

WEAR MECHANISM OF DUPLEX-COATED P/M VANADIS 6 LEDEBURITIC STEEL

MEHANIZEM OBRABE LEDEBURITNEGA JEKLA P/M VANADIS 6 Z DUPEKSNNO PREVLEKO

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The wear mechanism of duplex-coated P/M Vanadis 6 ledeburitic steel was investigated. Duplex layering by plasma nitriding and PVD CrN coating increased the wear resistance substantially; however, the scattering of the results was too large. The samples differed in the adhesion of the PVD overlay to the nitrided substrate and showed a very different wear resistance and wear mechanism, too. The specimens with good adhesion showed a critical load needed for coating delamination of more than 130 N, low wear, a relatively little damaged PVD layer, and a rapid wear of counterparts. If the adhesion of the coating was smaller, the wear rate increased rapidly and the worn surface showed symptoms of massive cracking and a total collapse of the coating, a strong decrease of the friction coefficient, and of the wear rate of the counterparts.

Key words: ledeburitic P/M steel, duplex coating, wear rate, wear mechanism, coating composition

Raziskan je bil mehanizem obrabe jekla P/M Vanadis 6 z dupleksno prevleko. Dupleksna prevleka iz plazemskega nitriranja in PVD CrNi-prekritja je pomembno povečala odpornost proti obrabi, vendar je bil pri tem prevelik raztros rezultatov. Vzorci so se razlikovali v adheziji med PVD-plastjo in podlago, zato sta bila različna odpornost proti obrabi in mehanizem obrabe. Vzorci z dobro adhezijo so imeli kritično silo za delaminacijo prekritja nad 130 N in relativno majhno obrabo, relativno malo poškodovano PVD-plast in hitro obrabo nasprotnega dela. Pri majhni adheziji prekritja, je hitrost obrabe hitro rastla, obrabljena površina pa je kazala simptome masivnega razpokanja in totalnega kolapsa prekritja, veliko zmanjšanje koeficienta obrabe in hitrosti obrabe nasprotnega dela.

Ključne besede: ledeburitno P/M-jeklo, dupleksno prekritje, hitrost obrabe, mehanizem obrabe, sestava prekritja

1 INTRODUCTION

Ledeburitic steels are widely used in industrial operations like metal cutting, but also in the cold working and sheet-metal forming of large series of parts for the automotive industry. The steels are usually used in the as-heat-treated condition and with a hardness of *HRC* 57–60. Nevertheless, in many cases the heat treatment itself cannot ensure a sufficient service time for the tools and they must be submitted to a surface treatment that also gives them a better resistance to environmental effects.

Thin layers prepared by various PVD (physical vapour deposition) methods are mostly used for the surface treatment of tool steels¹⁻⁶. PVD layers can be deposited over a large range of chemical compositions and mechanical properties, at low temperatures and without any influence on the core properties of the tools. Their common disadvantage is the adhesion to the steel substrate, since PVD layers differ considerably from steels in terms of mechanical and physical properties.

The surface has to meet many criteria to resist various mechanisms of wear. The first of these criteria, and established recently as not the most important, is hardness¹. The ratio of the hardness to the Young's modulus was established as being the most important

criterion for sufficient wear resistance¹. This ratio differs significantly for steels and for PVD layers, and may cause serious problems in the surface processing of tools. For example, the well-known layers such as TiN, TiAlN and TiB₂ have a hardness of about *HV* 3000 (four times more than tool steels) at a Young's modulus of 400 GPa (2 times higher than steel)³⁻⁵. Also, for the promising CrN layers, the Young's modulus is higher than that of steels (up to 240 GPa), whereas the hardness can be varied according to the actual chemical composition across a very wide range, from *HV* 1500 to approximately *HV* 2500⁶.

The heat treatment affects the Young's modulus in a very limited range. On the other hand, the hardness can be increased very significantly with an appropriate heat treatment. For the steel Vanadis 6, the hardness in the as-delivered state is about *HB* 255 (*HRC* 23)⁸, and after heat treatment a hardness of *HRC* 62 (*HV* 750) can be easily achieved. An additional surface hardness increase (and also the hardness/Young's modulus ratio) can be achieved by nitriding. As reported^{9,10} for the Vanadis 6 steel after plasma nitriding, a hardness of *HV* 1100 can be achieved. On the other hand, the fracture toughness is lowered very significantly by the nitriding^{11,12}. The minimizing of the undesirable effect of nitriding on the fracture toughness is a significant goal of our other

investigations. In this paper we report on the results of an investigation of the wear mechanism of CrN+ plasma-nitriding duplex-layers deposited on a Vanadis 6 substrate.

2 EXPERIMENTAL

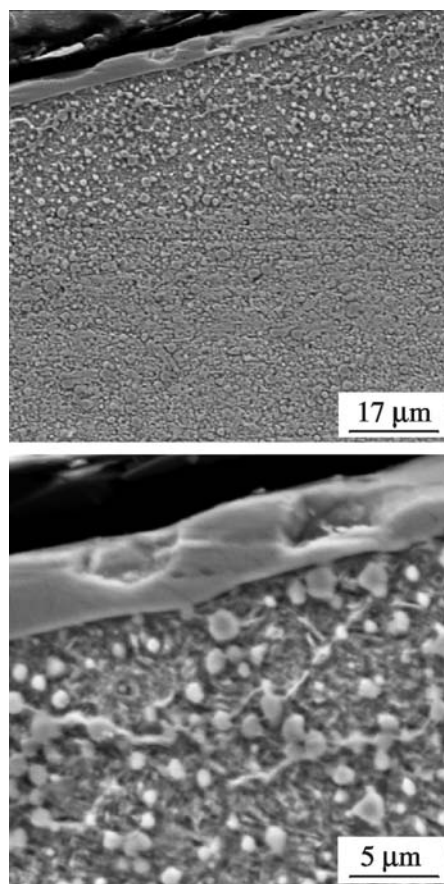
Specimens of the steel Vanadis 6 (2.1 % C, 7 % Cr, 6 % V, Fe bal.) were heat treated to a hardness of *HRC* 60, the recommended value for cold-work applications. Plasma nitriding was performed in a RUBIG Micropuls – Plasmatechnik® device at temperatures of 500 °C and 530 °C and processing times of 60 min and 120 min, respectively.

The CrN layers of thickness 2 µm or 5 µm were deposited with vacuum arc sputtering in a Balzers BAI 730M device, and the adhesion of the layers was examined with a scratch test. The microstructural examinations and the analysis of the worn surfaces were performed with scanning electron microscopy. Wear testing was carried out without lubrication using the ring-on-plate configuration of the counterpart and the tested specimen, at loads of 50 N and 150 N, respectively. Hollow cylinders from the ball-bearing steel 100Cr6 heat treated to *HRC* 60 were used as the counterparts. The total movement (sliding in a combination with slow rotation) distance was of 10 km, and the wear weight loss was determined also after (1, 2.5 and 5) km of sliding. Glow discharge optical emission spectroscopy (GDEOS) was used to determine the elements' depth profiles in the near-surface region. The phase constitution of the CrN layers was identified with X-ray diffraction.

3 RESULTS

In a previous paper¹³, the best nitriding parameters with respect to the adhesion of the PVD layers were found to be: a temperature of 530 °C, a processing time of 120 min and a reactive atmosphere with the composition $N_2 : H_2 = 1 : 3$. Tribological tests showed a large scatter in terms of the wear resistance. Therefore, it was decided to examine other combinations of nitriding parameters with the aim to reduce the dispersion of results and consider also the possibility of improving of the fracture toughness, although this was the main goal of other investigations.

An example of the microstructure of a duplex-layer (CrN + plasma nitriding) is shown in **Figures 1 and 2**. On the surface, a CrN layer of thickness 5 µm is deposited. The nitrided inter-layer has a thickness of about 40 µm. It differs from the non-nitrided material mainly in the topography caused by the etching. Also, there is a continuous or quasi-continuous layer of nitride found at the boundaries of the original grains. No inhomogeneities were found in the surface regions and the PVD layer was free of cracks and pores.



Figures 1, 2: SEM micrographs of duplex layer. Above – overview, below – detail

Slika 1, 2: SEM-posnetka dupleksnega prekritja. Zgoraj splošni videz, spodaj detajl

As shown in **Table 1**, all the newly used combinations of plasma nitriding and CrN layering produced a minimal adhesion of approximately 50 N. This indicates that the pre-treatment ahead of the plasma nitriding is a promising method for increasing the adhesion of the PVD CrN coating. On the other hand, the adhesion values again exhibited a large dispersion, even for specimens processed under the same conditions.

The results of the measurements of the wear resistance for a load of 50 N in **Figure 3**, demonstrate the good wear behaviour of the duplex-treated material in comparison to a material without surface processing, only nitrided or only CrN layered⁹. When compared to specimens pre-nitrided at 530 °C for 120 min and then CrN layered¹⁴, the results of the tests were not sufficiently reliable. However, it is clear that the pre-nitriding at 500 °C for 120 min. is more efficient for the wear behaviour of the duplex layer than the pre-nitriding at 530 °C for 60 min. Also, in contrast to previous tests,^{9,14} it was confirmed that a thinner CrN layer had a better wear resistance. This wear behaviour is to be expected, since internal stresses, often leading to coating damage, increase with the coating thickness under given testing conditions¹⁵.

The measurements gave a lower weight loss for the counterparts after sliding with the CrN layer with a thickness of 2 μm (Figure 4). No major changes in weight loss were found, which would indicate that no delamination of the coatings occurred during the sliding test. It is concluded that the cause of the different wear was the differences in the friction coefficient (Figure 5). A higher friction coefficient induced rather more wear (Figure 3).

Table 1: Measurements of adhesion with a scratch test. Yellow lines – results in^{9,14}

Tabela 1: Določanje adhezije s trgalnim preskusom. Rumene vrstice – rezultati v^{9,14}

Processing	Lc3/N	Lc4/N	Lc5/N
CrN 2 μm	18	44	60
CrN 5 μm	26	56	65
Nitriding 530 °C/120 min + CrN 2 μm	44	70	not identified
Nitriding 530 °C/120 min + CrN 5 μm	90	94	99
Nitriding 500 °C/120 min + CrN 2 μm	50, not identified	79, 156	116, -
Nitriding 500 °C/120 min + CrN 5 μm	55, 148	not identified	129, 158
Nitriding 530 °C/60 min + CrN 2 μm	46, 135	not identified, 100	108, 138
Nitriding 530 °C/60 min + CrN 5 μm	47, 52	not identified	98, 134

The use of a higher load (150 N) led to increasing differences in the wear behaviour between the specimens processed by various combinations of plasma nitriding and CrN layering (Figure 6), and also between specimens processed in the same batch (Figure 8). However, the general tendency of improved wear resistance after plasma nitriding was retained. The assessment of other

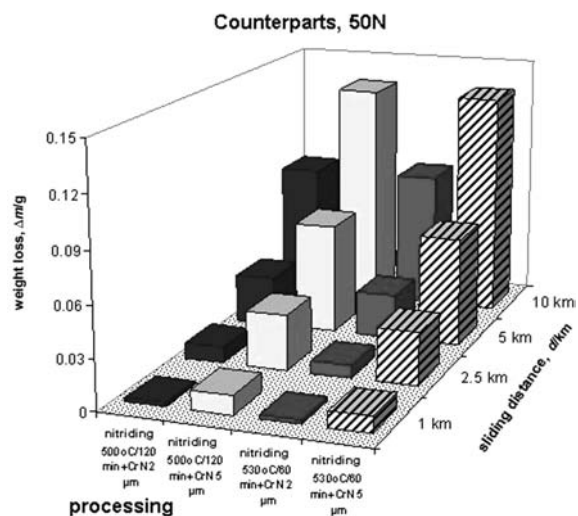


Figure 4: Weight loss of counterparts as a function of sliding distance and surface treatment, load of 50 N

Slika 4: Izguba mase nasprotnega dela v odvisnosti od dolžine drsenja in površinske obdelave; obremenitev 50 N

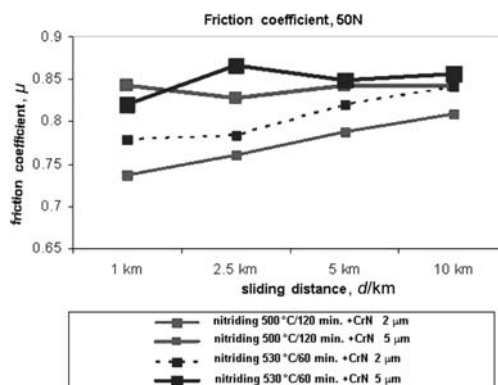


Figure 5: Friction coefficient as a function of sliding distance, load of 50 N

Slika 5: Koeficient trenja v odvisnosti od dolžine drsenja; obremenitev 50 N

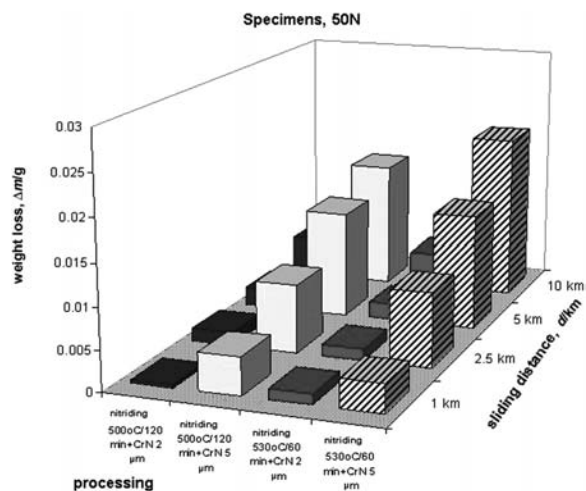


Figure 3: Weight loss of specimens as a function of sliding distance and surface treatment, load of 50 N

Slika 3: Izguba mase vzorcev v odvisnosti od dolžine drsenja in površinske obdelave; obremenitev 50 N

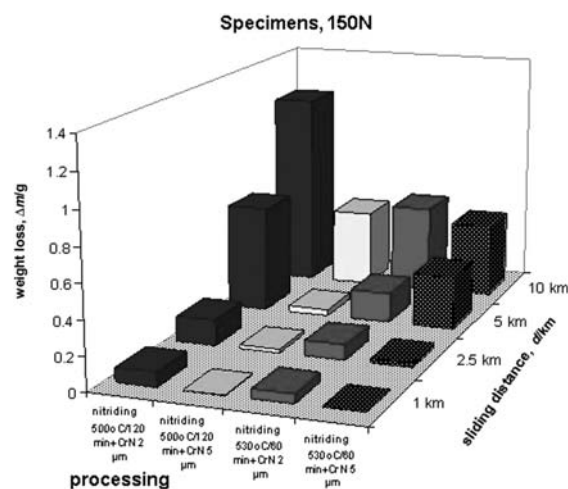


Figure 6: Weight loss of specimens as a function of sliding distance and surface treatment, load of 150 N

Slika 6: Izguba mase vzorcev v odvisnosti od dolžine drsenja in površinske obdelave; obremenitev 150 N

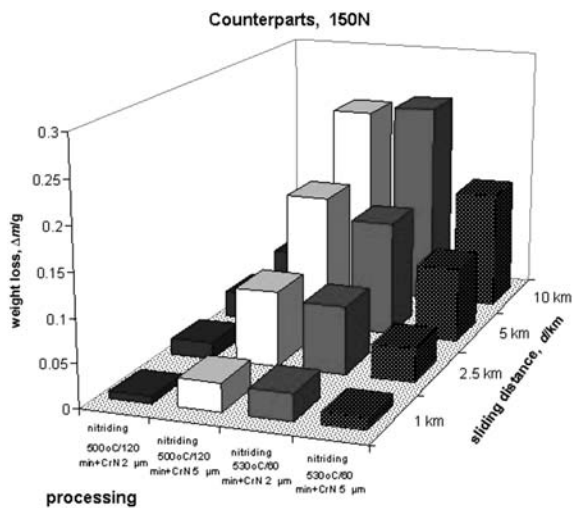


Figure 7: Weight loss of counterparts as a function of sliding distance and surface processing of the specimen, load 150 N

Slika 7: Izguba mase nasprotnega dela v odvisnosti od dolžine drsenja in površinske obdelave vzorca; obremenitev 150 N

effects on the wear resistance became more difficult due to sudden changes in the sliding surface due to the PVD-layer delamination. Therefore, the mean values from Figure 6 do not represent the wear behaviour accurately because of the large dispersion due to delamination on at least one specimen from the three tested.

Also, the figures of weight loss for the counterparts exhibited a large scatter. The mean values in **Figure 7** give only partial information about the process during the wear testing. In the cases of an absence of delamination, the weight loss of the specimen was minimal, the counterpart underwent intensive wear (**Figure 8**) and the friction coefficient increased slowly.

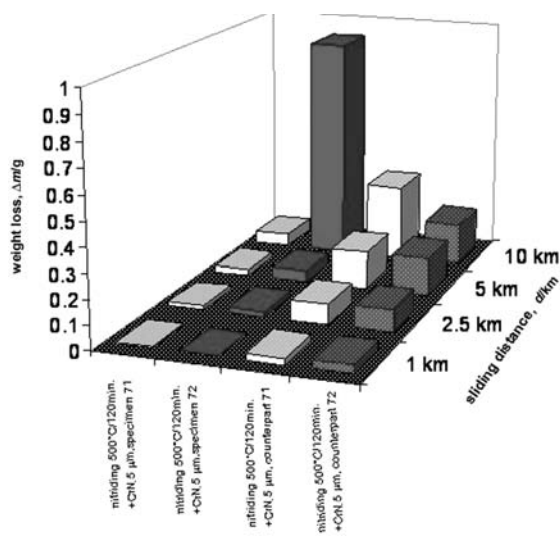


Figure 8: Wear of specimen and counterpart in the case of good (yellow) and poor (red) adhesion

Slika 8: Obraba vzorca in nasprotnega dela za primer dobre (rumeno) in slabe (rdeče) adhezije

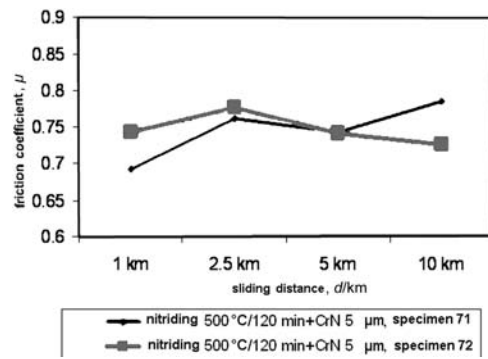


Figure 9: Friction coefficient for the specimen with good adhesion (yellow) and poor adhesion (red) of the CrN coating

Slika 9: Koeficient obrabe za vzorec z dobro (rumeno) in slabo (rdeče) adhezijo CrN-plasti

On the other hand, if the adhesion of the CrN layer was poor, the wear rate on the specimen's side changed suddenly from low to very high, and the weight loss of the counterpart became minimal. Simultaneously, the friction coefficient also decreased as a result of the decreased surface contact between the specimen and the counterpart (**Figure 9**). To understand these differences in wear behaviour, selected specimens with good (71) and worse (72) adhesion were subjected to a more detailed investigation.

These observations correspond well with the measurements of adhesion from the scratch test, although the type of contact differs for those during the scratch test (point contact of the diamond indenter) and during the wear-resistance test (linear contact of a hollow cylinder with approximately equal hardness) differs considerably. As shown already, the adhesion of 50 N, which is the minimum acceptable value for various industrial applications, was achieved for all the

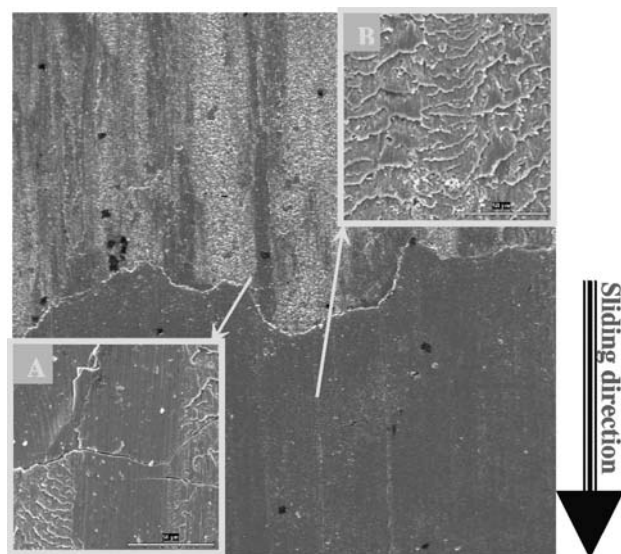


Figure 10: Worn surface of the specimen with good adhesion of the CrN layer

Slika 10: Obrabljena površina vzorca z dobro adhezijo CrN-plasti

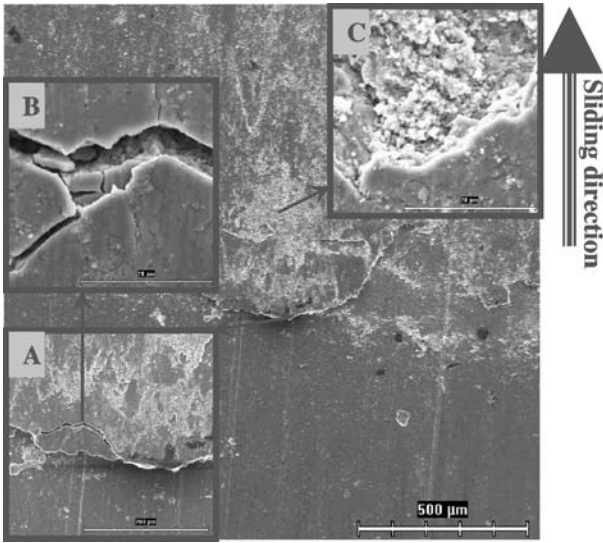


Figure 11: Worn surface with poor adhesion of the CrN layer
Slika 11: Obrabljena površina vzorca s slabo adhezijo CrN-plasti

specimens. Large differences in the wear resistance found for a load of 150 N can be attributed to the scattering of the adhesion values (ranged from 50 N to 148 N) determined during the scratch test.

Figure 10 shows the surface of a specimen with excellent PVD layer adhesion. The surface shows two areas of different micro-morphology. The first area (part A) is typical for the practically undamaged layer and only the friction producing parallel tracks in the sliding direction occurred during the wear test. On the second, "B", area a slightly deformed and cracked coating without clear marks of delamination is found. The preservation of the coating on the surface in spite of the high contact pressure confirms its excellent adhesion.

In the case of worse adhesion of the coating, the situation is significantly different (**Figure 11**). The wear process was probably equal to the case of good adhesion (**Figure 10**) until the occurrence of cracking in the CrN layer at various places. This was mostly, however, at the boundary between the worn and the unaffected parts of

the specimen (A). From this moment on the friction coefficient started to decrease (**Figure 9**). The degradation of the layer continued with its crumbling, predominantly in the vicinity of the primary cracks (B), up to the total material removal in selected areas (C). The removed fragments of the CrN coating accelerated the weight loss on the specimen's side due to the removal itself, while on the counterpart's side, the weight loss was practically stopped due to minimizing of the sliding contact (minimizing of the surface undergoing the wear) and lowering of the friction coefficient (**Figure 9**).

Specimens with good (71) and poor (72) wear behaviour also differ in the chemistry of the PVD layers and, as a consequence, in the phase constitution, too. The CrN layer on specimen 71 contains less chromium than that on specimen 72 (**Figures 12 and 13**), but it has more nitrogen. The difference is 1.5–2.0 %, depending on the depth. At the same time, the layer on specimen 71 consists mainly of the Cr₂N compound, while in the layer on specimen 72, the content of nitrogen in the chromium solid solution is significantly increased (**Figure 14**). The specimens also differ in terms of the texture of the layer. The diffraction peaks of Cr₂N₍₁₁₀₎ at 37° of the two-theta angle and Cr₂N₍₃₀₀₎ at 67° are higher for specimen 72, with a poor adhesion, than for specimen 71, with good adhesion. The differences indicate a different mechanism of growth for both layers. At first sight this is surprising, since the layers were prepared on specimens processed in the same way as in the heat-treatment stage and in the nitriding stage.

Another possible explanation of the different wear behaviour of the specimens, and also of those processed under the same processing conditions, is a possible exceeding of the load-carrying capacity of the substrate. Nevertheless, the scratch test indicated a large scatter of the critical load values and, therefore, the differences in the layer formation displayed by X-ray diffraction

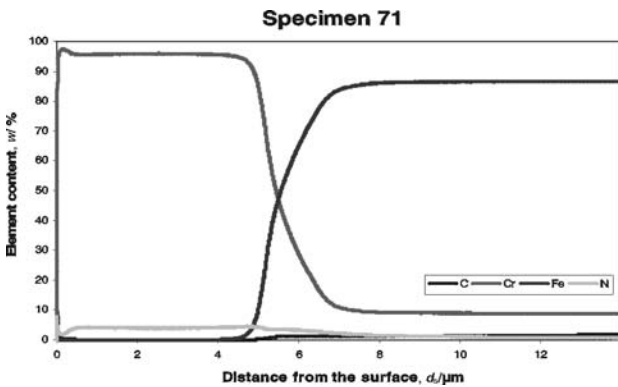


Figure 12: Depth profiles of elements throughout the near-surface region – specimen with good adhesion

Slika 12: Porazdelitev elementov v CrN-plasti blizu površine, vzorec z dobro adhezijo

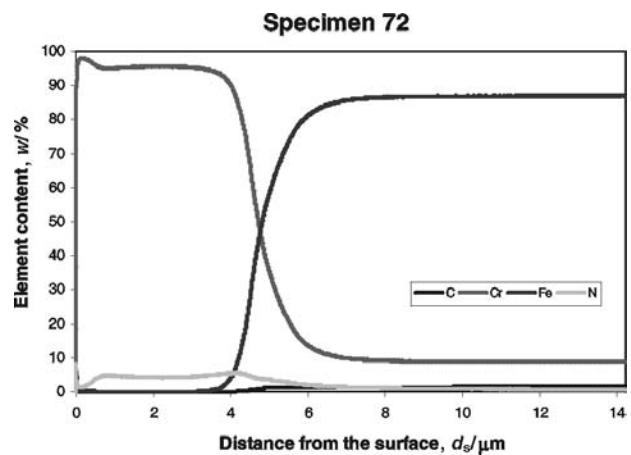


Figure 13: Depth profiles of elements throughout the near surface region – specimen with poor adhesion

Slika 13: Porazdelitev elementov v CrN-plasti blizu površine, vzorec s slabšo adhezijo

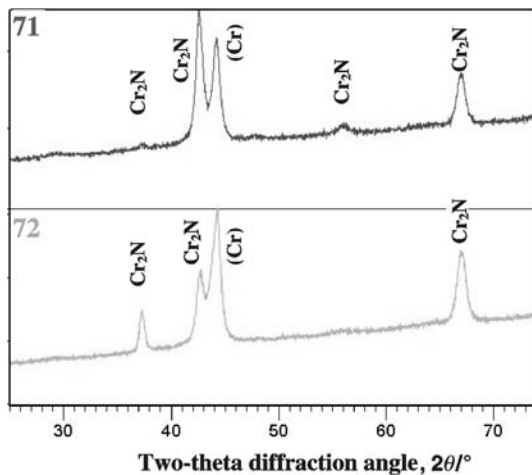


Figure 14: X-ray diffraction patterns from PVD CrN layers

Slika 14: Difraktogram rentgenskih žarkov u PVD-plasti

measurements are more probably the primary explanation for the different wear resistance of the specimens. The cause of different layer growth, in a given batch, too, can probably be attributed to the positional effect of the specimen with respect to the chromium source (target) during the sputtering. No other effects with a possible influence on the layer growth are known.

4 CONCLUSIONS

1) For all the combinations of plasma nitriding and CrN coating, the critical load measured with the scratch test was at least 50 N, which is considered to be an acceptable value for industrial applications. In some cases the adhesion was much better and the critical load exceeded 130 N. However, a large scatter of adhesion values was found, also for specimens processed under the same nitriding and/or coating conditions.

2) Wear testing at a load of 50 N did not show a significant scatter of results. The wear of specimens proceeded in a steady manner and slightly better results were found for the thinner CrN layers.

3) The use of a load of 150 N emphasized the difference in the wear behaviour of the specimens, also for those prepared under the same processing conditions. For the specimens with excellent adhesion, the wear rate on the specimen's side was low and on the counterpart's side it was much greater. The friction coefficient increased slightly with the increased sliding distance. For the specimens with poor adhesion, a sudden and dramatic increase in the wear rate on the specimen's side occurred during the test. This increase is connected with the decrease of the friction coefficient and the lowering of the wear on the counterpart's side.

4) In the case of specimens with excellent adhesion the CrN coating remained on the surface, although in a slightly damaged form. Coatings with small adherence cracked and were progressively removed from the contact surface.

5) Layers with excellent adhesion differ from those with poor adhesion in chemistry and phase constitution. In the first there was less chromium in the solid solution and more Cr₂N compound was found in the second. Moreover, the different texture probably also indicates a difference in the growth mechanism.

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