TiC-based coatings deposition using electric discharged plasma

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Abstract. The aim of this work was to research the possibility of using coaxial magneto plasma accelerator for TiC-coatings deposition on steel substrates. As a result, coatings with 0.01 m² area was deposited. They were researched using XRD, SEM; also, the nanohardness on cross section of coating was measured. The influence of energy and carbon load on phase content, average hardness and microstructure is shown. It is established that the finest microstructure and average nanohardness is 15.3 GPa are achieved at energy W = 46.7 kJ and carbon load of 2.0 grams.

1. Introduction

TiC-based coatings are characterized as wear resistant, hard, strength, corrosion and oxidation resistant material [1]. Moreover, TiC is refractory and thermally stable material, which has low friction coefficient and high thermal and electric conductivity [2]. TiC-based coatings can be deposited using different techniques such as laser cladding [1], laser assisted CVD [2] or plasma assisted CVD [3, 4], ion plating [5–7] and PVD [8].

There are certain problems with TiC deposition methods in spite of wide variety of them. They are low adhesion, residual stress, deposition time and substrate preheating to high temperature 500-1050 °C [3].

One of the possible ways to improve this is the implementation of coaxial magneto plasma accelerator (CMPA) generating supersonic jet of high current plasma. Previous investigations demonstrate possibility of using that kind accelerator for TiC/Ti-coatings on copper substrate with a high degree of adhesion [9]. In this work, TiC-coatings were deposited on steel substrates. The average hardness at cross section of coatings reached 15.3 GPa and were achieved at maximum energy of process W = 46.7 kJ. The influence of carbon load and energy output on coating hardness are shown.

2. Experimental

TiC-based coatings were deposited on the steel substrates using high voltage high current coaxial magneto plasma accelerator with power supply from capacity energy storage. CMPA unit is shown in Figure 1. CMPA is designed as a two coaxial titanium electrodes. One of them is central electrode placed in barrel electrode with insulated gap between them. Insulated gap plays a role of plasma structure forming channel. Electrodes are placed into the inductor which stabilizes and accelerates arc discharge after breakdown of electrodes gap. Carbon load was filled into the gap and provided initial

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electric conductivity for breakdown at a switching time. Thus, titanium electrodes were eroded under high current plasma flow, and the eroded material was reacting with filled carbon.

Carbon load was varied in the range of 0.4–2.0 g. Distance between the end of the barrel electrode and the substrate was 150 mm and was constant in all experiments. Area of the substrates was 0.01 m². Charging voltage and capacity in all experiments was 2.8 kV and 14.4 mF, respectively. Summary of initial experimental data is presented in Table 1. Energy parameters at process of synthesis were recorded using Tektronix TDS 2012B digital oscilloscope. Obtained waveforms were calculated respectively to transformation coefficients of Rogowski transformer and resistive voltage divider.

Deposited coatings were researched by methods of X-Ray diffractometry (XRD) using Shimadzu XRD6000S diffractometer. Samples were prepared as polished cross sections, and nanoharndess was measured on them using NANO Hardness Tester NHT-S under 300 mN load. After indentation, the samples were etched using a mixture of ammonium nitrate (65%), hydrofluoric acid HF (40%) and glycerin (87%). Etching mixture was applied to the polished side during 20 seconds and was washed by water flow. The microstructure of the etched samples was investigated using scanning electron microscope Hitachi TM3000 and Quanta 200 3D.



Figure 1. CMPA design (a) and waveforms of the process (b). 1 - central electrode; 2 - barrel electrode; 3 - central electrode insulator; 4 - carbon-filled gap; 5 - inductor; 6 - steel case; 7 - CMPA body; 8 - insulation; 9 - chamber; 10 - plasma jet; 11 - substrate.

3. Results and discussion

As a result, coated samples for different deposition conditions were obtained. Figure 2 presents typical coated samples obtained at different conditions respectively to the Table 1. Coatings change exterior from metallic to matted according to increasing of carbon load. Moreover, the relief of the coatings becomes more uniform.

#	m _C [g]	U _{ch} [kV]	C _{ch} [mF]	Uarc [kV]	Iarc [kA]	Parc [MW]	W [kJ]
1	0.4	2.5	14.4	0.8	140	122	24
2	0.5	2.8	14.4	1.3	133	132	35.7
3	1.5	2.8	14.4	1.55	117	145	42.0
4	2.0	2.8	14.4	1.6	141	180	46.7

Table 1. Initial experimental data and energy parameters of the deposition process.



Figure 2. Substrate surface after deposition under different carbon load: a) 0.5 g; b) 1.5 g; c) 2.0 g.

XRD-investigations demonstrate TiC fraction increase with carbon load increase. The level of released energy influence on TiC proportion also. Besides TiC in coating Ti, Fe and Fe₃C also exist. XRD-pattern in Figure 3 shows the absence of carbon in the coating. The presence of Ti is caused by the erosion of electrodes and shortage of carbon load. Iron appears due to the contact of high temperature plasma jet with substrate and consequent mixing, and the reaction between iron and carbon occurs with iron carbide synthesis.



Figure 3. XRD-pattern of coating obtained in experiment 4 (Table 1) and influence of energy on TiC fraction.

Nanohardness measuring on polished cross section of coating shows upward trend of average hardness with an increase of released energy level, carbon load and proportion of TiC in coating as demonstrated in Figures 4a, 4b and 4c.





Figure 4. Influence of experimental conditions on the average nanohardness of the coatings.

Etched cross-sections of deposited coatings were investigated using scanning electron microscopy, which allowed identifying their microstructure presented in Figure 5. The thickness of coatings varies in the range of $30-300 \,\mu\text{m}$ and grows with increasing of carbon load and released energy. SEM results demonstrate polycrystalline structure of the coatings. Microstructure and grain boundaries become more uniform and ordered, while carbon load and energy of the process are increasing. The finest microstructure was reached in experiment with m = 2.0 grams and $W = 46.7 \,\text{kJ}$ as it can be seen Figure 5c. On the basis of the image, it can be argued about uniform distribution of TiC grains in Timatrix, which allow reaching the highest level of nanohardness for described conditions.



Figure 5. Microstructure SEM-pictures of coatings deposited at different conditions.

4. Conclusion

Investigation results demonstrate the possibility of using coaxial magneto plasma accelerator of supersonic electrodischarged plasma jet for TiC- based coatings deposition with the thickness up to $300 \,\mu\text{m}$ in a short time cycle about $500 \,\mu\text{s}$. Influence of released energy and carbon load mass on coating thickness, microstructure and hardness is established.

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