C60 (H.C.P.): Synthesis by Cryoextraction From Solutions

- E.V. Skokan, I.V. Arkhangelskii, N.A. Zhukova, Y.A. Velikodnyi, N.B. Tamm, N.V. Chelovskaya, and L.N. Sidorov (Lomonosov Moscow State University)

Earlier, two methods were applied to obtain the hexagonal close packed (H.C.P.) phase of fullerite \(\text{C}_{60}\): cryosynthesis and precipitation of \(\text{C}_{60}\) from benzene solution using \(n\)-hydrocarbons. However, after heating, the products contained both H.C.P. and face-centered cubic (F.C.C.) phases.

The aim of this work is the development of a new method of the synthesis of the material with enhanced content of the H.C.P. phase. A precursor of the H.C.P. phase was prepared by low temperature (-20°C) extraction of benzene solution of \(\text{C}_{60}\) frozen with liquid nitrogen. \(n\)-Hexane, \(n\)-heptane, and acetone were used as extractors. Clathrate-like structures obtained were studied by XRD, TGA, DSC, and IR spectroscopy methods. It was found that only the materials extracted with \(n\)-hexane can serve as the precursors of the H.C.P. phase of fullerite \(\text{C}_{60}\). When the clathrates containing \(n\)-hexane or acetone were annealed in dynamic vacuum, the samples with distorted H.C.P. structure were obtained.

Annealing of the precursor yielded the sample with H.C.P. phase content significantly higher than that in earlier synthesized materials. We suggested that this sample is one of polytypic modifications, the terminal members of which are F.C.C. and H.C.P. structures (phases). Based on the data obtained, we proposed a possible mechanism of formation of the H.C.P. phase of fullerite \(\text{C}_{60}\).

This work was supported by the RFBR (project no 01-03-32994) and, in part, by the INTAS-RFBR (project no IR-97-1015).