© Strojniški vestnik 47(2001)4,152-162
ISSN 0039-2480
UDK 621.793:531.4/539.62
Izvirni znanstveni članek (1.01)
© Journal of Mechanical Engineering 47(2001)4,152-162
ISSN 0039-2480
UDC 621.793:531.4/539.62
Original scientific paper (1.01)

Vpliv kemotermične obdelave podlage na tribološke lastnosti trdih prevlek

Influence of Substrate Pretreatment on the Tribological Properties of Hard Coatings

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V raziskavi so bile določene tribološke lastnosti jekla 42CrMo4, nitriranega v plazmi in prekritega z različnimi zaščitnimi prevlekami (TiN, TiAlN in DLC). Pred tribološkimi preskusi smo z elektronsko in optično mikroskopijo, z merjenjem mikrotrdote ter oprijemljivosti prevlek vrednotili dotikalno tehnično površino. Tribološke preskuse smo opravili na napravi "valjček-disk" pri suhem drsenju. Za ugotovitev vpliva nitriranja, njegove globine in spojinske plasti na tribološke lastnosti sestave prevleka-podlaga smo trde zaščitne prevleke nanesli na kaljeno jeklo in na jeklo, nitrirano v plazmi. Samo nitriranje v plazmi je bilo izvedeno v različnih razmerah.

Rezultati kažejo, da v primerjavi s kaljenjem nitriranje jekla 42CrMo4 izboljša protiobrabno odpornost nanesenih trdih zaščitnih prevlek. Poleg tega natančno voden proces nitriranja v plazmi omogoča nastanek homogene spojinske plasti, ki ima pozitiven učinek na protiobrabne lastnosti trde zaščitne prevleke.

(Ključne besede: nitriranje plazemsko, prevleke trde, drsenje, obraba)

In the research, samples made of AISI 4140 steel pre-treated by plasma nitriding and coated with different PVD coatings (TiN, TiAlN and ta-C) were investigated in terms of their microhardness, scratch adhesion and dry sliding wear resistance. Wear tests, in which duplex-treated pins were mated to hardened ball bearing steel discs, were performed with a pin-on-disc machine. To examine the influence of the nitrided zone on the performance of the coating-substrate composite, coatings were deposited on hardened as well as on plasma nitrided samples, prepared under different nitriding conditions.

The results of the investigation showed improved mechanical and tribological properties of the plasma nitrided hard-coated specimens compared to the un-coated and pre-hardened ones. Furthermore, the compound layer was found to act as an intermediate hard layer leading to superior sliding wear properties of the composite.

(Keywords: plasma nitriding, hard coatings, dry sliding, wear)

0 UVOD

Zaradi nenehnih zahtev trga po večji produktivnosti, boljši zanesljivosti in obrabni obstojnosti ter seveda donosnosti mehanskih sistemov, so komponente sistemov vedno bolj obremenjene. Svetovne zaloge osnovnih surovin in cene končnih izdelkov zahtevajo od izdelovalcev uporabo cenenih in preprosto dosegljivih materialov, ki jih je nato moč oplemenititi z najrazličnejšimi postopki toplotne obdelave in nanosa trdih zaščitnih prevlek [1]. Je pa uporaba trdih zaščitnih prevlek omejena. V primeru strojnih elementov je glavni problem zmožnost sestave prenašati obremenitev. Ker so

0 INTRODUCTION

Due to market demands for mechanical systems with improved productivity, reliability, durability and wear resistance—as well as profitability—the elements of these mechanical systems are exposed to increasingly harsh mechanical environments. However, economic constraints require that these materials are inexpensive and easily available. In order to enhance the surface properties of today's materials, producers of components are turning to different surface treatments, and in particular to hard protective coatings [1]. What limits the more widespread use of hard thin coatings, especially in the case of prevleke v večini primerov zelo tanke, debele le nekaj µm ali celo manj, mora podlaga nositi večji del obremenitve. V primeru, da podlaga tega ne zmore, lahko pride do poškodbe prevleke in s tem strojnega elementa ([2] do [4]).

V zadnjih letih postaja tehnologija oplemenitenja površine "duplex", ki združuje kemotermično izboljšanje podlage ter nanos trde zaščitne prevleke, vedno bolj pomembna [5]. Pri tem se je izkazalo, da je ena od najbolj obetajočih tehnologij prav kombinacija nitriranja podlage ter nanosa trde zaščitne prevleke iz parne faze (NZP - PVD). Ta kombinacija ne vodi le do boljše nosilne zmožnosti površine temveč tudi do izboljšane odpornosti na utrujanje, temperaturne stabilnosti ter seveda boljših triboloških lastnosti dotikalne površine ([4], [6] do [10]).

Namen predstavljene raziskave je bil poiskati možnosti uporabe trdih zaščitnih prevlek na žilavih konstrukcijskih jeklih. V ta namen smo uporabili kombinacijo kemotermične priprave podlage z nitriranjem v plazmi in vakuumski nanos trde zaščitne prevleke iz parne faze (NZP). Vpliv kemotremične priprave podlage ter parametrov nitriranja na drsne protiobrabne lastnosti trdih zaščitnih prevlek je bil določen na primeru enoplastne (TiN) in večplastne (TiAlN) keramične prevleke ter prevleke na osnovi trdega ogljika (PTO - DLC).

machine elements, is the load-carrying capacity of the coated system. Because the hard coatings are very thin the substrate must carry most of the applied load. If the substrate is not capable of supporting the coating, plastic deformation will occur, leading to premature failure of the coating ([2] to [4]).

The challenge of improving the properties of hard protective coatings with a thermo-chemical pretreatment of the substrate has gained much attention in recent years, and this has led to the development of a new method called duplex treatment [5]. A combination of plasma nitriding and physical-vapour-deposition (PVD) processes has been found to be a very attractive method of duplex treatment, not only for increasing the load-carrying capacity, but also for improving the fatigue and temperature resistance as well as the tribological behaviour of the contact surfaces ([4], [6] to [10]).

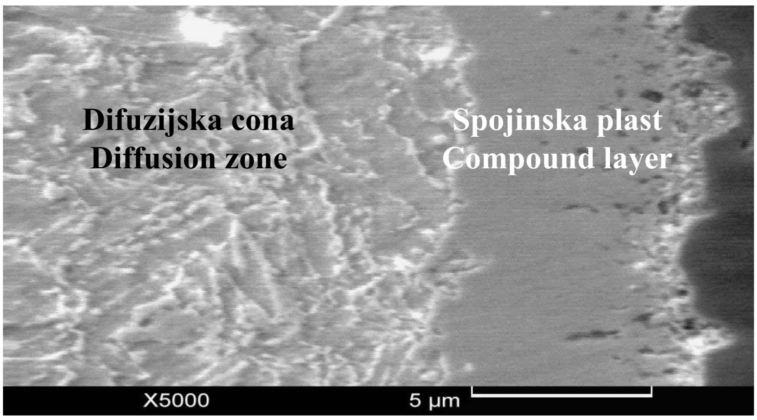
The aim of this paper was to investigate the possibilities of using hard protective coatings on structural steels. Therefore, a combination of plasma nitriding of a 42CrMo4 steel substrate and the deposition of a hard thin PVD coating was used. Different nitriding conditions were employed in order to investigate their effect on the sliding-wear properties of monolayer (TiN), multilayer (TiAlN) and a diamond-like carbon (DLC) coating.

1 PRESKUSI

Za osnovni material smo uporabili običajno konstrukcijsko jeklo 42CrMo4, pri katerem je moč, z uporabo ustreznega postopka izboljšanja, doseči relativno dobro trdoto površine ter žilavo jedro. Pred kemotermičnim izboljšanjem so bili preskušanci kaljeni in popuščani (650°C) na trdoto 300 $HV_{0.5}$ ter brušeni na stopnjo hrapavosti N5.

1 EXPERIMENTAL

The substrate material used was 42CrMo4 structural steel for hardening and nitriding. This material was chosen because it can be nitrided to a high surface hardness without losing its toughness properties, making it suitable for highly loaded machine parts. Samples were quenched and then tempered at 650°C to a mean hardness of 300 $HV_{0.5}$ and ground to a surface roughness of N5.



Sl. 1. *Mikrostruktura jekla 42CrMo4, nitriranega v plazmi (75%H_2-25%N_2)*
Fig.1. *Microstructure of plasma nitrided 42CrMo4 steel (75%H_2-25%N_2)*

Preglednica 1. *Uporabljeni kemotermični postopki in njihovi parametri*
Table 1: *Details of substrate treatment processes*

Postopek Treatment		Medij Atmosphere	Temperatura Temperature °C	Čas Time h	Globina Case depth mm
nitriranje v plazmi + γ' plasma nitriding + γ'	A	$75\%H_2$-$25\%N_2$	540	28	0,55
nitriranje v plazmi plasma nitriding	B	$99,4\%H_2$-$0,6\%N_2$	540	17	0,3
kaljenje hardening	C	olje oil	870/250	2/1	celoten prerez throughout
kaljenje hardening	D	olje oil	870/650	2/1	celoten prerez throughout

Kemotermična obdelava preskušancev je obsegala tri postopke. Prva skupina preskušancev je bila nitrirana v plazmi ($75\%H_2$-$25\%N_2$) 28 h pri temperaturi 540°C (postopek A), kar je vodilo do nastanka 5 μm debele spojinske plasti γ' ter globine nitriranja 0,55 mm (sl. 1). Druga skupina je bila nitrirana v plazmi z zelo majhnim deležem dušika ($99,4\%H_2$-$0,6\%N_2$) 17 h pri temperaturi 540°C (postopek B), s čimer smo preprečili nastanek spojinske plasti [10], [11] ter dosegli globino nitriranja 0,3 mm. Tretja skupina preskušancev je bila kaljena na trdoto 600 $HV_{0.5}$ (postopek C) ter skupaj z osnovnim materialom, izboljšanim na trdoto 300 $HV_{0.5}$ (postopek D), uporabljena kot primerjava. Detajli postopkov nitriranja in kaljenja, prikazani v preglednici 1, so bolj natančno opisani v literaturi [12].

Kemotermično izboljšani preskušanci so bili nato prekriti s trdo zaščitno prevleko TiN, TiAlN ali trdega ogljika, ki so bile nanesene po običajnem postopku PVD. Prevleki TiN in TiAlN, debeline ~ 4 μm, sta bili na kemotermično izboljšano podlago naneseni s postopkom reaktivnega naparevanja, pri čemer je bila temperatura podlage 400 do 450°C. Prevleka TiN je enoplastna prevleka prve generacije. Prevleka TiAlN, sestavljena iz ponavljajočih se plasti TiN in TiAlN debeline ~ 120 nm, pa je večplastna prevleka druge generacije [13]. Prevleka trdega ogljika (PTO - DLC), poznana tudi kot tetrahedralni amorfni ogljik (ta-C), debeline ~ 0,5 μm, je bila s pulznim obločnim naparevanjem nanesena na polirano podlago ($R_a \approx 0,02$ μm) temperature 20 do 80°C. Za zagotovitev ustrezne oprijemljivosti prevlek je bila, pred nanosom trde zaščitne prevleke, na podlago nanesena vmesna vezna plast titana debeline 30 nm.

Hrapavost površine pred nanosom in po nanosu trdih prevlek je bila izmerjena s stičnim merilnikom hrapavosti. Trdota prevlek je bila določena z metodo nano vtiskovanja [14]. Podlagi ter sestavi prevleka - podlaga pa smo trdoto izmerili z metodo Vickers pri obremenitvi 50 g. Za določitev oprijemljivosti prevleke na podlago je bil izbran preskus z razenjem [15], medtem ko so bile torne in

Three thermo-chemical processes were selected. The first group was plasma nitrided at 540°C for 28 h in a $75\%H_2$-$25\%N_2$ atmosphere (treatment A), which resulted in a diffusion zone of 0,55 mm and a dense γ' compound layer of about 5 μm, as shown in Fig. 1. The second group was bright nitrided (treatment B), realised by activating the surface and using a low nitrogen content in the plasma [11]. In this way a diffusion zone of 0,3 mm was obtained without any compound-layer formation. The third group of specimens was hardened to a surface hardness of ~600 $HV_{0.5}$ (treatment C) and together with pre-hardened samples (300 $HV_{0.5}$ - treatment D) used as a reference. The details of the nitriding and hardening processes are listed in Table 1 and described in more detail elsewhere [12].

The thermo-chemically treated samples were then coated with TiN, TiAlN or a DLC coating, deposited using commercial PVD processes. The TiN and TiAlN coatings, which were approximately 4-μm thick, were deposited using a commercial reactive-arc-evaporation technique at a substrate temperature between 400 and 450°C. Whereas the TiN coating is a single-layer coating, the TiAlN coating can best be described as a second-generation multilayer coating [13] composed of alternate TiN and TiAlN layers with individual layer thicknesses of ~120 nm. A 0,5-μm thick DLC coating, also known as tetrahedral amorphous carbon coating (ta-C), was deposited by a pulsed vacuum-arc deposition system at a deposition temperature in the range 20 to 80°C. DLC coatings were deposited on specimens, which were polished after the thermo-chemical treatment to an average surface roughness of approximately 0,02 μm. In order to ensure adequately reproducible adhesion of the coatings, all specimens were coated with a thin titanium interlayer (30 nm) prior to deposition of the coatings.

Surface roughness before and after the coating deposition was measured using stylus profilometry. The surface hardness was measured with a Vickers microhardness tester (50 g load) and nanohardness measurements [14]. The scratch test obrabne lastnosti sestave določene na napravi "valjček-disk". Pri triboloških preskusih je kemotermično izboljšan valjček, prekrit s trdo zaščitno prevleko, pritiskal ob rotirajoči disk iz orodnega jekla (90MnCrV8), izboljšanega na trdoto 700 $HV_{0.5}$ in brušenega na Ra ≈ 0,4 µm. Tribološki preskusi so bili izvedeni v razmerah suhega drsenja pri sobni temperaturi (20 °C) in relativni vlažnosti zraka 50 %, obremenitvi 60 N in drsni hitrosti 1 m/s.

2 REZULTATI IN OBRAVNAVA

2.1 Hrapavost in trdota površine

Povprečne vrednosti hrapavosti površine pred nanosom in po nanosu trde zaščitne prevleke so prikazane na sliki 2. Po nitriranju se je, glede na parametre nitriranja, hrapavost podlage povečala, kar je bilo ugotovljeno že v prejšnjih raziskavah ([12] in [16]). Nanos trde zaščitne prevleke na polirano (PTO) ali neobdelano kemotermično izboljšano podlago (TiN, TiAlN) pa ni vplival na izrazito spremembo hrapavosti površine (sl. 2).

V primerjavi s kaljenjem, nitriranje v plazmi poveča trdoto površine jekla 42CrMo4 ([12] in [16]). Po nitriranju v plazmi z zelo majhnim deležem dušika (postopek B, preglednica 1) je bila dosežena trdota površine ~ 700 $HV_{0.5}$. Povečanje deleža dušika ter podaljšanje časa nitriranja (postopek A, preglednica 1) je vodilo do večje trdote površine (~ 940 $HV_{0.5}$) in tudi do nastanka strnjene spojinske plasti (slika 1), katere trdota, izmerjena z metodo nano vtiskovanja, je bila prek 10,5 GPa. Poliranje površine pred nanosom PTO je povzročilo znižanje trdote nitrirane površine za približno 8 %, kar je razvidno s slike 3.

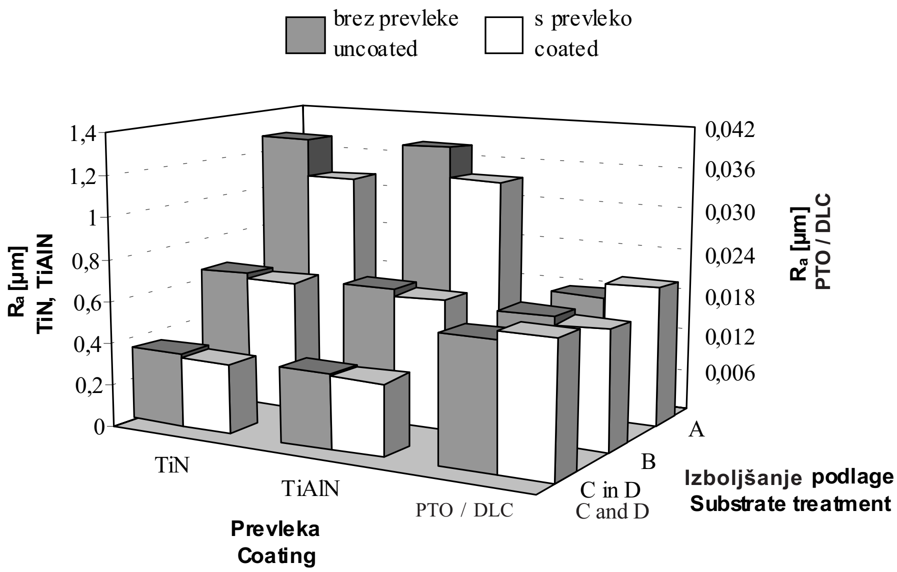
was chosen to evaluate the coating-to-substrate adhesion [15]. The wear resistance of the duplex-treated samples was tested using a pin-on-disc machine where a duplex-treated pin was loaded against an uncoated disc made of 90MnCrV8 steel, hardened to a surface hardness of 700 $HV_{0.5}$ and ground to R_a ≈ 0.4 µm. Unlubricated wear tests were carried out at a normal load of 60 N with a sliding speed of 1 m/s at room temperature (20°C) and a relative humidity of about 50%,.

2 RESULTS AND DISCUSSION

2.1 Surface roughness and hardness

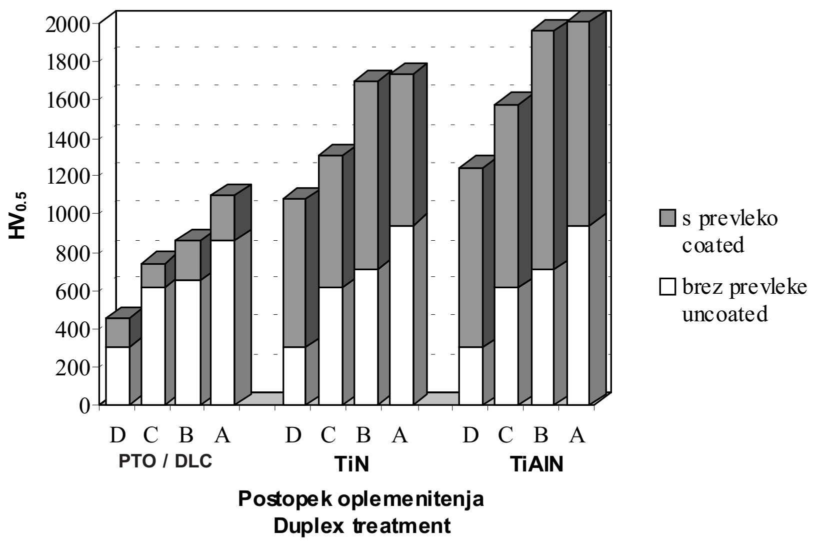
The average surface roughness of the 42CrMo4 steel samples before and after the coating deposition is shown in Fig. 2. Depending on the nitriding conditions, nitriding caused an increase in the surface roughness, as described in previous investigations ([12] and [16]). However, deposition of the PVD coatings on polished (DLC coating) or on "as thermo-chemically treated" substrates (TiN and TiAlN coatings) did not cause any measurable increase in the surface roughness, as shown in Fig. 2.

Compared to hardening, plasma nitriding increased the surface hardness of 42CrMo4 steel ([12] and [16]). In the case of plasma nitriding in a nitrogen-poor atmosphere (treatment B, Table 1) the surface had a peak hardness of ~700 $HV_{0.5}$. Increasing the nitriding time and the nitrogen concentration (treatment A, Table 1) led to a higher surface hardness of ~940 $HV_{0.5}$ and to the formation of a dense compound layer (Fig. 1), whose hardness, measured by a nano-intendation method was found to be over 10.5 GPa. Polishing of the substrate prior to the deposition of the DLC coating slightly reduced the surface hardness of the plasma-nitrided specimens, as shown in Fig. 3.



Sl. 2. *Hrapavost površine kemotermično izboljšanega jekla 42CrMo4 pred nanosom in po nanosu trde zaščitne prevleke*

Fig. 2. *Surface roughness of duplex-treated 42CrMo4 steel prior and after coating*



Sl. 3. *Trdota površine kemotermično izboljšanega jekla 42CrMo4, prekritega s trdo zaščitno prevleko*
Fig. 3. *Surface microhardness of duplex-treated 42CrMo4 steel*

Meritve trdote sestave prevleka - podlaga, izvedene z metodo Vickers, so pokazale, da nitriranje v plazmi poveča trdoto sestave, kar velja za vse tri raziskane prevleke (sl. 3). Najmanjši vpliv kemotermičnega izboljšanja podlage smo opazili v primeru PTO, kar je posledica zelo majhne debeline prevleke in relativno velike obremenitve. S povečanjem trdote podlage, nitriranje v plazmi izboljša nosilno zmožnost podlage ter zagotavlja dobro podporo prevleki [17]. Po drugi strani pa večja trdota podlage vodi do zmanjšanja gradienta trdote na meji med prevleko in podlago in s tem do manjših napetosti v prevleki med obremenitvijo ([10] in [11]).

Trdota in modul elastičnosti samih prevlek sta bila določena z metodo nano vtiskovanja. Za prevleko DLC s trdoto 70,1±7,4 GPa in modulom elastičnosti 571±51 GPa je bilo ugotovljeno, da je precej trša in bolj toga kakor prevleki TiN (H = 28,5±1,8 GPa, E = 386±26 GPa) in TiAlN (H = 31,4±2,8 GPa, E = 407±37 GPa).

Vickers microhardness measurements performed on coated specimens showed an improved composite hardness as a result of plasma nitriding the substrate: this was observed for all three coatings (Fig. 3). However, due to the low thickness of the DLC coating the substrate had a more pronounced effect on the measured hardness values of the DLC-coated specimens when compared to the TiN- and TiAl-coated specimens. By increasing the hardness of the substrate, plasma nitriding improves the load-carrying capacity of the substrate and provides good support for the coating [17] as well as reducing the large hardness gradient at the coating/substrate interface. Smoother hardness gradients would be expected to reduce stresses in the coating when it is loaded ([10] and [11]).

Nano-indentation analysis was used to determine the hardness and elastic properties of the coatings. The DLC coating with a hardness of 70.1±7.4 GPa and Young's modulus of 571±51 GPa was found to be much harder and stiffer compared to the TiN (H = 28.5±1.8 GPa, E = 386±26 GPa) and TiAlN (H = 31.4±2.8 GPa, E = 407±37 GPa) coatings.

2.2 Oprijemljivost prevleke na podlago

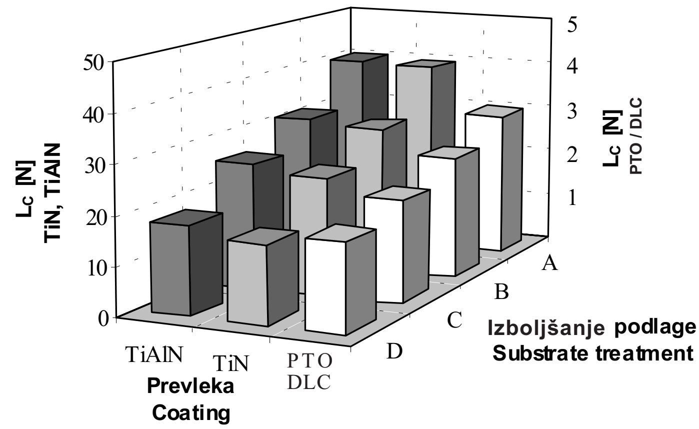
Oprijemljivost prevleke na podlago je bila določena z metodo razenja, kjer smo uporabili diamantno konico (Rockwell C) s polmerom 200 μm ter stopnjo obremenjevanja 10 N/mm. Slika 4 prikazuje kritično obremenitev L_C nastanka prvih razpok v prevleki naneseni na kemotermično izboljšano jeklo 42CrMo4.

V primeru prevlek TiN in TiAlN, nanesenih na jeklo, nitrirano v plazmi (postopek B), so se razpoke, ki so se širile od središča proti robu raze, pojavile pri kritični obremenitvi 35 N. S povečevanjem obremenitve se je pričela prevleka ločevati od podlage. Podoben mehanizem

2.2 Adhesion

A scratch tester equipped with a 200-μm radius Rockwell C diamond stylus and a loading rate of 10 N/mm was used to evaluate the coating-to-substrate adhesion. Figure 4 shows the critical load L_c for the first failure of the coating as a function of the substrate pre-treatment.

In the case of the TiN and TiAlN coatings deposited on a plasma-nitrided substrate (treatment B, Table 1) tensile cracking of the coating propagating from the bottom of the scratch towards the scratch-channel rims was observed at a critical load of approximately 35 N. Increasing the load led to coating spallation, observed at the rim of the scratch channel, and finally to complete removal of the coating. A



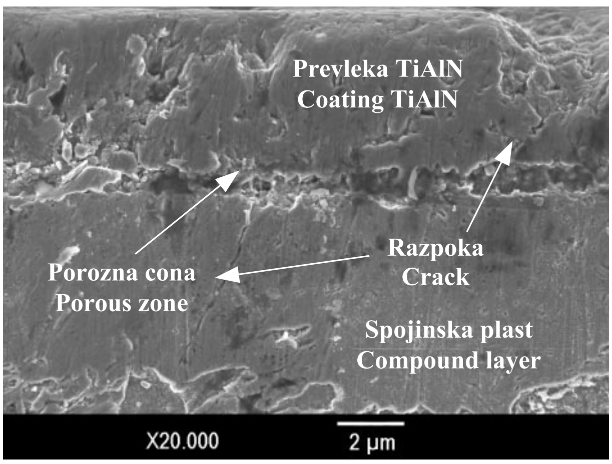
Sl. 4. *Kritična obremenitev nastanka prve poškodbe raziskanih prevlek*
Fig. 4. *Critical load for first failure of the investigated coatings*

poškodbe prevleke smo ugotovili tudi v primeru kaljene podlage (postopek C in D, preglednica 1), kjer pa so se prve razpoke v prevleki pojavile že pri precej manjši obremenitvi (sl. 4). Kritična obremenitev je bila pri PTO precej manjša kakor v primeru prevlek TiN in TiAlN. Nitriranje podlage pa je tudi v primeru PTO močno izboljšalo oprijemljivost prevleke na podlago, kakor prikazuje slika 4.

Spojinska plast, nastala med postopkom nitriranja, običajno povzroča poslabšanje oprijemljivosti prevleke na podlago, kar se pripisuje poroznosti in slabi oprijemljivosti spojinske plasti na difuzijsko cono ([8] do [11], [18] in [19]). Analiza rezultatov preskušanja z razenjem pa je pokazala, da spojinska plast, nastala pri natančno vodenem postopku nitriranja v plazmi, vedno ne vodi do poslabšanja oprijemljivosti prevleke na podlago. Še več, tanka porozna cona spojinske plasti lahko celo zavira neposredno širjenje razpok iz podlage v prevleko, kar je prikazano na sliki 5.

similar coating-failure mechanism was found for coated hardened substrates (treatments C and D, Table 1). Hardening, however, was found to decrease the critical loads when compared to plasma nitriding, as shown in Fig. 4. DLC coating was found lower when compared to the TiN and TiAlN coatings. However, as in the case of the TiN and TiAlN coatings, plasma nitriding of the substrate was found to yield increased critical loads for the DLC-coated specimens when compared to the hardened ones (Fig. 4).

It is normally claimed that the compound layer formed during nitriding reduces coating-to-substrate adhesion, and this is mainly attributed to the compound layer's porosity and its poor adhesion to the steel surface ([8] to [11], [18] and [19]). The results of the scratch-test analysis showed that although the compound layer displayed a small porous zone at the surface this did not necessarily lead to a reduced coating-to-substrate adhesion. Furthermore, it seems that the porous zone of the compound layer prevents any cracks that originate from the defects in the substrate from propagating directly into the coating, as shown in Fig. 5.



Sl. 5. *Širjenje razpoke v sestavi difuzijska plast – spojinska plast-trda prevleka*
Fig. 5. *Crack propagation in the composite diffusion zone-compound layer-hard coating*

2.3 Tribološke lastnosti

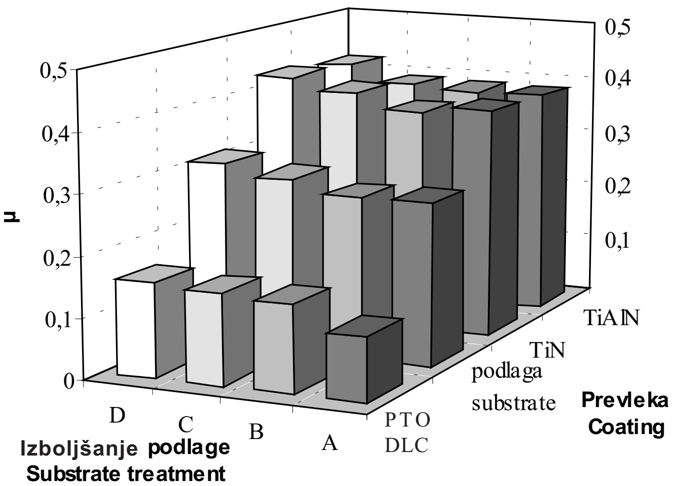
Slika 6 prikazuje povprečne vrednosti koeficienta trenja za kemotermično izboljšano jeklo 42CrMo4, katerega površino smo prekrili s trdo zaščitno prevleko. Za prevleki TiN in TiAlN se je izkazalo, da je koeficient trenja, s povprečno vrednostjo ~ 0,4, praktično neodvisen od postopka kemotermičnega izboljšanja podlage. Z nanosom PTO je koeficient trenja kemotermično izboljšanega jekla padel na vrednosti med 0,15 in 0,1, pri čemer postopek kemotermičnega izboljšanja podlage, vpliva na torne lastnosti sestave (sl. 6). V primeru kaljene podlage ter podlage nitrirane v plazmi z majhnim deležem dušika (postopek B, preglednica 1) se je koeficient trenja v začetni fazi preskušanja zvečeval, dokler ni dosegel stalne vrednosti 0,15. Najbolj enakomeren potek in najnižja vrednost koeficienta trenja (~ 0,1) sta bila dosežena v primeru PTO nanesene na nitrirano podlago s spojinsko plastjo (postopek A, preglednica 1). Zelo majhne vrednosti koeficienta trenja, dosežene v primeru prevlek DLC, je moč povezati z nastankom grafitnega sloja na dotikalni površini plošče ([20] do [23]), kar je razvidno s slike 7.

Nanos trde zaščitne prevleke je močno zmanjšal drsno obrabo kemotermično izboljšanih valjčkov, ki je odvisna od postopka kemotermičnega izboljšanja podlage (sl. 8). Drsna obrabna odpornost raziskanih prevlek se je s povečevanjem trdote podlage in zamenjavo postopka kaljenja z nitriranjem v plazmi izboljšala. Najboljšo drsno obrabno odpornost raziskanih prevlek pa smo dosegli v primeru nitrirane podlage s spojinsko plastjo (postopek A, preglednica 1). Boljšo obrabno odpornost kompozita, ki smo jo izmerili v primeru

2.3 Tribological properties

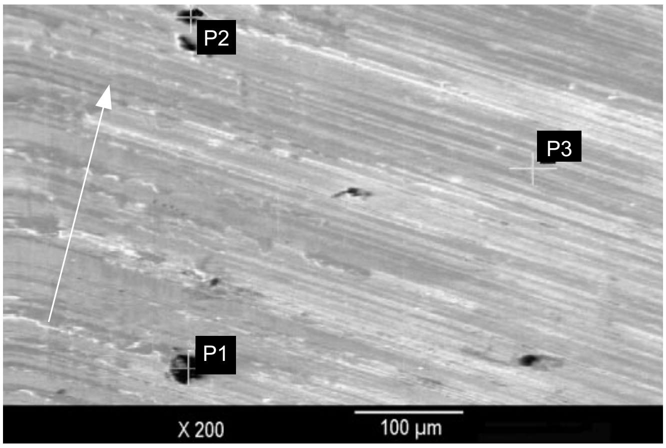
Average values for the coefficient of friction of the duplex-treated 42CrMo4 steel are shown in Fig. 6. Coating of surface-treated 42CrMo4 steel with TiN or TiAlN coatings increased the steady-state coefficient of friction from ~0,3 to ~0,4, which was found to be more-or-less independent of the substrate pretreatment used. On the other hand, deposition of the DLC coating decreased the steadystate coefficient of friction down to values between 0.1 and 0.15 (Fig. 6). In the case of the hardened-and-plasma-nitrided substrate that was nitrided in a nitrogen-poor atmosphere (treatment B, Table 1), the coefficient of friction increased during the early stages of the test before attaining a constant value of ~0.15. However, in the case of the plasma-nitrided substrate with a compound layer (treatment A, Table 1), the coefficient of friction showed a very constant value throughout the entire test with the lowest value (~0.1) among the substrate pre-treatments used. These low values of the coefficient of friction can be related to the formation of a carbon transfer film ([20] to [23]), observed on the counter surface of the disc (Fig. 7).

Deposition of the wear protective coating (TiN, TiAlN or DLC) decreased the pin wear rate significantly, as shown in Fig. 8. As in the case of the scratch test, wear of the duplex-treated pins was strongly influenced by the substrate pre-treatment (Fig. 8). The wear of the investigated coatings decreased with increased substrate hardness and a change of the substrate pre-treatment from hardening to plasma nitriding. Compared to the coated-and-hardened substrates, the nitrided-and-coated specimens showed improved sliding-wear resistance, which can be mainly attributed to higher substrate hardness and improved coating-to-substrate adhesion. Some earlier investigations have shown that the

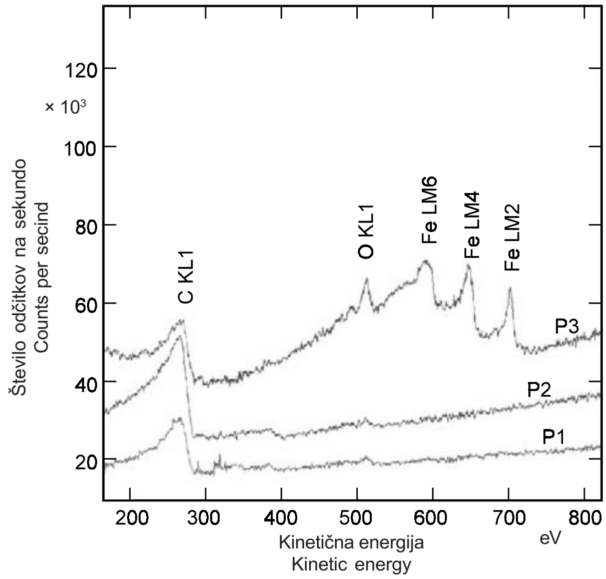


Sl. 6. *Povprečne vrednosti koeficienta trenja v odvisnosti od kemotermičnega izboljšanja podlage*
Fig. 6. *Average coefficient of friction of duplex-treated steel as a function of substrate treatment*

(a)



(b)



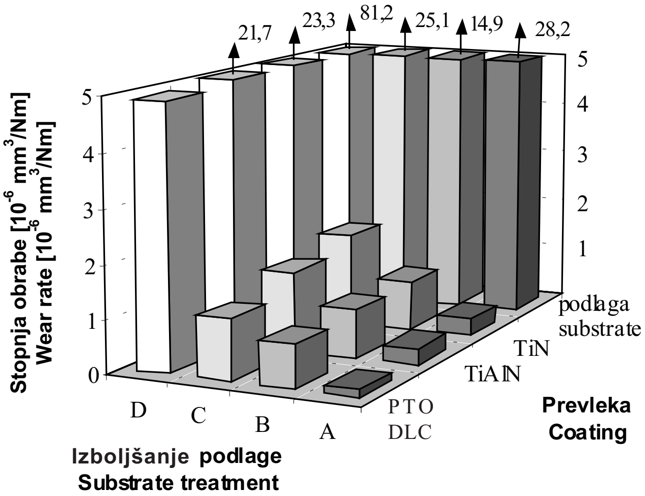
Sl. 7. *(a) Obrabljena površina plošče po drsenju s kemotermično izboljšanim valjčkom, prekritim s PTO in (b) analiza površine XPS – točki P1 in P2 znotraj obrabne sledi in P3 na neobrabljeni površini*
Fig. 7. *(a) Wear track on the steel disc running against a DLC coated pin and (b) XPS analysis of the surface (P1 and P2 inside wear track and P3 unworn surface)*

nitrirane podlage, je moč pripisati večji trdoti podlage ter boljši oprijemljivosti prevleke na podlago. Kljub temu, da so prejšnje raziskave pokazale negativen vpliv spojinske plasti na obrabno odpornost trdih prevlek ([8], [10], [11], [18] in [19]), pa rezultati te raziskave kažejo, da strnjena spojinska plast lahko deluje kot vmesna trda plast, ki močno izboljša drsno obrabno odpornost trdih zaščitnih prevlek (sl. 8).

Pozitiven vpliv nitriranja na drsno obrabno odpornost trdih zaščitnih prevlek je moč razložiti na naslednjih dveh primerjavah. Prvič, spojinska plast na nitrirani površini močno izboljša obrabno odpornost raziskovanih trdih prevlek. Razlika v obrabni odpornosti je v skladu z razliko v trdoti podlage ter dejstvom, da trša podlaga zagotavlja boljšo podporo trdi prevleki. Pri prenosu obremenitve večja trdota podlage

compound layer formed during nitriding has a negative effect on the coating-to-substrate adhesion and therefore reduces the durability of the wear protective coating ([8], [10], [11], [18] and [19]). However, results of this investigation have shown that when the compound layer has a relatively dense and uniform structure it can actually act as an intermediate hard layer, leading to a superior sliding-wear resistance of the hard coatings (Fig. 8).

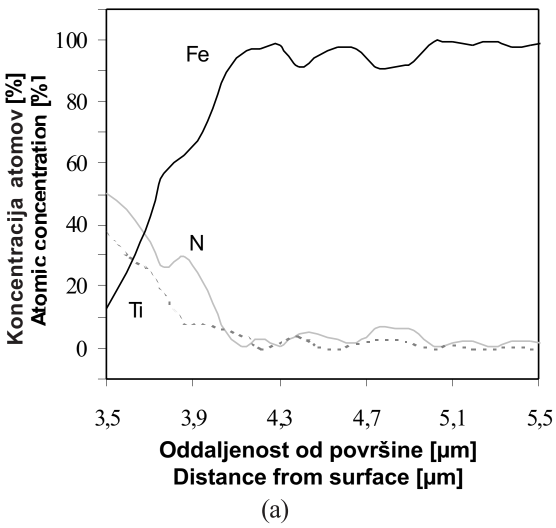
The positive effect of nitriding on the sliding-wear properties of hard coatings can be explained by considering the following two comparisons. First, a change of the nitriding parameters to produce a compound layer significantly improved the coating wear resistance. This difference in tribological behaviour is consistent with the difference in the hardness of the two substrates and the premise that a harder substrate is able to provide a more effective

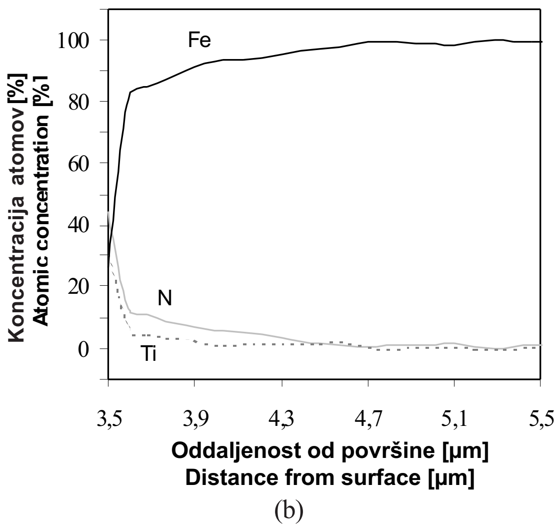


Sl. 8. *Stopnja obrabe raziskovanih prevlek v odvisnosti od kemotermičnega izboljšanja podlage (preglednica 1)*
Fig. 8. *Wear rate of the investigated coatings as a function of substrate treatment (Table 1)*

zniža verjetnost njene deformacije, zniža pa tudi gradient trdote in napetosti na meji med prevleko in podlago, kar poveča obstojnost prevleke. Drugič, kljub temu, da je bila s kaljenjem (postopek C, preglednica 1) in nitriranjem v plazmi z majhnim deležem dušika (postopek B, preglednica 1) dosežena približno enaka trdota podlage, pa nitriranje v plazmi zagotavlja boljšo obrabno odpornost trdih prevlek. Ta ugotovitev je v skladu z dejstvom, da poleg trdote podlage oprijemljivost prevleke igra zelo pomembno vlogo. V primeru nitriranega jekla je analiza AES (sl. 9) pokazala postopno zmanjšanje koncentracije N in Ti na meji med prevleko in podlago, medtem ko je pri kaljeni podlagi opažen takojšen padec koncentracije Ti (prevleka DLC) oz. N in Ti (prevleki TiN in TiAlN).

coating support. Substrate hardening reduces subsurface plastic deformation and a hardness gradient at the coating/substrate interface, thus improving the coating's durability. Second, although hardening (treatment C, Table 1) and plasma nitriding in a nitrogen-poor atmosphere (treatment B, Table 1) give almost the same surface hardness, plasma nitriding of the substrate led to an improved sliding-wear resistance for the investigated coatings. This observation is consistent with the finding that in addition to the hardness of the substrate, the adhesion also plays a very important role. AES analysis (Fig. 9) of hard-coated plasma-nitrided steel showed a gradual decrease in the Ti and N concentrations from the interface into the substrate, while a rapid drop in Ti (DLC) or Ti and N (TiN and TiAlN) at the coating/substrate





Sl. 9. *Porazdelitev koncentracije Fe, Ti in N v (a) kaljenem in (b) nitriranem jeklu 42CrMo4 (postopek B), prekritim s trdo zaščitno prevleko TiN*
Fig. 9. *AES concentration depth profiles of Fe, Ti and N measured from the interface for TiN coated (a) hardened and (b) plasma nitrided substrate (treatment B)*

Na podlagi rezultatov analize AES je moč sklepati, da vmesna plast Ti reagira z dušikom v nitrirani podlagi ter na ta način izboljša adhezijo med prevleko in podlago [11].

interface was observed in the case of the hardened substrate. Results of AES analysis suggest that the Ti interlayer reacts with the nitrogen in the nitrided substrate and therefore improves the adhesion between the coating and the substrate [11].

3 SKLEP

Uspešna uporaba trdih zaščitnih prevlek v mehanskih sistemih je odvisna od oprijemljivosti prevleke na podlago ter zmožnosti podlage, da daje prevleki zadostno podporo. Nitriranje v plazmi poveča trdoto podlage, s čimer se zmanjša deformacija podlage pri obremenitvi in izboljša nosilna zmožnost sestave. Prek interdifuzije dušika v nitrirani podlagi in Ti v vmesni plasti pa se izboljša tudi oprijemljivost prevleke na podlago.

Z natančno vodenim procesom nitriranja v plazmi je moč doseči nastanek strnjene spojinske plasti. V primeru površin, prekritih s trdo zaščitno prevleko, lahko strnjena spojinska plast deluje kot vmesna trda prevleka. Ta zniža gradient trdote in napetosti na meji med prevleko in podlago ter s tem privede do bolj ugodnega napetostno-deformacijskega polja. Tako se poleg izboljšane obrabne odpornosti površine izognemo potrebi po dodatni mehanski obdelavi nitrirane površine pred nanosom trde zaščitne prevleke.

4 CONCLUSION

Satisfactory performance of a coated component depends on the coating-to-substrate adhesion and the ability of the substrate to support the coating. By increasing the hardness of the steel substrate, plasma nitriding improves the load-carrying capacity of the substrate. In addition, plasma nitriding also improves coating-to-substrate adhesion, most probably through the inter-diffusion between the iron nitride at the nitrided surface and the thin Ti interlayer.

A uniform, dense and well-adhered compound layer can be obtained by precise control of the nitriding process. In the case of hard-coated components such a compound layer can act as an intermediate hard layer, which reduces large hardness differences between the coating and the substrate and consequently leads to a favourable strain-stress field and improved sliding-wear properties of the composite. Therefore, with a precisely controlled plasma-nitriding process the need for additional polishing or grinding of the nitrided surface prior to coating deposition can be eliminated.

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Prejeto:
Received: 28.11.2000

Sprejeto:
Accepted: 27.6.2001