ANNOTATION

of the dissertation for the degree of "Doctor of Philosophy" (Ph.D.) in the specialty 8D05301-Technical physics

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Title: "ROBOTIC MICROPLASMA SPRAYING OF FUNCTIONAL COATINGS WITH SPECIFIED STRUCTURE AND PROPERTIES"

Keywords: microplasma spraying (MPS), coating, porosity, surface roughness, adhesion, phase composition, biocompatibility, wear resistance, electrical conductivity, electron microscopy, regression analysis.

The main idea of the research: the dissertation is devoted to the problem of robotic microplasma spraying of functional coatings with a predictable structure and certain physical and mechanical properties. Microplasma spraying (MPS) refers to thermal plasma spraying of coatings that are used in various industries as protective coatings, ceramic coatings for electric heating elements, and biocompatible ceramic-metal coatings on medical implants. Among the various processes of thermal plasma spraying, MPS is characterized by a low power of the plasma source (up to 5 kW) and a small diameter of the spray spot (up to 10 mm), which together can reduce material losses during spraying and low heat input to the substrate. MPS, in contrast to conventional thermal plasma spraying, can provide coating of small parts and assemblies, including those with a complex shape, and allows coating thin-walled products without the risk of overheating and warping. Purposeful variation of the main parameters of the MPS, such as the electric current, the plasma-forming gas flow rate, the spraying distance, the feed rate of the sprayed material (wire or powder), the velocity of the plasma jet over the surface of the product, etc., in the long term makes it possible to obtain the required coating structure (in particular, with controlled porosity) and ensure good adhesive strength of the coating, since the degree of heating of the coating particles depends on the time of their stay in the plasma jet and can be controlled by selecting the MPS parameters. The use of robotic methods of plasma spraying makes it possible to accurately maintain such main parameters of the MPS as the spraying distance and the speed of movement of the microplasmatron along the part, increases the productivity and safety of the process.

In the dissertation, the selection of the main parameters of microplasma spraying was carried out using the methods of factorial planning of the experiment and regression analysis, implemented on the basis of a pilot plasma processing site using an industrial robot Kawasaki RS010L (Kawasaki Heavy Industries, Japan), followed by an experimental study of the microstructure and phase composition and testing a number of physical and mechanical properties of the obtained systems "coating-substrate".

The relevance of the research. The problem of obtaining materials with predictable (controlled) structure and properties is one of the main scientific challenges of materials science. The development of functional coatings may be a response to the demand for materials with new characteristics. The coating is a layer or film on the surface of the substrate, different in chemical composition or characteristics (for example, porosity) from the base material (substrate) and carrying a function that is not inherent in the base material. Thus, the development of new compositions of coatings and substrates and the production of new coatings is a promising direction in materials science: while retaining the functional properties of the substrate, the composition acquires new functional properties of the surface due to the coating. Based on the functions that need to be given to the substrate, the material and composition of the coating and methods of its production are selected. The operational reliability of the "substrate-coating" composition is ensured by a combination of mechanical and physical and chemical properties of its constituent materials and good adhesion of the coating to the substrate. Predicting the properties of coatings is also based on the possibility of forming certain microstructures and phases in a given composition. Improvement of methods for obtaining functional coatings with a given structure and properties using modern technological solutions is an important and urgent task. New technologies with the integration of industrial robots into spraying processes make it possible to reach a new level of production, to achieve a significant improvement in surface properties by forming coatings with special properties on the working surfaces of industrial products. Robotic systems for plasma spraying processes can significantly improve the accuracy and quality of spraying, optimize processing and shorten the production cycle. The development of robotic methods for microplasma spraying is an important scientific and technical task, the solution of which is necessary to obtain new methods and approaches of technical physics for the development of new materials with predictable properties.

This research was conducted within the framework of projects with state (grant) funding from the Committee of Science of the Ministry of Science and Higher Education of the Republic of Kazakhstan:

for 2022-2024 AP 13068317 “Development of new robot-manipulator control algorithms for 3D scanning technologies and additive microplasma spraying of coatings” (the project leader - Dr. Kadyroldina A.)

for 2022-2024 AP14869862 “Reliability Enhancement of Medical Implants Through Using Innovative Manufacturing and Coating Techniques” with grant funding from the Committee of Science of the Ministry of Science and Higher Education of the Republic of Kazakhstan (the project leader - Dr. Safarova Y.)

for 2018-2020 AP05130525 "Intelligent robotic system for plasma processing and cutting of large-sized products of complex shape" (the project leader - Dr. Prof. Alontseva D.)

The object of the research is coatings made of biocompatible or wear-resistant or thermoresistive materials (ceramic powders and metal wires) deposited on metal substrates by robotic microplasma spraying, as well as substrate materials.

The subject of the research is the patterns of formation of specified coating microstructures depending on the parameters of microplasma spraying (MPS); structural-phase states of coatings and substrates due to MPS; the effect of MPS parameters on the efficiency of the process and on the physical and mechanical properties of coatings, as well as the choice of optimal MPS parameters.

The research goal is to obtain functional coatings with controlled characteristics by the method of robotic microplasma spraying through ensuring the optimal MPS parameters.

Research objectives:

1) Analyze the current state of the problem of obtaining functional coatings with a controlled microstructure and desired properties, justify the choice of materials, process equipment and experimental research methods for this study.

2) Carry out the planning of a factorial experiment on the MPS of functional coatings to further establish the dependences of the characteristics of coatings on the parameters of the MPS using regression analysis methods

3) Conduct a study of the parameters of metallization figures and spray tracks to establish the distribution of the coating material during MPS and develop further recommendations for robotic MPS of coatings with a uniform and controlled thickness.

4) To carry out robotic MPS of coatings from biocompatible materials with selected MPS parameters on titanium alloy substrates to obtain experimental samples. To obtain by the MPS method a small-sized multilayer resistive electric heating element, consisting of an insulating and electrically conductive layers.

5) Conduct a study of the structure and properties (depending on the functional purpose) of coatings and substrates using optical and electron microscopy, X-ray diffraction analysis, adhesion tests, microhardness tests or measurements of electrical strength and conductivity and establish patterns of influence of MPS parameters on the material utilization factor and on porosity of coatings, to give a scientific justification for the choice of optimal MPS parameters, which make it possible to effectively obtain coatings with desired characteristics.

6) Perform approbation: perform robotic MPS of biocompatible coatings on implants according to the recommended optimal parameters and patent the method of robotic MPS of biocompatible coatings; to carry out a robotic MPS of a protective coating on the worn areas of the crusher plate made of Hadfield steel and to conduct production tests of the restored plate.

Main research methods: methods of planning a factorial experiment, regression analysis, methods of statistical processing of results, experimental methods for studying the structure and properties of materials: scanning and transmission electron microscopy, X-ray diffraction phase analysis, optical microscopy, X-ray energy-dispersive elemental analysis, adhesion tests, measurements of electrical strength of coatings.

Scientific provisions submitted for defense:

1) Patterns of the influence of MPS parameters on the characteristics of functional coatings and scientific justification for choosing the optimal MPS parameters, which make it possible to effectively obtain coatings for various functional purposes with specified characteristics.

2) The method of robotic MPS coating from a biocompatible material, which consists in applying 400 μm thick zirconium coatings with a porosity of 20.5 ± 2.00%, pore sizes up to 300 μm onto a titanium implant. The difference of the method is that the MPS is carried out using a microplasmatron mounted on a robotic arm moving along a given 3D model of the implant at a constant speed of 2.3 m/min, while the control of the thickness and porosity of the coatings is ensured by the exact observance of the MPS parameters selected in accordance with the experiment on factorial planning.

3) The totality of the results of approbation of the MPS robotic system, which has advantages in the accuracy and efficiency of the technological process compared to existing solutions.

Scientific novelty of the work and the main results proved in the dissertation and in publications on the research topic:

1) New patterns of influence of MPS parameters on the efficiency of spraying and characteristics of porosity of functional coatings made of biocompatible and thermoresistive ceramic materials have been established, it has been shown that the spraying distance, current strength and plasma-forming gas flow rate have the greatest influence on the characteristics of porosity and coefficient of utilization of the coating material. It has been confirmed that by controlling the MPS parameters it is possible to obtain coatings with the desired porosity characteristics and satisfactory adhesion to the substrate. Specific MPS parameters are recommended, which are optimal for obtaining a certain type of coatings.
2) A new method of robotic MPS of zirconium coating on a titanium base of a medical implant has been developed, which makes it possible to obtain a coating with a uniform thickness of 400 μm and with specified characteristics of 20% porosity and pore sizes up to 300 μm by moving a microplasmatron by a robotic manipulator along a given 3D model of an implant with constant predetermined speed and exact observance of the MPS parameters selected on the basis of factor planning.
3) A set of new results of testing the robotic MPS system has been obtained, showing the advantages in the accuracy of the execution of technological processes in comparison with existing solutions (semi-automatic, manual MPS). The robotic arm moves the microplasmatron along the 3D model of the workpiece being treated with plasma, accurately maintaining the spraying distance, movement speed and perpendicularity of the plasma jet falling on the workpiece surface, providing a given range of porosity and uniformity of the thickness of the sprayed coating.

The main results of the dissertation work were presented and discussed at 5 international conferences:

1) 6th International Thermal Spraying and Hardfacing Conference (ITSHC), September 22-23, 2022, Wroclaw, Poland;
2) 11th IEEE International Conference on Intelligent Data Acquisition and Advanced Computing Systems: Technology and Applications (IDAACS), September 22-24, 2021, Krakow, Poland (online);
3) The International Conference "Advanced materials manufacturing and research: new technologies and techniques" (AMM&R-2021 online), February 19, 2021, Ust-Kamenogorsk, Kazakhstan;
4) 7th International Congress on Energy Fluxes and Radiation Effects (EFRE), September 14-26. 2020, Tomsk, Russia;
5) VI International scientific and technical conference of students, undergraduates and young scientists "Creativity of youth for innovative development of Kazakhstan", April 9-10, 2020, EKTU, Ust-Kamenogorsk, Kazakhstan.

Publications. In total, there are 17 publications on the topic of the dissertation work, of which: 5 articles in journals recommended by the Committee of the Republic of Kazakhstan; 5 articles in international peer-reviewed journals indexed in the Scopus database and having a CiteScore percentile of at least 25% and (or) indexed in the data of the information company Web of Science Core Collection, Clarivate Analytics and (or) having a non-zero impact factor, 6 papers in the proceedings of international conferences (of which 3 papers are indexed in Scopus) and 1 patent for a utility model of the Republic of Kazakhstan.

The dissertation is of practical importance: a production test of an industrial product (crushing plate), restored and strengthened by robotic microplasma spraying (according to the parameters recommended as a result of a numerical experiment) of the powder coating of the composite alloy AN-35 (GOST 21448-75), was carried out, confirming the increase in service life plates of a jaw crusher with a microplasma coating of the worn surface (Production test certificate "IP Abakumov S.A.", No. 1 dated 01.10.2020).

Patent No. 5576 for a utility model "Method of robotic microplasma spraying of zirconium coatings" (according to application No. 2020/0547.2. Priority date 11/20/2020) is **proposed for implementation in practice**.

The structure and scope of the dissertation: the dissertation consists of an introduction, 4 main sections with conclusions for each section, a conclusion, a list of references from 143 titles, and 2 appendices. The main content of the dissertation is presented on 115 pages of computer text, includes 42 figures, 19 tables.