ELECTRO SPARK DEPOSITION OF TIB_2 LAYERS ON TI6AL4V ALLOY

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Received: 23.10.2015
Accepted: 24.02.2016

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Abstract
The electro spark deposition (ESD) method was used to create wear resistant layers of TiB_2 ceramic onto Ti6Al4V titanium alloy. Various deposition parameters and ways of deposition were employed in this study. It was showed that the TiB_2 layer on Ti6Al4Vcan be successfully created even using hand operating ESD equipment. Then, the microstructures of the obtained TiB_2 layers on Ti6Al4Vwere investigated using scanning electron microscope. Finally optimal conditions of the electro spark deposition were determined with respect to the obtained microstructure. It was also demonstrated that by using of multiple runs dense layer of almost pore free TiB_2 ceramic layer on Ti6Al4V titanium alloy can be successfully prepared.

Keywords: electro spark deposition, titanium alloys, titanium diboride, coating

1 Introduction
It is generally accepted that the protecting ability of the coated surface depends mainly upon the physical and chemical properties of the developing oxide film as well as structural heterogeneities on the substrate surface [1-2]. The Electro spark deposition (ESD) process is one of the most promising advances in metallurgical coatings for use in demanding environments on various substrates including titanium and its alloys [3-5].
Electro spark deposition enables to product hard and wear-resistant coatings on metallic substrates. The only essential conditions are that both, substrate and electrode materials, ought to be electrically conductive. Significant advantage is that due to the diffusion of electrode material into the substrate and vice versa good bonding between the layer and the substrate occurs there. The deposited layer is often composed from the substrate material, electrode material and also from the new phases due to the chemical reactions taking place during deposition and rapid cooling after deposition. The advantage of this method is the ability to melt and deposit material with high melting point and to achieve good bonding of such material also to low temperature melting substrates, all at low thermal input. Therefore the heat affected area is small, thus creating only minimal thermal stresses and minimum of cracks in deposited coating.
ESD is a pulsed micro-welding process that was developed originally for small scale and precision repair of high value components which were either worn or misfabricated [6, 7]. Basically the principal industrial applications include repair of defects in casting moulds and

DOI 10.12776/ams.v22i1.628 p-ISSN 1335-1532
e-ISSN 1338-1156

injection moulding tooling. In general, all ESD systems contain a capacitor-based power supply that produce short duration high current pulses through a vibrating wire consumable electrode - see **Fig. 1**. The consumable electrode material is deposited onto the work piece by means of electric sparks in a manner reverse to spark erosion (the electrode is the anode and the work piece is the cathode). When the capacitor energy is released, the direct current generates a plasma arc at a high temperature (8000 - 25000 °C) between the tip of the electrode and the work piece. The plasma arc ionizes the consumable and a small quantity of molten electrode material is transferred onto the work piece. The transfer of material is rapid and the self-quenching is extremely fast.

Fig. 1 Principal scheme of ESD process [8] and examples of possible deposition of coating by ESD method at desired surface areas - TiB_2 deposited on HSS steel drill cutting edge (middle) or on steel plate (right)

Thanks to the short duration, high current pulses, the ESD process imparts a low heat input to the substrate material, resulting in little modification of the substrate microstructure. Therefore, the process offers an advantage over fusion welding processes (including arc, laser and resistance welding) when repairing materials that are difficult to weld because of poor heat affected zone properties (e.g. cracking, high hardness, low toughness). Components can be restored to their original dimensions, because with such low heat input the bulk substrate material remains near to ambient temperature with thermal distortion, shrinkage and high residual stresses avoided. Moreover, the process generates a very good bonding between the coating and the substrate. Electro spark deposition can also be considered as a process to increase the wear and the erosion resistance of required surface areas or their parts as also indicates **Fig. 1**. Therefore ESD deposition is also important for various types of production technologies where the wear resistance of tools used during preparation of materials such as extrusion process [9, 10], ECAP process [11 – 13] and metal foam production [14, 15] will increase life time of tools [16] thus reducing the costs of preparation of final material.
This method is also suitable for the surface treatment of titanium and titanium alloys. ESD method will enable to use these materials (thanks to increase of their surface hardness and wear resistance) as sonotrode cutting tools [17] and also for biomedical applications in tooth impacts [18]. Therefore the main aim of this article was to study the way of ESD creation of hard and wear resistant TiB_2 layer on titanium alloy Ti6Al4V and resulting microstructure.

2 Experimental materials and methods

SZ 08 laboratory equipment (see **Fig. 2**) was used to perform ESD deposition of TiB_2 on Ti6Al4V substrate using TiB_2 working electrode. SZ 08 has maximal power of 600W at 220V

DOI 10.12776/ams.v22i1.628 p-ISSN 1335-1532
e-ISSN 1338-1156

AC input power supply. On this equipment the following parameters can be adjusted: working frequency from 50 to 500 Hz, the voltage 20V to 100V, power performance from 10% to 100%, direction of rotation of working electrode (right or left), rotational speed 0-60 rpm and there is also a possibility to use protective atmosphere.

As a substrate material CP Titanium Ti6Al4V (Grade 5) was used with chemical composition of components in wt. %: Al 6, V 4, Fe max 0.25, O max 0.2 and Ti balanced. Ti6Al4V has excellent biocompatibility, especially when direct contact with tissue or bone is required. Ti6Al4V has poor shear strength which makes it undesirable for bone screws or plates. It also has poor surface wear properties and tends to seize when in sliding contact with itself and other metals. Surface treatments such as nitriding and oxidizing are usually used to improve the surface wear properties.

The cylinders with diameter of 10 mm and length of 10 mm were prepared from bulk Ti6Al4V ingots by electrical discharge machining and turning to investigate the ESD deposition of TiB_2 onto this substrate. The used TiB_2 electrodes (diameter of 5 mm and length of 100 mm) were almost 100% dense and were obtained from company RHP-Technology GmbH, Austria.

At the beginning, the set of tests at different voltages: 16V, 20V, 30V, 40V and 60V at a constant power performance of 50 % , working frequency of 400 Hz , the rotational speed of 30 rpm (direction right), using a protective atmosphere of argon (with 99.996 % purity and flow rate of 14 l/min) were prepared. The only one deposit run was done for each voltage to create the TiB_2 layer onto the top surface of Ti6Al4V cylinder. The each run of the electrode above the substrate were performed close to each other to cover surface continuously.

Further, the possibility of multiple deposition of TiB_2 electrode material on previously deposited layer of TiB_2 on Ti6Al4V substrate was investigated. For this reason 5 and 10 layers were deposited at a voltage of 20V, 30V and 40V under the same condition as mentioned above. The each run of the electrode above the substrate/layer were performed close to each other to cover surface continuously and every next layer was applied after 90° rotation of the sample clockwise around axis of cylinder.

Fig. 2 ESD equipment SZ-08 for electro spark deposition

Hardness was measured using IndentaMet 1100 Series indenter. The measurements were realized according to EN ISO 6507-1. The parameters of measurement were: HV 0.1, the time of indentation was t = 10 s, applied force F = 0.9807 N. All test were performed at room temperature T = 22 °C. Microstructure of samples was observed using electron microscope JEOL 7600F, equipped with a Schottky thermal-emission cathode (thermal FEG -W-coated

DOI 10.12776/ams.v22i1.628 p-ISSN 1335-1532
e-ISSN 1338-1156

ZrO_2) as well as energy-and wavelength spectrometers from Oxford Instruments. At least 10 measurements of created TiB_2 layer thickness were performed at 5 different locations.

3 Results and discussion

At first only 1 deposition run at various voltages and above mentioned ESD conditions were performed. The deposited layer at 16V was discontinuous; the created layer evidently does not cover fully the substrate surface (**Fig. 3**). It indicates that the used voltage was insufficient to melt the amount of electrode material required for sound layer. The maximum observed thickness of such layer was between 13 - 36 μm (see **Table 1**). Besides that, under the coating layer a heat affected zone in the substrate approximately 30 - 35 μm thick was observed (see **Figs. 3, 4**).

When the voltage was increased to 20V the deposited layer becomes continuous, but with the enclosed pores in the coating layer. The significantly smaller heat affected zone was observed in this case 5 – 10 μm. The thickness of such layer was between 17 - 71 μm.

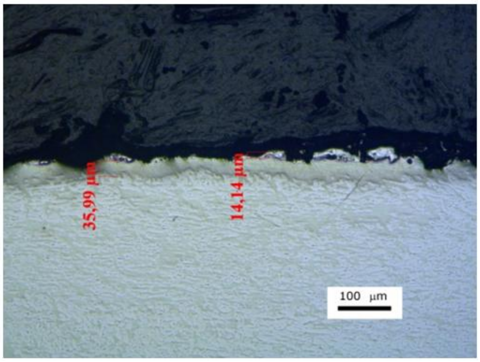

Fig. 3 TiB_2 layer on Ti6Al4V at 16V with some measurements of layer and HAZ thickness

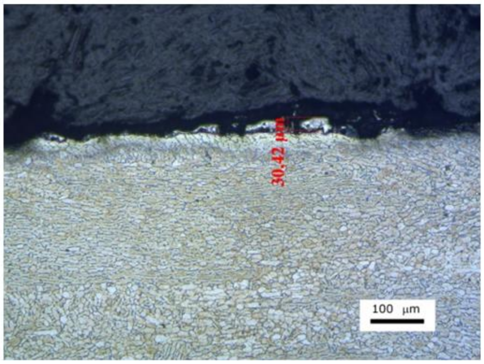

Fig. 4 TiB_2 layer on Ti6Al4V at 16V – etched, measurements of HAZ thickness

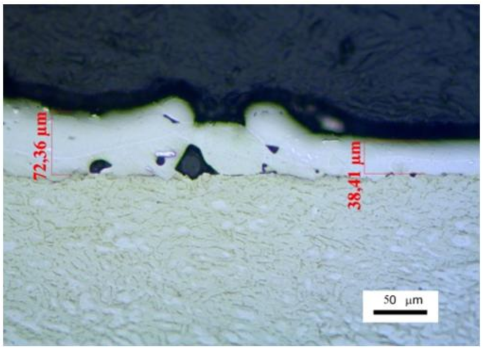

Fig. 5 TiB_2 layer on Ti6Al4V at 40V

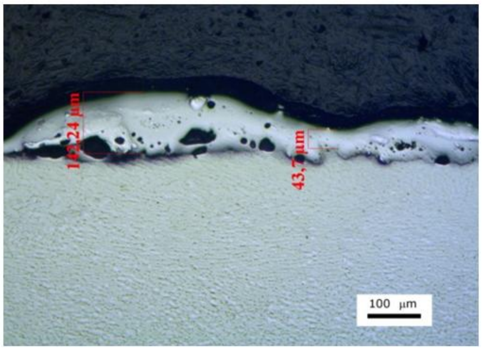

Fig. 6 TiB_2 layer on Ti6Al4V at 60V

The sample prepared at 40V satisfied the requirements for sufficient thickness and continuity of the deposited layers: Created layer adheres well to the base material and layer is continuous (**Fig. 5**). The thickness of such layer was between 32 - 80 μm. As showed later experiment these parameters are best for the application of TiB_2 layer on Ti6Al4V substrate.

DOI 10.12776/ams.v22i1.628 p-ISSN 1335-1532
e-ISSN 1338-1156

Finally the sample at 60V was also prepared. It was confirmed that with the increasing voltage the thickness of deposited layer also increases. The coating reached the thickness values in the range of 43 µm to 142 µm, however it possesses significantly high amount of small pores (see **Fig. 6**) in comparison with layer deposited at 40V. The presence of such small pores will considerably decrease layer mechanical and wear properties.

Table 1 Measured thickness of TiB_2 layers on TiAl4V after 1 deposition run

U [V]	Thickness of layer [µm]	Description of layer
16	13-36	Heat affected zone under layer 30 – 35 µm, discontinuous
20	17-71	Continuous, porous, HAZ of 5 – 10 µm
40	32-80	Continuous, very small porosity
60	43-142	Continuous, thick, higher small porosity

Table 2 Measured thickness of TiB_2 layers on TiAl4V after 5 and 10 deposition runs

U [V]	Thickness of layer after 5 runs [µm]	Thickness of layer after 10 runs [µm]
20	22-65	35-79
30	39-43	29-85
40	35-70	51-114

In the second group of experiments (5 and 10 runs deposition) it was confirmed that the thicknesses of layers after multiple runs increases with the number of runs and also with increasing voltage. Anyway, multiple runs better cover the surface of the substrate, thus increasing its properties. The heat affected zone was again observed for multiple runs at 20V (see **Fig. 7**). In this case the 62 µm thick heat affected zone was observed due to multiple runs deposition in comparison of 30 – 35 µm at 1 run. For higher voltages the heat affected zone was unable to observe. For this reason the element analysis map was performed on this sample to study the complexity of such layer structure (**Fig. 8**).

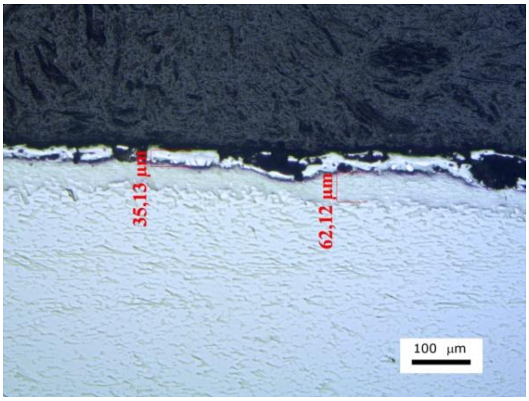

Fig. 7 TiB_2 layer on Ti6Al4V at 20V – 10 deposition runs, depicted layer thickness and heat affected zone measurements

Element maps clearly indicate approximately 30 µm thick coating layer (see boron and titanium maps in **Fig. 8**). The heat affected zone is not visible, except some variations in vanadium

DOI 10.12776/ams.v22i1.628 p-ISSN 1335-1532
e-ISSN 1338-1156

concentration distribution below created layer. The **Fig. 8** also confirmed that there is a diffusion bond between substrate (Ti6Al4V) and deposited material (TiB_2). During the ESD deposition Al is partially melted and is diffusing to the upper part of layer where it creates complex Al-Ti oxides. However, not only Al but also V is diffusing into the thick deposited layer of TiB_2. Unfortunately their concentration is so small that XRD analysis of them is very difficult to perform and it will be the goal of our future study on these layers.

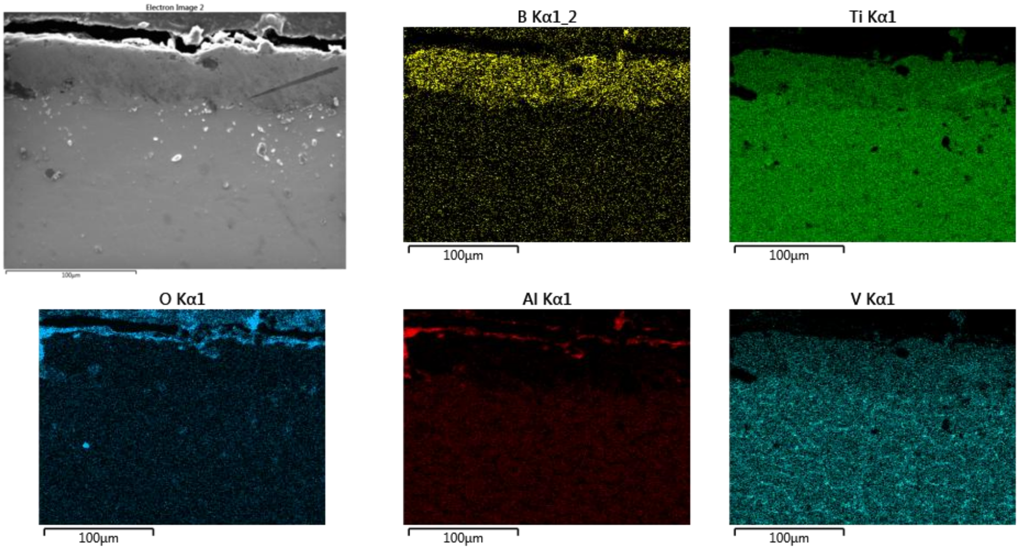

Fig. 8 Element map of TiB_2 deposited on Ti6Al4V at 20V – 10 deposition runs

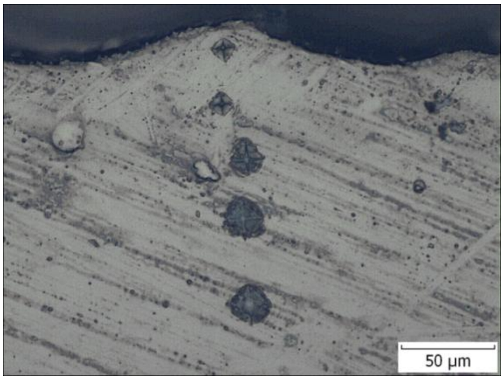

Fig. 9 Microhardness of TiB_2 deposited on Ti6Al4V at 40V and 10 deposition runs. The hardness values from top to bottom are: 1016, 951, 470, 347 and 355 MHV0.1

The measured hardness indicates that the ESD deposited layer significantly increases the hardness of Ti6Al4V surface - increase from approx. 350 HV in substrate up to 1000 HV in deposited layer (**Fig. 9**). Baksa et al. [19] has observed similar values of microhardnes 345 and 1190 for substrate and deposited TiB_2 layer, respectively. However for example the average hardness value of TiB_2 ESD deposited on steel was observed 2642 ± 223 MHV1 [20, 21]. The reason is simply the fact that during the ESD deposition of TiB_2 on Ti6Al4V more complex

DOI 10.12776/ams.v22i1.628
p-ISSN 1335-1532
e-ISSN 1338-1156

composite layer is created with new complex phases: From **Fig. 8** it is evident that diffusion of Al and V occurs during deposition. While V diffuses in a gradient way from substrate to the layer surface, Al creates rich region of alumina on the top of layer surface. Moreover TiB_2 can decompose during the deposition thus creating also hard TiB phase. It is evident that there are created new complex phases during ESD deposition can, but at very low concentration of them. Therefore they are very difficult to investigate. These phases affect the resulting hardness of the composite layer in comparison to the hardness HV 2600 of pure TiB_2. Anyway, due to this diffusion significantly good bonding between substrate and deposited layer is created.
Finally, it can be concluded that TiB_2 layer with high density and small porosity can be deposited onto Ti6Al4V substrate in protective argon atmosphere in the thickness of 50 – 120 mm at optimal conditions: 40V, constant power performance of 50 %, working frequency of 400 Hz, the rotational speed of 30 rpm (direction right), atmosphere of argon (with 99.996 % purity and flow rate of 14 l/min) and 10 deposition runs.

4 Conclusions

ESD method was successfully used for deposition of TiB_2 coating layers on Ti6Al4V substrate. EDX micro analysis confirmed the presence of boron from working electrode on the surface of substrate inside of deposited coating layer. EDX analysis also confirmed diffusion of aluminium and vanadium from substrate to the layer thus creating good bonding between layer and substrate. Significantly higher hardness was observed on the top of the layer 1000 MHV0.1 in comparison with the substrate hardness of 350 MHV0.1. The layer hardness is lower as is the hardness of TiB_2 due to the creation of alumina, TiB and other more complex phases during ESD process. Finally it was shown that TiB_2 layer with high density and small porosity can be deposited onto Ti6Al4V substrate in protective argon atmosphere in the thickness of 50 – 120 mm at optimal conditions: 40V, constant power performance of 50 %, working frequency of 400 Hz, the rotational speed of 30 rpm (direction right), atmosphere of argon (with 99.996 % purity and flow rate of 14 l/min) and 10 deposition runs.
The obtained results confirmed the possibility to improve the surface of Ti6Al4V thus enabling to this material new industrial applications.

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Acknowledgements

Authors are grateful for the support of experimental works by project of the Slovak Research and Development Agency under the contract VMSP II–0009–09 and Slovak Grant Agency under contracts VEGA 1/0189/12, 1/0385/15 and 2/0158/13.