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DUPLEX COATING OF P/M M2-GRADE LEDEBURITIC STEEL

LEDEBURITNO JEKLO VRSTE P/M M2 Z DUPLEKSNO TRDO ZAŠČITNO PREVLEKO

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P/M M2 tool steel was surface processed using plasma nitriding and duplex coating. Surface characteristics like the microstructure, the hardness, the nitrogen-concentration depth profiles and the phase constitution were investigated. Wear behaviour was tested using the ring-on-plate tribological method with various contact pressures and sliding distances. We determined that the temperature of the plasma nitriding has to be at least 500 °C and that a nitrogen-rich atmosphere has to be used to achieve a sufficient surface strengthening. Structural evaluation revealed good adhesion between the PVD overlay and the nitrided substrate. The effect of PVD TiN coating on the wear resistance was very positive when the specimens were heavily loaded, the wear rate was reduced to approximately 5 % in comparison with the non-coated samples. If a lower load was used then the improvement was not significant. This is due to changes in the wear mechanism when the testing load was increased from 50 to 150 N.

Keywords: P/M tool steels, heat treatment, duplex-coating, structural investigation, wear resistance

Površino orodnega jekla vrste P/M M2 smo utrdili s postopkom plazemskega nitriranja in za tem na nitrirano površino jekla nanesli s PVD-postopkom (angl.: Physical Vapour Deposition) še dupleksno trdo zaščitno prevleko na osnovi TiN. Določili in analizirali smo kemijske, mikrostrukturne in mehanske lastnosti izdelane trde prevleke. Določili smo globinski profil trdote in koncentracije dušika ter fazno sestavo površinsko obdelanega jekla. S tribološkim preizkusom "obroč na plošči" smo ugotavljali odpornost zaščitne prevleke proti obrabi pri različnih stičnih tlakih in drsnih hitrostih. Ugotovili smo, da mora biti temperatura plazemskega nitriranja najmanj 500 °C in da mora biti uporabljena zaščitna atmosfera, bogata z dušikom, če želimo doseči zadovoljivo površinsko utrditev. Mikrostrukturne preiskave so pokazale, da smo dosegli dobro adhezijo med PVD-prevleko in podlago iz nitriranega jekla. Ugotovili smo, da trda dupleksna zaščitna prevleka PVD na osnovi TiN pri velikih obremenitvah zelo ugodno vpliva na odpornost izbranega jekla proti obrabi. Hitrost obrabe jekla s PVD-prevleko se je zmanjšala za približno 5 % v primerjavi z jeklom, ki je imel samo plazemsko nitrirano površino. Pri manjših obremenitvah izboljšanje odpornosti proti obrabi ni bilo tako očitno. Mislimo, da je to zaradi spremembe mehanizma obrabe pri povečanju preizkusne obremenitve s 50 na 150 N.

Ključne besede: P/M-orodna jekla, toplotna obdelava, dupleksne trde zaščitne prevleke, struktura

1 INTRODUCTION

High alloyed ledeburitic steels and high speed steels (HSSs) can be produced by the conventional processing route (casting and hot rolling), as well as by the powder metallurgy (P/M) procedure [1]. The use of P/M made of these steels can provide many benefits to the tool industry. As a result, it is becoming increasingly important to introduce these materials to industrial processes because they are able to extend the lifetime of tools, which means significant costs savings. The cold-working tools made from these materials have to be heat treated before use. The hardening and subsequent tempering procedure increases the hardness usually to 700 - 800 HV, which permits the direct use of tools in the production line.

A post heat treatment such as PVD coating may further improve the quality of the manufactured tools [2]. Nevertheless, the substrate hardness range mentioned above may be insufficient to achieve an acceptable PVD-overlay-substrate interface properties in heavy loaded systems in particular and surface strengthening prior to the PVD is then needed. Plasma nitriding is a convenient pre-treatment method before PVD coating because it allows us to achieve well defined properties for the nitrided layers [3]. Since the processing temperature is lower than that used for the last tempering cycle, the microstructure and the properties of the core material remain practically unaffected. The diffusion inter-layer formed by such a process may give rise to mechanical support for the PVD overlay, due to an increased hardness [4,5]. As reported elsewhere, the adhesion of TiN-PVD coating on nitrided, conventionally produced, HSS was several times better than in the case of a non-nitrided substrate [6].

2 EXPERIMENTAL PROCEDURES

The surface processing was carried out on P/M M2 ledeburitic-type steel (0.9 %C, 6.1 %W, 5 %Mo, 4.14 %Cr, 2.02 %V, Fe bal.), see **Table 1**. Two types of specimens were prepared. The first type were plates of 70 x 18 x 8 mm for wear testing. The structural investigation was carried out on cylinders with a diameter 10 mm and a length of 12 mm. All the specimens were net-shape machined and afterwards subjected to a heat treatment bringing the hardness of 730 HV 10. The specimens intended for wear testing were finished by fine grinding, unless otherwise indicated*. The specimens for structural investigations were finished by polishing.

Table 1: The surface treatment of analysed specimens

Number	Plasma nitriding	PVD
1	500 °C, $N_2 : H_2 = 1:3$, t = 60 min. continuous processing	No
2	500 °C, $N_2 : H_2 = 1:3$, t = 60 min. continuous processing	TiN, 2.4 µm
3	500 °C, $N_2 : H_2 = 1:3$, t = 60 min. pulse processing	No
4	530 °C, $N_2 : H_2 = 1:3$, t = 120 min. pulse processing	No
5	530 °C, $N_2 : H_2 = 1:3$, t = 120 min. pulse processing	TiN, 2.4 µm
6*	500 °C, $N_2 : H_2 = 1:3$, t = 60 min. continuous processing	TiN, 2.4 µm

The structural analyse were realised with the optical and scanning electron microscopy. The phase constitution was investigated by the X-ray diffraction. The surface hardness and microhardness were tested using the Vickers method, with a load of 10 kg (HV 10) for the surface hardness and a load of 50 g (HV 0.05) for the depth profiles. The depth profiles of interstitials were established using VDA analysis. The wear tests were carried out using the ring-on-plate method (linear contact between sample and counterbody) under non-lubricated conditions at room temperature. The ČSN 41 4109 (100 Cr 6) ball-bearing steel processed to a hardness of 60 HRC was used as the counterbody material. The testing procedure was performed using lower (50 N) and higher (150 N) loadings. The total sliding distance was 10 km, however, the wear rate, represented by the weight loss of the specimens [g], was also examined after 1, 2.5 and 5 km.

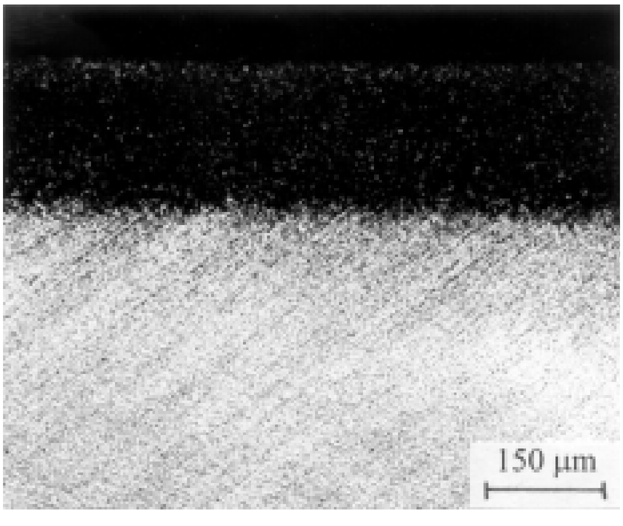

Figure 1: Microstructure of specimens nitrided at 530 °C for 120 min (without the PVD overlay)

Slika 1: Mikrostruktura vzorcev, nitriranih pri 530 °C/120 min (brez PVD-plasti)

3 RESULTS

Figure 1 shows the nitrided layer formed at 530 °C with an atmosphere containing $N_2 : H_2 = 1:3$ for 120 min. The real thickness of the layer is approximately 40 µm, which corresponds well with the recommended thickness for a duplex coating [5]. The layer consists of the metallic matrix, formed by tempered martensite in which fine

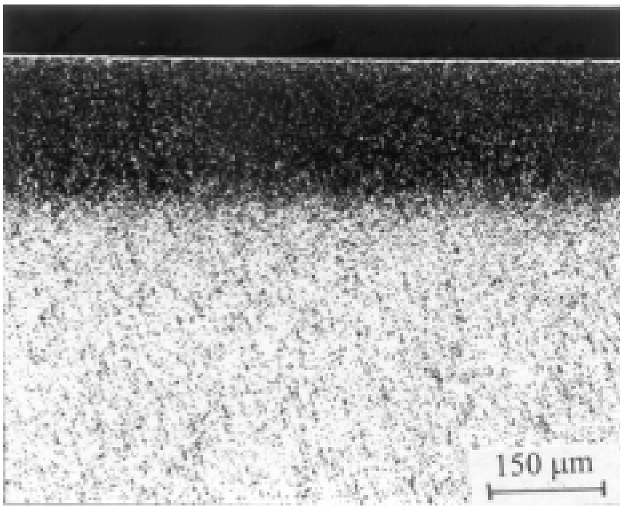

Figure 2: Microstructure of specimens nitrided at 530 °C for 120 min (PVD-layered)

Slika 2: Mikrostruktura vzorcev, nitriranih pri 530 °C/120 min (s PVD-plastjo)

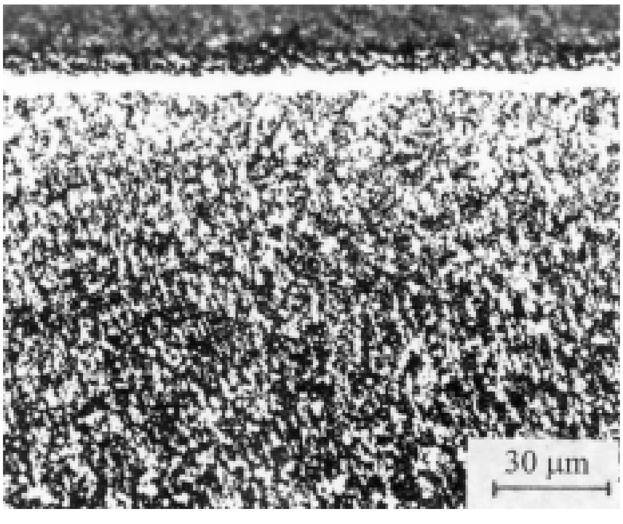

Figure 3: Microstructure of specimens nitrided at 530 °C for 120 min (detail from Figure 2)

Slika 3: Mikrostruktura vzorcev, nitriranih pri 530 °C/120 min (detajl s slike 2) carbides are uniformly distributed, and ultra-fine nitrides. **Figure 2** shows the microstructure of a duplex-coated specimen. At the surface, there is the TiN PVD overlay with a thickness 2.4 μm. The nitrided inter-layer consists of the same features as the layer shown in **Figure 1** because identical processing conditions were applied. The detail micrograph in **Figure 3** shows that inhomogeneities like pores or microcracks are visible neither at the interface nor in the PVD overlay. As a consequence, good adhesion of the overlay to the surface can be expected.

The X-ray diffraction determined the presence of the α solid solution enriched by nitrogen, M(C,N) carbonitride, M_6C carbide and various nitrides in all the specimens. In most parts of the specimens, the Fe_4N phase was observed, however, if a higher surface saturation by the nitrogen was achieved (more than 4 wt %) then the peaks of the Fe_3N nitride also appeared on the diffraction plots. In addition, the TiN phase diffraction peaks were found in the PVD-coated samples.

Figure 4 shows both the nitrogen- and the carbon-concentration depth profiles for the specimen processed at 500 °C, $N_2 : H_2 = 1:3$ for 60 min. The nitrogen content at the surface is 3.3 wt % and it decreases with an increasing distance from the surface. VDA analysis did not reveal nitrogen at distance of more than 9 μm from the surface. The relationship between the carbon content and the distance from the surface behaves in an interesting way: at the surface, the carbon concentration was lower than the nominal alloy composition, however, it increased weakly with increasing distance from the surface. The maximum carbon content was found at the distance below the surface where the nitrogen vanished. Beyond the concentration peak, the carbon content drops down slightly to that of the nominal steel concentration.

Figure 5 gives the concentration depth profiles for the specimen processed at 530 °C, $N_2 : H_2 = 1:3$ for 120 min. It is evident that the processing realised at a higher temperature for a longer time leads to a nitrogen surface saturation up to 5.5 %, and the region with high N content extends to a depth of 11 μm. As expected, this fact promotes a large and easily observable redistribution of carbon atoms. The cause of this carbon redistribution and its effect on the surface properties was published and discussed elsewhere [3].

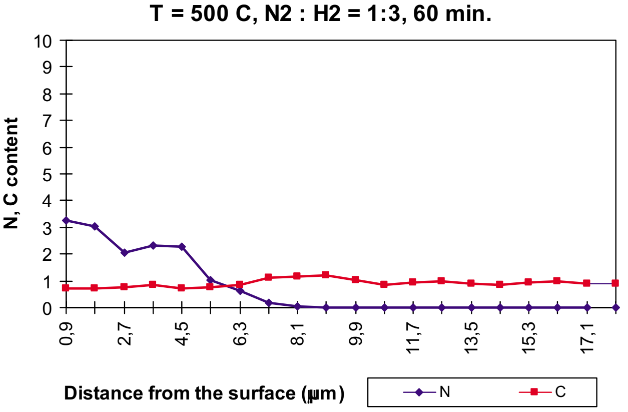

Figure 4: Concentration depth profiles for the specimen nitrided at 500 °C for 60 min

Slika 4: Globinski koncentracijski profil vzorcev, nitriranih pri 500 °C/60 min

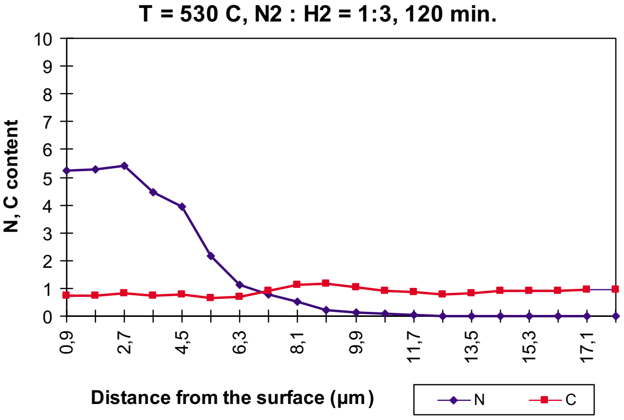

Figure 5: Concentration depth profiles for the specimen nitrided at 530 °C for 120 min

Slika 5: Globinski koncentracijski profil vzorcev, nitriranih pri 530 °C/120 min

Microhardness measurements revealed a high near-surface hardness in the specimens treated at 500 °C and 530 °C, **Figure 6**. Nevertheless, the hardness of the material processed at the lower temperature drops down significantly even at a short distance from the surface. On the other hand, only a slight hardness decrease was observed in the specimen processed at 530 °C for 120 min. These differences in the microhardness depth profiles are also reflected in the surface hardness, which is markedly lower for the specimens processed at 500 °C (1084 HV 10) than for those nitrided at 530 °C (1226 HV 10).

The wear rate differs for significantly for the different sets of nitrided specimens when a loading of 50 N was used for the testing, **Figure 7**. The highest wear

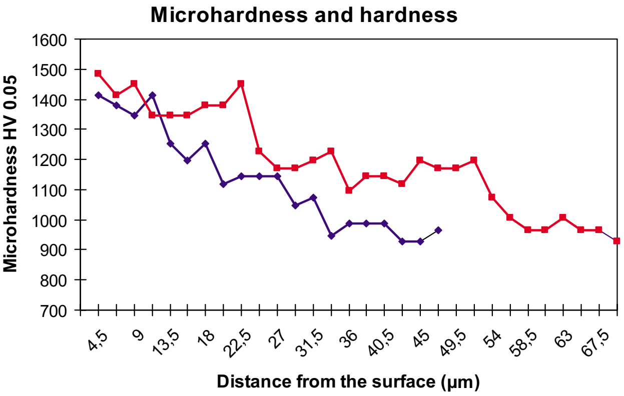

Figure 6: Microhardness depth profiles in nitrided surfaces

Slika 6: Globinski profil mikrotrdote vzorcev v nitriranem sloju

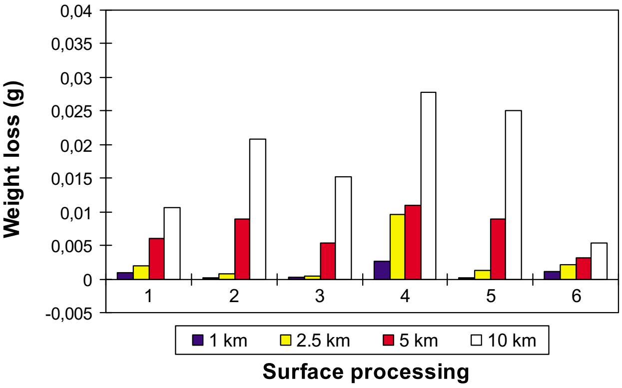

Figure 7: Weight loss as a function of sliding distance and surface processing, testing load of 50 N. Surface processing corresponds to that given in **Table 1**

Slika 7: Izguba mase v odvisnosti od drsne poti in površinske obdelave. Obremenitev vzorca med preizkusom 50 N. Način obdelave površine je podan v **tabeli 1**.

rate was found for the specimens that were pulse processed at 530 °C for 120 min. The specimens processed at 500 °C tended to show better wear resistance which is rather surprising because of their lower surface hardness. Wear testing with a load of 150 N did not reveal significant differences between the specimens that were plasma nitrided under different conditions, **Figure 8**.

At the early stages of testing, using the lower load, the weight losses observed on the PVD-layered specimens were negligible. However, these losses increased significantly after 2.5 km of sliding and the final wear losses were slightly higher than those found for the non-layered samples, **Figure 7**. For the testing with heavier load, the PVD-coated specimens had a much higher wear resistance than those without the PVD layer, **Figure 8**. The best wear resistance was found for polished and duplex-coated specimens.

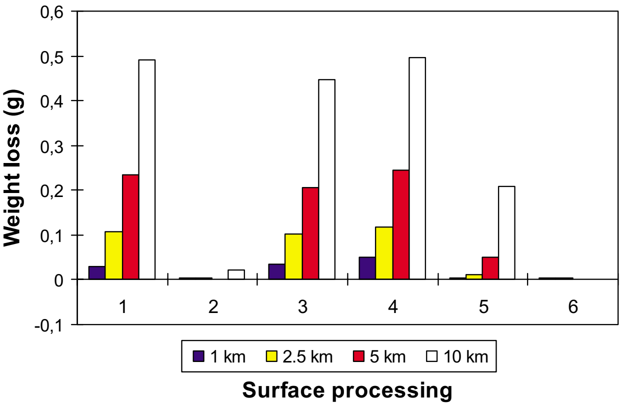

Figure 8: Weight loss as a function of sliding distance and surface processing, testing load of 150 N. Surface processing corresponds to that given in **Table 1**

Slika 8: Izguba mase v odvisnosti od drsne poti in površinske obdelave. Obremenitev vzorca med preizkusom 150 N. Način obdelave površine je podan v **tabeli 1**.

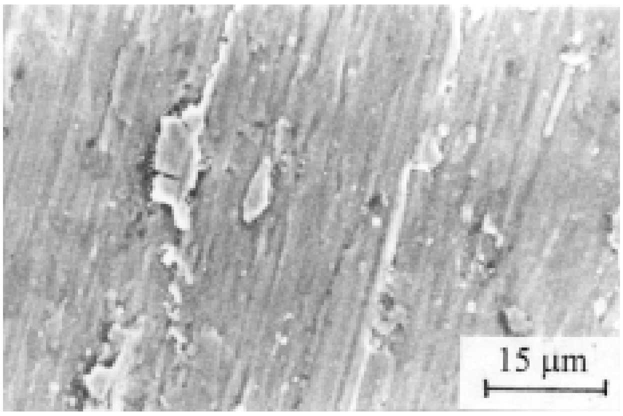

Figure 9: The changes in wear mechanism as a result of increasing testing load, (50 N)

Slika 9: Sprememba mehanizma obrabe kot posledica povečanja obremenitve (50 N)

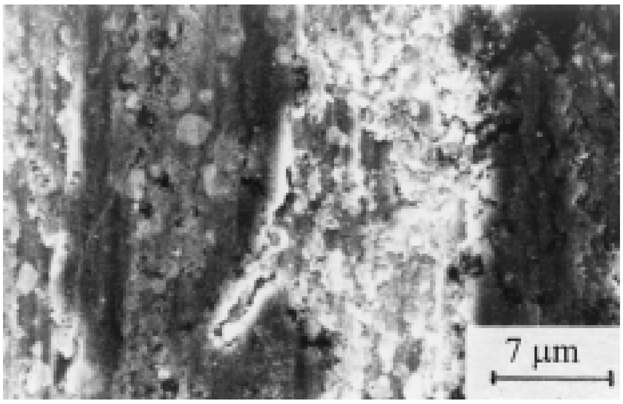

Figure 10: The changes in wear mechanism as a result of increasing testing load, (150 N)

Slika 10: Sprememba mehanizma obrabe kot posledica povečanja obremenitve (150 N)

4 DISCUSSION

These results indicate that the wear rate is not only a function of surface hardness but that the nature of the phases that coexist in the surface layer may also play an important role in the wear behaviour. With lower loads, the coarse, hard particles can easily be removed from the matrix of the specimens and the counterbodies and this may promote an enhanced abrasive wear, see **Figure 9**. If heavier loading is applied these particles are more likely to be crowded into the metallic matrix and the abrasive wear is then restricted in favour of friction, **Figure 10**. The change in the weight loss of counterbodies and a slight reduction in the coefficient of the friction support also such a hypothesis. During low loading the weight loss after 10 km of sliding ranged between 0.14 and 0.19 g, however, this was reduced to less than 50 % when high load was used for the testing. Such a radical difference in the wear rate may also be explained as follows: the wear rate of the PVD-layered samples is lower until the PVD overlay is partly removed from the surface. Subsequently, the weight loss probably does not differ from those measured on non-layered specimens, however, the weight of the PVD overlay (approximately 0.007 g) has to be included in the total weight loss.

5 CONCLUSIONS

a) Plasma nitriding leads to considerable surface strengthening due to the fine nitrides and carbo-nitrides formed in the near-surface region. The surface hardness increase is 350-500 HV 10.
b) TiN overlay has good adhesion to the nitrided surface
c) TiN coating improves the wear resistance of the material, particularly under heavy loading conditions. If less severe testing conditions were used then no improvement in the wear resistance was found, except for the polished specimens which show the best wear resistance for both loadings applied.
d) Changes in the wear mechanism, from abrasion to friction, were found. These changes probably occurred due to increased loading and this is also the principal explanation for the very different wear behaviour of the duplex-coated specimens under various testing conditions.

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