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Abstract

Full Text

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A High-Pressure and High-Temperature Apparatus with Several Pairs of Electrical Leads

Recently, descriptions of the operation of high-pressure and high-temperature apparatuses with conical punches have appeared in the domestic and foreign literature (1-3). The considerable working volume of the apparatuses (up to 1 cm^3 and more), which makes it possible to introduce a graphite or metal furnace into the high-pressure chamber, enables numerous problems to be solved in studying the effect of high pressure (up to 100000 kg/cm^2) and high temperature on the physical and physicochemical properties of solids. One of the main shortcomings of apparatuses of the indicated design is the difficulty of introducing electrical leads into the region of high pressure and high temperature. The authors have succeeded, to a considerable extent, in avoiding this shortcoming by using a fundamentally new method of introducing electrical leads.

Fig. 1. Section of a high-pressure and high-temperature apparatus with several pairs of electrical leads

Description of the apparatus and principle of its operation. Figure 1 shows a section of the apparatus. Specimen 4 is placed in the high-pressure chamber 5 and is compressed by two punches 9, having the shape of truncated cones. Between the conical surfaces of the chamber and the punches, pyrophyllite seals 2 are inserted. Specimen 4 consists of the substance under investigation, placed in a cylindrical cup made of metal or graphite, which serves as the heating furnace. The substance is heated by passing an electric current through the furnace. The current is supplied to the punches, which are electrically insulated from one another and from the chamber. Contact between the furnace and the punches is effected by means of two steel disks 3. The punches, made of hard alloy VK-6, VK-8, are pressed into sets of steel supporting rings 10 and 11. The high-pressure chamber 5, made of ShKh-15 steel heat-treated to a hardness of

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$R_c = 64$, is inserted into a set of supporting rings 6, 7, 8. The inner surface of the chamber consists of a combination of the surfaces of two truncated cones joined by their small bases, 9 mm in diameter. The chamber is split and consists of four identical parts 2, Fig. 2. These parts are insulated from one another and from the supporting rings by mica 1 and 3, Fig. 2. All sharp edges of the parts are beveled to eliminate the possibility of electrical contact between them during operation of the apparatus. When force is applied

pyrophyllite flows into the gaps l, Fig. 2, over a short distance. The flow ceases because of the large internal friction of the pyrophyllite and the smallness of the gap (< 0.1 mm), after which compression of the working substance begins.

The apparatus was calibrated for pressure up to 50,000 kg/cm^2 on the Bridgman scale (see Fig. 3). The calibration was carried out by the known jumps in electrical conductivity of the following metals: Bi I–II 25,600 kg/cm^2; Bi II–III 27,000 kg/cm^2; Tl 45,000 kg/cm^2. During calibration, the absence of electrical contact between the parts of the chamber was systematically checked.

Fig. 2. Top view of the high-pressure chamber with steel supporting rings

Fig. 3. Pressure calibration curve of the apparatus

Introduction of electrical leads into the high-pressure and high-temperature chamber

Each electrode leading from the high-pressure and high-temperature zone is brought into contact with the inner surface of one of the parts of the chamber. The ends of the wires going from the measuring or generating instruments to the apparatus are placed between the parts of the chamber and the Teflon ring 1, Fig. 1. When force is applied to the punches, reliable ohmic contacts are obtained. Naturally, the number of electrodes is determined by the number of parts of the chamber. The apparatus described has three pairs of electrodes (including a pair of insulated punches). If desired, the number of electrodes can be increased by increasing the number of parts of the high-pressure chamber 5. It should be noted that making the high-pressure chamber of a separable construction strengthens it, since under these conditions the material works in compression. In addition, the heat treatment of the chamber material is improved, especially for one having large dimensions.

The use of hard alloy as the chamber material will make it possible to substantially raise the pressure range.

It will apparently be expedient, between the chamber and the supporting rings, to use a separable ring that has a cylindrical inner surface and a conical outer surface.

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