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The structure and nanoindentation of C_{60} films

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The epitaxial (111) oriented fullerite films have been prepared by condensation of C60 molecules onto mica subsrtates in a quasi-closed volume. The hardness H = 0.55±0.17 GPa and Young's modulus E = 15±1 GPa of the fullerite films along the (111) direction of the film fcc lattice have been determined.

Конденсацией молекул C_{60} на подложках из слюды в квазизамкнутом объеме получены эпитаксиальные (111)-ориентированные пленки фуллерита. Определены твердость H = 0.55±0.17 GPa и модуль Юнга E = 15±1 GPa пленки фуллерита вдоль направления (111) ГЦК решетки.

To study the fullerite mechanical characteristics was always a rather complicated problem because of certain difficulties in preparation of bulk samples for micro-mechanical tests. The first complete experimental measurements of the fullerite elastic constants had been carried out using the sound speed measurements in single crystals of various orientations [1]. However, such measurements became possible only after obtaining the C_{60} single crystals of sufficiently large size and high quality. With development of the hardness testing technique using the indentation depth [2], it becomes possible to determine the hardness of films. In such tests, the displacement of a diamond indentor is registered, while the indentor load is increased and decreased. As the indentor unloading process is elastic, it is possible to estimate the elastic modulus of the sample. The growing of oriented fullerite films of a high perfection degree in combination with nanoindentation technique makes it possible to measure the elastic characteristics of fullerene in a film state.

The single crystalline epitaxial fullerite films were obtained using the vacuum condensation of C_{60} molecules in a quasi-closed volume onto mica substrate of 50 mm diameter heated to 573 K. The hardness measurements were carried out using a Nano Indenter II (MTS Systems Inc.) instrument using a Berkovich indentor under the load (P) of $20{\cdot}10^{-5}$ N. The hardness and the elastic modulus were calculated using the method described by Oliver and Pharr [2]. Nano-hardness tester "Nano Indenter II" of MTS Systems Inc., USA, was equipped by a diamond Berkovich indentor shaped as a trihedral pyramid. The instrument provides the indentor displacement to be measured to within ±0.04 nm, and the indentor load to within ±75 nN. The instrument is able to measure the hardness in the load range of from $0.1{\cdot}10^{-5}$ to 0.15 N. The minimum indentation depth sufficient for hardness and elastic modulus determinations is about 30 nm.

The structural investigations of the fullerite films of about 20 μm thickness has shown that the objects have a face-centered cubic (fcc) structure with the crystallite size at least 40 μm and the axis of (111) texture normal to the growth surface (Fig. 1). The texturing degree can be judged from the obtained width of the (111) line swinging

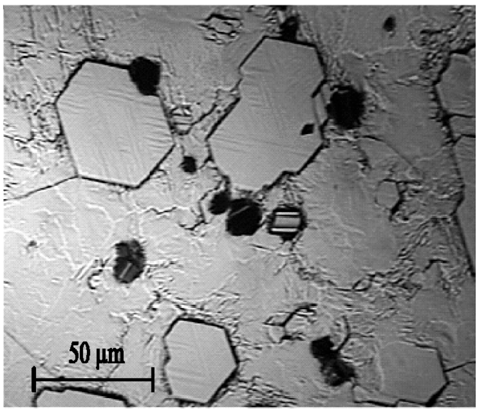

Fig. 1. Surface image of epitaxial fullerene film grown on mica.

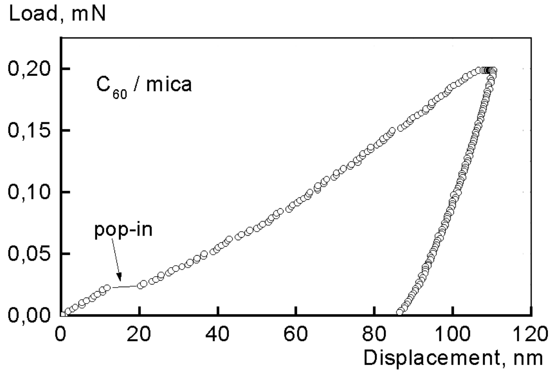

Fig. 2. Loading-indentation diagram for a high-perfection (111) oriented fullerene film.

curves taken from 2×3 cm^2 area varying within the range of 0.210 to 0.370.

The hardness (H) and the Young's modulus (E) determined from the nanoindentation measurements of the fullerite films are 0.55±0.17 GPa and 15±1 GPa, respectively. The results obtained agree well with the theoretical calculations for (111) direction in fcc fullerite lattice (E = 16.3±1.2 GPa), basing on the sound speed measurements in the crystals [2].

An important feature of the loading curves of the fullerite films grown on mica consists in the presence of a step (the so-called "pop-in effect") (Fig. 2) related to the homogeneous nucleation of dislocations in the contact [3]. In the perfect single crystals with the contact depth of about 20 nm, the distance between the dislocations becomes considerably greater than the indentation size, i.e. there are no dislocations in the contact and the plastic deformation cannot start. The homogeneous nucleation of dislocations takes place only under high stress in the contact, which is close to the theoretical shear strength of the material. Because of the strong overstrain, an avalanche-like multiplication of dislocations takes place within a fraction of a second. As a result, the indentation depth increases sharply [4, 5]. Thus, before the step, the strain in the contact is elastic. After the step, the elastic-plastic strain starts in the contact.

The observed pop-in evidences a high structural perfection of C_{60} single crystals. In imperfect crystals, the plastic strain starts easily in the contact, and the step does not appear in the curve (heterogeneous nucleation of dislocations).

Thus, highly perfect (111)-oriented fullerite films have been obtained. For the first time the direct measurements of the hardness and the Young's modulus were carried out on the fullerite films along (111) direction in fcc lattice.

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Структура та наноіндентування плівок C_{60}

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Конденсацією молекул C_{60} на підкладках зі слюди у квазізамкненому об'ємі виготовлено епітаксійні (111)-орієнтовані плівки фулериту. Визначено твердість H = 0.55±0.17 GPa та модуль Юнга E = 15±1 GPa плівки фулериту вздовж напрямку (111) ГЦК решітки.