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Properties of Nanoscale Carbon Coatings Obtained by the Pulsed Vacuum-Arc Method on Silicon

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The complex of properties including the structure, adhesive strength, internal stresses, tribological properties, microhardness and crack-resistance of nanoscale carbon coatings obtained by the pulsed vacuum-arc method on single-crystal silicon substrates was investigated. Two types of samples of the carbon coating: type (i) formed at the normal location of the substrate relative to the geometric axis of the plasma flow ($\theta = 0°$); type (ii) obtained at an angle $\theta = 70°$ were studied. The analysis of the experimental results showed, that the angle of plasma flow incidence relative to the substrate drastically affects the properties of carbon coatings. The structure, adhesion, internal stresses, wear resistance, crack resistance are interrelated and determined by the radiation-diffusion sealing during the process of carbon coating deposition from the carbon plasma flow. Nanoscale carbon coatings can significantly improve the strength and tribological properties of different tools, parts and products.

Keywords: Nanoscale carbon coating, Pulsed vacuum-arc method, Properties.

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1. INTRODUCTION

Applications of amorphous carbon coatings are defined by their high microhardness, low friction coefficient, chemical inertness, transparency in the infrared radiation range, bioinertia, unique emission characteristics, etc. Currently, many researchers are interested in carbon coatings with thickness less than 100 nm [1-3], that are promising for applications in various fields of science and technology. Thus, we have developed technology for deposition of nanoscale carbon coatings doped with nitrogen [2] that has been successfully used to improve the operational characteristics of scanning probe microscopes microprobe manufactured by NT-MDT. One of the most promising deposition methods of nanoscale coatings is a pulsed vacuum arc method, which allows obtaining coatings with required thickness by giving a certain number of pulses, dosing of the thermal load on the substrate by changing the pulse repetition rate, and, in addition, does not require the applying of the accelerating potential to the substrate.

2. EXPERIMENTAL RESULTS

The complex of properties including the structure, adhesive strength, internal stress, tribological properties, microhardness and crack-resistance of nanoscale carbon coatings obtained by the pulsed vacuum-arc method on single-crystal silicon substrates KEF-4.5 with the orientation (100) was investigated. Two types of samples of the carbon coating: type (i) formed at the normal location of the substrate relative to the geometric axis of the plasma flow ($\theta = 0°$); type (ii) obtained at an angle $\theta = 70°$ were studied.

The properties of carbon coatings are defined by the content in the amorphous carbon matrix of atomic bonds formed by sp^2 and sp^3- hybridization of electron orbitals. Structural studies of nanoscale carbon coatings by electron energy loss spectroscopy by using a transmission electron microscope Tecnai G2 F20 S-TWIN showed that the content of the atomic bonds with sp^2 - hybridization in coatings (ii) is higher than in coatings (i) [4].

The adhesive / cohesive strength of the carbon coatings with thickness 100 nm on silicon substrate with using tester REVETEST of CSM Instruments company was investigated. Diamond spherical "Rockwell C" indenter was used for making scratches at continuously increasing load for registration of acoustic emission and friction coefficient. The adhesive strength criterion was the critical load, leading to the complete destruction of the coating up to the substrate material. The value of the adhesive strength was determined visually using an optical microscope, as well as the coefficient friction and acoustic emission variation curves. Adhesive strength of the coatings (i) was 7 H, and coatings (ii) – 12 N.

The significant difference in the adhesion strength of coatings (i) and (ii) can be explained by the experimental results of the value of internal stresses remaining after the deposition of the carbon coating on the cold substrate from the low-temperature carbon plasma flow. Value of the internal compressive stresses in the coatings (i) was 10 GPa, and in coatings (ii) – 4 GPa. Hence, the anomalously high values of the internal compressive stresses tending to tear the coating from the substrate cause the lower adhesion strength of coatings (i).

Explanation of the effect of the plasma flow angle relative to the substrate on the formation of the structure, appearance and partial relaxation of the internal stresses [4] based on the concepts of radiation-diffusion sealing is proposed. The decrease of the internal stress approximately 2 times with increasing angle θ from 0°

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to 70° is due to the smaller depth of radiation-induced defects in the case of the plasma flow tilted relative to the surface of substrate on which the coating is formed. This leads to greater likelihood of internal stress relaxation a result of defect migration to sinks (the coating surface), due to the decrease of the ion effect seal. The increase of sp^2- phase content in the carbon matrix is caused by the reduction of the ion sealing effect.

The complex tribological study of the initial silicon substrate and the coating samples by the standard wear test scheme "ball-disc" (ASTM G99-959 and DIN 50324) using an automated friction machine (Tribometer, of CSM Instruments) and an analysis of the coatings friction track and the wear scar on the counter body (sapphire ball diameter of 6 mm) were performed. The wear traces of the coatings on Si samples and the wear scar on the balls were studied after the tests using an inverted optical microscope Olympus GX 51. Measurement of the average cross-sectional area and the depth of the friction track were performed in 4 opposite regions of samples using automated precision contact profilometer Surtronic 25. It was found that nanosized carbon coating has a low friction coefficient (< 0.1), high wears resistance, and is the ideal protection of the silicon product from premature wear in friction. Wear factor (mm^3 / (N × m)) for the coating (i) is significantly lower than for the coating (ii).

The micro-hardness and fracture toughness of the initial silicon and the "coating-substrate" system on a DM-8 microhardness gage, equipped with a Vickers pyramid, at loads of 10, 25 and 50 g were investigated. For the crack-resistance criterion the average length of cracks formed during the microindentation and determined for each load from a series of prints was adopted At a 10 g load the micro-hardness of "coating-substrate" system was 1750 HV, microhardness of the initial silicon – 1010 HV. It was revealed that nanosized carbon coating significantly improves the strength properties of silicon. Crack resistance of the "coating-substrate" system is significantly higher compared with the initial silicon substrate. Minimum values of the average cracks length correspond to the coating type (i). Thus, the more stress state of the coating corresponds to a smaller length of cracks. This fact is consistent with our previous studies of internal stress and crack-resistance of carbon coatings doped by different elements [3].

3. CONCLUSION

Analysis of the study results leads to the following conclusions:

- Angle of the plasma flow incidence to the substrate influences dramatically on the properties of the carbon coatings.
- Structure, adhesive strength, internal stress, wear- and crack-resistance are interconnected and determined by the radiation-diffusion sealing in the process of coating deposition from the carbon plasma flow.
- The nano-sized carbon coatings deposited by pulsed vacuum-arc method allow significantly increase the strength and tribological properties of various tools, parts and products.

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