

## WEAR BEHAVIOUR OF NITRIDED MICROSTRUCTURES OF AISI H13 DIES FOR HOT EXTRUSION OF ALUMINIUM

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Nitriding of bearing surfaces on dies (tools, AISI H13) for hot extrusion of aluminium is technologically a very sensitive process with regard to achieving a constant quality of the nitrided layers. This study was based on the analysis of microstructure on dies with intentionally prepared deep and narrow gaps which were nitrided by various manufacturers of equipment for gas and ionic nitriding. The manufacturers chose their own nitriding parameters in order to achieve an optimal wear resistant microstructure. The microstructures obtained showed differences with regard to the presence or absence of a compound layer (white layer), its thickness and its  $\epsilon/\gamma'$  phase ratio (XRD), nitriding depth and microhardness profile. The measured nitriding depths and the maximum microhardness values on nitrided surface layers were quite similar on dies of the same manufacturer, while for different manufacturers these values differed. Differences with regards to compound layer characteristics were also found on the same die. The die samples with these various nitrided microstructures were then laboratory tested for wear resistance using equipment that provides simulation of the tribological conditions during hot extrusion of aluminium. The wear testing results show differences in behaviour of the nitrided samples. The differences in the actual structures, microstructures, hardness, etc. explain the high level of scattering in die life in actual industrial applications.

**Key words:** *aluminium hot extrusion, H13 die steel, nitrided microstructures, compound layer, XRD, wear resistance*

**Obradne karakteristike alata AISI H13 s nitriranom mikrostrukturom pri toploj ekstruziji.** Nitriranje alatnih (AISI H13) površina pri toploj ekstruziji aluminija je tehnološki veoma osjetljiv proces obzirom na poteškoće postizanja konstantne kvalitete nitriranog sloja. Studija se temelji na analizi mikrostrukture površine u namjerno pripremljenim uskim i dubokim utorima alata koji su bili nitrirani kod različitih proizvođača opreme za plinsko i ionsko nitriranje. Svaki proizvođač je nitrirao više alata i pri tome postigao različitu mikrostrukturom nitriranog sloja s prisustvom bijelog sloja ili bez njega. Postignute su čak razlike u debljini bijelog sloja kod istog proizvođača s obzirom na broj alata. Svi alati bili su laboratorijski testirani na obradni otpor na sopstveno izrađenom simulatoru triboloških uvjeta koji su prisutni pri toploj ekstruziji aluminija. Rezultati testiranja potvrđuju razlike u ponašanje obradnog otpora nitriranih uzoraka. Razlike u prisutnoj strukturi, mikrostrukturom, tvrdoći, dubini nitriranja i omjeru  $\epsilon/\gamma'$  faza pojašnjavaju visoku razinu trajanja životnog doba alata kod ove industrijske primjene.

**Ključne riječi:** *topla ekstruzija aluminija, H13 alatni čelik, nitrirana mikrostruktura, bijeli sloj, XRD, obradni otpor*

### INTRODUCTION

Due to their lightness and good mechanical properties various Al-alloy products obtained by casting, rolling and hot extrusion are still gaining in importance. Hot extrusion of Al-alloys (Figure 1.a) enables the production of a very varied range of profile shapes, which, due to the above

mentioned excellent properties are used mainly in the automotive and electro-industries, in aeronautics, in the building trades, in household goods, etc. The opening on the die (bearing surface, Figure 1.b), through which the extruded piece leaves, gives the extruded profile the desired shape and dimensions. The most frequently used die steel is AISI H13. Beside the quality of the bearing surface its tribological conditions determine the surface quality of the product. Namely, the die life is determined by allowable tolerances of the cross sectional dimensions and by the appearance of the surface on the extruded piece [1 - 4].

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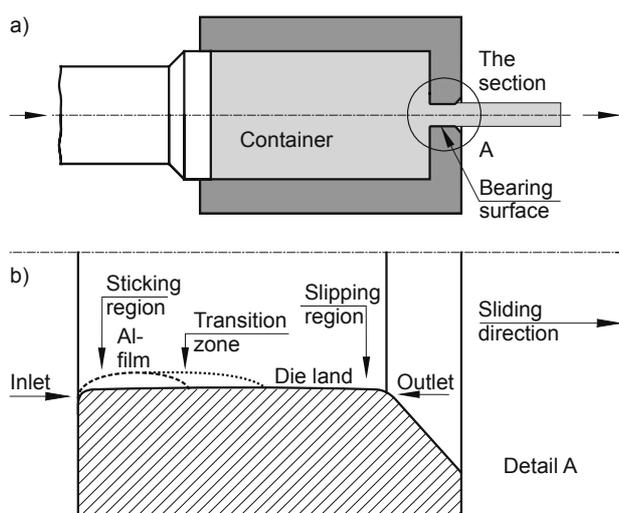


Figure 1. Principle of hot extrusion: a) and bearing surface on the die b)

Slika 1. Princip toplje ekstruzije: a) i klizna površina na alatu b)

Dies for hot extrusion of aluminium are exposed to high mechanical, thermal, chemical and tribological loads. Indeed, the process of hot extrusion takes place at billet temperatures of 450 °C - 490 °C and at exit speeds of the extruded piece between 5 and 50 m/min. Contact pressures on the bearing surface of dies reach values up to 50 MPa, and temperatures can locally even exceed 600 °C, since during the process of extrusion large amounts of heat are generated due to volume compression of the billet and due to friction on the contact between the extruded profile and the surface of the die [5 - 9].

Dies for hot extrusion of aluminium are required to possess good resistance to abrasion and adhesion, mechanical fatigue and corrosion, as well as chemical stability against hot aluminium. Better wear resistance of the die and consequently prolongation of its life can be achieved by an additional refinement of the die (tool) surface; usually this is done by gas, ionic or salt bath nitriding, by CVD or PVD procedures, and by duplex treatment, etc. The weakness of salt bath nitriding lies first of all in its harmful influence on the environment, while the limits of the PVD procedure are related to difficulties in coating the surfaces, which lie in deep and narrow gaps. These deep and narrow gaps are present on dies to give extruded profiles complex shapes with thin walls (higher moment of resistance of extruded profile). Thus the gas nitriding is still common, since it is effective for such narrow and deep gaps in dies [10 - 14, 23, 27].

During gas nitriding the formation of a compound layer often occurs on die surfaces. The chemical composition of the dies, as well as the appropriate setting of the parameters of nitriding (gas mixture and its flow, temperature, time, etc.), influence the formation and the growth of the compound layer, the phase ratio ( $\epsilon/\gamma'$ ) in the compound layer and the

diffusion of nitrogen into the die surface layer. Opinions in the literature about whether the compound layer has a positive or a negative influence on the wear resistance of dies are very divided. Even the manufacturers of equipments for ionic and gas nitriding do not share the same opinion about which nitrided microstructure has the best wear resistance. Thus, some manufacturers avoid a compound layer, but on the contrary, other manufacturers recommend nitrided microstructures with a 3 - 5  $\mu\text{m}$  thick compound layer. It is also known from experience that die life varies very much even in the extrusion of very similar profiles [15 - 20, 26].

In this contribution nitrided microstructures of the surfaces in intentionally prepared deep and narrow gaps of dies that have been nitrided by different manufacturers of ion and gas nitriding equipment are analysed. Furthermore, the study includes phase analysis (XRD), microhardness measurements and the results of wear testing. The wear tests performed on nitrided surfaces indicate which microstructures are potentially more appropriate in achieving a longer service-time of the die.

#### ANALYSIS OF WEAR ON THE BEARING SURFACE OF A NITRIDED DIE

In general three different zones occur on the bearing surface of a die; namely the sticking zone, the transition zone and the sliding zone (Figure 1.b). The transition zone is the most exposed to wear, because just at this location periodic sticking between extruded material and die surface occurs (and consequently chemical reactions) and afterwards to detachment of mentioned connection. The consequence is pitting, which leads later to the formation of small craters (Figure 2.a). These craters slowly extend during the operation of extrusion to the whole transition zone and then continue in the sliding direction of the Al profile in the slipping zone up to the outlet edge of the bearing surface (Figure 2.b). In this way wear mechanisms such as chemical reaction of Al with the die surface, abrasion and adhesion take place.

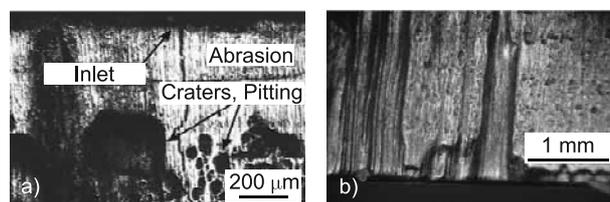


Figure 2. Micrographs of phases in the progression of wear on the die bearing surface for hot extrusion of a round profile: chemical reaction in the transition zone (pitting) and formation of craters a), extension of craters towards the outlet of the bearing surface b), sliding direction ↓

Slika 2. Mikrografi faza razvoja obrade na kliznoj površini alata tijekom toplje ekstruzije okruglog profila: kemijska reakcija u prijelaznom području i tvorba kratera a), produžavanje kratera u smjeru izlaska profila b), smjer klizanja ↓

**EXPERIMENTAL NITRIDING OF DIE SURFACES IN DEEP AND NARROW GAPS**

For evaluation of the efficiency of ionic and gas nitriding of die surfaces in deep and narrow gaps, performed by selected (acknowledged) equipment manufacturers, it was first of all necessary to produce a die (AISI H13) of a particular shape with gaps 1 mm and 4 mm wide and 12 mm deep (Figure 3.). In front of these gaps the die has a

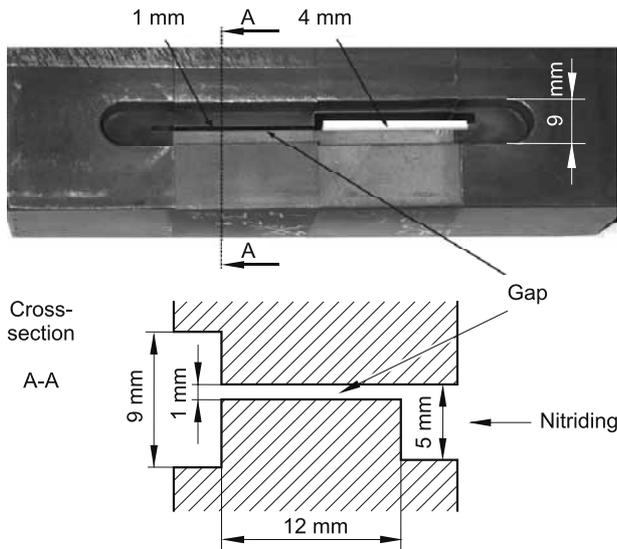


Figure 3. Die with 1 and 4 mm gap and cross-sectioned A-A  
Slika 3. Alat sa 1 i 4 mm visokim utorom i poprečni presjek A-A

gap of 5 mm, and at the back a gap of 9 mm. The chemical composition of AISI H13 (commercial name of the die steel producer METAL RAVNE is UTOPMO2EFS) steel

Table 1. Chemical composition of the die steel used, (AISI H13, METAL Ravne - UTOPMO2EFS)  
Tablica 1. Kemijski sastav upotrebljenog čeličnog alata, (AISI H13, METAL Ravne - UTOPMO2EFS)

C	Si	Mn	Cr	Mo	V	Fe
0,40	1,00	0,40	5,05	1,28	0,95	rest

is given in Table 1. Table 2. gives a list of the manufacturers of nitriding equipments, but for the sake of protecting their interests, they are denoted only by a symbol, and

Table 2. List of manufacturers of ionic (ION) and gas (GSN) nitriding, and dies  
Tablica 2. Lista proizvođača koji su izvršili ionsko i plinsko nitiranje

Manufacturer	I.	II.	III.	IV.	V.	VI.	VII.	VIII.
Type of nitriding	ION	ION	GSN	GSN	GSN	GSN	GSN	GSN
Number of die	1,2	3,4	5-7	8-10	11-14	15-16	17-18	19-20

for the same reason, the tested die is represented by a number. Each equipment manufacturer (altogether eight manufacturers) nitrided at least two dies.

The following requirements were fulfilled:

- all dies were manufactured from the same charge in the same way and in the same workshop;
- nitriding was carried out by eight external manufacturers of equipment for gas or ionic nitriding according to our instructions (the time of the nitriding process, including heating and cooling, did not exceed 12 h, and the depth of nitriding was at least 120 μm);
- the manufacturers themselves, based on their experience with extrusion dies, chose the optimal gas mixture, gas pressure, nitriding temperature, cooling, etc.

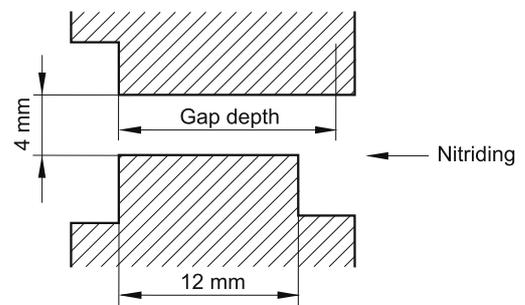
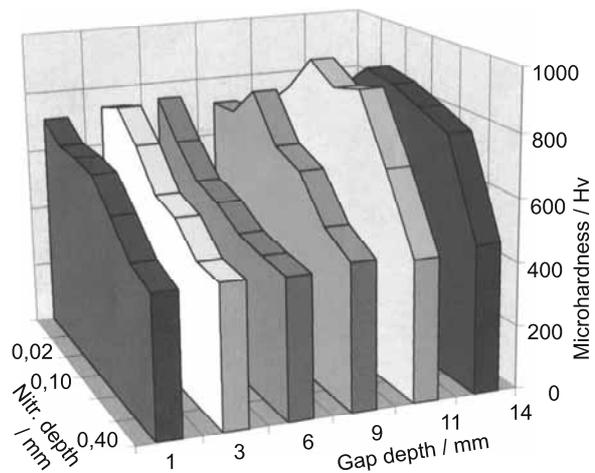


Figure 4. Microhardness obtained in the 4 mm gap (ionic nitriding), die 4, manufacturer II  
Slika 4. Dobivene mikrotvrdoće u 4 mm utoru kod ionskog nitiranja, alat 4, proizvođač II

The use of 1 and 4 mm wide gaps was chosen because the gap depth and its narrowness, in particular, reduce the efficiency of ionic nitriding. Comparison of its efficiency with the gas nitriding of dies is appropriate, since this procedure, in principle, depends much less on the depth of the gap and its width. It is well known that gas nitriding is also effective in deep and narrow gaps, which is why this procedure is still today used in 95 % of cases for improving the wear resistance of bearing surfaces of dies.

## MICROSTRUCTURES, MICROHARDNESS AND XRD ANALYSIS OF NITRIDED SURFACES

**Ionic nitriding:** It is typical of ionic nitrided dies that none of the equipment manufacturers (I and II, die 1 and 3) succeeded in carrying out nitriding in a 1 mm gap (Figure 3.). This is especially true of manufacturer I: the effects of nitriding by manufacturer I range up to cca. 1 - 2 mm in gap depth, and for manufacturer II up to cca. 2 - 3 mm in gap depth; microhardnesses in the 5 mm gap, which lies

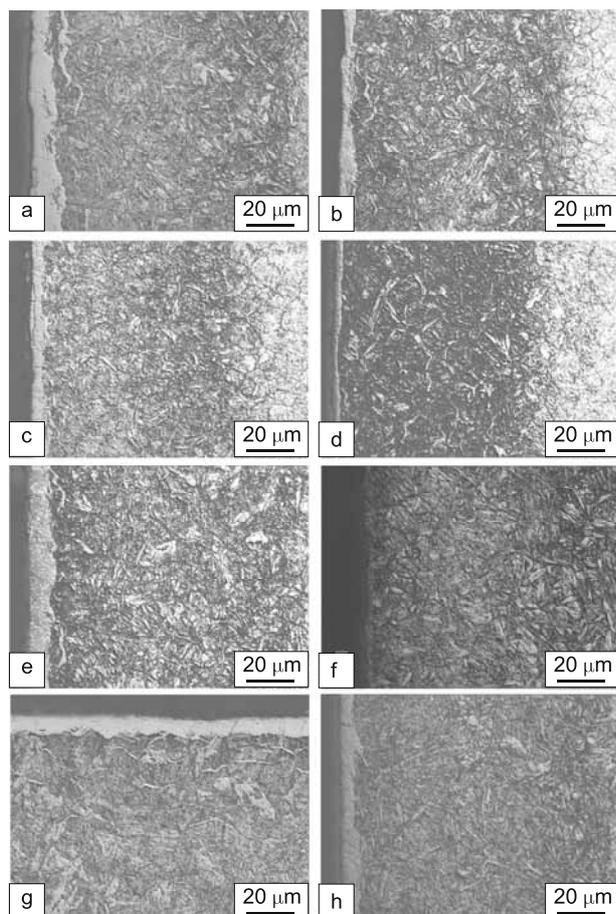


Figure 5. Optical microscopy of gas nitrided microstructures; manufacturer III (a-b), manufacturer IV (c-d), manufacturer V (e-f), manufacturer VI and VII (g), manufacturer VIII (h)

Slika 5. Optička mikroskopija plinski nitrirane mikrostrukture, proizvođač III (a-b), proizvođač IV (c-d), proizvođač V (e-f), proizvođač VI i VII (g), proizvođač VIII (h)

before the 1 mm gap, exceeded the value of 1200 Hv for both manufacturers. In the case of the 4 mm gap, manufacturer II (die 4) was again relatively more successful than manufacturer I (die 2). Manufacturer II achieved a more uniform effect of nitriding over the whole gap and therefore homogeneous values for microhardness (the maximum amounted to cca. 750 Hv), and the depth of ni-

triding to cca. 150  $\mu\text{m}$  (Figure 4.). Manufacturer I achieved microhardnesses of only around 600 Hv in the gap in 12 mm of length, while before the gap microhardnesses were cca. 1100 Hv. The main characteristic of the ionic nitrided layer was the absence of a compound layer.

**Gas nitriding:** It is typical of gas nitrided surfaces that microstructures were uniform over the whole gap depth and that in the great majority of cases a compound layer was present on the nitrided surfaces; the thickness of this layer varied with regard to the manufacturer, as well as to the nitrided dies of the same manufacturer. Figures 5.a-h show the typical microstructures obtained on gas nitrided surfaces by manufacturers III-VIII, and Figure 6. gives the

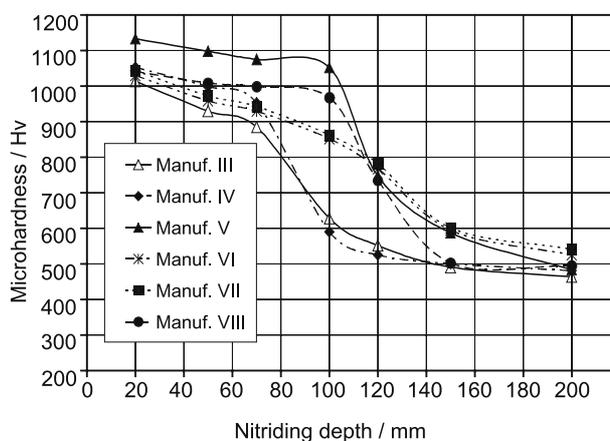


Figure 6. Microhardness of gas nitrided dies of manufacturers III - VIII

Slika 6. Mikrotvrdoća plinski nitriranih alata proizvođača III - VIII

values of the measured microhardness profiles. The results of phase analysis (XRD) for some microstructures with a compound layer as shown in Figures 5.a-h are given on Figure 7.a, but the most important feature is difference in the quantity of  $\text{Fe}_3\text{N}$  ( $\epsilon$ ) and  $\text{Fe}_4\text{N}$  ( $\gamma'$ ) phases and their quantitative ratio. It is known that the  $\epsilon$  phase is hard and remarkably brittle, while the  $\gamma'$  phase is hard and tough [15, 22] and that the usefulness of the compound layer depends on two factors; the homogeneity (higher ductility) of the compound layer (structure) and its thickness. Thus the optimum will be obtained when compound layer is mono-phased ( $\epsilon$  or  $\gamma'$ ) and has the minimum thickness, which is necessary to meet specific property requirements such as wear resistance. Influence of various  $\epsilon/\gamma'$  ratio (mixed structure) of the compound layer on its ductility is not known. A mixed structure of compound layer could accelerate the scaling of the compound layer already in the starting phase of extrusion, and negatively contribute to the wear resistance of the nitrided surface [24].

From manufacturer III we obtained in one case a very thick compound layer (cca. 15 - 18  $\mu\text{m}$ , die 5, Figure 5.a), in a second case (cca. 8 - 10  $\mu\text{m}$ , die 6, Figure 5.b) and

in a third case no compound layer at all on the nitrided surface (die 7). Microhardnesses did not exceed a value of 1000 Hv, and the depth of nitriding amounted to cca. 120  $\mu\text{m}$ . Phase analysis by XRD (Figure 7.a) of the nitrided surfaces of dies 5 and 6 confirmed the presence of  $\varepsilon$  and  $\gamma'$  phases, with a higher content of  $\varepsilon$  phase.

of the nitrided surface of dies 8 and 9 showed the presence of  $\varepsilon$  and  $\gamma'$  phases, and the ratio  $\varepsilon/\gamma'$  was in favour of the  $\varepsilon$  phase and greater to that of manufacturer III. A general characteristic of the microstructures of nitrided surfaces from manufacturers III and IV was a noticeable mesh of iron nitrides on the grain boundaries.

Diffractometer type: PW 1840 Generator tension: 40 / kV Start angle (2 theta): 20 ° Step (2 theta): 0,026 ° Divergence slit: 1,000  
Tube anode: Cu Generator current: 30 / mA End angle (2 theta): 120 ° Time of steps: 15 / s Receiving slit: 0,2

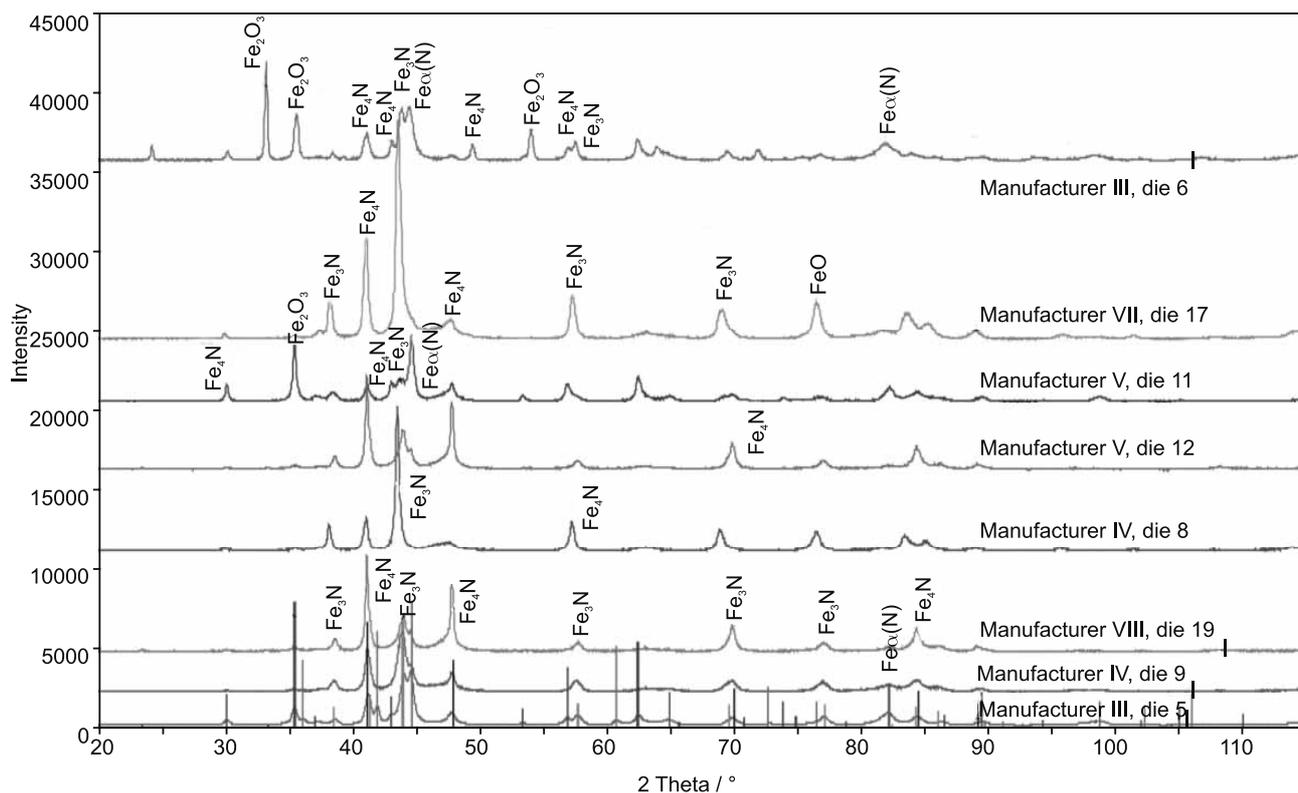


Figure 7.a XRD patterns of gas nitrided surface on dies of manufacturers III - VIII  
Slika 7.a XRD analiza površina plinsko nitriranih alata proizvođača III - VIII

From manufacturer IV we also detected variations in the thickness of the compound layer (Figures 5.c-d, dies 8-10), which on average was thinner than from manufacturer III. A difference in the microstructures of the nitrided layers was also noticeable between the 1 and 4 mm gaps of the same die: inside the 4 mm gap we obtained a 3 - 7  $\mu\text{m}$  thick compound layer (die 9, Figure 5.d), but inside the 1 mm gap a cca. 10  $\mu\text{m}$  thick compound layer (die 8, Figure 5.c). These differences in the thickness of the compound layer (of the same die) are most probably due to differences in gas mixture flow inside the 1 and 4 mm gaps, and consequently the gradients of the influential parameters (gas concentrations, temperature, etc.) were different. Microhardnesses attained a value of cca. 1050 Hv, which was a little higher than from manufacturer III (cca. 1000 Hv), and the nitriding depth extended up to cca. 130  $\mu\text{m}$ . On die 10 the compound layer was not detected. The XRD phase analysis (Figure 7.a)

From manufacturer V we obtained a similar picture as with manufacturers III and IV, that is to say, dies 11 (Figure 5.e) and 12 had a compound layer, with a thickness of 13 - 15  $\mu\text{m}$  and 6 - 9  $\mu\text{m}$ , respectively), but dies 13 and 14 were without a compound layer. The main characteristic of the nitrided surfaces from this manufacturer was that the nitriding depths and the maximum microhardnesses on die surfaces were a little higher than from other manufacturers, providing a good support to the compound layer on loading. Furthermore, on the nitrided surface of the fourth die of the same manufacturer (die 14) there was no mesh of iron nitrides on the grain boundaries (Figure 5.f). XRD phase analysis (Figure 7.a) of nitrided surfaces of dies 11 and 12 confirmed the presence of  $\varepsilon$  and  $\gamma'$  phases; the quantity of  $\gamma'$  phase was a little higher and the phase ratio  $\varepsilon/\gamma'$  was lower in comparison to that of manufacturers III and IV and this is an indication of the lower brittleness of the compound layer.

Manufacturers VI (dies 15 - 16) and VII (dies 17 - 18) nitrided only two dies each. Common characteristics of the microstructures obtained are their similarity regarding the thickness of the compound layer and the nitriding depth, as

parameters, including the  $\varepsilon/\gamma'$  ratio. Furthermore, noticeable differences in  $\varepsilon/\gamma'$  ratio and especially in  $\text{Fe}_\alpha(\text{N})$  between 1 mm and 4 mm gap were found also on same die as it is presented on Figure 7.b. The observed differences in the

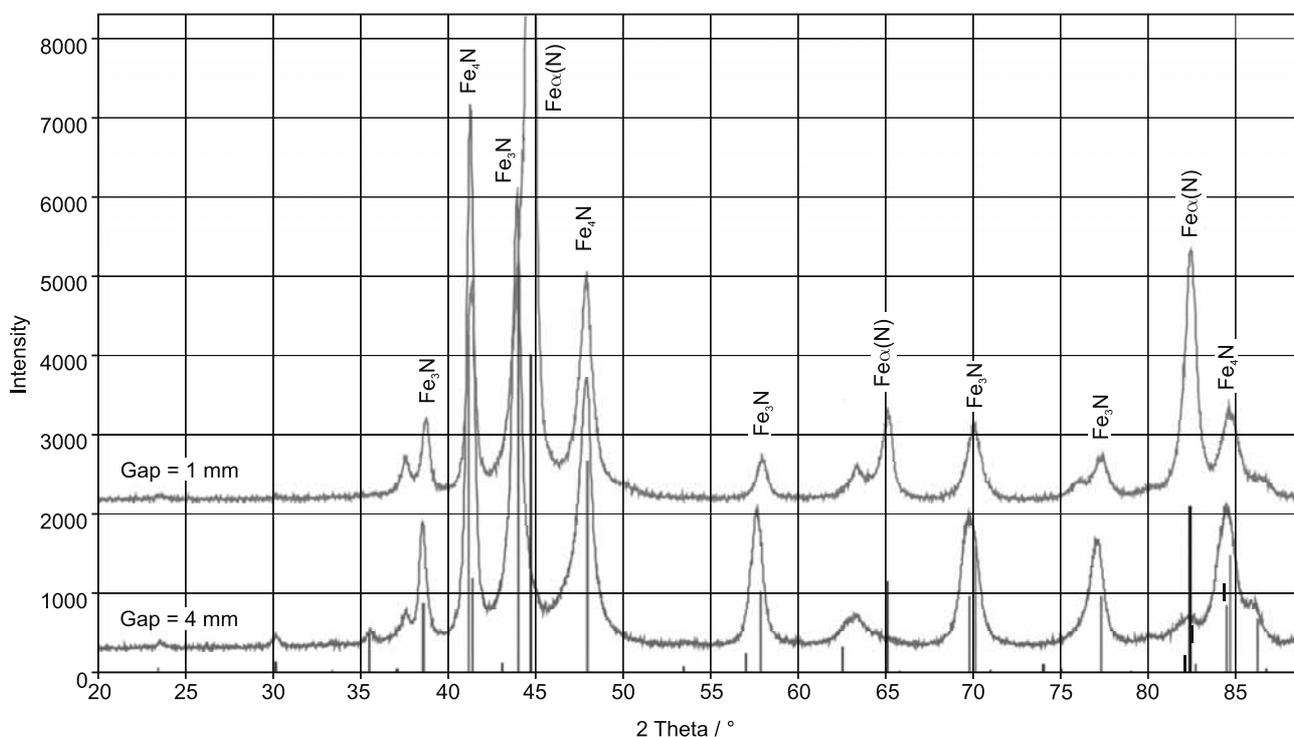


Figure 7.b Comparison between XRD patterns of gas nitrided surface in 1 mm and 4 mm gap of die  
Slika 7.b Usporedba između XRD primjeraka plinskog nitiranja 1 mm i 4 mm istog alata

well as their measured microhardness profile (Figure 6.). Namely, both manufacturers obtained a compound layer thickness of cca. 10  $\mu\text{m}$  (Figure 5.g) for the first die and cca. 6  $\mu\text{m}$  on the second die. The nitriding depth reached a value of cca. 150  $\mu\text{m}$ . XRD phase analysis (Figure 7.a) of the nitrided surfaces on dies 15-18 confirmed the presence of  $\varepsilon$  and  $\gamma'$  phases but with the highest content of  $\varepsilon$  phase (close to mono-phased) of all the manufacturers.

In the case of manufacturer VIII (dies 19 - 20), the thickness of the compound layer amounted to cca. 12  $\mu\text{m}$  for die 19 (Figure 5.h) and cca. 8  $\mu\text{m}$  for die 20. XRD phase analysis (Figure 7.a) of die 19 also confirmed the presence of  $\varepsilon$  and  $\gamma'$  phases, with a higher share of  $\varepsilon$  phase. The  $\varepsilon/\gamma'$  ratio was similar to that of manufacturer V.

We can conclude that the nitriding depth and maximal values of microhardness are relatively similar on dies nitrided by the same manufacturer, while differences occur with regard to the presence or absence of the compound layer and its thickness. The highest microhardness and nitriding depth values were exhibited by the dies of manufacturer V, which also showed the lowest  $\varepsilon/\gamma'$  ratio. The differences between manufacturers are considerable in all the mentioned

nitrided microstructures are in general a consequence of the choice of nitriding parameters, but the variations within the same manufacturer probably originate from the different conditions (parameters) at the micro and macro level inside the nitriding equipment (gas flow in furnace).

## TESTING OF WEAR RESISTANCE

In order to evaluate the wear resistance of the nitrided surfaces (blocks), a "block on cylinder" test equipment (Figure 8.) was applied, where the rotating cylinder (dimensions:  $\varnothing$  146 mm  $\times$  35 mm) made of Al-alloy represents the extruded part, and the block (30 mm  $\times$  30 mm  $\times$  20 mm) represents the die [21]. The chemical composition of the Al cylinder is presented in Table 3. and that of the tested die steels in Table 1. The Al-cylinder, which is tightly inserted between two Cu-discs, is inductively heated to the desired temperature, measured and controlled by a point pyrometer on the Cu-disc. The thin Cu-discs (Cu has a high radiation coefficient) allow good control of the temperature of the Al-cylinder and prevent any plastic deformation of the Al-cylinder at the sides due to high contact pressures. An inert

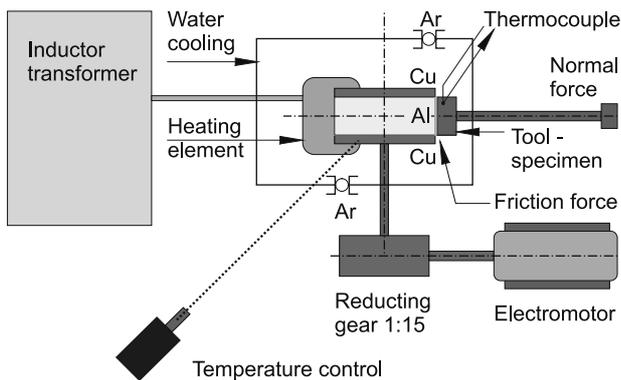


Figure 8. Applied "block on cylinder" testing equipment  
Slika 8. Upotrebjeni "blok na cilindru" ispitni uređaj

atmosphere preventing Al oxidation is achieved by a supply of argon (chemical composition: O<sub>2</sub> max 5 vppm, H<sub>2</sub>O max 10 vppm, CO<sub>2</sub> max 0,5 vppm, N<sub>2</sub> max 20 vppm; vppm is volumetric parts per million) into the chamber.

Table 3. Chemical composition of the AA 6063 Al-alloy cylinder  
Tablica 3. Kemijski sastav AA 6063 cilindričnih uzoraka

Mg	Si	Fe	Mn	Al
0,50	0,50	0,19	0,05	rest

During wear testing our observation was focused on the time of spalling of the compound layer from the die surface and consequently on the reaction of the exposed surface with hot aluminium. The process of spalling of

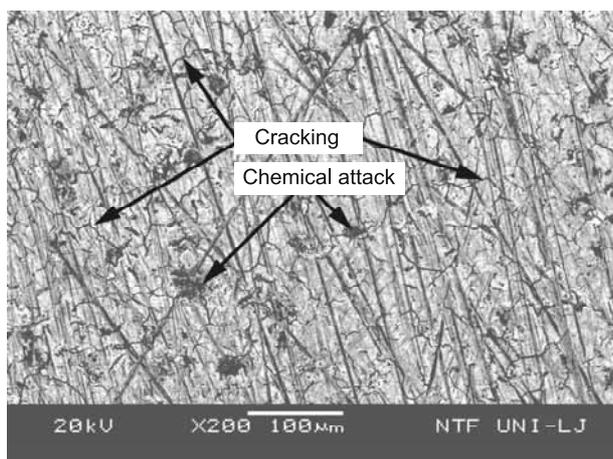


Figure 9. Appearance of cracking of compound layer before its spalling and beginning of chemical reaction with hot Al in some cracks,  $T_{Al} = 510\text{ }^\circ\text{C}$ ,  $v = 25\text{ m/min}$ ,  $F_N = 2200\text{ N}$ , argon atmosphere, gas nitrided, direction ↓, JEOL JSM 5610  
Slika 9. Pojava pukotina na bijelom sloju prije ljuštenja i početak kemijske reakcije s vrućim Al u nekim pukotinama,  $T_{Al} = 510\text{ }^\circ\text{C}$ ,  $v = 25\text{ m/min}$ ,  $F_N = 2200\text{ N}$ , argon atmosfera, plinski nitrirano, smjer klizanja ↓, JEOL JSM 5610

compound layer began with its cracking as shown on Figure 9.; beginning of a process of chemical reaction with hot Al in some cracks is also visible. The surfaces of the tested blocks were deliberately made more coarse (rough - whetting with paper,  $R_a \sim 0,21\text{ }\mu\text{m}$ ) since we wanted to match the surface roughness of real nitrided dies of a specified producer of extruded profiles. On Figure 10. a

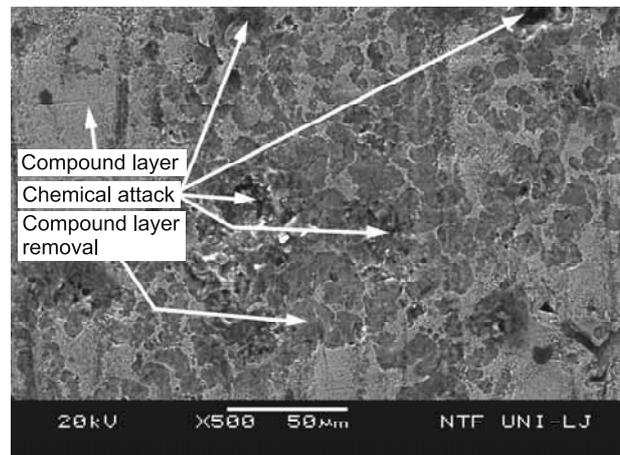


Figure 10. Typical SEM image of the tribologically loaded surface after compound layer removal,  $T_{Al} = 510\text{ }^\circ\text{C}$ ,  $v = 25\text{ m/min}$ ,  $F_N = 2200\text{ N}$ , argon atmosphere, gas nitrided, sliding direction ↓, JEOL JSM 5610  
Slika 10. Tipičan SEM prikaz tribološki opterećene površine poslije uklanjanja bijelog sloja,  $T_{Al} = 510\text{ }^\circ\text{C}$ ,  $v = 25\text{ m/min}$ ,  $F_N = 2200\text{ N}$ , argon atmosfera, plinski nitrirano, smjer klizanja ↓, JEOL JSM 5610

typical example of reaction of nitrided surface at the site of compound layer removal is presented; accelerated chemical attack by hot aluminium on the exposed die surface is visible. This can be explained by the irregular thickness of the compound layer leading to increased roughness [25] (after compound layer removal) and consequently to an increase of the active surface for chemical reactions with hot aluminium. These chemical reactions (pitting) were scarcely visible at the sites where the compound layer was not removed which indicates that the compound layer is chemically more stable against hot aluminium in comparison to the nitrided surface. It also proves the occurrence of a lower frequency of pitting on nitrided surfaces without compound layer after the nitriding process. Table 4. shows the approximate time of compound layer removal (as shown

Table 4. Approximate time of partial removal of white layer of some dies  
Tablica 4. Približno vrijeme za djelimično uklanjanje bijelog sloja s nekih alata

	Man. III: Die 5, Man. VII: Die 17	Man. IV: Die 8, Man. VIII: Die 19	Man. V: Die 11
Time	1h	2h	3h

in Figure 10.) for some manufacturers and testing periods. The best results with regard to the time of compound layer spalling were exhibited by the microstructures presented in Figures 5.c-d (manufacturers IV) and Figure 5.e of manufacturer V. Despite of mixed structure of the compound layer at manufacturer V (die11) the process of spalling was observed after two hours of testing. The reasons are probably due to higher values of microhardness and of nitriding depth at manufacturer V in comparison to the other manufacturers. Combination of low nitriding depth and microhardness value with thick and mixed structure of compound layer is more prone to earlier spalling of compound layer and vice versa (Table 4).

In the later phase of wear testing the formation of craters occurred (similar as presented in Figures 2.) that were the starting points for formation of grooves especially at the site where the craters were more frequent. For die 11 these processes occurred after eight hour of testing. At other dies the mentioned behavior was observed sooner depending on the time of the removal of compound layer and microhardness profile.

Due to the higher chemical stability of the compound layer against hot aluminium, its retention on die surfaces is desirable. Earlier spalling of compound layer thus leads to an accelerated wear process and consequently to a decreased die life. On the basis of the above results we could claim that there are still reserves in prolongation of service life of gas nitrided extrusion dies by better combination of nitriding depth, microhardness, structure of compound layer (mono-phased is desired) and its thickness.

## CONCLUSIONS

For elucidation of efficiency of gas and ionic nitriding in narrow gaps, special dies with 1 and 4 mm wide and 12 mm deep gaps were made. These dies were than nitrided by eight different manufacturers of equipment for gas and ionic nitriding. Analysis of the microstructures on nitrided surfaces showed the relatively non-effectiveness of nitriding by ionic nitriding and the effectiveness of gas nitriding. The major differences between the microstructures obtained relate to the presence or absence of the compound layer, its thickness and  $\varepsilon/\gamma'$  ratio, the nitriding depth, as well as its measured microhardness profile. The majority of these differences were also present on dies nitrided by the same manufacturer. The variety of nitrided microstructures originate from differences in the values of the nitriding parameters in the furnaces at the macro as well at the micro level. Wear tests revealed that the compound layer protects the die surface from chemical reaction with hot aluminium. In the case of compound layer removal, accelerated wear process (pitting) begins on the site of exposure. The characteristics of the nitrided microstructures, i.e. the presence or absence of a compound layer and its

thickness,  $\varepsilon/\gamma'$  ratio, and microhardness profile influence the time of compound layer removal, and consequently contribute to considerable scattering in die life during the process of hot extrusion of aluminium.

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