Diagnostics of the structure of thin films of polymerized C₆₀ formed via electron-beam dispersion method

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Polymerized fullerene C_{60} forms a whole new promising class of carbon materials. Structure of polymerized C_{60} varies from dumb-bell- to peanut-shaped polymers which in turn can form dimers, linear chains, two-dimensional or even three-dimensional networks. The physical-chemical properties of C_{60} polymers vary in a wide range respectively. However, the diagnostics of the exact polymer composition and structure of such materials is often quite a complicated experimental task. Unlike C_{60} monomer, C_{60} polymers hardly can be dissolved. The polymer insolubility provides a quick and convenient technique to check the polymerization. However, on the other hand, it also makes measurement of the polymer composition even more complicated. Therefore, thorough analysis and comparison of the results obtained using different measurement techniques is necessary.

In the presented report, polymer composition and structure of thin C_{60} polymer films, deposited via electron-beam dispersion of pristine fullerite C_{60} in vacuum [1], have been studied using Raman and FTIR spectroscopy, laser desorption/ionization (LDI) and matrix assisted laser desorption/ionization (MALDI) mass spectrometry, X-ray photoelectron spectroscopy.

We have used a precise Lorentzian lineshape analysis to decompose Raman spectra of the deposited coatings into components characteristic of dumb-bell-shaped fullerene dimers, 1D and 2D polymers and thus, to analyse the polymer composition of the coatings. The results have been also proved by FTIR spectra analysis. However, we have also shown that estimations of the polymer composition based on the vibrational spectra clearly overestimate the content of fullerene dimers due to end C_{60} molecules in polymer chains and clusters.

It has been shown that LDI mass spectrometry allows for detection of polymer clusters in the deposited coatings. However, C_{60} polymers readily dissociate to monomers upon desorption/ionization process. Therefore, a soft mass spectrometry technique, MALDI was used to obtain information about the polymer composition of the coatings. Polymer clusters with the size of up to 7 monomer units have been found in the mass spectra. The results of the mass spectrometry measurement are compared to estimations based upon vibrational spectra as well as to XPS data. Peculiarities of each experimental technique in the measurement of thin C_{60} polymer film composition are discussed.

[1] V. Kazachenko, I. Ryazanov, Technical Physics Letters 34, 930 (2008).