Abstract – It is analyzed how the surface morphology of nitrogen-containing diamond-like carbon CN_x coatings changes with their thickness. A distinctive feature of the experiment is that the coatings having the same composition but different thicknesses are sputtered in a single vacuum cycle. The obtained results confirm the composition structure of the CN_x coatings. It is found that the disordered formation of domains having different densities is followed by the growth of the average roughness (R_a) and the average step of the profile irregularities (S_m) with increasing thickness of the coatings. DLC, CN_{0.25} and CN_{0.5} coatings are studied using a SMM-2000T atomic force microscope and a method for analysis of electrical characteristics. As a result, mapping of the relief is accompanied by point-by-point plotting of one more map, which provides information about electrical conduction. The measurement results support the earlier supposition that boundaries between unlike domains in the composite structure only have the property of conduction.

1. Introduction

The DLC and CN_x coatings have intensively been studied with the aim to show up properties and promise to practical application [1, 2]. Hard diamond-like (~ 80% sp^3 bonds) coatings (DLCs) possess an excellent set of properties for modification of the surface of tools and friction parts so as to extend their lifetime and expand the fields of their practical application. DLCs have the hardness of about 100 GPa and the stationary friction coefficient of not over 0.1. However, large internal stresses (~10 GPa) weaken their adhesion to the substrate. Doping of DLC by nitrogen reduces the internal stresses and friction coefficient. According to literature data and our previous results, the limiting concentration of nitrogen in the CN_x coatings was established to make up 33% (the CN_{0.5} composition) and physical properties of the CN_{0.3-0.5} coatings can best be explained in terms of a composite that consists of the DLC and CN_{0.5} nanodomains [3–6]. While interpreting the properties of the CN_x coatings as those of a composite, a special point is explanation of their conductivity dependence on nitrogen concentration [5]. The resistance of the DLC and CN_{0.5} coatings (1–3) μm thick amounts to more than 10^6 Ohm, whereas for compositions CN_{0.2-0.4} it is less by several orders of magnitude. Involving the concepts on the composite structure of the coatings, this fact can be accounted for by a high conductivity of the boundaries only between different (DLC and CN_{0.5}) structure domains of the coating. The results gained by such model calculations are in good agreement with experiment [7].

2. Experimental details

A distinctive feature of the experiment is that the coatings having the same composition but different thicknesses are sputtered in a single vacuum cycle. For this purpose, a special sample holder with a rotating shaped sector diaphragm is designed. In this case, the minimal thickness of a deposited coating was 9 times as low as that in the case without the diaphragm. The ion etching of substrates and deposition of an adhesion titanium sublayer was performed for all samples through opened sectors of the diaphragm being fixed, whereas the deposition of the coating, under its rotation. The mass thickness of the coatings for each sample was controlled by the weight increment of the sample when it was not closed by the rotating diaphragm with a subsequent recalculation by the open angle of the diaphragm. The linear thickness was controlled with allowance for the coating density.

DLC, CN_{0.25} and CN_{0.5} coatings are studied using a SMM-2000T atomic force microscope and a method for analysis of electrical characteristics. By this method, the current passing through the cantilever with a thin conductive coating on its edge and through a local point on the sample is measured. As a result, mapping of the relief is accompanied by point-by-point plotting of one more map, which provides information about electrical conduction.

A surface relief of coating was measured by means of AFM microscope. Four scans 40×40 μm of each sample were obtained. Main surface roughness characteristics (R_a, S_m) were calculated using static AFM profile images processing.

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To investigate a comparative conductivity (to measure current between the coating surface and cantilever) a special AFM-based technique was employed which operates with a contact movement of the conducting cantilever at a constant load. To this end, the conducting coatings were applied onto the cantilever and their wear resistance was controlled by the stability of current measurements upon scanning the sample surface. The best of the tested coating was the platinum one with a thickness of 20 nm.

3. Results and discussion

According to the AFM data the size of CNx structure domains in the coatings was 10–100 nm over the whole range of nitrogen concentrations (Fig. 1).

![AFM images of CN0.25 (a) and CN0.5 (b) coatings](image1)

The average surface roughness Ra increased with nitrogen concentration in the series from CN0 (DLC) to CN0.5, as well as with the thickness of all coatings. However, the rate of growing Ra and Sm as a function of the coating thickness for DLC and CN0.5 was about half that for CN0.25 (Fig. 2).

This experimental observation can be judged involving the concept on the formation of the composite coating consisting of different (DLC and CN0.5) domains that possess different growth rates under equal conditions of deposition.

Before interpreting the results of investigation of the electrical conductivity of the coating surfaces, the expected peculiarities of the current-coordinate dependences along the scanning direction should a priori be pointed. These peculiarities are caused by the very technique, namely, by the cantilever being driven in contact with the not smooth surface under a constant load. On such conditions, the pressure on the spot of contact will be the lower, the smaller the angle between the cantilever axis and the surface spot under contact. This can result in deterioration of the contact perfection and, as a consequence, in a decrease in the cantilever-sample current. At an equal electrical conductivity of the spots along the line of scanning, including domain boundaries, this should yield a qualitative match of the relief profile with that of the current passing through the cantilever. It is just the match that has always been observed upon scanning the surface of the DLC and CN0.5 coatings (Figs. 3, a and b).

![Dependences of average roughness Ra on coating thickness for DLC (*), CN0.15 (●), CN0.25 (▲), and CN0.5 (□)](image2)
Modification of Material Properties

Modification of Material Properties

Fig. 3. Profiles of the relief and current through the cantilever along the line of scanning for DLC (a), CN_{0.5} (b), and CN_{0.25} (c).

The same character was peculiar to the current-coordinate dependence at the periphery of the domains in CN_{0.25}. However, in half the cases, an increase in the current at the boundary between the structures domains was registered (Fig. 3, c). Allowing for the tip angle of 60°, the size of the contact spot, which is more than 10 nm at a crystallographic width of the boundary of ~0.5 nm, and the fact that just at this boundary the surface forms the deepest hollow, a conclusion can be made that the observed increase in the current must be traceable to a very high electrical conductivity of the thin boundary portion in contact spot. This conclusion is well supported by the comparison of the electrical maps of the DLC, CN_{0.5}, and CN_{0.25} (Fig. 4).

In the case of DLC, occasional current increases were observed that were ascribed to the effect of the graphite particle inevitably incorporated into the coating under conditions of an inhomogeneous beam of carbon ions. In some quite rare cases (in comparison with CN_{0.25}) the current increase was observed in CN_{0.5} too, which, just as in CN_{0.25}, was localized at the boundaries and can be caused by the presence in the coating of the DLC domains.

4. Conclusion

Experimental observation of surface relief of CN_{x} coatings confirms the concept on the formation of the composite coatings consisting of different (DLC and CN_{0.5}) domains that possess different growth rates under equal conditions of deposition.

Thus, it was established that in the CN_{0.25} coatings, there indeed take place highly conductive spots at the domain boundaries. The number of such spots grows with increasing the probability of emergence of the boundaries between the domains that are different in structure, i.e., at an intermediate nitrogen concentration between DLC and CN_{0.5}.

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References


