
SURFACE AND THIN FILMS

Structure and Conditions for the Formation of Fullerite Crystallites in Sn-C₆₀ Films

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Abstract—The structure of tin–fullerite films with different thicknesses of the Sn coating layer (50, 100, 200, 300, 450, and 700 nm) and the conditions for the formation and growth of fullerite crystallites on the tin surface during sample exposure in air have been investigated. The methods of X-ray diffraction; scanning electron, transmission electron, and atomic force microscopy; and X-ray microanalysis were used to reveal changes in the structure and phase composition of the tin–fullerite films. Fullerite crystallites in the form of plates and bolts grown under internal stress have been found on the surface of tin films with thicknesses of 50, 100, and 200 nm. The incubation period of crystallite formation is established to be 12–22 months, depending on the thickness of the tin layer.

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INTRODUCTION

Crystals shaped as filaments and fibers, found in nature, have attracted much attention due to their unusual appearance. Crystalline needles, fibers, filaments of various silicates, carbonates, sulfides, oxides, and many other chemical compounds have been found at different times and in different places. The spontaneous growth of filamentary crystals (referred to as whiskers) has also been revealed in tin-based artificial devices in the first half of the 20th century.

It is known that whiskers of metals with low melting temperatures (Sn, In, etc.) can grow on thin metal layers without any external effect at room temperature. Fullerite is a crystal with a low (650 K) sublimation temperature; therefore, fullerite whiskers can spontaneously grow at room temperature. In [1–3], flowerlike, leaflike, and filamentary formations were found on the surface of tin–fullerite and chromium–fullerite films kept in desiccators for over two years.

The purpose of this study was to determine the incubation period and establish the growth mechanism of fullerite crystallites in tin–fullerite films with tin layers of different thicknesses.

EXPERIMENTAL

The films were prepared by thermal evaporation in a vacuum. First, a 300-nm-thick fullerite film was deposited on an oxidized Si(100) substrate, and then tin layers of different thicknesses ($d = 50, 100, 200, 300, 450$, and 700 nm) were deposited on the fullerite films. The topography of the sample surface was investigated with a Solver P47 PRO scanning probe microscope in the

semicontact mode. Standard silicon cantilevers with a tip radius of 10 nm were used. The phase composition of the films was analyzed on a DRON-4.13 diffractometer in Cu K_α radiation. The film structure was investigated with an LEO-1455 VP scanning electron microscope at an accelerating voltage of 20 kV and a PEM-100 transmission electron microscope with an accelerating voltage of 75 kV.

RESULTS AND DISCUSSION

Atomic force microscopy (AFM) study of the film surface topography immediately after deposition showed that as-prepared fullerite films have a granular structure (Fig. 1a) with a root-mean-square roughness of 4 nm. Condensation of tin layers with thicknesses of 50 and 100 nm on fullerite films leads to the formation of islands, as can be seen well in the AFM images (Fig. 1b) obtained in the phase-contrast mode [4]. An increase of the tin film thickness to 200 nm leads to the merging of islands and the growth of a continuous film. A further increase in the condensed Sn layer thickness facilitates the formation of the crystal structure of the film, and the crystallite size increases from 600 nm at $d_{\text{Sn}} = 200$ nm to 900 nm at $d_{\text{Sn}} = 700$ nm (Figs. 1c, 1d).

Analyzing the AFM images of tin–fullerite films with Sn layers of different thickness, one can select the following characteristic growth stages:

- (i) the formation of individual nuclei and their growth with the formation of an island film structure;
- (ii) the formation of a bound tin network with the existence of many unfilled sites on the substrate in the form of channels and voids;