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CHANGES TO THE FRACTURE BEHAVIOUR OF MEDIUM-ALLOYED LEDEBURITIC TOOL STEEL AFTER PLASMA NITRIDING

SPREMEMBE V NAČINU PRELOMA SREDNJE LEGIRANEGA LEDEBURITNEGA JEKLA ZARADI PLAZEMSKEGA NITRIRANJA

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Three-point test specimens made from VANADIS 4 Extra cold-work steel were heat treated using two basic regimes and a different hardness was obtained in each case. The cross-section of the specimens was 10 mm × 10 mm. Plasma nitriding was carried out using various combinations of temperature, processing time and atmosphere. Fracture-toughness tests using the method of static three-point bending showed the dominant role of the presence of a nitrided layer on both the bending strength and the fracture mechanism. Only if the material was not plasma nitrided did the role of the austenitizing temperature become clear, and in this case the higher the temperature, the lower the bending strength. The initiation and the propagation of the fracture were low-energy ductile for the steel that was hardened and tempered. The presence of the plasma-nitrided region on the surface changed the initiation as well as the propagation mechanism to that of cleavage. The thickness of the cleavage region increased as the nitrided region became thicker, which additionally lowered the bending strength.

Key words: Vanadis 4 cold work steel, heat treatment, plasma nitriding, three-point bending strength, fracture surface

Tritočkovni preizkušanci iz jekla Vanadis 4 Ekstra za hladna orodja so bili toplotno obdelani na dva osnovna načina na različno trdoto. Prerez preizkušancev je bil 10 mm × 10 mm. Nitriranje v plazmi je bilo izvršeno z različno kombinacijo temperature, procesiranja in atmosfere. Preizkusi žilavosti loma po metodi tritočkovnega upogiba so pokazali dominantno vlogo nitrirane plasti na upogibno trdnost in na mehanizem preloma, le pri jeklu, ki ni bilo nitrirano, je prišel do izraza vpliv temperature: čim višja je bila temperatura, tem nižja je bila upogibna trdnost. Začetek in propagacija preloma sta bila duktilna-maloenergijska pri kaljenem in popuščenem jeklu. Zaradi nitrirane plasti sta se spremenila začetek in propagacija razpoke v cepljenje. Debelina cepilne plasti je bila večja pri večji debelini nitrirane plasti, kar je dodatno zmanjšalo upogibno trdnost.

Ključne besede: jeklo Vanadis za hladna orodja, toplotna obdelava, nitriranje, tritočkovni upogib, površina preloma

1 GENERAL REMARKS

The ledeburitic steels made via the powder metallurgy (P/M) technique have a considerably finer and much more isotropic microstructure than materials with the same chemical composition produced by the conventional ingot-fabrication route. The favourable structural parameters are reflected in the fracture toughness, which is several times greater than that of conventionally produced steels. To improve the surface hardness and to increase the wear resistance, the steels are nitrided, PVD- or CVD-layered or duplex-coated. The occurrence of surface layers formed by various diffusion processes affects the mechanical properties, markedly improving the wear resistance, hardness, and in many cases also the fatigue strength; however, these layers also lower the fracture toughness. Nevertheless, it is very important to determine exactly the extent of the lowering of the fracture toughness, since this property is a very important parameter for the end-user of the tools.

The powder metallurgy of rapidly solidified particles, which is a common name for the production of the group of materials with an excellent combination of microstructure and properties, is a rapidly expanding area in metallurgy. Many newly developed materials are introduced to industry every year. For these materials, the understanding of their behaviour under the condition of surface layering is of essential importance. In this paper, the results of an investigation of fracture behaviour for the newly developed cold-work steel Vanadis 4 Extra processed with plasma nitriding are presented and discussed.

2 EXPERIMENTAL

Specimens of the steel Vanadis 4 Extra (1.37 % C, 0.43 % Si, 0.38 % Mn, 4.66 % Cr, 3.47 % Mo, 3.65 % V, 0.08 % Cu, Fe bal.) were heat treated (hardened and tempered) in a vacuum furnace to different hardnesses, **Table 1**. Next, the specimens were plasma nitrided in a RUBIG – Micropuls plasma furnace using various combinations of temperature and dwell time (**Table 1**).

The fracture toughness was determined with a three-point bending test, with a distance between the supports of 80 mm. The specimens were loaded at the central point with a loading speed of 1 mm/min up to fracture.

The nitrided layers were investigated using light microscopy (the thickness of the diffusion layer), microhardness tests (depth profiles of the microhardness, Nht*), a WDX analyser (concentration depth profiles), X-ray diffraction (phase constitution of the surface). Scanning electron microscopy was used for the examination of the surface of the fractures.

Table 1: Nitriding of the specimens
Tabela 1: Nitriranje preizkušancev

Specimen set	Hardness HRC	Plasma nitriding
1–4	57	470 °C/30 min/N_2:H_2 = 1:3, 500 °C/60 min/N_2:H_2 = 1:3, 530 °C/120 min/N_2:H_2 = 1:3, 470 °C/30 min + 470 °C/75 min, N_2:H_2 = 1:10
5–8	60	470 °C/30 min/N_2:H_2 = 1:3, 500 °C/60 min/N_2:H_2 = 1:3, 530 °C/120 min/N_2:H_2 = 1:3, 470 °C/30 min + 470 °C/75 min, N_2:H_2 = 1:10

3 RESULTS AND THEIR DISCUSSION

The microstructure of the substrate steel after quenching and tempering to a hardness of *HRC* 57 is shown in **Figure 1**. It consists of a martensitic matrix and fine (several microns) globular carbide particles (Figure 1). The microstructure of the steel processed to a hardness of 60 HRC is similar to that with the hardness of *HRC* 57 (**Figure 2**).

The nitrided region differs from the substrate strongly in terms of etching sensitivity due to the nitride precipitates formed in the near-surface layer; this is typical for all nitrided ledeburitic steels. For the steel processed at a low temperature, the nitrided layer is free of a compound sub-layer (**Figure 3**). For the materials processed at a higher temperature and/or for a longer

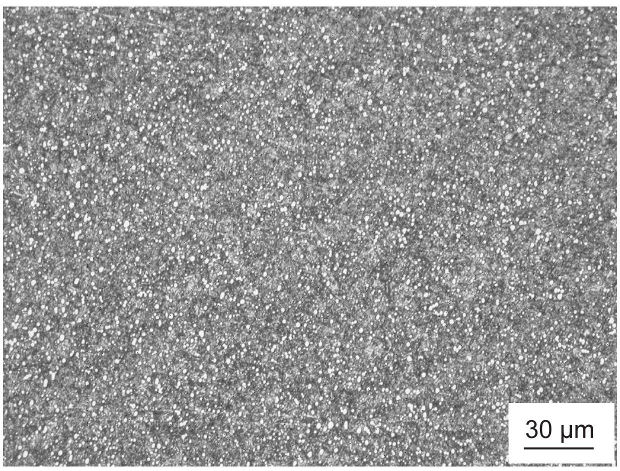

Figure 1: Microstructure of the steel heat treated to *HRC* 57
Slika 1: Mikrostruktura jekla, ki je bilo toplotno obdelano na trdoto *HRC* 57

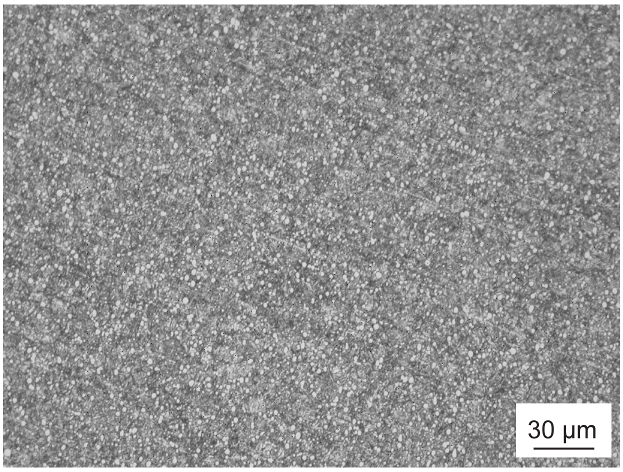

Figure 2: Microstructure of the steel heat treated to *HRC* 60
Slika 2: Mikrostruktura jekla, ki je bilo toplotno obdelano na trdoto *HRC* 60

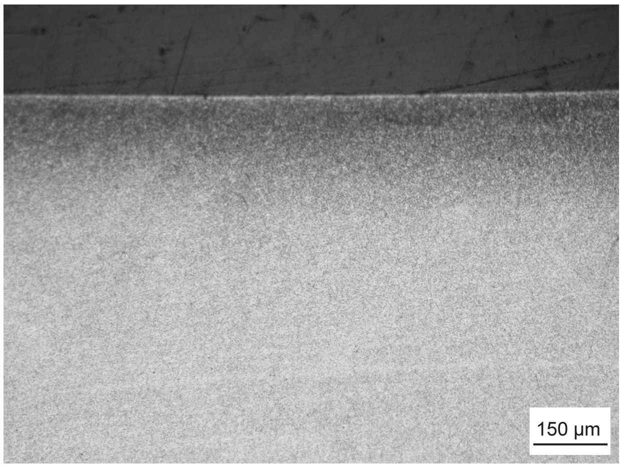

Figure 3: Microstructure of the steel heat treated to *HRC* 57 and nitrided at 470 °C for 30 min
Slika 3: Mikrostruktura jekla, ki je bilo toplotno obdelano na *HRC* 57 in nitrirano 30 min pri 470 °C

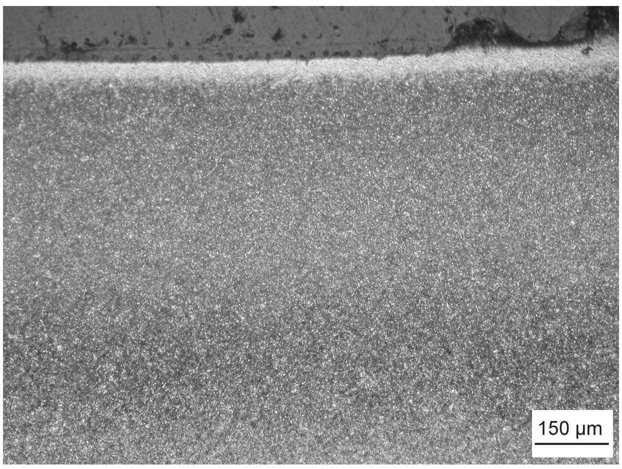

Figure 4: Microstructure of the steel heat treated to *HRC* 57 and nitrided at 530 °C for 120 min
Slika 4: Mikrostruktura jekla, ki je bilo toplotno obdelano na *HRC* 57 in nitrirano 120 min pri 530 °C

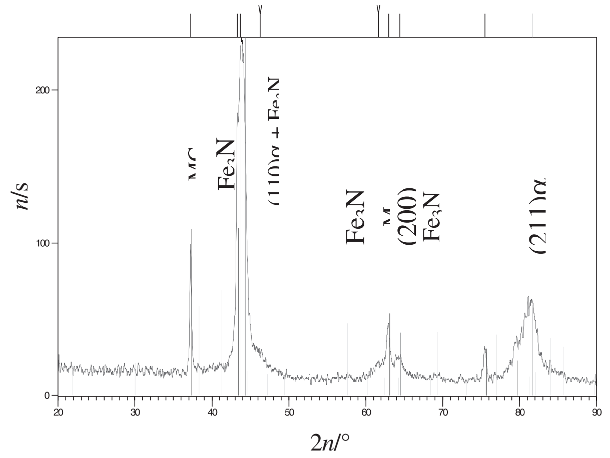

Figure 5: X-ray patterns of the steel, nitrided at 470 °C for 30 min
Slika 5: Rentgenski spekter jekla, ki je bilo nitrirano 30 min pri 470 °C

time, a compound "white" layer is also obtained (**Figure 4**).

The X-ray diffraction spectra show no presence of a compound layer on the surface of the specimens processed at 470 °C for 30 min. The surface microstructure consists of martensite and 13 % is the ε-nitride (**Figure 5**). On the other hand, up to 70 % of the ε-nitride was found in the case of the specimen processed at 530 °C for 120 min. This definitely indicates the presence of a compound layer with a thickness of several μm (**Figure 6**).

The input of nitrogen into the surface induces a considerable surface hardness increase. The hardness is also increased below the surface and the thickness of the region with elevated hardness is related to the diffusion depth of nitrogen. The initial hardness of the material does not have any substantial effect on the surface hardness, but influences slightly the depth of the nitrided region, according to the criterion: core hardness $HV_{0.05}$ = 50 (**Figures 7 and 8**).

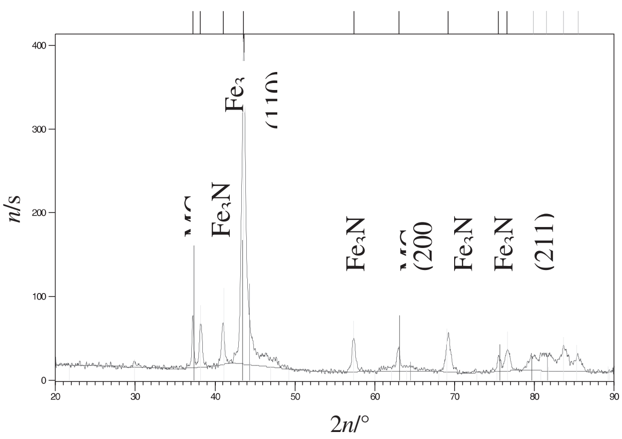

Figure 6: X-ray patterns of the steel, nitrided at 530 °C for 120 min
Slika 6: Rentgenski spekter jekla, ki je bilo nitrirano 120 min pri 530 °C

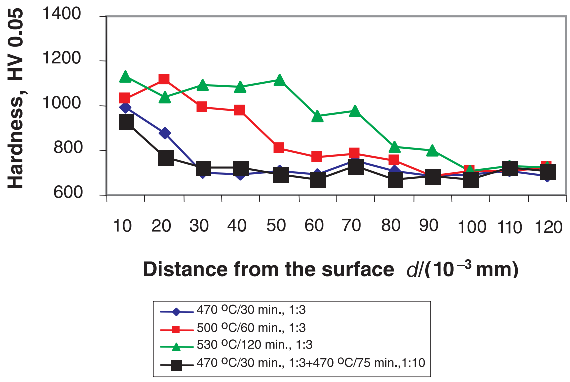

Figure 7: Hardness depth profiles of the nitrided steel with a core hardness of *HRC* 57
Slika 7: Globinski profil trdote nitriranega jekla s trdoto jekla *HRC* 57

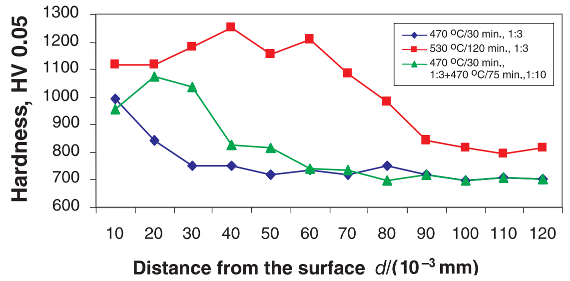

Figure 8: Hardness depth profiles of the nitrided steel with a core hardness of *HRC* 60
Slika 8: Globinski profil trdote nitriranega jekla s trdoto jekla *HRC* 60

Figure 9 shows how the bending strength changes when the core hardness, nitriding temperature and the processing dwell time are increased. It is evident that the austenitising temperature, resulting in a different core hardness, is a relevant factor influencing the three-point bending strength only when the steel is not nitrided. In the nitrided steel, the presence of the nitrided layer by itself lowers the bending strength considerably and the austenitizing temperature does not play a significant role. The bending strength is decreased if the thickness of the nitrided region is increased. It is also important that the diffusion annealing in a nitrogen-poor atmosphere (the

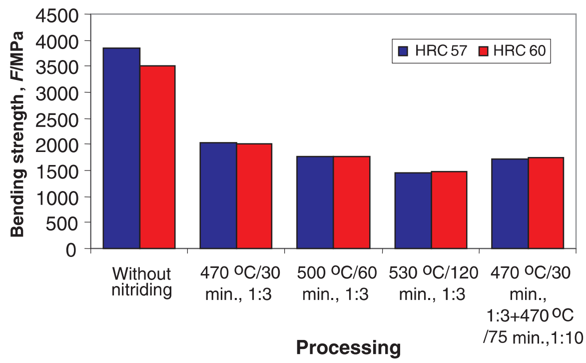

Figure 9: Bending strength as a function of nitriding parameters and core hardness
Slika 9: Upogibna trdnost pri različnih parametrih nitriranja in trdoti jekla

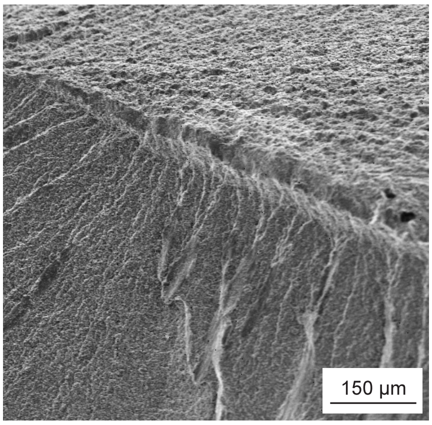

Figure 10: Fracture surface of un-nitrided specimen, processed to *HRC* 57
Slika 10: Prelomna površina preizkušanca s trdoto *HRC* 57, ki ni bil nitriran

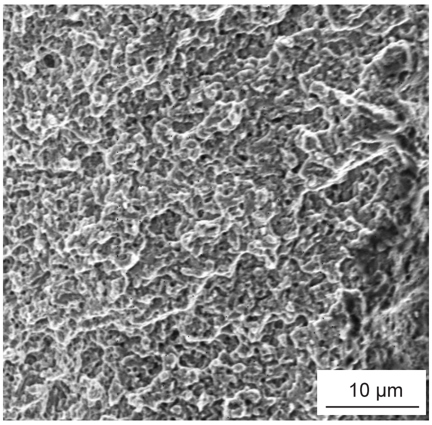

Figure 11: Fracture surface of the specimen in **Figure 10**
Slika 11: Prelomna površina preizkušanca s **slike 10**, detail

last two columns) does not lead to an improvement in the bending strength.

The bending strength of the non-nitrided Vanadis 4 Extra steel is higher than that of Vanadis 6 and comparable with that of M2-type steel [5]. The nature of the difference was not explained so far; however, it can be assumed that Vanadis 4 Extra differs from Vanadis 6 in the molybdenum content and that molybdenum nitride particles can affect the bending strength in an undesirable way. Nevertheless, the M2-type steel also contains molybdenum and the bending strength of the nitrided material remained much higher. Further and more detailed investigations are needed to make a more reliable conclusion about the cause of the change in the bending strength after nitriding.

The fractographical analysis was designed to investigate the fracture initiation and propagation for specimens with and without a nitrided region of different thickness.

In all of the specimens the fracture is initiated on the tensile strained side, in several centres, and propagated in the specimen (**Figure 10**). The crack propagation is different for the non-nitrided and nitrided samples. In the case of the non-nitrided material, the propagation of the crack occurs with the de-cohesion at the carbide-matrix interface and the fracture surface exhibits a shallow dimpled morphology (**Figure 11**). The crack propagation does not consume a large amount of energy, since the dimples are relatively flat and the plastically deformed volume of steel is not large. For this reason, this type of fracture is low-energy transcrystalline. Similarly, as for steel Vanadis 6, a lower austenitizing temperature did not change the mechanism of the failure and only some secondary cracks were observed at the surface [5].

The steel austenitized at a lower temperature had a higher fracture toughness because of its smaller grain size. The austenite grain size increased with the temperature and the products of the austenite decomposition (like martensite) were coarser, too. These phenomena are well known as the limiting ones for the fracture toughness and can explain the obtained results of the three-point bending strength.

The mechanism of fracture initiation in the case of the nitrided specimens differs a great deal from that of the non-nitrided specimens. The fracture clearly exhibits the characteristics of transcrystalline cleavage (**Figure 12**) with the thickness of the cleavage layer corresponding to that of the nitrided region. At higher magnification, small steps are visible on the cleavage facets, indicating the microcracks' propagation at different levels of the same lattice plane. The investigations on various ledeburitic steels showed that the microstructure of the nitrided steel consisted of martensitic platelets containing nitrogen, and ultra-fine nitride particles [6]. Coarser nitride particles are broken during the propagation of the crack and can act as nuclei for the crack re-initiation. In the SEM micrograph in **Figure 14**, the case of a brittle particle with spokewise cracks propagating in the surrounding area is shown. The steps on the cleavage facets can be related to the platelet shape of martensite. **Figure 15** shows that in the core

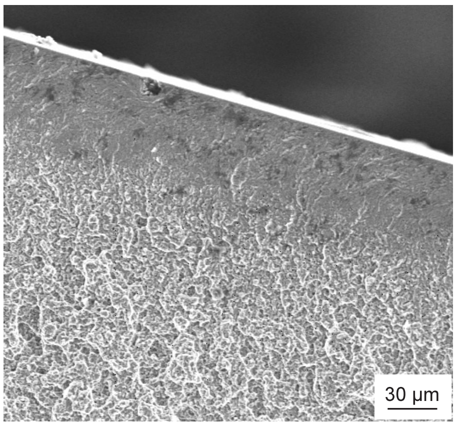

Figure 12: Fracture surface of specimen nitrided at 530 °C for 120 min
Slika 12: Prelomna površina preizkušanca, ki je bil nitriran 120 min pri 530 °C

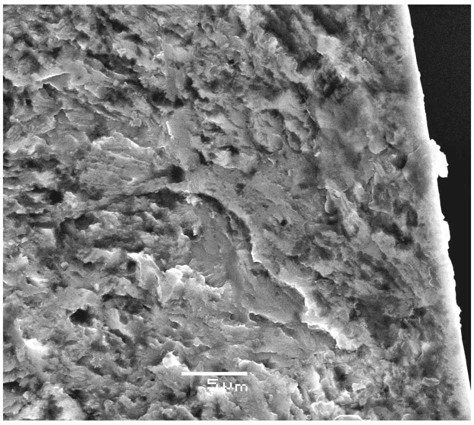

Figure 13: Fracture surface of the specimen in **Figure 12**
Slika 13: Prelomna površina preizkušanca s **slike 12**, detail

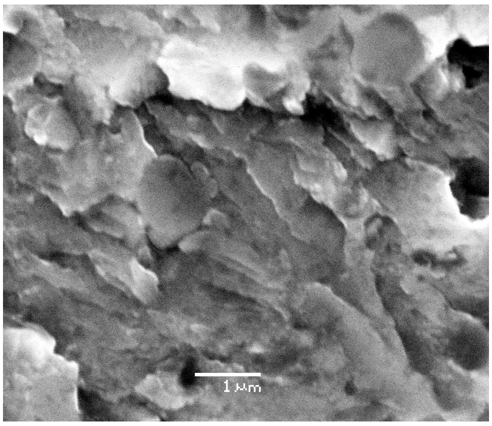

Figure 14: Fracture surface of the specimen in **Figure 12**, detail of cleavage facets
Slika 14: Prelomna površina preizkušanca s **slike 12**, detail s cepilnimi facetami

material, the fracture propagates again according to the trancrystalline low-energy ductile mechanism with de-cohesion at the carbide-matrix interface and a small plastic deformation.

The lowering of the bending strength after the plasma nitriding is due to the fact that cleavage crack propagation requires only a negligible plastic deformation. All of the energy input into the material is spent only for the formation of two new surfaces. This is a different, when compared to the non-nitrided material, where a low plastic deformation occurs throughout the specimens and, as a consequence, the three-point strength was considerably higher. The lowering of the fracture toughness at an increased nitriding temperature and/or time can be explained by the fact that the area of cleavage of the total cross-sectional area is greater due to the thickness of the nitrided layer.

Based on the results presented in this work as well as in the papers published previously [5,6] it seems that the lowering of the bending strength, and fracture toughness in general, due to the occurrence of a nitrided region of the surface, is a systematic phenomenon and cannot be

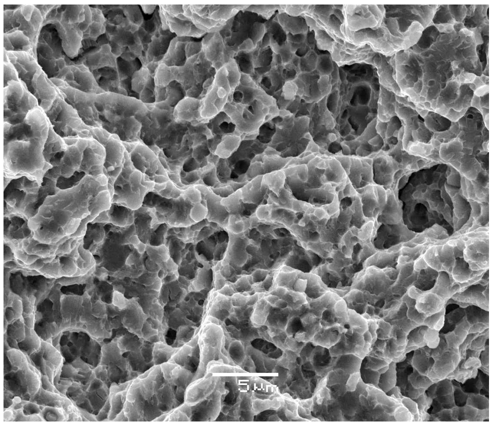

Figure 15: Fracture surface of the specimen in **Figure 12**, core steel
Slika 15: Prelomna površina preizkušanca s **slike 12**, jedro preizkušanca

avoided completely. On the other hand, the nitriding brings several beneficial effects to the materials and components, such as an increase in the fatigue lifetime of the specimens and tools, and an improvement in the wear resistance, corrosion resistance, adhesion of thin PVD layers, etc. Therefore, the nitriding will be required from industrial producers and/or users of tools. It is, therefore, necessary to minimize the lowering of the fracture toughness, through the optimization of the nitriding process.

4 CONCLUDING REMARKS

- In the case of the non-nitrided steel, the austenitizing temperature has an important influence on the fracture behaviour. The three-point bending strength decreases as the austenitizing temperature increases because of the grain coarsening at the higher austenitizing temperature.
- The main mechanism of the fracture initiation is the nucleation of dimples at the carbide-matrix interface in the case of non-nitrided specimens. The fracture propagation is ductile and low energy.
- The presence of the plasma-nitrided layer at the surface lowers significantly the bending strength. The thicker is the nitrided layer the lower is the fracture toughness, since the cleavage region, where a small amount of energy is spent for the crack propagation, in greater with a thicker nitrided region.
- The lowering of the bending strength for the steel Vanadis 4 Extra is more remarkable than that for the steels Vanadis 6 and M2, processed in the same nitriding conditions.
- Transcrystalline cleavage was found to be the main mechanism of crack propagation in the case of nitrided layers. The thickness of the cleavage regions corresponds well with the thickness of the nitrided regions determined by metallographic methods.

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