

Changes in the Structures and Phases of Tin–Fullerite Films during Annealing

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Abstract—The patterns of change in the structure and phase composition of tin–fullerite films subjected to thermal annealing in vacuum at different temperatures (430, 450, 470, and 490 K) are studied using scanning electron microscopy, atomic-force microscopy, X-ray diffraction, and X-ray spectrum microanalysis. It is established that annealing at $T = 450$ K or higher leads to significant changes in the structures and phases of films; namely, new phases Sn_xC_{60} are formed in the form of threadlike crystals with lengths of $6 \mu\text{m}$ containing 98 at % C and 2 at % Sn and ball-shaped isolations with diameters up to $12 \mu\text{m}$ consisting of 50 at % C and 50 at % Sn.

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INTRODUCTION

From the first days of the discovery of fullerenes, scientists' efforts were directed toward the synthesis and study of new phases based on them. Studies showed that new phases can be formed by substituting different atoms for the carbon atom in the molecule skeleton, by penetrating inside the fullerene cell (endohedral fullerenes), or by adding atoms or other molecules to the fullerene skeleton from outside (exohedral fullerenes). Alkali metal–fullerene complexes exhibiting the properties of high-temperature superconductivity have been studied most thoroughly. Much less is known about the interaction of fullerenes with other metals. The authors of [1–5] showed that, in the case of coprecipitation of metal atoms ($\text{Me} = \text{Ag}, \text{Mg}, \text{Cu}, \text{Pd}, \text{Nb}, \text{and Ti}$) and C_{60} molecules, the chemical bond $\text{Me}-\text{C}_{60}$ can form as a result of their interaction. The authors of [6–8] revealed that, in tin–fullerite films obtained during successive precipitations of the fullerite and metal layers, fullerite crystallites form during aging as a result of relaxation of internal mechanical stress. This paper is a continuation of the studies of structure and phase transformations in the tin–fullerite system.

The aim of this paper is to reveal the pattern of change in the structures and phases of tin–fullerite films as a result of thermal annealing at different temperatures.

EXPERIMENTAL PROCEDURE

Tin–fullerite films were obtained by the method of thermal evaporation in a vacuum. First, a fullerite film with a thickness of 300 nm was condensed on a substrate

made of oxidized monocrystalline silicon [9]; then, a tin film with a thickness of 500 nm was also condensed. These films were annealed in a low-inertia vacuum furnace at a pressure of residual gases of $1.3 \times 10^{-3} \text{ Pa}$. The annealing temperatures were 430, 450, 470, and 490 K, and the duration of annealing was 30 min. Changes in the structure were controlled by scanning electron microscopy and atomic-force microscopy. The changes in the phase and elemental composition were studied using a DRON-4.13 X-ray diffractometer for $\text{CuK}\alpha$ radiation and a Roenteg energy-dispersive SiLi microanalyzer.

RESULTS AND DISCUSSION

Our studies showed that newly prepared fullerite films had a homogeneous granular structure with an average granule size of 100 nm . When tin is condensed on the underlying fullerite layer, a labyrinth-like crystal structure of the film forms; in this case, the average size of crystallite is 500 nm (Fig. 1a). X-ray diffraction patterns of newly prepared tin–fullerite films contain intense maxima corresponding to reflections from the planes of tetragonal tin ($a = 0.5831$ and $c = 0.3182 \text{ nm}$) and fullerite of the hexagonal modification with the lattice parameters $a = 1.002$ and $c = 1.638 \text{ nm}$ (Fig. 2).

The samples were annealed at different temperatures to study the solid-phase interaction between tin and C_{60} fullerene. The choice of the maximum annealing temperature was limited by a low Sn melting temperature of 502 K . Annealing of films at $T = 430 \text{ K}$ led to a insignificant increase (by 20 nm) in the average crystallite size as a result of recrystallization. As the annealing temperature was increased up to 450 K , iso-