

Tribološke lastnosti jekla AISI 4140 nitriranega v plazmi in pulzirajoči plazmi

Tribological Properties of Plasma and Pulse Plasma Nitrided AISI 4140 Steel

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V raziskavi so bile tribološke lastnosti jekla AISI 4140 (42CrMo4), nitriranega v plazmi in pulzirajoči plazmi, določene za primer suhega drsenja. Pri tem je bilo kaljenje uporabljeno kot referenca in primerjava nitriranja. Vpliv nitriranja je bil določen z uporabo metalografije, merjenjem mikrotrdote in raziskavo topografije površine, in sicer pred preskusi in tudi po njih. Tribološke lastnosti so bile določene na napravi "valjček-disk", pri čemer je bil določen tudi vpliv drsne hitrosti in obremenitve na tribološke lastnosti jekla po nitriranju.

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(Ključne besede: jeklo AISI 4140, nitriranje plazemsko, lastnosti tribološke, obraba)

In our study the friction and the wear behaviour of plasma and pulse plasma nitrided AISI 4140 (42CrMo4) steel was evaluated under dry sliding conditions, where hardened samples were used as a reference. The nitrided samples were fully characterised before and after the wear testing using metallographic, microhardness and surface examination techniques. After surface characterisation, dry sliding wear tests were performed on a pin-on-disc machine in which hardened ball bearing steel discs were mated to nitrided pins. The influence of sliding speed and contact load on the response of the surface treated pins was determined.

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0 UVOD

Nitriranje je termokemični postopek, ki se uporablja za izboljšanje drsnih, protiobrabnih in korozijskih lastnosti površin. Postopek nitriranja temelji na difuziji dušika v jeklo, kjer se veže z legirnimi elementi in tvori nitride. Nastali nitridi povzročijo nastanek tlačnih napetosti in s tem utrditev površine. Sorazmerno nizke temperature nitriranja, običajno med 480 in 570 °C, omogočajo poboljšanje in utrditev elementov z minimalno verjetnostjo nastanka deformacij ter sprememb izmer nitriranega elementa. V primerjavi s tradicionalnimi termičnimi in kemotermičnimi postopki, kakršna sta kaljenje in ogljičenje, nitriranje zmanjša, v nekaterih primerih pa celo odpravi potrebo po dragih poznejših fazah obdelave površine ([1] in [2]).

Med postopkom nitriranja prihaja do nastanka difuzijske plasti, pri običajnem plinskem nitriranju pa tudi do nastanka tanke spojinske ali bele plasti na sami površini. Spojinska plast, sestavljena iz faz γ' (Fe_4N) in ϵ ($\text{Fe}_{2,3}\text{N}$), je zelo tanka, običajno ne debelejša od 25 μm , in vsebuje velike notranje napetosti [3]. Čeprav ima spojinska plast relativno veliko trdoto, do 1200 HV [4], je zaradi velikih notranjih napetosti krhka in drobljiva, kar pogosto privede do njenega kršenja v tribološkem dotiku

0 INTRODUCTION

Nitriding is a ferritic thermochemical treatment used to enhance the performance in terms of fatigue and wear resistance of very highly stressed mechanical components. During the treatment, nitrogen is supplied to the surface of the steel and diffuses into the material where it combines with alloying elements to form surface layers with high hardness. Therefore the response of a steel to the nitriding treatment depends principally on its composition and process temperature. The relatively low temperature of nitriding, usually in the range 480 to 570°C, allows for fully stabilised, hardened and tempered components to be surface hardened with only minimum risk of distortion and dimensional variations. In comparison to traditional thermo and thermochemical surface treatments such as hardening and carburizing, nitriding allows for the reduction or even elimination of the need for a subsequent expensive machining phase ([1] and [2]).

Conventional nitriding causes the formation of a relatively thick diffusion layer with fine dispersion of alloy nitrides as well as a thin compound or white layer on the component surface. The white layer, consisting of a heterogeneous mixture of γ' (Fe_4N) and the ϵ ($\text{Fe}_{2,3}\text{N}$) phase together, is clearly undesirable [3]. It is very hard (1200HV) and contains high internal stresses, thus making it brittle and friable [4]. Therefore such layers have to be removed

med samim delovanjem elementa. Prav zaradi tega je spojinska plast nezaželena in jo je treba pred uporabo odstraniti z delovne površine.

Nitriranje v plazmi in proces ionske vsaditve [5] omogočata nitriranje površin brez nastanka spojinske plasti, zaradi česar je mogoča takojšnja uporaba nitriranih elementov brez potrebe po dodatni obdelavi površine. Nitriranje v pulzirajoči plazmi, najnovejši proces nitriranja, pa omogoča še nižje temperature in bolj natančno vodenje procesa nitriranja.

Namen predstavljenega dela je bil raziskati tribološke lastnosti jekla AISI 4140, nitriranega v plazmi in pulzirajoči plazmi ter jih primerjati z lastnostmi jekla po kaljenju. Za razmere suhega drsenja smo raziskali tudi vpliv spojinske ali bele plasti na obrabno obnašanje jekla po nitriranju.

1 PRESKUSI

1.1 Priprava preskušancev

Za izdelavo preskušancev smo uporabili orodno jeklo AISI O2 (90MnCrV8) in jeklo za poboljšanje in nitriranje AISI 4140 (42CrMo4). Jeklo AISI O2 smo zaradi njegove široke namembnosti in pogoste uporabe v triboloških primerih uporabili za izdelavo diskov (a na sliki 1). Vsi diski so bili zaradi primerjalne narave preskusov kaljeni in popuščani na trdoto 700 HV, po toplotni obdelavi pa brušeni na stopnjo hrapavosti N5.

Valjčki, (b na sliki 1) so bili izdelani iz jekla za poboljšanje in nitriranje AISI 4140, pri čemer je kemotermična obdelava valjčkov vključevala nitriranje v plazmi in nitriranje v pulzirajoči plazmi, kot referenco pa tudi kaljenje. Parametri posamezne kemotermične obdelave valjčkov so prikazani v preglednici 1. Pred nitriranjem so bili valjčki poboljšani na trdoto 300 HV in s čelne strani brušeni na stopnjo hrapavosti N5.

by chemical or mechanical means from load-bearing surfaces before the components can be used in service.

Plasma nitriding [5] permits a fully automated and controlled nitrogen-diffusion process, which makes it possible to nitride the steel surfaces without compound or white layer formation. The most recent nitriding process, called pulse plasma nitriding, allows for even lower temperatures of the work pieces as well as shortening the time of nitriding.

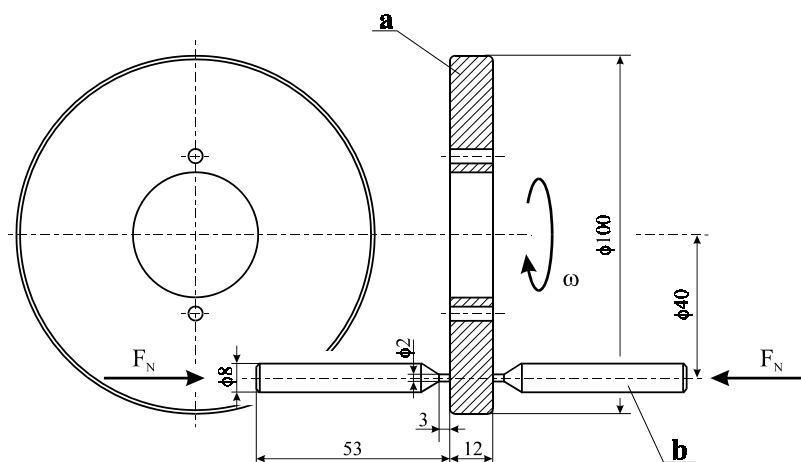
The purpose of this paper was to study the wear properties of plasma and pulse plasma nitrided AISI 4140 steel surfaces in comparison to hardened surfaces, as well as to determine the influence of the compound or white layer on wear behaviour under dry sliding conditions.

1 EXPERIMENTS

1.1 Preparation of samples

The materials used for this study were AISI O2 steel (90MnCrV8) and AISI 4140 steel (42CrMo4). Throughout the series of wear tests, disc samples produced from ball bearing steel AISI O2 were used, Figure 1a. The discs were quenched and tempered to give a hardness of 700 HV and ground to an average roughness value N5.

Pin samples, Figure 1b, were made from steel for hardening and nitriding AISI 4140, and were ground in order to obtain the desired shape and surface roughness (N5). One group of pin specimens was plasma nitrided, another batch was pulse plasma nitrided, while a third group was hardened and used as a reference. The details of the nitriding and hardening processes used in this study are listed in Table 1. All samples prepared for nitriding were vacuum hardened prior to nitriding to obtain a surface hardness of ~ 300 HV.



Sl. 1. Shematičen prikaz modelnega preskuševališča "valjček-disk"; a - disk, b - valjček
Fig. 1. Schematic representation of the pin-on-disc test configuration; a - disc, b - pin

1.2 Tribološki preskusi

Tribološke preskuse smo opravili na modelnem preskuševališču "valjček-disk", pri katerem kemotermično obdelana valjčka pritiskata ob rotirajoči disk (slika 1). Pred preskušanjem, ki je potekalo v razmerah suhega drsenja ter relativni vlažnosti zraka ~50% in sobni temperaturi ~20°C, so bili preskušanci očiščeni v ultrazvočni kopeli. Določitev triboloških lastnosti raziskovanega jekla je vključevalo preskušanje pri dveh drsni hitrostih, 0,1 in 1 ms⁻¹, ter treh obremenitvah 60, 80 in 100 N. Med preskusi smo spremljali tako spremembo sile trenja kakor tudi vzdolžno obrabo valjčkov.

Po končanem preskusu, ki je v odvisnosti od razmer pri preskušanja in kemotermične obdelave preskušancev trajal od 30 sekund do 5 ur, smo analizirali obrabljeno površino ter nastale obrabne delce.

2 REZULTATI IN DISKUSIJA

2.1 Lastnosti površine

Na sliki 2 je prikazana metalografska raziskava vpliva postopka ter parametrov nitriranja v plazmi na spremembo mikrostrukture jekla AISI 4140. Kakor je vidno s slike 2a, je osnovna poboljšana mikrostruktura preučevanega jekla visoko popuščeni bainit s karbidi v feritu, ki imajo orientacijo bainita. Pri nitriranju so se nitridi izločili deloma po mejah kristalnih zrn in deloma med karbidi v obliki drobnih zrn (sl. 2b). V primeru nitriranja v 99,4%H₂-0,6%N₂ plinski mešanici, postopek B in C, je bila na površini opazna neenakomerna spojinska ali bela plast, debeline 0,5 do 1,5 μm (sl. 2b). S povečanjem deleža dušika v plinski mešanici in povečanjem časa nitriranja

1.2 Wear tests

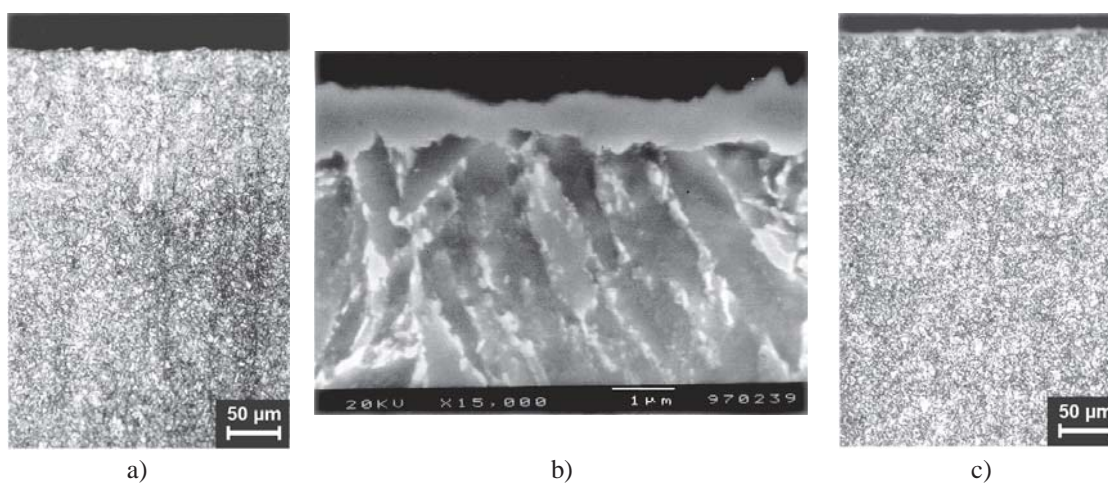
The wear experiments were performed on a pin-on-disc machine with the pin pair loaded against the disc, Figure 1. After ultrasonic cleaning, parts of pins and disc were inserted into the machine. A particular load was applied and the rotation of the disc started. Experiments were carried out at two sliding speeds of 0.1 and 1 ms⁻¹, and at each sliding speed three different loads (60, 80 and 100 N) were applied to the pins under unlubricated conditions. The atmosphere used was air with a relative humidity of ~50% and a room temperature of ~20°C. Two experimental data were recorded continuously during the sliding test: the linear wear of the pins and the friction force.

After completion of the wear test (30 s to 5 h, depending on the test conditions) the wear surfaces and generated wear debris were analysed by means of profilometry as well as by optical and scanning electron microscopy.

2 RESULTS AND DISCUSSION

2.1 Surface properties

Figure 2 shows the optical microstructures of cross-sections of plasma nitrided specimens nitrided in a 99.4%H₂-0.6%N₂ gas mixture for 17 h (Fig. 2a and 2b) and in a 75%H₂-25%N₂ gas mixture for 28 h (Fig. 2c), respectively. During nitriding, nitrogen diffuses into the steel surface and combines with alloying elements to form a diffusion zone containing a fine dispersion of nitride precipitates (Fig. 2b). In the case of plasma nitriding in a 99.4%H₂-0.6%N₂ gas mixture (treatment B and C), a nonuniform compound layer 0.5 to 1.5 μm thick was formed on the nitrided surface. In the case of plasma nitriding in a 75%H₂-25%N₂ gas mixture for 28 h (treat-

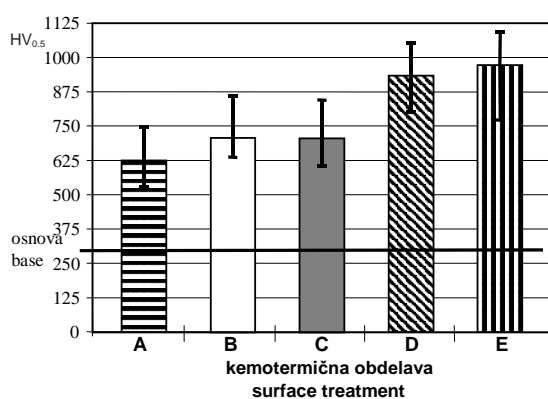


Sl. 2. Mikrostruktura jekla AISI 4140 po nitriranju v plazmi v 99,4%H₂-0,6%N₂ plinski mešanici pri 200 (a) in 15.000-kratni (b) povečavi in 75%H₂-25%N₂ plinski mešanici pri 200-kratni povečavi (c)
Fig. 2. Microstructure of plasma nitrided AISI 4140 steel, nitrided in a 99,4%H₂-0,6%N₂ gas mixture at a magnification of 200x (a) and 15.000x (b) and nitrided in a 75%H₂-25%N₂ gas mixture at a magnification of 200x (c)

Preglednica 1. Uporabljeni postopki kemotermične obdelave in njihovi parametri

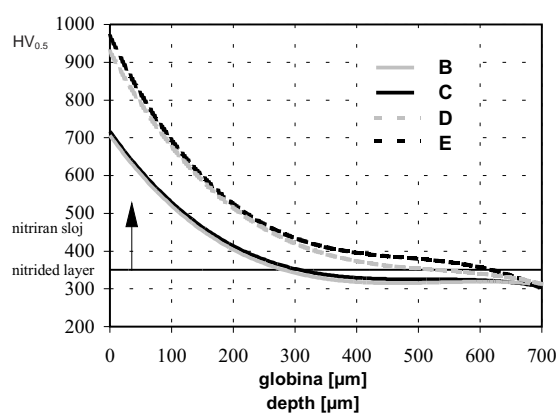
Table 1. Details of used surface treatment processes

Postopek Treatment	medij atmosphere	T °C	t h	pulz puls s	plast/layer	
					difuzijska difusion mm	spojinska compound µm
kaljenje hardening	A	olje/oil	870/250	2/1	-	-
nitiranje v plazmi plasma nitriding	B	99,4% H_2 -0,6% N_2	540	17	-	0,3
	D	75% H_2 -25% N_2	540	28	-	0,5
nitiranje v pulz. plazmi pulse plasma nitriding	C	99,4% H_2 -0,6% N_2	540	17	0,48/0,02	0,3
	E	75% H_2 -25% N_2	540	28	0,48/0,02	0,5



Sl. 3. Mikrotrdota površine v odvisnosti od kemotermične obdelave (preglednica 1)

Fig. 3. Surface microhardness as a function of surface treatment used (Table 1)



Sl. 4. Porazdelitev mikrotrdote z globino za različne postopke kemotermične obdelave (preglednica 1)

Fig. 4. Subsurface microhardness distribution for different surface treatments (Table 1)

(postopek D in E; preglednica 1), s čimer smo dosegli večjo debelino nitiranega sloja, je postala spojinska plast na površini povezana, povečala pa se je tudi njena debelina, ki je znašala 3 do 5 µm (sl. 2c). Primerjava postopka nitiranja v plazmi in nitiranja v pulzirajoči plazmi je pokazala povsem identično mikrostrukturo materiala, kar pomeni, da pulzni način nitiranja nima nikakršnega vpliva na spremembo mikrostrukture v primerjavi z običajnim nitiranjem jekla v plazmi.

Povprečne vrednosti izmerjene mikrotrdote površine ter vpliv nitiranja na njeno spremembo so v odvisnosti od postopka in parametrov nitiranja prikazane na sliki 3, medtem ko je sprememba mikrotrdote z globino prikazana na sliki 4. Tako meritve mikrotrdote površine kakor tudi njena porazdelitev z globino so bile narejene po Vickersovi metodi pri statični obremenitvi 0,5 N.

Z nitiranjem v plazmi utrdimo površinski sloj. V primeru nitiranja v plazmi in nitiranja v pulzirajoči plazmi v 99,4% H_2 -0,6% N_2 plinski mešanici, postopek B in C, se je trdota površine s 300 HV povečala na 705 HV. S povečanjem deleža dušika v plinski mešanici in podaljšanjem časa nitiranja, s čimer dosežemo večjo globino nitiranja, se je povečala tudi mikrotrdota površine kakor tudi raztros rezultatov (sl. 3). V tem primeru je bila mikrotrdota površine 935 HV po nitiranju v plazmi, postopek D, ter 970 HV po nitiranju v pulzirajoči plazmi, postopek E (sl. 3).

Za jeklo AISI 4140, nitirano v plazmi in pulzirajoči plazmi, je na sliki 4 prikazana sprememba

ment D and E; Table 1), a uniform 3 to 5 µm thick compound layer was observed on the nitrided surface with increased nitriding depth (Fig. 2c). Microstructure comparison of the plasma and pulse plasma nitrided samples showed no difference between both nitriding processes, regardless of the nitriding parameters.

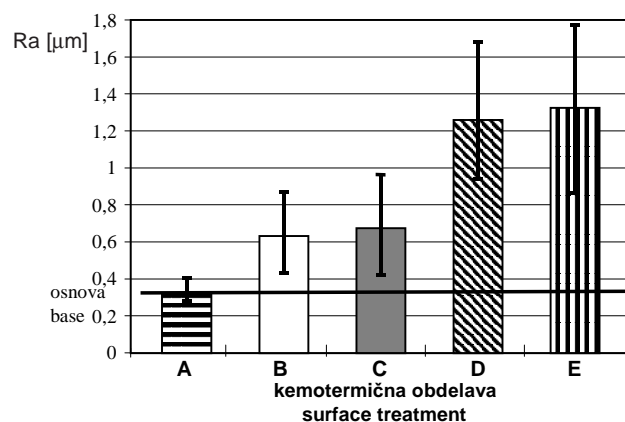
The characteristic Vickers microhardnesses, measured on the surface of the samples under 0.5 N load, are presented in figure 3. By contrast, figure 4 shows the microhardness curves plotted against the distance from the surface for plasma and pulse plasma nitrided AISI 4140 steel.

In the case of plasma as well as pulse plasma nitriding for 17 h in a 99.4% H_2 -0.6% N_2 gas mixture (treatment B and C), the surface hardness increased from 300 to 705 HV. When the nitriding parameters were changed to produce a thicker compound and diffusion layer, the surface hardness as well as scattering of the results increased. The surface hardness increased to 935 HV in the case of plasma nitriding (treatment D) and to 970 HV in the case of pulse plasma nitriding (treatment E), as shown in Figure 3.

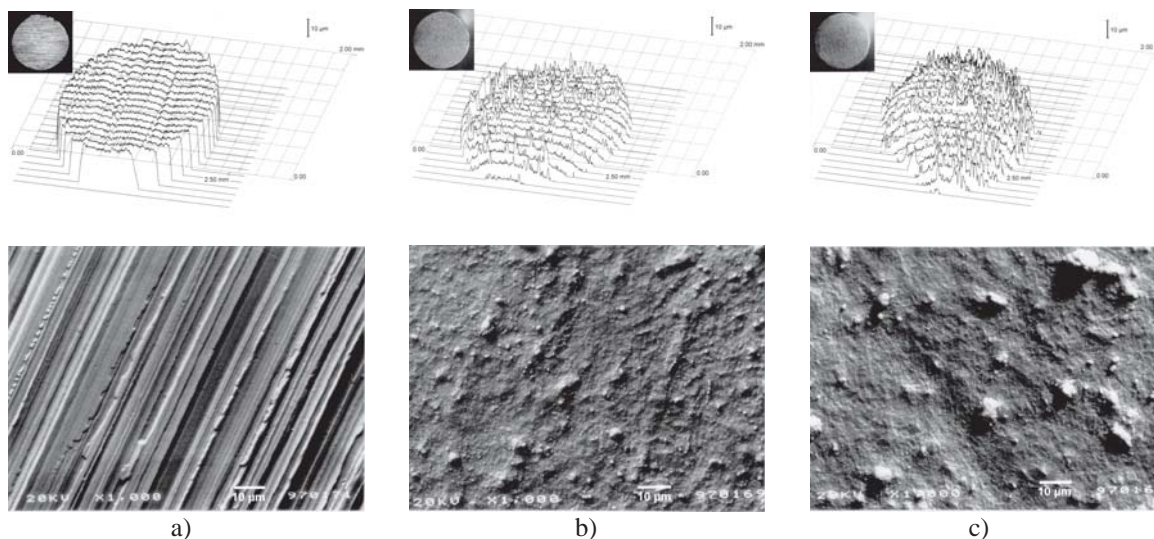
In Figure 4 the microhardness curves plotted against the distance from the surface are shown

mikrotrdote z globino, ki smo jo uporabili za določitev globine nitriranja. Pri tem je globina nitriranja določena kakor tista globina, pri kateri je trdota materiala še za 50 HV večja od trdote osnovnega materiala [6]. V vseh primerih se trdota zvezno zmanjšuje z globino. Po nitriranju v plazmi v 99,4% H_2 -0,6% N_2 plinski mešanici, postopek B, je dejanska globina nitriranega sloja enaka 290 μm , po nitriranju v pulzirajoči plazmi, postopek C, pa 310 μm . S povečanjem časa nitriranja in deleža dušika v plinski mešanici se je dejanska globina nitriranja povečala na 550 μm v primeru nitriranja v plazmi, postopek D, in na 600 μm v primeru nitriranja v pulzirajoči plazmi, postopek E. Iz rezultatov, prikazanih na sliki 3 in 4, je razvidno, da nitriranje v pulzirajoči plazmi omogoča doseganje večjih globin nitriranja kakor tudi večjih mikrotrdot površine ob rahlo večjem raztrosu rezultatov. Pri določanju triboloških lastnosti so prav tako kakor podatki o trdoti

for plasma and pulse plasma nitrided AISI 4140 steel, which was also used for determination of the nitriding depth. In all cases the hardness gradually decreases with the increase in distance from the surface. After plasma and pulse plasma nitriding for 17 h in a 99.4% H_2 -0.6% N_2 gas mixture (treatment B and C) the case depth was 0.29 and 0.31 mm, respectively, and with increased nitriding time and N_2 content in the gas mixture (treatment D and E) it increased to 0.55 and 0.6 mm, respectively. From a tribological point of view it is equally important to obtain information about surface hardness as well as about surface roughness and topography. Both surface roughness measurements and 3D topographic analysis were carried out using a stylus profilometer. The results showed that nitriding causes the increase of surface roughness, as shown in Figure 5. In the



Sl. 5. Hrapavost površine v odvisnosti od kemotermične obdelave (preglednica 1)
Fig. 5. Surface roughness as a function of surface treatment used (Table 1)



Sl. 6. Topografija površine po kaljenju in brušenju (a), nitriranju v plazmi (b) in nitriranju v pulzirajoči plazmi (c) v 99,4% H_2 -0,6% N_2 plinski mešanici
Fig. 6. Surface topography after hardening and grinding (a), and plasma nitriding (b) and pulse plasma nitriding (c) in a 99.4% H_2 -0.6% N_2 gas mixture

površine pomembni tudi podatki o hrapavosti in topografiji površine. Meritve hrapavosti in topografije površine po nitriranju v plazmi in nitriranju v pulzirajoči plazmi smo naredili z dotikalnim merilnikom hrapavosti. Po nitriranju sta se povečali tako vrednost srednjega odstopanja profila kakor tudi največja višina neravnin, kar prikazuje slika 5. Hrapavost osnovne brušene površine se je po nitriranju v 99,4% H_2 -0,6% N_2 plinski mešanici, postopek B in C, z 0,35 μm povečala na 0,65 μm , medtem ko povečanje deleža dušika in časa nitriranja, postopek D in E, privede do še večjih vrednosti hrapavosti 1,30 μm , ter večjega raztrosa rezultatov. Primerjava hrapavosti površine, nitrirane v plazmi in nitrirane v pulzirajoči plazmi, kaže večji raztros rezultatov kakor tudi rahlo večje vrednosti srednjega odstopanja profila (~3%) v primeru nitriranja v pulzirajoči plazmi (sl. 5).

Slika 6 prikazuje topografijo izvorne brušene površine (sl. 6a), kakor tudi topografijo površine po nitriranju v plazmi (sl. 6b), in po nitriranju v pulzirajoči plazmi (sl. 6c). Po nitriranju površine se na osnovni brušeni površini (sl. 6a), tvorijo koničasti vrhovi, katerih gostota in velikost sta odvisni od samega postopka in parametrov nitriranja (sl. 6b,c). S povečevanjem deleža dušika in časa nitriranja se, tako v primeru nitriranja v plazmi kakor tudi v primeru nitriranja v pulzirajoči plazmi, gostota in velikost vrhov povečata, kar se kaže tudi na povečani hrapavosti površine (sl. 5). Opazna je tudi razlika med postopkom nitriranja v plazmi in nitriranja v pulzirajoči plazmi. V primerjavi z nitriranjem v plazmi povzroči nitriranje v pulzirajoči plazmi nastanek bolj grobe površine z večjimi vrhovi neravnin (sl. 6b,c).

2.2 Tribološke lastnosti

Koeficient trenja

Izmerjene vrednosti in potek koeficienta trenja so za različne postopke ter parametre kemotermične obdelave površine valjčka prikazane na sliki 7. Slika 7a prikazuje rezultate preskušanja pri drsni hitrosti 0,1 ms^{-1} in normalni obremenitvi 80 N, medtem ko so na sliki 7b prikazani rezultati preskušanja pri enaki normalni obremenitvi in drsni hitrosti 1 ms^{-1} . Povprečne vrednosti koeficienta trenja pa so v odvisnosti od razmer preskušanja in postopka kemotermične obdelave valjčkov prikazane na sliki 8.

Neodvisno od uporabljenega postopka kemotermične obdelave površine leži koeficient trenja znotraj ozkega pasu, kakor prikazuje slika 7. Pri drsni hitrosti 0,1 ms^{-1} se vrednost koeficienta trenja giblje med 0,5 in 0,65 (sl. 7a), medtem ko je pri drsni hitrosti 1 ms^{-1} na začetku preskusa opazen skok koeficienta trenja, nakar zavzame konstantno vrednost med 0,25 in 0,3 (sl. 7b). S slike 8 je razvidno, da se s povečevanjem obremenitve poveča tudi povprečna vrednost koeficienta trenja, medtem ko ostaja njegov potek nespremenjen. Tako na potek, kakor tudi na povprečno vrednost koeficienta trenja pa v največji meri vpliva sprememba drsne hitrosti. Rezultati meritev koeficienta trenja, prikazani na slikah 7 in 8, kažejo na dva različna obrabna mehanizma pri drsni

case of nitriding in a 99.4% H_2 -0.6% N_2 gas mixture for 17 h (treatment B and C), the average roughness of the original smooth surface ($R_a = 0,35 \mu m$) increased to 0,65 μm , meanwhile nitriding in a 75% H_2 -25% N_2 gas mixture for 28 h (treatment D and E) caused an even worse situation, with the average roughness increasing to approximately 1,30 μm . Comparison of surface roughness values after plasma and pulse plasma nitriding shows larger scattering of the results as well as slightly higher roughness values (~3%) in the case of pulse plasma nitriding (Fig. 5).

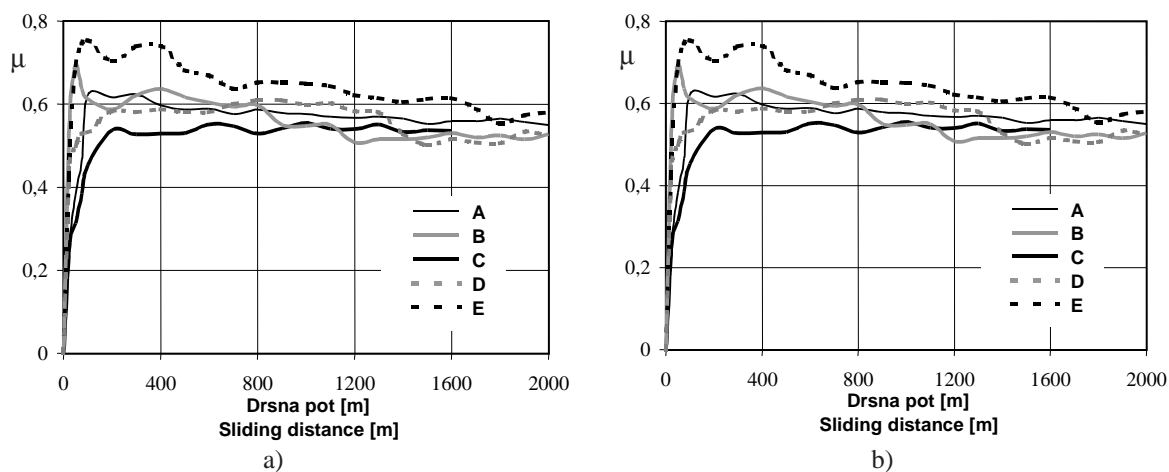
Figure 6 shows the topographic features of the original smooth surface (Fig. 6a) and the corresponding surfaces after plasma (Fig. 6b) and pulse plasma nitriding (Fig. 6c). It can be seen that nitriding causes the formation of specific topography of the surface characterised by many conical asperities. In the case of plasma nitriding (treatment B) many small conical asperities are formed at the surface. Changing the nitriding parameters to produce a thicker compound layer (treatment D) led to the formation of larger asperities with a few abnormally high ones, which also caused an increase of surface roughness (Fig. 5). The same tendency was present in the case of pulse plasma nitriding. However, compared to conventional plasma nitriding, pulse plasma nitriding caused the formation of larger asperities (Fig. 6b and 6c).

2.2 Tribological properties

Coefficient of friction

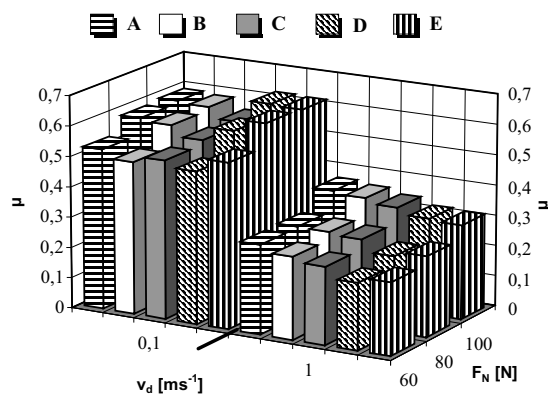
Figure 7 shows the coefficient of friction for various surface treated steel pins mated with a hardened ball bearing steel disc at a sliding speed of 0.1 ms^{-1} (Fig. 7a) and 1 ms^{-1} (Fig. 7b) and a load of 80 N. Average values of the coefficient of friction are shown in figure 8 for all surface treatment processes and different testing conditions.

Regardless of the surface treatment used, the results fall into a narrow band, thus indicating that frictional characteristics are largely independent of differences among the surface treatments used in this study (Fig. 7). In the case of low sliding speed (0.1 ms^{-1}) the coefficient of friction had a relatively constant value during the test, lying between 0.5 and 0.65 (Fig. 7a), meanwhile at a higher sliding speed of 1 ms^{-1} the coefficient of friction increased rapidly in the early part of the test before assuming a constant value between 0.25 and 0.3 (Fig. 7b). It was found that increasing the test load caused an increase in the coefficient of friction, meanwhile increasing the sliding speed caused considerable reduction of the coefficient of friction, indicating that the contact conditions had changed with sliding speed (Figs. 7 and 8).



Sl. 7. Potek koeficienta trenja pri drsni hitrosti $0,1 \text{ ms}^{-1}$ (a) in 1 ms^{-1} (b) ter normalni obremenitvi 80 N v odvisnosti od kemotermične obdelave (A do E, preglednica 1)

Fig. 7. Coefficient of friction vs. sliding distance at a sliding speed of $0,1 \text{ ms}^{-1}$ (a) and 1 ms^{-1} (b) and 80 N test load for different surface treatments used (A to E, Table 1)



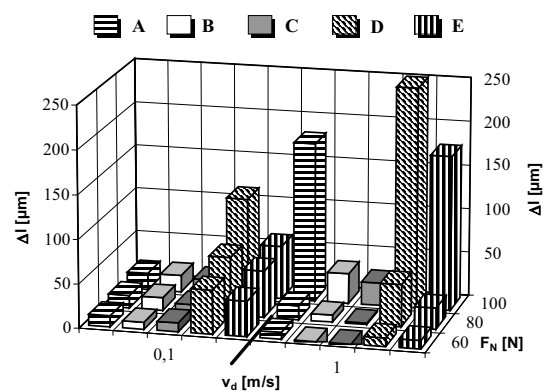
Sl. 8. Povprečna vrednost koeficienta trenja v odvisnosti od drsne hitrosti in obremenitve prikazana za različne postopke kemotermične obdelave (A do E, preglednica 1)

Fig. 8. Average coefficient of friction as a function of load and sliding speed for different surface treatments used (A to E, Table 1)

hitrosti $0,1$ in 1 ms^{-1} , medtem ko ostaja obrabni mehanizem neodvisen od obremenitve in postopka kemotermične obdelave raziskovanega jekla. V primerjavi s kaljenjem se koeficient trenja nitriranih površin zmanjša tudi za 10%, medtem ko je razlika med površinami nitriranih v plazmi in nitriranih v pulzirajoči plazmi minimalna (sl. 8). Ob tem ima spojinska plast različen vpliv na vrednost koeficienta trenja. V primeru drsne hitrost $0,1 \text{ ms}^{-1}$ povzroči spojinska plast dvig koeficienta trenja, ki celo preseže vrednosti kaljenih površin, medtem ko povečanje drsne hitrosti na 1 ms^{-1} še dodatno zniža vrednost koeficienta trenja.

Obraba

Merjenje obrabe je potekalo prek meritve pomika valjčkov, pri čemer pomeni izmerjena obraba skupno obrabo valjčkov in diska. Ker je bila obraba diska zanemarljiva v primerjavi z obrabo valjčka, smo



Sl. 9. Vz dolžna obraba para valjčkov v odvisnosti od drsne hitrosti in obremenitve prikazana za različne postopke kemotermične obdelave (A do E, preglednica 1)

Fig. 9. Linear wear of pin pair as a function of load and sliding speed for different surface treatments used (A to E, Table 1)

In relation to hardening, the nitriding process slightly decreased the coefficient of friction (up to 10%), meanwhile there was practically no difference between plasma and pulse plasma nitriding (Fig. 8). The presence of a compound layer decreased the coefficient of friction to even lower values in the case of high sliding speed (1 ms^{-1}). Whereas, in the case of low sliding speed ($0,1 \text{ ms}^{-1}$) test conditions the compound layer had a negative effect on friction, causing even higher values of the coefficient of friction than hardened surfaces.

Wear

The amount of wear was determined by the measurement of the pin lengths during each wear test, where measured wear represents the wear of the pin pair and the disc. Since the disc wear depth was very

obrado diska zanemarili in tako je izmerjena vrednost obrabe, prikazana na sliki 9, kar vzdolžna obraba valjčka.

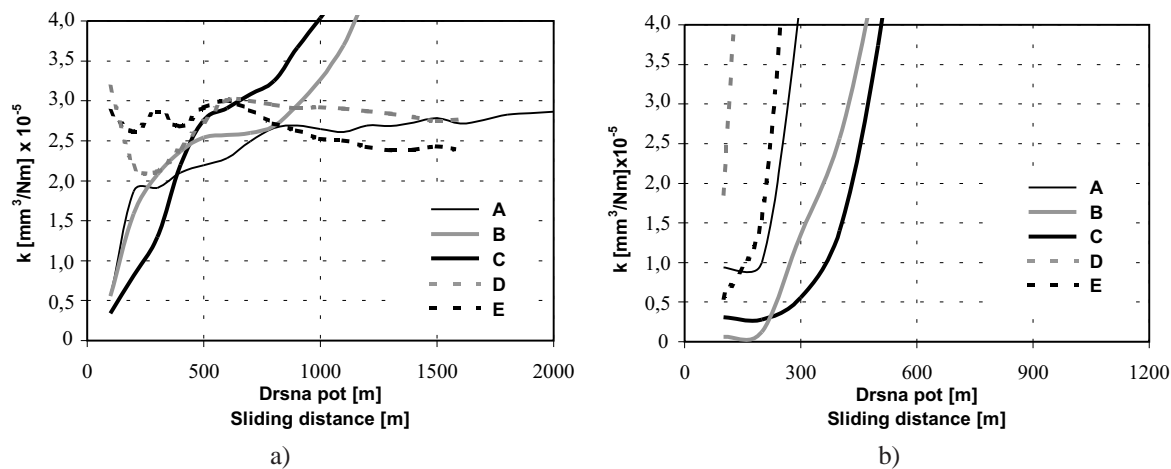
Na sliki 9 je prikazan vpliv pogojev preskušanja ter uporabljene kemotermične obdelave na obrabo valjčkov po 100 m drsenja. Razvidno je, da na obrabo valjčkov vplivajo tako razmere pri preskušanju kakor tudi postopek in parametri kemotermične obdelave. V skladu s pričakovanimi rezultati se v vseh primerih obraba z obremenitvijo povečuje, do bolj izrazite spremembe obrabe pa privede sprememba drsne hitrosti, kar kaže tudi potek koeficienta trenja (sl. 8). Primerjava kemotermičnih postopkov priprave raziskovanega jekla je pokazala, da v primerjavi s kaljenjem nitriranje v plazmi in nitriranje v pulzirajoči plazmi v 99,4% H_2 -0,6% N_2 plinski mešanici (postopek B in C) izboljšata protiobrabne lastnosti jekla AISI 4140, kar je še posebej izrazito v primeru višjih obremenitev in večje drsne hitrosti (sl. 9). Spojinska plast, ki nastane pri nitriranju v 75% H_2 -25% N_2 plinski mešanici (postopek D in E), pa zaradi svoje porozne in krhke strukture [6] celo močno poslabša protiobrabno odpornost raziskovanega jekla. Iz primerjave nitriranja v plazmi in nitriranja v pulzirajoči plazmi je vidno, da imajo površine, nitrirane v pulzirajoči plazmi, nekoliko boljše protiobrabno odpornost (sl. 9).

Vpliv postopka in parametrov nitriranja na koeficient obrabe je pri obremenitvi 80 N in drsni hitrostih 0,1 in 1 ms^{-1} prikazan na sliki 10a,b. V primeru drsne hitrosti 0,1 ms^{-1} je za valjčke, nitrirane v 99,4% H_2 -0,6% N_2 plinski mešanici (postopek B in C) značilen enakomeren potek koeficienta obrabe, medtem ko je pri valjčkih, nitriranih v 75% H_2 -25% N_2 plinski mešanici (postopek D in E), opazna močna začetna obraba, ki se s časom ustali (sl. 10a). Za preizkušanje pri drsni hitrosti 1 ms^{-1} pa je, neodvisno od postopka in parametrov nitriranja, značilno področje utekanja, ki po določenem času

small compared to pin wear it was ignored in this study, and the measured wear was taken as a representative linear wear of the pin.

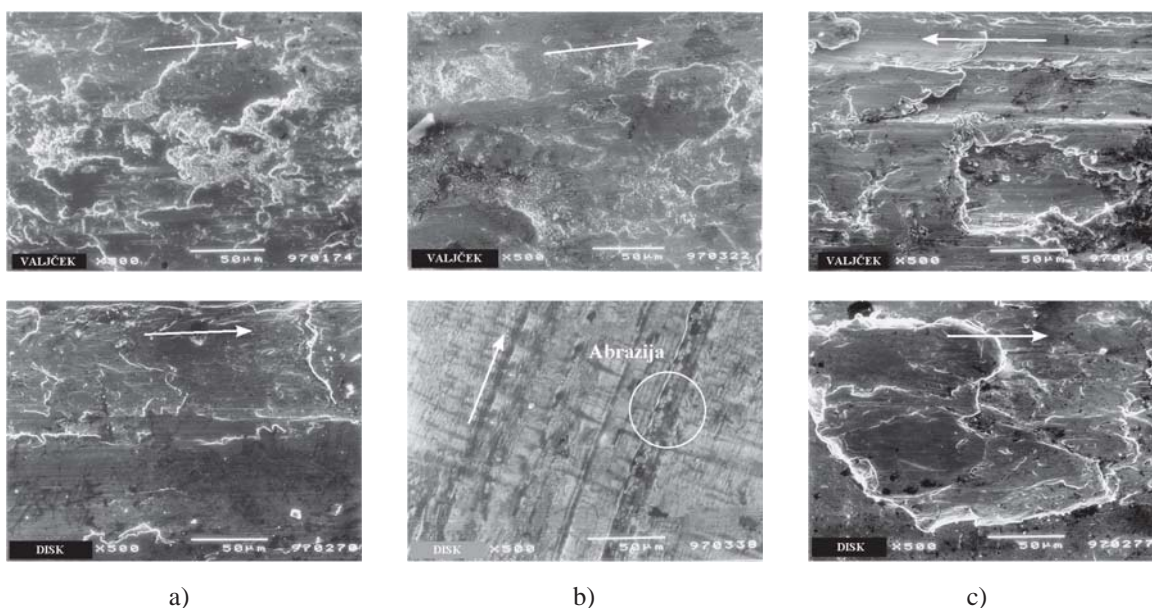
Figure 9 shows the amount of wear for different surface treated pins as a function of test load and sliding speed after 100 m of sliding. The wear increases with the test load for all surface treated samples and both sliding speeds, as expected. The results shown in Figure 9 indicate that plasma and pulse plasma nitriding (treatment B and C) improve the wear resistance of the steel surface compared to the hardened samples, which become more apparent when the test conditions become more and more severe (Fig. 9). However, nitriding in a 75% H_2 -25% N_2 gas mixture for 28 h (treatment D and E), which causes the formation of thicker and uniform compound layer over the diffusion one, even causes impairment of the wear resistance. It was found that a thicker compound layer increases the wear because of its porous and brittle structure [6]. Comparison of the wear of plasma and pulse plasma nitrided steel surfaces indicates slightly better wear resistance of the latter (Fig. 9).

Wear rate curves for different surface treated samples tested under 80 N load and at two sliding speeds 0.1 (Fig. 10a) and 1 ms^{-1} (Fig. 10b) are shown in Figure 10. In the case of low sliding speed (0.1 ms^{-1}) samples nitrided in a 99.4% H_2 -0.6% N_2 gas mixture for 17 h (treatment B and C) showed uniform wear rate over the whole test. Whereas, samples nitrided in a 75% H_2 -25% N_2 gas mixture for 28 h (treatment D and E) showed severe wear in the initial stage of sliding, followed by steady state wear behaviour (Fig. 10a). The results of testing at 1 ms^{-1} sliding speed showed initial mild wear regime in the first part of the test,



Sl. 10. Potek koeficienta obrabe pri drsni hitrosti 0,1 ms^{-1} (a) in 1 ms^{-1} (b) ter normalni obremenitvi 80 N v odvisnosti od kemotermične obdelave (A do E, preglednica 1)

Fig. 10. Wear rate vs. sliding distance at a sliding speed of 0,1 ms^{-1} (a) and 1 ms^{-1} (b) and 80 N test load for different surface treatments used (A to E, Table 1)



Sl. 11. Obrabljena površina diska in valjčka nitriranega v pulzirajoči plazmi v 99,4% H_2 -0,6% N_2 plinski mešanici (a) in v 75% H_2 -25% N_2 plinski mešanici (b) po drsnem preizkusu pri $v_d = 0,1\text{ms}^{-1}$, $F_N = 60\text{N}$, drsna pot 2000m, ter nitriranega v pulzirajoči plazmi v 99,4% H_2 -0,6% N_2 plinski mešanici po drsnem preskusu pri $v_d = 1\text{ms}^{-1}$, $F_N = 100\text{N}$, drsna pot 300m (c)

Fig. 11. Worn surface of disc and pulse plasma nitrided pin, nitrided in a 99,4% H_2 -0,6% N_2 gas mixture (a) and in a 75% H_2 -25% N_2 gas mixture (b) after a dry sliding wear test ($v_s = 0,1\text{ms}^{-1}$, $F_N = 60\text{N}$, sliding distance 2000 m), as well as of pulse plasma nitrided one in a 99,4% H_2 -0,6% N_2 gas mixture (c) after a dry sliding wear test ($v_s = 1\text{ms}^{-1}$, $F_N = 100\text{N}$, sliding distance 300 m)

preizkušanja preide v močno obrabo (sl. 10b). Tudi v tem primeru je opazen vpliv parametrov nitriranja, saj je pri površinah nitriranih v 75% H_2 -25% N_2 plinski mešanici (postopek D in E) področje utekanja minimalno in se močna obraba prične že praktično na samem začetku preskusa. Iz rezultatov je razvidno, da ima navzočnost krhke in porozne spojinske plasti negativen vpliv na protiobrabno odpornost nitriranih površin [4], [6] in [7].

Obrabni mehanizem

Pod vplivom obrabnega mehanizma pride do spremembe topografije površine. Na podlagi analize obrabljenih površin ter nastalih obrabnih delcev smo določili tudi prevladujoče obrabne mehanizme. Na sliki 11 je prikazana obrabljena površina nitriranega valjčka in pripadajočega diska, preskušanih pri drsni hitrosti $0,1\text{ms}^{-1}$ in obremenitvi 60 N (sl. 11a,b) ter drsni hitrosti 1ms^{-1} in obremenitvi 100 N (sl. 11c). Rezultati analize obrabljene površine kakor tudi izmerjene vrednosti koeficienta trenja (sl. 8) in koeficienta obrabe (sl. 10), kažejo, da je vpliv obremenitve minimalen in ne povzroča spremembe obrabnega mehanizma, temveč le spremembo intenzivnosti obrabe. Na drugi strani pa sprememba drsne hitrosti povzroči spremembo obrabnega mehanizma, in to pri vseh kemotermično obdelanih valjčkih. V primeru drsne hitrosti $0,1\text{ms}^{-1}$ (sl. 11a,b) je neodvisno od postopka in parametrov nitriranja prevladujoč obrabni mehanizem tribooksidacija [8]

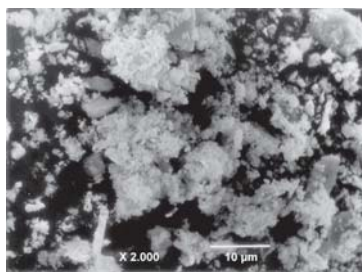
followed by a severe wear regime (Fig. 10b). In the case where a thicker compound layer was present on the surface (treatment D and E), the wear curve reached severe wear regime almost at the beginning of the sliding. These results indicate that presence of a brittle and porous compound layer decreases the wear resistance of nitrided surfaces [4], [6] and [7].

Wear mechanism

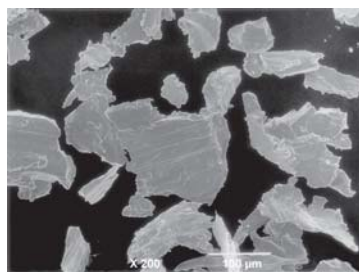
The wear process causes a change in surface topography. Therefore worn surfaces, as well as generated wear debris, were analysed in order to determine the prevailing wear mechanism. Figure 11 shows the worn surface of a plasma nitrided pin (99.4% H_2 -0.6% N_2 for 17h) as well as of the counterpart disc, after a wear test at a sliding speed of $0,1\text{ms}^{-1}$ and test load of 60 N (Fig. 11a and 11b) as well as at a sliding speed of 1ms^{-1} and test load of 100 N (Fig. 11c). It can be seen that load change did not cause a change in wear mechanism, but only caused a change in wear intensity. On the other hand, changing the sliding speed also caused the change in wear mechanism, found for all surface treated samples and confirmed by the measured coefficient of friction (Fig. 8) and wear rate (Fig. 10). In the case of $0,1\text{ms}^{-1}$ sliding speed (Fig. 11a and 11b) the prevailing wear mechanism was tribooxidation ([8] and [9]), meanwhile in the case of 1ms^{-1} sliding speed (Fig. 11c) it

in [9]), medtem ko se pri drsni hitrosti 1 ms^{-1} pojavi utrujanje površine (sl. 11c) ([8] in [10]). Oba obrabna mehanizma je potrdila tudi analiza nastalih obrabnih delcev, prikazanih na sliki 12.

Pri analizi obrabljenih površin valjkov, nitriranih v $75\% \text{H}_2$ - $25\% \text{N}_2$ plinski mešanici (postopek D in E) in pripadajočih diskov, smo ugotovili, da navzočnost spojinske plasti povzroči nastanek abrazijskih raz (sl. 11b), ki jih drugače nismo zasledili (sl. 11a). Ti rezultati potrjujejo predpostavko, da se krhka spojinska plast prične luščiti in drobiti že v začetni fazi preizkusa, čemur sledi nastajanje trdih abrazijskih delcev, ki poslabšajo dotikalne razmere med potekom preskusa.



a)



b)

Sl. 12. Obrabni delci nastali pri drsnem preskusu; (a) drsna hitrost $0,1 \text{ ms}^{-1}$ in obremenitev 60 N ; (b) drsna hitrost 1 ms^{-1} in obremenitev 100 N

Fig. 12. Generated wear debris; (a) sliding speed $0,1 \text{ ms}^{-1}$, test load 60 N ; (b) sliding speed 1 ms^{-1} , test load 100 N

3 SKLEPI

1. Raziskava jekla AISI 4140 nitriranega v plazmi in nitriranega v pulzirajoči plazmi je pokazala povsem enake mikrostrukturalne spremembe ter zelo podobne spremembe hrapavosti in mikrotredote površine neodvisno od uporabljenega postopka nitriranja.
2. V primerjavi z običajnim postopkom nitriranja v plazmi omogoča nitriranje v pulzirajoči plazmi doseganje enakih globin nitriranja v krajšem času.
3. Rezultati triboloških preskusov so pokazali, da nitriranje v plazmi in nitriranje v pulzirajoči plazmi izboljša tribološke lastnosti jekla AISI 4140, če je nitriranje izvedeno v plinski mešanici z zelo majhnim deležem dušika. S povečevanjem težavnosti preskušanja postaja izboljšanje triboloških lastnosti nitriranih površin nasproti kaljenim vedno bolj očitno.
4. Navzočnost trde in krhke spojinske plasti povzroči poslabšanje dotikalnih razmer in povečanje obrabe nitriranih površin, ki se pojavi zaradi luščenja in drobljenja spojinske plasti ter nastanka trdih abrazivnih delcev v dotiku. Zaradi tega se je treba pri realnih strojnih elementih spojinski plasti izogniti, kar dosežemo z nitriranjem v plinski mešanici z zelo majhnim deležem dušika.

3 CONCLUSIONS

1. Comparison of plasma and pulse plasma nitrided AISI 4140 steel shows no difference in the steel microstructure after nitriding, and nearly the same surface roughness and surface microhardness values.
2. Compared to conventional plasma nitriding, pulse plasma nitriding allows shorter nitriding times for obtaining the same case depths.
3. Dry sliding wear experiments showed that plasma and pulse plasma nitriding improves the tribological properties of AISI 4140 steel compared to the hardened one, which is more and more apparent when testing conditions become more severe.
4. However, the presence of a hard and brittle compound layer can even impair the contact conditions compared to hardened pins. This is due to its brittleness and fracture in the initial stage of sliding followed by the formation of hard abrasive particles. Therefore, nitriding of the steel parts should be carried out in such a way that the formation of a hard and brittle compound layer is avoided, which can be done by limiting the content of nitrogen in the gas mixture.

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