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FRACTURE CHARACTERISTICS OF THE Cr-V LEDEBURITIC STEEL VANADIS 6

PRELOMNE ZNAČILNOSTI LEDEBURITNEGA Cr-V-JEKLA VANADIS 6

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The P/M Vanadis 6 is a very popular tool steel used for different cold work applications. The specimens for the three point bending tests and fracture toughness test were austenitized at two different temperatures, quenched with or without the sub-zero treatment and then double tempered. The bending strength was lower by higher austenitizing temperature and also with the use of sub-zero treatment. The average fracture toughness was lower by higher austenitizing temperature also and increased slightly with the sub-zero treatment. The lowering of bending strength can be considered as logical result because of the austenitic grain growth, as well as increased internal stresses in the case of sub-zero processed samples. For fracture toughness, the situation seems to be more complex. On one side, the effect of austenitizing temperature on fracture toughness is similar as for bending strength, on the other side, the sub-zero treatment induces a rather slight increase of fracture toughness. The paper presents some details on the experiments performed including possible explanation of the material's behaviour considering microstructure investigations.

Key words: ledeburitic Cr-V PM steel, heat treatment, sub-zeto treatment, bending strength, fracture toughness, fracture surface

PM-jeklo Vanadis 6 je zelo popularno orodno jeklo za različno uporabo v hladnem. Preizkušance za tri točkovni upogib in žilavost loma smo avstenitizirali pri dveh različnih temperaturah, kalili z obdelavo in brez nje pod temperaturno ničlo in dvakrat popustili. Ugotovili smo, da je upogibna trdnost nižja po kaljenju z višje temperature in po obdelavi pod ničlo. Žilavost loma je bila nižja po kaljenju z višje temperature in je bila nekoliko večja po obdelavi pod ničlo. Zmanjšana upogibna trdnost je logičen rezultat povečanja velikosti avstenitnih zrn in povečanja notranjih napetosti po obdelavi pod ničlo. Primer žilavosti loma je bolj kompleksen, kajti na eni strani temperatura avstenitizacije vpliva podobno na upogibno trdnost in žilavost loma, po drugi pa obdelava pod ničlo poveča žilavost loma.V članku so predstavljeni detajli o opravljenih preizkusih in predstavljena je razlaga vedenja materiala z upoštevanjem mikrostrukture.

Ključne besede: ledeburitno PM CrV-jeklo, toplotna obdelava, obdelava pod ničlo, upogibna trdnost, žilavost loma, površina preloma

1 GENERAL REMARKS

Ledeburitic steels have a high wear resistance and hardness and are used in many industrial operations like: metal cutting, wood cutting, fine blanking, bending etc. In these operations, the material must meet various requirements. It has to withstand compressive stresses, abrasive and/or adhesive wear, but also chipping and total tool collapse.

To meet these demands, the ledeburitic steels must have an optimal chemistry, as well as phase constitution. Moreover, a proper heat treatment must be performed before the use. The ledeburitic steels contain a high content of carbon and alloying elements that form carbides responsible not only for high hardness and wear resistance but also for the capability of the material to achieve a high strength and acceptable fracture toughness after heat treatment. For the resistance to chipping and total collapse, two basic characteristics are important: three-point bending strength as a measure for the resistance against the crack initiation and the fracture toughness that characterises the resistance of the material against crack propagation.

The three-point bending strength is sensitive to various factors. First of all it is important how the material was made. It is known that the large carbide networks, clusters or bands are responsible for a significant lowering of three-point bending strength and its anisotropy [1,2]. This is also the main reason why the steels made via the powder metallurgy (P/M) of rapidly solidified particles have higher bending strength than the materials of the same chemistry made by conventional metallurgy. The three-point bending strength is also influenced by the material cleanless. It was reported that oxides, sulphides and other impurities can decrease the three-point bending strength significantly even at relatively low quantity [3]. The quality of the surface should also not be neglected. Some investigations have confirmed that the three-point bending strength decreases as the surface roughness increases [4]. The three point-bending strength decreases with the increase of the austenitizing temperature because of the coarsening of the austenitic grains during austenitization [5].

On the other hand, the fracture toughness depends mainly on the matrix hardness and its ductility. Generally, it also depends to its relation to the carbide network (cohesive strength at carbide-matrix boundary i.e. shape, size and size distribution of carbide particles, carbide clusters, distance among the particles etc). Ledeburitic steels have to be heat treated up to approximately *HRc* 60 (or more in some cases); and their fracture toughness is relatively low. For the conventionally manufactured chromium ledeburitic steels, as well as high speed steels, it decreases with the increasing austenitizing temperature and also with increasing tempering temperature, but only up to the maximum of secondary hardness [6]. The orientation of carbides and/or the state of the carbides (networks or bands) play only minor role [4,7,8]. On the other hand, the role of carbide distribution is highlighted in the soft state of the steels [7,8] the microstructure with carbide network had the lower fracture toughness, although the wrought material had better values. No data, except the early work of Olsson and Fischmeister [9], are known on the assessment of fracture toughness of P/M made ledeburitic steels. However, results published in the mentioned paper are negatively influenced by the state of manufacturing technique, porosity of the consolidated material, and therefore they did not have a sufficient value for today's manufactured and used materials.

The ideas about the sub-zero treatment on the mechanical properties of ledeburitic steels was developed over last few decades [10,11]. This technique has early gained scientific interest, but the attempts of its application did not produce doubtless results. Kulmburg et al. [11], for instance, reported for the M2-type steel a reduction of the amount of retained austenite after sub-zero treatment at –196 °C for 1 h and slight lowering of three point bending strength. Berns [10], on the other hand, reported a significant hardness increase for the sub-zero processed X290Cr12 ledeburitic steel. Last decade has brought new accurate investigations on the nature of possible improvement of material characteristics after the sub-zero processing. Stratton [12] pointed out, that especially for the D2 ledeburitic steel, the improvement of wear resistance due to the sub-zero processing could be increased to extremely high values. According to investigations of Collins and Meng [13–15], it is assumed that deep cooling leads to an arrangement of the structure responsible for the hindering of the dislocation movement. This induces a strengthening of the material. In addition, Meng assumed [14] that martensite originated at very low temperatures can differ from that transformed at higher temperatures in the lattice parameter. The martensitic transformation is probably superposed with the precipitation of nanosized carbides particles coherent with the matrix. However, up to now the principal explanation of the positive effect on essential properties (hardness, wear resistance) is not entirely clear. Moreover, also an optimal processing route of the cryogenic processing is not clearly determined and the opinions about it are different.

The main goal of our experimental effort was a reliable investigation on the influence of various factors (heat treatment, sub-zero treatment, tempering) on fracture toughness and three point bending strength of the PM ledeburitic steels. The Vanadis 6 P/M steel produced by Uddeholm was selected as example because of its relatively simple chemistry.

2 EXPERIMENTAL

The experimental material was the ledeburitic steel Vanadis 6 with nominally 2.1 % C, 1.0 % Si, 0.4 % Mn, 6.8 % Cr, 1.5% Mo, 5.4 % V and Fe as balance, manufactured with P/M (HIP of rapidly solidified particles) and soft annealed to the hardness of HV_{10} = 284.

Two types of specimens were prepared for the investigations. Samples for the three point bend testing (numbered 1 to 28) had a cross section of (10 × 10) mm and a length of 100 mm. They were ground to obtain a final surface roughness of 0.2–0.3 μm. The second type of specimens were the circumferentially notched and pre-cracked specimens (numbered 33 to 69) prepared according to the method described in [6,16] (see also **Figure 1**).

The specimens were then submitted to the heat treatment procedure that included vacuum austenitization up to temperature 1000 °C or 1050 °C, nitrogen gas quenching at 5 bar pressure and double tempering, each cycle at 550 °C for 1 h. In selected cases, the sub-zero treatment in liquid nitrogen was inserted between the quenching and tempering. The parameters of heat-treatment, the average K_{IC} and final hardness and are summarized in **Table 1**.

The fracture toughness was determinated according to the method published elsewhere [6,16]. At least eight samples were tested for a given parameter of the heat treatment. Three point bending tests have been carried out at following parameters: distance between supports of 88.9 mm, loading in the central region and loading rate of 5 mm/min up to fracture. The results of bend strength testing and hardness measurements are given in **Table 2**.

Figure 1: Precracked and fractured V notched cylindrical samples for K_{IC} determination
Slika 1: Prelomljeni valjasti preizkušanci z V zarezo za določitev K_{IC} z vnaprejšnjo razpoko

Table 1: K_{IC} for V notched cylindrical samples
Tabela 1: K_{IC} valjastih preizkušancev z V-zarezo

Specimens	Testing conditions			K_{IC}	Notch strength	Rockwell hardness
	Preheating and quenching	Subzero treatment in liqu. N_2	Tempering	Mpa √m	MPa	*HRc* *
33–40	650/850 °C → 1000 °C/30 → N_2 (5 bar)	–	2-times 550 °C/1 h	12.9 ± 0.9/1.3 (11.2 at $\boldsymbol{x}_0$)	283.3 ± 19.8/29.0	57.5 ± 0.3/0.7
41–50	650/850 °C → 1000 °C/30 → N_2 (5 bar)	−196 °C/24 h		13.2 ± 2.4/1.9 (9.5 at $\boldsymbol{x}_0$))	289.4 ± 51.6/42.0	56.4 ± 0.6/0.5
51–60	650/850 °C → 1050 °C/30 → N_2 (5 bar)	–		10.9 ± 1.5/1.2 (8.1 at $\boldsymbol{x}_0$))	238.0 ± 33.8/26.2	61.3 ± 0.4/0.8
61–69	650/850 °C → 1050 °C/30 → N_2 (5 bar)	−196 °C/24 h		12.5 ± 1.4/1.9 (9.1 at $\boldsymbol{x}_0$))	273.1 ± 31.2/40.9	58.8 ± 0.3/0.3

* One (1) Rockwell is added as a correction for rounded (cylindrical) samples

Table 2: Bend strength of samples with square section
Tabela 2: Upogibna trdnost preizkušancev s kvadratnim prerezom

Specimens	Testing conditions			Bend strength	Rockwell hardness
	Preheating and quenching	Subzero treatment in liqu. N_2	Tempering	MPa	*HRc*
1–7	650/850 °C → 1000 °C/30 → N_2 (5 bar)	–	2-times 550 °C/1 h	3626 ± 186/259	56.3 ± 0.7/1.2
8–14	650/850 °C → 1000 °C/30 → N_2 (5 bar)	−196 °C/24 h		3587 ± 143/169	55.3 ± 0.8/1.0
15–21	650/850 °C → 1050 °C/30 → N_2 5 bar	–		3474 ± 357/514	59.9 ± 0.8/1.1
22–28	650/850 °C → 1050 °C/30 → N_2 (5 bar)	−196 °C/24 h		3352 ± 385/307	58.1 ± 0.5/0.4

The microstructure of the material was investigated with light microscopy (LM) and scanning electron microscopy (SEM). The fractures surfaces were investigated with SEM. Hardness measurements were made using the Vickers method at a load of 98.1 N (HV_{10}) as well as using the Rockwell "C" method (*HRC*).

3 RESULTS AND DISCUSSION

The microstructure of the as-received material is shown in **Figure 2a** and **2b**. The LM micrograph (**Figure 2a**) gives us the overview. One can see that the material consists of the matrix and carbides particles that are very fine and uniformly distributed throughout the material. The SEM micrograph, (**Figure 2b**) shows that carbide particles of different size with the maximum size of particles is around 3 μm and the smallest have a size less than 0.5 μm.

The steel microstructure after heat processing is shown in **Figure 3a–d**. It consists of the martensitic matrix and of eutectic and part of secondary carbide particles. These particles are very fine and uniformly distributed in the matrix. The amount of carbides is slightly smaller for the specimens processed at higher austenitizing temperature. This is natural as higher temperature normally leads to the dissolution of large amount of secondary carbides.

Heat treatment leads to a substantial hardness increase. With increasing austenitizing temperature the hardness increases. It is natural because higher austenitizing temperature induces a better dissolution of secondary carbides and higher supersaturation level of martensite after the quenching. On the first sight, it is rather surprising that the sub-zero processing reduces the hardness only slightly. Several authors, for instance Berns [10], Kulmburg et al. [11], reported either slight or

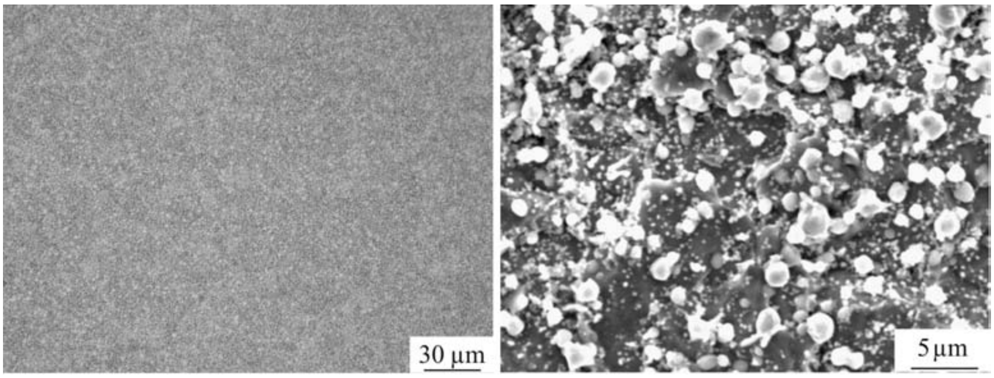

Figure 2: Microstructure of the investigated steel in as-received condition: a) LM micrograph and b) SEM micrograph
Slika 2: Mikrostruktura jekla v dobavljenem stanju; a) optični posnetek, b) SEM-posnetek

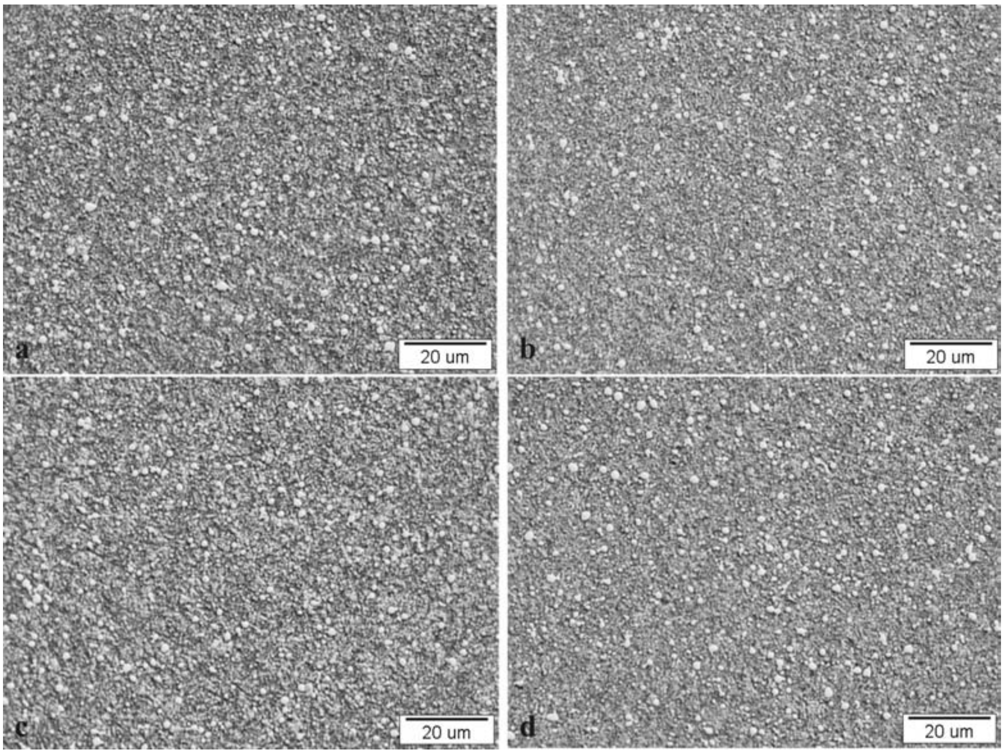

Figure 3: Microstructure of the Vanadis 6 steel after the heat treatment: a) austenitizing 1000 °C + quenching + 2x tempering, b) austenitizing 1000 °C + quenching + sub-zero + 2x tempering, c) austenitizing 1050 °C + quenching + 2x tempering, d) austenitizing 1050 °C + quenching + sub-zero + 2x tempering,

Slika 3: Mikrostruktura jekla po toplotni obdelavi: a) avstenitizacija pri 1000 °C + kaljenje + dvakratno popuščanje, b) avstenitizacija pri 1000 °C + kaljenje + obdelava pod ničlo + dvakratno popuščanje, c) avstenitizacija pri 1050 °C + kaljenje + dvakratno popuščanje, d) avstenitizacija pri 1050 °C + kaljenje + obdelava pod ničlo + dvakratno popuščanje.

more significant hardness increase of hardness after sub-zero treatment. However, in the mentioned papers, other ledeburitic steels were investigated, the sub-zero processing was carried out at different parameters and the tempering was shorter. It is assumed also, that two processes were happening during the deep freezing related to high stresses and strains; i.e. retained austenite to martensite transformation (hardness increase) and mechanical annealing (hardness decrease). Anyway, the reasons for the reduction in hardness requires more detailed TEM investigation.

The three point bending strength (**Table 2 and Figure 4a**), decreases with the increasing austenitizing temperature. This was expeced because at increased austenitizing temperature grains coarse structural constituents are changed and the bending strength is decreased. Also after the sub-zero processing, the three point bending strength is decreased. Similar results were published also by other authors [10,11,14] that did not suggest a reliable explanation. The bending strength is lower because of the increased internal stresses after quenching and deep freezing. These stresses are relieved by the

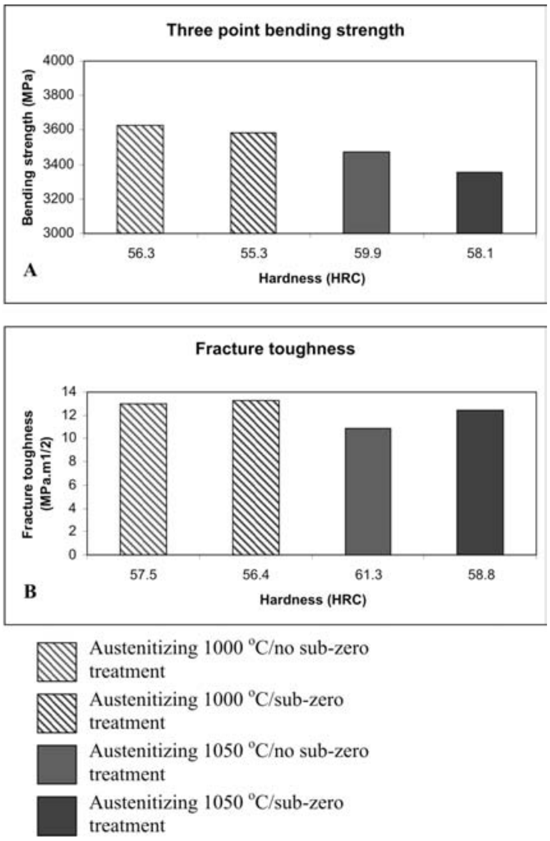

Figure 4: Graphical presentation of obtained experimental results for a) three-point bend strength and b) average fracture toughnes for different hardness
Slika 4: Eksperimentalni rezultati za a) tri točkovni preizkus upogibne trdnosti in b) žilavosti loma pri različni trdoti

tempering and this supports the conclusion that the sub-zero treatment decreases the material's resistivity to crack initiation. A possible explanations could be in the increased surface roughness because of martensite transformation (lattice reorientation and deformation) and mechanical annealing (dislocation glide and annihilation). Further investigations should prove this explanation.

On the other hand the results of fracture toughness (**Table 1** and **Figure 4b**) show slightly opposite tendency. Normally, it can be expected that higher austenitizing temperature would lead to a lower fracture toughness, since it is function of the matrix hardness. The principal explanation would the higher hardness of the matrix after higher austenitizing temperature. Nevertheless, the average fracture toughness after heat processing of Vanadis 6 steel was found to be slightly higher for sub-zero processed material. This is rather contradictory to the generally expected behaviour, as it is normally assumed that not only the resistance against initiation, but also the resistance against propagation of the crack should be lowered. However, it seems that the situation can be considered to be more complex, the more complete transformation of austenite into the martensite would be in favour of lower fracture toughness. On the other hand, the so-called effect of mechanical annealing (dislocation glide to the grain boundaries and a decrease of dislocation concentration inside the grains) can induce a resistance to the crack propagation resulting in the improvement of fracture toughness.

It is noticed that former investigations have shown a linear correlation between so called weak spot (x_0) and fracture toughness K_{IC}. In the case of tool and high speed steels this linear correlation shows a relatively large statistical reliability ($r^2 > 0.7$) and as the relevant material's K_{IC} value is used, the K_{IC} value at $x_0 = 0$ [18–20] are very conservative. In the case of materials with larger K_{IC} (for example hot work tool steels) this correlation is not so distinct and the mean value of all the determined K_{IC} values is usually used. In **Table 1** both approaches of K_{IC} results are given. For the $K_{IC} = f(x_0)$ approach a relatively good statistical reliability of results is obtained for three cases ($r^2 = 0.7–0.9$), except for the first set of experiments (specimens 33–40, **Table 1**). The obtained K_{IC} values at $x_0 = 0$ seems also to be relatively too conservative, therefore mean values (arithmetic average) of K_{IC} are used as relevant.

The fracture surface of not sub-zero processed sample after three point bending test is in **Figure 5a**. The fracture was initiated on the tensile strained side of the specimen (left margin of the micrograph) and propagated downwards the material. Some propagation lines are visible in the micrograph. In the direction from the tensile side to the core of the sample the fracture surface becomes a slightly more marked topography. As reported [5], such fracture surface (typical for hard steels) is known as low energetic ductile because some energy is also spent for the plastic deformation. However, the plastic energy is relatively low and corresponds to a relatively flat surface relief of the surface. More detailed micrograph, **Figure 5b**, shows that the fracture was propagated by two main mechanisms. The first is the cracking of coarser carbide particles (see **Figure 5c**) and the second is the de-cohesion at the carbide/matrix interfaces which can be attributed to different plasticity of these two microstructural constituents. In some places dimples are (tracks of extracted carbides) visible with in vicinity a plastically deformed matrix, **Figure 5d**.

The fracture surface of sub-zero processed material is shown in **Figure 6**. On the overview, **Figure 6a**, also propagation lines on the tensile strained side are visible. Detail micrograph, **Figure 6b**, confirms that the mechanism of fracture propagation is very similar to that of the sample processed without sub-zero period. Both, cracking of carbide particles and de-cohesion on the matrix/particle interfaces are clearly visible in the micrograph.

To explain the fracture propagation more precisely, the fracture surface of one specimen processed with a sub-zero period was examined to more in detail. **Figure 7a** shows a SEM micrograph at high magnification.

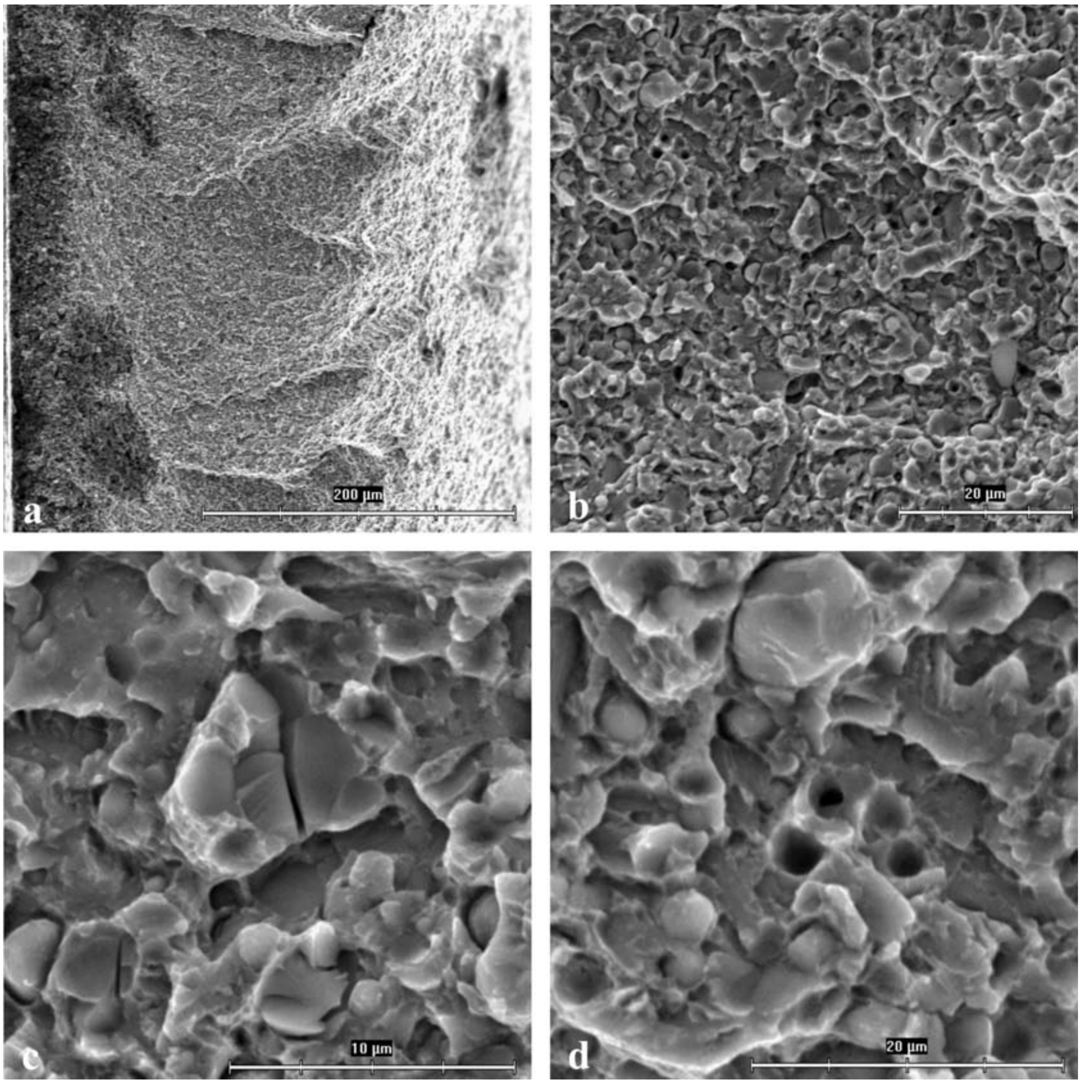

Figure 5: Fracture surface of the steel Vanadis 6 without a sub-zero treatment by different magnification
Slika 5: Površina preloma jekla brez obdelave pod ničlo; EM-posnetki pri različnih povečavah

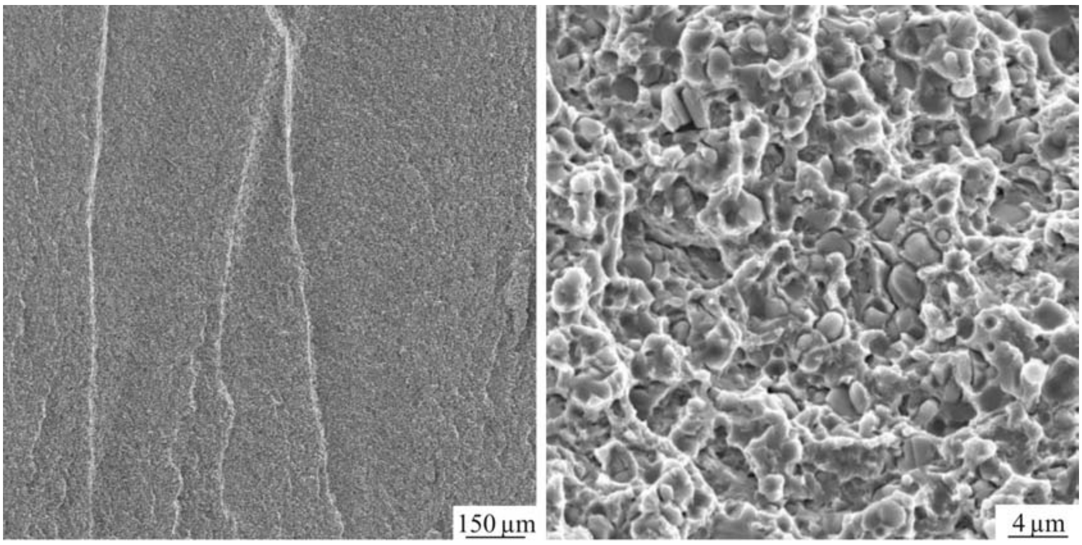

Figure 6: Fracture surface of the steel Vanadis 6 processed with a sub-zero period by different magnification
Slika 6: Površina preloma jekla po obdelavi pod ničlo; SEM-posnetka pri različni povečavi

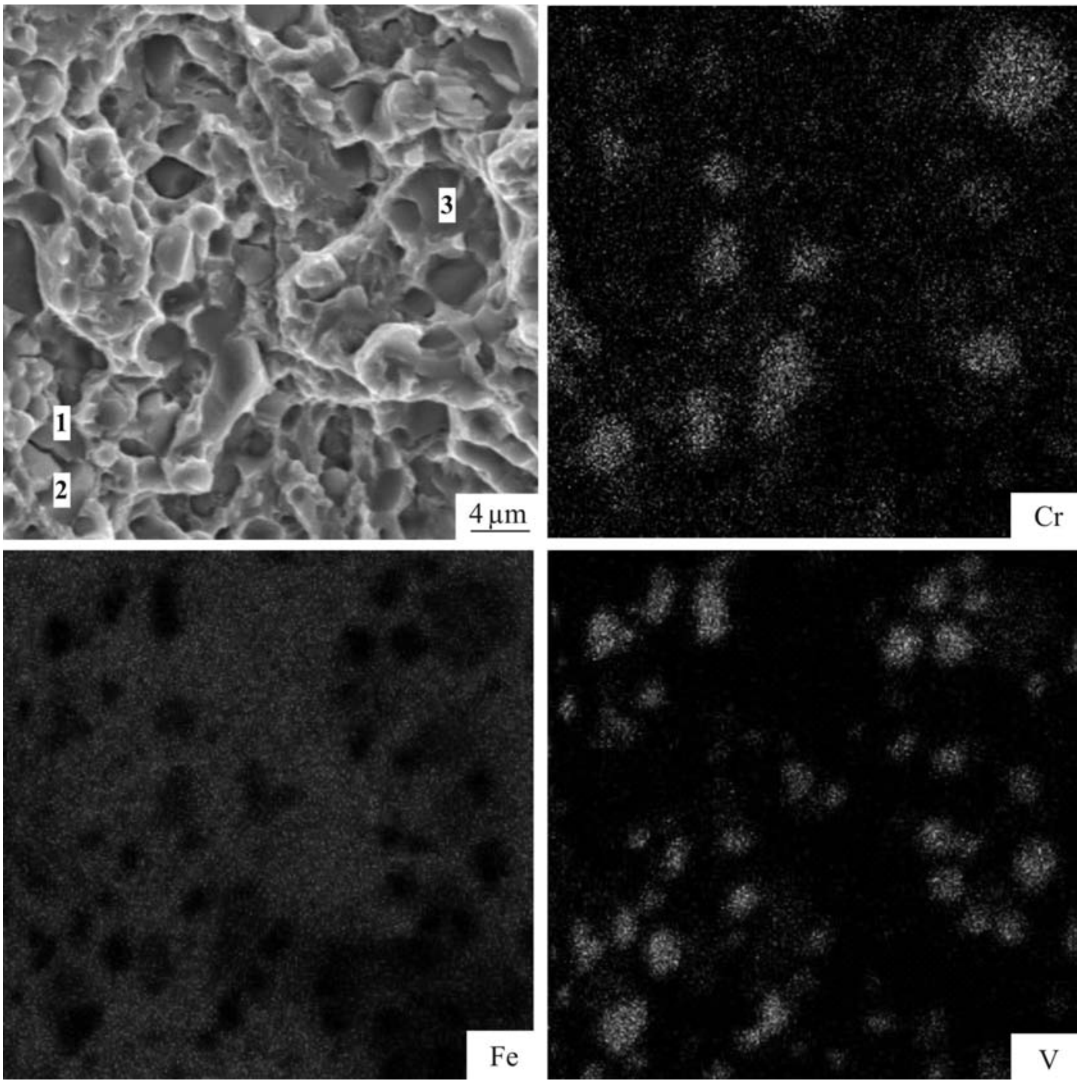

Figure 7: Fracture surface of the steel Vanadis 6 processed with a sub-zero period, fracture surface and EDS mapping for main alloying elements
Slika 7: Površina preloma jekla po obdelavi pod ničlo; prelomna površina in rasterski EDS-posnetek za osnovne legirne elemente

Three characteristic areas are marked. The first and second are carbide particles. The particle "1" was broken at fracture propagation. The particle "2" did not break and at the interface with the particle "1" decohesion is visible. The place "3" is a typical cleavage area in the matrix. EDX-mapping shows that the particle 1 is a chromium rich phase, probably of the M_7C_3-type. Its chemistry was estimated to 39 % Cr, 40 % Fe, 12 % V. The particle "2" contains more vanadium and less other elements – EDX fixed following chemistry: 13 % Fe, 61 % V, 9 % Cr. The cleavage region "3" can be referred as matrix with the chemical composition 89 % Fe, 6 % Cr, 1.7 % V.

4 CONCLUSIONS

The microstructure of the as-received material consisted of the matrix and carbide particles uniformly distributed in the material. The maximal size of particles is around 3 μm, but there are also particles with a size below of 0.5 μm (pearlitic carbides) are found.

After the heat-treatment the microstructure consists of the martensitic matrix, eutectic and part of secondary carbide particles. The amount of particles is smaller than in the as-received condition. For the heat processed material, the portion of carbide is slightly smaller for the specimens processed at higher austenitizing temperature.

The heat-treatment leads to a substantial hardness increase. With increasing austenitizing temperature the hardness increases, while, the sub-zero processing leads to slight decrease in hardness. This is rather surprising and inconsistent with earler observations. In this moment it is not clear why the sub-zero processing lowers the hardness of the steel Vanadis 6 steel and the explanation requires a more detailed investigation.

The three point bending strength decreases with the increasing austenitizing temperature. Also, with the application of sub-zero processing the three point bending strength is lowered.

The fracture toughness is lowered with increased austenitizing temperature. It is rather surprising that the sub-zero treatment increases the average fracture toughness slightly. Similarly to the hardness behaviour, this is not entirely clear and needs detailed TEM examination.

The fracture surfaces of the sub-zero- and the not sub-zero processed samples have similar basic characteristics. Both surfaces shown clearly dimple morphology. In some areas details of crack propagation like breaking of carbide particles and decohesion at the phase interfaces occur, also.

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