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FULLERENES AND ATOMIC CLUSTERS

Structural Phase Transformations in Tin–Fullerite Films

L. V. Baran[a], G. P. Okatova[b], and V. A. Ukhov[c]

[a] *Belarussian State University, Minsk, 220050 Belarus*
e-mail: brlv@mail.ru

[b] *Institute of Powder Metallurgy, Belarus State Scientific Production Concern of Powder Metallurgy, National Academy of Sciences of Belarus, Minsk, 220071 Belarus*

[c] *Belmikrosistemy Scientific Research Design-and-Technology Republic Unitary Enterprise, Minsk, 220064 Belarus*

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Abstract—X-ray diffraction, scanning electron microscopy, electron probe x-ray spectrum microanalysis, and Auger-electron spectroscopy were used to study the variations in the structure and elemental and phase compositions of tin–fullerite films stored in air. A new phase Sn_xC_{60}, tin whiskers, and fullerite "flowers and petals" were found to form under internal compressive stresses.

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1. INTRODUCTION

The discovery of fullerenes in 1985 has initiated wide-scale investigations of this new allotropic form of carbon. The discovery of the superconductivity of alkali-metal fullerides [1–3], the anhysteretic ferromagnetism of the BrC_{60} and IC_{60} compounds, and the nonlinearity of their optical properties [4] has stimulated research and study of the properties of the fullerides of other metals.

This paper presents the results of studying the structure and phase transformations in tin-fullerite films under storage in air.

2. EXPERIMENTAL

The films under study were prepared by sequential deposition from the vapor phase using a VUP-5M device. Oxidized single-crystal silicon plates were used as substrates. First, a 150-nm-thick fullerite film was condensed on a substrate and then a 130-nm-thick tin film.

X-ray phase study was performed using a DRON–3.0 diffractometer (CuK_α radiation). The film structure was studied using a LEO–1455 VP scanning electron microscope at accelerating voltages of 10 and 20 kV. The elemental composition of the films in depth was determined by the Auger-analysis method using a PHI–660 scanning electron spectrometer (Perkin Elmer, United States). The films were sputtered by Ar^+ ions (E = 3.5 keV) at a rate of 15 nm/min. The Auger peaks were recorded at an accelerating voltage U = 3 kV and electron irradiation dozes not exceeding 10^{16} cm^{-2}. An analysis of the elemental composition of new formations was performed by the electron probe x-ray spectrum microanalysis method using a Röntec energy-dispersion microanalyzer. The concentration of light elements (C, O) was measured from the intensity of the K-series lines with an accuracy better than 5 at %, and the tin concentration was measured from the L-series lines with an accuracy better than 1 at %.

3. RESULTS AND DISCUSSION

The as-prepared fullerite films have a fine-grained structure with an average grain size of 30 to 50 nm (Fig. 1a). When condensed on a C_{60} layer, tin forms prolonged grains 0.7 to 1.2 μm long, which accrete to form a complex labyrinth structure (Fig. 1b).

In the x-ray diffraction patterns of the as-prepared films, tin is characterized by narrow intense lines (Fig. 2) which are indexed in the tetragonal system (space group $I4/amd$). The tin lattice parameters as calculated from the center of gravity of the (103) and (400) reflections are a = 0.5778 nm and c = 0.3194 nm, which are 0.9% larger and 0.4% smaller than the parameters a and c of massive β tin, respectively. In a tin film condensed on a fullerite layer, internal stresses can occur due to a lattice parameter misfit between the contacting materials (fullerite and tin), a difference between the coefficients of thermal expansion ($\alpha_{C_{60}} = 40 \times 10^{-6}$ K^{-1}, $\alpha_{Sn} = 30 \times 10^{-6}$ K^{-1}), and structural defects.

In a low-angle region in the x-ray pattern, a halo formed by the reflections from fullerite lattice planes is observed. Some of the reflections are indexed in the hexagonal system, though the granules of the powder used for sublimation had an fcc lattice. The transformation of the fcc lattice to an hcp lattice is caused by a high deposition rate of C_{60} molecules [5]. The fullerite layer is subjected to compressive stresses, as demon-