ORIGINALNI NAUČNI ČLANCI
ORIGINAL SCIENTIFIC PAPERS

PROPERTIES AND STRUCTURE OF TUNGSTENCARBIDE – COBALT COATINGS DEPOSITED BY THE APS – PLASMA SPRAY PROCESS

Mihailo R. Mrdak, Research and Development Center IMTEL Communications a. d., Belgrade

DOI: 10.5937/vojtehg61-2215

FIELD: Chemical Technology
ARTICLE TYPE: Original Scientific Paper

Summary:

The aim of this study was to optimize the parameters of the plasma spray and to deposit WC17Co layers with optimal structural - mechanical characteristics. The powder was deposited by the plasma spraying process at the atmospheric pressure (APS). When choosing the parameters, the flow of the He plasma gas was taken as the basic parameter. In relation to other gases, helium does not react with the powder,it produces a denser plasma with a lower heat content and it incorporates less ambient air into the plasma jet which reduces decarburization of the powder. The study shows three groups of samples obtained with three plasma gas flows of 12, 22 and 32 l/min He. The coating with the best properties was deposited on the shaft sleeve of the main rotor of the Gazelle H42 helicopter, in order to reduce the influence of vibrations and bearings on sleeve wear up to 500°C. The estimates of the WC17Co layers of the coating were made on the basis of their structural - mechanical properties. The surface morphology of the WC17Co powder particles was examined on the SEM. The mechanical properties of the deposited coatings were tested in accordance with the 'TURBOMECA'standard. The estimate of the mechanical properties of layers was done by examining microhardness with the method $HV_{0.3}$ *and bond strength with tensile testing. Metallographic assessment of the pore proportion in the layers of the WC17Co coating (image analysis) was performed with the technique of light microscopy in accordance with the 'Pratt & Whitney' standard. Studies have shown that the rate of the plasma gas flow significantly affects the mechanical properties and the structure of coatings.*

Key words: *properties, powder, plasma, flow tate, cobalt, coating.*

ACKNOWLEDGEMENT: The author is thankful for the financial support from the Ministry of Education and Science of the Republic of Serbia (national projects OI 174004, TR 34016).

miki@insimtel.com

Introduction

The plasma spray process is applied in many industries with the aim to improve the characteristics of components or to extend their working life. This process has found a wide application in the aviation industry and it is one of the most applicable processes for thermal coatings. The process configuration can vary so that particle jets can have a wide range of temperature and velocity values. The resource of coatings can be influenced by the characteristics of the deposited material, its phase composition, proportion of pores and unmelted particles as well as by cohesion and adhesion strength. These properties are closely related to the conditions of the deposition and a significant improvement can be expected when optimizing the deposition parameters (Vencl, et al., 2006, pp.151-157), (Dorfman, 2002, pp.47-50), (Brossard, et al., 2010, pp.1599-1607). WC17Co coatings deposited with thermal spray processes always show change in the phase composition in relation to the composition of the starting powder. This is something that cannot be avoided. Changes of the phase composition in deposited carbide coatings were described by Saha, G.C., et al. (Saha, et al., 2010, pp. 592-595) who explained the cause of phase changes. The examinations of the phase of the nanocrystalline WC17Co coatings deposited by the HVOF thermal process and polycrystalline coatings deposited by the process of the plasma spraying have shown that, in both coatings, the starting phase WC decomposes, more or less, into the phase of W_2C and W. This decomposition is more pronounced in plasma due to higher temperatures. In the structure of the polycrystalline coating deposited by plasma, besides the phase WC, the phases W_2C, W, W_3C and mixed carbide ή - Co_3W_3C (Saha, et al., 2010, pp.592-595) are always present. The type of the applied plasma gas, flow and the power of supply are important parameters affecting the quality of the deposited carbide coatings. In the case of the application of H_2 as a plasma gas, the WC-Co powder is more exposed to decomposition because of the combination of decarburization, oxidation and the reactions between WC and cobalt, which leads to the formation of hard and brittle phases such as W_2C, CoxWyCz, and even WO_3 and W (de Villiers Lovelock, 1998, pp.357-373). The decarburization of one powder particle takes place as follows. As temperature increases, cobalt melts and WC also passes to the liquid state. Carbon is removed from the melted particle by some reaction with oxygen on the liquid/gas boundary or through the diffusion of oxygen into the melted particle, which leads to the formation of carbon monoxide (Stewart, et al., 2000, pp.1593-1604). Due to the incorporation of air into the plasma jet and this influence on the carbon reduction from carbide, the powder deposition is done with an average power of supply of the plasma gun and with Helium as the plasma gas. Helium, as a noble gas, has great advantages as a plasma gas in comparison to other gases. Experimental investigations of plasma jet characteristics have shown that the isotherms near the anode exit have a smaller diameter for the Ar-He plasma than for the Ar-H_2. The isotherms are also shorter in the Ar-He plasma because of less specific enthalpy and because of higher viscosity when compared to Ar-H_2. Denser plasma, such as Ar-He, can substantially reduce the incorporation of ambient air into the plasma jet. Mixing of the plasma jet with the ambient air increases with the increasing intensity of the electric arc and with the plasma gas flow (Roumilhac, et al., 1988, pp.105-119), (Roumilhac, Fauchais, 1988, pp.121-126). Helium, because of these characteristics, allows deposition of carbide coatings with a reduced process of decarburization and with a lower content of pores. The properties of coatings are directly related to the deposition parameters. The WC17Co powder was developed for the aviation industry. The WC17Co-based coatings are resistant to: wear, abrasion, erosion, corrosion and cavitation up to 500˚C (Material Product Data Sheet, 2011). Cubic monokarbid WC contains 6.13%C and has the microhardness of about 2700$HV_{0.3}$, while W_2C contains 3.16%C and has the microhardness of about 3000$HV_{0.3}$ while being more brittle than WC. The W_2C brittle phase degrades the coating and reduces the coating resistance to abrasive wear (Valiev, Nature, 2002, pp.887-889). The highest wear resistance of carbide phases can be retained in coatings through the optimization of the spray process parameters (Li, et al., 1996, pp.785-794). Despite the fact that W_2C is metastable below 1250°C, it is often present in WC-Co, even after slow cooling. The metastable WC_{1-x} phase, however, can be found only at room temperature when the material is quickly quenched (de Villiers Lovelock, 1998, pp.357-373), (Mrdak, et al., 2004, pp.407-421). The decomposition of WC carbide is incomplete because of a short retention of powder particles in the plasma jet. Depending on the heat quantity exchanged between the plasma jet and the powder, decomposition of carbides occurs to a higher or lower extent. Therefore, the coating has the microhardness in a range of 800-1300 $HV_{0.3}$, since the composition of the coating deviates from the initial composition of the powder In the coating, besides carbide WC, W_2C, there is a complex carbide W_3Co_3C whose microhardness is in a range of 600-1300 $HV_{0.3}$. Depending on the deposition parameters, W with the microhardness of about 400 $HV_{0.3}$ and Co with the hardness below 200 $HV_{0.3}$ may appear in the coating. In order to understand the metallurgical processes that take place when carbide WC is deposited, it is important to know the W-C binary system, shown in Fig. 1 (ASM Handbook, 1992). Tungsten and carbon build W_2S and WC hexagonal carbides as well as β-WC cubic carbide. Tungsten carbide, with the WC hexagonal structure, has the melting temperature of 2785 ± 10°C and, in the cooling process, builds eutectic with tungsten at 2715 ± 5°C. Stability of the carbide decreases with the decreasing temperature and at 1250°C it decomposes to pure tungsten and WC hexagonal monocarbide. This carbide has a very narrow area of stability and is not used for deposition.Consequently, WC cubic monocarbide which has a wide area of stability (Mrdak, et al., 2004, pp.407-421), (Mrdak, 2010, pp.43-52), (Mrdak, et al., 2003, pp.125-128). is used for deposition.

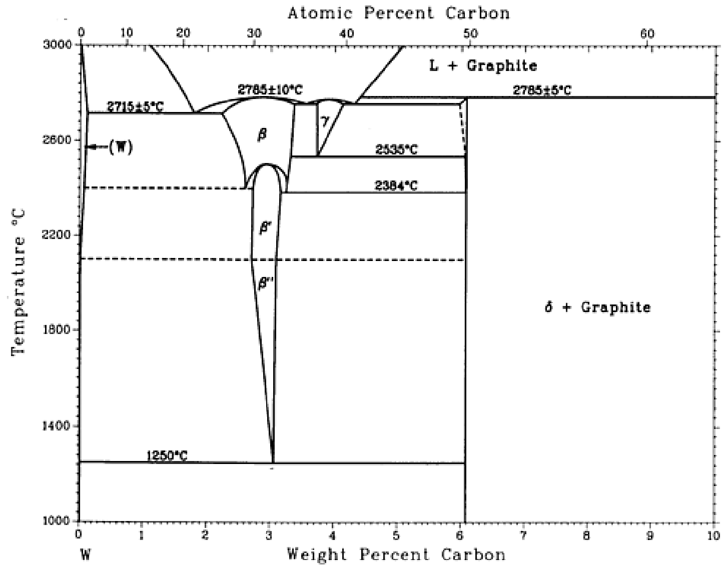

Figure 1 – W – C binary sistem
Slika 1 – Binarni sistem W – C

This paper shows the results of experimental research on the influence of the plasma gas flow (He) on the mechanical properties and on the microstructure of WC17Co layers. The main aim was to replace the old technology of shaft sleeve cementation with depositing WC17Co coatings using the plasma spray technology. The mechanical properties and the microstructure of coatings were analyzed in order to select a coating with the best properties. Coatings with the best mechanical and structural characteristics were tested and homologated on the shaft sleeve of the main rotor of the Gazelle H42 helicopter, in flight tests in the period of 50 hours in the VZ „Moma Stanojlović“ – Batajnica.

Testing Materials and Samples

The Metco 73F-NS-1 powder by the ’Sulzer Metco’company was used for the production of coatings. For powder, the technique of dry dispersion/sintering with a content of 83 wt.% WC and 17 wt.% Co was used. The powder used in the experiment had a granulation span from 11 μm to 53 μm (Material Product Data Sheet, 2011). Fig. 2 shows the scanning electron micrography (SEM) of the morphology of WC17Co powder particles. The powder particles are porous and spherical.

The bases on which coatings for assessing the structure and for testing microhardness were deposited were made of Č.4171 (X15Cr13 EN10027) steel in the thermally unprocessed state with the dimensions 70x20x1.5mm (Turbojet engine-standard practices manual TURBOMECA). Also, the bases for testing bond strength were made of Č.4171 (X15Cr13EN10027) steel in the thermally unprocessed state with the dimensions Ø25x50mm (Turbojet engine-standard practices manual TURBOMECA).

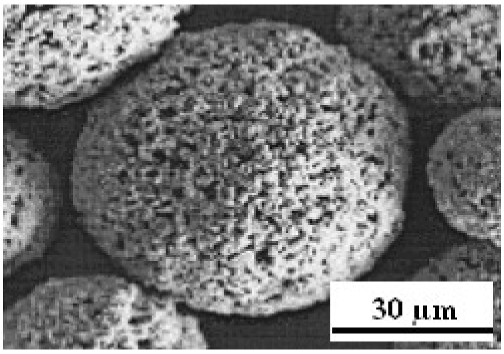

Figure 2 – (SEM) Scanning electron micrograph of WC17Co powder particles
Slika 2 – (SEM) Skeningelektronska mikrografija čestica praha WC17Co

Examination of microhardness, bond strength and microstructure

Mechanical characterizations of WC17Co coatings were made in accordance with the TURBOMECA standard (Turbojet engine-standard practices manual TURBOMECA). For microhardness testing and metallographic tests, 70 × 20 × 1.5mm samples were used, while for tensile strength tests, Ø25 x 50mm samples were used.

The measurements of microhardness were done by using the Vickers diamond pyramid indenter and 300 grams of load ($HV_{0.3}$). The measurements waere done along the lamellar structure, in the middle and at the ends of the samples. Five readings were performed at three places and the results were averaged.

The tensile strength tests were done at room temperature on the hydraulic equipment with a speed of 10 mm/min. Two samples were used in pairs, but the coating was deposited only on one of them. For each group of WC17Co coatings five samples were tested, and the results were averaged.

The metallographic evaluation of the pore proportion (image analysis) in the WC17Co coating layers was made using the light microscopy technique in accordance with the 'Pratt & Whitney' standard (Turbojet Engine, 2002). The morphology of the powder particles was done under the SEM (scanning electron microscope).

Powder deposition

The process of depositing layers on the metal matrix was done by the plasma spraying process at atmospheric pressure (APS). The coatings were deposited on the steel bases roughened with white corundum Al_2O_3 with the granulation sizes of 0.7-1.5 mm. The 'Plasmadyne' atmospheric plasma spray (APS) system was used for making coatings.The powder was deposited with the plasma gun 'SG -100' which consisted of a cathode type K 1083A-129, an anode type A 2083-175 and a gas injector type GI 1083-113. Argon was used as a gas in combination with Helium and the power of supply was 40 KW. The plasma gas flow of Helium was the main parameter for the powder deposition. Three different flows of Helium were used (12 l/min, 22 l/min and 32 l/min). The optimum flow of Helium enables minimum decarburazition, oxidation and a reaction between WC and cobalt, which leads to the formation of hard and brittle phases. The detailed values of plasma spray parameters are shown in Table 1. The layers are deposited on the substrates of total thickness of 0.020 to 0.025mm, with the plasma gun speed of 500 mm/s. The WC17Co coating with the best structural and mechanical properties was deposited on the shaft sleeve of the main rotor of the Gazelle H42 Helicopter.

Table 1 – Plasma spray parameters
Tabela 1 – Plazma-sprej parametri

Deposition parameters	Values
Plasma current, I (A)	700
Plasma Voltage, V (V)	35
Primary plasma gas flow rate Ar (l/min)	47
Secondary plasma gas flow rate He (l/min)	12 / 22 / 32
Carrier gas flow rate (l/min)	8
Powder feed rate (g/min)	40
Stand-off distance (mm)	90

Results and discussion

The results of testing the microhardness and bond strength of WC17Co coating layers, depending of the plasma Helium gas flow, are shown in Figs. 3 and 4.

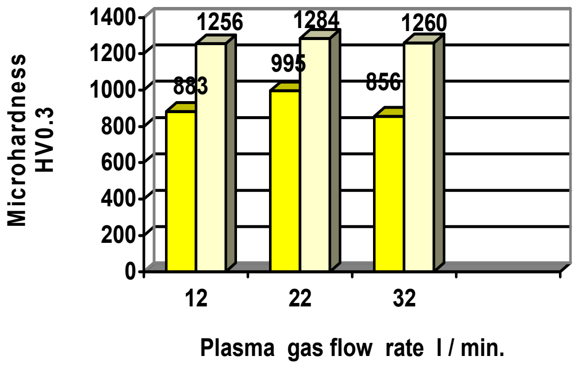

Figure 3 – Microhardness of WC17Co layers
Slika 3 – Mikrotvrdoća WC17Co slojeva

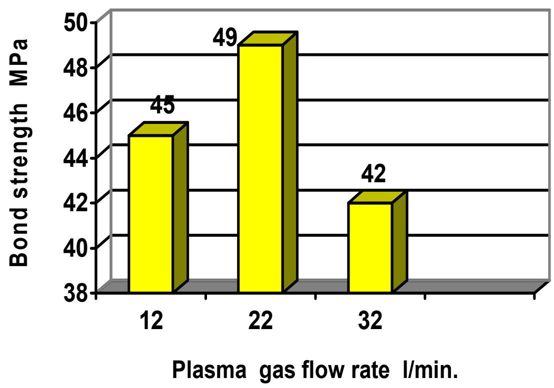

Figure 4 – Bond strength of WC17Co layers
Slika 4 – Čvrstoća spoja WC17Co slojeva

The values of the microhardness and bond strength of the deposited layers are directly related to the flow of Helium as a plasma gas. All deposited layers have the values of microhardness within the prescribed limits of 850 - 1300 $HV_{0.3}$ (Material Product Data Sheet, 2011), (Turbojet engine-standard practices manual TURBOMECA).

The most uniformly distributed microhardness was found in the layers deposited with the plasma He gas flow of 22 l/min. These layers had the smallest difference between the maximum and the minimum of the microhardness values (289$HV_{0.3}$). The biggest distribution of microhardness was found in the layers deposited with the highest plasma He gas flow of 32 l/min. These layers had the biggest difference between the maximum and the minimum of the microhardness values (404 $HV_{0.3}$). A higher distribution of microhardness was also found in the layers with the smallest plasma He gas flow of 12 l/min. The difference between the maximum and the minimum of the microhardness values for these layers was (373 $HV_{0.3}$). The layers of WC17Co coating deposited with the optimal plasma He gas flow of 22 l/min showed the best microstructure with the smallest proportion of lamellar pores. This was confirmed by the image analysis of the microstructure of coating layers under the light microscope.

The tensile strength of bonding is directly related to the plasma He gas flows. The values of bond tensile strength in the deposited layers, with the plasma He gas flows of 12 l/min and 22 l/min, are in the prescribed limits per standard (min. 45 MPa) (Turbojet engine-standard practices manual TURBOMECA).

These layers had good adhesion strength with the substrate and good lamellar cohesive strength. The highest value of bond strength of 49MPa was found in the layers deposited with the plasma He gas flow of 22 l/min, which had the lowest distribution of microhardness. These layers had the smallest proportion of pores. The lowest value of bond tensile strength of 42 MPa was found in the layers deposited with the highest plasma gas flow of He. These layers had the greatest distribution of microhardness and the greatest propotion of micro pores. The testing of bond tensile strength showed that, for all deposited coatings, the failure mechanism was on the interface between the substrate and the coating. This indicates good melting of powder particles and their bonding to the substrate for all three plasma gas flow of He. These values were confirmed by the image analysis of the coating microstructure under the light microscope.

Figs. 5, 6 and 7 show the microstructure of WC17Co coating layers deposited with the plasma gas flows He of 12 l/min, 22 l/min and 32 l/min. The figures under (a) show the substrate/ coating interface and (b) shows the middle of the layer. The quantitative analysis of the total content of pores in WC17Co layers shows that the measured values are directly related to the plasma gas flows of He. In the shown micrographs, different proportions of pores in the deposited layers are clearly recognizable. The smallest proportion of pores was in the layers of WC17Co coating deposited with the plasma gas flow He of 22 l/min. In these layers, the total proportion of pores was 1%. In the layers deposited with the plasma gas flow He of 12 l/min, the total proportion of pores was 1.3%. The greatest proportion of pores was in the layers deposited with the plasma gas flow He of 32 l/min. In these layers, the pores are more prominent and coarser with the proportion of pores of 2.5%. In all layers, the total proportion of pores was within the prescribed limits of 0.5 - 3% (Material Product Data Sheet, 2011), (Turbojet engine-standard practices manual TURBOMECA).

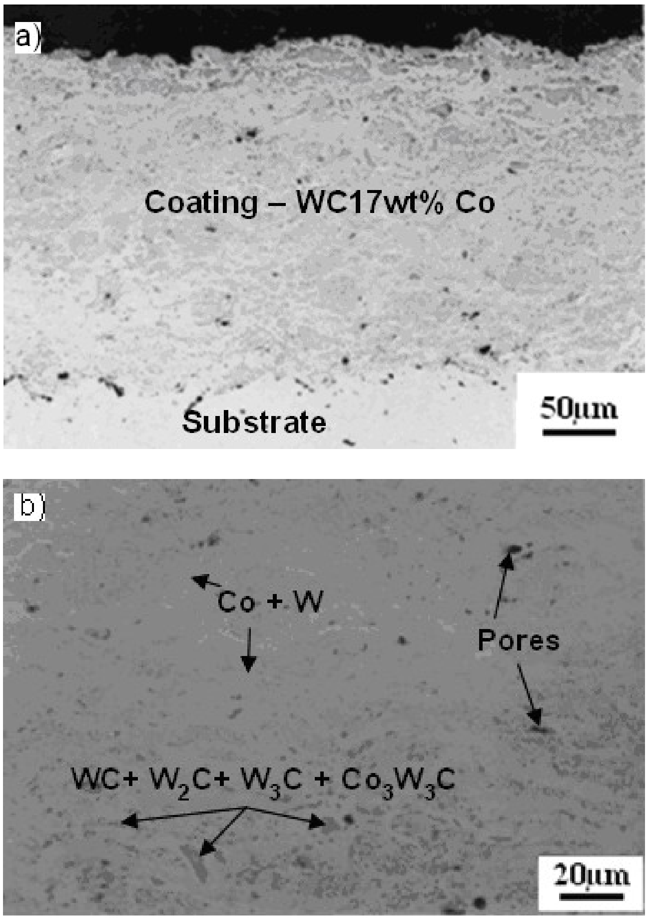

Figure 5 – Microstructure of the WC17Co coating with a plasma gas flow of 12 l / min. He (a) substrate / coating interface and (b) middle layer

Slika 5 – Mikrostruktura prevlake WC17Co sa protokom plazma gasa 12 l/minHe, (a) interfejs substrat/prevlaka i (b)sredina prevlake

The measured values of the total content of pores in the WC17Co layers are consistent with the measured microhardness values.

The qualitative analysis of all deposited WC17Co layers showed that there is a negligible proportion of residual particles of corundum Al_2O_3 left from roughening at the substrate/coating interface. The bond of coatings with the substrates is niform without the separation of coating layers from the substrate. Along the interface, between the substrate and the coating, there are no micro cracks and macro cracks. Fig. 6 shows the micrographs of WC17Co coatings whose layers had the best mechanical properties and the smallest proportion of micro pores.

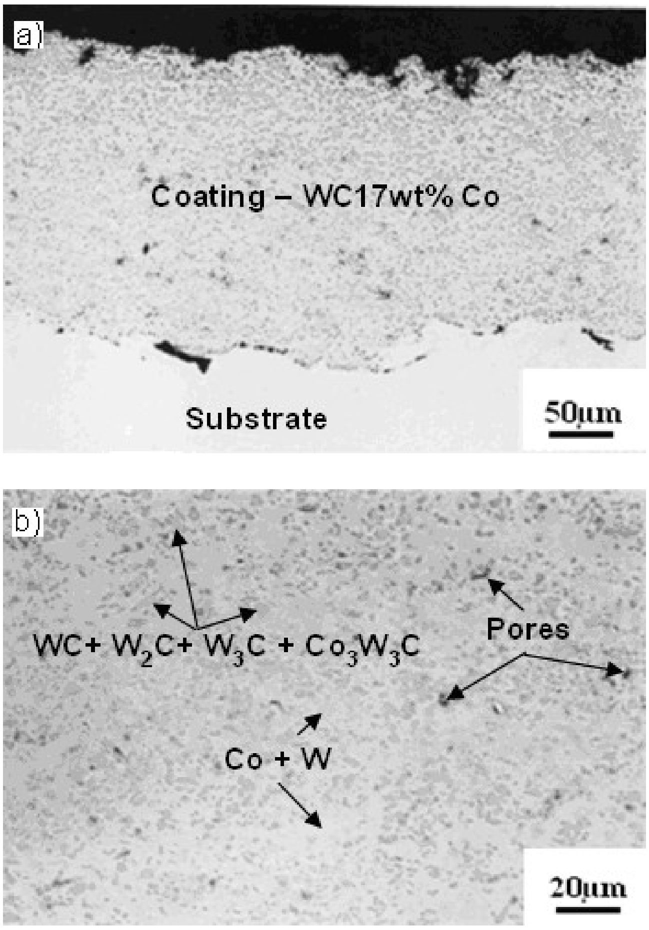

Figure 6 – Microstructure of the WC17Co coating with a plasma gas flow of 22 l / min.He (a) substrate / coating interface and (b) middle layer
Slika 6 – Mikrostruktura prevlake WC17Co sa protokom plazma gasa 22 l/min He, (a) interfejs substrat/prevlaka i (b) sredina prevlake

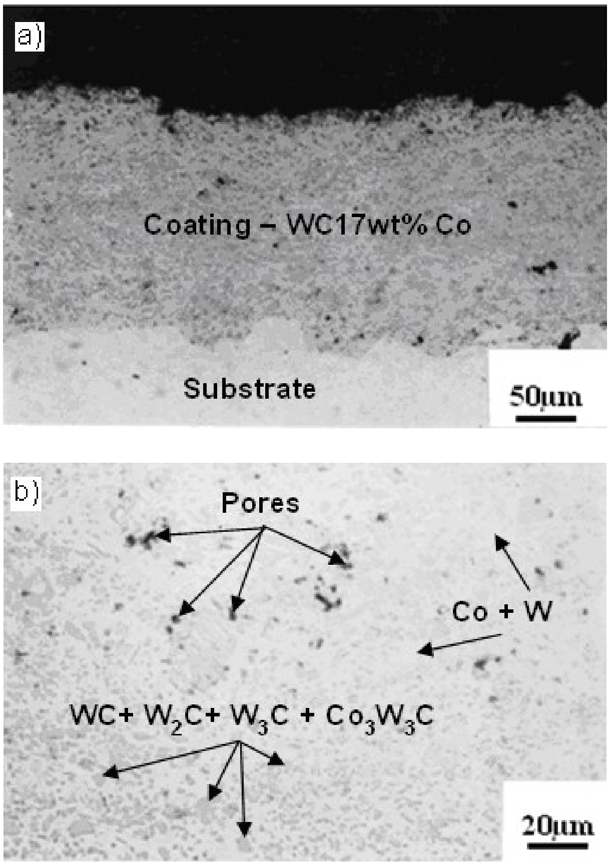

Figure 7 – Microstructure of the WC17Co coating with a plasma gas flow of 32 l / min.He (a) substrate / coating interface and (b) middle layer
Slika 7 – Mikrostruktura prevlake WC17Co sa protokom plazma gasa 32 l/minHe, (a) interfejs substrat/prevlaka i (b) sredina prevlake

These layers are deposited with the plasma gas flow He of 22 l/min. The microstructure of all coatings is lamellar. The micrographs show that the melted powder particles are uniformly distributed. The coating layers were deposited continuously without present micro cracks and macro cracks through the layers. In the layers, there are no unmelted powder particles, precipitates, and inter-lamellar pores. In the deposited layers of all coatings,uniformly distributed carbide phases are clearly seen in the tough cobalt base. The light fields of metal phases and the dark-gray fields of carbide phases are clearly seen in all micrographs. Light fields contain the initial metallic phase Co and the metallic phase W which is derived from a partial degradation of the initial cubic monocarbide WC

(Saha, et al., 2010, pp.592-595), (de Villiers Lovelock, 1998, pp.357-373). In gray fields, there is the initial phase of cubic monocarbide WC and the carbide phases resulting from the decomposition of cubic carbide WC into the carbides of type W_2C and W_3C, and the mixed carbide ή - Co_3W_3C (Saha, et al., 2010, pp.592-595), (de Villiers Lovelock, 1998, pp.357-373).

Conclusion

The atmospheric plasma spray (APS) process was used for depositing WC17Co coatings with the plasma gas flow He of 12 l/min, 22 l/min and 32 l/min. The mechanical properties of deposited layers were analysed as well as the microstructure under the light microscope. The morphology of powder particles was investigated under the scanning electron microscope (SEM). The performed analyses led to the following conclusions.

The morphology of WC17Co powder particles is of a spherical shape and is typical for powder particles produced with the technique of dry dispersion/sintering. The values of microhardness and bond strength of the deposited layers were directly related to the flows of Helium as a plasma gas. All deposited layers had the values of microhardness in the prescribed limits of 850 - 1300 $HV_{0.3}$. The most uniform distribution of microhardness was found in the layers deposited with the plasma gas flow He of 22 l/min. These layers had the smallest difference between the maximum and the minimum microhardness values (289 $HV_{0.3}$).

The values of bond tensile strength of the layers deposited with the plasma gas flows He of 12 l/min and 22 l/min were within the prescribed limits in accordance with the standard (min.45 MPa). These layers had good adhesion of strength with the substrate and good lamellar cohesive strength. The highest value of the bond strength of 49MPa was found in the layers deposited with the plasma gas flow of He, which had the lowest distribution of microhardness. These layers had the smallest proportion of pores. The lowest value of bond tensile strength of 42 MPa was seen in the layers deposited with highest plasma gas flow of He. These layers had the greatest distribution of microhardness and the greatest proportion of micro pores.

The quantitative analysis of the total content of pores in the WC17Co layers showed that the measured values are directly related to the plasma gas flows of He. The smallest proportion of pores was seen in the WC17Co coating layers deposited with the plasma gas flow He of 22 l/min. In these layers, the total proportion of pores was 1%. In the layers deposited with the plasma gas flow He of 12 l/min, the total proportion of pores was 1.3%. The greatest proportion of pores was in the layers deposited with the plasma gas flow He of 32 l/min. In these layers, the pores are more prominent and coarser with the proportion of pores of 2.5%. In all layers, the total proportion of pores was within the prescribed limits of 0.5 - 3% .

The microstructure of all coatings is lamellar.The coating layers were deposited continuously without present micro cracks and macro cracks through the layers. In the layers there are no unmelted powder particles, precipitates, and inter-lamellar pores. In all deposited coating layers, the uniformly distributed carbide phases are clearly seen in the tough cobalt.base The light fields contain the initial metallic phase Co and the metallic phase W which is derived from a partial degradation of the initial cubic monocarbide WC. In gray fields, there is the initial phase of cubic monocarbide WC and the carbide phases resulting from the decomposition of cubic carbide WC into the carbides of type W_2C and W_3C, and the mixed carbide ή - Co_3W_3C .

The obtained results showed that the plasma gas flow of He significantly affects the mechanical properties of layers and the proportion of pores in the coatings. The tests confirmed that the best layers are those deposited with the plasma gas flow He of 22 l/min. The oating with the best mechanical and structural characteristics was tested and homologated on the shaft sleeve of the main rotor of the Gazelle H42 helicopters during flight tests in the period of 50 hours in the VZ "Moma Stanojlović" - Batajnica.

Literature

ASM Handbook, 1992, Volume 3, Alloy Phase Diagrams, ASM International, Printed in the United States of America, Metals Park.

Brossard, S., Munroe, P.R., Tran, A.T.T., Hyland, M.M., 2010, Study of the effects of surface chemistry on splat formation for plasma sprayed NiCr onto stainless steel substrates, Surface and Coatings Technology, 204, 9-10, pp.1599-1607.

de Villiers Lovelock, H.L., 1998, Powder/processing/structure relationships in WC-Co thermal spray coatings: a review of the published literature, *Journal of Thermal Spray Technology*, 7(3), pp.357–373.

Dorfman, M.R., 2002, Thermal spray basics, Advanced Materials & Processes, 160(7), pp.47-50.

Li, C.-J., Ohmori, A. and Harada, Y., 1996, Effect of WC-Co powder structure on the structure of thermally sprayed WC-Co coatings, J. Mater. Sci., 31, pp.785-794.

Material Product Data Sheet, 2011, Metco 73F-NS-1 Tungsten Carbide-17% Cobalt Powder, DSMTS – 0030.2, Sulzer Metco.

Mrdak, M., Kakaš, D., Pović, Đ., 2004, Karakterizacija plazma-sprej prevlaka $Cr_3C_2$25NiCr i WC17Co otpornih na habanje i koroziju, Istraživanje i Razvoj Mašinskih Elemenata i Sistema, IRMES '04, Kragujevac, pp.407-421.

Mrdak, M., 2010, Karakterizacija WCCo/NiCrBSi prevlake otporne na habanje, Vojnotehnički glasnik 2/10, pp.43 - 52.

Mrdak, M., Kakaš, D., Pović, Đ., 2003, Ispitivanje strukturnih i mehaničkih osobina prevlaka sa povišenom otpornošću na habanje, 8th Internacionalna konferencija o tribologiji, Beograd, Srbija, pp.125 -128.

Roumilhac, Ph., Coudert, J.F., Leger, J.M., Fauchais, P., 1988, Characterization of a D.C.Spraying Plasma Torch using Optical and Thermal Diagnostics, Proceedings of the 1st Plasma-Technik Symposium, Switzerland, Lucerne, 1, pp.105-119.

Roumilhac, Ph., Fauchais, P., 1988, Optical and Thermal Diagnostics Regarding the Working Conditions of a Plasma Spray Mini Torch, Proceedings of the 1st Plasma-Technik Symposium, Switzerland, Lucerne, 1, pp.121-126.

Saha, G.C., Mateen, A., Khan, T.I., 2010, Tribological Studies of Conventional Microcrystalline and Engineered Near-Nanocrystalline WC-17Co HVOF Coatings, NSTI – Nanotech 2010, ISBN 978-1-4398-3401-5 (1), pp.592-595.

Stewart, D.A., Shipway, P.H., and McCartney, D.G., 2000, Microstructural evolution in thermally sprayed WC-Co coatings: comparison between nanocomposite and conventional starting powders, *Acta Materialia*, 48(7), pp. 1593– 1604.

Turbojet engine-standard practices manual TURBOMECA.

Turbojet Engine – Standard Practices Manual (PN 582005), 2002, Pratt & Whitney, East Hartford, USA.

Valiev, R., 2002, Nature, 419, pp.887-889.

Vencl, A., Mrdak, M., Cvijović, I., 2006, Microstructures and tribological properties of ferrous coatings deposited by APS (Atmospheric Plasma Spraying) on Al-alloy substrate, FME Transactions, 34(3), pp.151-157.

SVOJSTVA I STRUKTURA WOLFRAMKARBID-KOBALT PREVLAKE DEPONOVANE PLAZMA-SPREJ POSTUPKOM

OBLAST: hemijske tehnologije
VRSTA ČLANKA: originalni naučni članak

Sažetak:

Cilj istraživanja bio je da se optimizacijom plazma-sprej parametara deponuju WC17Co slojevi optimalnih strukturno-mehaničkih karakteristika. Prah je deponovan plazma-sprej postupkom na atmosferskom pritisku (APS).Pri izboru parametara kao osnovni parametar uzet je protok plazma gasa He. Helijum u odnosu na druge gasove ne reaguje sa prahom, proizvodi gušću plazmu sa manjim toplotnim sadržajem i manje inkorporira okolni vazduh u mlaz plazme, što smanjuje dekarburizaciju praha. U istraživanju su korišćene tri grupe uzoraka dobijene sa tri protoka plazma gasa od 12, 22 i 32 l/min He. Prevlaka sa najboljim karakteristikama deponovana je na rukavcu vratila glavnog rotora helikoptera "gazela H42" da bi se

smanjio uticaj ležaja i vibracija na habanje rukavca do 500°C. Procene WC17Co slojeva prevlake urađene su na osnovu njihovih strukturno-mehaničkih karakteristika. Morfologija površine čestica praha WC17Co ispitana je na SEM-u. Mehaničke karakteristike deponovanih prevlaka ispitane su u skladu sa standardom TURBOMECA. Procena mehaničkih karakteristika slojeva urađena je ispitivanjem mikrotvrdoće metodom $HV_{0.3}$ i čvrstoće spoja ispitivanjem na zatezanje. Metalografska procena udela pora u slojevima WC17Co prevlaka (image analiza) urađena je tehnikom svetlosne mikroskopije u skladu sa standardom Pratt & Whitney. Istraživanja su pokazala da protok plazma gasa bitno utiče na mehaničke osobine i strukture prevlaka.

Uvod

Plazma-sprej postupak je zastupljen u mnogim industrijama radi poboljšanja karakteristika komponenti ili produženja njihovog radnog veka. Ovaj postupak našao je široku primenu u vazduhoplovnoj industriji i jedan je od najprimenjenijih postupaka za termičko nanošenje prevlaka. Na resurs prevlaka utiču karakteristike deponovanog materijala, njegov faznih sastav, udeo pora i nestopljenih čestica, koheziona i adheziona čvrstoća. Deponovane prevlake WC17Co termosprej postupcima uvek pokazuju promenu u faznom sastavu u odnosu na sastav polaznog praha, što se ne može izbeći. Promenu faznog sastava u deponovanim karbidnim prevlakama opisao je Saha,G.C. i dr. autori (Saha, et al., 2010, pp. 592-595) koji su objasnili uzrok faznih promena. Ispitivanjem faza nanokristalne prevlake WC17Co deponovane termičkim postupkom HVOF i polikristalne prevlake deponovane plazma-sprej postupkom, ustanovljeno je da u obe prevlake dolazi do, manje ili više, razgradnje polazne faze WC u fazu W_2C i W. Ovo razlaganje je izraženije kod plazme zbog viših temperatura. U strukturi polikristalne prevlake deponovane plazmom, pored faze WC uvek su prisutne i faze W_2C,W,W_3C i mešoviti karbid ή – Co_3W_3C (Saha, et al., 2010, pp. 592-595). Zbog inkorporiranja vazduha u mlaz plazme i njegovog uticaja na redukciju ugljenika iz karbida, depozicija praha radi se srednjom snagom napajanja plazma-pištolja i sa helijumom kao plazma gasom. Helijum kao plemeniti gas ima velike prednosti kao plazma gas u odnosu na druge gasove. Eksperimentalna istraživanja karakteristika mlazeva plazmi pokazala su da izoterme blizu izlaza iz anode imaju manji prečnik za plazmu Ar-He u odnosu na Ar-H_2. Dužina izotermi je takođe manja kod plazme Ar-He zbog manje specifične entalpije i većeg viskoziteta u odnosu na Ar-H_2. Gušća plazma, kao što je Ar-He, može znatno da smanji inkorporiranje okolnog vazduha u mlaz plazme. Mešanje mlaza plazme sa okolnim vazduhom povećava se sa povećanjem jačine strujnog luka i protokom plazma gasa (Roumilhac, et al., 1988, pp. 105-119), (Roumilhac, Fauchais, 1988, pp. 121-126). Helijum zbog ovih osobina omogućuje deponovanje karbidnih prevlaka sa umanjenim procesom dekarburizacije i sa manjim sadržajem pora. Osobine prevlaka su u direktnoj vezi sa parametrima depozicije.

Prah WC17Co razvijen je za potrebe vazduhoplovne industrije. Prevlake na bazi WC17Co su otporne na habanje, abraziju, eroziju, koroziju i kavitaciju do 500°C (Material Product Data Sheet, 2011). U ovom radu predstavljeni su rezultati eksperimentalnih istraživanja uticaja protoka plazma gasa (He) na mehanička svojstva i mikrostrukturu slojeva WC17Co. Glavni cilj je bio da se stara tehnologija cementacije rukavaca vratila zameni deponovanjem WC17Co prevlake plazma-sprej tehnologijom. Analizirane su mehaničke karakteristike i mikrostrukture prevlaka da bi se odabrala prevlaka najboljih karakteristika. Prevlaka sa najboljim mehaničkim i strukturnim karakteristikama je testirana i homologovana na rukavcu vratila glavnog rotora helikoptera "gazela H42" letnim ispitivanjem u trajanju od 50 časova u VZ „Moma Stanojlović" u Batajnici.

Materijali za ispitivanje i uzorci

Za izradu prevlaka upotrebljen je prah firme „Sulzer Metco" sa oznakom Metco 73F-NS-1. Za izradu praha korišćena je tehnika suvo raspršavanje/sinterovanje sa sadržajem od 83 tež.%WC i 17 tež.%Co. Prah koji se koristio u eksperimentu imao je raspon granulacije od 11 μm do 53 μm (Material Product Data Sheet, 2011). Čestice praha su porozne i sfernog oblika.

Osnove na koje su deponovane prevlake za procenu strukture i za ispitivanje mikrotvrdoće izrađene su od čelika Č.4171 (X15Cr13 EN10027) u termički neobrađenom stanju dimenzija 70x20x1,5mm. Osnove za ispitivanje čvrstoće spoja takođe su izrađene od čelika Č.4171(X15Cr13EN10027) u termički neobrađenom stanju dimenzija Ø25x50 mm (Turbojet engine-standard practices manuel TURBOMECA).

Mehaničke karakterizacije prevlaka WC17Co urađene su prema standardu TURBOMECA (Turbojet engine-standard practices manuel TURBOMECA). Za merenje mikrotvrdoće i metalografska ispitivanja korišćeni su uzorci dimanzija 70 × 20 x 1,5 mm, dok su za ispitivanje zatezne čvrstoće korišćeni uzorci Ø25 x 50 mm.

Merenja mikrotvrdoća izvršena su korišćenjem Vikers dijamant piramide indenter i 300 grama opterećenje (HV0.3). Merenje je urađeno u pravcu duž lamela, u sredini i na krajevima uzorka. Na tri mesta sprovedeno je pet očitavanja, a rezultati su bili usrednjeni.

Testovi zatezne čvrstoće rađeni su na sobnoj temperaturi na hidrauličnoj opremi sa brzinom od 10 mm/min. Korišćena su dva uzoraka u paru, od kojih je prevlaka deponovana samo na jednom od njih. Za svaku grupu WC17Co prevlaka ispitano je po pet uzoraka, a dobijeni rezultati su usrednjeni.

Metalografska procena udela pora (image analiza) u slojevima WC17Co prevlaka urađena je tehnikom svetlosne mikroskopije u skladu sa standardom Pratt & Whitney (Turbojet Engine – Standard Practices Manual (PN 582005), 2002, Pratt & Whitney, East Hartford, USA). Morfologija čestica praha urađena je na SEM-u (skening elektronskom mikroskopu).

Depozicija praha

Proces deponovanja slojeva na metalne osnove urađen je plazma-sprej postupkom na atmosferskom pritisku (APS) firme Plasmadyne. Depozicija praha urađena je plazma pištoljem SG-100 koji se sastojao od katode tipa K 1083A -129 , anode tipa A 2083-175 i gas-injektora tipa GI 1083-113. Kao gas korišćen je argon u kombinaciji sa helijumom i snaga napajanja od 40 KW. Protok plazma gasa helijuma bio je osnovni parameter za deponovanje praha. U eksperimentu su korišćena tri različita protoka helijuma od 12 l/min, 22 l/min i 32 l/min. Slojevi su deponovani na supstratima ukupne debljine od 0,020 do 0,025 mm sa plazma pištoljem brzine 500 mm/s.

Rezultati i diskusija

Vrednosti mikrotvrdoće i čvrstoće spoja deponovanih slojeva su u direktnoj vezi sa protocima helijuma kao plazma gasa. Svi deponovani slojevi imaju vrednosti mikrotvrdoće u propisanim granicama od 850 do 1300 $HV_{0.3}$ (Material Product Data Sheet, 2011), (Turbojet engine-standard practices manuel TURBOMECA). Najravnomerniju raspodelu mikrotvrdoće imali su slojevi deponovani sa protokom plazma gasa od 22 l/min.He. Ti slojevi su imali najmanju razliku mikrotvrdoće između maksimalnih i minimalnih vrednosti (289 $HV_{0.3}$). Najveću raspodelu mikrotvrdoće pokazali su slojevi deponovani sa najvećim protokom plazma gasa od 32 l/min He. Ti slojevi imali su najveću razliku mikrotvrdoće između maksimalnih i minimalnih vrednosti (404 $HV_{0.3}$). Višu raspodelu mikrotvrdoće imali su i slojevi sa najmanjim protokom plazma gasa od 12 l/min He. Razlika između maksimalnih i minimalnih vrednosti mikrotvrdoće za te slojeve bila je 373 $HV_{0.3}$.

Zatezna čvrstoća spoja je u direktnoj vezi sa protocima plazma gasa He. Vrednosti zatezne čvrstoće spoja deponovanih slojeva sa protocima plazma gasa od 12 l/min i 22 l/ min He, u propisanim su granicama po standardu (min. 45 MPa) (Turbojet engine-standard practices manuel TURBOMECA). Ti slojevi imali su dobru adhezionu čvrstoću sa supstratom i dobru međulamelarnu kohezionu čvrstoću. Najveću vrednost čvrstoće spoja od 49MPa pokazali su slojevi deponovani sa protokom plazma gasa od 22 l/min He, koji su imali najmanju raspodelu mikrotvrdoće. Ti su slojevi imali najmanji udeo pora. Najmanju vrednost zatezne čvrstoće spoja od 42MPa imali su slojevi deponovani sa najvećim protokom plazma gasa He. Ti su slojevi imali najveću raspodelu mikrotvrdoće i najveći udeo mikropora. Ispitivanja zatezne čvrstoće spoja pokazalo je da je za sve deponovane prevlake mehanizam razaranja bio na interfejsu između substrata i prevlake.

Kvantitativna analiza ukupnog sadržaja pora u WC17Co slojevima pokazala je da su izmerene vrednosti u direktnoj vezi sa protocima plazma gasa He. Na prikazanim mikrofotografijama jasno se uočavaju različiti udeli pora u deponovanim slojevima. Najmanji udeo pora bio je u slojevima WC17Co prevlake deponovane sa protokom plazma gasa od 22 *l/ min He. U tim slojevima je ukupan udeo pora bio 1%. U slojevima deponovanim sa protokom plazma gasa od 12 l/min He, ukupan udeo pora je bio 1,3%. Najveći udeo pora je bio u slojevima deponovanim sa protokom plazma gasa od 32 l/min He. U tim slojevima pore su izraženije i grublje sa udelom od 2,5%. U svim slojevima ukupan udeo pora bio je u propisanim granicama od 0,5 do 3% (Material Product Data Sheet, 2011), (Turbojet engine-standard practices manuel TURBOMECA).*

Mikrostruktura svih prevlaka je lamelarna. Na mikrofotografijama se vidi da su istopljene čestice praha pravilno razlivene. Slojevi prevlake deponovani su kontinualno bez prisutnih mikro i makropukotina kroz slojeve. U slojevima nisu prisutne neistopljene čestice praha, precipitati i interlamelarne pore. U deponovanim slojevima svih prevlaka jasno se uočavaju ravnomerno distribuirane karbidne faze u žilavoj osnovi kobalta. Na svim mikrofotografijama jasno se uočavaju svetla polja metalnih faza i tamnosiva polja karbidnih faza. U svetlim poljima prisutna je polazna metalna faza Co i metalna faza W koja potiče od delimične razgradnje polaznog kubnog monocarbidea WC (Saha, et al., 2010, pp.592-595). U sivim poljima prisutna je polazna faza kubnog monokarbidea WC i karbidne faze koje su nastale razlaganjem kubnog karbida WC u karbide tipa W_2C,W_3C i mešoviti karbid ή - Co_3W_3C (Saha, et al., 2010, pp.592-595), (de Villiers Lovelock, 1998, pp.357–373).

Zaključak

Plazma-sprej postupkom (APS) deponovane su prevlake WC17Co sa protocima plazma gasa od 12 l/min, 22 l/min i 32 l/ min He. U radu su analizirane mehaničke karakteristike deponovanih slojeva i mikrostrukturne karakteristike na svetlosnom mikroskopu. Morfologija čestica praha ispitana je na (SEM) skening elektronskom mikroskopu. Na osnovu izvršenih analiza došlo se do određenih zaključaka.

Morfologija čestica praha WC17Co je sfernog oblika i tipična je za čestice praha koje se proizvode tehnikom suvo raspršavanje/sinterovanje.

Vrednosti mikrotvrdoće i čvrstoće spoja deponovanih slojeva bili su u direktnoj vezi sa protocima helijuma kao plazma gasa. Svi deponovani slojevi imali su vrednosti mikrotvrdoće u propisanim granicama od 850 do 1300 $HV_{0.3}$. Najravnomerniju raspodelu mikrotvrdoće imali su slojevi deponovani sa protokom plazma gasa od 22 l/min He. Ti su slojevi imali najmanju razliku mikrotvrdoće između maksimalnih i minimalnih vrednosti (289 $HV_{0.3}$).

Vrednosti zatezne čvrstoće spoja slojeva deponovanih sa protocima plazma gasa od 12 l/min i 22 l/ min He bile su u propisanim granicama po standardu (min. 45 Mpa). Ti su slojevi imali dobru adhezionu čvrstoću sa supstratom i dobru međulamelarnu kohezionu čvrstoću. Najveću vrednost čvrstoće spoja od 49 MPa pokazali su slojevi deponovani sa protokom plazma gasa He, koji su imali najmanju raspodelu mikrotvrdoće. Ti su slojevi imali najmanji udeo pora. Najmanju vrednost zatezne čvrstoće spoja od 42 MPa imali su slojevi deponovani sa *najvećim protokom plazma gasa He. Ti su slojevi imali najveću raspodelu mikrotvrdoće i najveći udeo mikropora.*

Kvantitativna analiza ukupnog sadržaja pora u WC17Co slojevima pokazala je da su izmerene vrednosti u direktnoj vezi sa protocima plazma gasa He. Najmanji udeo pora bio je u slojevima WC17Co prevlake deponovane sa protokom plazma gasa od 22 l/min He. U tim slojevima je ukupan udeo porao bio 1%. U slojevima deponovanim sa protokom plazma gasa od 12 l/min He, ukupan udeo pora bio je 1,3%. Najveći udeo pora bio je u slojevima deponovanim sa protokom plazma gasa od 32 l/ min He. U tim slojevima pore su izraženije i grublje sa udelom od 2,5%. U svim slojevima ukupan udeo pora bio je u propisanim granicama od 0,5 do 3%.

Mikrostruktura svih prevlaka je lamelarna. Slojevi prevlake su deponovani kontinualno bez prisutnih mikro i makropukotina kroz slojeve. U slojevima nisu prisutne neistopljene čestice praha, precipitati i interlamelarne pore. U deponovanim slojevima svih prevlaka jasno se uočavaju ravnomerno raspoređene karbidne faze u žilavoj osnovi kobalta. U svetlim poljima prisutna je polazna metalna faza Co i metalna faza W koja potiče od delimične razgradnje polaznog kubnog monokarbidea WC. U sivim poljima prisutna je polazna faza kubnog monokarbidea WC i karbidne faze koje su nastale razlaganjem kubnog karbida WC u karbide tipa W_2C*,*W_3C *i mešoviti karbid ή –* Co_3W_3C.

Dobijeni rezultati su pokazali da protok plazma gasa He bitno utiče na mehaničke karakteristike slojeva i udeo pora u prevlakama. Ispitivanja su potvrdila da su najbolji slojevi deponovani sa protokom plazma gasa od 22 l/min He. Prevlaka sa najboljim mehaničkim i strukturnim karakteristikama je testirana i homologovana na rukavcu vratila glavnog rotora helikoptera „gazela H42" letnim ispitivanjem u trajanju od 50 časova u VZ „Moma Stanojlović" u Batajnici.

Ključne reči: *prevlake, kobalt, depoziti, gas, protok gasa, plazma, prah, osobine.*

Datum prijema članka/Paper received on: 06. 07. 2012.
Datum dostavljanja ispravki rukopisa/Manuscript corrections submitted on: 23. 02. 2013.
Datum konačnog prihvatanja članka za objavljivanje/ Paper accepted for publishing on: 25. 02. 2013.