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Shock-induced phase transitions of C_{70} fullerite

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Abstract

Shock-induced phase transitions of C_{70} fullerite were studied at the pressure range up to ~52 GPa with use of recovery assemblies of planar geometry. The starting material consists of two crystalline phases: phase with hexagonal close-packed (HCP) and phase with rhombohedral structure. We have found that C_{70} fullerite undergoes a series of polymorphic phase transitions in conditions of step-like shock-wave compression. In the specimens, recovered from 9, 14 and 19 GPa, a dominant phase was fullerite C_{70} with cubic structure. Also, some amount of C_{70} with HCP structure was observed. The quantity of HCP phase was decreasing with increasing of intensity of shock loading. With further growth of shock pressure, destruction of C_{70} molecules occurs. In the samples, recovered after shock loading, the main phase was graphite with a low degree of three-dimensional regularity.

Keywords: Fullerite; Shock waves; Phase transitions

1. Introduction

In comparison with fullerite C_{60} very little is known about the high-pressure properties of fullerite C_{70}. One reason is that its price is significantly higher, and a second reason is that C_{70} is much more complicated material regarding molecular, translational, orientational and rotational properties [1].

At ambient conditions, C_{70} (depending on way of obtaining) is a face centered cubic (FCC), hexagonal close-packed (HCP) or rhombohedral molecular crystal [1].

In works [2–4], areas of thermal stability of various crystalline modifications of fullerite C_{70} were studied at low pressure ($P \leq 1$ bar). It was found that monoclinic C_{70} structure is stable at temperatures below ~280 K, rhombohedral structure—in a range from ~280 up to ~345 K, and FCC structure is stable at temperatures higher than ~345 K.

At static conditions, phase transitions of fullerite C_{70} were studied in a range of pressures up to 12.5 GPa with use of diamond anvil cells and high pressure chambers of different types [5–11].

The equilibrium phase diagram of fullerite C_{70} (considered as individual molecular compound, instead of as the allotrope of carbon), constructed on the basis of experiments [2–10] (see also review [1]), is presented in Fig. 1. It is necessary to note regarding the phase diagram, that there is no full consensus on the high-pressure phase boundaries of C_{70}, nor is the structural evolution of C_{70} with pressure [1].

It is known that a series of specific transformations takes place at static compression of fullerite C_{70} in conditions of increased temperature. The result of these transformations is the drawing together C_{70} molecules and formation of intermolecular covalent bonds (polymerization) [9–11]. In works [10,11], polymerized structures of C_{70} fullerite were synthesized with use of "toroid"-type apparatus. Specimens were subjected to the action of pressures in a range from 4 to 12.5 GPa and to the action of temperatures in a range from 300 to 1770 K in atmosphere of argon. Thermodynamic parameters of the synthesis of polymerized C_{70} structures are presented in Fig. 1, too.

In the present work, shock-induced phase transitions of C_{70} fullerite were studied in a range of pressures up to 52 GPa with use of recovery assemblies of planar geometry.

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2. Starting material

In our experiments we used polycrystalline C_{70} powder with purity not less than 99.5%. The powder was made in G. A. Razuvaev Institute of Organometallic Chemistry (Nizhny Novgorod, Russia). The X-ray powder diffraction study (Cu K_a irradiation) of starting C_{70} fullerite has shown that the material consists of two crystalline phases. The first phase was HCP structure with lattice parameters $a=1.068$ nm, $c=1.734$ nm. The quantity of this phase in a crystalline part of the material was ~47 vol.%. The second crystalline phase was characterized by diffraction peaks corresponding to interplanar distances 0.941; 0.841; 0.548; 0.509; 0.435; 0.419 nm. It is possible to assume that these peaks correspond to a phase with rhombohedral structure and lattice parameters (in hexagonal axes) $a=1.018$ nm and $c=2.822$ nm. The quantity of this phase in a crystalline part of the material was ~53 vol.%. Also, the starting material contained a small amount of X-ray amorphous substance characterized by a halo with a maximum of intensity near angles $2\theta=42-45°$. Probably, this halo belongs to monomolecular component of C_{70}.

3. Experimental procedure

Starting C_{70} powder was pressed between two copper disks (with a thickness of 1 and 2 mm) into a cavity of a stainless steel liner. A density of the specimens was 1.64 ± 0.01 g/cc. A diameter of the specimens was about 15 mm; a thickness was about 1 mm. The liner was placed into a stainless steel recovery ampoule with an external diameter of 57 mm and a thickness of 20 mm. The ampoule was mounted into a steel ring (momentum trap) with an external diameter of 150 mm and a thickness of 20 mm. The assembly was placed on a massive steel basis. The recovery assemblies were loaded by impact of aluminum plates with

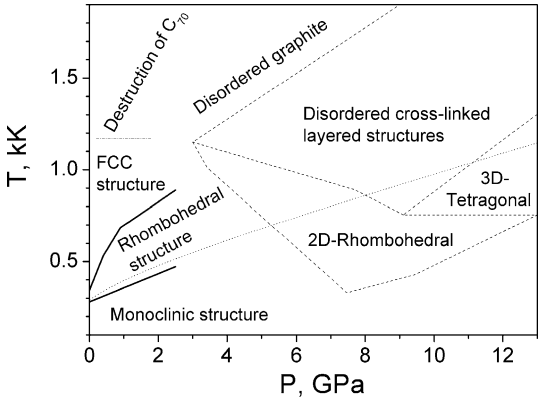

Fig. 1. The phase diagram of fullerite C_{70}, constructed on the basis of works [1–8]. Phase boundaries are shown by solid lines. Superimposed on the phase diagram: areas of thermodynamic parameters of synthesis of polymerized C_{70} structures [10,11] (are bounded by dashed lines); Hugoniot of porous graphite (with a density of 1.64 g/cc) [12] (dotted line).

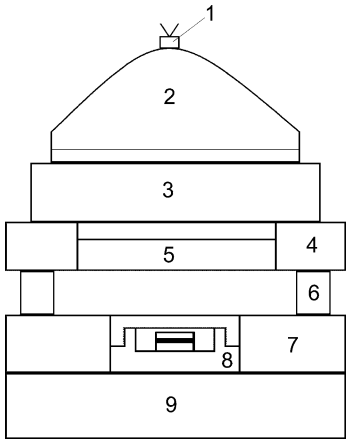

Fig. 2. Scheme of an experimental assembly. Explosive projectile system: 1—detonator; 2—plane shock wave generator; 3—explosives; 4—focusing ring; 5—flyer; 6—supports. Recovery assembly: 7—momentum trap; 8—recovery ampoule; 9—baseplate.

a diameter of 90–100 mm and a thickness of 4–10 mm, accelerated by calibrated explosive projectile systems. Impact velocities were ranged up to 3.35 km/s. Scheme of an experimental assembly is presented in Fig. 2.

In the specimens, maximal shock pressures were reached after several reverberations of the waves between the walls of the recovery ampoule (step-like shock-wave compression) and were ranged from 9 to 52 GPa. A duration of action of pressure pulse on the specimen was 0.6–1.9 μs (depending on type of the explosive projectile system). The characteristic dependence of pressure in an explored material versus time of loading is presented in [13].

Microstructure of the specimens, recovered after shock-wave loading, was examined by means of powder X-ray diffractometry (Cu K_α-irradiation).

4. Experimental results

X-ray scans of the starting C_{70} powder and materials, recovered after shock compression, are presented in Fig. 3.

In the material, recovered from ~9 GPa, a dominant phase was fullerite C_{70} with face-centered cubic structure (FCC) ($a=1.499\pm0.001$ nm) and an apparent grain size of ~8 nm. The second crystalline phase was C_{70} fullerite with HCP structure. The quantity of this phase in a crystalline part of the material was ~27 vol.%. Also, X-ray amorphous substance (characterized by halo in a field of angles $2\theta=35-60°$) was observed.

In the material, recovered from ~14 GPa, a dominant phase was fullerite C_{70} with FCC structure ($a=1.500\pm0.002$ nm). Some amount of C_{70} fullerite with HCP structure (~16 vol.%) was observed, too. Also, the recovered material contained X-ray amorphous substance characterized by halo in a field of angles $2\theta=35-59°$. Integral intensity of this halo surpassed intensity of the similar halo in a spectrum of the starting powder approximately in 4.5 times.