Superhigh pressures produced by using carbonado anvils (polycrystalline diamonds)

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A new design for a high-pressure chamber is proposed and possible operating modes of high-pressure chambers made from carbonado are analyzed, taking into account the obtained experimental results and the well-known data in the literature.

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Lately, a number of papers on generation of pressures in the megabar range have appeared.[1-3] Kawai and Endo[1] used a gang assembly to obtain pressures of the order of 1 Mbar. Vereshchagin *et al.*[2] used the pressure generated between the conical anvil with a rounded top on the cone and the plane anvil of polycrystalline synthetic carbonado diamond. In these experiments the change in the electrical conductivity of the sample was recorded by the change in the electrical conductivity of the circuit consisting of a cutting edge, a plane anvil, and a sample placed between them. The pressure evaluated in terms of the amount of the applied force and the area of the stamping lies, in our opinion, in the megabar range. Moo and Bell[3] used anvils of optically transparent diamonds.

In this work we used the following chamber design for obtaining high pressures and for studying the properties of high pressure chambers made from carbonado.

In contrast to the high-pressure chambers having axial symmetry, which are usually employed, the chamber we developed consists of two anvils, one of which (Fig. 1) has the shape of a truncated pyramid with a circular edge and a working-area dimensions $a = 0.01$ mm and $b = 0.1$ mm. The vertex angle of the pyramid is $\alpha = 169°$. The

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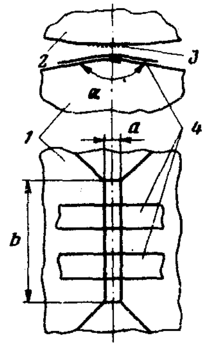

FIG. 1. High-pressure chamber.

second anvil is plane. The sample 3 in the form of a thin film is sputtered (or deposited) on the plane anvil surface. We used as measuring electrodes 10- to 15-μm wide lead plates 4, which were spray-coated in a vacuum at right angles to the edge of the pyramid 1.

This design allowed us to measure the electrical resistance of the sample along the edge of the anvil 1, eliminating the effect of possible short circuits between the anvils 1 and 2 during compression. The lead electrodes are used simultaneously to evaluate the pressure from displacement of the temperature T_c of a superconducting transition in some of the electrodes which are located in the high-pressure zone. Since T_c of Pb decreases monotonically with increasing pressure, the temperature at the end of the superconducting transition corresponds to the maximum pressure in the chamber (on the axis of the edge). Figure 2 shows typical curves for the superconducting transition in a lead manometer produced as a result of applying different forces.

Our studies showed that the compression conditions in the carbonado chambers have a number of peculiarities connected primarily with the fact the carbonado material is highly inhomogeneous. It is a fine-grained, polycrystalline accretion of diamonds with inclusions of graphite, metal catalysts, and compounds based on them. Therefore,

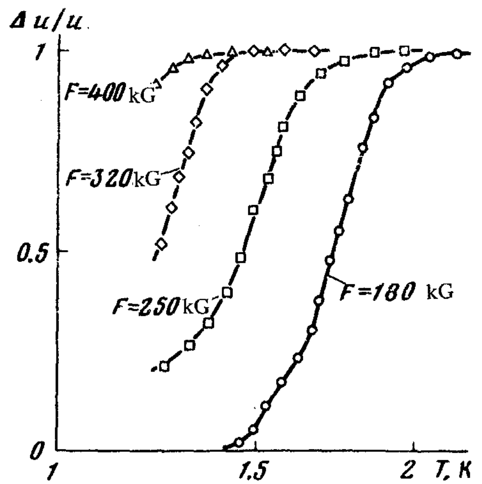

FIG. 2. Superconducting transitions of lead manometer.

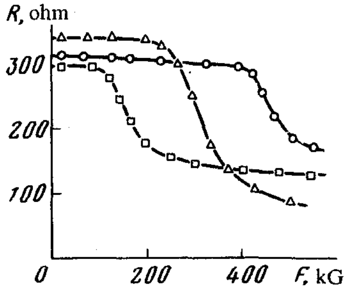

FIG. 3. Dependence of the electrical resistance of the high-pressure zone of the three pairs of anvils with identical dimensions of the working area on the force.

first, the carbonado anvils cannot withstand large stresses during compression. Despite the fact that diamond micrograins are much harder than the VK-3 hard alloy, the limiting loads that the carbonado anvils with a surface diameter of 1.5 to 2 mm can withstand exceed only slightly the permissible loads of the VK-3 anvil of the same size.

Second, because of the inhomogeneity of the material, the surface of the anvils [the surface of the point or the edge[2] (Fig. 1)] is a collection of projecting diamond grains of different hardness, which are cemented by a softer and more plastic bond, as a result of which the carbonado chambers can operate in two different modes.

A. The thickness of the sample's layer is much greater than the height of the microirregularities on the surface of the anvils. In this case the irregularities on the surface are insignificant, and a certain average "quasi-homogeneous" pressure, whose average value can be determined from the applied pressure and contact area, is produced in the sample as a result of compression. According to our estimates, the maximum "quasi-homogeneous" pressure attainable in the carbonado anvils operating in mode A is 400–600 kbar (according to the 1975 scale). The anvils are destroyed with further increase of the pressure.

B. The thickness of the sample's layer is of the order of the height of the microirregularities on the surface of the anvils. The specific property of case B is that in addition to some average "quasi-homogeneous" pressure, which does not exceed the value indicated above, the local pressures, which may greatly exceed the average pressure, are set up between the separate projections operating under conditions of strong side support.

The results of measurements of the electrical conductivity of the high-pressure region of the anvils during their compression indicate the onset of large local pressures. Figure 3 shows the dependence of the electrical resistance between the electrodes 4 (Fig. 1) on the acting force for anvils without a sample at $T = 4.2$ K. A decrease of the electrical resistance is apparently attributable to partial metallization of the anvil's material. Thus, the onset of metallization is not connected directly with the average pressure at the contact. This fact indicates that metallization occurs in micro regions, and the conditions for its onset are determined by the configuration of the microirregularities on the surface of the contact.

The dimensions of the high-pressure regions can be evaluated approximately by using the data of Refs. 4 and 5, if we assume that the observed transitions in Xe [4] and NaCl [5] are superconducting. Taking into account that a 1-K shift of T_c by the current $J = 200\ \mu\text{A}$ observed in Ref. 4 is equivalent to the magnetic field $H \approx 100$ Oe (since $dH_c/dT \sim 100$ Oe·deg^{-1} for soft superconductors) and, using the known ratio H (Oe) $= 0.2\,J$ (A)/r (cm), we can see that at least one of the dimensions r of the high-pressure region should be of the order of 4×10^{-7} cm. Analogous estimates based on NaCl data[5] give the values $r \sim 10^{-7}$ cm. We note that if NaCl is considered to be a rigid superconductor with a much higher critical field, then the values of r become unreasonably small. Therefore, the large values of H_c for NaCl indicate that the regions going over to the superconducting state are small.

Thus, it is considered that the dielectric-metal transitions, observed in Refs. 4 and 5 and in a number of other papers, occur only in microvolumes of the sample, whose dimensions do not exceed several tens of angstroms. Thus, it is basically impossible to equate the transition with a specific pressure estimated from the contact surface and the force. Second, it should be borne in mind that the conditions of the phase transitions and the properties of the new phase of the material in such small volumes can differ from the conditions for the equilibrium phase transitions and from the properties of bulk samples.

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