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Abstract

Full Text

PHYSICS

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OPTICAL METHOD FOR DETERMINING THE MELTING TEMPERATURE OF GRAPHITE AS A FUNCTION OF PRESSURE UP TO 40,000 atm

The method for determining the dependence of the melting temperature of graphite on pressure up to 40,000 atm is a further development of the method for determining this same dependence up to 3000 atm. As in the preceding work ([1]), the apparatus consists of a high-pressure chamber, inside which melting takes place, and of a recording photoelectric device. However, the high-pressure chamber for these measurements differs substantially from the chamber used for measurements up to 3000 atm. We used Drickamer' s idea ([2]) of employing sodium chloride as a pressure-transmitting and optically transparent medium.

The high-pressure chamber shown in Fig. 1 is a cylinder 1, whose outside diameter is 44 mm and height 30 mm. The diameter of the internal opening is 5 mm and its height is 5 mm. The upper and lower parts of the internal opening are divided by a 90-degree cone.

The chamber has two lateral openings 2, located along the diameter of the section in such a way that the central part of the chamber lies in the light path between them.

Fig. 1. Diagram of the high-pressure chamber for studying the melting curve of graphite as a function of pressure up to 40,000 atm.

The openings widen outward by means of four shoulders. The dimensions of the shoulders along the diameter are somewhat increased in comparison with the dimensions in Drickamer' s apparatus. These openings are filled with sodium chloride and serve as windows of the high-pressure chamber. From the outside

Fig. 2

Figure 2: Fig. 2

these windows are supported by glass 3 and by an obturator 4, which we usually use ([3]) for optical investigations at high pressures. The glass and obturator are placed in the outer support of the high-pressure chamber 5. The chamber is made of 45KhNMFA steel, hardened to R_c = 58—60. The chamber support is made of 45KhNMFA steel, hardened to R_c = 45—48. The pressure in the chamber is produced by pistons 6. The piston diameter is 5 mm, the height 10 mm. The piston material is U-12 steel, hardened to R_c = 65. The pistons are supported by steel holders 7 made of 45KhNMFA steel, hardened to R_c = 45—48, by inserts 8 made of 45KhNMFA steel, hardened to R_c = 45—48, and by cushions 9 made of KhVG steel, hardened to R_c = 48—50. The lower piston is stationary and is insulated from the body by mica glued to the metal surface with bakelite varnish. The insulation of the piston is shown in Fig. 2. Insulation of one of the pistons in our apparatus is necessary, since the pistons also serve as current leads for the current required to heat the specimen under investigation. The upper piston, which is movable, is not insulated from the body and serves to create high pressure in the chamber.

The entire inner part of the chamber and the stepped windows in the chamber walls are filled with sodium chloride. We tried filling the windows with molten salt, with salt powder ground in a mortar, and also with windows etched from salt crystals followed by compression under high pressure both from the outside and from the inside. The following method proved to be the best. The chamber is filled with cylinders etched in parts, corresponding in size to the shoulders of the openings, followed by external compression at a pressure of 10,000 atm for 10-15 min. Then the inner part of the chamber is likewise filled with an etched cylinder and compressed between the pistons at a pressure of 15,000 atm for 1 hour. After this operation the inner part of the chamber becomes transparent when viewed through the windows.

Fig. 2. Enlarged image of the piston-electrode lead, insulated from the body. 1—insulation with mica plates glued with bakelite varnish; 2—insulation with a polyvinyl chloride tube.

The graphite specimen 10 under investigation, in the form of a rod 1 mm in diameter, is placed in a salt tablet located between two pistons. For this purpose, in the salt tablet, previously pressed in the chamber to transparency, a through hole 1 mm in diameter is drilled along the axis, into which the etched graphite rod is inserted. Both these operations are carried out while the salt tablet is inside the chamber. On the outside, near the end of the rod, a small amount of powdered graphite is poured in to provide better electrical contact between the rod and the end of the piston. Steel pads 11 are placed between the pistons and the tablet; their diameter is equal to the diameter of the pistons and their height is 1.5 mm. The pad adjacent to the lower piston along the generatrix

and, correspondingly, the piston, are insulated from the lateral surface of the opening by a very thin layer of mica glued with bakelite varnish. For strength, this pad is pasted over the mica with one layer of cigarette paper. The paper is likewise glued with bakelite varnish. Heating of the graphite specimen up to melting is carried out by alternating electric current supplied from the mains through an autotransformer for several seconds. During the experiment the current is increased to 40 A and above at a voltage of 20-30 V. Molten graphite under pressure diffuses very strongly into the salt, and soon after melting the intensity of the glow begins to fall sharply because of absorption in the salt layer surrounding the specimen and containing inclusions of graphite particles. At the end of the experiment, if it was not possible to stop the supply of electrical energy in time, a certain amount of salt was often expelled from under the piston, which was accompanied by an impact with a metallic sound.

Examination of the tablet with the specimen after the experiment showed that the diameter of the graphite rod had apparently increased (in fracture), gradually becoming lighter toward the edges, as a result of diffusion of graphite.

The apparatus recording the temperature and melting is essentially no different from the apparatus used by us in measurements of the melting temperature of graphite up to 3000 atm ([1]), except that in the present work there are no absorbing filters of grade NS, since the intensity of the radiation emerging outward is considerably reduced because of the strong absorption of light by sodium chloride, and also because the minimum diameter of the aperture in the windows in this case is five times smaller. Since in our temperature calculations we use the ratio of the intensities of two spectral lines, the overall decrease in intensity is of no essential importance to us.

Calibration of the apparatus for pressure was carried out using polymorphic transitions under pressure in bismuth and thallium by the method of measuring electrical resistance ([4]).

We measured the melting temperatures of graphite at various pressures up to 40,000 atm. The results of the measurements are shown in Fig. 3. As can be seen from the figure, the melting temperature increases with pressure, reaching 6500°K at 40,000 atm.

The numerical material and the calculation of the temperatures will be presented by us in a subsequent communication.

Fig. 3. Dependence of the melting temperature of graphite on pressure up to 40,000 atm. (The points on the curve up to 3000 atm were taken from the smoothed curve of the $T-P$ dependence for graphite, measured in a gas medium (1).)

It should be noted that the melting temperatures measured by us somewhat exceed the melting temperatures obtained by Bundy ([5]) by measuring the electric current heating the specimen.

T. V. Shcheglakov took part in the work.

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Note: Figure translations are in progress. See original paper for figures.