics, enabling this treatment with the application of low shot velocities.

Shot-peening of hard nitrided layers without compound layer is a new trend and calls for the development of totally different parameters from those used for treating typical applications. The aim of the investigations was the optimum selection of type and size of shot, shot peening velocity and time of application.

3 RESULTS

Observations of the topography of sample surface after nitriding and after the 2-stage treatment of combined nitriding and shot peening were carried out with the aid of a scanning electron microscope and presented in **Figures 1-2**.

Results of HV0.5, HV1, HV5 and HV10 hardness measurements are presented graphically in **Figure 3**. It was determined that for all the versions investigated there occurs an increase in hardness after shot peening, as measured under all test loads used.

As the result of the shot-peening carried out, a rise occurred in the residual compressive stresses in the superficial layer – see **Figure 4**.

4 CONCLUSIONS

The examinations carried out indicated the possibilities of raising the mechanical properties of diffusion nitrided layers by the implementation of dynamic surface strengthening treatment (shot-peening).

By the implementation of low energy shot-peening (i.e. with low shot velocities) a rise in the hardness and an increase in the state of compressive stresses in nitrided layers were obtained.

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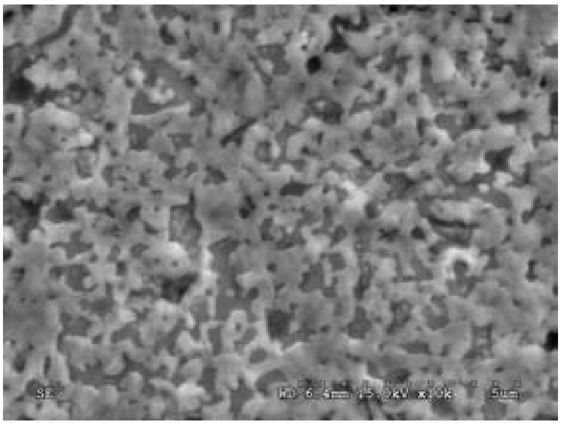

Figure 1: Surface morphology of nitrided layer produced at 470°C, for 16h

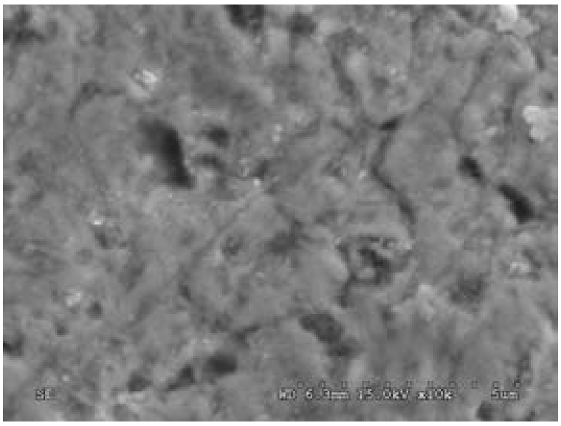

Figure 2: Surface morphology of nitrided layer after shot-peening treatment

It was also established that when small shot velocities are employed, no significant damage occurs to the surface of nitrided layers.

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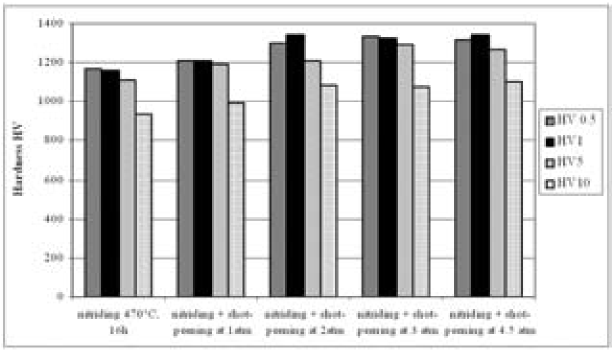

Figure 3: Surface hardness HV0.5, HV1, HV5, HV10 for nitrided layers and surface layers after duplex treatment /nitriding+shot-peening/

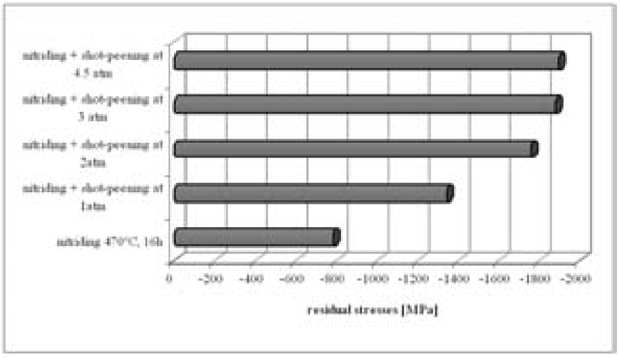

Figure 4: Residual compressive stresses values in the surface layers

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NITROOXIDATION OF TOOLS MANUFACTURED FROM HIGH-SPEED STEEL

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ABSTRACT

This article presents the technology for nitrooxidation of tools manufactured from high-speed steels. There have been discussed the current ways of fabricating the nitrooxide layers, which have been developed in IMP. The examples of applications in the industry are shown. The morphology of the fabricated layers and operational properties of the nitrooxidated tools are demonstrated.

Keywords: nitrooxydation, technology, high-speed steels, tool steels, applications, characteristics

1 INTRODUCTION

In the Institute of Precision Mechanics (IMP) first original technology of nitrooxydation gas treatment for high-speed steels was developer, under the name of oxynitriding. His treatment entered the industry at the beginning of 1970. Since 1978, fluid modification of his process, known as fluidal nitrooxydation, TERMO-FLUID, has been introduced for the industry and is still in use. At the end of 1990, on the order from one of the tool manufacturers, the modified version of the gas treatment for high-speed steel tools was developed and is known as nitrooxydation AG.

2 EQUIPMENT

Gas Nitooxydation (oxynitriding)

For oxynitriding the low temperature soaking pits were used. These pits had leaktight retorts, which were inserted into the pits only during treatment process. For oxynitrinding the low temperature pits type PEH-2B and PEGat-700 manufactured by LZT Elterma were used. Retorts for the PEH-2B furnaces with workspace capacity about 120 dmł, stock instrumentation, WAP generators and PGO culverts were all constructed and produced by IMP.

Fluidal Nitrooxydation TERMOFLUID

Fluidal nitrooxydation was developed as TERMOFLUID system, which covers both technology and equipment. For fluidal nitrooxydation low temperature fluidal furnaces were used, of the type UF2-1 and UF4-1, both constructed and produced by IMP. Also, there were used furnaces reconstructed to fluidal, from the base of crucible furnaces PET-35 and PET-50 types, constructed by LZT Elterma. At the moment, for fluidal nitrooxydation fluidal furnaces FP-700 type and fluidal furnace-tubs FPW-700 type are used. They have workspace capacity from 35 to 100 dmł and working temperatures about 700 °C. To control of the furnaces and the process, the sets of swithboxes FSE and FSG are used, they can be controlled manually or automatically. Automatic control is realized with the microprocessor controller with the possibility to visualize and registry of the process parameters.

Nitrooxydation AG

For AG nitrooxydation low temperature atmosphere soaking pits GOAT-700 type with retractable retort are used. These pits are manufactured by IMP. GOAT furnaces have the up-to-date, light construction based on fibrous heat insulation materials. Retorts are equipped with the efficient fans to ensure the atmosphere's circulation, which provides uniform distribution of temperature in the workspace. The pits are built with retorts, that provide workspace capacity from 10 to 350 dm^3. To control the pits and the processes, there are used sets of swithboxes SE +SG manufactured for both automatic and manual control. In the manual control version, only temperature of the nitrooxydation is controlled automati-

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cally and the rest of the parameters; time and intensity of the atmosphere factor's flow are controlled manually.

3 TECHNOLOGY

Nitrooxydation can be performed on available tools after a typical heat and mechanic treatment.
In the gaseous form of nitrooxydation, the stock, after precise degreasing, is loaded to the retort outside of the furnace, or inside of the cold furnace. Before heating the stock to the process temperature it is very important to rinse out air from the working space. After sufficient rinsing out the retort and heating up to the process temperature, the atmosphere is supplied. After the process time the supply of the atmosphere stops and after rinsing out the rest of the atmosphere and cooling down the retort with stock, the stock is taken out into air. Different from the first developed nitrooxydation, in the AG nitrooxydation phase of nitriding and phase of oxidizing were separated, to eliminate generation of ammonia water in the waste gases. In the process of nitrooxydation AG the waste gases emitted to atmosphere contain inactive components: nitrogen, water vapour and carbon dioxide. Figure 5 shows the sight of a pallet with screw-taps after taking out from a retort in the nitrooxydation AG process.
Fluidal nitrooxydation TERMOFLUID is realized in completely different way, using free access to the furnace's workspace with atmosphere, similar to bath treatment.
The process of fluidal nitrooxydation uses cheap atmosphere generated from the mixture of ammonia and air, which works the same as the more expensive atmosphere generated from a mixture of ammonia, nitrogen and separated oxidizer. It is possible because in the bed explosive combustion of the gases does not occur and it is possible to safely combine ammonia and air in any temperature.
So for fluidal nitrooxydation the stock with appropriate instrumentation is put into the bed, which is fluidized with atmosphere and after all processing phases the stock is moved outside of the furnace to free air.
The last element of all of the discussed types of treatment is the covering of the tool's surfaces with the similar to oil, anti-corrosion preparate, which increases the corrosion resistance and intensity of the dark brown color.

4 CHARACTERISTICS OF NITROOXIDIZED TOOLS

As a result of nitrooxydation the diffusion layers form themselves on the high-speed steel tool's surface. They are composed of the outermost oxide zone and deeper positioned zone enriched with nitrogen. The typical microstructure of the diffusion layer fabricated on the high-speed steel SW7M as a result of nitrooxydation in the temperature of 520 °C composes of oxides zone A and zone enriched witch nitrogen B. The outermost oxide zone (A) is built from Fe_3O_4 and it has two sub zones. The outermost sub zone (A1) is porous and is generated on the tool's surface. The innermost sub zone (A2) is compact and it appears directly under the tool's surface.
Fabricated diffusion layers are characterized with improved surface hardness and increased temperature emissivity and friction resistance. As an effect the tool's durability increases due to increased wear resistance and corrosion resistance. Simultaneously the dark brown surface layer of the oxides gives the aesthetic looks to the tool's surface.

MECHANICAL AND PHYSICAL PROPERTIES OF TOOL AND DIE MATERIALS

2nd INTERNATIONAL CONFERENCE ON HEAT TREATMENT AND SURFACE ENGINEERING OF TOOLS AND DIES
Bled, 25–28 May 2008

TOOLS MATERIAL BEHAVIOR AT ELEVATED TEMPERATURES

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ABSTRACT

In this paper experimental data related to material mechanical properties as well as material behavior at elevated temperatures are obtained. Tensile tests at different elevated temperatures are carried out and also for some of these temperatures one-dimensional short time creep tests for different constant loads are made. Experiments are made using modern computer controlled 400 kN materials testing machine equipped by high temperature furnace and the corresponding high temperature extensometer. Some of material creep responses are also numerically modeled. The curves of mechanical properties and creep responses, engineering stress-strain diagrams, as well as curves of specimen's elongation and reduction in area are also presented.

Keywords: tool steel, mechanical properties, short-time creep, elevated temperatures

1 INTRODUCTION

Knowledge of material mechanical properties and behavior at certain environmental conditions is of great interest for both structure design and its manufacturing procedure. In present time exists a large number of material types, design procedures, manufacturing processes, service life conditions, etc. Thus the role of material specialists in all of mentioned activities is complex, varied and changing [1].

Mechanical properties are function of temperature [2, 3]. Therefore, it is very important to have data about these properties at tool working temperature. Also, creep resistance of these steels is of utmost concern.

2 EXPERIMENTAL SETUP

All the mentioned tests, uniaxial ($\sigma-\varepsilon$) tests and short creep tests are carried out using the mentioned material testing machine (Zwick Roell Z400E, 400 kN), equipped by the high temperature furnace Maytec HTO-08 900 °C and the high temperature extensometer Maytec LVTD.

Heat was generated by the heating elements for each of the three heating zones. Three internal thermal couples, each in one zone, are located inside the furnace to measure the air temperature. The temperature accuracy is very high, so the temperature difference between the zones and the required test temperature did not exceed 1 °C. The heating rate of the furnace is 15 °C/min. The gauge length of the high temperature extensometer was 25 mm.

The specimens were prepared according to the ASTM standard E8M-04, EN-10002-1:2001 [4]. Two types of testing procedures are carried out. The first one is the uniaxial tensile test made at room temperature and the uniaxial tensile test performed at elevated temperatures. Using such tests, material mechanical properties are determined. The tests are made according to the EN 10002-1 (ASTM E8) and EN 10002-5 (ASTM E21) standards. At elevated temperatures every specimen was heated up to a specified temperature. After the specified temperature had been reached, the test was not started until the temperature regarding the mentioned standards became stable. After that the tensile load was applied to the specimen. The obtained results are given as engineering stress σ (MPa) versus strain ε (%). Second testing procedure is the short time creep test. Several one-dimensional creep tests are carried out regarding to ASTM E139. Each of these creep tests is made under constant load at specified constant elevated temperature. The constant load level is selected as percentage of force $F_{0.2}$ corresponding to $\sigma_{0.2}$. Force $F_{0.2}$ is obtained as a product of yield strength $\sigma_{0.2}$ and initial area of specimen cross-sectional area A_0. The cross-sectional area A is

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Presentation type: Oral

taken as the initial one because its changes till yield point may be neglected.

3 CONCLUSIONS

Based on previously described experimental setup, evaluation of mechanical properties of a tool steel at elevated temperatures was carried out. In addition, short-time creep response is also given through the strain – time curves. This creep behaviour of tool steels is also numerically modelled.

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BEHAVIOUR AT ELEVATED TEMPERATURE OF 55NiCrMoV7 TOOL STEEL

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ABSTRACT

Mandrels used in ring rolling usually work under static and dynamic forces, and they are constantly subjected to shokc and vibrations. To investigate failure causes of these mandrels lab tests able to reproduce working conditions have been carried out as thermal fatigue and hot wear tests. As-delivered material is used to perform lab test but hardened and tempered samples are used too to compare different results.

Keywords: thermal fatigue, hot wear, tool steel, tribology

1 INTRODUCTION

Aim of present work is to investigate the behavior at high temperature of 55NiCrMoV7 steel used in mandrels for ring rolling. This tool is exposed to static and dynamic forces as shocks and vibrations. Hot wear, oxidation and thermal fatigue are also possible causes of failure or damage during service.

In this work samples required for lab tests were obtained from a broken mandrel (200mm diameter). Mechanical properties as hardness and resilience have been investigated and related to microstructure. Technological tests tailored to reproduce working conditions and damaging (as hot wear and thermal fatigue tests) have been carried out too, to investigate reasons of damaging.

The microstructure of the steel in the as-delivered state consisted of tempered martensite and bainite, the fraction of the last one increasing moving to the inner of the mandrel. As original material had too low hardness (34 HRC), because of the thermal softening occurred during service, a new set of samples has been obtained after hardening and tempering steel specimens to a higher hardness (41 HRC). Heat treatment comprised vacuum austenitizing at 870 °C for 1 hour followed by cooling in a controlled N2 atmosphere at 7 Bar. Two tempering were then carried out at 560 °C for 2 hours. After heat treatment the microstructure consisted entirely of tempered martensite. Tempering curves were also determined using different tempering temperatures (200 °C, 400 °C, 500 °C, 550 °C, 600 °C, 650 °C).

All tests performed at the University of Trento have the purpose to describe a sort of material history regarding mechanical properties, wear and thermal shock behavior and correlations between them to understand failures occurred to mandrels and to improve this material too.

Resilience and hardness of the material have been investigated and then lab tests to verify behavior of samples under hot wear conditions and thermal shocks conditions have been performed too.

2 EXPERIMENTAL SETUP

HOT WEAR TEST. Hot wear tests have been carried out to reproduce, as likely as possible, the damage mechanism occurring during actual ring rolling. Due to experimental limits however, the maximum temperature of the rolled material is lower than the real one. The test facility is an AMSLER tribometer with two shafts on which disc sample (40mm external diameter) is mounted with its counterpart (50mm external diameter). Wear test were carried out using a disc on disc configuration under rolling-sliding contact. As counterpart material a C40 plain carbon steel was selected, the temperature of which was raised up to 700 °C by means of an induction system. The temperature was controlled by an infrared pyrometer pointing on it. The total length of the test was 3 hours with stops at every hour to weight the sample. The wear curve, i.e., the curve reporting the cumulative mass loss versus the rolling distance, gave a linear trend. The wear rate (Wr, g/m) was determined as the slope of the line in-

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Presentation type: Oral

terpolating the experimental points of the cumulative mass loss. Data on friction coefficient were collected by a pc connected to the tribometer and the data acquisition is 2 points per second. Samples in the as-delivered state, as those after hardening and tempering, have been tested at two different temperatures, i.e., 600°C and 700°C. Wear and friction data have been correlated to microstructure, taking into account the wear mechanism acting during the test. Microstructural analysis carried out on the wear tracks revealed the occurrence of triboxidation accompanied by adhesion and even by abrasion. Results were also discussed in relation with old data obtained at the University of Trento on different materials.

THERMAL FATIGUE TEST. Thermal fatigue test facility consists in a rotating shaft where the sample, a disc like that used in the wear tests, is mounted on. The disc is heated by an inductor up to 670°C (±10°C), controlled by an infrared pyrometer; the shaft has a rotational velocity of 4 rounds per minute. The heated sample is quenched by a direct water jet at a flow rate of 2 liters per minute. Every hour (about 250 cycles) the sample was dismounted, mirror polished on a lateral surface, allowing the thermal radial cracks to be measured by means of an optical microscope. Following a standard procedure just adopted in the past at Trento University the following parameters were used to describe the thermal fatigue resistance of the steel: crack density ρ, mean crack length (lm), maximum crack length (lmax) and the so called pyrocracking factor (P = ρ x lm x lmax). A material with many but small cracks is more desirable than a material with few but long cracks and so tempered and not tempered material was investigated to show different behavior under thermal fatigue conditions.

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MODERN PRE-HARDENED TOOL STEELS IN DIE-CASTING APPLICATIONS

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ABSTRACT

Die-casting is an attractive method to mould metallic components. In such moulds the most common failure mechanism is heat-checking. Care must be taken by the steel producer to minimize this failure risk, i.e to control strength at elevated temperatures, thermal fatigue properties, thermal softening and impact toughness at elevated temperatures etc. New pre-hardened tool steels have been introduced on the market where control of the abovementioned properties has been taken in combination with elimination of the need of heat treatment in die/mould manufacturing; i.e a much faster die/mould manufacturing is possible.

Keywords: die-casting, heat-checking, thermal fatigue, thermal softening, impact toughness

1 INTRODUCTION

Pressure die-casting moulds are normally manufactured in W.Nr 1.2343/1.2344. A major drawback, however, in traditional mould manufacturing is heat treatment (quenching & tempering). Not only shape distortions then take place, but there is also a substantially cracking risk. Furthermore, the impact toughness, important to reduce the heat-checking risk, can vary due to differences in the heat treatment parameters. The new pre-hardened tool steel, TOOLOX 44, of the same hardness level as is used in die-casting, 45 HRC, is based on another alloying philosophy/steel production route and also heat treated to its final hardness by the steel producer; thereby, in die-casting, important properties such as impact toughness, tensile properties, thermal fatigue properties etc. can be carefully controlled. The new alloying composition also results in an improved heat conductivity, beneficial to reduce the heat-checking risk.

With pre-hardened tool steels moulds can not only be manufactured much faster, but also with more consistent and improved properties thanks to the pre-hardening of the new steel grades carried out by the steel producer.

2 STEEL PROPERTIES AT ELEVATED TEMPERATURES

By careful processing control the new tool steel has improved Charpy-V impact toughness, at room temperature as well as at elevated temperatures. A comparison to traditional grades generally used is given in **Figure 1**.

Also compressive yield strength at elevated temperatures is equal in the new pre-hardened tool steel and in the traditional grades as is demonstrated in **Figure 2**.

As is shown in the figures the properties of the new pre-hardened steel makes it very suitable in die-casting. Thermal softening shows the same softening characteristics as in W.Nr 1.2344 (45 HRC). Simulation of thermal fatigue has been carried out, T_{peak} = 710°C / T_{min} = 200°C, 10,000 cycles. No differences in heat-checking could be found between TOOLOX 44 and W.Nr 1.2344-ESR (45 HRC).

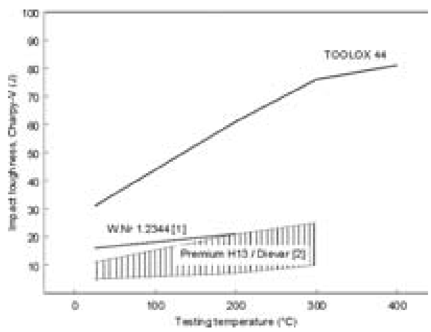

Figure 1: Charpy-V impact toughness in regular grades and in TOOLOX 44

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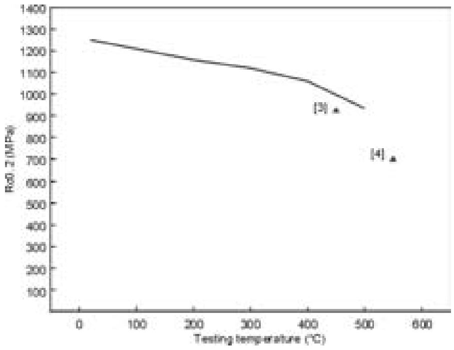

Figure 2: Compressive yield strength at elevated temperatures

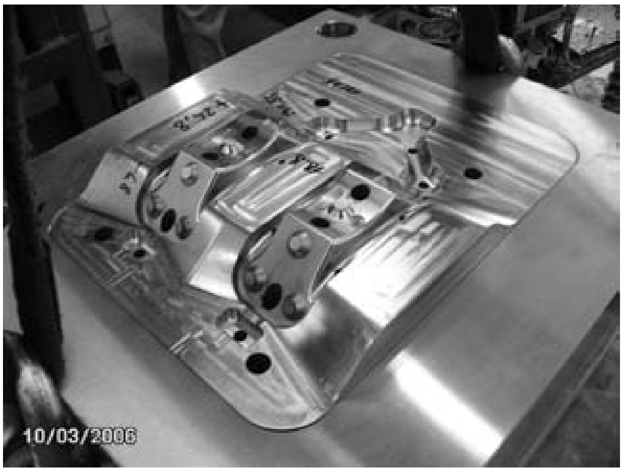

Figure 3: Aluminium pressure die-casting mould

3 MOULD MANUFACTURING

As the heat treatment step in mould manufacturing can be eliminated a mould/die can be manufactured in much shorter time when using a pre-hardened tool steel. The total manufacturing time when producing the mould shown in **Figure 3** was only 40%, 12 days instead of 30 days, of the time consumed when using W.Nr 1.2344.

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INNOVATIVE TESTING METHOD FOR THE EVALUATION OF THERMAL SHOCK AND MECHANICAL WEAR

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ABSTRACT

Hot working tools are subjected to a complex load collective. During the usage, the material has to sustain thermal fatigue and thermal shock, as well as mechanical and tribological load, resulting in abrasive, adhesive and impact wear. These kinds of wear are the main cause for tool failure, necessitating the replacement of the tools. As a consequence, an untimely failure of tools results in high costs, leading to a negative effect on product prices and quality. Therefore, the evaluation of fatigue occurring in hot working tools is inevitable for the prediction of life-time and can also be very helpful in improvement and invention of new tool materials and coating technologies.

Keywords: thermal shock, thermal fatigue, mechanical fatigue, wear

1 INTRODUCTION

Up to now, there exist only few standardised test methods for the investigation of wear of hot working tools that are able to simulate both mechanical and thermal fatigue. But with the presented testing apparatus it is possible to simulate the thermal shock behaviour, as well as the adhesive and abrasive load on the tested material simultaneously. Therefore a test bench was designed, by which both kind of wear can be tested on hot working tool material, which can be modified prior to testing e.g. by application of coatings or different heat treatments.

2 EXPERIMENTAL SETUP

The thermal load is applied on a disc-like rotating sample by a sudden local heating of the circumferential sample surface by means of an acetylene-oxygen burner. Afterwards, the material is quenched rapidly by water spraying. The mechanical load on the investigated material is granted by a disc roll that is pressed with constant pressure on the rotating sample. The disc roll is turning synchronously or in counter direction with the rotating sample, so that adhesive wear can be simulated. A principle of the apparatus for thermal and mechanical fatigue testing is shown in **Figure 1**.

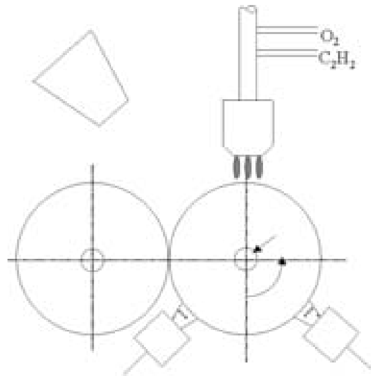

Figure 1: Principle of the apparatus for thermal and mechanical fatigue testing

3 RESULTS

Finally, the described testing method can be used for the investigation and comparison of different tool materials and coating technologies. An estimation of fatigue can be achieved after a few hours to avoid expensive and ex-

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tended test runs of real tools. Furthermore, a quantitative investigation of wear is possible, e.g. by means of counting the cracks and measuring their depth. Thus, the forecast of the tool's life-time, especially for drop forging die plants, can be improved. The possibilities of investigation methods and results available by this test method will be shown on hot working tool material with view to different nitriding treatments and surface engineering methods.

HEAT-RESISTANT CASTINGS IN CARBURISING FURNACE

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ABSTRACT

The present study discusses the problem of degradation of cast elements carrying charge in carburising furnaces. The wear and tear of castings, which finally results in decohesion of walls, is due to an effect of the carburising atmosphere and thermal fatigue.

Keywords: austenitic cast steel, thermal fatigue, failure, carburising

1 INTRODUCTION

Various accessories used to carry and transport the batches of charge in carburising furnaces (the charge-carrying elements) are mainly produced from austenitic cast steel. The conditions of their performance are directly defined by temperature variations during carburising and quenching of steel parts.

The performance life of the charge-carrying elements decides about the reliability, and hence, about the operating cost of a carburising furnace [1÷3]. Basing on the results of an analysis of the operating conditions, three groups of interrelated processes deciding about the wear and tear of these elements have been indicated [2,3]: high temperature corrosion, mainly carburising, thermal fatigue due to rapid heating and cooling of the charge, creep due to static loads, i.e. the weight of the charge and own weight.

This study gives the results of an analysis of the damages suffered by charge-carrying elements operating in chamber and pusher-type carburising furnaces. The analysis was made basing on visual assessment of the scrap.

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2 EXPERIMENTAL

The main charge-carrying element is a thin-walled openwork grate usually made from creep-resistant 36%Ni-17%Cr cast steel [1÷3]. It plays the role of a pedestal on which one can:

1. Place a fixture holding inside the parts to be carburised (**Figure 1a**).
2. Construct by means of auxiliary grates and supporting rods a structure which enables stable fixing of parts to be carburised (**Figure 2**).

On performance, the charge-carrying elements are exposed to heavy thermal shocks which, during cyclic operation, lead to damages caused by thermal fatigue. The fields of own stresses formed in castings are caused by temperature gradient which occurs in external and internal areas and on the cross-section of a single wall. An immediate effect of the thermal fatigue is deformation and cracks formed in the walls (**Figures 1b-1,2 and Figures 2-1,2**) and, at the final stage of degradation, local or total decohesion of casting (**Figure 2-1**). Due to this, the grate has to be withdrawn from use before its structure breaks down completely, otherwise a seizure may occur, followed by failure in furnace operation and rejection of the carburised parts [2].

The shocks of purely thermal nature affecting the casting are further aggravated by the aggressive effect of carburising atmosphere. The carburised layer formed in the sub-surface region of castings (**Figure 1b-2**) is characterised by the mechanical and physical properties definitely inferior in respect of the non-carburised core [2,4].

The process of degradation of the grate assemblies is much more complex than it is in the case of one single grate. The decohesion in the corners of a lower (base) grate (**Figure 2-1**) and in an upper – intermediate grate is the result of external bounds, i.e. of the reaction of constraints. Variations in mass and in the wall cross-sections are the reason why the time of their heating/cooling is different. From the point of view of the resistance to

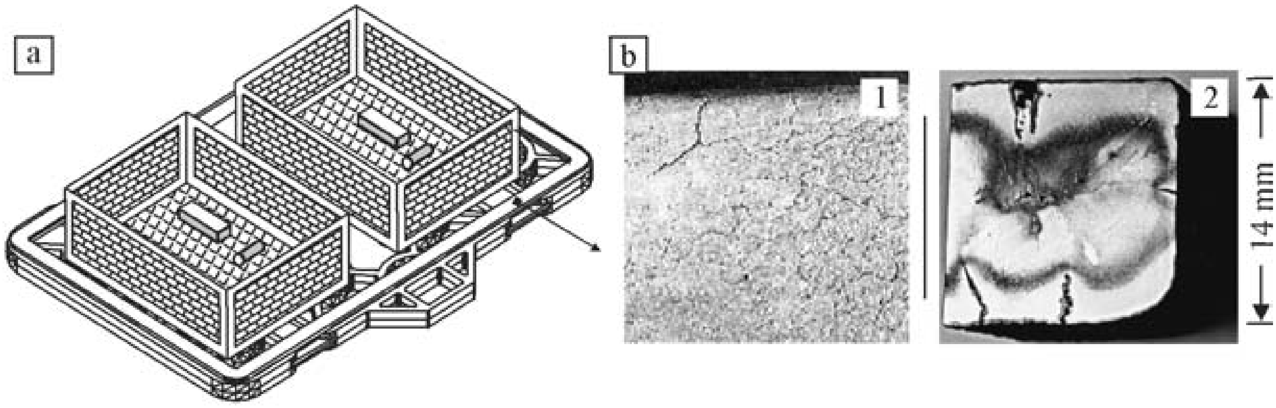

Figure 1: The grate from a carburising chamber furnace withdrawn from use: a) schematic diagram, b) surface cracks (1), macrostructure on the wall cross-section (2) with well visible carburised layer, cracks and axial porosity

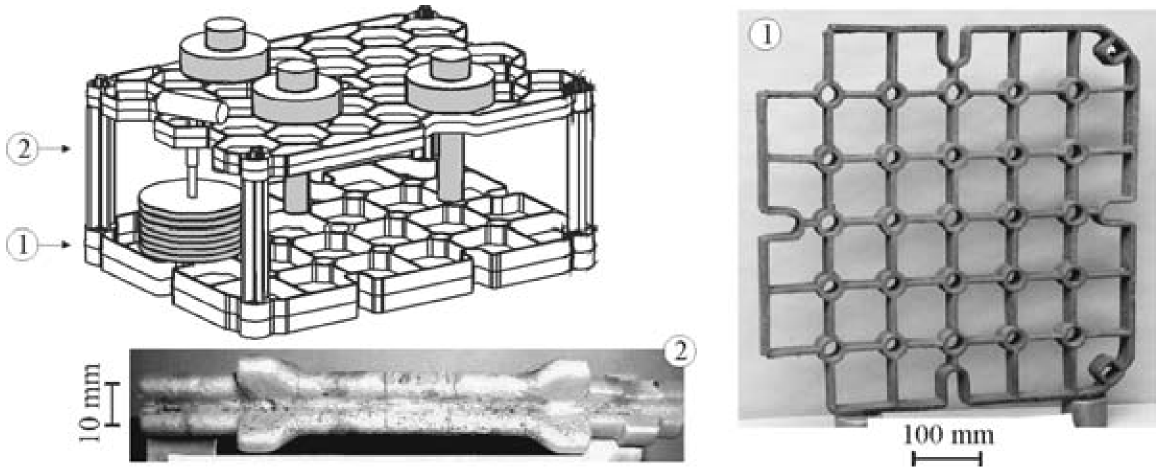

Figure 2: An assembly of grates withdrawn from use in pusher-type furnace: 1) scrap base grate, 2) supporting rod with cracks and deformations

thermal fatigue, very disadvantageous are the differences in shape and mass between the base grate and the intermediate grate. This is not only because of the function they are expected to perform but also because of the economic reasons – the weight of the accessories should be reduced to minimum in respect of the weight of the carburised charge. The intermediate grates with walls usually thinner get heated/cooled in a time much shorter than the base grate. During of the charge preheating process, the supporting rods joining them are bent to the outside, and during cooling to the inside. Since these operations are repeated in regular cycles, the result is, on one hand, breaking out of the corners, and on the other – the deformation of rods (**Figure 2**). The next factor speeding up the degradation of castings are external and internal casting defects (**Figure 1b-2**).

3 CONCLUSION

From the above described process of degradation of the charge-carrying elements three general conclusions follow, and they define the procedure which should be followed in order to increase the life of the charge-carrying elements:

1. Since it is impossible to totally eliminate the process of casting carburising, of an utmost importance for the life on performance of this casting will be improving the cast steel resistance to thermal fatigue under the conditions of an effect of the carburising atmosphere.
2. In the process of degradation of the charge-carrying elements very important role is played by the thermal fatigue behaviour under the conditions of both external as well as internal constraints, because the fields of own stresses forming in castings originate from factors present in these two groups. The presence of casting defects both on the surface and inside the casting accelerates the thermal fatigue and carburising processes.
3. No permanent deformations have been observed to occur in castings due to the creep effect. The examined examples of the grate degradation show that nei-

ther the static (bending) loads caused by the charge, nor the creep effect can be regarded as an immediate reason of the grate deformation and failure.

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FRACTOGRAPHIC EVALUATION OF GIGACYCLE FATIGUE FAILURE OF A HIGH Cr ALLOYED COLD WORK TOOL STEEL

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ABSTRACT

In order to shed more light on gigacycle fatigue crack nucleation and propagation, fracture surfaces of AISI D2 type (DIN NR. 1.2379) cold work tool steel were studied as obtained in fatigue testing using ultrasonic resonance testing. Macroscopically, flat surfaces were observed showing fish-eye type appearance around the crack origins. Primary carbides and carbide clusters at/near the surface and in the interior of the specimens gave rise to fatigue cracks. Five zones of the fatigue process could be distinguished exhibiting different surface morphologies. The size of these zones was evaluated and probable relationships to the applied stress amplitude and cycle number to failure were investigated.

Keywords: tool steels, gigacycle fatigue, fractography, carbide clusters, ultrasonic fatigue testing

1 INTRODUCTION

As can be concluded from a literature review (presented in the introduction of ref. [1]) of fatigue studies on high strength steels, material failure has been observed also below the conventional fatigue limit e.g. in high strength bearing steels in the very high cycle regime above 10^6 cycles, where specimens failed due to fatigue cracks originating in the interior, forming so-called "fish-eye" features at the fracture surface. Internal crack origins were mostly nonmetallic inclusions. However, most of the research [1] was focused on high strength structural steels, strengthened only by martensitic matrix, but containing a low amount of other phases. That represents a major difference to tool steels, which contain numerous primary and secondary carbides responsible for high abrasion resistance and hot hardness. Summarizing the limited studies on fatigue of tool steels [1], fatigue failures occurred due to large primary carbides, and due to nonmetallic inclusions or carbide clusters in case of very clean powder metallurgically produced tool steels. However, it is noted that none of the studies described tests for more than 10^7 loading cycles. Thus, the present contribution aims to provide evaluation of fatigue fracture surfaces of AISI D2 type cold work tool steel, up to N = 10^{10} cycles.

2 EXPERIMENTAL

A wrought cold work tool steel (Böhler grade K110 in cylindrical, annealed bars (ϕ 15.5mm); DIN Nr. 1.2379; AISI D2 type; 1.55%C, 0.32%Si, 0.32%Mn, 12.7%Cr, 0.85%Mo, 0.89%V, 0.12%W, 0.12Co) was fatigue tested employing an ultrasonic resonance testing system [1] operating at 20 kHz in fully reversed tension-compression mode. Hour glass shaped fatigue specimens were machined to desired geometry (with axis in rolling direction, gauge diameter 4 mm) and longitudinally ground in annealed condition, and then heat treated (austenitizing at 1040°C-25 min and tempering at 530°C-2 hrs) under high purity nitrogen atmosphere. Subsequently, the samples were ground and polished to mirror-like finish in the longitudinal direction using two procedures: For test series K110L-I the specimens were ground (600 mesh SiC paper) and polished (12 µm Chromium oxide paste). Specimens of series K110-II were ground (240 mesh Al_2O_3 paper) and subsequently material removal to depth

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of 30-50 µm was performed using 15 µm diamond suspension. Here, for finish polishing 6 µm diamond suspension was used. Axial residual stresses (RS) were measured by means of XRD and fracture surface investigated using SEM.

3 MATERIALS CHARACTERIZATION

After the heat treatment, uniform tempered martensitic microstructure was obtained. XRD proved that the RA content was very low, a few percent maximum. Prior austenite grain size turned out to be 13±2 µm. Steel hardness, T.R.S. and Young's modulus were 58±2 HRC, 3600±350 MPa and 210±7 GPa, respectively. The numerous primary carbides of type $(Fe,Cr)_7C_3$ (confirmed by XRD) were found to be distributed relatively uniformly in the transverse section, whereas in the longitudinal section they were elongated due to the rolling operation (carbide bands). Numerous very small secondary carbides representing finely dispersed precipitates, generated during the tempering process, were also observed. Furthermore, high compressive surface RS of -800±100 MPa were measured for K110L-I specimens, while K110L-II specimens showed only very low surface RS in the range of -170 to +40 MPa. Detailed discussion of the microstructure, mechanical properties, and residual stresses has been presented elsewhere [1].

4 RESULTS AND DISCUSSION

The S-N data showed that surface compressive RS increased significantly the fatigue strength of the material. Test series K110L-I exhibited internal crack origins, which were carbide clusters (**Figures 1a,c**), up to $6x10^6$ cycles,. Due to relaxation of RS a transition from internal to near/at surface failure took place with increasing life time. K110L-II specimens revealed predominantly near/at surface crack origins. Here, carbides and carbide clusters located at or just beyond the surface gave rise to fatigue cracks. (Half) fish-eye appearance was observed around the crack origin. Five different zones of crack propagation – regardless of crack origin location – were identified according to **Figures 1a,b**. In the surrounding of primary carbides (clusters) of crack origin a granular area (GA-**Figures 1c,d**) was observed which is assumed to be of importance for small crack formation, according to a model proposed by Shiozawa et al. [2]. Multiple microcracks seem to be formed – through decohesion of matrix from numerous finely dispersed secondary carbides – which then grow and coalesce until appropriate size of GA is reached. Then, small fatigue cracks, which can be observed in area "2a", are initiated. Apart from these small cracks the area "2a" exhibited a rather flat surface morphology. Stages "2b" and "2c" mark further fatigue crack propagation revealing roughened surface with multiple cracks. The border of the fish-eye marks the occurrence of final fracture (area "3"). Detailed evaluation of the sizes of the different stages of crack growth showed strong relationships with applied stress amplitude and cycle number to failure [3]. Furthermore, it was possible to calculate stress intensity factors using the obtained size data, which were then used for estimation of probable fatigue limit [3].

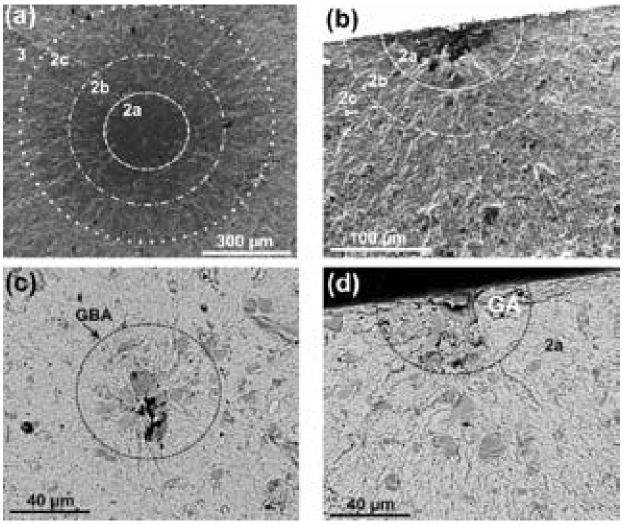

Figure 1: Zones of fatigue crack nucleation and propagation in (a), (c) internal and (b), (d) near-surface failures

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ANISOTROPY EFFECTS ON GIGACYCLE FATIGUE BEHAVIOR OF 12%CHROMIUM ALLOYED COLD WORK TOOL STEEL

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ABSTRACT

The gigacycle fatigue behavior of wrought cold work tool steel of AISI D2 type (DIN NR. 1.2379) was studied employing an ultrasonic fatigue testing system operating at 20 kHz in fully-reversed tension-compression mode. This contribution focuses on the effect of anisotropy and degree of deformation on the fatigue behavior. Primary carbides and carbide clusters located near/at the surface acted as fatigue crack initiation sites. Macroscopically, the fracture surfaces revealed rather flat surface morphology, and around each crack origin a semicircular fish-eye feature was obtained. Around the carbide particles a granular area (GA) was observed followed by a very flat zone. The obtained S-N data showed considerable anisotropy of the fatigue behavior. Specimens with their axis perpendicular to the rolling direction revealed about 150 MPa lower fatigue endurance strength at 10^{10} loading cycles compared to samples with their axis in the rolling direction.

Keywords: tool steels, gigacycle fatigue, material anisotropy, carbide clusters, ultrasonic fatigue testing

1 INTRODUCTION

Cast, forged and rolled materials generally reveal directional mechanical properties due to anisotropy of the material microstructure that can be e.g. crystallographic textures, elongated grain structure, or additional phases such as alloy carbides or nonmetallic inclusions. The cold work steel studied here is a conventionally cast and then hot worked grade containing numerous primary chromium carbides, responsible for high abrasion resistance. However, these hard phases are elongated in rolling direction due to the hot working (rolling) process. Thus, anisotropy of the mechanical properties of this tool steel can be expected, which affects also the fatigue behavior of the tool steel. Summarizing the limited studies on fatigue of tool steels [1] (no tests longer than 10^7 loading cycles have been described), fatigue failures occurred due to large primary carbides, and due to nonmetallic inclusions or carbide clusters in case of very clean powder metallurgically produced tool steels. This contribution provides new information about anisotropy of gigacycle fatigue behavior of AISI D2 type cold work tool steel.

2 EXPERIMENTAL

A wrought cold work tool steel (Böhler grade K110 in cylindrical, annealed bars (ϕ bar A 15.5 and bar B 106 mm); DIN Nr. 1.2379; AISI D2 type; 1.55%C, 0.32%Si, 0.32%Mn, 12.7%Cr, 0.85%Mo, 0.89%V, 0.12%W, 0.12Co) was fatigue tested employing an ultrasonic resonance testing system [1] operating at 20 kHz in fully reversed tension-compression mode. Hour glass shaped fatigue specimens with axis parallel to (K110L-barA, LL-barB) and perpendicular to (K110TT bar B) the rolling direction (RD) were machined to desired geometry and longitudinally ground in annealed condition and then heat treated (austenitizing at 1040°C-25 min and tempering at 530°C-2 hrs) under high purity N_2 atmosphere. Subsequently, the samples were ground (240 mesh Al_2O_3 paper) and polished to mirror-like finish (15&6 µm diamond susp.) in the longitudinal direction. Surface axial residual stresses (RS) were measured by XRD.

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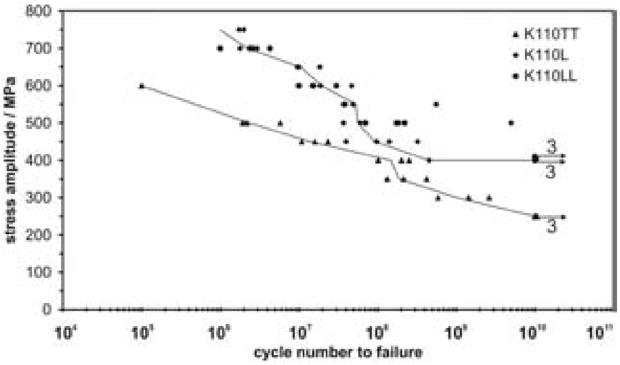

Figure 1: S-N data of AISI D2 type wrought cold work tool steel.

3 MATERIALS CHARACTERIZATION

After the heat treatment, uniform tempered martensitic microstructure was obtained. XRD proved that the RA content was very low, a few percent maximum. Prior austenite grain size turned out to be 13±2 µm. Steel hardness, T.R.S. and Young's modulus were 58±2 HRC, 3700±400 MPa (parallel to RD) and 1950±150 MPa (perpendicular to RD), and 208±9 GPa, respectively. The numerous primary carbides of type $(Fe,Cr)_7C_3$ (as proved by XRD) were found to be distributed relatively uniformly in the transverse section, whereas in the longitudinal section they were elongated due to the rolling operation (forming carbide bands). Numerous very small secondary carbides representing finely dispersed precipitates, generated during the tempering process, were also observed. Surface RS turned out to be -195 to +35MPa (K110L,LL) and -340 to -195MPa (K110TT).

4 RESULTS AND DISCUSSION

S-N data showed that sample life of K110L and LL specimens was similar, which indicates that the degree of deformation has no effect in the longitudinal direction. In contrast, K110TT samples exhibited significantly lower fatigue strength.

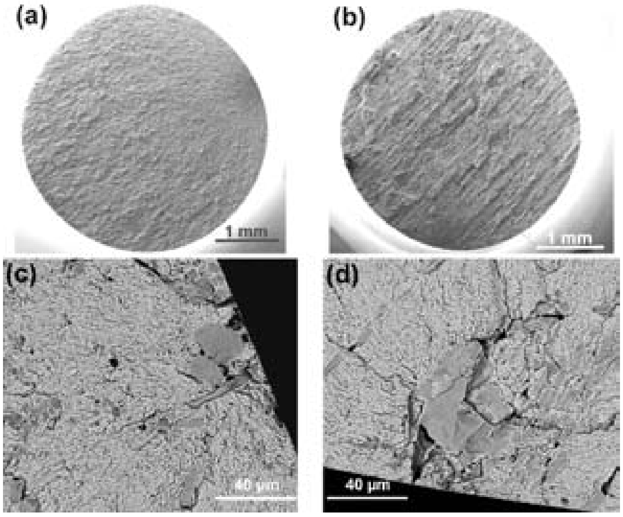

Figure 2: Fracture surface overviews of ASIS D2 type wrought cold work tool steel: (a) K110LL, (b) K110TT; primary carbide clusters to be crack origins of (c) K110LL and (d) K110TT specimens.

The fracture surface morphology was completely different in the longitudinal and transverse directions except of the fact that around the crack origin half fish-eye feature was formed. In case of K110L,LL fractures numerous cracks point back to the crack origin (**Figure 2a**), whereas in K110TT fractographs numerous cracks oriented parallel to carbide bands (**Figure 2b**) were observed. Crack origins were primary carbides and carbide clusters located near/at the specimen surface for all three test series (**Figure 2b,d**). However, in case of K110TT specimens the clusters were significant larger, and often elongated species were found which seemed to be deformed during machining/polishing. Detailed evaluation and discussion can be found elsewhere [2].

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LOAD ON PUNCH DURING FINEBLANKING INCONEL 718

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ABSTRACT

We measured the force that act on punch during fineblanking parts for turbocharger for automotive diesel engine. Force was measured out with strain gauges that we installed them on punch. Sheet metal from which we were producing segments was alloy Inconel 718, witch has rather high mechanical properties and high ratio of strengthening. We were interested in dependence of force from time and vertical moving of punch. Furthermore, the influences of different heat treatment and with this different mechanical property of alloy Inconel 718 on load of tool were investigated.

2 EXPERIMENTAL SETUP

Alloy Inconel 718 was solution annealed at temperature 960 °C and 1050 °C and then quenched in water. With tensile test and measurement of hardness we designated the mechanical properties (**Table 1**) of different solution annealed alloy Inconel 718.

Table 1: The influence of different heat treatment on mechanical properties of Inconel 718

Tempera-ture of so-lution an-nealing	Average values				
	R_m [MPa]	$R_{p0,2}$ [MPa]	A [%]	n [/]	HV 0,3
960 °C	877	443	48	0,39	220
1050 °C	757	293	59	0,46	185

We installed four strain gauges on punch of tool and connected them in measuring system (**Figure 1**), witch enable to measure the force that was employed on punch during fineblanking of alloy Inconel 718. As well, we installed in tool also the inductive probe for measuring the vertical moving of punch through sheet metal of alloy Inconel 718 during fineblanking process.

3 RESULT AND DISCUSSION

Mechanical properties of alloy have influence on force of fineblanking. Punch is during fineblankig of alloy A (**Figure 2**) loaded with force of 255 kN. On the other hand, during fineblanking of alloy B (**Figure 2**) punch is loaded with force of 235 kN. Reasons for this are smaller mechanical properties of alloy B, witch was solution annealed on higher temperature.

Oscillating is seen in initial part of both curves (**Figure 3**). This is result of air gap of press. In second part of curve suddenly fall is seen. This kind of fall is characteristic for conventional stamping, where the tensile stress of material is overcome and the material is brake down.

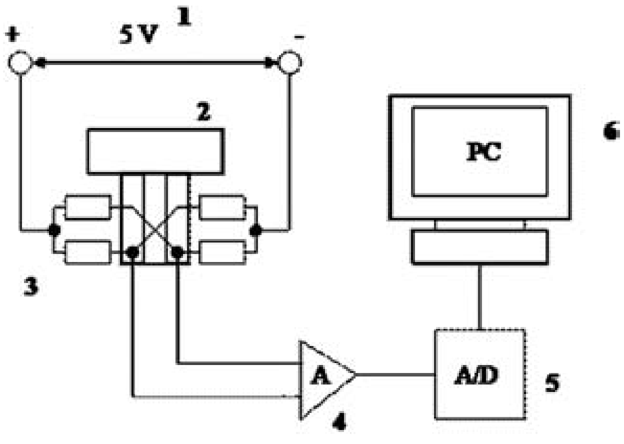

Figure 1: Measuring system; 1 – source of electricity, 2 – punch, 3 – strain gauges connected in full Weatstonov bridge, 4 – amplifier, 5 – converter of impulse, 6 – personal computer.

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Topic: D – Mechanical and Physical Properties of Tool and Die Materials
Presentation type: Oral

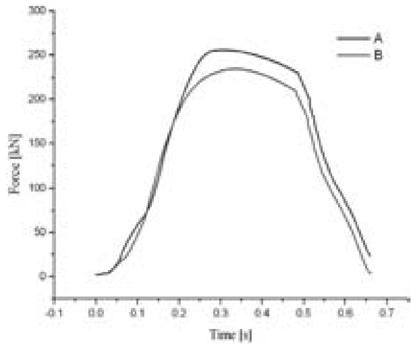

Figure 2: Curves for force versus time during process of fineblanking; A – solution annealed alloy Inconel 718 at temperature 960°C, B – solution annealed alloy Inconel 718 at temperature 1050°C

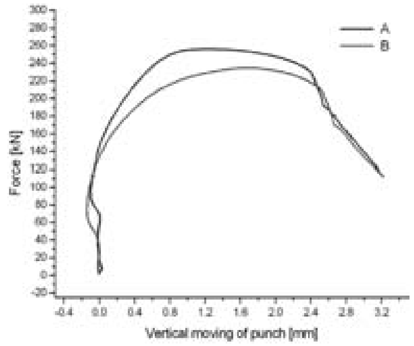

Figure 3: Force as function of vertical moving of punch through sheet metal; A – solution annealed alloy Inconel 718 at temperature 960°C, B – solution annealed alloy Inconel 718 at temperature 1050°C

4 CONCLUSION

The load on tool depends upon mechanical properties of raw material. From view of technology of fineblanking is the most appropriate solution annealed alloy on temperature 1050 °C because it has the lowest hardness, yield strength, tensile strength and highest elongation. Lower mechanical properties mean lower load on tools and more quality cutting surface.

During fineblanking process there is no brake down of raw material. Maximal force and line of force with which is loaded punch between fineblankin is important information for choice tool steel and construction of tool.

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THE EFFECT OF SERVICE CONDITIONS ON CRACK INITIATION AND PROPAGATION IN WELDED JOINT OF HIGH-ALLOY STEEL X20

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ABSTRACT

The aim of these investigations was to establish the effect of service conditions (service life, temperature) on behaviour of base metal and welded joint of high alloy X20, designed for operation at elevated temperatures and affected by variable loading. The effect of service conditions was analysed by testing the new material and material that had been in service for 116,000 hours. Investigations of the new material and base metal that had been in service as well as of the components of welded joint (weld metal and heat-affected zone) made of high alloy steel included determination of the parameters of fatigue-crack growth both at room and at elevated temperature.

The results of these investigations should provide practical contribution toassessment of quality of base metal of the components of welded joint of steel X20, aimed at revitalisation i.e. assessment of strustural integrity and extension of service life of vital components, high-alloy steel thermal power plants operating at elevated temperatures.

Keywords: high alloy steel X20 CrMoV 12-1, Paris law, crack-propagation rate, fatigue threshold

1 INTRODUCTION

For in-service safety of the components of processing equipment for operation in thermal power plants, the most important characteristics are those describing crack initiation and growth. This particularly relates to the cracks initiated because of service conditions and effects of variable loading. Initiation and growth of a crack induced by variable loading, i.e. Paris' law of crack growth establishing the dependence of crack growth per cycle on acting variable loading i.e. corresponding range of stress intensity factor, is nowadays widely accepted as, basically, it describes the micromechanical behaviour of a growing crack [1].

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Topic: D – Mechanical and Physical Properties of Tool and Die Materials
Presentation type: Poster

For better comprehension of the causes and mechanisms of crack initiation and growth in welded joints of steels designed for operation at elevated temperatures and high pressures, it is obligatory to establish the effect of heterogeneity of structure and mechanical properties of welded joint on crack initiation and growth, as well as on the parameters of fatigue-crack growth [2]. Testing of the specimens with initiated fatigue cracks in base metal, weld metal and heat-affected zone at room temperature, operating temperature of 545°C and max operating temperature of 570°C will enable assessment of the effect of heterogeneity of structure and mechanical properties of welded joint on the parameters of fatigue-crack growth, i.e. on the parameters of the Paris equation obtained from the dependence log (da/dN) – log (ΔK) [3].

2 EXPERIMENTAL SETUP

For the analysis of the effect o temperature and service life on mechanical, structural and service properties of base metal and welded joint of high-alloy steel X20 designed for manufacture of vital components of thermal-power plants, two samples were available: sample N, new pipe-tube ∅450 x 50 mm approx. 400 mm long, and sample U, welded used pipe-tube with weld metal in the middle, ∅450 x 50 mm approx. 500 mm long. Sample U was taken from a steam line for fresh steam in a thermal-power plant, having been in service for about 116,000 hours. The results of the chemical analysis of the samples of the new pipe and used pipe are shown in **Table 1**.

The effect of service conditions (service time and temperature) on the parameters of fatigue-crack growth in base metal and components of welded joint of steel X20 was analysed by testing the samples of the new pipe and pipe that had been in service for 116,000 hours. Three types of specimens were prepared: specimens with a notch in base metal (BM), specimens with a notch in weld metal (WM) and specimens with a notch in heat-affected zone (HAZ).

Based on the test procedure, log da/dN = log(ΔK) are plotted. An example of the diagram representing fatigue-crack growth rate, da/dN – variation of stress-intensity factor range, ΔK, for the specimens prepared from the sample BM of the new pipe and exploited pipe tested at room temperature, operating temperature of 545 °C and max operating temperature of 570 °C is given in **Figure 1** [1].

Investigations were conducted on the specimens with crack tips and in characteristic zones of welded joint. It is obvious that the location of the notches and crack initiation, as well as service conditions (temperature and service life) have decisive effect on the values of fatigue threshold ΔK_{th}, fatigue-crack growth rate and parameters of Paris equation.

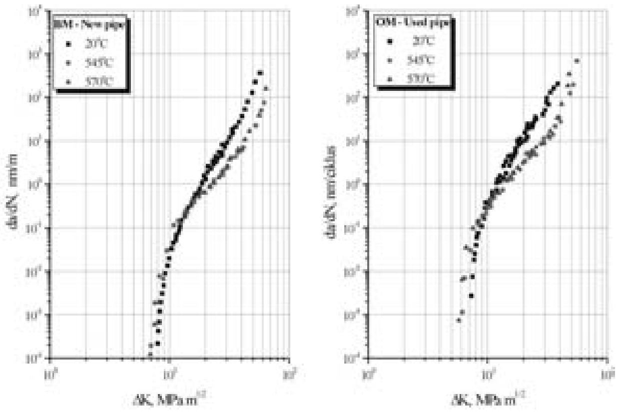

Figure 1: Log-log diagram da/dN – ΔK for specimens BM [1]

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Table 1: Chemical composition of the sample of pipe of high-alloy steel X20

Batch	Mass %								
	C	Si	Mn	P	S	Cr	Mo	Ni	V
Sample – N	0.21	0.27	0.56	0.017	0.006	11.70	1.019	0.601	0.310
Sample – U	0.22	0.31	0.54	0.019	0.005	11.36	1.033	0.551	0.314

MECHANICAL PROPERTIES OF BORONIZING STEELS AS REPERCUSSION OF BORON PHASES

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ABSTRACT

Results of the boride surface layers investigation, obtained by the torsion and bending methods, point to the better ductility of the mono-phase Fe_2B boride layers then the binary-phase $FeB + Fe_2B$ boride layers. This is especially outstanding in the case of peaces that are under higher thermal and mechanical load, where the binary-phase layer because of higher local stresses has higher tendency to fall of, respectively to shell.

Keywords: boronizing, boron phases, boride layers, mechanical properties

1 INTRODUCTION

Boronizing is a thermochemical process in which the chemical element boron diffuses in the surface layer of steel substrates. In the surface layer of steel substrate, boron forms with the iron Fe_2B, as desirable compound, and FeB as undesirable compound. Boride layers have extraordinary high hardness. FeB is a little bit harder (1800–2100 HV0,1) than Fe_2B (1400–1600 HV0,1), but much brittle, so its forming have to avoid in the practice.

During boronizing of the forging matrixes, drawing tools and shaping tools it is needed, among other, to determine the toughness of the boronized material [1 to 3].

The aim of the conducted investigation was to determine the ductility of the boron phases using methods in which the influence of the core on the boride layers will bee lower (lower result scatterings). For the ductility determination of the boride layers two independent methods were chosen: by the torsion method and by the bending method.

2 INVESTIGATION

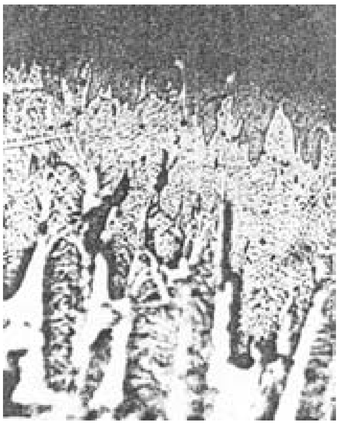
Torsion method used was detection of the cracks appearance of the boride layer during torsion cylindrical specimen. Specimens were made from C 15 and 20 CrMo 5, designated in accordance with data in **Table 1**, and boronized with the appropriate regimes and in the conditions which in one way led to forming of the binary phase $FeB + Fe_2B$ boride layer, and in the other way to forming mono phase boride layer Fe_2B.

Boronizing was performed in solid mediums. First boronizing regime and conditions, which resulted in mono phase boride layer Fe_2B formation, include: boronizing agent with low activity (SiC, B_4C, KBF_4) using lower boronizing temperature T_B = 850 °C and boronizing time t_B = 4 hours. Second borinizing regime and conditions, in which the binary phase boride layer $FeB + Fe_2B$ is formed, include boronizing agent with increased activity ($Na_2B_4O_7$, B_4C, KBF_4) using higher boronizing temperature T_B = 950 °C and lower boronizing time t_B = 2 hours. Besides classical metallographic technique (**Figure 1 and Figure 2**), the examination of the formed boride layers were performed by the scanning electron microscopy – microsonde. For the boride layer type determination, surface layer of the specimens were also examined by the X-ray diffraction

20 CrMo 5 100x
Figure 1: Boride layer photomicrograph

Table 1: Specimen identification numbers

Steel C 15		Steel 20 CrMo 5	
Boron phases		Boron phases	
$FeB+Fe_2B$	Fe_2B	$FeB+Fe_2B$	Fe_2B
Specimen identification Nr.		Specimen identification Nr.	
0,1	1,1	2,1	3,1
0,2	1,2	2,2	3,2
0,3	1,3	2,3	3,3
0,4	1,4	2,4	3,4
0,5	1,5	2,5	3,5
0,6	1,6	2,6	3,6



20 CrMo 5 500x

Figure 2: Boride layer photomicrograph

method. Surface layer, formed on the specimen, has structure and parameters of FeB and Fe_2B boride compounds, so as some of Fe_3 (B, C) and Fe_{23} (B, C)$_6$ carbide type compounds. Ductility examination by torsion method was done with testing machine and additional equipment.

From results of the ductility examination by torsion method can bee concluded that the torsion angle of both specimen, one made from carbon steel C 15 and the other from alloyed steel 20 CrMo 5, in case of mono phase boride layer Fe_2B is higher than in case of binary phase boride layer FeB+Fe_2B. It can be seen from the mean value of torsion angle x_1=22,67° and y_1=29,17° for binary phase boride layers, so as x_2=32,67° and y_2=34,5° for mono phase boride layers. Bending method used for ductility determination is bending deformation of the tin flat specimen (0,85x10x100 mm), previously boronised in suitable mediums, described before.

The results boride layers ductility examination by the bending are given in **Figure 3**.

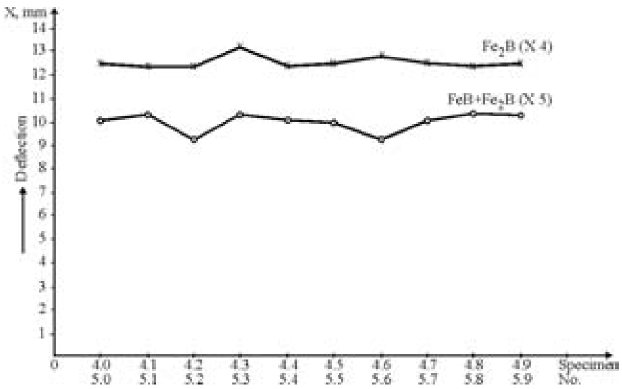


Figure 3: Results boride layers ductility examination by bending

3 CONCLUSIONS

Values obtained by measuring the deflection x_4 and x_5 (Figure 3) show that method of boride phase ductility determination by bending (so as previous method) gives better results in the case group of specimen (x_4) with mono phase boride layer Fe_2B comparing to the group of specimen (x_5) with binary phase boride layer Fe_2B+FeB. The most probable cause of better mechanical properties, ductility as wearing resistance, mono phase comparing to binary phase boride layers (even though the hardness is higher by binary phase FeB+Fe_2B boride layers) is different thermal expansion coefficients. During work pieces (specimens) treatment cooling forms compression stresses are in the Fe_2B layer and tension stresses in FeB layer. The stresses with different sign can produce micro cracks in boride phase vertical to the layers as between two layers.

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2nd INTERNATIONAL CONFERENCE ON HEAT TREATMENT AND SURFACE ENGINEERING OF TOOLS AND DIES
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EFFECT OF HEAT TREATMENT ON THE MECHANICAL PROPERTIES OF TOOL STEELS

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1 LOADING, DAMAGE AND LIFETIME OF TOOLS

In service tools and tool materials are normally subjected to (1) mechanical loads, including thermo-mechanical loads, (2) tribological loads, and (3) thermal loads.
Mechanical/thermo-mechanical loads cause cyclic stresses and strains which lead to fatigue type damage effects. If the stresses are high enough cyclic plastic strains occur in addition to the cyclic elastic strains. Tribological loading takes place at the tool surface and causes stresses, strains and often also chemical interaction which lead to damage at and near the surface. Thermal loads may lead to microstructural changes, e.g. precipitation of dissolved alloying elements, phase transformation (e.g. retained austenite to martensite) or coarsening of precipitates.
Damage mainly takes place in the highest loaded regions of a tool. The damage process is strongly depending on the stresses and strains induced by the manufacturing process for which the tool is used on the one hand and on the microstructure and the mechanical properties of the tool material which depends on the tool steel grade and on the heat treatment on the other.
Tool steels are multiphase materials which are consisting of a metallic matrix with embedded hard phases, mainly carbides, nitrides or carbonitrides. Due to the high strength level of tool steels even small plastic strains lead to the formation of micro-cracks, e.g. by cracking of carbides or carbide/matrix interfaces. The performance of tools is strongly related to the behaviour of these micro-cracks. It should be pointed out in this regard that micro-crack tip loading does not only result from the stress range caused by the manufacturing process for which the tool is used but also from the above mentioned residual stresses. The initial size and the crack tip loading conditions determine whether these micro-cracks are able to grow or in other words whether the damage process stops or proceeds after the micro-crack formation.
There is no consistent definition available for the end of tool life. The failure criterion varies strongly with the application and the type of tools. In some cases tools have to be replaced if the first small cracks appear which are imprinted on the workpiece, whereas in other cases the total tool failure is relevant. But as a general rule reaching a critical crack size, which is characteristic for a specific application, can be taken as a feasible lifetime criterion.

2 MATERIAL RESPONSE TO THE MECHANICAL LOADING AND DEFINITION OF RELEVANT MATERIAL PROPERTIES

Determination of tool loads in terms of stress, strain and temperature cycles is a highly challenging task. It requires a coupling of the simulation of the manufacturing process for which the tool is applied with the finite element calculation of the stresses, strains and temperatures in the tool.
Most tools exhibit some highly stressed areas in which cyclic plastic deformation occurs very localised. Due to the high hardness these plastic strains occurring in tools are normally small. If cyclic straining is not fully reversible, which is often the case, this is leading to the development of residual strains. These residual strains lead to the formation of residual stresses. Due to the high strength level of tool steels even low residual strains may lead to high residual stresses. These residual stresses are superposed to the stresses induced by the manufacturing process for which the tool is applied. The residual stresses result in a change of the stress ratio which is known to have a significant effect on the growth behaviour of the micro-cracks and the whole damage process.

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Title: Effect of heat treatment on the mechanical properties of tool steels
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Topic: D – Mechanical and Physical Properties of Tool and Die Materials
Presentation type: keynote lecture

A detailed analysis of the behaviour of tools in service requires finite element calculations that are taking the static and the cyclic elastic-plastic material behaviour into account. Such advanced finite element simulations require powerful material models which allow a realistic prediction of the material response under the very specific loading conditions occurring in tools. The material models must be able to describe among others the static stress-strain curve, isotropic and kinematic hardening, elastic and plastic shakedown, ratchetting, visco-plastic behaviour, or stress relaxation.

Complex test procedures and time consuming and expensive material modelling procedures are necessary to derive suitable material models (constitutive equations). A significant drawback is that material models available today are mostly based on phenomenological principles. Most of the parameters in these models can be hardly interpreted on the basis of well defined physical meanings. Due to the high effort necessary for the determination of parameters for the constitutive equations and the missing physical interpretation only very limited information is available in literature. Most activities in this respect were performed on the more ductile hot work tool steels, for high speed steels which have lower ductility only a few activities are known to the authors of this paper.

For a prediction of tool lifetimes also the effect of cyclic stresses and strains on the damage process and on the lifetime is required. The fatigue lifetime behaviour is often described by means of the double-log strain amplitude versus cycles to failure curve which allows a description of the low cycle fatigue behaviour, the high cycle fatigue behaviour and the "fatigue strength" in one diagram. Most literature on the fatigue properties is focussing on the high cycle fatigue regime and the fatigue strength. It is well known that the lifetime in the high cycle fatigue regime is controlled by defects. This means that micro-crack forming microstructural elements, e.g. non metallic inclusions, or machining induced micro-defects control the fatigue behaviour whereas the hardness has only a minor or even no effect. In most tool applications the tool materials are subjected to fatigue loading under high compressive mean stress. Unfortunately, almost no literature is available on the fatigue behaviour of tool materials under such conditions.

Assuming that linear damage accumulation is applicable (Miner rule) the double-log strain amplitude versus cycles to failure curve can be also taken as basis to calculate the damage increment produced by one single load cycle. This can be used further to calculate the number of cycles necessary to produce a crack with a critical length. Again, there is also very little information available in literature regarding the effect of loading on the fatigue life of tool steels. This is especially true for the effect of heat treatment.

In practice the service properties of tool steels are still characterised mainly by their hardness. Material data determined in uniaxial tension tests are standard only in case of the more soft and ductile materials, e.g. hot work tool steels. For the very hard tool steel grades with low ductility, e.g. high speed steels, it is even hard to get reliable static material data from tension and compression tests. Also cyclic material data are very rare; this is also true for the effect of heat treatment.

Goal of this paper is to give some basic information on the mechanical properties which are relevant for the tool performance and to point out the effect of heat treatment on these properties.

3 EFFECT OF TOOL STEEL GRADE AND HEAT TREATMENT ON THE LIFETIME RELEVANT MATERIAL PROPERTIES

The properties of tool steels are controlled by the tool steel grade on the one hand and on the heat treatment on the other. The steel grade determines the strength and ductility potential of the tool steel and the heat treatment determines to which extent this potential is used.

The heat treatment of tool steels consists of several steps, the most relevant steps and the controlling factors are: (1) Austenitizing (temperature, time), (2) Quenching (cooling rate), and (3) Tempering (tempering temperature, cooling temperature, number of tempering cycles).

In the present paper the effect of heat treatment (austenitizing temperature, quenching rate, tempering temperature) will be demonstrated for various steel grades with focus on tool steels with medium and higher volume fractions of carbides. The investigations concentrate on the static mechanical properties (characteristic strength and ductility values, stress-strain behaviour, fracture toughness) and cyclic mechanical properties (cyclic stress-strain behaviour, strain amplitude versus cycles to failure curve, fatigue crack growth behaviour).

Furthermore the application of these data for advanced finite element calculations of the evolution of loading conditions (evolution of stresses and strains including residual stresses) and the damage of tools will be demonstrated.

DETERMINATION OF LOWER BOUND OF FRACTURE TOUGHNESS OF SUSPENSION SPRING MATERIAL

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ABSTRACT

Modern suspension springs of vehicles are made from one single spring, which should carry all loads. It is well known that spring materials have quite low fracture toughness. It makes that spring are very sensitive on presents of inclusions. Mechanical properties and fracture toughness are determined on specimens cut out tested spring sample, as is shown in **Figure 1**. The mechanical properties are determined on mini tensile specimens (MTS).

The fracture toughness testing was performed on specimens cut from tested springs. The tested fracture toughness specimens exhibited brittle behaviour (as is shown in **Figure 2**) and condition for fracture toughness parameter K_{Ic} are completed.

Critical sizes of inside fatigue crack around inclusions are determined by using SINTAP procedure. It is assumed that the circular fatigue crack appeared around inclusions (e.g. Al_2O_3) (**Figure 3**).

The aim of our paper is find lower bound of fracture toughness in order to accept regular size of inclusion close the surface of bended spring. Theoretical fracture toughness values had been calculated for fixed inclusion size and constant mechanical properties.

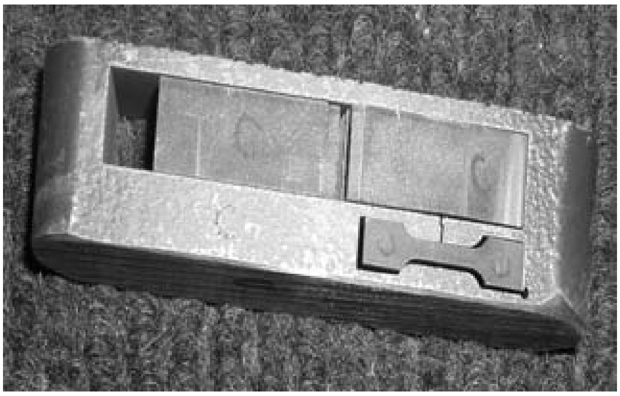

Figure 1: Orientation and position of specimens for determination mechanical properties and fracture toughness

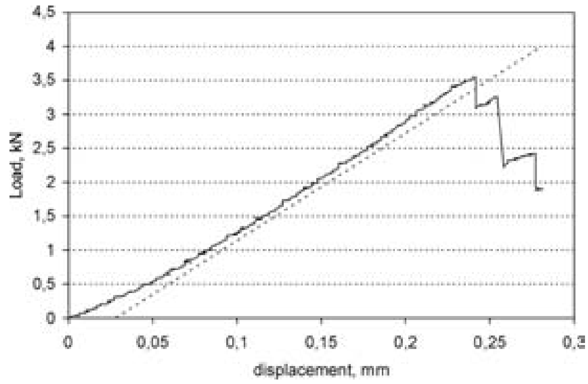

Figure 2: Fracture behaviour of single edge bend specimen

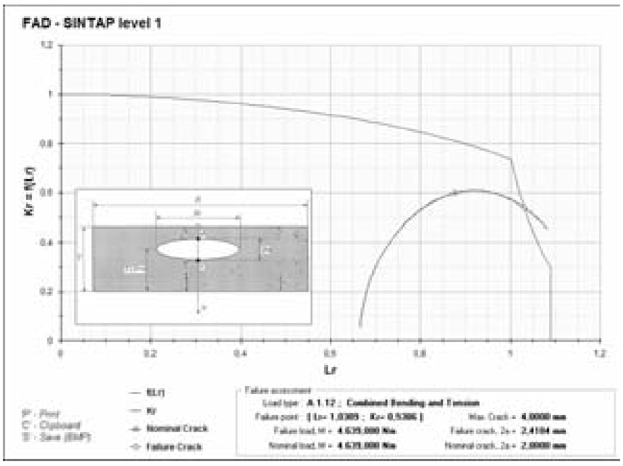

Figure 3: Result of spring integrity analysis by using SINTAP procedure for inclusion

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Topic: D – Mechanical and Physical Properties of Tool and Die Materials
Presentation type: Oral

ADVANCED TOOL MATERIALS

FATIGUE RESISTANT PM TOOL STEELS

Ziya Devrim Caliskanoglu*, Jochen Perko, Hubert Lenger

Traditional, conventionally manufactured cold work tool steels e.g. DIN 1.2379 (X155CrVMo12-1), are often incapable of withstanding the high loads in high-performance cold work applications. The reason for early failure of the material obviously depends heavily on the load profile of the particular application. Typically occurring failure mechanisms are abrasive wear, adhesive wear, cyclical plastic deformation, the formation of fatigue cracks and spalling of flat surfaces and/or edges. To increase the abrasive wear resistance of tool steels, the matrix strength must be increased and a certain percentage of hard carbides must be present. To increase the adhesive wear resistance, the average distance between carbides in the high-strength matrix of the tool steel should be minimised. To avoid plastic deformation and spalling at the edges due to cyclical loading, the tooling material must possess both a high-strength matrix and high ductility.

The failure mechanisms described can occur either alone or in combination. For this reason, high-performance powder metallurgical tool steels with a chemical analysis, high homogeneity, high cleanliness, homogenous carbide size and distribution, and high reproducibility of those properties optimised for the particular loading situation, should be used for highly stressed tooling. In this presentation, we demonstrate how the material properties can be influenced by the manufacturing process and the chemical analysis, using selected high-performance steels as examples.

Powder metallurgy tool steels and high speed steels used for highly stressed tooling must have a high toughness as well as a high strength and high wear resistance, and must above all posses the best possible fatigue properties in order to ensure a suitable resistance to fracture. To increase the working life of tooling it is therefore necessary to drastically reduce the number of fracture-initiating defects such as non-metallic inclusions and carbide clusters. The negative influence of primary carbide clusters or those formed eutectically from the melt in highly alloyed, conventionally manufactured steels can be largely prevented using a powder metallurgy manufacturing route. Non-metallic inclusions remain as the limiting defects reducing the working life of tooling. The main sources of this type of inclusion are the furnace slag and the top slag in the tundish.

For this reason, the traditional PM process was modified during construction of the Böhler-Uddeholm PM plant in Kapfenberg. The installation of an electro-slag heating unit which heats the top slag in the tundish, in combination with an electromagnetic stirrer, protects the melt from oxidation. In addition this means that inclusions caused by the furnace slag can be precipitated out by stirring, and a constant melt temperature and cleanliness level can be maintained.

It is known that in the area of low-cycle-fatigue, at high strain amplitudes and a low number of cycles, the resistance to fatigue fracture can be improved by increasing the ductility of an alloy. In the area of high-cycle-fatigue, at low strain amplitudes but a high number of cycles, an improvement in the resistance to fatigue fracture can in principle be achieved by increasing the strength. The linear correlation between strength and high-cycle-fatigue strength is however only valid to a strength R_m of approx. 1000MPa. High-strength tool steels have a significantly higher strength than this, at which level the fatigue strength is no longer a function of the strength but of the defect size in the material. In powder metallurgy materials, defects are primarily caused by non-metallic inclusions, but also by oversized primary carbides and carbide clusters.

These theoretical principles were put into practice in the development of new microclean® grades. Due to their versatility, M4-type steels manufactured using the powder metallurgy route are used often. In many cases however a more exact matching of the properties of the tool material to the stress/loading profile of the application is necessary to optimise the tool life. For this reason, Böhler Edelstahl developed the highly ductile, low-carbide steel K890 microclean®, and the high-strength, carbide-rich steels S390 microclean® und S290 microclean® from the PM-M4 material. This enabled the properties of the materials to be optimally adapted to fit the stress/loading profile of the application.

In will be shown how the highly ductile material K890 microclean® was developed from the standard

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Topic: E – Advanced Tool Materials
Presentation type: Oral

PM-M4 steel type with a reduced primary carbide content and a high-strength matrix in order to achieve a higher resistance to fracture in applications with higher bending stresses. It will be demonstrated how an increase in the primary carbide content and the matrix strength compared to PM-M4 types resulted in the higher compressive strength and wear resistance of the steels S390 microclean® und S290 microclean®. Thanks to its universal property profile, S390 microclean® has established itself on the market as a problem solver which can be used in a wide variety of applications. In contrast, the highly-alloyed S290 microclean® can be seen as a tougher alternative to cemented carbides.

DEVELOPMENT OF A HYBRID TOOL STEEL PRODUCED BY SPARK PLASMA SINTERING

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ABSTRACT

The in-service performance of a tool can be correlated to the material properties and its production process. In the case of tool steels, involving high-speed and hot work tool steels, both factors contribute in determining the microstructure, on which properties as hardness, toughness, strength, wear and thermal fatigue resistance are dependent.
The microstructure of tool steels is constituted by a hardenable *metallic matrix* strengthened by a dispersion of *hard particles*, usually carbides. "Primary" carbides precipitate during solidification and have quite large size. With a given alloy chemistry, their phase composition, size, shape and distribution are determined by the course of solidification, in conjunction with the process of hot working the ingot to a mill shape. "Secondary" carbides precipitate in solid phase during solidification and even during following quenching-tempering heat treatment. In the case of hot work tool steels just secondary carbides are present.
The tool performance is determined by the primary carbide population and by the degree of secondary hardening of the metallic matrix. The distribution of the blocky carbides also has a decisive influence on the toughness of the tools, because large carbide clusters nucleate the microcracks which eventually cause rupture. I order to produce high speed steels with higher toughness the Powder Metallurgy process was introduced. This technology allows a finer and more homogeneous distribution of primary carbides, obtained during the atomization process of the elementary powder. PM steels shows considerable higher toughness/hardness ratio than the corresponding forged alloys. Their toughness, however, remains largely lower that that of hot work tool steels, which still represent the best product in all such application where a high toughness is required.
Hans Berns, professor at the University of Bochum (Germany), as first proposed a "hybrid" steel, obtained by mixing proper quantities of a coarse spherical high speed steel powder *II*, containing a high percentage of dispersed carbides, with a fine hot work tool steel powder *I*, of a low carbide content. After hot isostatic pressing (HIP) wear resistant areas II were dispersed in a tougher area I. After hardening and tempering, a double dispersion with 60%II-40%I showed 50% increase in fracture toughness over a reference PM high speed steel of comparable wear resistance and bending strength.
The aim of this work is the production of a steel having similar features, by means of a different sintering technique, namely the Spark Plasma Sintering (SPS). This method provides the consolidation-sintering aided by a current flow. The heating by Joule effect with pulsed continuous current allows very high heating rates of the graphite die (up to several hundreds degrees Centigrade per minute) where the powder to be sintered is placed. The magnetic fields and the micro plasma produced by the peculiar current wave forms enhance sintering, through the surface activation of the powder particles and favouring diffusion processes. The laboratory of metallurgy at Trento University in 2005 acquired one of the two SPS units in Italy. This technology offers very interesting advantages:

1. over consolidation techniques like hot isostatic pressing HIP it allows the direct production of the tool, thus avoiding the fabrication of a semi-finished product;
2. it allows to obtain full density components by using lower times and temperatures than those employed in HIP. In this way this techniques is an excellent candidate for sintering of nanostructured and/or nanosized powders, allowing to limit grain growth;
3. it allows to work under controlled atmosphere and in high vacuum;

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4. it permits to establish temperature gradients in the die, thus allowing the realization of multilayered or Functionally Graded Materials.
5. It allows to sinter mixed powders and to obtain "composite microstructures" like those to be produced within this project.

Two commercial available powders (Ospray) were selected for this work, namely an AISI H13 hot work tool steel and a AISI M2 high speed steel, having particle mean size lower than 20 microns. X-ray diffraction and metallographic analysis allowed to determine the powder constitution given by ferrite, austenite and carbides.

Samples in forms of 30mm diameter discs, 4-5mm height were produced by Spark Plasma Sintering under vacuum conditions. Optimum sintering conditions were individuated by varying both, sintering temperature and time: near-full density samples, preserving quite small grain size could be obtained at 1100°C for 5 minutes at a pressure of 60MPa. Starting from the base AISI H13 powder four different *blends* were obtained by adding 20, 40, 60, 80%wt AISI %M2. Samples where then vacuum heat treated by austenitizing at 1050°C x 30min and double tempering at 625°C x 2hours each. The base steels reached a final hardness of 430HV10 (~44HRC, AISI H13) and 493HV10 (~49HRC, AISI M2), respectively. The hardness of the *blends* increased linearly with increasing amount of AISI M2 powder.

An apparent fracture toughness Ka was determined using a procedure just proposed to overcome the difficulties in introducing fatigue precrack to small fracture toughness specimens. Notches depth with root radii of 50µm were electro discharge machined in 6x3x25.2mm^3 specimens. The ratio of notch depth to the specimen width (a/w) was set at 0.5. The decrease in stress triaxiality with increasing ρ causes a decrease in the stress that would initiate fracture, so that the Ka value is higher than that of K_{IC} measured on precracked sample. The present values highlight a good toughness level for AISI M2 (45MPam-1/2) whilst a comparatively low value could be observed for AISI H13 (50MPaxm-1/2). Values of 45MPa and 80MPa could be expected for the two steels on the base of experimental data obtained by the authors in the past. The toughness drops by increasing %AISIM2, i.e., by increasing hardness, even if not continuously. Low and highly dispersed values of Ka were observed by 60-40% and 40-60% blends. Fractographic analysis on AISI H13 allowed to detect a general ductile fracture accompanied by a strong intergranular component. The fracture path easily follows the particle boundary in correspondence of which a lot of round micropores were revealed by scanning electron microscopy (SEM). Micropores were also detected at the interface between AISIH13 particles and high speed steel. Accordingly, blends containing large fractions of AISI H13 behave worse than those containing high fractions of AISI M2. The large scatter for the blends having intermediate composition can be interpreted on the base of their intermediate fracture behaviour, influenced by almost interconnected fracture path of AISI H13. The present results suggest that sintering conditions can be further optimized for AISI H13.

Dry sliding tests were carried out using a block on disc configuration. A counterpart disc (40mm external diameter, 10mm width) of ASP2052 salt bath quenched and tempered to 70HRC was selected. The sliding speed was 0.55 m/sec and a load of 180N was applied for a total sliding distance of 3000m. The wear volume was determined by a stylus profilometer, after measuring the worn track profile in three different positions. The wear mechanism was given mainly by abrasion, as evidenced by the big scratches on the rubbing surface after the test. Traces of oxidation were also observed. According to the hardness increase, the wear resistance of the hybrid steels increased linearly with increasing %AISI M2. Microstructural analysis of the worn cross sections revealed a strengthening mechanism after which the plastic deformation of the relative soft AISI H13 regions is counteracted by the hard AISI M2 particles. Hence, the thickness of the plastically deformed layer linearly decreased with increasing amount of AISI M2.

STRESS STATE OF 12% Ni MARAGING STEEL AFTER A MODIFIED PROCEDURE OF PRECIPITATION HARDENING

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ABSTRACT

Compressive residual stresses in a surface layer can essentially improve fatigue resistance of the surface layer of a material of which exacting thermo-mechanically and dynamically loaded machine components and tools are made. In order to establish such a stressed state, a combination of cold deformation and heat treatment of maraging steel, which is suitable for tool manufacture, was applied. Mechanical hardening of the surface consists in shock peening, i.e. roto peening. Local plastic deformations of the surface layer of maraging steel in the soft state produce a higher density of dislocations and increase compressive residual stresses.

The combined application of cold deformation and precipitation annealing to specimens results in the formation of very fine precipitated phases. The microstructure obtained improves mechanical properties of the material concerned and provides the required variations of compressive residual stresses.

Keywords: maraging steel, residual stresses, roto-peening, precipitation hardening

1 INTRODUCTION

Tools for die casting of non-ferrous alloys consist of components subjected to strong dynamic and thermo-mechanical loads. The most critical tool components are elements of mould cavity, where different damages such as wear, micro cracks, and other imperfections will occur.

Maraging steels intended for tool production exhibit numerous technological advantages over common tool steels. Hardening of maraging steel consists in phase precipitation in a matrix of comparatively soft saturated nickel martensite. The process is relatively slow and related to the mechanism of diffusion, which proceeds uniformly through the tool-component cross-section. Consequently, the final properties of maraging steel are homogeneous across the entire tool cross-section. After precipitation hardening, relatively low compressive residual stresses will be present. This is the reason for looking for possibilities to improve residual stress variation in the thin surface layer. In the course of precipitation hardening of maraging steel, phases will precipitate in the matrix. They interfere with gliding of dislocations across a glide plane and thus essentially change mechanical properties of the material. A change of hardness in the initial phases of precipitation was described with an exponential equation by Squired in Wilson [1]. Hardness, indeed, has a strong influence on wear resistance of a material, particularly with die casting tools. Grum and Zupančič [2] found that after precipitation annealing at a temperature of 450 °C for two hours, operating hardness of a tool of around 44 HRC, tensile strength R_m of 1250 MPa and impact toughness KU of 16 J was achieved. It is proposed by the authors to apply the combination of cold deformation and precipitation annealing to improve the properties [3].

2 EXPERIMENTAL PROCEDURE

Specimen preparation: The initial state of maraging steel was a forged bar of 80 mm in diameter, from which flat specimens were cut in the transverse direction. The specimen preparation was followed by the first set of measurements of residual stresses.

The second set of measurements of residual stresses was performed after cold deformation of the surface by roto-peening using different parameter combinations, which provided different treatment intensity. The treatment intensity is determined by a kinetic energy of solid

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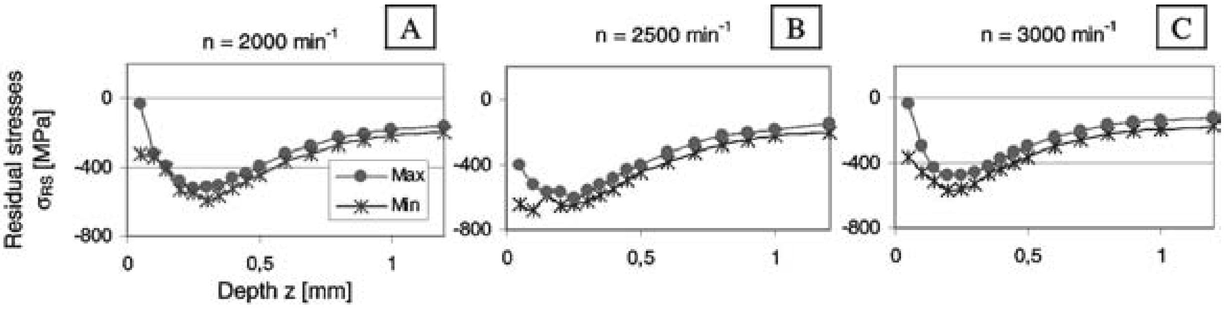

Figure 1: Residual-stress profiles of maraging steel after cold deformation of surface layer and after subsequent precipitation hardening at 450°C for2 hours.

particles impacting the specimen surface. It is given by the number of flap rotations (n = 2000, 2500, and 3000 min^{-1}).

The third set of measurements of residual stresses was performed after cold deformation of the specimens, which were additionally hardened by precipitation annealing. In the course of precipitation annealing very fine phases precipitated. They hardened the material and provided its final mechanical properties.

Residual stresses in the surface layer: The through-depth residual-stress profiles of the specimens were determined using the relaxation hole-drilling method. The method consists in measurement of deformations occurring in the hole vicinity. From the latter, equivalent homogeneous or incremental residual stresses are calculated. **Figure 1** shows three residual-stress profiles, i.e. the initial state, state after cold-deformation of the surface, and the state after subsequent additional precipitation annealing.

The »initial state« curve represents a profile of maximum residual stresses in the maraging steel specimens after having been cut from the bar. The profiles measured are located predominantly in the tensile zone and range between 100 and 200 MPa. Such an unfavourable stress state is a consequence of specimen cutting and subsequent flat grinding, which produces mainly tensile residual stresses in the surface layer. The stress state measured was a starting point for specimen comparison after hardening by cold deformation (the second set of measurements) and after hardening by combined hardening (the third set of measurements) respectively.

The residual-stress profiles after roto peening will shift to the compressive zone, which is represented by the »Roto 2500« curve. From the viewpoint of the stress variation the change indicates essential improvement of wear resistance of the material. The compressive stress zone is present through depth *z* of 1.0 mm, and attains the highest value, i.e. 1100 MPa, in a depth of 0.12 mm.

Cold deformation by roto peening was followed by additional precipitation annealing at a temperature of 450 °C for 2 hours. Subsequently, the highest compressive stress, i.e. 550 MPa, was obtained in a depth of 0.25 mm.

A comparison with the state prior to precipitation annealing showed the following differences:

– The residual stresses in the surface layer were essentially reduced. This was to be expected since this was a result of both the relaxation stresses at elevated temperatures and diffusion processes generating new phases with a changed crystal lattice.
– The difference between the minimum and maximum stresses will get smaller after precipitation, which is a result of the processes in precipitation annealing.
– After precipitation annealing the residual-stress profiles show a considerably lower value of the compressive stresses (550 MPa) with increased specimen hardness (44 HRC).

3 CONCLUSIONS

The investigation conducted on the combined hardening of maraging steel showed that treatment of maraging steel with preliminary mechanical surface hardening was efficient. Mechanical hardening introduces considerable compressive residual stresses in the surface layer. The latter interfere with the initiation and propagation of micro cracks and, in certain cases, essentially improve resistance of tools or machine components to thermo-mechancial fatigue.

The chosen maraging steel with 12% of nickel, which is suitable for the production of thermo-mechanically loaded tools, was treated by roto peening using three different parameter combinations. The flap rotation speeds of roto-peening flap *n* were equal to 2000, 2500, and 3000 min^{-1}. After treatment the measurements performed showed high values of the compressive residual stresses. After additional precipitation annealing of a specimen, hardness increases whereas the residual-stress variations will be reduced. The residual stresses persist to be of the compressive character, which ensures good wear resistance and fatigue resistance of the material.

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TRIBOLOGY

WEAR BEHAVIOUR OF PLASMA – SPRAYED Al-12Si/SiC COMPOSITE COATINGS UNDER DRY AND WATER – LUBRICATED SLIDING

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ABSTRACT

Powders of Al – 12Si with 25 and 50 wt %SiC particles were ball milled in a conventional rotating ball mil with alumina balls for 2h. The milled powders were plasma sprayed on to aluminum and stainless steel substrates to obtain Al matrix composites with high SiC weight fraction. The microstructure characteristics of the sprayed composites were investigated by metal microscopy, x-ray diffraction and scanning electron microscopy. Surface roughness, coating thickness and micro hardness tester were measured. The obtained results point out that an increase of SiC particles in Al – 12Si coatings was caused on the rising of the micro hardness values. The wear behavior of the coatings under dry – sliding and water – lubricated sliding against WC/Co counter material was studied using ball on disc for different loads. Increasing SiC content of coatings increase the wear resistant. Finally, the highest wear resistances of coatings were also found in the %20 SiC coating.

Keywords: plasma spray coating, aluminium matrix composite, microstructure, wear, water lubrication

1 INTRODUCTION

SiC particle reinforced aluminium matrix composites (SiC_p/Al MMCs) are widely applied in aerospace, transportation and electronic industries due to their excellent combinations of high specific strength, high specific stiffness, low coefficient of thermal expansion and excellent wear resistance [1]. The high wear resistance is generally attributed to the presence of the hard silicon particles distributed throughout the matrix [2].
Plasma sprayed coatings are used in a wide range of industrial applications primarily for wear resistance, frequently in a corrosive environment [3]. Recently, plasma spraying has been osed to produce coatings and near-net shapes of aluminium matrix composites [4].

2 EXPERIMENTAL SETUP

Al-12Si (METCO 52C-NS) powders with an average size of 45 μm were used as matrix material. The reinforcements consisted of SiC particles with an average sizes of 8 μm and 24 μm. The powder mixtures of aluminium with 25 and 50 wt %SiC particles, that is, Al12Si/25SiC_8, Al12Si/25SiC_{24}, Al12Si/50SiC_8, Al12Si/50SiC_{24} powder, were ball milled as plasma spray feedstock.
Plasma spraying was carried out using plasma equipment (Sulzer Metco MN 40kW) with 3MB – II type gun. The plasma spray conditions are listed in **Table 1**. The microstructure characteristics of the sprayed composites were investigated by metal microscopy, x-ray diffraction and scanning electron microscopy. Microstructure of Al12Si/50SiC_8 composite coating is shown in **Figure 1**. Surface roughness, coating thickness and micro hardness tester were measured. The coating exhibited a thickness of about 400 μm.
Wear test samples with size of Φ45mm are used. The wear behavior of the coatings under dry – sliding and water – lubricated sliding against WC/Co counter material was studied using ball on disc for different loads.

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Presentation type: Oral

3 TABLES AND FIGURES

Table 1: Conditions for plasma spraying process

Parametreler	
Voltage, V	60 – 70
Current, A	500
Primary gas, Ar, (L/min)	47
Secondary gas, H_2, (L/min)	7
Powder feed rate, (g/min)	90
Spray distance, mm	130

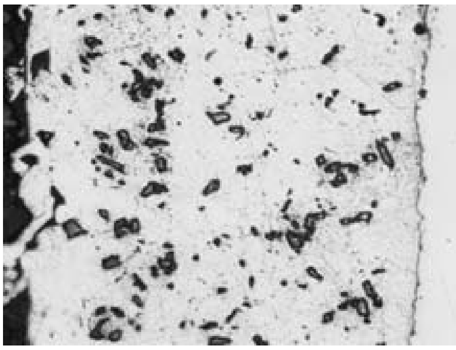

Figure 1: Microstructure of Al12Si/50SiC_8 composite coating

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ANALYSIS OF ABRASIVE WEAR RESISTANCE OF THE D2 TOOL STEEL IN RELATION TO HEAT TREATMENT

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ABSTRACT

Heat treatment significantly influences wear resistance of the tool steels. D2 is suitable for the cutting and forming cold work tools. Heat treatment of the parts must be in accordance with the function of the tool positions. When high austenitized, it is suitable for application of PVD coatings which further enhance its operating characteristics. This article refers to relationship between the hardened-and-tempered condition of this tool steel and its abrasive wear resistance. The samples were subjected to the different heat treatment parameters, then were tested using dry sand and rubber wheel method. Microstructural analysis of the representative samples is presented and related to the obtained results of the dry sand / rubber wheel test.

Keywords: D2 tool steel, heat treatment, abrasive wear resistance

1 INTRODUCTION

D2 steel is one of the most commonly used steels in tool making. It is suitable for production of cutting inserts, as it can reach high hardness level and its abrasive resistance is good. When the cutting inserts have bigger dimensions, the same steel type can be used, but with the different heat treatment parameters, so that higher toughness is obtained. Goal of the article is to correlate the heat treatment parameters (austenitizing and tempering) temperature with the abrasive resistance.

2 EXPERIMENTAL SETUP

Dry sand and rubber wheel test (ASTM G 65-94) was used for determination of the abrasive wear resistance. The samples along with the volume loss are specified in **Table 1**.
Microstructural analysis of the following sample numbers were performed: 1, 2, 5, 7, 8, 10, 11, 12, 15, 17, 20. These samples were selected in accordance with the results of the dry sand and rubber wheel test.

3 CONCLUSION

The abrasion resistance of this steel is influenced primarily by the portion of retained austenite and carbide in the microstructure: content, form and size and distribution of Cr and W-carbides (**Figure 1**). The austenitizing and especially tempering temperature determined dynamics and formation of suitable microstructure for optimum abrasive resistance in defined working condition.

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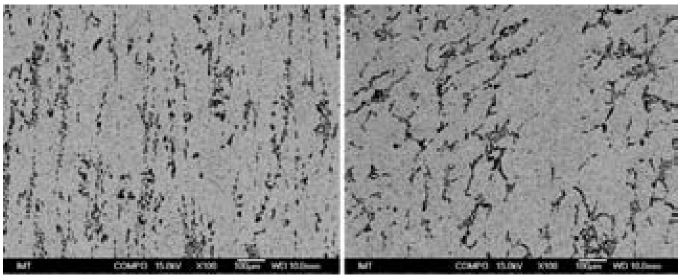

Figure 1: Distribution of carbides (SEM micrographs) in two samples; left: No. 2 (low volume loss) and right: No. 10 (higher volume loss)

Table 1: Volume loss of the samples used for dry sand and rubber wheel test

Sample No.	Austenitizing temperature, °C	Tempering temperature, °C	Hardness, HRC	Average hardness, HRC	ΔV, mm^3	Average volume loss, mm^3
1	1030	160	61,5	61,7	10,3694	8,82
2	1030	160	61,5		6,1783	
3	1030	160	62		9,9108	
4	1030	200	60	60	11,3885	12,77
5	1030	200	60		12,4968	
6	1030	200	60		14,4331	
7	1080	500	61,5	61,5	7,6943	6,54
8	1080	500	61,5		4,9300	
9	1080	500	61,5		7,006	
10	1080	515	60	60,2	13,0573	10,59
11	1080	515	60		12,1911	
12	1080	515	60,5		6,5222	
13	1030	220	60,5	60,2	11,6561	10,44
14	1030	220	60		9,7452	
15	1030	220	60		9,9108	
16	1030	250	60	59,7	11,2739	11,11
17	1030	250	60		11,9618	
18	1030	250	59		10,0892	
19	1030	350	59,5	59,5	13,9745	10,97

INFLUENCE OF DEEP-CRYOGENIC TREATMENT ON TRIBOLOGICAL PROPERTIES OF P/M HIGH-SPEED STEEL

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ABSTRACT

The aim of the present investigation was to determine the influence of deep-cryogenic treatment parameters (treatment time and temperature) on the tribological performance of powder-metallurgy (P/M) high-speed steel. Special emphasis was put on abrasive wear resistance and resistance to galling under dry sliding conditions. Tribological tests indicate that deep cryogenic treatment contributes to improved abrasive wear resistance as well as to better galling properties of P/M high-speed steel.

Keywords: deep-cryogenic treatment, high-speed steel, galling, friction

1 INTRODUCTION

In metal forming industry tools can be exposed to very complex and surface demanding conditions, which are the result of different effects (mechanical, thermal, chemical or tribological loading). Therefore tool materials need to fulfill many requirements, which, to a certain extent, are not mutually compatible, i.e. high hardness and high toughness. Beside the material's intrinsic properties, tribological properties of the tool surface, including abrasive wear resistance, coefficient of friction and resistance to galling, will determine tool's operating lifetime. It has been reported that the microstructure of the tool steels can be substantially modified by vacuum heat treatment in conjunction with a deep-cryogenic treatment and that the ratio between the hardness and the fracture toughness can be optimized. On the other hand, plasma nitriding was found to form surface layers of exceptional tribological properties, exhibiting low friction and reduced tendency to pick-up work material. By combining deep-cryogenic treatment and plasma nitriding, individual effects could be combined, resulting in greatly improved mechanical and tribological properties of forming tools.

The aim of our work was to investigate the influence of deep-cryogenic treatment parameters (treatment time and temperature) on the tribological performance of powder-metallurgy (P/M) high-speed steel. Special emphasis was put on abrasive wear resistance and resistance to galling under dry sliding conditions.

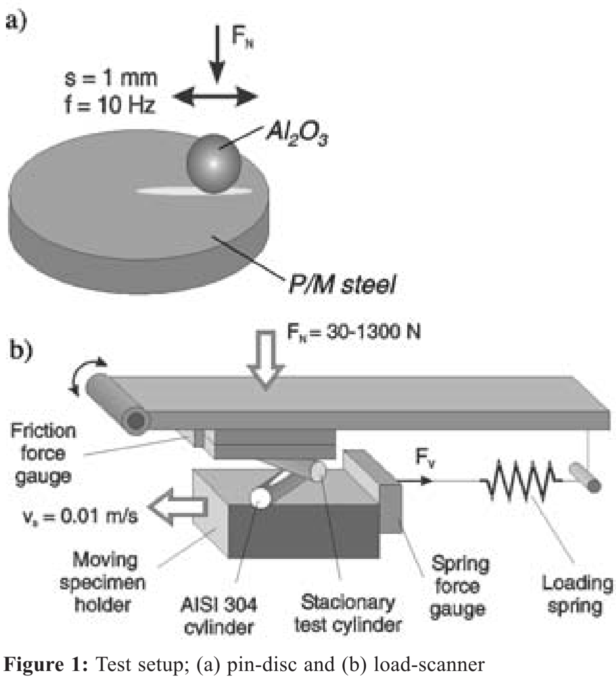

Figure 1: Test setup; (a) pin-disc and (b) load-scanner

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Presentation type: Oral

2 EXPERIMENTAL SETUP

Abrasive wear resistance of P/M high-speed steel S390 was tested under reciprocating sliding conditions using alumina ball (**Figure 1a**), while galling resistance against austenitic stainless steel was determined in a load-scanning test rig (**Figure 1b**). Tribological test were evaluated in terms of high-speed steel wear volume, coefficient of friction under reciprocating sliding, friction variation with load, and critical load for galling initiation and stainless steel transfer layer formation.

Tribological tests indicate that deep cryogenic treatment contributes to improved abrasive wear resistance as well as to better galling properties of P/M high-speed steel.

WEAR RESISTANCE OF THIN PROTECTIVE LAYERS IN ABRASION CONDITIONS UNDER HIGH-PRESSURES

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[2]FSB, Zagreb

On parts with dimensional criteria failure allows maximum wear-out 0.1 to 0.2mm alternative is application of thin protective layers but higher hardness then abrasive.

That possibility is examined on usually used carbonated layers comparision with vanadium alloy (duplex procedure), bor alloy, PVD CrN layer and SiC+DLC protective layers. Research is carried out:

I- laboratory method dry sand / rubber wheel (ASTM G65-94) and II- in practice on press example for draining sunflower oil.

It was determined that wear resistance depends not only about surfice layer hardnes but about connection quality and underlayer hardnes as well.

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EVALUATION METHODS AND SURFACE ENGINEERING TECHNIQUES FOR IMPROVED GALLING PROPERTIES OF FORMING TOOLS

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ABSTRACT

In recant years the main focus on improving tribological properties of forming tools has been on modifying the surface of the tool, including new forming tool steels, improved surface finish, and application of surface treatments and coatings. In order to evaluate, compare and select suitable materials and/or surface engineering techniques for forming tools simple but reliable and repeatable test methods for galling evaluation are needed. Recent results show that the galling tendency can be greatly reduced by proper surface engineering of the tool surface, with the selection being greatly dependant on the type of work material.

Keywords: galling, surface engineering, tribological evaluation, friction

1 INTRODUCTION

Surface finish concerns are critical for the tolerances of many components made of metal sheet. Major obstacles to the generation of acceptably smooth surfaces are smudge, orange peel and galling, with galling also being one of the major causes for tool failure in metal forming processes. Generally, lubricants are used in order to avoid such problems. However, the very high stresses and high degree of plastic deformation experienced in forming will inevitably lead to metal-to-metal contact, high coefficient of friction and galling initiation. Galling develops gradually as an accumulation of material pick-ups on the tool surface during the forming operation, causing aesthetic problems with the work-peace and establishing unstable friction conditions for the proceeded forming process.

In recent years the main focus on improving tribological properties of forming tools has been on modifying the surface of the tool, including new forming tool steels, improved surface finish, and application of surface treatments and coatings. In order to evaluate, compare and select suitable materials and/or surface engineering techniques for forming tools simple but reliable and repeatable test methods for galling evaluation are needed.

The aim of this paper is to compare and evaluate different test methods in terms of galling properties assessment of forming tool material. Furthermore, different surface engineering techniques, including different grades of grinding and polishing, plasma nitriding and different hard coatings, are compared in terms of galling properties of forming tool steel when sliding against different work materials. The Pin-on-Disc configuration was included as the most simple and generally used model test in tribology research. The second method was a modified Block-on-Cylinder test, where, it is possible to scan the contact load during each test run through a spring loading system. A load-scanning test configuration, which involves two crossed cylinders forced to slide against each other under a constant speed and gradually increasing load, was used for surface engineering techniques comparison and evaluation. It gives the capability to compare different tool steels, surface finishes and treatments, and hard coatings in terms of friction increase, material transfer intensity and critical load for galling initiation.

2 EXPERIMENTAL

Material used in this investigation was a powder metallurgy cold work tool steel, VANADIS 6 (2.1%C, 1.0%Si, 0.4%Mn, 6.8%Cr, 1.5%Mo, 5.4%V) from Uddeholm Tooling AB, Sweden. Test samples in the shape of cylinders (ϕ 10mm, 100mm long) and discs (ϕ 30mm) were

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cut out from cylindrical bars, hardened and tempered to 850 HV, and ground to an average roughness value of ≈ 0.25 μm. Additionally, three groups of VANADIS 6 steel specimens were prepared in terms of surface engineering. First group was thermo-chemically treated, including plasma nitriding with and without compound layer, and hardening. Second group was prepared using different grades of grinding and polishing, while the third group of specimens was coated with five different coatings (TiN, TiB2, VN, TaC and DLC) using commercial PVD processes.

Test specimens were investigated in terms of galling properties assessment, using three different test method. The pin-on-disc configuration was included as the most simple and generally used model test in tribology research. The second test method was a modified Block-on-Cylinder test, where it is possible to scan the contact load during each test run through a spring loading system. Finally, a new load-scanning test method [1,2], was included, involving two crossed cylinders which are forced to slide against each other under a constant speed and gradually increasing load, where each point along the contact path of both specimens experience a unique load. Effect of different surface engineering technique was evaluated against AISI 304 steel, Ti6Al4V alloy and Al98Mn1Mg1 alloy.

3 RESULTS

The load-scanning test rig was found to be the most suitable method to evaluate galling properties of forming tool steels. It gives the capability to compare different

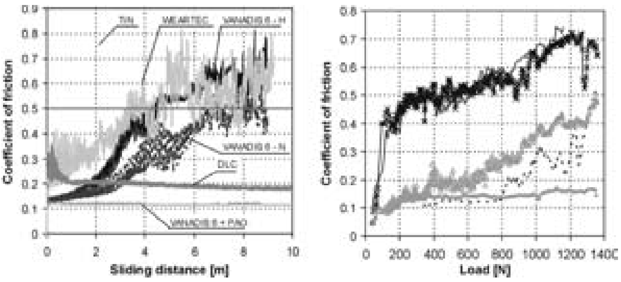

Figure 1: Galling test results, performed on (a) Pin-disc and (b) Load-scanner test rig

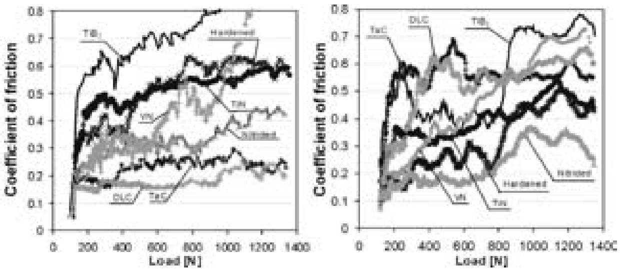

Figure 2: Galling properties of surface engineered VANADIS 6 steel against (a) stainless steel and (b) aluminium alloy

tool steels, surface finishes and treatments, and hard coatings in terms of friction increase, material transfer intensity and critical load for galling initiation. Besides being very simple it also provides results representing a whole range of loads during one single test run (**Figure 1**).

Results from load-scanner indicate that the galling tendency can be greatly reduced by proper polishing of the tool surface (**Figure 1b**). If polished after treatment, plasma nitrided surface will provide further improvement in friction and wear properties of the tool steel and especially for aluminium and titanium alloys reduce the galling tendency. On the other hand, coating selection for improved galling performance of coated forming tool steel greatly depends on the type of work material. In the case of stainless steel, carbon-based coatings provide the best protection against the work material transfer, while forming of aluminium and titanium based alloys, requires nitrides rich surface (**Figure 2**).

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INFLUENCE FROM TOOL ROUGHNESS ON THE RISK OF WORK MATERIAL ADHESION AND TRANSFER

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ABSTRACT

The influence from surface roughness of a forming tool surface on its ability to pick up work material has been investigated. One hardened powder metallurgical forming tool steel was treated by polishing, grinding and tumbling to achieve different surface roughnesses. Dry laboratory load scanning tests were performed against work materials generally known to be difficult to form; one austenitic stainless steel and one aluminium alloy. Post test examination of the tool material surfaces was performed by scanning electron microscopy (SEM). The following main conclusions are made.

Adhesion and transfer can occur almost instantaneously. A single passage and a very short sliding distance is enough to transfer a fully covering layer and even larger lumps. The tested work materials exhibited different dependence on the tool roughness, and there is no generally valid critical surface roughness below which adhesive transfer will not occur.

Keywords: surface roughness, forming tool, load-scanner, adhesion

1 AIM

The influence from surface roughness of a forming tool surface on its ability to pick up work material has been investigated. One aim was to investigate if there exists a tool roughness below which there is no risk of work material adhesion [1, 2].

2 EXPERIMENTAL

Dry laboratory load scanning [3] was performed with one hardened powder metallurgical (PM) forming tool steel tested gainst two work materials generally known to be difficult to form; austenitic stainless steel (SS) and one aluminium alloy (Al), see Table 1. The PM steel was treated by polishing, grinding and tumbling to achieve different surface roughnesses R_a [µm]; PM A: 0.01, PM B: 0.03, PM C: 0.10, PM D: 0.12 and PM E 0.10. A and B were polished, C and D ground and E tumbled. Tested surfaces were examined by SEM.

Table 1: Designation, nominal composition and mechanical properties of the test materials

Materials	Abbreviation	Alloying elements (wt %)	Hardness $(HV)_{5N}$	Elastic modulus [GPa]
PM tool steel	PM	8.5 V, 4.5 Cr, 3.7 W, 3.2 Mo, 1.1 C	700	220
304L	SS	0.02 C, 18.5 Cr, 9.5 Ni	380	200
Al1MgSiPb	Al	1.0 Mg, 0.6 Si, 0.6 Pb	120	70

3 RESULTS

Generally, the coefficient of friction and scatter in the friction values increases with the roughness of the PM steels, see **Figure 1** [1]. It is also seen that the Al alloy gave the highest friction. The smoothest PM surface gave a very low and usually stable friction level for SS. Note also that the tumbled PM E gave a lower friction than the ground PM C though their R_a-values were the same.

A strong correlation between friction level and work material adhesion to the PM steel surface was found by observations in the SEM. Material adhered in the form of a thin layer and/or small patches (SS/PM A and SS/PM B), extensive patches (SS/PM C-E) or as fully covering layers (Al/PM A, C and D).

4 CONCLUSIONS

Testing in the load scanner has proven that adhesion and transfer can occur almost instantaneously. A single pas-

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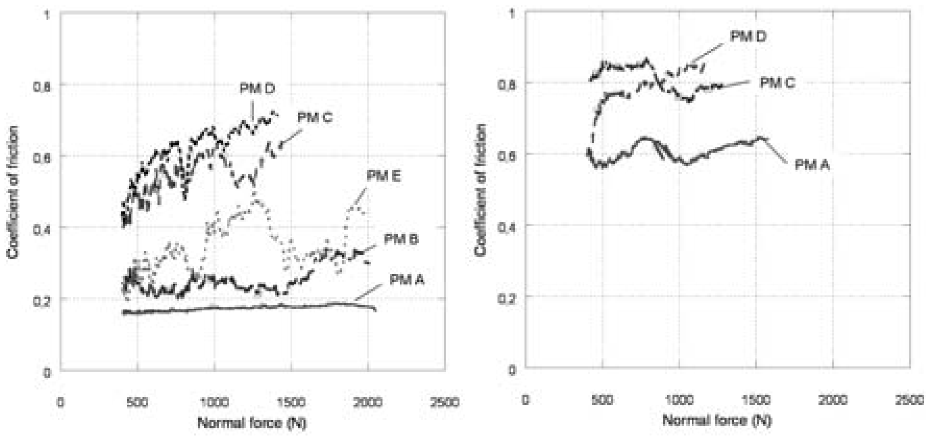

Figure 1: Typical coefficient of friction vs. normal load curves. a) SS against PM A-E b) Al against PM A, C and D.

sage and a very short sliding distance is enough to transfer a fully covering layer and even larger lumps.

There is no generally valid critical surface roughness below which adhesive transfer will not occur independently of the material combination. The tested work materials exhibited very different dependence on the tool roughness.

The transferred material may form thin layers, thicker layers or lumps. The formation of thin layers is generally associated to relatively low friction while the two other forms are accompanied by high friction.

The SS work material is exceptional by its sensitivity to the roughness of the tool surface. Against the smoothest tool surface it gives very limited metal transfer and low friction while against the rougher ones it gives severe transfer and high and irregular friction, and a critical roughness exists around R_a 0.01 probably exists.

The aluminium showed adhesion and transfer against all surfaces, even for the lowest load. Obviously, for this material even the finest polishing of the tool surface (Ra as low as 0.01 μm) is not enough to avoid adhesion.

The tumbling operation showed some advantage over grinding when comparing tool surfaces of very similar Ra. Both the duplex and the austenitic stainless steel showed significantly less adhesion and transfer against the tumbled than against the ground tool rod. The reason being the absence of sharp peaks in the tumbled surface.

Two models for material and transfer are proposed, one for work materials with relatively soft oxides, and on for materials with oxides significantly harder than the parent metal.

The critical surface roughness to avoid adhesion and metal transfer for materials with soft oxides is related to the oxide thickness. Any roughness peaks in the tool surface must not penetrate the oxide.

For the materials with hard oxides such as the Al alloy, the oxide layer may break up and hard oxide fragments may scratch the tool surface to activate chemical bonding and also to give a mechanical "grip" between tool and work material.

Instead of striving for the finest tool polishing, applying a coating to the tool with a hardness exceeding that of the metal oxide is recommended when forming Al-alloys and other metals with hard oxides.

Acknowledgements

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APPLICATIONS OF NANOTECHNOLOGY

2nd INTERNATIONAL CONFERENCE ON HEAT TREATMENT AND SURFACE ENGINEERING OF TOOLS AND DIES
Bled, 25–28 May 2008

CHARACTERIZATION OF MULTILAYER PACVD COATINGS FOR HOT-WORKED TOOL STEELS USING ELECTRON SPECTROSCOPY TECHNIQUES

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ABSTRACT

Multilayer Ti(B-N) layers coating was deposited by plasma-assisted chemical vapour deposition (PACVD) on treated AISI H11 steel and have been characterized using electron spectroscopy techniques. The thickness of the total coating is 1.6 µm and comprised 21 layers. Earlier studies of such coatings using X-ray diffraction (XRD), energy dispersive spectroscopy (EDS), and wavelength dispersive spectroscopy (WDS) suffer from their relatively large analysis depths. In this work, Field-emission Auger electron spectroscopy (FE-AES) was used to examine the composition of the multilayered films since it has a smaller analysis depth. AES line-scans across cross-sectioned samples and AES depth profiling were used and are shown to be well suited for characterizing these multilayered coatings

Key words: plasma-assisted chemical vapour deposition (PACVD); hard TiN/Ti(B-N) coating, AISI H11 tool steel, Field emission Auger electron spectroscopy (FE-AES)

1 INTRODUCTION

Hard thin films, such as Ti(B-N), are well known for providing surfaces with a high hardness, and good corrosion and wear resistance, giving them a wide range of industrial applications [1-5]. Duplex treatments consisting of plasma nitriding the steel surface first and then using plasma-assisted chemical vapour deposition (PACVD) to deposit the hard coating has proven successful in improving the wear, fatigue and corrosion resistance, as well as the load-carrying capability, of steel substrates [16-21]. The increasing industrial demand for improved hard coatings with tailored properties for die casting and forging tools requires the development of multi-element and/or multi-phase hard films, as well as a better understanding of their composition.

Most of the published studies of Ti(B-N) film compositions use scanning electron microscopy (SEM), wavelength-dispersive electron-probe microanalysis (EPMA) [6-8], Rutherford backscattering spectroscopy (RBS) [5], Bragg-Brentano X-ray diffraction (XRD) [2,4-8] and in some cases transmission electron microscopy (TEM) [4, 6]. These techniques suffer from their relatively large analysis depths or lack of depth resolution. In this investigation we have studied PACVD-deposited thin films using Auger electron spectroscopy, which is better suited for Ti(B-N) multilayer characterization We demonstrate that a combination of techniques such as AES depth profiling and FE-SEM give a better insight into the chemistry and structure of multilayered Ti(B-N) thin films. WDS measurements were made on the same multilayered structure for comparison.

2 EXPERIMENTAL

AES depth-profile analysis

The AES instrument used was a Thermo Scientific VG Microlab 310-F composed of two ultra-high-vacuum chambers, one for sample insertion and one for the analysis. The electron gun has a thermally assisted Schottky field-emission source that provides a stable electron beam in the range of 0.5 to 25 keV. The electron energy analyzer is of the double-focusing spherical secureor type with an electrostatic input lens and can provide an energy resolution of between 0.02% and 2%. The spectrometer has five electron detectors and spectra were ac-

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Topic: G – Applications of Nanotechnology
Presentation type: Oral

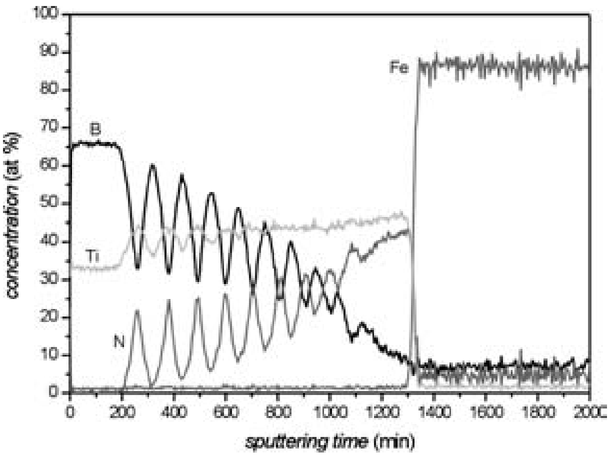

Figure 1: AES sputter depth profile of the TiB_2/Ti(N-B)/TiN

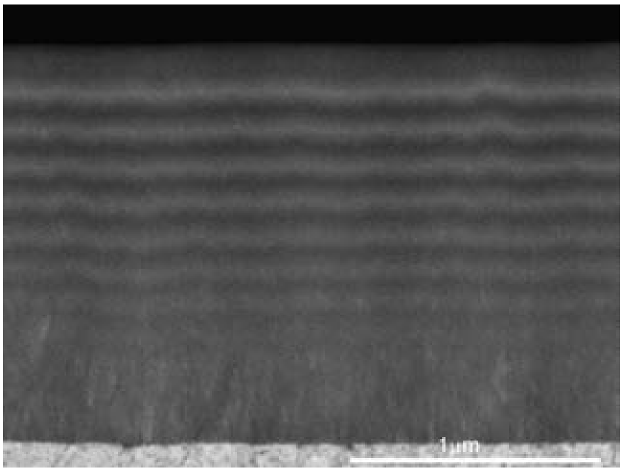

Figure 2: BEI of the nanostructure multilayered coating of a multilayered coated hard sample in cross-section

quired with a constant retard ratio (CRR) of 4, which provides an energy resolution that is 0.5% of the pass energy. For the cross-section studies, samples were cross-sectioned using a JEOL Cross Section Polisher, Model SM-09010, and analyzed using an AES linescan at 10 keV beam energy. AES depth profiles of the hard coatings were also measured with a 10 keV electron beam and the sample was sputtered with a 1.2 nA current of Ar ions at 3 keV. The AES data were acquired using Eclipse V2.1 ver07 software and processed using CasaXPS software.

3 RESULTS

The Auger sputter depth profile is shown in **Figure 1**. The Auger spectra were processed using equation. The TiB_2 layer can be seen at the first part of the depth profile. The nine boron-depleted and nine boron-rich regions can be seen in the sputter depth profile, and these are much better resolved than the corresponding regions in the Auger linescan Further, not all 18 alternating layers were resolved in the Auger linescan. The close agreement with the boron concentration behaviour in the Auger line scan and the Auger sputter depth profile indicates the absence of preferential sputtering in the depth profile.

4 CONCLUSION

We have shown that for studies of multilayer TiN/Ti(B-N)/TiB_2 hard coatings, Auger sputter depth profiling are excellent suited to determine their composition.

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DESIGN OF RECYCLE PROCESS OF COLOR FILTER USING ARC-FORM TOOL

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ABSTRACT

Current study presents a new process of electrochemical machining using a design precision recycle system and an arc-form electrode offering faster performance in the removal of the color filter surface's ITO layer. Through establishing an ultra precise recycling process removes the microstructure of ITO layer, this helps the semiconductor optoelectronic industry to reduce both production costs and pollution. In the current experiment, the author takes a 5^{th} Generation TFT-LCD for instance. The design features of the removal processes for a thin-film microstructure and the tool design of the electrodes are of major interest. The design of tool electrodes is used with continuous and pulsed direct current in the electrochemical machining experiment. A thin thickness with a smaller end radius of the arc-form tool provides an effective current density and a larger discharge space and better removal effect. High flow velocity of the electrolyte elevates the discharge mobility and improves the removal effect. Pulsed direct current can improve the effect of dregs discharge and is advantageous to associate with the fast feed rate of the workpiece. A color filter with a fast feed rate is combined with enough electric power to provide highly effective removal. A precision recycling process is presented using an effective arc-form tool in electrochemical machining. It just needs a short period of time to remove the ITO layer easily and cleanly.

Keywords: color filter, ITO layer, microstructure, recycle process, arc-form tool, electrochemical machining

1 INTRODUCTION

Flat panel LCD's are the most common displays now in use in many environments. The largest market by volume and value remains the PC (Personal Computer) monitor market in LCD applications, and the market is still expanding. [1]. The primary cause for a decrease in yield rate for LCD production is "dust". When these dust particles become attached to the LCD substrate they impair its function causing breaks in the circuit, short-circuits or poor performance. [2]. Electrochemical machining (ECM) can be applied to electrolytic components (silicon chips, VLSI/ULSI chips). This technique is based on the electrochemical reaction between an electrode and a workpiece. The main difficulty lies in the design of tool electrodes considering the complicated process of metal removal [3]. Datta [4] showed that the gap width between electrode and workpiece directly influences the current condition and the discharge dreg of the electrolyte. In ECM, good surface quality of the workpiece was obtained through the arrangement of the experimental conditions [5]. This research presents a new design modus using electrochemical machining and an arc-form tool as a precision recycling process of ITO layer removal from optoelectronic flat panel displays' color filter surface. An effective and low-cost recycling process is developed.

2 EXPERIMENTAL SETUP

The experimental set-up of the precision recycle process of ITO layer removal from color filters is schematically illustrated in **Figure 1**. The workpiece material uses the 5^{th} Generation LCD panel (1300×1100mm; 0.7mm). The electrolyte is $NaNO_3$ of 15%wt and PO4-3-P 5%wt. The amount of the removal reduction from the color filter surface after electrochemical machining for ITO layer is 150nm. The current rating is 50, 100, 150, 200A. The feed rate of workpiece (color filter) ranges from 150 to 500 mm/min. The flow rate of electrolyte is 5, 10, 15, 20, and 25 L/min. The temperature of the electrolyte is 40, 50, 60, and 70 °C. The diameter of the positive-electrode is ϕ30mm. The thickness (t) of the arc-form tool (negative-electrode) is 5, 10, 15, and 20mm. The end radius of the arc-form tool is 0.5, 1, 1.5, 2, and 2.5mm. The pulsed

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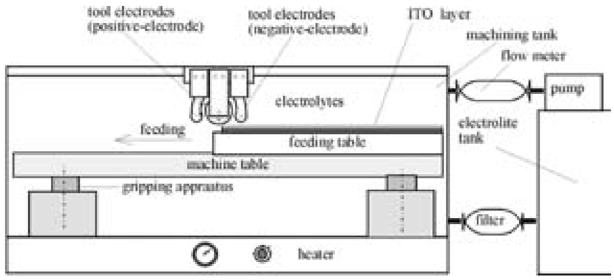

Figure 1: Experimental set-up

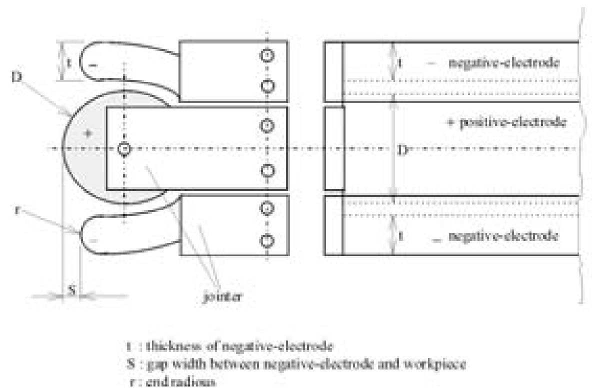

Figure 2: Geometry of electrode

period (on/off time) uses 100 ms / 100 ms compared with the continuous direct current. Their settings are shown in **Table 1**. All workpieces are put to water cleaning after the recycling process and then air-dried. The produced ITO layer is measured in more than two locations byα-step.

3 TABLES AND FIGURES

Table 1: Experimental parameters

current rating (A)	50, 100, 150, 200
feed rate of workpiece (mm/min)	150 to 500
pulsed period (on/off time) (ms / ms)	100 / 100, 100 / 200, 100 / 300, 100 / 400 100 / 500
flow rate of electrolyte (L /min)	5, 10, 15, 20, 25
temperature of the electrolyte (°C)	40, 50, 60, 70
pulsed period (on/off time) (ms / ms)	100/100, 100/200, 100/300,100/400, 100/500
thickness of the arc-form tool (mm)	5, 10, 15, 20
End radius of negative-electrode (mm)	0.5, 1, 1.5, 2, 2.5

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MATHEMATICAL MODELLING AND PROCESS SIMULATION

GENETIC PROGRAMMING AND JOMINY TEST MODELLING

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ABSTRACT

The paper presents the use of genetic programming method for Jominy test modelling for 51CrV4 (DIN) spring steel. The experimental data on chemical composition, distance from the specimen face and Jominy test results of 75 batches were collected. On the basis of experimental data set, mathematical model for Jominy test was developed by genetic programming method. The model developed during the simulated evolution was tested also on the basis of experimental data on 871 batches. The results show that the proposed concept can be easily used also in practice.

Keywords: hardenability, Jominy test, modelling, genetic programming

1 INTRODUCTION

Hardenability is a steel property which describes the depth to which the steel may be hardened during quenching. The Jominy test is a method for determining the hardenability of steel which involves heating a test piece from the steel (25mm diameter and 100mm long) to an austenitising temperature and quenching from one end with a controlled and standardised jet of water. After quenching the hardness profile is measured at intervals from the quenched end.

Several attempts for Jominy test modelling have been made [1-4] with also included artificial intelligence approach [3].

In the paper the genetic modelling method for Jominy test is proposed. Genetic programming has been successfully implemented into several manufacturing processes [5, 6].

2 EXPERIMENTAL SETUP

The experiment was performed with 51CrV4 (DIN) spring steel specimens. Distance from the specimen face (1.5mm, 9mm, 15mm, 30mm, 50mm) and chemical composition (percentage of *C*, *Si*, *Mn*, *P*, *S*, *Cr*, *Mo*, *Ni*, *Al*, *Cu*, *Ti*, *V*, *Sn*, *Ca*, *N*) were used for mathematical modelling of Jominy test.

Training data set (74 batches) was used for surface roughness and tool life prediction, whereas the testing data set (871) was used for verifying the model. The average chemical composition of 51CrV4 (DIN) spring steel used in the research is shown in **table 1**.

3 JOMINY TEST MODELLING

Genetic programming is probably the most general evolutionary optimization method. The organisms that undergo adaptation are in fact mathematical expressions (models) for Jominy test prediction consisting of the available function genes (i.e., square root and basic arithmetical functions) and terminal genes (i.e., independent input parameters, and random floating-point constants). In our case the models consist of: function genes of addition, subtraction, multiplication, division and square root operation, terminal genes distance from specimen face *D* and chemical composition (percentage of *C*, *Si*, *Mn*, *P*, *S*, *Cr*, *Mo*, *Ni*, *Al*, *Cu*, *Ti*, *V*, *Sn*, *Ca*, *N*).

Random computer programs of various forms and lengths are generated by means of selected genes at the beginning of simulated evolution. Afterwards, the varying of computer programs during several iterations, known as generations, by means of genetic operations is performed. After completion of varying of computer programs a new generation is obtained that is evaluated and compared with the experimental data, too. The process of changing and evaluating of organisms is repeated until the termination criterion of the process is fulfilled. This was the prescribed maximum number of generations.

With the previously mentioned genes, the simulated evolution of the genetic programming system produced the following best model for prediction of the Jominy test:

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Table 1: The average chemical composition of 51CrV4 spring steel used in the research

Training data set	%	Average	Stdev	Testing data set	%	Average	Stdev
	C	0,524	0,012345		C	0,520875	0,011203
	Si	0,280667	0,03214		Si	0,276695	0,033691
	Mn	1,006267	0,060607		Mn	0,997613	0,064439
	P	0,012973	0,0022		P	0,012589	0,00226
	S	0,005133	0,003087		S	0,004957	0,002668
	Cr	1,104933	0,068353		Cr	1,103564	0,063644
	Mo	0,038533	0,020139		Mo	0,043035	0,023867
	Ni	0,102667	0,019982		Ni	0,10568	0,020528
	Al	0,016587	0,005825		Al	0,016961	0,005442
	Cu	0,160133	0,029319		Cu	0,161231	0,029322
	Ti	0,003493	0,003126		Ti	0,004577	0,005245
	V	0,139067	0,022609		V	0,141328	0,021955
	Sn	0,011093	0,001595		Sn	0,011248	0,001757
	Ca	0,001283	0,000365		Ca	0,001283	0,000374
	N	0,010587	0,001908		N	0,010857	0,002206

$$
\begin{gathered}
29.27 + 60.38\,C - D\,Si + \\
Mo\left(Mo - Mn^2 (60.38 + 60.38\,C + Mn)\,Mo + Si\left(120.76\,Si + 120.76\,Si^2\right)\right. \\
+ Si^2\left(60.38\,Si + 120.76\,Si^2 + Mn\,Si^7\right) \\
\left. + Si\left(120.76\,Si + 362.28\,Si^2 + Si\left(120.76\,Si + 181.14\,Si^2\right)\right)\right)
\end{gathered}
$$

with average percentage deviation for training data (47 batches) 4.22%.

The average percentage deviation for testing data (871 batches) is 4.37%.

4 CONCLUSIONS

In this paper prediction of the Jominy test by genetic programming was performed. Prediction models were developed on the basis of experimental data on the chemical composition and distance from the specimen face of the 51CrV4 (DIN) spring steel. Training data set (74 batches) was used for surface roughness and tool life prediction, whereas the testing data set (871) was used for verifying the model. Genetic programming model predicts Jominy test with average percentage deviation for training data (47 batches) 4.22% and 4.37% for testing data (871 batches). Research also shows that the influence of the percentage of *Ca*, *Cr*, *Sn*, *V* and *Ti* is relatively small so further investigation of chemical composition influence is needed. The proposed evolutionary approach is efficient for Jominy test prediction.

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PREDICTION OF HARDNESS DISTRIBUTION WITHIN AXIALLY SYMMETRICAL WORKPIECES THEREUPON HIGH PRESSURE GAS QUENCHING

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ABSTRACT

The aim of a joint project among: the Stiftung Institut für Werkstofftechnik (IWT) the University of Bremen; the company Ipsen International, and the Faculty of Mechanical Engineering, the University of Zagreb is to develop a 2-D computer programme for prediction of hardness on the axial section of axially-symmetrical workpieces of any complex shape, thereupon high pressure gas quenching.

The hardenability for two steel grades and the cooling dynamics under two gas pressures are measured using the unique facility at IWT.

Keywords: high pressure gas quenching, hardenability, hardness distribution

1 INTRODUCTION

High pressure gas quenching in vacuum furnaces offers the following advantages in relation to other quenching methods [1]: hardness uniformity, smaller distortion, clean and bright surface, higher level of automation and possibility to control the quenching dynamics. Because of the many influential factors like: steel hardenability, workpiece dimensions and geometry, mass and loading arrangement of the batch, vacuum furnace characteristics, parameters of the quenching medium (art, pressure and velocity of the gas), accurate prediction of hardness distribution is a difficult and complex task.

Except the initial results of investigations [4] there is a lack of hardenability data for different steel grades and measured data of heat transfer coefficient for various heat treatment conditions and parameters, which are the prerequisites for any complex shape hardness prediction. The first purpose of this project is to establish a hardenability database for different structural and tool steels cooled with various gases pressures and flow velocities. The hardenability of steels when gas quenched cannot be measured by the known Jominy test, which is applied when workpieces are quenched in liquid quenchants.

The main issue of this investigation is to develop a 2-D computer programme for prediction of hardness distribution on the axial section of axially-symmetrical workpieces of any complex shape, thereupon high pressure gas quenching, which is based on comparison between the measured heat extraction dynamics on test specimen and the corresponding one at the workpieces quenched in vacuum furnace in real condition.

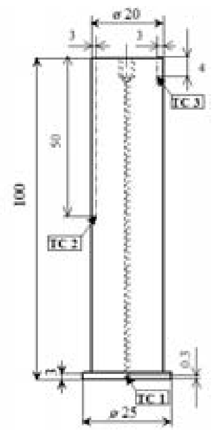

Figure 1: Modified Lohrmann's test specimen

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Presentation type: Oral

2 DESCRIPTION OF MEASUREMENT SYSTEM

In this investigation hardenability of steels, for different parameters of the high pressure gas quenching was measured by a modified test specimen, according to Lohrmann [4], of 20 mm diameter and 100 mm length, with three thermocouples placed at: the middle of the quenched end (0.3 mm below surface), 50 mm from the quenched end (3 mm from the surface) and at the top of the probe (3 mm from the surface) – **Figure 1**.
Like with the Jominy test specimen, the heat conduction axially through the quenched end is taken as boundary condition for calculation of cooling curves and the relevant cooling times 800-500 °C at different distances from the quenched end. The hardness after quenching at these distances is measured in the same way as with the Jominy specimens.

For quenching the described specimens under different cooling parameters (pressure and gas velocities) a unique facility at IWT was used [2] (**Figure 2**).

The specimens are heated to the austenitizing temperature by induction. Pressure in the vacuum chamber of the facility can be varied up to 20 bars with varying gas velocities.

Two steel grades (one structural steel EN-42CrMo4, and one tool steel EN-90MnCrV8) have been chosen for this investigation. Nitrogen was used as cooling gas under pressure between 3 and 18 bar and gas velocity between 10 and 70 m/s respectively.

From the other side batches of workpieces are quenched in a vacuum furnace under the same cooling parameters as the specimens, and the heat transfer coefficient is established using the proprietary Ipsen-Flux-sensor [3].

Using the obtained heat transfer coefficient cooling curves and cooling times 800-500 °C are calculated for every arbitrary point on the axial section of the workpiece.

3 CONCEPT FOR PREDICTION OF HARDNESS DISTRIBUTION

The principle of hardness prediction is based on the old known rule: "If during quenching two bodies at certain points have equal cooling times 800-500 °C (i.e. equal average cooling rates in this region exist), equal microstructure and equal hardness can be expected at these points". Because equal cooling parameters are used for quenching the specimens and the bach of workpieces, the cooling times 800-500 °C can be mutually correlated. This enables to read the hardness at relevant cooling times 800-500 °C in different distances from the quenched end of the specimen and transfer it to the corresponding point with the same value of the cooling time 800-500 °C at the axial section of the workpiece. In this way the 2-D computer programme enables to obtain the predicted hardness distribution within the whole axial section of any axially symmetric shape.

4 CONCLUSION

Four combinations of minimum and maximum cooling parameters have been chosen for preliminary investigation: pressure of 3 and 18 bar and gas velocity of 10 and 70 m/s. Two steel specimens from each steel grade (42CrMo4 and 90MnCrV8) for each given combination of pressure and gas velocity have been quenched and the cooling curves at three locations in the specimen (see Figure 1) have been recorded.

The average values of recorded cooling times from 800 to 500 °C at 0.3 mm below the surface of the specimen's lower end (Table 1) show the expected significant differences between the milder and more severe quenching conditions.

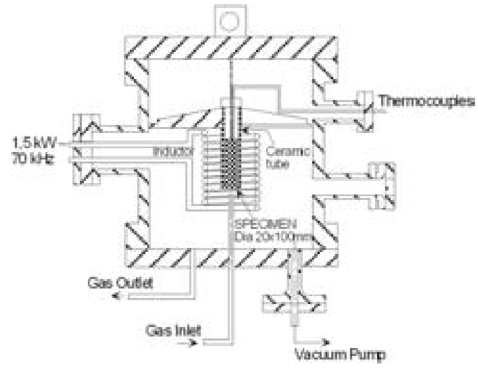

Figure2: Facility for quenching the specimens (proprietary IWT) [2]

Table 1: The average values of recorded cooling times from 800 to 500 °C at 0.3 mm below the surface of the probe's lower end

Steel type	cooling times 800-500 °C, s			
	3 bar and 10 m/s	3 bar and 70 m/s	18 bar and 10 m/s	18 bar and 70 m/s
42CrMo4	136	-	34	8
90MnCrV8	128	31	53	9

Recording of quenching intensity within a batch of real workpieces under different quenching parameters in a vacuum furnace will be the next step of described investigation.

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2nd INTERNATIONAL CONFERENCE ON HEAT TREATMENT AND SURFACE ENGINEERING OF TOOLS AND DIES
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UTILIZATION OF THE KUYUCAK METHOD TO SIMULATE LABORATORY AND COMMERCIAL QUENCHING PROCESSES

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ABSTRACT

A report was recently published describing the use of an alternative to the commonly used thermocouple-probe assemblies for gathering time-temperature data to simulate microstructure, hardness and residual stresses generated in large castings of crack-sensitive steel alloys. This process involves the measurement of the increase of the water temperature in the quenching tank as a function of time as if the quench tank were a macro-calorimeter. From this data, cooling curves may be calculated which are then used to predict microstructure and hardness. However, no details of the actual modeling process used in that work have been published to date. This paper describes the results of a laboratory study which was recently performed using a round bars of different steel alloys to evaluate and confirm the feasibility of using water temperature increase during a quenching to generate a cooling curve for property prediction by simulation in a manner similar to that reported earlier. In addition to this work, computational simulation of the data obtained from this study to examine residual stress and as-quenched hardness is also discussed. In addition to reviewing the numerical modeling process involved in these simulations, the potential applicability of this approach relative to currently utilized modeling processes to simulate quenching processes conducted in real time in actual commercial heat treating installations will also be discussed in detail.

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COMPUTER SIMULATION OF MECHANICAL PROPERTIES OF STEEL DIES

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ABSTRACT

The algorithm of estimation of yield strength and fracture toughness on the base of steel hardness HV has been made. The hardness distribution in quenched die was estimated based on relevant cooling time from 800 °C to 500 °C, as well as on results of modified Jominy-test, which is designed for hardenability prediction of high-hardenability steels. The computer simulation of mechanical properties was made for steel 60WCrV7 (DIN).

Keywords: computer simulation, mechanical properties, quenching, steel dies, modified Jominy-test

1. INTRODUCTION

Each type of engineering component failures is related with characteristic mechanical property, and in designing of engineering components it is necessary to understand types of failure. To understand lot of mechanical metallurgy problems it is necessary to understand theories of elasticity and plasticity and strength of materials. Generally, engineering components can fail due to fracture, or due to excessive deformation. Yield and crack resistance are fundamental properties, which are used in design of safety engineering components applications.
The algorithm of estimation of yield strength, R_e, and fracture toughness, K_{Ic}, on the base of steel hardness HV will be presented in this paper.

2 ESTIMATION OF MECHANICAL PROPERTIES

Mechanical properties of quenched steel directly depend on the degree of quenched steel hardening. Oriented values of investigated mechanical properties are estimated by following algorithm [1, 2].

$$R_e = 2.3\ HV,\ \mathrm{Nmm^{-2}} \tag{1}$$

$$R_m = \frac{R_e - 170C + 200}{0.8 + 0.1C},\ \mathrm{Nmm^{-2}} \tag{2}$$

$$\varepsilon_f = ln\left(1 - \frac{Z}{100}\right)^{-1} \tag{3}$$

$$Z = 96 - (0.062 - 0.029C)R_m,\ \% \tag{4}$$

$$\frac{R_m}{R_e} = \left(\frac{n}{0.002e}\right)^n \approx 0 \tag{5}$$

$$K_{IC} = \sqrt{\frac{e_f n^2 E R_e}{60}},\ \mathrm{MPam^{1/2}} \tag{6}$$

R_m is the tensile strength, C is the ratio between actual hardness and martensite hardness in HRC, ε_f is the true fracture strain, n is the strain-hardening exponent, E is the modulus of elasticity in $\mathrm{Nmm^{-2}}$, and Z is the reduction of area.

3 APPLICATION AND RESULTS

The presented algorithm is applied in computer simulation of the mechanical properties of quenched die made of steel 60WCrV7. Geometry of quenched die is shown in **Figure 1**. One of the most important factors for efficient simulation of hardening is the proper selection of representative of the cooling phenomena that is relevant for microstructure transformation. It was implied that the cooling time from 800 °C to 500 °C, time $t_{8/5}$, is relevant for microstructure transformation of high-hardenability steel 60WCrV7 (**Table 1**).
To achieve the times $t_{8/5}$ between 200 and 1000 s, the modified Jominy-specimen (JM®-specimen) was designed for high-hardenability steels, i.e. steels for tools and dies [3]. The hardness at grid points is estimated by

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Presentation type: Oral

Table 1: Chemical composition of steel 60WCrV7 (DIN)

Chemical composition [wt. %]	C	Si	Mn	P	S	Cr	Mo	Ni	V	W
	0.55	0.94	0.34	0.015	0.012	1.27	0.05	0.12	0.18	2.10

Table 2: Predicted mechanical properties of quenched die

Field in Figure 2	**A**	**B**	**C**	**D**	**E**	**F**	**G**	**H**
Hardness HV	820-774	773-744	743-714	713-684	683-654	653-624	623-594	593-564
Yield strength R_e, Nmm^{-2}	1886-1780	1778-1711	1709-1642	1640-1573	1571-1504	1502-1435	1433-1366	1364-1297
Fracture toughness K_{Ic}, MPam$^{1/2}$	72-76	76-84	84	84-88	88-91	91	91-95	95-100

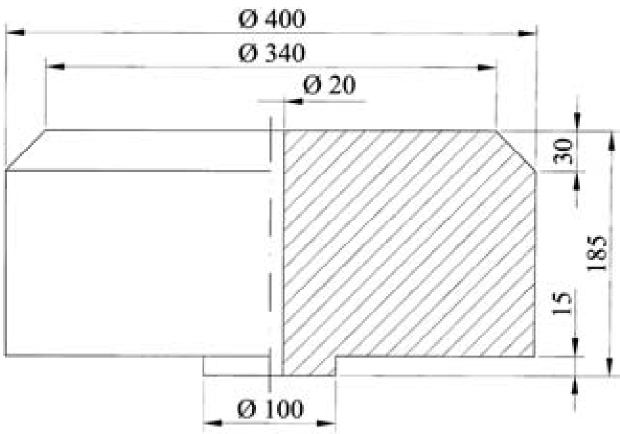

Figure 1: Die

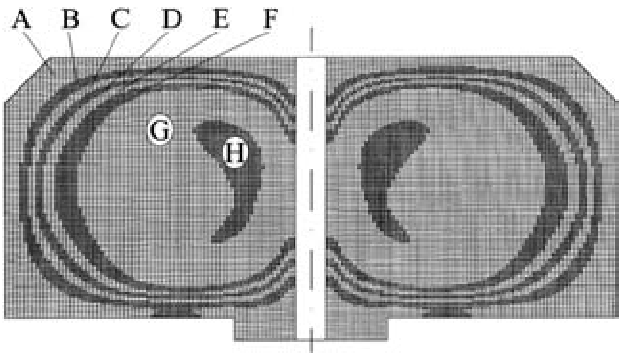

Figure 2: Distribution of mechanical properties fields

the conversion of time $t_{8/5}$ results to hardness by using both, the correlation between time $t_{8/5}$ and JM® distance from the quenched conical tip, as well as the Jominy hardenability curve.

After heating to 950°C for 2 hours, die was quenched in agitated oil with the severity of quenching $H = 0.45$. Distribution of mechanical properties fields is shown in **Figure 2**. The predicted values of mechanical properties are given in **Table 2**.

4 CONCLUSIONS

Yield and crack resistance are fundamental properties, which are used in design of safety engineering components applications. The algorithm of estimation of yield strength and fracture toughness on the base of steel hardness HV has been made. The hardness distribution in quenched die was estimated based on relevant cooling time from 800 °C to 500 °C, as well as on results of modified Jominy-test, which is designed for hardenability prediction of high-hardenability steels. The computer simulation of mechanical properties was made for steel 60WCrV7. It can be concluded that by proposed method mechanical properties of quenched steel dies can be successfully calculated.

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RAPID PROTOTYPING OF TOOLS AND DIES

2nd INTERNATIONAL CONFERENCE ON HEAT TREATMENT AND SURFACE ENGINEERING OF TOOLS AND DIES
Bled, 25–28 May 2008

POTENTIALS OF LENS TECHNOLOGY

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ABSTRACT

This paper has a threefold purpose. The first part deals with introduction of innovative high technology LENS (Laser Engineered Net Shaping) that offers breakthrough in manufacturing, maintaining and repairing products. The second part deals with a group of companies that are currently acquiring LENS technology and focuses on organisational issues needed for conquering the technology. The final part presents a comparison of a product manufactured by conventional technologies and the same product manufactured by LENS technology. The product is a medical implant from titanium alloy.

Keywords: LENS technology, laser, industrial cluster, medical implant, titanium alloy

1 INTRODUCTION

At least three conditions have to be fulfilled to manufacture a product with a high added value. We need a sophisticated technology that offers possibilities to manufacture the most demanding products from different materials, we need organisational capabilities to conquer and use the technology and we need business ideas to apply the technology in areas that contribute the most to prosperity of firms and whole regions. This paper will address all three aspects with a focus on introduction of LENS technology and its applications.

Layer manufacturing, also called rapid prototyping, techniques have about 20 years of history. These techniques directly fabricate, layer-by-layer, physical models from 3-D solid models produced in CAD [1]. One of most promising one is LENS technology that is used to fabricate fully dense metal parts directly from computer-aided design (CAD) solid models. The process is similar to traditional laser-initiated rapid prototyping technologies such as stereolithography and selective laser sintering in that layer additive techniques are used to fabricate physical parts directly from CAD data. By using the coordinated delivery of metal particles into a focused laser beam, a part is generated. The laser beam creates a molten pool of metal on a substrate into which powder is injected. Concurrently, the substrate on which the deposition is occurring is moved under the beam/powder interaction zone to fabricate the desired cross-sectional geometry. Consecutive layers are additively deposited, thereby producing a three-dimensional part [2,3,4]. This process exhibits enormous potential to revolutionize the way in which metal parts, such as complex prototypes, tooling, and small-lot production parts, are produced. The result is a complex, fully dense, near-net-shape part. Parts can be fabricated from stainless steel, nickel-based alloys, titanium etc.

LENS is a technology that is gaining in importance and is in early stages of commercialization. Its strength lies in the ability to fabricate fully-dense metal parts with good metallurgical properties at reasonable speeds. A lot of research is still being done in USA laboratories. There are only three installations of LENS in Europe: UK, France and Slovenia – a consortium of companies within Toolmaker cluster of Slovenia. The full paper will present potential and use of LENS technology in tool-making industry and for medical purposes in Slovenia.

2 ORGANISATIONAL ISSUES IN CONQUERING LENS TECHNOLOGY

The second part of the paper investigates the collective processes of acquiring and implementing advanced technology in a network of medium size firms that constitute

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the Slovenian Toolmakers Cluster. These firms lack the necessary knowledge for a smooth adoption of an advanced technology and have limited experience in investing in technologies that promise radical changes in technological capability. This paper identifies those capabilities that are needed for such a network of medium size companies to successfully acquire and implement advanced technology. This part of the paper also discusses the possible relevance of clustering for dealing with emergent technologies and increasing competitiveness within medium sized firms. We will describe the chronology and complexity of acquiring technology by a network of firms requiring new knowledge, new capabilities and high financial investment. We will also highlights what is a somewhat thorny balancing between collaboration and competition amongst the involved firms as they seek to conquer emerging technology and develop a new business model relevant to their sometimes diverging aspirations.

3 EXPERIMENT WITH MEDICAL Ti-IMPLANT

The biggest focus is on the last part of the paper where we will present the use of LENS technology for tool-making and medical purposes and present its advantages over conventional processing technologies such as turning, milling and drilling. Modern medical implants are products with pretentious requirements regarding materials, machining technologies and their functionality. They are regulated and classified in order to ensure safety and effectiveness to the patient. One of the most favourite biomaterial used for biomedical applications is titanium alloy Ti6Al4V due to its combination of the most desirable characteristics including immunity to corrosion, biocompatibility, shear strength, density and osteointegration. The excellent chemical and corrosion resistance of titanium is to a large extent due to the chemical stability of its solid oxide surface layer to a depth of 10 nm [5]. Under »in vivo« conditions the titanium oxide (TiO_2) is the only stable reaction product whose surface acts as catalyst for a number of chemical reactions.

In this paper an example of machining an Intramedullary nail (IM) for fixation of the head (caput radii) of the radius bone in the elbow joint with conventional and LENS technology is going to be presented. Material used for machining of IM nail is titanium alloy Ti6Al4V in classical form (bars) for conventional processing technologies and in powder form (45 μm grain size) for LENS technology. With conventional machining technologies (turning, milling and drilling) we get full nail form with two lines of fixation screw holes on the head of the nail and two stabilizing screw holes in lower part of the nail (**Figure 1**).

Figure 1: IM nail form produced with conventional machining techniques

Nail produced with LENS technology is hollow, thin walled with two stabilizing screw holes in the lower part. Fixation of the fragments of the caput radii is done with drilling screw holes during surgical operation to the head of the nail coincidentally in the best possible way to gain primary stabilization of the fracture. This is possible because of the thin walls of the nail. IM nail prototypes produced with both technologies (conventional and LENS technology) will be tested with computer simulations (Finite Element Analysis-FEM) and mechanical tests on human cadaver parts. The results of the biofunctionality tests will be compared and conclusions regarding both technologies will be given.

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MICROSTRUCTURE AND MECHANICAL CHARACTERISTICS OF DMLS TOOL-INSERTS

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ABSTRACT

Direct metal laser sintering (DMLS) of metal powders is already a well-known and established process for fast and effective manufacture of prototypes and tool-inserts in smaller series. The final goal is to achieve the quality of DMLS products enabling production of larger series of injection moulded polymer-based products or even to produce those which can sustain more rigorous conditions during pressure die casting of Al-based or other non-ferrous alloys. Therefore, permanent improvement of equipment, process and metal powders for DMLS is necessary. A commercial metal powder mixture was used for the laser sintering experiments. Different substrates in the form of simple blocks and complex testing tool-inserts for pressure die casting of Al-based alloy were prepared at different process conditions and post-surface treatments. Additional improvement of surface quality was obtained with the hard coating.

In the present contribution the basic principles of laser sintering are presented. Morphological and microstructure properties of metal powders and sintered products are also discussed. The results show that relatively porous and heterogeneous microstructures are obtained during laser sintering. However, improved properties of the laser-sintered products can be obtained by careful selection and control of powder properties, optimisation of DMLS conditions and by the hard coating of the sintered product.

Keywords: direct metal laser sintering, microstructure, mechanical properties, tool-inserts

1 INTRODUCTION

A stereo-lithography and laser sintering process are two main rapid prototyping processes which are commercially used from the beginning of the nineties. The laser sintering based process is more appropriate for the manufacture of larger and complex parts because they are built into and supported by the powder itself. A lot of problems existed in the beginning with the sufficient dimensional accuracy and mechanical strength of sintered parts. However, the quality of sintered products significantly increased in the last ten years with the unsurpassed development of laser technology and systems, computer hardware and software, as well as powder materials for laser sintering[1]. Nowadays, laser beam based processes are certainly the most advanced processes for rapid prototyping, rapid tooling, rapid machining/cutting, rapid manufacturing, laser welding, as well as alloying and microstructure refinement.

DMLS has been developed by the Electrical Optic System (EOS)[2]. Three different systems and machines are used for the manufacture of: (a) models and prototypes from thermoplasts, (b) tool-inserts for injection moulding or die casting and parts from metal powders and (c) for box-less production of moulds and cores from foundry sand for metal casting. In the case (b), the income of heat of laser beam is high and sufficient to stick the metal powder particles together, layer by layer with the thickness of approximately 20 to 50 μm directly by their partial melting and sintering. After each pass over the surface layer the laser beam creates a two-dimensional shape of the desired product. With the movement of the powder container and additional supplement of the next layer of powder a three-dimensional form of the product is produced. The advantage of DMLS is fully automatic, PC controlled process with the direct use of models, made in any of commercially applicable software. This enables a rapid and direct manufacturing of very complex near-net shapes from metals and their al-

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Presentation type: Oral

loys with very good dimensional stability and small shrinkage because no polymer binder and additional thermal post processing at high temperature. Additional advantage of the process is that the structure of sintered product can be controlled and changed over its whole cross section. Disadvantage of the process are the limitations relating to metal alloy compositions because the metal powder mixture has to be prepared from the constituents which form low-melting-point phases enabling fast liquid-phase sintering process. Therefore, sintered products have characteristic cast, porous structure resulting worse mechanical properties, which can be improved to certain extent by the additional surface treatment like shot-pinning, polishing and hard facing.

In Slovenia tooling for PIM and PDC of non-ferrous alloys has a long tradition and is well developed. The equipment (EOSINT M 250X) for DMLS, purchased and installed at regional R&D Centre (RTCZ) Hrastnik, has enabled Slovenia to make also a breakthrough in the field of rapid prototyping and manufacture of DMLS prototypes and tools[3]. This demands good knowledge of metal powders, of the possible mechanisms of their sintering and the characteristics of the sintered product.

2 EXPERIMENTAL

2.1 Characteristics of DMLS powders

In the first step of our investigations complete chemical and microstructure investigations of commercial powders for DMLS were performed. Commercial DMLS powders are mainly prepared on the basis of two different chemical compositions. The first one is Cu-Ni based powder with additions of Sn and P. The second one is Fe-Ni-Cu based powder with addition of P. The chemical compositions are carefully selected in the way that the additions of Sn, P and Cu act as liquid phase sintering promoters, as well as alloying (hardening) elements. Metal powders for DMLS must have a narrow particle size distribution with as possible small average particle size and regular particle shape. Such powders have small interparticle friction and good flowability, as well as large apparent and tap density. This enables undisturbed powder transport and its good deposition in the form of thin layers during the DMLS process. Therefore, the control of engineering properties of DMLS powders is also very important. Average particle size of both powders is approximately 30 μm and particle size range is between 2 and 200 μm. Morphological and microstructure characteristics of individual particles of investigated powders were analysed with the light and scanning electron microscope. Cu-Ni based powder is a powder mixture consisted from relatively regular particles of at least three different materials. However, Fe-Ni-based powder is a powder mixture consisted from much more irregular particles.

2.2 Sintering experiments

The investigated powders were laser sintered with new DMLS equipment into a simple form of blocks, as well as into a complex testing tool-inserts[4, 5] for pressure die casting of Al-based alloy (AlSi9Cu3) at different process conditions and post-surface treatments (**table 1**). The latter have to be performed in order to achieve adequate surface roughness and improvement of mechanical properties.

Post-processing include shot pinning with steel shots and fine ceramics for increasing surface hardness, smoothness of sharp corners and stress relaxation. Manual grinding and polishing can also be performed for the improvement of surface quality with special attention to vertical walls and slots; and consequently second shot pinning with fine ceramics to induce compressive stress to the surface and to improve the behaviour of the lubricant film. Additional improvement of surface quality is obtained with hard coating. In our case two different coatings were used. First thicker layer is Cu-P based hard coating and second thinner one is PVD coating (TiN for PIM or CrN for PDC)[6]. Microstructural investigations of tool-inserts have shown that in the case of inadequate (too fast) sintering conditions rough and for open porosity characteristic surface is formed. Therefore, special so called up-skin sintering procedure is performed. It enables to obtain almost full dense, pore-free and relatively smooth surface of sintered product and DMLS parts, respectively.

Sintered tools were then tested under industrial conditions of pressure die casting (molten metal temperature 690 °C, pressure 780 bars and entrance velocity 50m/s). Visual control of tool-inserts has been performed after every five to ten shots if cracks are already appeared in

Table 1: The characteristics of testing tool-inserts for pressure die casting of Al-based alloy[5]

Sample designation	DMLS inserts (S I)	Finish machining (S II)	Coatings (S III)	Up-skin (S IV)
Post-processing	SPSH*	SPSH + P	SPSH + P	SPSH + P + C
Coating	no	no	10μm Ni+ 3.5μm CrN	no
Surface roughness Ra (μm)	3	0.2	1	0.1
Surface hardness HV_{20}	270	230	1300-2400	380
Number of parts without cracks	50	60	210	150
Number of parts with first crack	100	150	250	210

* SPSH … shot pinning (steel shots), P … polishing, C … shot pinning (ceramics)

the most critical regions. Microstructure and microchemical investigations of exploited tool inserts were also performed[4,5].

3 DISCUSSION AND ANALYSIS OF SINTERING EXPERIMENTS

Sintering of product usually starts on the basic steel plate where the first layer of powder is melted by laser beam. The powder type is selected on the basis of sintered product. Highly mechanically and thermally loaded tool-inserts for pressure die casting demand sintered product of the best quality with the lowest possible porosity, the largest hardness and strength, which guarantee the highest tools' wear resistance. However, it distinctively influences the costs of DMLS process and sintered part, respectively. The sintering time is defined by the size of the part and by the sintering speeds (between 2 and 15 mm^3/s). The latter depends on type of powder, as well as desired quality (porosity, hardness) and shape complexity of the sintered product. High-pressure die-casting toolmakers urgently require solutions that will give a faster return on a tool. This means that production of at least 1.000 castings is required for short run tooling and only 100 castings for technical prototypes. Therefore, our goal was to obtain at least 1.000 good cast parts before tool starts to crack. However, as it is shown in **Table 1**, we did not reach more than 250 good parts in spite of the additional post-surface treatment and improved surface characteristics. The results of our investigations show that deposited hard nickel coating improves the surface roughness of the tool, as well as fills most cavities (pores) and cracks of sintered material. With additional deposition of hard PVD CrN layer thermal resistance and high surface hardness is achieved. However, the performed coatings did not give expected results and significantly improved tool life. With the application of melted up-skin layer (two melted layers of thickness approx. 0.94 mm, last very thin layer performed with the laser speed of 50 mm/s) almost the same characteristics as with the coated one were achieved.

4 CONCLUSIONS

The investigated powders were direct laser sintered with EOSINT M 250X equipment into a simple form of blocks, as well as into complex testing tool-inserts for pressure die casting of Al-based alloy. The experiments have shown that cast, relatively porous and heterogeneous microstructures are formed. The tool-insert life improvement is obtained with the optimization of process parameters and new approach forming of remelted final up-skin layer. Additional improvement of wear resistance and thermal stability of the sintered product was obtained by the appropriate surface finishing and hard coating.
Our experiments shows that the obtainable real life of laser sintered tool-inserts for pressure die casting of Al and Mg-based alloys is below 1.000 parts. The development of still finer metal powders and new chemical compositions, as well as applications of new multilayer-based hard coatings enable prospects for further improvement of their wear and temperature resistance.

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APPLICATIONS AND MATERIAL SELECTION FOR TOOLS AND DIES

IMPORTANCE OF SELECTION OF THE TOOL STEEL GRADES AND PVD COATINGS IN COLD WORK TOOLS

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ABSTRACT

Tool steel selection and selection of PVD coatings is of key importance for the tool longevity. Therefore it is very important to recognize all present wear mechanisms in tool operation and select the most influential one, which enables proper selection of the tool steel types for the active tool positions. The case study of certain tool steel selections has been provided, along with presentation of the selection of PVD coatings in certain cases. The feedback received from exploitation of tools is included as well.

Keywords: tool steel, PVD coating, abrasion, adhesion

1 INTRODUCTION

Numerous guidelines related to tool steel selection are available, in different forms and from different sources. Each steel manufacturer specifies suitability of certain steel types for application in particular tools. Based on it, steel selection seems to be quite linear. However, it is to emphasize that wear mechanisms in cold work tools must be properly recognized. Adhesion and abrasion are the two main wear mechanisms which determine choice of tool steel and suitable surface modification treatment [1, 2]. This is of utmost importance and the decisions related to steel selection which are made in design phase of the cold work tool will be decisive for the tool life. There are many known examples demonstrating that the improper steel selection can reduce the tool life or dramatically increase the tool maintenance costs, which both increase the product cost.

2 CASE STUDIES

The example for reducing the maintenance costs further to proper selection of the substrate steel and PVD coating is a tool for Baking Tray, **Figure 1**. The tool performance at 3 000 pieces before regrinding was needed, when the uncoated D2 steel was used. Change of the substrate steel to Sleipner increased the performance to 4 500 pieces, which was still unsatisfactory. For this tool, the most significant improvement was obtained by PVD coating, which prolonged the tool performance to 30 000 pieces before regrinding. It has dramatically influenced the maintenance costs – it was calculated that the saving was 27.000 EURO/year.

The another example refers to the tool for VTC Frame. This part is a cosmetic part, which is well visible to the customer buying the cooker. Therefore, its shape must be perfect, the material must be formed that way that the surface is perfectly smooth. No wrinkles, buckling and similar imperfections are allowed, which means that the maximal forming pressure must be applied in order to get the smoothest possible outer surface. The parts from two steel grades were produced in this tool, from carbon steel and stainless steel. However, stainless steel was covered with the protective foil, therefore its adhesive nature was much diminished by it. In this case, sticking of the steel sheet to the tool steel substrate surface was expected, particularly in the corners where forming is

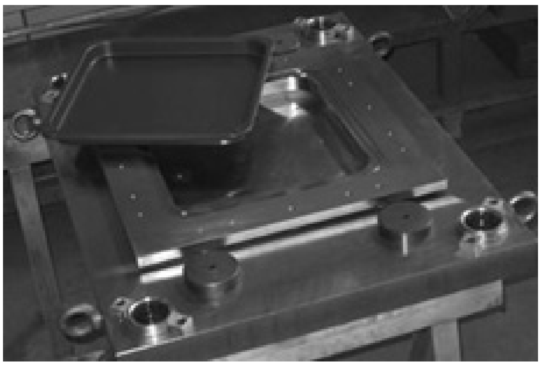

Figure 1: Tool for Baking Tray

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Figure 2: Uncoated t ool corner area after 100 pieces

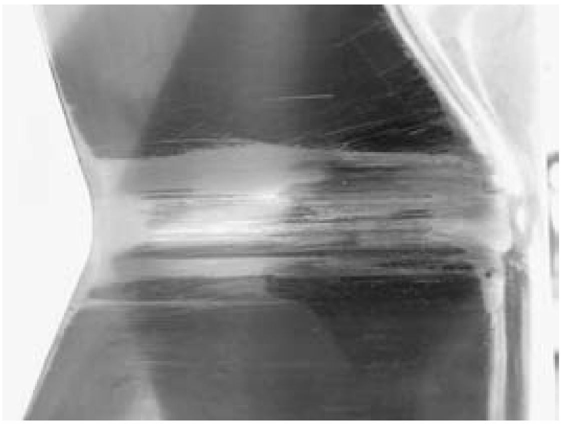

Figure 3: TiCN coated tool corner area after 53.000 pieces

most severe. The first step done in tool steel selection was application of the Sleipner steel grade for the forming corners, as this steel would bring more resistance towards the steel build-ups.

After production of as few as 100 pieces, the surface was affected, **Figure 2**. There were significant build-ups, it was to expect that this tool should be repolished each 200 pieces. For mass production, this frequently maintenance is absolutely unacceptable, as it would dramatically increase the production cost. The solution guaranteeing a continuous performance of this tool and production of large quantities was required. It was decided to coat the corner area with TiCN PVD coating.

This coating has significantly contributed to tool performance and enabled tool functioning for thousands of cycles. After 53 000 parts, the tool corner is still in acceptable condition and is further functioning quite well, without any need for maintenance or recoating, **Figure 3**.

Although this solution was already quite good and economical, it was considered how to further improve the tool operation and simplify tool maintenance. Namely, PVD coating required recoating with time, whereby the costs are generated. The tool steel type used for production of new corners, possessing high resistance towards adhesive loading is a new steel grade Vancron 40 by Uddeholm, whose specific advantage is that no coating is needed. The result obtained did not exceed performance of the uncoated Sleipner steel. However, this is quite easy to understand. Although sticking of the material to the tool insert surface in the corner was observed, the wear mechanism was not adhesion. Due to extremely high contact pressures that arise due to the massive forming forces needed to produce the perfectly smooth surface, high abrasive loading was generated in the corners. These high abrasive loads damaged the surface first and then the material was building up on the steel insert surface due to the combined of the abrasive and adhesive loads.

3 CONCLUSION

In toolmaking, selection of tool steels is of extreme importance, as it directly influences lifetime and efficacy of the tools, as well as the tool maintenance costs generated during production. For tool steel selection, thorough knowledge about wear mechanisms is essential. The dominant wear mechanism must be properly defined, as it directly affects steel selection. Nowadays, the steels are foreseen and produced for ever more specific purposes, therefore any mistake in defining the wear mechanism can be very misleading – if using the steel which is not proper for the subject purpose, although it is basically the top quality steel, it will not fulfil the demands and the tool will not operate as expected and required.

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2nd INTERNATIONAL CONFERENCE ON HEAT TREATMENT AND SURFACE ENGINEERING OF TOOLS AND DIES
Bled, 25–28 May 2008

EXPERIMENTAL ANALYSES OF THE INFLUENCE ON CORROSIVE ENVIRONMENT IN SEAWATER OF VLORE BAY TO THE CENTRE ON FATIGUE OF STEEL A -3

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ABSTRACT

This article is about the role that corrosive environment plays to the centre on fatigue at steel used in outward covering of means of naval transport. By the complex study of these phenomena is lowered the fatigue resistance as a result of temperature, pressure, waving, salting and maneuverable factors during the navigation and floating of means of naval transport at different time periods.

The specification of factors, the direct study of the role and the influence of environmental factors on the fatigue resistance are specified and concreted through the fulfillments of experiments in the seawater of Vlore Bay known for its specific characteristics.

Keywords: fatigue, fatigue test, steel, low cycle fatigue, stress ratio, mean stress, amplitude stress, stress minimum, stress maximum.

1 INTRODUCTION

The problem of centre on fatigue of metal materials and the influence of different technologic and constructive factors (microstructure, inferior tensions, superficial treatments, geometric shapes et-c) as well as the factors of surrounding environment (temperature and corrosive environment) consist one of the most important issues of the fatigue theory of materials in the constructions field and in the choice of materials

The presence of elements or structures in aggressive corrosive environments is accompanied by a sensitive low on fatigue resistance of metal materials and their links. While the combinations of the simultaneous acting of dynamic loadings as well as the corrosive influence of aggressive environment are accompanied by a more emphatic low on their fatigue resistance in comparison with damaged effects as separated factors.

The corrosive fatigue comes from the joint acting and as a result of the most destroying mechanism of damage of pure mechanic fatigue with the further implementation of the right corrosion .The continuous exposure of un protective surfaces to the corrosion is helped by micro and macro deformations and the displacements for effect of cyclic loading. These factors hasten the rising of micro fractures and their quick advance toward fatal fractures.

2 THE METHOD OF THE APPROACH

To know the influence of the reduction of the fatigue resistance by corrosive fatigue, there have been constructed samples from the metallic structures of the sea means in the level of the constructive water line, there where it is supposed to be maximal action of the dynamic effects of the load from the weavings and with different degree of corrosion.

The samples being prepared according to the demand of the Italian standard are the shape of slabs of dimensions h x b x t; (150mm x 30mm) with two thickenings of 8 and 10mm The features research of the corrosive fatigue resistance has been performed in the machinery for tests in conventional fatigue, type: "Schneck", organized in four groups A, B, C, D which divide the samples different thickening (The experiments have been executed in the Department of the Design and Industrial Products, Polytechnic, Bari, Italy.)

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The effect of loading because of the weavings has been brought closer to the dynamic nature of the sinusoidal cycles with its characteristics of minimal load against the maximal relativity low equally (R= 0,1) (The samples are steel A-3, material which is used for structures of the sea floating means.

For the tests there have been combined also selections of the differentiation levels of the loading providing sufficient aperture of the density level, accompanied with figures of cycles from breaking the samples which have enough variation. Therefore, for about 50 samples, for amplitude tension, calibers 64 – 180 N/mm^2, have been defined the destructive cycles. Also the levels of the functional frequencies in tests which change from 20 to 29 Hers exclude their influence in the deformation of the results.

3 THE TESTS RESULTS

Table 1: Summary of tests results

The thickens 10 mm						The thickens 8 mm					
The sample different thickening from corrosion 7-13%	Group A		The sample different thickening from corrosion 6-14%	Group B		The sample different thickening from corrosion 6-16%	Group C		The sample different thickening from corrosion 3-14%	Group D	
	Alternating stress σ_a (N/mm^2)	Number of cycles N		Alternating stress σ_a (N/mm^2)	Number of cycles N		Alternating stress σ_a (N/mm^2)	Number of cycles N		Alternating stress σ_a (N/mm^2)	Number of cycles N
	160,5	85676		175,5	15 878		142,8	10514		159,6	17266
	142,6	236074		159,6	62 287		94,8	133647		110,5	266518
	128,4	354282		141,3	123753		117,4	65478		88,0	405239
	117,1	462777		130,99	242 960		120,5	45473		83,3	542622
	95,3	1443908		108,3	486 992		83,3	195054		64,1	2189125
	78,9	2540973		96,5	768923		107,1	127937		95,8	525129
	182,8	21752		78,9	1189855		66,2	1021629		135,4	47172

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2nd INTERNATIONAL CONFERENCE ON HEAT TREATMENT AND SURFACE ENGINEERING OF TOOLS AND DIES
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SELECTION OF TOOL MATERIALS FOR COLD FORMING OPERATIONS USING A COMPUTERIZED DECISION SUPPORT SYSTEM

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ABSTRACT

A decision support system (DSS) is outlined which is designated primarily to select the proper tool steel grades for various cold forming operations (sheet metal and bulk material forming). This computerized system contains four data base: data base of cold forming operations and tools, of work materials, of cold forming tool materials and of wear resistant PVD coatings. The decision strategy (algorithms) makes it possible to select appropriate tool materials taking into consideration their most important properties (wear resistance, toughness, compressive yield strength, tempering resistance, dimensional stability, hardenability, machinability (grindability) and material price. The practical advantages of using the developed decision support system is demonstrated on examples of applications.

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2nd INTERNATIONAL CONFERENCE ON HEAT TREATMENT AND SURFACE ENGINEERING OF TOOLS AND DIES
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AN ANALYSIS OF RELATIONSHIPS BETWEEN BEHAVIOUR AND MICROSTRUCTURE CONSTITUTION OF HOT-WORK TOOL STEEL

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ABSTRACT

Influence of microstructure on mechanical behaviour of hot-work steels is analysed on sub-micro, micro and macro scale. Methods of determination of working stress of hot work-steels have been presented. In optimisation and selection of hot-work tool steel, proper failure criteria have to be recognized based on possible specific failure micromechanism of tool or dies in application. Moreover, the specific failure micromechanisms have to be modelled and numerically defined in terms of time and temperature. Except suitable phase distribution and microstructure refinement, proper alloy chemistry and melting practice, are so important in hot-work steel optimisation.

Keywords: hot-work steel, mechanical behaviour, working stress, plastic deformation, fracture

1 INTRODUCTION

When mechanical behaviour is understood in terms of metallurgical structure, it is generally possible to improve the mechanical properties of metals or at least to control them. Hot-work steels are medium to high carbon steels which are quenched and tempered to obtain the desired strength, toughness and hardness. Generally, hot-work tool and die steels should have resistance to excessive deformation and distortion, shock, wear, softening, and to heat checking during application at high working temperature [1]. Very complex interactive influence of chemical composition and microstructure composition on hot-work steel properties gives opportunities to develop hot-work tool steels with more specific properties. There are three main groups of hot-work steels, chromium, molybdenum and tungsten hot-work steels. Chromium hot-work steels usually are used for forging and die casting applications. Tungsten hot-work steels are used for extrusion and press dies for higher temperature melting alloys. Molybdenum hot-work steels has stable microstructure when exposed to higher working temperature and offers an alternate choice to tungsten and chromium hot-work steels [1].

Yield and crack resistance as well as modulus of elasticity are fundamental properties, which are used in design of safety of engineering components applications. Since working parts of hot-work tools are working in relative motion between tools and working parts, hot-work steels are characterized with high resistance to wear. In addition hot-work steels have to be considered in respect of temperature and time. The microstructure and mechanical properties of hot-work steels may continuously change with time during the hot-working processes. Workpiece mechanical behaviour at elevated temperature is so important for hot-work steels selection. During the thermal processes, dies are affected bay many degradation processes, such as, creep, high-temperature fatigue and thermal fatigue. Analysis of distortion and fracture appearances due to residual stresses is important part of mechanical metallurgy of hot-work steels. In microstructure design and property prediction of hot-work tool steels precise relationships between properties, microstructure chemical compositions have to establish [2].

2 DETERMINATION OF WORKING STRESS

The mechanical behaviour of metals can be generally predicted as a function of stress-strain state, strain rate and temperature. Generally, engineering components can fail due to excessive deformation or due to fracture [3]. Since stresses and strains have to be determined to solve an elastic-plastic problem, in order to carry out such calculations in thermal processes it is necessary to know the material properties in terms of temperature. Since high

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strength materials usually have a low crack resistance, the conventional design criteria are non adequate when there cracks are exist. In this case, equations of fracture mechanics have to be used. Criteria equation for fracture was developed already in 1921 by Griffith. The catastrophic crack growth occurs when stress exceeds a certain critical value σ_c: $\sigma_c\,(\pi a)^{1/2} = (EG_{Ic})^{1/2} = (1-\nu)^{1/2}\,K_{Ic}$, where a is the crack length, G_{Ic} is the so called critical value of elastic energy release rate per crack tip, K_{Ic} is referred to as the fracture toughness, E is Young's modulus, and ν is Poisson's ratio. Since creep and fatigue effects could not be successfully predicted based on short time yield and fracture resistance, failure risk due to excessive deformation or fracture of hot-work steels has to be predicted based on log time material properties.

3 MICROSTRUCTURAL ASPECTS OF HOT-WORKING STEELS OPTIMIZATION

Hall and Petch have been established that the tensile yield stress was related to grain size by [3]:

$$\sigma_Y = \sigma_i + k_y d^{-1/2}$$

where si has the mining of yield stress of monocrystal, k_y is the constant, and d is the grain diameter. The strengthening due to grain boundaries results from mutual interference to slip within the grains. The Hall-Petch equation has been found to express the grain-size dependence of the flow stress at any plastic strain out to ductile fracture. For brittle fracture, fracture stress can be expressed by: $\sigma_f = (4\mu\gamma_s)/(k_y d^{1/2})$. In this case microcrack length is related to d. Constant μ is the shear modulus. Effective surface energy γ_s includes energy of plastic deformation in specific field scale.

General expression of microstructural aspect for nucleation and propagation of brittle crack can be written in form [4]: $(\tau_i d^{1/2} + k_y)\,k_y = \beta\mu\gamma_s$. Factor β depends on stress-strain condition and τ_i is equal to the friction stress opposing motion of dislocation. A high value of friction stress ti lowers ductility. If the left side in this equation is smaller than the right side a microcrack can be formed but it cannot grow. If the left side of the equation is greater than the right, brittle crack will propagate at stress equal to yield stress. The effective surface energy will depend on the contribution of plastic deformation which depends on number of available slip systems and the number of mobile dislocations at the tip of the crack. Strengthening mechanisms based on dislocation locking, such as, increasing dislocation density and presence of fine second phase particles result in brittleness. Increasing of vacancies concentration with temperature can result in greater mobility of dislocations and material can softening. The unique characteristic of grain refining strengthening lies in that it is the only one strengthening method that also increases the toughness. In addition to the grain size, the value of the gradient ky in the Hall-Petch relationship plays an important role in controlling the mechanical properties of steels. Since particle distance determines the slip distance, for optimum metal alloys properties interparticle spacing have to be in correlation with refinement of grain size. In steels, most solutes decrease the ductility, and interstitial solutes have commonly many more effects than substitutional solutes. Hot-work steels must have the fine grained microstructure with optimum distribution of fine carbides in a matrix of tempered martensite. Proper alloy chemistry and melting practice are so important for hot-work steels application ability. Carbon is most important element for steel strength and hardness, but carbides locations are places for cracks nucleation. Nickel lowers transition temperature and provides better toughness. Chromium is solid solution strengthener but strong carbide former. Molybdenum is solid solution strengthener, strong carbide former and suppresses temper embrittlement. Silicon increases yield strength and transition temperature. Manganese prevents hot cracking and lowers transition temperature. Cobalt increases martensite formation and precipitation hardening. Titanium is carbide and nitride former and in steels is used for precipitation hardening.

Materials optimisation and tools has to perform simultaneously, based on failure physics, mechanical and materials engineering, material science, and quantum chemistry and mechanics knowledge [2].

4 CONCLUSIONS

Safety of hot-work steel has to be designed taking into account all aspects of specific application at high temperature. During the thermal processes, tools are affected bay many degradation processes, such as, creep, high-temperature fatigue and thermal fatigue. Failures appearances analysis due to residual stresses has to be taken into account. The mechanical behaviour of hot-work steels can be improved by nearly of all known mechanisms of steel strengthening, such as, solid solution hardening strengthening by heat treatment, grain refinement, second-phase hardening and precipitation hardening by undeformable particles and deformable particles. Composition of hot-work steel has to allow long time and high temperature consistence of mechanical properties of hot-work steel. Hot-work steels must have the fine grained microstructure with optimum distribution of fine carbides in a matrix of tempered martensite.

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HEAT TREATING OF H13 DIES ACCORDING TO THE NADCA AND GM POWERTRAIN SPECIFICATION

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ABSTRACT

- Intro
  - What is “acceptable” die life?
- Get the facts
  - GM Powertrain increased their average die life from 90,000 shots to over 220,000 shots.
  - Steel & heat treatment represent about 15% of the cost of a die, but are responsible for about 50% of die failures.
  - Good steel & bad steel may machine the same and harden the same, but even at the same hardness, they don’t perform the same
  - Heat treating to meet a hardness specification while minimizing distortion is exactly the wrong way to harden a die.
- Development of H13 Specifications
  - GM Powertrain cost saving impact
  - The “Evolution” of technical requirements (NADCA / Chrysler / Ford / GM PT Specifications)
  - Die Material development
- Premium Heat Treat of H13 Dies
  - Faster Quenching
  - The GM 16” cube test
  - Surface Treatments
- Conclusion

BODYCOTE, GLOBAL LEADER IN THERMAL PROCESSING

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ABSTRACT

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