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EFFECT OF HEAT TREATMENT ON THE MICROSTRUCTURE AND PROPERTIES OF Cr-V LEDEBURITIC STEEL

VPLIV TOPLOTNE OBDELAVE NA MIKROSTRUKTURO IN LASTNOSTI LEDEBURITNEGA JEKLA Cr-V

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Duplex coating of high-alloyed tool steels is the newest and most promising technology that allows improvements to tools in terms of wear resistance and service time. However, the deposition of thin films onto the nitrided surface is associated with problems that are not yet solved. To clarify the problem of the adhesion and mechanical properties of the system substrate/coating it is necessary to prepare the substrate with well-defined microstructural parameters. The Vanadis 6 PM ledeburitic steel was chosen as a substrate for surface processing. It was austenitised, quenched and double tempered to a desired hardness of *HRC* = (57–58). Some specimens were also sub-zero treated in order to improve the transformation of austenite to martensite. The microstructure of the heat-processed steel was examined using optical microscopy and scanning electron microscopy. The resistance to nucleation of fracture was assessed with the three-point bending-strength test and the fracture surfaces were subjected to a fractographic analysis.

Keywords: Sub-zero treatment, hardness, ledeburitic tool steel

Dupleksno pokritje visokolegiranih orodnih jekel je nova in obetajoča tehnologija za povečanje obrabne obstojnosti in dobe uporabnosti orodij. Vendar je nanos tanke plasti na nitrirano površino povezana s problemi, ki še niso rešeni. Za razlago problema adhezije in mehanskih lastnosti sistema podlaga-pokritje je treba pripraviti podlago z dobro opredeljenimi parametri mikrostrukture. Jeklo Vanadis 6 PM je bilo izbrano za podlago za nadaljevanje procesiranja. Bilo je avstenitizirano, kaljeno in dvakrat popuščeno na želeno trdoto *HRC* = (57–58). Nekateri preizkušanci so bili tudi podhlajeni zaradi povečanja obsega premene avstenita v martenzit. Mikrostruktura je bila preiskana z optično in vrstično elektronsko mikroskopijo. Odpornost proti začetku razpoke je bila preverjena s tritočkovnim upogibom, površina prelomov pa fraktografsko preiskana.

Ključne besede: podhladitev, trdota, ledeburitno jeklo

1 INTRODUCTION

Generally, heat treatment improves a steel's toughness and hardness, and it is absolutely necessary for the proper functioning of tool steels. The heat treatment usually consists of austenitizing, quenching and is followed by multiple tempering. After this procedure the tool steels gain properties that are suitable for industrial applications.

In tool steels, retained austenite is an undesired component because it lowers the hardness after the heat treatment. Additionally, it can transform into martensite and/or bainite at elevated working temperatures or under high loading. This worsens the operational suitability of tools and strong efforts are made to minimize the amount of retained austenite.

With this aim, the sub-zero treatment was introduced in the 1960s. This treatment hinders the retained austenite stabilization in steels with the M_f temperature lower than room temperature. A typical example of these steels is the group of ledeburitic steels, with a higher carbon content and with a large amount of alloying elements.

Nevertheless, the opinions on the effect of the sub-zero treatment on the working utility of tools are diverse because the nature of the process has not yet been entirely clarified. In 1962 Rapatz concluded that there is no advantage in using these methods for ledeburitic high-speed steels [1]. Also, Hoyle did not recommend this type of heat treatment for ledeburitic steels [2]. On the other hand, Berns stated different recommendations for ledeburitic steels. He found that it was possible to significantly increase the hardness of chromium ledeburitic steels with an immediate sub-zero treatment after quenching [3]. He stated that the immediate sub-zero treatment after quenching from the usual temperatures and tempering allowed a hardness increase of the low-carbon-content steels. For the steel X155CrVMo 12 1, it is possible to reach *HRC* = 67 and in the case of superhard ledeburitic steel X290Cr even *HRC* = 69. Thus, it is evident that older studies focused on the research of this type of heat treatment did not produce definite results. On top of that, in the case of ledeburitic high-speed steels the results were not even optimistic. Nevertheless, in recent years the experiments engaged in sub-zero treatment have become more realistic.

The goal of this study was to investigate what happens when Cr-V ledeburitic steels are plasma nitrided. For this purpose, it was necessary to prepare a substrate with a well-defined microstructure and properties. In this paper, the Vanadis 6 steel processed using various heat treatments, including a sub-zero period, is investigated.

2 EXPERIMENTAL

2.1 Material

The samples were made from Vanadis 6 – a ledeburitic cold-work tool steel with the chemical composition of 2.1 % C, 7 % Cr, 6 % V and an initial hardness of HV_{10} = 284 Fifteen specimens for three-point bend tests were net-shape machined to dimensions of (10 × 10 × 100) mm and then submitted to the heat treatments in **Table 1**.

2.2 Methods

Several measurements and experiments for determining the material and structural properties were performed. The hardness was measured with the EMCOTEST M4C 075G3 hardness tester using the Vickers HV_{10} and Rockwell HRC methods. These two methods were selected because of the expected high material surface hardness. The resistance against crack initiation was determined with a three-point bending test. The microstructure of the steel in the as-received state and of heat treated steel were examined with optical microscopy and scanning electron microscopy, applying a Neophot 32 optical microscope and a Jeol JSM 5410 scanning electron microscope. Also, the quantitative analysis of the carbides' dissolution during the austenitizing, which consisted of determining the particle count and the size classes, was performed. The first out was executed using the micrograph of the as-received material and the second one using the micrograph after the heat treatment A. Within the fractography study, micrographs of two material states were taken: after heat-treatment A (i.e. without the sub-zero treatment) and after heat treatment C (i.e., with sub-zero treatment –196 °C; 7 h).

3 EXPERIMENTAL RESULTS

3.1 Optical and scanning microscopy

The as-received steel Vanadis 6 has a microstructure with fine carbide particles uniformly distributed throughout the ferritic matrix. These carbides are of eutectic, secondary and eutectoid origin, as shown in the micrographs in **Figure 1** and **Figure 2**.

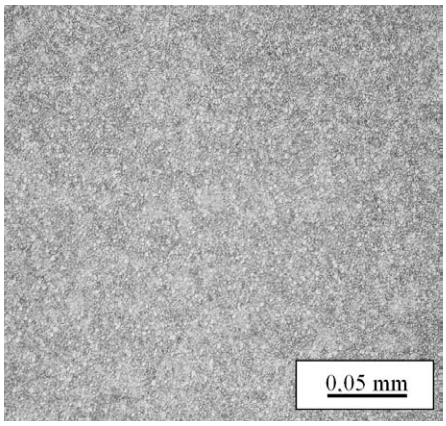

Figure 1: Microstructure of the as-received material state (optical microscopy)
Slika 1: Mikrostruktura prejetega jekla, optični posnetek

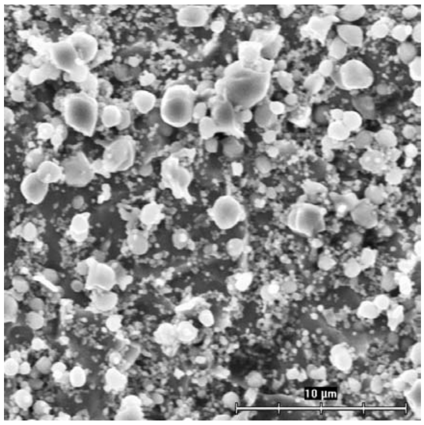

Figure 2: Microstructure of the as-received state (SEM)
Slika 2: Mikrostruktura prejetega jekla, SEM-posnetek

After the heat treatment (**Figure 3**), the matrix consists of tempered martensite and particles of the carbides M_7C_3 and MC.

The material submitted to heat treatment B, i.e., with sub-zero treatment, shows, at first glance, a similar microstructure, MC and M_7C_3 carbide particles in a martensitic matrix with particles of carbide M_7C_3 fewer and smaller than after heat-treatment A, **Figure 4**.

The results of the quantitative analysis of the carbide particles are presented in **Figure 5** (as-received) and

Table 1: Heat treatment details
Tabela 1: Podatki o toplotni obdelavi

Specimen	Heat-treatment marking	Heat-treatment process details			
		Austenitizing	Quenching $p(N_2)$/bar	Sub-zero treatment	Tempering
1 – 5	A	1030 °C; 30 min	6	–	2 × 530 °C; 1 h
6 – 10	B	1030 °C; 30 min	6	–196 °C; 4 h	2 × 530 °C; 1 h
11 – 15	C	1030 °C; 30 min	6	–196 °C; 7 h	2 × 530 °C; 1 h

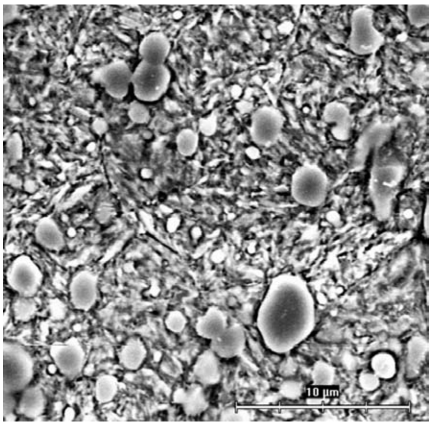

Figure 3: Microstructure of Vanadis 6 after the heat-treatment A
Slika 3: Mikrostruktura jekla Vanadis 6 po toplotni obdelavi A

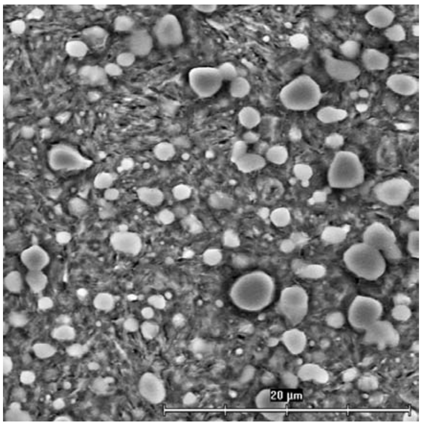

Figure 4: Microstructure of Vanadis 6 after the heat-treatment B
Slika 4: Mikrostruktura jekla Vanadis 6 po toplotni obdelavi B

Figure 6 (after heat treatment). It is obvious that after the heat treatment the number of carbide particles is lower and their size is smaller in comparison to the as-received material. The main reason is that the as-received material contains a larger number of ultra-fine eutectoid carbides that undergo a complete dissolution in the austenite. In addition, also a part of the secondary carbides is dissolved in the austenite during the heating up to the final austenitizing temperature

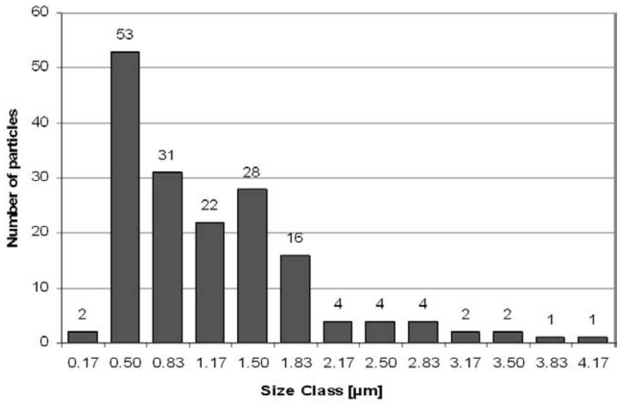

Figure 5: Results of the analysis of the carbide particles' sizes and the number in the as-received material
Slika 5: Rezultati določitve velikosti in števila karbidnih zrn v prejetem jeklu

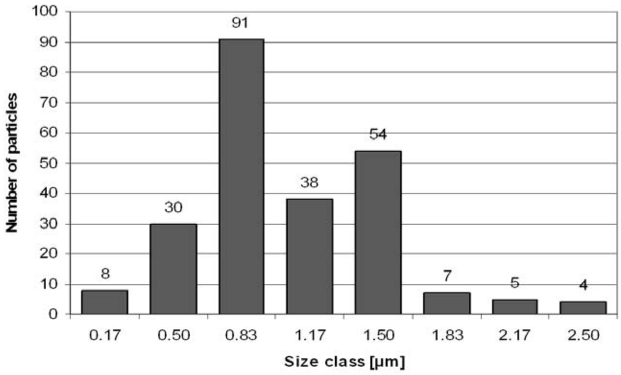

Figure 6: Results of the analysis of the carbide particles' sizes and the number in the material after the heat treatment
Slika 6: Rezultati določitve velikosti in števila karbidnih zrn v toplotno obdelanem jeklu

3.2 Hardness

The results of the hardness measurements obtained from the samples of all the heat treatments are presented in **Table 2**. The hardness of the as-received material was HV_{10} = 284 and the average hardness after heat treatment without a sub-zero treatment was of HV_{10} = (748 ± 6.9) (HRC = (60 ± 0.2)). The process with the 4-h sub-zero treatment at –196 °C leads to an average surface hardness of HV_{10} = (734 ± 6.9) (HRC = (58 ± 1.0)). Finally, the process including the 7-h sub-zero treatment at –196 °C leads to the average surface hardness of HV_{10} = (721 ± 5.6) (HRC = (58 ± 0.4)).

It is evident that after the sub-zero treatment the hardness is slightly lower – of the order of tens of Vickers units compared to the heat treatment without this treatment. This hardness decrease is higher with a longer duration of the sub-zero period. These findings do not agree with [2,4], which stated that the hardness increased after this procedure.

Table 2: Average hardness and standard deviation after different heat treatments
Tabela 2: Povprečna trdota in standardna deviacija po različni toplotni obdelavi

Measurement Method	HV_{10}			HRC		
Heat Treatment	A	B	C	A	B	C
Average Value of Hardness	748	734	721	60	58	58
Standard Deviation	6.9	6.9	5.6	0.2	1.0	0.4

3.3 Three-point bending strength

The three-point bending strength was used to determine the materials' resistance to fracture initiation. The measured parameters were the maximum load until fracture and the bending strength. The tests were performed on all the samples and the average values of the samples with the same heat treatment are presented in **Table 3**. The bending strength was calculated from the F_{max} using the formula: $R_{Mo} = \frac{3F_{max} \cdot L}{2a^3}$, where: F_{max}/N – load at the fracture, L/mm – length of the sample, a/mm – width of the sample with square intersection.

Table 3: Results of the three-point bending-strength test
Tabela 3: Rezultati tritočkovnega upogiba

Heat treatment	Average value of bending strength /MPa	Average value of the maximum load /kN
A	2436	16.24
B	2961	19.74
C	3217	21.45

The relationships between the load and the deformation (F-s) of the samples are shown in the following diagrams. **Figure 7** shows the F-s diagram for the sample processed with the processing route A (no sub-zero treatment). The F-s relation is practically linear, therefore only a minimum plastic deformation until fracture can be expected to occur. Also, the three-point bending strength and the maximum load F_{max} were relatively low (**Table 3**); the fracture appeared when the load reached only 16.24 kN. In **Figure 8**, the behavior after the B heat treatment (–196 °C, 4 h sub-zero treatment) is shown. The F-s relation is not a completely linear shape, especially at high loading, and indicates that some plastic deformation took place during the testing. The fracture in this case appeared at a load of 19.74 kN. **Figure 9** shows the results of the three-point bending test of the sample processed with the C treatment (–196 °C, 7 h sub-zero treatment). The dependence of the load deformation is even less linear because of the greater portion of plastic deformation. The fracture occurs at 21.45 kN.

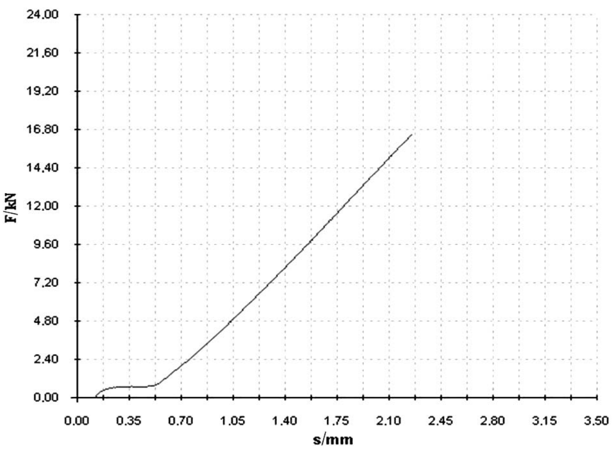

Figure 7: Dependence of F-s for the three-point bending test of the sample processed using the A treatment (without the sub-zero treatment)
Slika 7: F-s-odvisnost za tritočkovni upogib po toplotni obdelavi A (brez podhladitve)

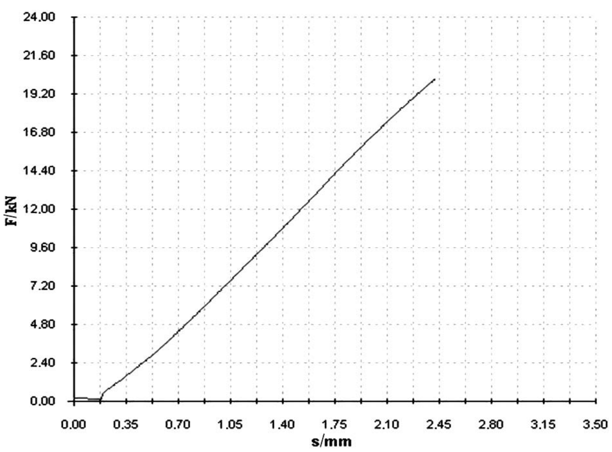

Figure 8: Dependence of F-s for the three-point bending test of the sample processed using the B treatment (with –196 °C, 4 h sub-zero treatment)
Slika 8: F-s-odvisnost za tritočkovni upogib po toplotni obdelavi B (podhladitev –196 °C, 4 h)

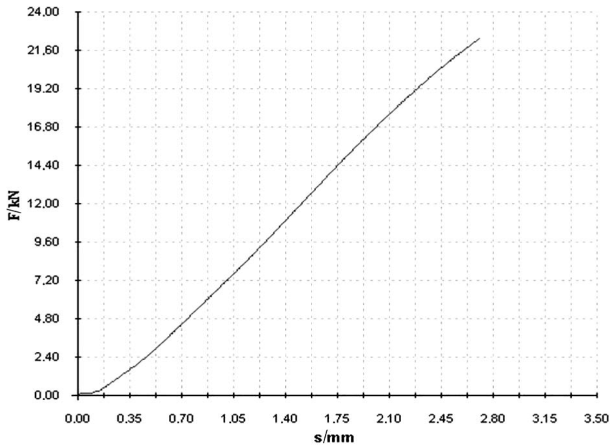

Figure 9: Dependence of F-s for the three-point bending test of the sample processed using the C treatment (with –196 °C, 7 h sub-zero treatment)
Slika 9: F-s odvisnost za tritočkovni upogib po toplotni obdelavi C (podhladitev –196 °C, 7 h)

From these results, it seems that the sub-zero period has a relatively small, but positive, effect on the three-point bending strength and it agrees with the changes in hardness, as with a higher bending strength, the hardness is lower.

3.4 Fractography

The fracture of the specimen processed using the processing route A is shown in **Figure 10**. The fracture was initiated on the tensile-strained side of the specimen and propagated throughout the specimen. Step-like propagation tracks are visible in the region near the surface of the specimen. At a depth of approx. 1 mm, a great step is also found.

The micrograph in **Figure 11** at a higher magnification shows that the fracture surface presents a dimpled morphology with some areas of microcleavages and indicates that a very low energy was spent for the crack propagation.

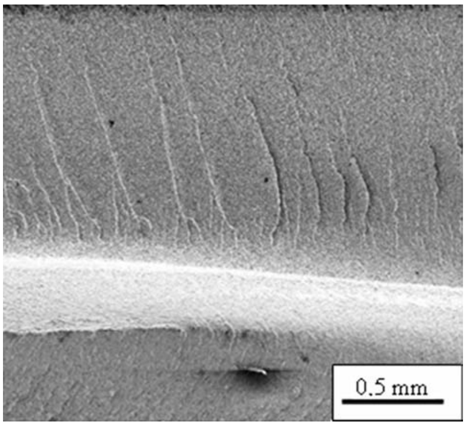

Figure 10: Fracture surface of the sample processed using the A treatment
Slika 10: Površina preloma po toplotni obdelavi A

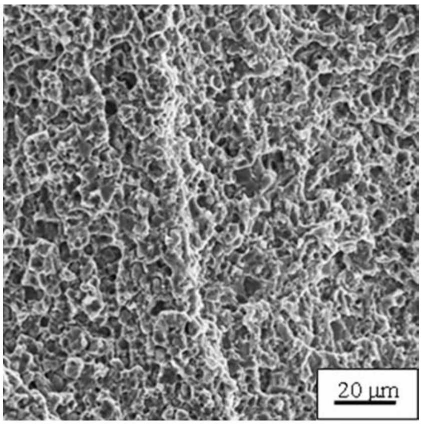

Figure 11: Detail of the fracture surface of the sample processed using the A treatment
Slika 11: Detajl površine preloma po toplotni obdelavi A

The fracture surface of the sample processed using the C treatment sample at 35-times magnification is presented in **Figure 12**, and at 1000-times magnification is shown **Figure13**. The dimples in the fracture surface of the sample processed using the C treatment are deeper and the step-like propagation tracks are longer than on the sample processed using the A treatment (**Figure 10**). Considering the *F*-*s* diagrams, showing that for the sample processed using the C treatment a greater amount of plastic deformation energy was consumed, it is possible to say that performed sub-zero treatment improves slightly the ductility of the tool steel.

4 CONCLUSIONS

1) After all of the performed heat-treatment processes the microstructure of he material consisted of tempered martensite and undissolved carbides.
2) The hardness was slightly higher for the steel without the sub-zero treatment than for the steel that underwent this process.

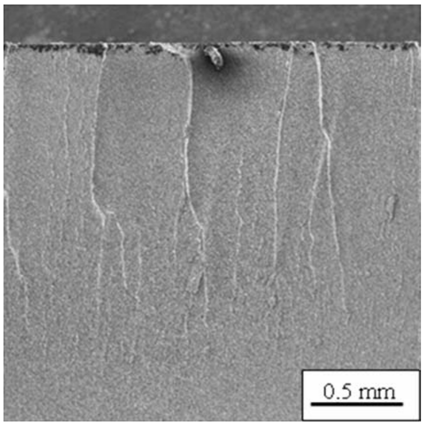

Figure 12: Fracture surface of the sample processed using the C treatment
Slika 12: Površina preloma po toplotni obdelavi C

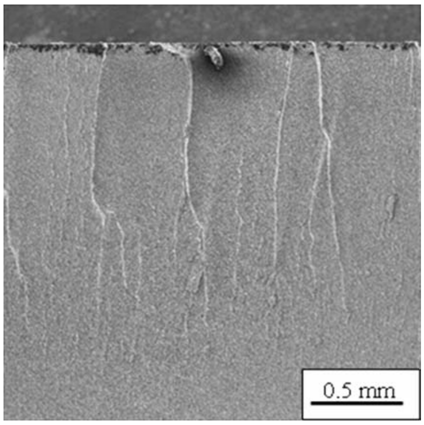

Figure 13: Detail of the fracture surface of the sample processed using the C treatment
Slika 13: Detajl površine preloma po toplotni obdelavi C

3) The sub-zero processed samples had a slightly higher three-point bending strength. The reason is that the crack propagation is connected with a higher consumption of plastic energy until the fracture.
4) The results of the mechanical tests are rather unexpected and so further, and more accurate, tests and examinations will be necessary to determine and verify the causes of this unexpected observation.

Acknowledgements

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5 LITERATURE

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