CODEN STJSAO ISSN 0562-1887
ZX470/1495 UDK 669.14.018.252:620.178.1:621.893

PACVD Duplex Coating for Hot Forging of High Strength Steels for Automotive Applications

Vojteh LESKOVŠEK[1]***, Monika JENKO***[1]***,***
Bojan PODGORNIK[2]

1) Institute of Metals and Technology, Lepi pot 11, 1000 Ljubljana,
Republic of Slovenia

2) University of Ljubljana, Center for Tribology and Technical Diagnostics, Bogišičeva 8, 1000 Ljubljana,
Republic of Slovenia

vojteh.leskovsek@imt.si

Original scienfic paper

The tribological load is the load on forging tools resulting from the relative motions between the plastically deformed workpiece and the die. In comparison to many other forming processes hot die forging has an especially disadvantageous tribological system. The advantages of the application of hard coatings, which are well known for cutting tools, are to a much lesser extent explored for casting, extrusion, moulding and forging tools. Increasing the lifetime of these tools is an important task in surface engineering because of the complex loading conditions and often complicated tool geometry. The plasma-assisted chemical-vapour-deposition (PACVD) technique is well suited to depositing hard coatings onto large dies and moulds. The aim of this study is to present and discuss results obtained on a duplex PACVD hard coating in an industrial application such as hot forging of automotive parts. The results presented here are from a preliminary investigation. The experimental results indicate that introduction of a proper duplex PACVD hard coating will lead to improved wear resistance and longer lifetime for hot-forging dies. Furthermore, by using a hard, lowfriction coating excellent anti-sticking property can be obtained. At this stage of the investigation it is also necessary to carry out pilot trials to determine the wear resistance and tool life in practice.

PACVD duplex prevlake za toplo kovanje visoko čvrstih čelika za automobilsku primjenu

Izvornoznanstveni članak

Tribološko opterećenje je opterećenje na alatu za kovanje koje je rezultat kretanja između plastično deformiranog dijela i alata. U usporedbi s mnogim drugim postupcima oblikovanja, kod kovanja u toplom postoji nepovoljan specifičan tribološki sistem. Prednosti primjene tvrdih prevlaka, koje su poznate za alate za rezanje, manje su izražene za ljevačke, ekstruzijske i kovačke alate. Povećanje radnog vijeka tih alata je važan zadatak u inženjerstvu površina zbog složenih uvjeta opterećenja i često komplicirane geometrije alata. Plazmom potpomognuto taloženje u parnoj fazi kemijskim postupkom (PACVD) je vrlo prikladno za taloženje tvrdih prevlaka na velike kalupe. Cilj ovog istraživanja je predstaviti i prodiskutirati dobivene rezultate na duplex PACVD tvrdim prevlakama u industrijskoj primjeni kao što je kovanje automobilskih dijelova. Predstavljeni rezultati dobiveni su uvodnim istraživanjima. Eksperimentalni rezultati navode da će uvođenje prikladne duplex PACVD tvrde prevlake dovesti do poboljšanja otpornosti na trošenje i na produljenje životnog vijeka alata za kovanje. Nadalje, korištenjem tvrde prevlake s malim trenjem mogu se postići izvrsna svojstva ne-lijepljenja. U ovoj fazi istraživanja također je nužno iznijeti početne pokušaje (pilote) za određivanje otpornosti na trošenje i određivanje životnog vijeka alata u praksi.

Keywords
Adhesion,
Drop-forging die
Hardness
Microstructure
PACVD coating
Hot-work tool steel

Ključne riječi
Adhezija
Alati za rad u toplom
Mikrostruktura
PACVD prevlake
Prokaljivost
Tvrdoća

Received (primljeno): 2009-09-10
Accepted (prihvaćeno): 2010-12-30

1. Introduction

The advantages of hard coatings, which are well known for cutting tools, are to a much smaller extent explored for forging dies. Wear of the die not only results in a short lifetime, but also in unacceptable surface conditions of the product. In recent years, heavy demands for lower costs, increasing productivity and higher product quality have stimulated the development of hard coatings for these applications. During the forging process dies are heavily loaded due to a complex load combination that consists of four fundamental loads, i.e., thermal, mechanical, tribological and chemical [1,2]. However, although it is an important task in surface engineering, increasing the lifetime of forming tools is often difficult due to the complex loading conditions and often complicated tool geometry. These peculiarities impose very specific requirements on the deposition process itself. In this

Symbols/Oznake

PACVD	- plasma-assisted chemical-vapour deposition - plazmom potpomognuto taloženje u ranoj fazi	HRC	- Rockwell C hardness - tvrdoća po Rockwellu C
		R_a	- Average roughness - Prosječna hrapavost
ESR	- electro slag remelted - električno pretaljena šljaka	HV	- Vickers hardness - tvrdoća po Vickersu

work, the technique of plasma-assisted chemical vapour deposition (PACVD), suitable for hard-coating deposition on forging dies, and its specific limitations, are described.

2. Experimental materials and methods

2.1. Material and vacuum heat treatment

The commercial electro slag remelted (ESR) hot-work tool steel grade AISI H11 was delivered as a 25-mm plate, which was cut from a forged-and-soft-annealed master block, with the following chemical composition (mass content in %): 0.39 % C; 1.07 % Si; 0.34 % Mn; 0.013 % P; 0.0004 % S; 4.93 % Cr; 1.26 % Mo; 0.35 % V. The specimen, in the form of bar φ 25 x 75 mm, cut from the plate in the short transverse direction was used, Figure. 1.

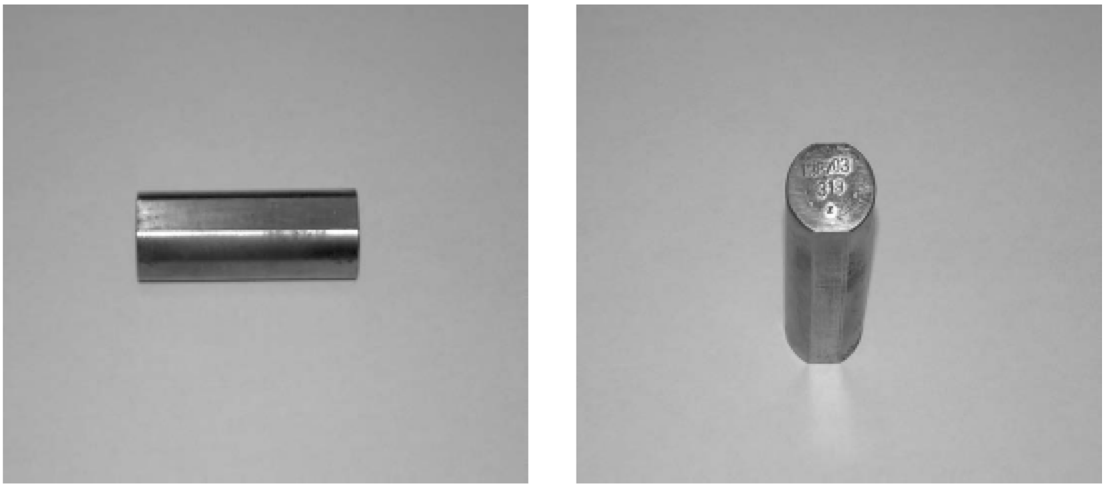


Figure 1. Specimen of ESR AISI H11 hot-work tool steel in the form of bars Ø 25 x 75 mm

Slika 1. Uzorak čelika ESR AISI H11 za rad u toplom u formi šipke Ø 25 x 75 mm

The test specimens were heat treated as inserts together with the forging dies made from the same tool steel. A uniform high-pressure gas-quenching heat treatment was performed in a horizontal vacuum furnace using nitrogen (N2) at a pressure of 5 bar and a cooling parameter $\lambda_{800-500}$ = 8.11. After the last pre-heating (850 °C) the specimens were heated to the austenitizing temperature of 1000 °C (10°C/min), soaked for 30 minutes, gas quenched to a temperature of 100 °C, and then double tempered, first at 540 °C, and then at 585 °C, each time for 4 hours. The vacuum heat-treated specimen was further machined into Ø 25 x 15 mm discs.

2.2. Pulse plasma nitriding and PACVD treatment

The convection and plasma heating to the process temperature of 540 °C took approximately 3.5 h, with the time of nitriding being 24 hours. Before introducing the process gas, the chamber was evacuated to a pressure of 2 Pa. The 4kHz pulse plasma was produced in a total pressure of 3.3 hPa. For nitriding, a negative bias potential of 530 V was applied to the specimens, which act as the cathode.

The specimens were nitrided in 95 vol-% H2: 5 vol-% N2 gas mixture at a nitriding temperature of 540 °C, which resulted in a nitrided case depth of about 0.25 mm without the formation of a compound layer. Stylus profilometry was used to measure the actual change in the surface roughness caused by the pulse-plasma nitriding as well as by the coating deposition. After nitriding the average roughness value, Ra, of the original ground surface increased from 0.01 μm to approximately 0.116 μm.

The Rockwell-C hardness (HRC) was measured before and after nitriding, and before and after the PACVD treatment, using a Wilson 4JR hardness machine. The microhardness depth profiling and the surface hardness measurements were performed using a Fischerscope H100C apparatus at a load of 10N (1000 g), while the near-surface hardness was measured on cross-sections at a load of 1N (~100 g).

Prior to the PACVD coating deposition the surfaces of the test specimens were prepared as shown in Table 1 and sputter-cleaned. PACVD deposition was applied to samples denoted B-D, while sample A was used as a reference. The PACVD-deposited coating was a standard TiN/Ti-B-N duplex coating developed by Rübig and deposited by a bipolar-pulsed glow discharge at the processing temperature of 530°C and pressure of 200 Pa. The multilayer TiN/Ti-B-N coating was composed of a base TiN monolayer, a multilayer TiN/Ti-B-N zone with Ti-B-N layers containing 23 at.% B and a top TiB_2 layer. Thus, the boron content was varied between single-phase TiN and single-phase TiB_2.

The overall coating thickness was 1.8 µm. The 23 at.% boron content was selected because of the small grain size, typically 5-7 nm, resulting in increased resistance to plastic deformation and abrasion when compared to TiN [3]. The characterization of the coating morphology was performed using optical and scanning electron microscopy.

3. Results and discussion

The compound-layer thickness and the nitriding depth were determined on substrate A, which was prepared according to conventional metallographic techniques and etched with 3 % nital. Diffusion layers of different thickness were formed with no compound layer visible on the surfaces. The metallographically determined case depth of the diffusion zone was 208.3 µm. Figure 2 shows hardness profile of the diffusion layer for specimen A, nitrided at 540 °C for 24 hours. A surface hardness of the nitrided steel specimen is ≈ 1350 HV 0.1, with the nitriding depth (Nht), determined according to the DIN 50 190-79 standard, being about 220 µm. It should be noted that due to the size of the indentation and interference effects from the free surface, hardness measurements with a load of 1N (~100 g) are only possible within ~20 µm of the surface.

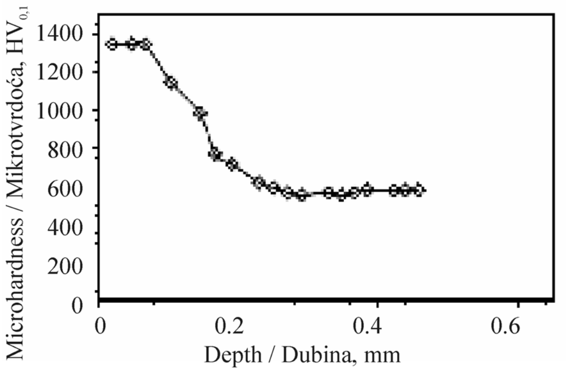


Figure 2. Micrograph showing microhardness depth profile of nitrided AISI H11 substrateat a load of 1N

Slika 2. Graf pokazuje mikrotvrdoću profila nitrirane AISI H11 podloge pri opterećenju od 1N

Figure 3, shows optical microphotographs of the duplex coating consisting of a plasma-nitrided substrate and a PACVD-deposited TiN/Ti-B-N hard coating (substrates B, C and D) at 500 x magnification. As can be seen from Figure 3a, the PACVD coating deposited on the as-nitrided surface (substrate B) shows a fine and dense structure, which is periodically interrupted with bulges.

After the deposition, the average roughness value, Ra, of the original ground surface increased from 0.116 m to approximately 0.437 m. In the case of sample C, Figure 3b, with the nitrided and polished substrate coating showed a fine and dense structure without bulges, and a uniform thickness of ~1.8µm. The average roughness value, *R*a, of the originally ground surface of sample C increased from 0.01µm to approximately 0.132 µm. The coating on sample D, with a nitrided and sand-blasted substrate, used the same process as for forging dies before

Table 1. Condition of the surface, the average roughness, the Rockwell-C hardness and the surface hardness of the substrates after nitriding

Tablica 1. Stanje površine, prosječna hrapavost, tvrdoća u Rockwell-C i površinska tvrdoća podloge nakon nitriranja

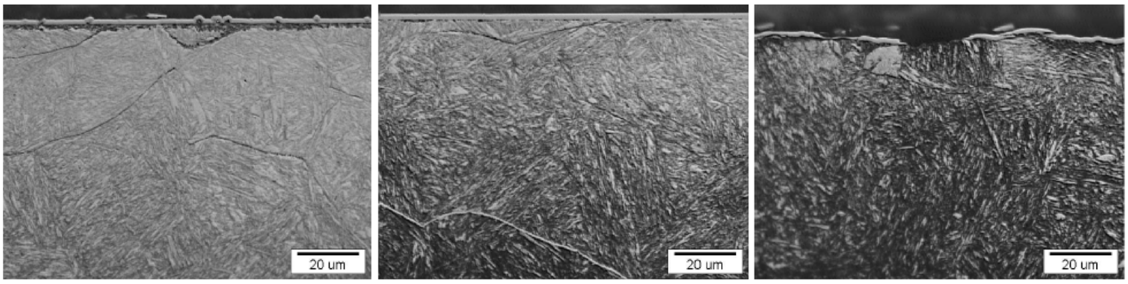
Sample symbol / Simbol uzorka	Condition of substrate surface / Stanje površine supstrata	Average roughness / Prosječna hrapavost R_a, µm	Rockwell-C hardness after nitriding / Tvrdoća po Rockwellu-C psolije nitriranja, HRC	Surface hardness after nitriding / Površinska tvrdoća poslije nitriranja, $HV_{0.1}$
A	as nitrided	0.116	47,5	1350
B	as nitrided	0.116	47,5	1350
C	nitrided + polished	≤ 0.01	47,5	1350
D	nitrided + sand blasted	> 0.116	47,5	1350

coating, Figure 3d, showed a non-uniform thickness and discontinuities. The average roughness value, Ra, of the coated sample D's surface is 0.52 μm. For all the samples (B-D) a thin layer (1.7 μm) with some closed porosity that etches more intensively can be observed below the hard coating and into the diffusion zone. Depending on the deposition time, a layer thickness of ~1.8 μm was observed on all the substrates. An individual layer thickness of approximately 85 μm was calculated for the 21-layer coating, which is in good agreement with the coating's total thickness measurement as well as being confirmed by the SEM cross-section analysis, shown in Figure 4.

a much smoother surface and a more distinct and flat interface compared to sample B.

A high-magnification bulge-morphology examination (Figure 4a) shows some microcracks originating in a bulge foot under an angle of 45 °, which go over the entire thickness of the coating. Inside the bulges a structure in the shape of a comb was found at the substrate surface. There is also an irregular etching effect immediately beneath the coating, which is much more pronounced than for substrate C.

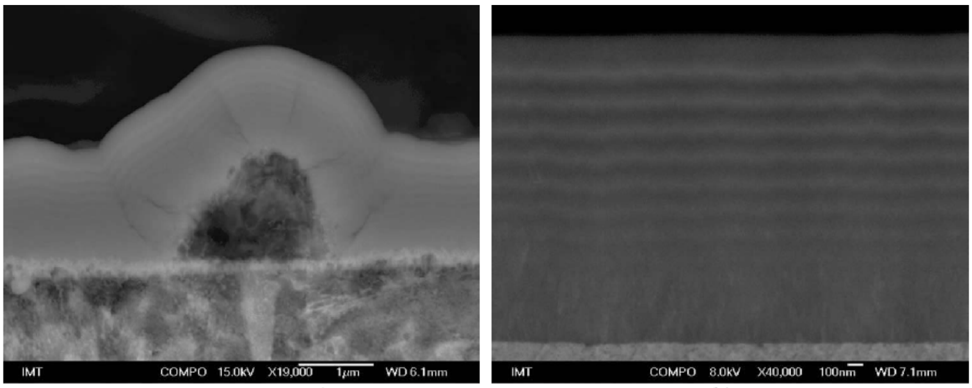
The coating-surface microhardness was measured on all the samples with a Fischerscope H100 C and was 50 GPa, corresponding to ~5000 HV0.003, with the elastic-modulus of the coating being in the range 380–430 GPa. The adhesion of the coating deposited on the substrates B-D was evaluated with a Revetest equipped with a 200-μm-radius Rockwell-C diamond stylus. In the case of the asnitrided substrate (sample B), the coating flaked instantaneously at loads below 6.7 N, which indicates poor adhesion of the coating containing bulges. In the case of the nitrided and polished substrate (sample C), the first failure of the coating in the form of cracking and spallation on the scratch channel rim was detected in the load range 12–16 N. For the nitrided and sand-blasted substrate (sample D) the scratch-test results are not relevant due to the non-uniform and discontinuous coating.



a) b) c)

Figure 3. Optical cross-section of duplex-treated AISI H11 substrates B-D

Slika 3. Optički poprečni presjek duplex obrađene AISI H11 podloge B-D



a) b)

Figure 4. SEM cross-sections of AISI H11 substrate and PACVD TiN/Ti-B-N coatings B and C after plasma pre-treatment

Slika 4. SEM poprečni presjek AISI H11 podloge i PACVD TiN/Ti-B-N prevlake B I C nakon predobrade plazmom

As shown in Figure 4b the base and top mono layers can be distinguished by the phase contrast between the TiN/Ti-B-N and TiB_2 layers. TiN, which is the base layer at the interface, shows a more fibrous structure and can be distinguished from the smooth surface of the Ti-B-N phase as well as from the top TiB2 phase. Moreover, a sharp interface between the 21 individual layers was formed.

The SEM cross-section micrograph of sample B shows a periodic coating interruption with bulges, which confirms the observations with the optical microscope. On the other hand, the cross-section of sample C reveals a very uniform coating thickness of 1.8 μm as well as

Friction coefficients of 0.7–1.0 are reported for sputtered Ti-B-N and TiB2 coatings [4,5]. PACVD

Ti-B-N coatings show a similar friction coefficient at low humidity, while at high humidity the friction coefficient drops to 0.3–0.4 [4]. TiB_2 is also soluble in iron at elevated temperatures, which favours chemical wear [5,6]. Therefore, although not attractive for the machining of steel, TiB-based coatings could be used for drop-hammer forging dies. Processes that take place during forging in the tool are substantially influenced by the intermediate zone, which mainly consists of hard scale parts, lubricant and residues of lubricants and abrasion particles of the tool and workpiece [1]. Therefore, the investigated Ti-B-N coating with a high hardness of ~50 GPa, a relatively low friction coefficient at high humidity and relatively good adherence, shows good potential for reducing the predominantly abrasive wear of forging dies when deposited on a polished nitrided surface [7].

For an industrial test, PACVD-coated forging-die inserts, made from ESR AISI H11 hot-work tool steel, were used (Figure 5a). Four coating variants (TiCN-duplex, TiN/Ti-B-N-duplex, TiN/Ti-B-N-duplex-low B, TiCN) were tested and evaluated under industrial conditions. Fig. 5b, shows the surface topography of an as-coated TiCN duplex-coated insert (TiCN-duplex).

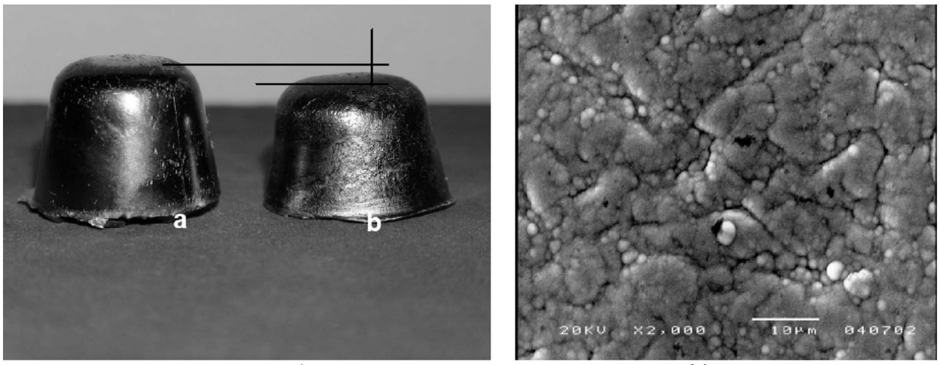
As can be seen from Figure. 5b, after the nitriding and deposition of the TiCN duplex coating the surface of the insert is relatively rough, with the surface roughness being increased 3–4 times, i.e., from 0.105 to 0.314 μm. After the industrial test the TiCN duplex-coated insert was examined and compared with a standard nitrided forging-die insert, operated under the same production conditions.

The wear criterion for the insert change was determined by the height of the insert, which was checked with a reference gauge. As can be seen from Fig. 5a, the height of the coated insert after 13500 forged pieces is equal to the initial height, while the heights of the nitrided forging-die inserts decrease by 1/5th of the initial height. Due to the very high thermal loading of the insert the initial substrate hardness of the coated insert decreased during the production run from 42 HRC to 38.1 HRC, and for the standard nitrided one, from 47 to 42 HRCc.

A visual and SEM inspection of the coating (Figure 6) showed that although it was locally damaged, it covers most of the loaded part of the insert (a), while the coating which was not exposed to the highest loads looks more or less intact (b). Delamination or flaking of the coating, which was observed at a critical part of the insert (Fig. 6b), could be a result of the rough surface, which together with high compressive residual stresses led to initiation of the cracks, located at the stress risers. The presented results are preliminary, and a detailed characterization of the above coatings is the subject of [8].

4. Conclusions

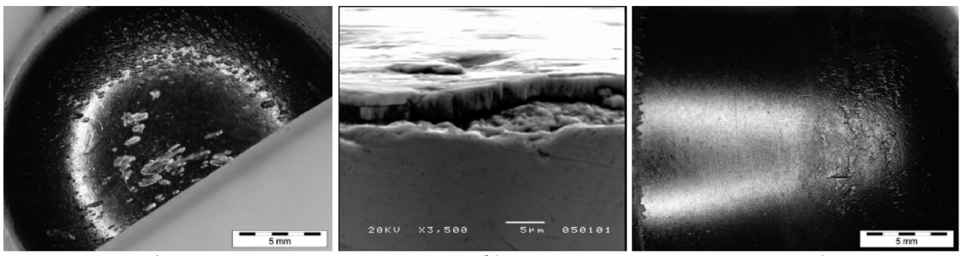
A PACVD TiN/Ti-B-N duplex coating was deposited on vacuum-heat-treated and plasma-nitrided substrates from ESR AISI H11 hot-work tool steel. The surfaces of the substrates C and D were mechanically treated before coating, while the surface of the substrate B was coated



a) b)

Figure 5. Forging die inserts: (a) coated by the TiCN duplex process (aa) and as nitride (bb), (b) SEM images of TiCN duplex coating showing the surface topography

Slika 5. Dodaci kalupu za kovanje: (a) prevučen TiCN duplex postupkom (aa) i kao nitrid (bb), (b) SEM slika TiCN duplex prevlaka pokazuje topografiju površine



a) b) c)

Figure 6. Coating on the loaded part of the insert (a, b) and at the foot of the insert (c)

Slika 6. Prevlaka opterećenog dijela dodatka (a,b) i otisak dodatka (c) as nitrided. From this investigation it can be concluded that the substrate pre-treatment of the surface has a crucial influence on surface topography and the adhesion properties of the coating. In the case of the investigated PACVD TiN/Ti-B-N duplex coating the lowest surface roughness and good adhesion properties of the coating were obtained in sample C. In this case the nitrided surface of the substrate was polished before deposition of the coating.

For the industrial test a range of four PACVD coatings were available at this time. Our first results

(the testing of the remaining three inserts is still in progress) show that the deposition of a composite layer of the nitrided H11 steel and the TiCN coating prepared by PACVD are promising techniques for the surface modification of forging-die inserts made of H11 steel. This plasmaduplex treatment processing for drop-forging die inserts made of the investigated steel used in automotive forgings production was successful at improving the lifetime when compared to the conventional technique.

The results presented above are from preliminary investigations. The aim of future developments in the field of PACVD coatings will be to up-scale the process to even bigger drop-forging dies, to expand the spectrum of coatings available and to explore new applications of VMR hot-work tool steels for hot-forging processes.

REFERENCES

[1] SEIDEL, R.; LUIG H.: *Friction and wear processes in hot die forging*, Proceedings of theInternational European Conference on Tooling Materials, September 7-9, 1992 Bochum,pp.467-480.

[2] KLIMEK K.S.; GEBAUER-TEICHMANN, A.; KAESTNER, P.; RIE K.-T.: *Duplex-PACVD coating of surfaces for die casting tools*, Surface and Coatings Technology, In Press, Corrected Proof, Available online 22 August 2006.

[3] STOIBER. M.; BADISCH, E.; LUGMAIR, C.; MITTERER, C.: *Low-friction TiN coatings deposited byPACVD*, Surface&Coatings Techhnology, 163-164 (2003),pp.451-456.

[4] STOIBER, M.; PERLOT, S.; MITTERER, C.; BESCHLIESSER, M.; LUGMAIR, C.; KULLMER, R.: *PACVD TiN/Ti-B-Nmultilayers:from micro-to nano-scale*, Surface&Coatings Techhnology, 177-178 (2004), pp.348-354.

[5] KULLMER, R.; LUGMAIR, C.; FIGUERAS, A.; BASSAS, J.; STOIBER, M.; MITTERER, C.: *Microstructure, mechanical and tribological properties of PACVD Ti(B,N) and TiB2 coatings*, Surface&Coatings Techhnology, 174-175 (2003), pp.1229-1233.

[6] MITTERER C.; HOLLER, F.; REITBERGER, D.; BADISCH, E.; STOIBER, M.; LUGMAIR, C.; NÖBAUER, R.; MÜLLER, Th.; KULLMER, R.: *Industrial applications of PACVD hard coatings*, Surface&Coatings Techhnology,163-164 (2003), pp.716-722.

[7] HE Y.; APACHITEI, I.; ZHOU, J.; DUSZCZYK, J.: *Effect of prior plasma nitriding applied to a hotwork tool steel on the scratch-resistant properties of PACVD TiBN and TiCN coatings*, Surface and Coatings Technology, In Press, Corrected Proof, Available online 9 June 2006.

[8] JENKO, M.; LESKOVSEK, V.; MANDRINO, D.; GODEC, M.: *Characterizatin of PACVD Duplex TiN/TiB_2 Coatings on Hot Work Tool Steels by Integrated Electron Spectroscopy Techniques*, International Conference on Metalurgical Coatings and Thin films, San Diego 1-5 May 2006.