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CHANGES TO THE FRACTURE BEHAVIOUR OF THE Cr-V LEDEBURITIC STEEL VANADIS 6 AS A RESULT OF PLASMA NITRIDING

SPREMEMBA NARAVE LOMA LEDEBURITNEGA JEKLA Cr-V VANADIS 6, NITRIRANEGA V PLAZMI

František Hnilica[1], Josef Čmakal[2], Peter Jurči[3]

[1]Czech Technical University, Faculty of Mechanical Engineering, Karlovo nám. 2, 121 35 Prague 2, Czech Republic
[2]UJP PRAHA, a. s., Nad Kamínkou 1345, 156 10 Prague–Zbraslav, Czech Republic
[3]ECOSOND, s. r. o., Křížová 1018, 150 21 Prague 5, Czech Republic
hnilica@fsik.cvut.cz

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Three-point test specimens made from VANADIS 6 ledeburitic steel were heat treated using two basic regimes to produce different hardness values. The cross-sections of the specimens ranged between (10×10) mm and (1×10) mm – this range was chosen in order to evaluate the influence of the portion of nitrided material on the fracture toughness. The plasma nitriding was carried out for various combinations of temperature and dwell time. The testing of the fracture toughness with static three-point bending established the dominant role of the presence of the nitrided layer on both the bending strength and the fracture mechanism. Only if the material was not plasma nitrided was the role of the austenitizing temperature clearly shown – the higher the temperature, the worse the three-point bending strength. The character of the initiation and propagation of the fracture was low-energy ductile when the material 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 induced an additional lowering of the bending strength.

Key words: ledeburitic steel, plasma nitriding, fracture toughness, crack propagation

Žilavostni vzorci, izdelani iz ledeburitnega jekla VANADIS 6 so bili toplotno obdelani po dveh režimih, ki sta zagotovila različno trdoto. Prerez vzorcev je bil razvrščen v področja od (10 × 10) mm do (1 × 10) mm, da bi ugotovili vpliv deleža nitriranega materiala na lomno žilavost. Nitriranje v plazmi je bilo izvršeno pri različnih kombinacijah temperature in časa nitriranja. Preizkus lomne žilavosti z metodo tri točkovnega upogibanja je pokazal prevladujoč vpliv prisotnosti nitrirane plasti na upogibno trdnost in mehanizem preloma. Samo pri materialu, ki ni bil nitriran v plazmi, se je pokazal vpliv temperature avstenitizacije – čim višja je bila temperatura, tem nižja je bila upogibna trdnost. Ko je bil material kaljen in popuščan, je bila iniciacija in propagacija razpoke značilno nizkoenergijska, duktilna. Prisotnost področja, nitriranega v plazmi, na površini, je spremenila iniciacijo kot tudi mehanizem propagacije, ki je postal cepilen. Debelina cepilnega področja narašča z naraščanjem debeline nitriranega področja, to pa povzroči dodatno zmanjšanje upogibne trdnosti.

Ključne besede: ledeburitno jeklo, nitriranje v plazmi, udarna žilavost, napredovanje razpoke

1 GENERAL REMARKS

Ledeburitic steels made via a powder-metallurgy (P/M) route have many advantages over steels with the same chemical composition that are produced by a conventional ingot-fabrication route. This relates particularly to their mechanical properties and to their isotropy. P/M-made materials have a many-times refined microstructure, and do not contain any carbide clusters and/or bands that could lead to an embrittlement of the material. As a result, P/M ledeburitic steels are very important in applications where a high fracture toughness in conditions of dynamic loading is required.

In some cases, however, an additional surface improvement is required – mostly with respect to the hardness increase, wear resistance and lowering of the friction coefficient. The application of various PVD methods, in combination with plasma nitriding, has been successfully applied in order to meet these requirements [1,2]. What should be taken into account, however, is that the plasma nitriding leads to the presence of hard layers on the surface. These layers have an elevated hardness, improved wear resistance and fatigue life, on the one hand, but can influence the fracture toughness in an undesirable manner, on the other. Therefore, the main goal of this research was to assess what happens on the surface if the tool material is plasma nitrided, from the point of view of fracture mechanics, fracture initiation and propagation. The Vanadis 6 ledeburitic steel, which is known as a promising tool material, was chosen as an appropriate material for the experimental studies.

2 EXPERIMENTAL

Specimens with various cross-sections, and with a length of 100 mm, made from the ledeburitic VANADIS 6 steel (2.1% C, 7% Cr, 6% V, Fe bal.) were heat treated (hardened and tempered) in a vacuum furnace to the resulting hardness values given in Table 1. Next, the specimens were plasma nitrided in RUBIG–Micropuls plasma equipment. Various combinations of temperature and dwell time were used, as listed in **Table 1**.

Table 1: List of processings applied for the treatment of specimens
Tabela 1: Spisek uporabljenih procesnih parametrov pri pripravi vzorcev

Set of specimens	Dimensions (mm)	Heat treatment / plasma nitriding
1	1×10×100	1000 °C/30 min + 2 × 550 °C/1 h, 57 HRC, 470 °C/30 min, 500 °C/60 min, 530 °C/120 min
2	1×10×100	1050 °C/30 min + 2 × 550 °C/1 h, 60 HRC, 470 °C/30 min, 500 °C/60 min, 530 °C/120 min
3	3×10×100	1000 °C/30 min + 2 × 550 °C/1 h, 57 HRC, 470 °C/30 min, 500 °C/60 min, 530 °C/120 min
4	3×10×100	1050 °C/30 min + 2 × 550 °C/1 h, 60 HRC, 470 °C/30 min, 500 °C/60 min, 530 °C/120 min
5	10×10×100	1000 °C/30 min + 2 × 550 °C/1 h, 57 HRC 470 °C/30 min, 500 °C/60 min, 530 °C/120 min
6	10×10×100	1050 °C/30 min + 2 × 550 °C/1 h, 60 HRC, 470 °C/30 min, 500 °C/60 min, 530 °C/120 min

The testing procedure was carried out using the three-point bending-test method. The distance between the supports was 80 mm. The specimens were loaded in the central region, at a loading speed of 1 mm/min, up to the moment of the fracture.

The main characteristics of the nitrided layers were established using an optical microscope (the thickness of the diffusion layer), microhardness tester (depth profiles of the microhardness, *Nht**), WDX analyser (concentration depth profiles). A SEM was used for the analysis of the fracture surfaces.

3 RESULTS AND DISCUSSION

A detailed study of the substrate material and the surface layers has been published elsewhere [3,4], and for this reason only the basic information is presented here. The substrate material consists of the matrix and carbides. A TEM investigation showed that the matrix is formed by both the dislocations and the twin martensite. Two types of carbides – M_7C_3 and MC – were found. Previous papers have also shown that the development of the phase constitution in nitrided regions corresponds well with the iron–nitrogen equilibrium diagram. During the first stage of the nitriding only the saturation of the martensite proceeds and, after the solid solubility limit of the nitrogen in the martensite is reached, disperse nitrides are formed. If a higher temperature and/or longer dwell time are applied, then also a thin, discontinuous network was apparently shown close the surface. The formation of the described structural features can be related to the level of the nitrogen saturation of the surface, whereas a close relationship between the surface nitrogen concentration, the phase constitution and the appearance of nitrogen-rich structural constituents was observed. In addition, the plasma nitriding resulted in a considerable increase in surface hardness.

Figure 1 demonstrates how the bending strength changes as the nitriding temperature and the processing dwell time increase. What is also evident is that the austenitising temperature is a relevant factor influencing the three-point bending strength. This was mainly evident if the material was not nitrided. In other cases the presence of the plasma-nitrided layer itself is the main factor influencing the fracture toughness, and the effect of the austenitizing temperature is less significant. The fracture toughness decreases again as the thickness of the nitrided layer increases. However, the decrease of

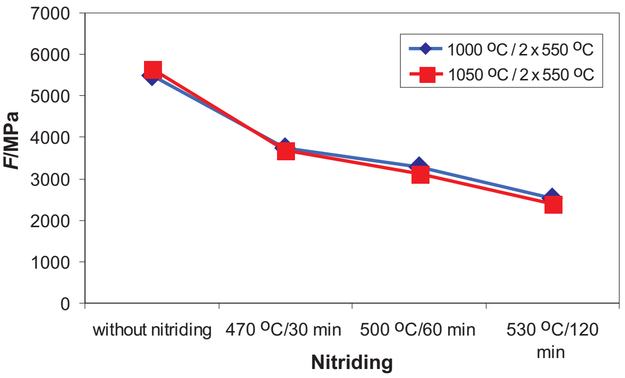

Figure 1: Three-point bending strength *F* as a function of the austenitizing temperature and parameters of the nitriding: specimens (10 × 10 × 100) mm
Slika 1: Upogibna trdnost *F* kot funkcija temperature avstenitizacije in parametrov nitriranja, vzorci (10 × 10 × 100) mm

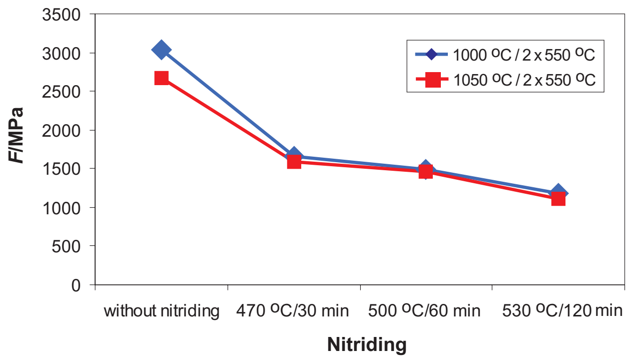

Figure 2: Three-point bending strength *F* as a function of the austenitizing temperature and parameters of the nitriding: specimens (3 × 10 × 100) mm
Slika 2: Upogibna trdnost *F* kot funkcija temperature avstenitizacije in parametrov nitriranja, vzorci (3 × 10 × 100) mm

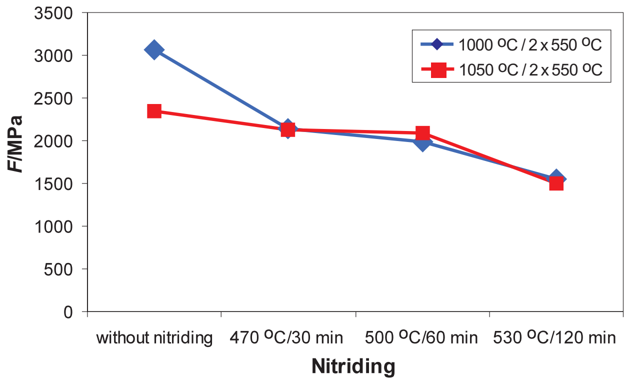

Figure 3: Three-point bending strength *F* as a function of the austenitizing temperature and parameters of the nitriding: specimens (1 × 10 × 100) mm

Slika 3: Upogibna trdnost *F* kot funkcija temperature avstenitizacije in parametrov nitriranja, vzorci (1 × 10 × 100) mm

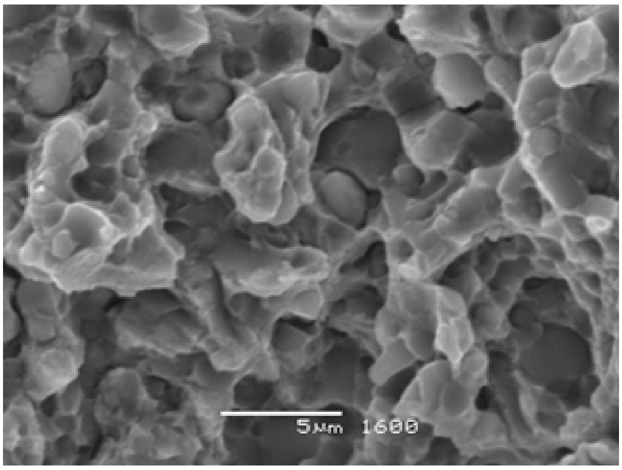

Figure 5: SEM micrograph showing a detail from **Figure 4**.

Slika 5: SEM-posnetek, ki prikazuje detajl s **slike 4**

the bending-strength toughness caused by the thickening of the nitrided regions is not as clear as that induced only by the presence of the nitrided layer itself.

The difference in the fracture toughness of the non-nitrided samples, generated by different austenitizing temperatures, is reduced as the cross-section of the specimens decreases, see **Figures 2, 3**. Concerning the influence of the nitrided layer, no differences between the specimens with various cross-sections were found; compare **Figure 1 to Figures 2 and 3**.

The fractographical observations were oriented mainly to an investigation of fracture initiation and propagation in the specimens with and without the presence of the nitrided layers of a different thickness. The effect of the different austenitising temperature was also investigated.

In all of the specimens with no nitrided layer the fracture surface exhibits clear symptoms of a so-called low-energy ductile fracture, with a dimple morphology on the surface. **Figure 4** demonstrates the fracture surface of the specimen austenitized at 1050 °C. As shown, it appears that the initiation of the fracture is realised through a decohesion at the carbide–matrix interfaces, as well as by the cracking of carbides. These processes proceed mainly at the surface, in several centres, and on the tensile-strained side, as demonstrated in the SEM micrograph in **Figure 5**. One difference between the specimens austenitized at different temperatures (and having a different hardness) from the macroscopical point of view is that the fracture surface of the specimen quenched from the lower temperature has a lot of secondary cracks, **Figure 6**. However, from the microscopical point of view, these differences could hardly be observed.

The reason why the material austenitized at a lower temperature has a higher fracture toughness can be considered as natural. It is commonly known that the austenitic grain size increases as the temperature is

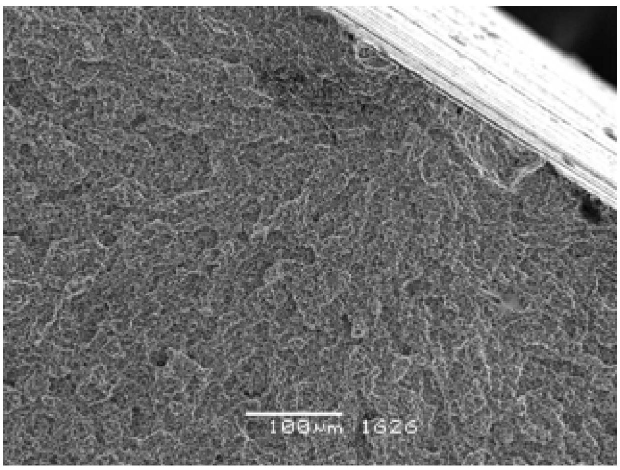

Figure 4: SEM micrograph showing the fracture surface of the non-nitrided specimen austenitized at 1050 °C

Slika 4: SEM-posnetek površine preloma nenitriranega vzorca; avstenitizacija pri 1050 °C

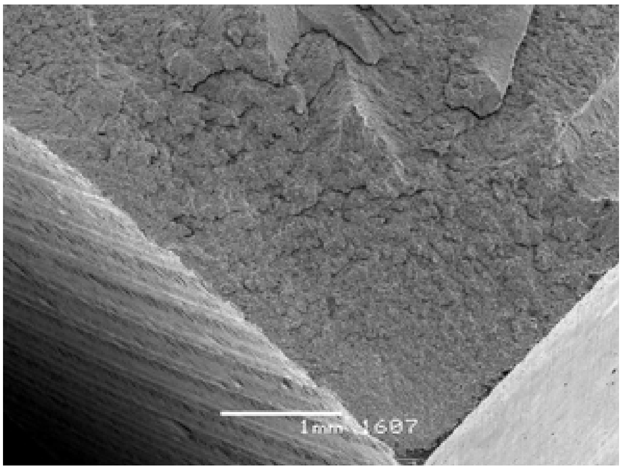

Figure 6: SEM micrograph showing the fracture surface of the non-nitrided specimen austenitized at 1000 °C

Slika 6: SEM-posnetek površine preloma nenitriranega vzorca; avstenitizacija pri 1000 °C

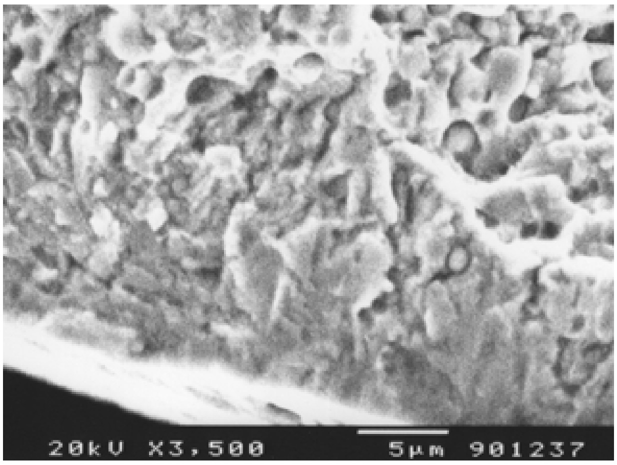

Figure 7: SEM micrograph showing a cleavage fracture of the nitrided layer, developed at 470 °C for 30 min
Slika 7: SEM-posnetek cepilnega loma nitrirane plasti, po 30-minutnem nitriranju pri 470 °C

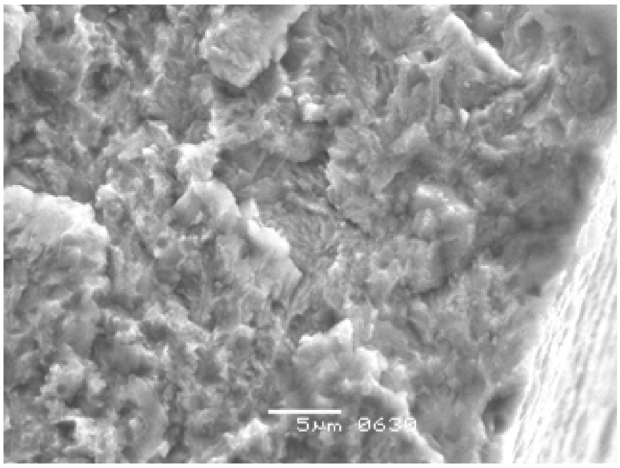

Figure 9: SEM micrograph showing a detail from **Figure 8**, close to the surface
Slika 9: SEM-posnetek, detajl s **slike 8** blizu površine

Table 2: Main parameters of nitrided regions (*the nitriding case depth *Nht* is determined as a core hardness + 50 HV 0.05)
Tabela 2: Glavni parametri nitriranega področja (*globina nitriranja *Nht* je določena kot trdota jedra + 50 HV 0.05)

Nitriding parameters	Surface nitogen content w/%	Nitrogen diffusion depth d/μm	Surface hardness HV 0.05	Nht* /μm
470 °C/30 min.	4.03	13.2	1283	12
500 °C/60 min.	4.8	42	1346	23
530 °C/120 min.	5.78	67	1648	60

raised, and so the products of the decomposition of the austenite (like martensite) can be expected to have a coarser structure. These phenomena are well known as the limiting ones for the fracture toughness and can clarify the measured values of the three-point bending strength.

The mechanism of fracture initiation in the case of nitrided specimens differs clearly from that of the non-nitrided samples. The initiation is, in a way similar to the non-nitrided material, located in a tensile strained face, but the fracture clearly exhibits the characteristics of trans-crystalline cleavage. **Figure 7** shows a SEM micrograph of such a cleavage fracture in the nitrided layer of the smallest thickness; **Figure 8** shows a specimen with the largest thickness. As shown, the thickness of the cleavage region corresponds well with the thickness of the nitrided layer, determined by the microhardness measurement, see **Table 2**.

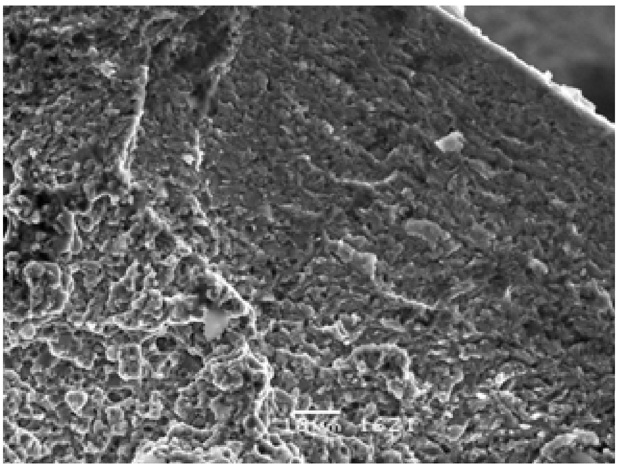

Figure 8: SEM micrograph showing a cleavage fracture of the nitrided layer, developed at 530 °C for 120 min
Slika 8: SEM-posnetek cepilnega loma nitrirane plasti po 120-minutnem nitriranju na 530 °C

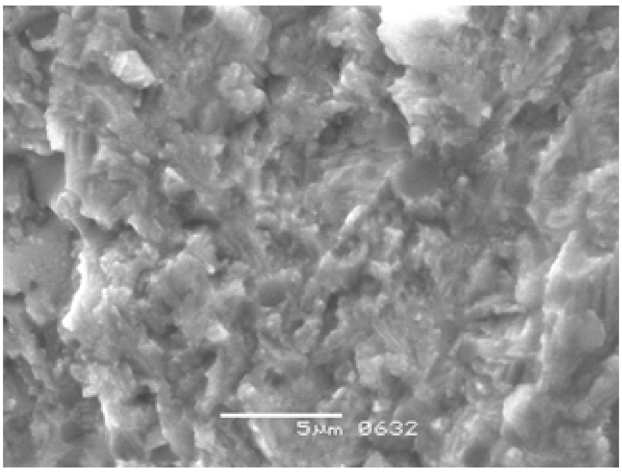

Figure 10: SEM micrograph showing a detail from **Figure 8**, from approximately 25 μm below the surface
Slika 10: SEM-posnetek, detajl s **slike 8**; okrog 25 μm pod površino

Details of the cleavage facets from various places in the nitrided region are shown in **Figures 9 and 10**. The SEM micrograph in **Figure 9** shows the cleavage region at the surface; **Figure 10** shows the cleavage in the central area. The cleavage facets form small steps (**Figure 8**), which is probably connected with the structure of the surface layer. Previous experiments revealed that the layer consists of martensitic needles containing nitrogen and ultra-fine nitride particles. Relatively coarse carbo-nitride particles are broken during the propagation of the crack and they can also serve as the nuclei for crack re-initiation. The SEM micrograph in **Figure 10** shows such a case of brittle particles, around which a crack is propagated in a spoke-like manner.

SEM micrographs showing the fracture surface of the nitrided regions clearly indicate that the cleavage propagation of cracks is connected with 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 difference, compared with the non-nitrided material, where a low plastic deformation of the material was found with all the specimens and, as a consequence, the three-point strength was a lot higher. The lowering of the fracture toughness with an increasing nitriding temperature and/or time can be explained by the fact that the portion of cleavage region from the total cross-sectional area increases as the thickness of the nitrided layer increases.

Although it shows that the presence of a nitrided layer at the surface lowers the fracture toughness of the material, i.e., it acts in an undesirable manner, the nitriding cannot be assessed only from this point of view. Previous experiments confirmed that nitriding increases the fatigue life of the specimens and tools made from P/M ledeburitic steels, which led to a significant prolonging of the life of the tools. Another important aspect is an improvement in the wear resistance, or, in many cases, also an improvement in the adhesion of thin PVD layers developed on the pre-nitrided surface.

4 CONCLUSION

- In the case of non-nitrided material, the austenitizing temperature plays a dominant role in the fracture behaviour. The three-point bending strength reduces as the austenitizing temperature increases.
- Only from macroscopical point of view was a difference found between the specimens quenched from various temperatures. This is based on the number of secondary cracks that appear on the fracture surface of the specimen quenched from the lower temperature.
- The main mechanism of fracture initiation is the nucleation of dimples at the carbides in the case of the non-nitrided specimens. The fracture was propagated in a ductile, low-energy manner, and the surface exhibits a clearly dimpled morphology.
- Even the presence of the plasma-nitrided layer at the surface results in a significant lowering of the bending strength. The thicker the nitrided layer is, the lower the fracture toughness, since the cleavage region, where a little energy is spent for the crack propagation, increases as the nitrided region becomes thicker.

A trans-crystalline cleavage was found as the main mechanism of crack initiation and propagation in the case of nitrided layers. The thickness of the cleavage regions corresponds well with the thickness of the nitrided regions measured by metallographical methods.

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