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Manufacturing and characterization of Tantalum microplasma Coatings for Biomedical Application

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The relevance of research

Currently, there has been growing interest in the use of biocompatible tantalum (Ta) as materials for medical implants. Due to its high chemical stability and hardness, Ta is very promising as an orthopedic biomaterial.

According to modern research in this field, coatings for orthopedic implants should be porous; the recommended range of pore sizes from 20 microns to more than 100 microns, so that bone tissue and blood vessels can grow through them.

The use of robotic microplasma spraying (MPS) allows for the precise spraying of coatings on implants of complex shape, such as endoprostheses of the elbow and hip joints.

The goal of this study is

the microplasma spraying (MPS) of Ta wire onto Ti medical implants for obtaining the hard coatings of a specified thickness and porosity

Research background

the research was performed in the sector of high-speed air-plasma cutting and spraying of the Center for Advanced Development (CER) VERITAS EKSTU named after D. Serikbayev

The main aim of this research is developing cheaper and patient specific medical implants (Ti-based alloy/ Ta coating)

We pursue two approaches:

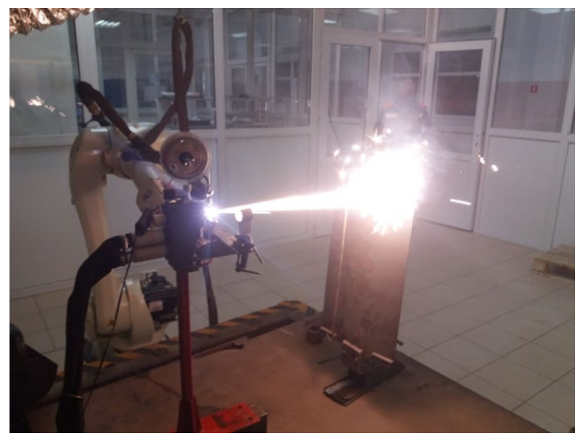
1) Implant lathing by numerically controlled machines, followed by surface purification and quality control.
2) Microplasma spraying of biocompatible Ta or Ti or Zr wires and hydroxyapatite (HA) powders onto implants using an industrial robot.

Goal: process optimizing & prototype development

Samples of specific implants were provided by the Kazakhstan Research Institute of Traumatology and Orthopedics

Manufacture of medical implants with biocompatible coatings

Hip joint and elbow joint endoprosthesis modified by the Research Institute of Traumatology and Orthopedics of the Republic of Kazakhstan.



Implant lathing at Computerized and Numerically Controlled machines produced by DMG MORI (Germany)

Microplasma spraying (MPS) of biocompatible Ta wires onto Ti implants using an industrial robot.

Implants with metal biocompatible coatings

Material and Methods

- To manufacture the medical implants the CTX 510 ecoline CNC turning and milling machine and DMU 50 CNC milling machine (DMG MORI AG, Germany) have been used.
- MPS of tantalum coatings on titanium alloy substrates and directly on the elbow joint prosthesis was performed with an industrial robot-manipulator Kawasaki RS-010LA (Kawasaki Robotics, Japan) and a microplasmatron MPN-004 manufactured by the Institute of Electric Welding named after E.O. Paton (Ukraine).
- High purity tantalum wire 0.3 mm in diameter was applied to Grade 5 ELI titanium alloy substrates, standard composition in wt.% (Max): Ti - base, Fe -0.25-0.40, N - 0.05%, O - 0.13-0.20, Al - 5.50-6.75, C - 0.08, V- 3.50-4.50, N - 0.015.
- In order to reveal microstructural features, samples of mechanically polished tantalum coated titanium cross sections were further subjected to chemical etching for 5 seconds using a solution of 13 ml of HNO_3 + 2 ml of HF + 35 ml of H_2O.
- To assess the porosity of the coatings, the images of their microstructure were processed using the ImageJ computer program, which allows to measure pore sizes and porosity of the coating using ASTM E2109-01 standard (i.e., the percentage of pores in the coating over the area per detected pores relative to the entire section area coatings), highlighting inclusions that differ in shades of gray or brightness.
- **No clinical tests on humans or animals have been carried out.**
- Before microplasma spraying, **a gas-abrasive treatment of substrates was carried out** followed by ultrasonic cleaning in medical alcohol for 15 minutes.

The scheme of preliminary robotic 3D scanning of an implant for generating a robot movement program during microplasma spraying of a coating on an implant of a complex shape

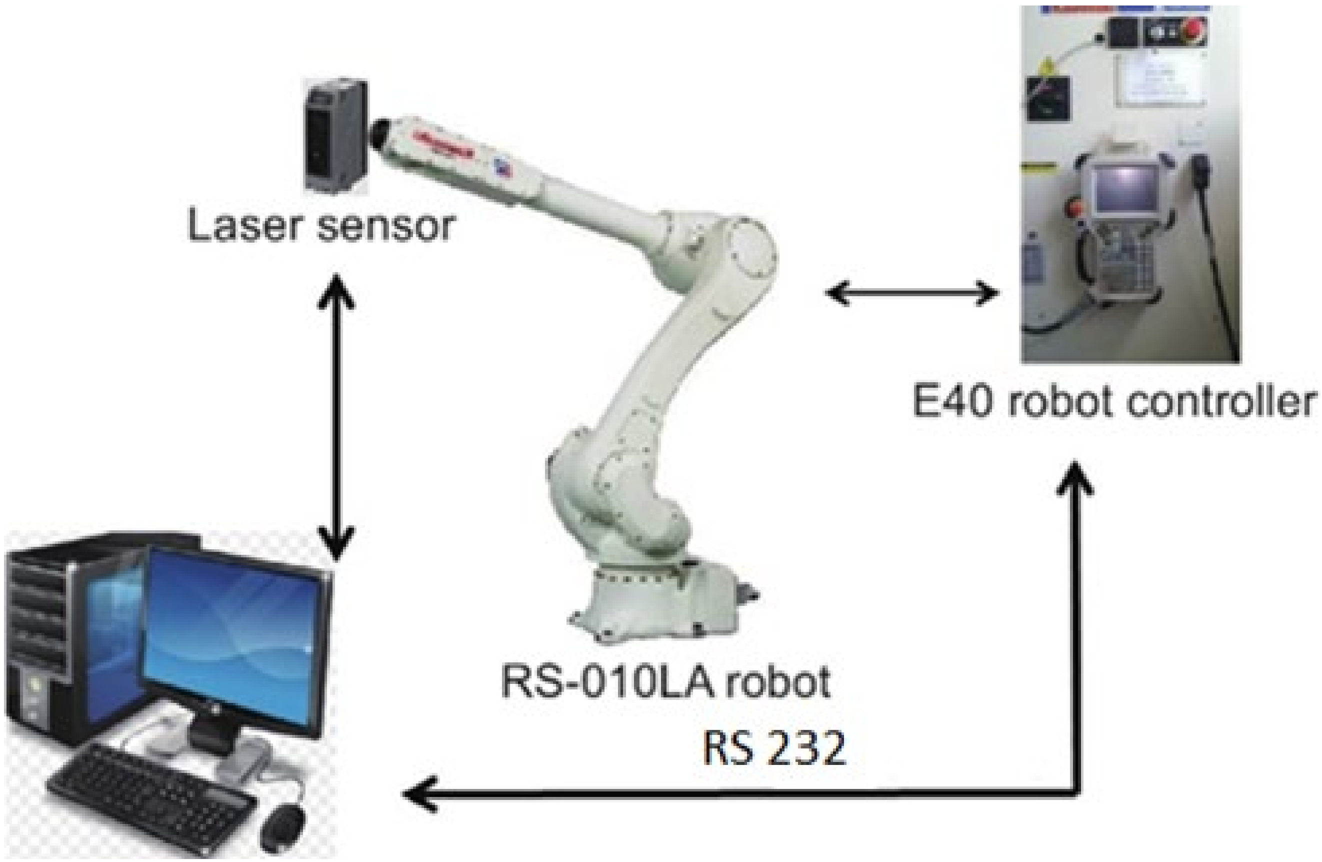
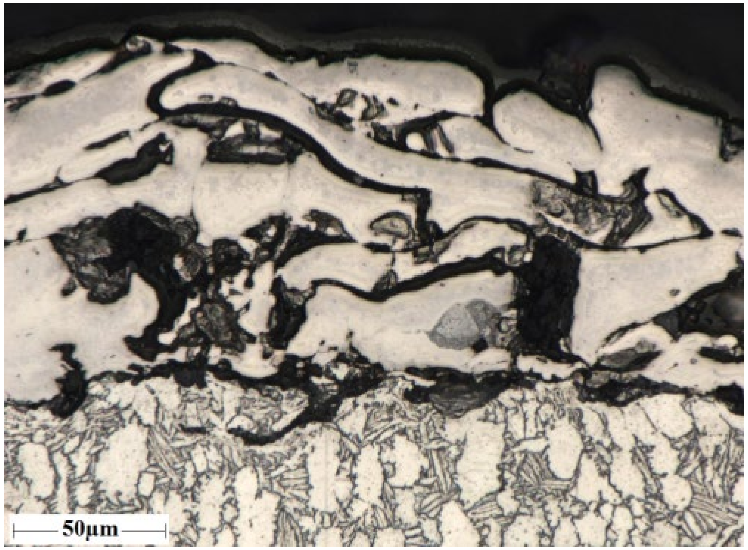
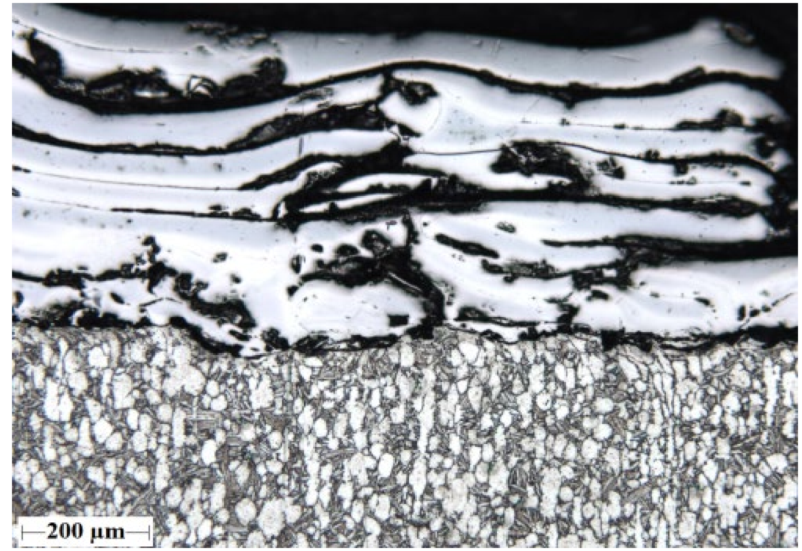


Table 1. The parameters of microplasma spraying (MPS) of Ta coating

Parameters, (units)	Parameter Value	
	Mode 1	Mode 2
Current, (A)	45	35
The consumption of plasma-forming gas Ar, (slpm)	300	300
Spraying distance, (mm)	80	80
Wire consumption (m/min)	6	6
The number of passes of the plasma jet	3	2

Microstructure of Ta-coatings on a Ti alloy substrate

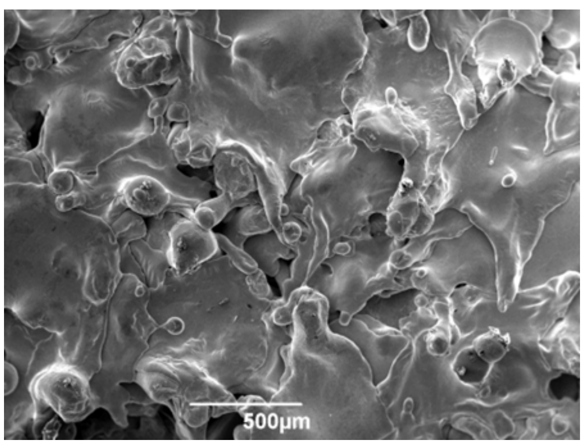




Mode 1

Mode 2

The cross sections of the coatings applied in Mode 1 and Mode 2 (Table 1)

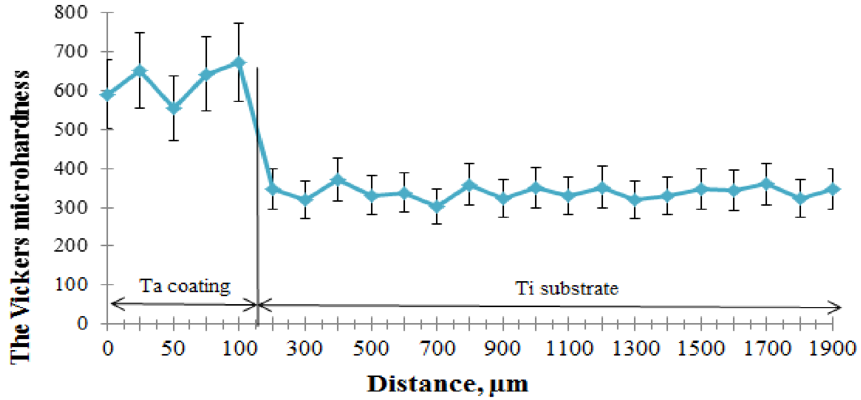


- The Ta coatings have a layered (lamellar) structure formed by the Ta wire particles melted and deformed upon impact on the substrate. Cracks in the coating and substrate are not observed.
- The pore size in the coatings varies from 20 µm to 50 µm, reaching 200 microns on the surface of the coating (open pores)

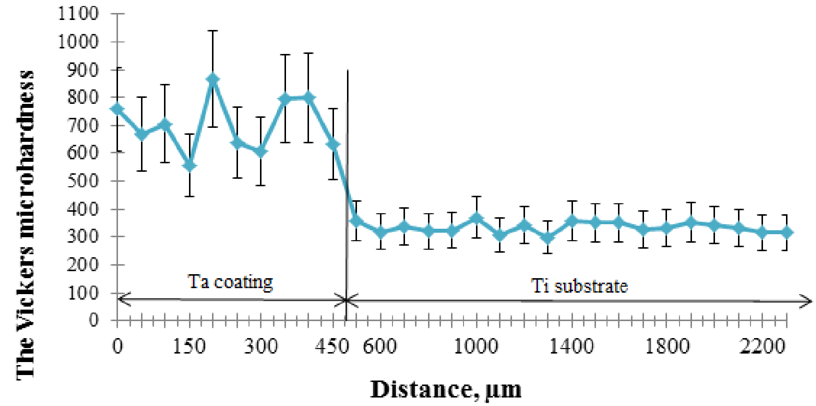
SEM image of the surface of Ta-coating applied in Mode 1

The experimental profiles of the distribution of microhardness in depth from the surface of Ta coatings deposited on Ti alloy substrates according to the modes indicated in Table 1

Mode 1



Mode 2



The Vickers microhardness was measured using a DuraScan-20 microhardness tester (EMCO TEST, Austria) in depth from the coating surface at the cross sections of Ta-coated Ti samples using a load of 200 g with an exposure at a maximum load of 5 s, with the calculation average value according to the results of 10 measurements.

The endoprosthes of the elbow joint made of titanium alloy before and after MPS of Ta coating

The average microhardness of Ta - coatings deposited
in mode 1 was 622.4 ± 28 HV,
in mode 2 it was 702.3 ± 80 HV,
which was higher than the average hardness of Ti substrate
(337.6 ± 14 HV).

Discussion

- A significant variation in the microhardness of the Ta coating is due to the presence of pores in the coating and the interface between the Ta wire particles melted and deformed upon impact with the substrate.
- The microhardness distribution profiles in depth from the surface show that the Ta microhardness is somewhat lower at the surface, which may be due to the presence of open pores on the coating surface. At the same time, regions with increased microhardness of the substrate (390 ± 2 HV) are observed at the Ti boundary of the coated substrate, which may be due to hardening of the solid solution as a result of dissolution of Ta in Ti during the MPS. It can be assumed that during the deposition of coatings, the substrate surface did not heat up to high temperatures, since there is no increased porosity at the coating boundary.
- As Balla at al noted in the paper [5], the presence of increased porosity in the boundary regions of Ta coatings on titanium is apparently related to the boiling of Ti, since the boiling temperature of Ti (3287°C) is close to the melting temperature of Ta (3017°C). Measurements of the porosity of the coatings showed that it varies from 10% (Mode 1) to 20% (Mode 2) and is determined by the MPS parameters.
- In order to determine the dependence of porosity on the spraying parameters, additional research is required, but it is obvious that this coating parameter can be controlled, which is very promising for the development of the technology for creating coatings from refractory biocompatible materials.

Conclusions

- The possibility of forming hard coatings of a given thickness and porosity by the method of robotic microplasma spraying (MPS) of Ta wire is shown.
- The parameters of MPS are determined, which ensure the formation of tantalum coatings with a thickness of up to 500 μm and with the presence of pores in the coating with sizes from 20 μm to 200 μm.
- It was found that the microhardness of a microplasma tantalum coating is on average 2 times higher than that of a titanium alloy substrate.
- Robotic MPS of a tantalum wire onto titanium alloy elbow joints implants was carried out using new robot manipulator control algorithms.
- The research results are important for a wide circle of researchers developing technologies for thermal spraying of biocompatible coatings.

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Thank you for watching this!