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Annealing Effect on the Structure, Phase Composition, and Nanohardness of Titanium/Fullerite Films

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Abstract—The effect of heat treatment on the structure, phase composition, and nanohardness of titanium/fullerite films of different thicknesses produced by thermal evaporation in vacuum has been studied using atomic force microscopy, X-ray diffraction, and nanoindentation. The results demonstrate that annealing at 570 K increases the grain size of the films and leads to the formation of a Ti_xC_{60} phase. The nanohardness of the annealed films varies little with depth owing to the interdiffusion of Ti atoms and C_{60} molecules.

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INTRODUCTION

Titanium–fullerene films are potentially attractive as protective and antifriction coatings for biomedical and electronic applications [1, 2]. There is substantial evidence [3–5] that the addition of fullerenes to titanium and nickel coatings raises their hardness and wear resistance. At the same time, a key role in determining the strength of a material is played by its structure, which depends on the coating procedure, doping level, and further processing (e.g., heat treatment, laser processing, or ion bombardment).

This paper reports the structure, phase composition, and nanohardness of titanium/fullerite films in relation to their thickness, substrate temperature, and annealing conditions.

EXPERIMENTAL

Films were grown by thermal evaporation in a VUP-5M vacuum system. The starting materials used were VT1-0 titanium and 99.9%-pure C_{60} fullerite powder. First, a fullerite film was grown on an oxidized single-crystal silicon substrate, and then a titanium film was produced. A number of samples were prepared by coevaporation at a titanium content in the flow of 40 wt %. The films were annealed in a low thermal mass vacuum furnace at a residual air pressure of 1.3×10^{-3} Pa. The phase composition of the films was determined by X-ray diffraction (XRD) on a DRON-3.0 powder diffractometer with CuK_α radiation. The surface morphology of the films was examined by atomic force microscopy (AFM) on a Solver P47-PRO scanning probe microscope using standard silicon cantilever probes with a tip radius of 10 nm.

The mechanical properties of our samples were studied by nanoindentation on a Shimadzu DUH-202 dynamic hardness tester using a Berkovich (three-sided pyramid) diamond indenter. The indent depth was measured with an accuracy of ±1 nm, and the indenter load was determined with an accuracy of ±196 nN. Indents were placed 50 μm apart, and ten measurements were made for each sample. The measurement uncertainty was within 10%. Each test comprised two cycles. The first cycle was intended to determine the dynamic hardness of the sample. The nanoindentation depths were 100, 200, and 500 nm. The loading rate was maintained at 5 nm/s until the desired indentation depth was reached, and the dwell time was 10 s. In the second cycle, the dwell time was 10 s at a constant load of 1 mN, and then the load was reduced by 90%. In this way, an elastic–plastic loading curve was obtained.

RESULTS AND DISCUSSION

Fullerite films 500 and 2500 nm thick grown on unheated oxidized single-crystal silicon substrates had granular structures with lateral grain sizes of 30 and 120–180 nm, respectively (Fig. 1). The rms surface roughness was within 3 nm for the 500-nm-thick C_{60} film and 20 nm for the 2500-nm-thick C_{60} film. The 2500-nm-thick fullerite film grown at a substrate temperature of 420 K consisted of faceted grains 1–2 μm in size, with an rms surface roughness value as high as 80 nm. Titanium films 60 and 200 nm thick grown on fullerite layers differing in surface roughness were found to replicate the surface topography of the fullerite underlayers (Figs. 1b, 1d, 1f), with a slightly increased roughness.

The XRD pattern of a film with a C_{60} layer thickness of 500 nm (Fig. 2a) shows a weak halo at small angles, due to the fullerite layer. There are also the strongest lines of