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## Abstract

## Full Text

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# AN OPTICAL METHOD FOR DETERMINING THE MELTING TEMPERATURE OF GRAPHITE AS A FUNCTION OF PRESSURE UP TO 3000 atm

The change in the state of graphite as a function of temperature and pressure was considered more than 30 years ago. As early as 1933, Basse, studying the possibility of obtaining artificial diamonds from graphite under the action of high pressures and temperatures, investigated the dependence of the melting temperature of graphite on pressure <sup>(1,2)</sup>. In one of these works, in a study carried out up to 11,500 atm, a phase diagram was constructed and the triple point of the solid, liquid, and gaseous states of graphite was determined. Basse

Fig. 1. Schematic of the apparatus for studying the melting curve of graphite under pressure

measured the melting temperature of the sample with an optical pyrometer with a disappearing filament, and melting was recorded in each experiment after releasing the pressure and disassembling the apparatus, by the traces of melting on the remaining parts of the graphite rod.

In 1959, Noda <sup>(3)</sup>, studying the crystallographic properties of molten graphite, methodically reproduced Basse's measurements up to 160 atm and found similar values of the pressure and temperature of the triple point.

Taking into account that the determination of the moment of melting by previous authors was qualitative in character, and that measurements of the temperatures corresponding to melting with an optical pyrometer—especially with visual observation—cannot be regarded as reliable, we carried out measurements of the melting temperature of graphite as a function of pressure at higher pressures by an optical method with automatic recording of the temperature at the moment of melting.

Fig. 2. Oscillogram of the change in intensity of two spectral lines with time at constant pressure

Figure 2: Fig. 2. Oscillogram of the change in intensity of two spectral lines with time at constant pressure

Our apparatus (see Fig. 1) for studying this dependence up to 3000 atm consists of a high-pressure vessel 1, inside which melting occurs

...melting of graphite, and an automatically recording photoelectric system. The high-pressure vessel is equipped with two electrodes 2, 3 and two quartz viewing windows 4, 5. A description of the operating principle of the high-pressure vessel, the windows, and the electrode leads was given by us in work with an electric arc under pressure [4]. In the present work, however, sealing of the electrodes was carried out according to the principle of an uncompensated area by means of contact between two lapped planes (as was also the sealing of the windows in our work [4]). Insulation is provided by mica plates 6, 7, inserted between two lapped planes.

Fig. 2. Oscillogram of the change in intensity of two spectral lines with time at constant pressure

...planes. Pressure is supplied to the bomb from a gas compressor. Argon is the pressure-transmitting medium. The dimensions of the high-pressure vessel are identical to the dimensions of the vessel used in the work with the electric arc.

The graphite sample under study 8, in the form of a rod 10 mm long and 1.5 mm in diameter with a neck in the middle 0.8 mm in diameter, was placed in the middle part of the inner opening of the bomb between two holders connected to the inner ends of the electrodes.

Heating of the graphite to melting and above was carried out by an alternating electric current supplied from the mains through an adjustable autotransformer. Heating took place over several seconds with current of increasing power until the molten graphite at the melting site flowed apart, breaking the electrical circuit. During the experiment the current rose to 40 A and somewhat higher at a voltage of 20-30 V.

The melting of the sample, as well as the temperature at the moment of melting, were recorded by a loop oscillograph in the form of an automatic recording (Fig. 2) of the dependence of the change in intensity of two spectral lines on time. From the ratio of the intensities of this pair of spectral lines at the moment of melting, the melting temperature was determined. Melting was fixed by the cessation of the increase in intensity of these spectral lines, which corresponds to the cessation of the temperature increase in graphite under increasing electric-current power.

The light from the incandescent graphite rod was focused by lens 9, located inside the high-pressure bomb, and through window 5 in obturator 10 fell on

Fig. 3. DC amplifier circuit

Figure 3: Fig. 3. DC amplifier circuit

slit 11 of an FEU-22 photomultiplier, passing on its way alternately through two interference light filters, which cut out spectral bands of the order of 2 mμ from the continuous emission spectrum of graphite. The symmetrically positioned window 4 served for visual observations. Passage of the light alternately through two light filters was achieved by placing these filters on a rotating disk 12. In the path of the light beam, absorbing gray light filters 13 of the NS brand were placed to reduce the intensity of the light falling on the photomultiplier slit. The use of these filters was necessitated by the increase in the intensity of graphite radiation during melting with increasing pressure. The signal from the output of the photomultiplier...

the multiplier was amplified and fed to an MPO-2 loop oscillograph. Amplification is carried out according to the circuit shown in Fig. 3.

To eliminate the effect of scattering due to fluctuations caused by convection currents in the gaseous medium, we used a quartz light guide 14, one end of which adjoined the central part of the collecting quartz lens 9, while the other end was directed toward the central part of the graphite rod so that the neck of the graphite rod and the axis of the cylinder lay on the common optical axis of the system. The distance between the graphite rod and the end of the light guide was 2 mm. The light guide was a fused-quartz rod 7 mm in diameter and 29 mm long, with strictly parallel and well-polished end faces.

### Fig. 3. DC amplifier circuit

The lens placed inside the high-pressure bomb, with  $f = 33$  mm, magnified the image of the rod neck in the plane of the photomultiplier slit by a factor of 20. This made it possible for us to obtain the luminous flux in each experiment from the middle part of the neck of the graphite rod.

Adjustment of the setup for each experiment was carried out by projecting the shadow image of the rod onto the plane of the photomultiplier slit while passing a beam of light from outside through the bomb via the window for visual observation.

When, in the absence of the light guide, the image of a rod heated to incandescence was projected onto the plane of the photomultiplier slit at atmospheric pressure, at the first moment we observed its bright image. Then, against the background of the luminous image, all clearly visible shadow images of convection currents in the gas appeared. With increasing pressure the intensity of the convection currents gradually increased so much that, at a pressure of 500 atm, no transmission of light through the medium scattering in this manner was visually observed. A similar decrease of the luminous flux in a gaseous medium was observed by Ya. A. Kalashnikov and L. F. Vereshchagin (5).

The use of the light guide made it possible for us to carry out measurements up to 3000 atm without a noticeable absorption effect. However, we considered it risky to perform optical measurements on this setup at higher pressures.

By measurements using this method we found that the melting temperature of graphite, with an increase in pressure up to 3000 atm, rises very slowly, from 4650 to 4750° K.

A more detailed description of the measurement results will be given in subsequent communications.

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

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