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ON THE CHANGE OF NOTCH TOUGHNESS TRANSITION TEMPERATURE OF STRUCTURAL STEELS AFTER STRAIN AGEING

O SPREMEMBI PREHODNE TEMPERATURE ZAREZNE ŽILAVOSTI KONSTRUKCIJSKIH JEKEL PO DEFORMACIJSKEM STARANJU

Franc Vodopivec[1], Bojan Breskvar[1], Jelena Vojvodič-Tuma[1], Savo Spaić[2], Boštjan Markoli[2]

[1]Institute of Metals and Technology, Lepi pot 11, 1000 ljubljana, Slovenija
[2]Faculty of Natural Sciences and Engineering, 1000 Ljubljana, Slovenija
franc.vodopivec@imt.si

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Based on experimental data and transmission electron micrographs an explanation of the mechanism of the change of Charpy notch transition temperature after strain ageing is proposed. The explanation involves the interplane ordering of carbon atoms at ageing annealing at 250 °C as a synergy of the dislocation structure and the redistribution of carbon atoms in solid solution in ferrite. The induced internal stresses decrease the cleavage fracture strength of the ferrite matrix and increase the Charpy notch toughness transition temperature.

Key words: structural steels, strain ageing, dislocation structure, segregation of carbon atoms, internal stresses.

Na podlagi eskperimentalnih podatkov in posnetkov s presevnega elektronskega mikroskopa je predložena razlaga mehanizma spremembe prehodne temperature Charpy zarezne žilavosti po deformacijskem staranju. Razlaga vključuje medploskovno ureditev atomov ogljika med staranjem pri 250 °C kot sinergijo dislokacijske strukture in prerazdelitve atomov ogljika v trdni raztopini v feritu. Inducirane notranje napetosti znižajo cepilno trdnost feritne kristalne mreže in povišajo prehodno temperaturo Charapy žilavosti.

Ključne besede: konstrukcijska jekla, deformacijsko staranje, dislokacijska struktura, prehodna temperatura zarezne žilavosti, segregacija ogljikovih atomov, notranje napetosti

1 INTRODUCTION

The diffusion of carbon in ferrite can alter the mechanical properties by three mechanisms[1]: stress induced ordering of carbon atoms among the possible interstitial sites[2], segregation of carbon atoms to form atmospheres[3] and precipitation of iron carbide particles. All three kinds of carbon redistribution occur during the strain ageing of steel[4]. The strain ageing mechanism was investigated and explained on the base of the suggestion that for an interstitial atom situated in the centre of an edge dislocation the binding energy is greater than if the same atom was bound to iron carbide or nitride[5,6]. Interstitials atoms are initially distributed randomly among the mid points of (100), (010) and (111) cube edges and it is energetically advantageous for them to move in plastically deformed lattice of ferrite to particular positions if the resulting tetragonality decreases the stress due to the dislocation[7]. At a large distance from the dislocation thermal agitation would over-ride this redistribution of interstitial atoms[7]. According to[8] a tensile strain of 0.005 induces substantial ordering of interstitials at room tempereture and such strain is to be found within about 20 atomic spacings of a dislocation. In the first stages of ageing interstitial atoms diffuse to free dislocations and occupy sites where intereaction is strong. In moderately deformed, slowly cooled steels 0.005-0.001 % of interstitial in solution is sufficient to complete the locking of dislocations, while, contributing rather little to the lower yield stress in strain aged steels[9]. From measurements of internal frixion it was estimated that between 10 and 50 atoms have segregated to each atomic plane after the completion of the ageing of a steel with more than 0.01 % C deformed plastically for less than 10 %[10]. The segregation of about 0.001 % of carbon or nitrogen is sufficient to complete atmosphere locking in a moderately deformed low carbon steel[11], however, the solute effect decreases as the precipitation develops further. After strain ageing the steel mechanical properties are changed and solutes segregation to atmoshere is more effective in rising the yield stress. Also tensile strength and the work hardening exponent are increased, while the elongation to fracture is decreased[11]. The increase of yield stress and the decrease of uniform elongation are much greater for structural steels with a microstructure of polygonal ferrite and pearlite[12,13] than with a fine grained steel with a quenched and tempered ferrite and pearlite and a steel with a microstructure of tempered martensite. For structural steels with the yield stress in range from 265 MPa to 1000 MPa and a microstructure of polygonal ferrite and pearlite, quenched and tempered fine grained ferrite and pearlite and tempered martensite a numerical equation for the increase of yield stress after strain ageing (ΔE_{sa}) was deduced in [14].

For the same steels after strain ageing the uniform elongation at tensile test is diminished to below ¼, while the reduction of area is decreased very little. It is decreased f.i. for a polygonal ferrite pearlite structural steel with the room temperature yield stress of 364 MPa from 68 % to 63 %. This difference indicates that strain ageing affects strongly the deformability with uniaxial stressing, while it affects less the deformability with triaxial stressing[15].

After strain ageing the Charpy transition temperature, defined as temperature for 50 % of the upper shelf Charpy notch toughness, is significantly increased and the propensity of the steel to brittle fracture shifted to a higher temperature. The increase of transition temperature depends on the initial yield stress, thus of the level of ferrite strengthening. For the mentioned structural steels the relation toughness transition temperature (T_t) versus the initial yield stress was proposed in [15].

Brittle cleavage fracture of steel occurs when the emission of dislocations required for the blunting of the crack tip, essential for ductile crack propagation, is prevented. The movement of dislocation is hindered with the Peierls-Nabarro force, which increases proportionally to the content of carbon and nitrogen in solid solution in α iron and it is greater at lower temperature[16].

The content of carbon in solid solution in ferrite is greater after annealing at 550 °C than after annealing at 400 °C, while, the transition temperature was virtually equal in both cases and it is higher than in non strain aged steel with a greater content of carbon in solid solution[17]. The yield stress of ferrite with 44 ppm of carbon in solid solution increases exponentially with the decrease of temperature and it is of 25 MPa at 300 K, of 72 MPa at 200 K and of 209 MPa at 100 K[18]. The figures show that the locking effect of interstitials on dislocations is smaller for a lower temperature, while the mobility of dislocation is greater. For ductile decohesion to propagate a determined dislocation mobility is required, this being greater by higher temperature, it is clear that the increase of transition temperature can not be explained in terms of Peierls-Nabarro force.

The effect of annealing temperature and of deformation, separately, as well as strain ageing on notch transition temperature was investigated on the normalised steel with 0.12 % C, 0.26 % Si, 0.59 % Mn, 0.027 % Al and ≈0.007 % N[17]. A selection of tests results are presented in **Table 1**.

The ratio aluminium over nitrogen Al/N = 4 is sufficient to assume safely that strain ageing effects in the investigated steels were due mostly to carbon remained in solid solution in ferrite after air cooling from the austenitising temperature of 905 °C. Nitrogen is in interstitial solid solution in ferrite and it solubility and atomic radius are also similar to those of carbon and, as established in several of the quoted references, residual nitrogen atoms behave in the strain ageing mechanism in the same way as carbon atoms do.

Table 1: Results of Charpy notch toughness tests of a ferrite pearlite structural steel

Tabela 1: Rezultati preizkusov Charpy zarezne žilavosti feritno konstrukcijskega jekla z mikrostrukturo iz ferita in perlita

Heat treatment	$C_{sol.}$	Notch tough.[1]	Temp.[2]	Δ temp.[3]	Hard-ness
	ppm	J	°C	°C	HV 5
Normalised	>100	183	–63	–	124
Norm., ageing 0.5 h 250 °C	0.04	178	–51	12	129
Norm., ageing 2 h 550 °C	27	198	–55	8	131
Norm., ageing 2 h 550 °C, def. 27	191	-50	12	179	
Norm., 2 h 550 °C, strain ag.	0.04	171	–5	58	183
Norm., ageing 2 h 400 °C	2.4	190	–51	12	132
Norm., 2 h 400 °C, strain ag.	0.04	185	–16	47	183
Quench., ageing 2 h 550°C	27	240	–126	-63	211
Quench., 2 h 550 °C, strain ag.	0.04	236	–89	-44	213

[1] Upper shelf notch toughness.

[2] Temperature for the notch toughness of 50 J.

[3] Difference to the 50 J. temperature after normalisation

After normalisation the number of cementite particles in the interior of ferrite grains was very small and no intergranular precipitation of cementite was observed. It was therefore assumed that the content of carbon in solution in ferrite after cooling of the steel from the normalisation temperature was above 100 ppm. This level amounts to less than a half of the solubilty of carbon in ferrite at the eutectoid temperature.

Experimental data on the solubility of carbon in ferrite from ref.[19] can't be used because valid for a steel maintained for an indefinite time at the coiling temperature. For this reason, the solubility of carbon in ferrite in **Table 1** was calculated using the dependence[20]

$$\% \, C_w = 240 \exp. (-77300/RT) \qquad (1)$$

with % C_w – wt % of carbon in solid solution in ferrite, R – universal gas constant and T – temperature in K.

After annealing at 550 °C the calculated theoretical content of carbon in solid solution in ferrite was of 14.4 ppm, of 2.4 ppm after annealing at 400 °C and of 0.04 ppm at the ageing temperature of 250 °C. The precipitation of cementite at 550 °C, 400 °C, 250 °C and the double annealing first at 550 °C than at 250 °C increases only slightly the hardness, while after 10 % of plastic deformation the hardness is strongly increased[17]. This was expected according to the finding that for a normalised steel with a microstructure of polygonal ferrite and pearlite the yield stress increased from 290 MPa to 546 MPa after 10 % of plastic deformation and to 580 MPa after 30 min. of annealing at 250 °C[21]. This indicates that cementite particles precipitated from carbon in solid solution in ferrite after normalisation, produce only a minor precipitation hardening when compared to the work hardening caused by the cold rolling of the steel. This is not in favour of the explanation that the increase of the Charpy notch toughness transition temperature is related to the precipitation of cementite at the annealing at 250 °C following the plastic deformation of the steel. On the other hand, the plastic deformation alone did not affect significantly the transition temperature. This finding differs from the conclusion that the straining affects significantly the cleavage fracture and the transition temperature[22,23].

After intermediate tempering of the steel at 550 °C and 400 °C and strain ageing the increase of toughness transition temperature was slightly smaller than that for the normalised and strain aged steel. The hardness after strain ageing was in all cases similar to that after 10 % of plastic deformation. With water quenching from normalisation temperature a very fine grained acicular ferrite pearlite microstructure was obtained with a higher hardness and upper shelf Charpy toughness and a significantly lower transition temperature[17]. Thus, the propensity to strain ageing is not affected by the steel grain size and shape, while, the decrease of transition temperature is in agreement with the finding that for a mild steel there is a linear dependence transition temperature versus grain size with lower transition temperature for the steel with smaller grain size[24].

Brittle fracture occurs if the yield stress of a steel is higher than its cleavage strength and it occurs if the apllied stress is greater than the critical stress[25]:

$$\sigma_c \approx 4\, G\, \gamma / \alpha\, k\, d^{1/2} \qquad (2)$$

with G – shear modulus, γ – fracture surface energy, α – constant, k – the constant in the relation $E = k' + k\, d^{1/2}$ with d – average linear grain size.

For two steels the dependence load versus deflection was determined for the temperature interval above the upper to below the lower shelf notch toughness threshold. From the recorded curves the average Charpy brittle fracture stress of 1325 MPa was deduced for a steel with a microstructure of polygonal ferrite and pearlite and the yield stress of 364 MPa at room and 475 MPa at nil ductility temperature[26] (NDT). For a similar steel the cleavage strength of 1250 MPa was determined with tensile tests of notched specimens[27] and the term relative to the share of grain size in uniaxial yield stress at room temperature was kd^2 = 135 MPa[28]. Provided that the fracture is slip induced, the cleavage strength is substatially independent on the temperature[29].

The quoted references show that the mechanism of strain ageing and it effect on mechanical properties as well as on the transition temperature ductile to brittle fracture was widely investigated and explained. So far none experimentally confirmed explanation was proposed for the mechanism of the increase of the Charpy toughness transition temperature brittle to ductile fracture, which was the subject of this article.

2 EXPERIMENTAL WORK AND RESULTS

The experimental work consisted of the verification of the findings in table 1 with a 0.10 % C, 0.25 % Si, 0.62 % Mn, 0.0047 % N, 0.036 % Al, 0.002 % S, 0.001 % P, steel with a microstructure of polygonal ferrite and pearlite and yield stress R_E = 304 MPa, tensile strength R = 425 MPa, elongation A_5 = 33.5 % and reduction od area 68.2 % as well as transmission electron microscopy of thin foils of this strain aged steel and of that in table 1. Before the Charpy tests the specimens were normalised and submitted to three different treatments: 10 % of cold rolling deformation, 30 min of ageing at 250 °C and strain ageing combining 10 % of cold deformation and ageing at 250. Thin foils for transmission electron microscopy were cut out from the area of uniform elongation of a strain aged tensile specimen fractured at the nil ductility temperature of –120 °C[27] and from a strain aged Charpy specimen from **Figure 1**.

The dependence Charpy notch toughness versus testing temperature is shown for the three kinds of specimens of the same stee in **Figure l**. After cold deformation and ageing alone the transition temperature

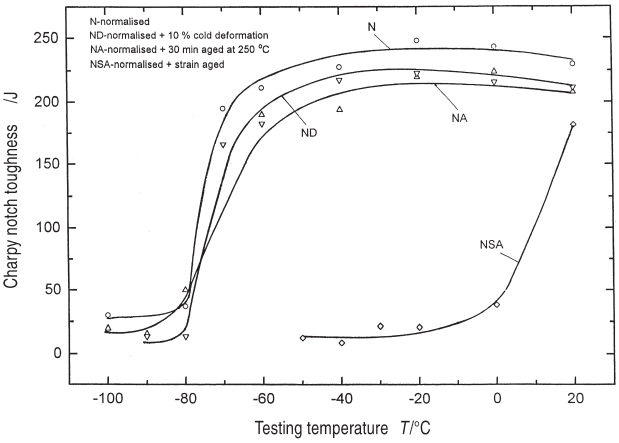

Figure 1: Dependence Charpy notch toughness versus testing temperarture for a 0.1 % C, 0.25 % Si, 0.62 % Mn, 0.036 % Al and 0.0047 % N steel after different heat treatment and ev. strain ageing: a) normalisation, b) normalisation and 10 % plastic deformation, c) normalisation and 30 min ageing at 250 °C and d) normalisation and strain ageing.

Slika 1: Odvisnost med temperaturo in Charpy zarezno žilavostjo za jeklo z 0,1 % C, 0,25 % Si, 0,62 % Mn, 0,036 % Al in 0,0047 % N po različnih obdelavah: a) normalizacija, b) normalizacija in 10 % plastične deformacije, c) normalizacija in 30 min žarjenja pri 250 °C in d) normalizacija in deformacijsko staranje (10 % plastične deformacije in 30 min žarjenja pri 250 °C.

remains unchanged with respect to that after normalisation, while after strain ageing it is strongly increased. Thus, the findings in **Table 1** are confirmed.

In the this foil cut out from the tensile specimen a dislocation network was found. Its typical feature were dislocations, which in the {211} plane rich in prepitates approached to other dislocations to a minimal distance and were than deflected with the angle of ≈ 80° (**Figure 2**) and a periodical array of diffused lines (**Figure 3**). In an earlier investigation[30] in the foil cut out from the aged Charpy specimen from table 1 a less distinct periodical array of parallel and diffused lines was observed, also. Similar arrays were observed in martensite tempered at low temperature and explained as indications of stress fields caused by the clustering of carbon atoms in specific crystal planes, which occurs in the first stages of the tempering of martensite[31,32]. The average interline distance in **Figure 3** corresponds to 9 (211) interplane distances.

3 CHANGE OF CHARPY NOTCH TOUGHNESS TRANSITION TEMPERATURE

Based on ref.[30] and the **Figures 2 and 3**, it is assumed that after strain ageing the movement of dislocations is impaired because of parallel fields of elastic stresses stress in the {211} planes situated at an equal mutual distance. The prepitation of cementite alone causes a very low precipitation hardening and has only a negligible effect on the transition temperature brittle to ductile fracture. On the other hand, plastic deformation produces a strong strain hardening and does not also affect significantly the transition temperature, as shown in **Table 1**. The increase of transition temperature of the strain aged steel would on principle favour the ductile fracture because of the diminished Peierls-Nabarro force. All these facts considered, it seems logical to assume that the rise of notch toughness transition temperature is due to a synergistic effect of the dislocation structure and the redistribution of carbon atoms in equililibrium solid solution in ferrite at the ageing temperature with their alignment in the {211} planes interspaces or a specific, deformation induced precipitation of a carbide phase in the same interspaces.

The carbide precipitation of the carbon left in solid solution after normalisation does not affect virtually the transition temperature. The coherency of the matrix and the precipitate lattices is necessary for the formation of elastic, lattice acomodating stresses.

Let us assume that of all possible iron carbides[33] only cementite is formed at the strain ageing annealing. Only particles with the cube lattice and the lattice parameter very near to that of α iron and of sufficiently small size can grow coherently with the ferrite lattice and produce a much stronger precipitation hardening than the same quantity of the precipitated phase in form of coarser particles, f.i. niobium carbides in microalloyed steel[34] and molybdenum carbide in tool steels[35]. Cementite has an orthorombic lattice with the parameters $a = 0.451$ nm, $b = 0.507$ nm and $c = 0.673$ nm and the coherency with the ferrite lattice would be connected with unaittanable accomodating elastic stresses. These would greatly increase the hardness, which is similar after cold deformation and strain ageing. Cementite particles are stable at sufficient size and the calculation shows that 30 min of annealing at 250 °C is sufficient for the growth of

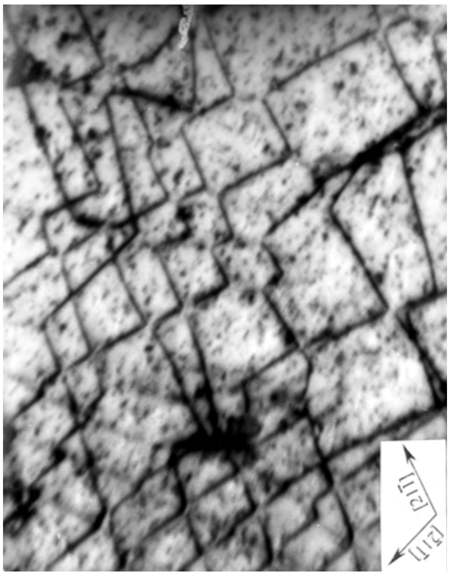

Figure 2: Thin foil. Array of dislocation in the uniform elongation part of a tensile specimen tested at the nil ductility temperature of –120 °C. Strain aged steel from **Figure 1**

Slika 2: Tanka folija. Splet dislokacij v enakomerno deformiranem delu preizkušanca, ki je bil pretrgan pri temperaturi –120 °C. Deformacijsko starano jeklo s **slike 1**

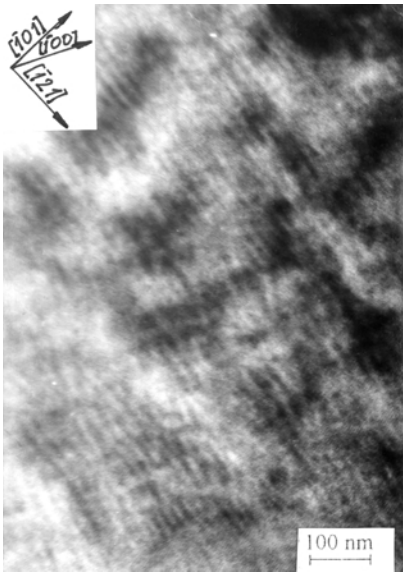

Figure 3: Thin foil. Array of diffused lines in the normalised and strain aged steel from **Figure 1**

Slika 3: Snopje difuznih črt v normaliziranem in deformacijsko staranem vzorcu jekla iz **slika 1**

cementite particles[13] of size of a few nm, to coarse to accomodate coherently in the {211} interspaces. The formation of intermediate carbide phases, as with the tempering of martensite[31], is not probable in the present case because it occurs in a very different matrix, with a content of carbon higher by more than 10^4 and at a lower temperature.

It seems, therefore, that the increase of transition temperature after strain ageing can not be ascribed to the precipitation of cementite or other iron carbide phases, but more probably to a redistribution of carbon atoms at the annealing at 250 °C in the steel submitted previously to 10 % of plastic deformation. With the redistribution ordering of solute atoms is introduced in the ferrite lattice which and the increased interplane distance decreases the cleavage strength and increases the propensity of steel to brittle fracture as well as the notch toughness transition temperature.

The octahedric site of the ferrite lattice with the size of 0.078 nm[36] is preferred site for the insertion of carbon atom with a diameter of 0.09 nm. The insertion extends the lattice and induce elastic stresses and the solid solution strengthening[37]. The interaction energy E_C of the introduction of an interstitial carbon atom, which causes a volume change of ΔV, is[38]

$$E_C = K\,\theta\,\Delta V \tag{3}$$

With K as bulk modulus and θ the local dilatation strain.

Let us assume that the strain of insertion of a carbon atom in the lattice cell with length $L = 0.286$ nm produces a dilatation equivalent to the extension of $\Delta L = 0.001$ nm of the length of the cube edge with the resulting strain of aproximately $\Delta L/L = 3.5\cdot10^{-3}$. The resulting decrease of insertion energy is deduced to $\Delta E_C = 1.84\cdot10^{-20}$ J rsp. 0.11 eV per lattice cell. Let further assume that with the cooling from 400 °C to 250 °C half of the carbon atoms, thus 1.2 ppm C or. $3.3.\cdot10^{17}$ at·cm^{-3} in solid solution at higher temperature are bound to cementite and half remain inserted in the lattice. If the insertion occurrs in the previously elastically strained lattice cells, the lattice elastic energy of α iron would be decreased for $\Delta E_C = 7.6\cdot10^{-3}$ J/cm^3. An approximate value of the energy involved in the insertion of carbon atoms in the α iron lattice can be deduced also from the increase of yield stress. This is increased for 4.6 MPa/0.001 wt.% C in solid solution[39], it is thus of 0.55 MPa/1.2 ppm C. Assuming the elastic behaviour of the lattice the stored energy is of $0.55\cdot10^{-2}$ J/cm^3 On the other side, the free energy for the formation of cementite from 1.2 ppm of carbon is of $5.5\cdot10^{-4}$ J/cm^3. Thus, the insertion of interstitials atoms in the previously strained lattice would produce a decrease of the elastic energy of the lattice even higher than the free energy of the binding of an equal number of carbon atoms to cementite.

The fact that the annealing alone of the steel at 250 °C does not change appreciably the notch toughness transition temperature supports the assumption that for the ordering of the solid solution of carbon atoms in the α iron lattice elastic stresses due to the plastic deformation are necessary.

4 CONCLUSION

After strain ageing of a mild structural steel yield stress and tensile strength are increased, reduction of area very slightly and uniform elongation strongly decreased while, the transition temperature for Charpy notch toughness is significantly increased. Strain hardening and ageing alone do not shift appreciably the transition temperature. This shift is virtually independent upon the quantity of cementite precipitated at strain ageing annealing and before it, it is independent upon the temperature of intermediate annealing after normalisation and of the grain size. In a trasmission electron micrograf elongated diffused spots parallel to the {211} lattice planes of ferrite were found. The proposed explanation, for all experimental findings, is that the change of the lattice structure after plastic deformation allows the redistribution of carbon atoms in solid solutionin α iron and their segregation in {211} interplanes. A partially ordered structure is produced with a greater prodecrease of cleavage strength and the increase of the Charpy notch toughness transition temperature.

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