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ANALYSES OF THE REASONS FOR THE DECREASED SERVICE TIME OF CRN - COATED DIE FOR ALUMINY HOT EXTRUSION – A CASE STUDY

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Presented study shows that considerable reserves still exist for increasing the service times (lifetimes) of CrN - coated dies for Al hot extrusion. The main reasons for the decreased service times are revealed and explained regarding the selected CrN - coated die for hot extrusion, i.e. why the service time of the coated-die is not in accordance with the wear resistance of the CrN - coating. The shaping of the bearing surface and presence of the scratches, size and amount of nonmetalic inclusions in the die steel, nodular defects in the CrN - coating, as well as thicknesses uniformity of CrN - coatings along the bearing surface, are relevant influential parameters.

Key words: CrN - coated, demage, Al hot extrusion, physcial vapor deposition coating, die wear, service time

INTRODUCTION

Aluminium hot extrusion is a metal-forming process for the manufacturing of long products with different sectional shapes such as beams, rods, tubes, wires, etc. This is carried out on a hydraulic press where a heated billet is pushed by a ram through the profile opening of the die. Extrusion dies are exposed to high mechanical, thermal (up to 600 ºC), chemical and tribological (sliding velocities up to 100 m/min) loads. The mentioned loads in relation to the relevant characteristics of dies, i.e. wear resistance, shaping of the die (flow of material, etc.), quality of die steel, heat treatment, etc. influence the occurrences of different damages on the dies, such as e.g. wear, cracking, fracture, plastic deformation, etc. which influence the qualities of the extruded profiles and consequently the service times of the dies [1-10].

In order to increase the wear resistances of the extrusion dies usually nitriding [2, 4-8, 11-15], physcial vapor deposition (PVD), chemical vapor deposition (CVD), etc. procedures are applied [15-24]. However, despite the fact that the PVD- coated bearing surfaces of the dies are much more wear resistant in comparison to only gas nitrided, they usually do not reach service times which correspond to their wear resistance characteristics [17-22]. Moreover in industrial exploitation, nitrided as well as PVD - coated dies, Large differences in industial service times can be observed for the extrusion of similar or even for the same profiles between nitrided and PVD - coated dies. Till now the reasons for such behaviour were not sufficiently explained.

Therefore in this contribution the main reasons for the decreased service times of dies for Al extrusion for the selected CrN PVD coated die are revealed and explained. In order to evaluate the service time of a CrN - coated die, a die which was only gas nitrided was used for comparison.

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EXPERIMENTAL

Applied extrusion die

The length of the bearing surface was 5,5 mm and the thickness of the extruded hollow profile was 1,5 mm. In Figure 1 two spots denoted by A and B are also shown, where a detailed examination of the area from the inlet to the outlet edge along the bearing surface was carried out. These postions were selected since most

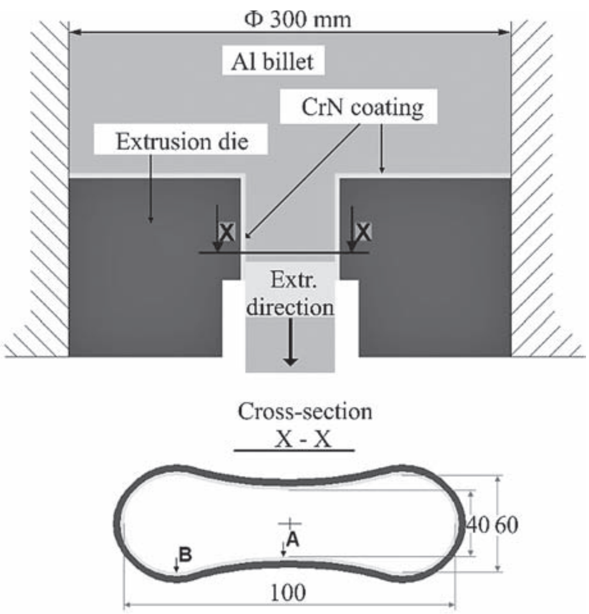

Figure 1 Schematic presentation of CrN PVD-coated die (die opening) used for the analysis of wear progress

damages were observed there. CrN - coating was deposited by the magnetron sputtering system CC800/7 (CemeCon). The coating was deposited on the die as well as on ground, polished, and ultrasonically cleaned substrate made from H11 tool steel. Prior to deposition substrates were RF ion etched. During deposition power on the targets was 4 kW. The flow-rates of N_2, Ar and Kr were 140, 140 and 80 ml/min, respectively. The temperature during deposition was 450 °C. The DC bias volatage on the substrates was 100 V.

Characterisation of the CrN PVD coated die

Microstructures etched with Nital were obtained with an OLYMPUS BX60M optical microscope. The thicknesses of the PVD coating were determined by the ball cratering technique (calotest) and by SEM fracture cross-sections. A Taylor-Hobson Talysurf 2 profilometer was used to measure surface roughness S_a. Vickers microhardness HV and indentation modulus were determined by micro-indentation (Fischerscope H100C). The morphologies of the coating were characterised by field emission scanning electron microscopy (JEOL JSM 5800). The coating topography was measured by 3D - profilometer and atomic force microscope. X-ray diffraction was conducted in a diffractometer using Bragg-Brentano geometry. The coating adhesion was evaluated with a scratch tester equipped with a 200 μm radius Rockwell C diamond stylus (CSEM REVETEST) on a scratch length of 10 mm, table speed 10 mm/min, loading rate 200 N/min and loading scale 0 - 200 N.

RESULTS AND DISCUSSION

Characteristics of CrN coating

The CrN coating thickness on the test samples was 4,0 ± 0,5 μm and surface roughness S_a = 43 ± 5 nm, 60 % higher in comparison with the non-coated polished substrates (cf. Figure 2 a). Increased roughness was a consequence of formations of the nodular defects. The density of the nodular defects, considered to be those with a height above 0,5 μm, was typically in the range

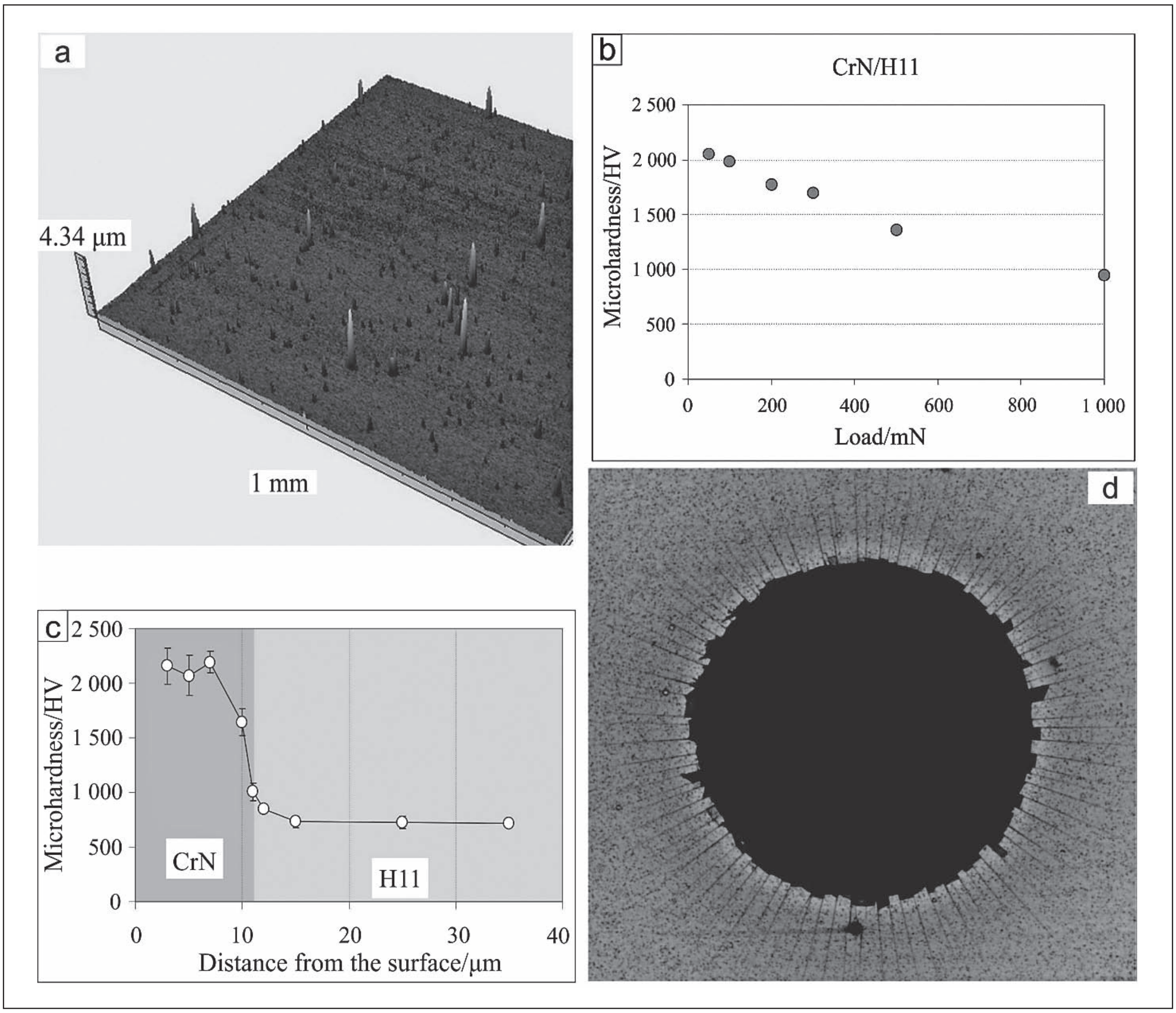

Figure 2 Characteristics of CrN – coating: a) 3D - profilometer image of a CrN - coating deposited on a H11 tool steel substrate, (the peaks are nodular defects), b) micro-hardness of CrN-coating on H11 as a function of indentation load (25 – 1 000 mN), c) and micro-hardness depth profile on a perpendicular cross-section of CrN -coating on H11 (load 10 mN), d) Rockwell indent confirmed a good adhesion of CrN - coating deposited on a H11 tool steel substrate

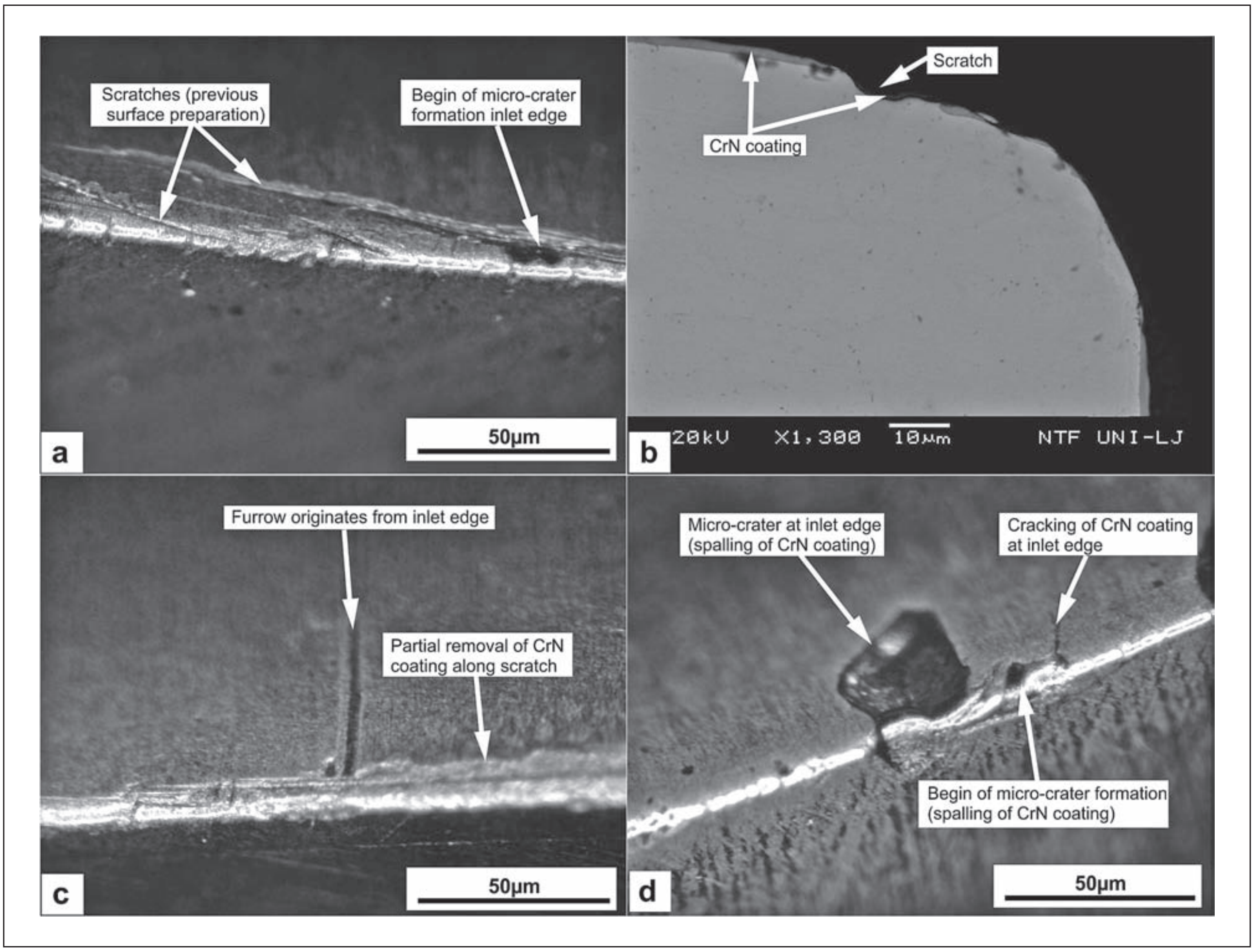

Figure 3 Damages on the inlet edge of a CrN - coated die bearing surface at spot A (see Figure 1): a) tangential coarse scratches at inlet edge, b) cross-section of inlet edge (SEM), c) extending of initial micro-craters at inlet edge in the sliding direction of Al profile, d) and formations of micro-craters at the inlet edge due to its too high sharpness

80 mm^{-2}. It was found that CrN - coating is sub-stoichiometric with an average N/Cr ratio of 0,9 ± 0,1. TEM analysis showed a columnar microstructure with a column diameter of about 160 nm. According to the XRD spectra the CrN - coatings exhibited the FCC structure with a pronounced (200) texture. A small amount of Cr_2N phase was also detected. The lattice parameter for the FCC CrN phase was 0,414 nm. The results of the Vickers micro-hardness measurements performed on coatings deposited on H11 tool steel substrate are presented in Figure 2 b. Micro-hardness depth profile using loads of 10 mN is presented in Figure 2 c. Both the indentation test (see Figure 2 d) and the scratch test showed that adhesion of the CrN fulfilled the standards for technological applications, i.e. critical load > 50 N.

Using the scratch test the adhesion was evaluated by measuring critical loads for the well-defined failures of the coating, for the beginning of acoustic emission and for the rapid increase in scratch force. The critical load for total delamination of the coating, Lc5, which correlated with the scratching force jump, LcF_5, showed the best reproducibility of the results. Six scratches were made on each sample. The critical loads LcF_5 were more than 75 N for all samples.

Service times of the dies

The expected service time of the analysed PVD - coated dies (CrN) in industrial exploitation measured with length is arround 200 000 m but the applied die sustained only 102 000 m. In contrast, the gas nitrided die used for extrusion of the same profile sustained extrusion of 58 100 m. In general service time of the CrN-coated dies were approximately twice of the gas nitrided die. However, this is still considerable lower than one would expect when comparing their wear characteristics.

Analysis of wear on a CrN - coated die

The main damages on the PVD - coated die were found predominately at the inlet as well as exit edges of the bearing surface (spots A and B, as depicted in Figure 1). For spot A main damages were at the inlet edge of the bearing surface, as presented in Figure 3a where coarse scratches are clearly visible in the tangential direction originating from previous surface preparation. The cross-section of these scratches are presented in Figure 3b, where it could be seen that residues of the CrN - coating are still present on the bottom of scratch-

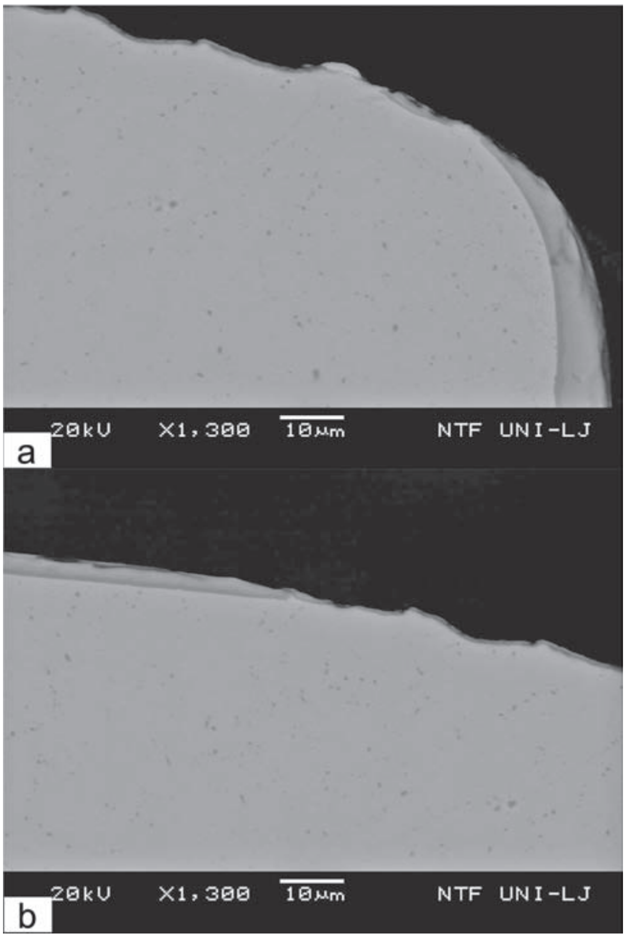

Figure 4 Damages on the inlet edge of the die's bearing surface at spot B: a) cross-section of inlet edge with visible tangential scratches and the remaining CrN - coating within the scratches, b) cross-section of the area after the spot of the presence of scratches

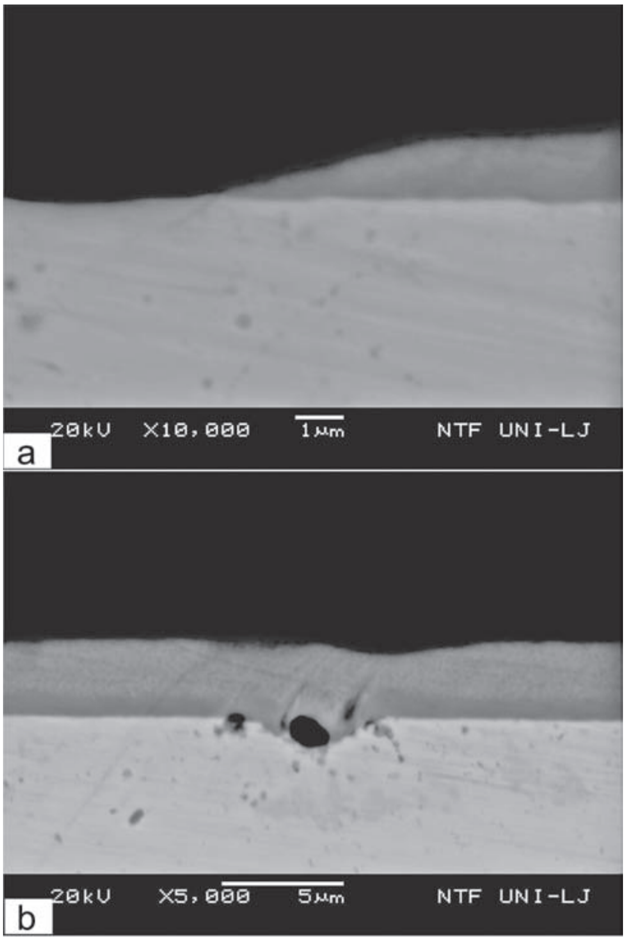

Figure 5 Damages on a cross-section of CrN-coating in the central part of the bearing surface at spots A and B denoted in Figure 1: a) spot of entirely removed CrN-coating, b) and non-metallic inclusion on substrate surface, (SEM)

es, which indicate that they were present before the coating procedure, i.e. they originate from previous surface preparation. All the topographical features of substrates before deposition are retained and due to the shadow effects are often magnified after deposition. Furthermore, these topographical irregularities on the coating's surface represent obstacles for the sliding of Al during extrusion, which leads to increased local tribological loads on the coating and consequently to premature partial removal of the CrN layer, i.e. forming of micro-craters. Furthermore, it should be emphasised that normal contact pressures have the highest values near or at the inlet edge of the bearing surface and then gradually decrease towards its exit edge [9]. Thus these initial scratches on the coating at the inlet edge serve as initial spots for the formation of micro-craters (see Figure 3a), which then extend along the entire bearing surface towards the outlet edge during the extrusion process (see Figure 3c). On the other hand, Figure 3d shows cracking of the inlet edge and its partial removal as a consequence of its too high initial sharpness. Namely, at the sharper initial edge the stiffness is decreased which consequently accelerates its cracking during extrusion and can also lead to formation of grooves, as presented in Figure 3 c. Thus in order to utilise the coating wear resistance on the entire bearing surface, its shaping, preparation and maintenance are essential.

Similar damages can also be observed at the inlet edge (spot B shown in Figure 1) as shown on the cross-section in Figure 4a. These damages are related to tangential scratches which originate from previous mechanical pretreatment, as the residues of CrN - coating in these scratches were visible again. Figure 4b shows the spot that lies in the sliding direction after the spot of the appearance of scratches and exhibits considerably lower removal of CrN - coating, whereas no scratches can be observed on the initial surface. Again it can be concluded that initial scratches at the inlet edge of the bearing surface are responsible for premature CrN - coating removal due to increased loads (normal contact pressures) due to increased roughness. Bearing surface thickness of the CrN - coating at the beginning is around 5 – 6 µm (see Figure 4b) whilst on the surface perpendicular to the bearing surface, i.e. before the inlet edge, it is almost 10 µm (see Figure 4a). In the central part of the bearing surface this value is about 4 – 5 µm (see Figure 5) whilst on areas closer to the exit edge is is 2 – 3 µm (see Figure 6). The decreased coating thickness towards the outlet edge can be attributed to the well-known 'line of sight' effect. Damages observed in

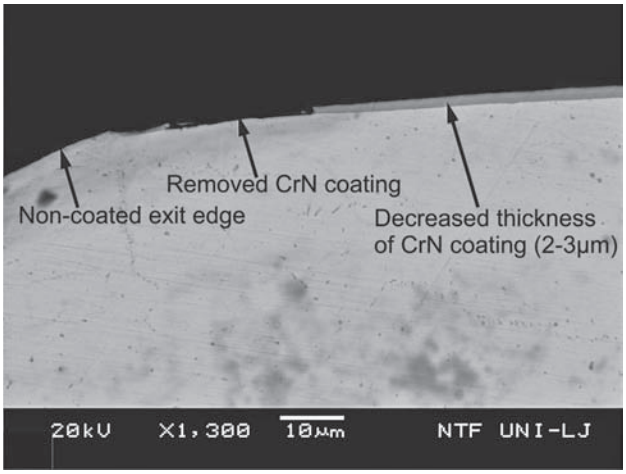

Figure 6 Cross-section of bearing surface: removal of CrN - coating with non-coated area at exit edge, (SEM)

the central part of the bearing surface, i.e. in sliding direction regarding spots A and B are predominately lower in comparison to the spots at the initial edge. On the some spots in the central part of bearing surface, CrN - coating is almost entirely removed as can be seen in Figure 5a, but this area did not suffer significant damages. Loss of the coating during the initial stages of wear is caused by the spalling of nodular defects that are always present in the as-deposited coatings. Nodular defects have a strong negative impact on the tribological performances of the coatings during sliding contact, causing the workpiece material to pick-up on the coating's surface. They also poorly adhere to the surrounding material and can be pulled out during the extrusion process. The nodular defects originate from surface irregularities (asperities, scratches, steps) in the substrate and from different foreign particles. The origins of these particles are different: part of them remain on the substrate surface after pretreatment (e.g. dust, polishing residue, debris, impurities), whilst others are generated during the deposition process. The formation of nodular defects in PVD - coatings is caused by shadow effects initiated by all these seed particles or asperities on the substrate surface during the deposition process since evaporation and sputtering are line-of-sight processes. The weak points of the coated sample are also at the positions where different non-metallic inclusions are present on the substrate surface (see Figure 5b). Present study confirmed that especially MnS inclusions reduce the substrate-coating interfacial strength.

The fact that in central part the damages were not extended to the exit edge can be attributed to delayed occurrence of damages in comparission to the damages at the inlet edge. It should be emphasised that local removal of the CrN - coating does not considerably decrease the surface finish of the extrudate, but the formation of grooves along the entire bearing surface (see Figure 3c), as well as damage at the exit edge (see Figure 6), can have a detrimental influence on its surface finish. At the exit edge the non-coated areas, as well as those areas with complete removal of CrN - coating, are observed (see cross-section in Figure 6). Furthermore, the lower thickness of the CrN - coating at the exit edge, its decreased stiffness and the presence of the scratches at the exit edge leads to premature removal of coating and considerable decreased service times of the extrusion dies. The inner surface of the die hole can be coated but it should not have a depth greater than its diameter. Depending on the geometry of the tool, thickness of the coating may decrease as depth of the die hole increases. It is very important that the inner surface of the die is directly exposed to the flux of sputtered atoms from the source. One or two-fold rotations are necessary to obtain uniform coating. Furthermore, more attention should be paid to avoid sharpness when producing the radii of the inlet and exit edges, i.e. radii of these gravures should be increased, which will lead to an increased stiffness and to the reduction of contact pressures on the sharp edges.

CONCLUSIONS

From the analysis of damages of the selected industrial CrN - coated die for Al hot extrusion, main reasons for the decreasing service time are discussed and the following conclusions can be drawn:

- Considerable reserves for increasing the dies' service times of CrN - coated dies for Al hot extrusion exist and consequently also the possibilities for increasing the economy of this forming process.
- Regarding its wear characteristics, the analyzed CrN - coated die sustained considerable lower service time as expected, i.e. 102 000 m of extrusion profile vs. expected 200 000 m.
- Analysis of damages on extrusion die contributed to understanding the complex relationships between loads, characteristics of CrN - coating, shape and surface qualities of bearing surfaces, the used die steel and the service times of the dies.
- Appropriate shaping of the inlet edge of the bearing surface, i.e. avoiding too high sharpness would increase its stiffness, which would prevent its chipping.
- Bearing surface preparation prior to CrN - depositing, i.e. avoidance of producing scratches perpendicular to the sliding direction and die maintenance are very important parameters for the utilizing of higher wear resistances of CrN - coated dies in comparison to nitrided ones.
- Irregularities on the coated bearing surface lead to locally increased tribological loads and consequently to earlier removal of coating.
- Die steel with lower fraction and smaller size of non-metallic inclusions should be selected for extrusion dies.
- PVD - deposition procedure should be improved in such a way as to reduce nodular defects in CrN - coating and to ensure uniform thickness along the entire bearing surface length. The latter can be achieved by one or two-fold rotations of the die during the PVD process.

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