Optical investigations in the megabar range using a chamber consisting of a rounded cone and a flat foundation

M. I. Eremets, E. S. Itskevich, A. M. Shirokov, and E. N. Yakovlev
Institute of Physics of High Pressures, Academy of Sciences of the USSR

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A system of diamond punching dies with a rounded cone-flat plane geometry is used for optical studies of the R luminescence line shift in ruby under pressure and of the pressure distribution in the system. The largest shift occurs at 0.7 Mbar.

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Progress in the physics of high pressures in recent years is usually associated with the development of apparatus, in which high pressures of the order of 1 Mbar are created between diamond indenters. The pressures in this case are generated in a small volume ($\lesssim 10^{-4}$ mm^3). Two types of chambers are mainly used: 1) a chamber consisting of a rounded cone and a flat foundation[1-3] and 2) a chamber with flat anvils.[4-6]

The rounded cone-flat plane chamber was first used for physical investigations in Ref. 1 (see also the review article in Ref. 2). In this chamber, the electrical resistance of specimens is measured in order to observe insulator-metal transitions and superconductivity. The rounded cone and the flat plane, serving as the electrodes, consist of a conducting synthetic diamond of the carbonado type[1,2] or of natural diamonds with a sputtered metallic film.[3]

The volume of the chamber with the flat diamond anvils is large enough to conduct various physical investigations: optical, x-ray diffraction, and others.[4] The motivation for the development of such chambers was the possibility for measuring pressure with the help of an accurate and convenient method, namely, according to the shift in the R luminescence line of ruby under pressure.[4]

The rounded cone-flat plane type of construction is attractive due to its simplicity and potential from the point of view of the maximum attainable pressures, created between diamond plunger dies.[7] However, a characteristic of this type of chamber, which makes it difficult to carry out physical investigations, is the smallness of the volume in which the pressure is created: It is 2 to 3 orders of magnitude smaller than in chambers with flat anvils.

The purpose of the present work is to investigate the possibility of creating chambers for the megabar pressure range with the rounded cone-flat plane geometry, in which, in spite of the smallness of the high-pressure region, it would be possible to perform optical measurements and to determine the pressure using the ruby technique.

In the experiments described here, the luminescence spectra of ruby grains, placed in different points of the chamber in order to study the pressure distribution in it, were measured. One of the elements of the chamber, namely, the rounded cone or the flat base, was made of transparent diamond, while the other element was made of carbonado (Fig. 1). The ruby power (1% Cr), consisting of micron-size grains, was deposited on capacitor paper with a thickness of 12 μm, which served as a quasihydrostatic medium for the ruby.

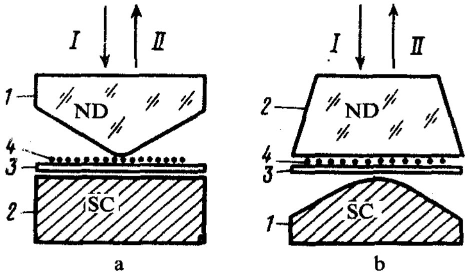

FIG. 1. Schematic diagram of the optical high-pressure chamber. (1) Rounded cone, radius of curvature $r = 0.2$ mm (a), $r = 0.8$ mm (b); (2) flat base; (3) capacitor paper; (4) ruby powder. I—Exciting radiation; II—luminescence radiation; ND is the natural diamond and SC is the synthetic carbonado.

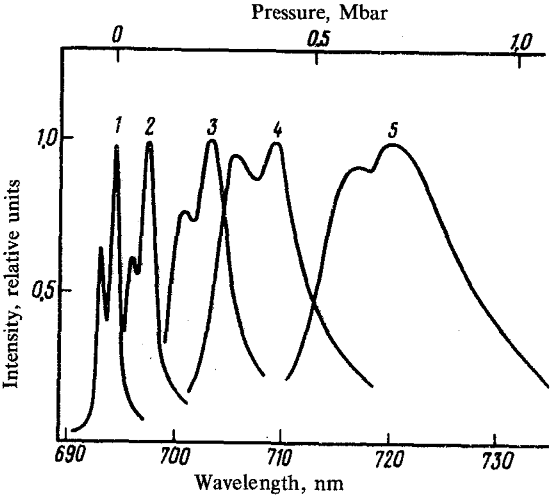

FIG. 2. Luminescence spectra of ruby, located at the center of the chamber (spectra 1–5 were obtained in different experiments).

The luminescence was excited with the help of a He–Cd laser ($\lambda = 441.6$ nm). The laser beam was focused on a spot with diameter 2–3 μm. The luminescence radiation out of this region was passed through an MDR-2 monochromator (resolution of 5 cm^{-1}) and recorded with the help of a FÉU-100 photomultiplier. The luminescence spectra from ruby grains with sizes less than 1 μm are recorded reliably. By moving the high-pressure chamber perpendicular to the laser beam to within ± 1 μm, it was possible to record the luminescence spectra at different points of the pressurized region.

Similar experiments on the investigation of the pressure distribution in the high-pressure chamber with flat diamond anvils were performed in Ref. 5. However, in Ref. 5, the minimum area from which the luminescent signal was recorded was $\simeq 400\,\mu\text{m}^2$, i.e., two orders of magnitude larger than necessary to perform the experiment in the rounded cone-flat plane chamber.

Figure 2 shows the luminescence spectra of ruby for different pressures, obtained in the experiment. The pressure was determined from the shift of the R_1 luminescence line ($d\lambda / dP = 0.0365$ nm kbar^{-1}). Under pressure, the R lines are broadened and the relative intensity of the R_1 lines decreases.

The maximum pressure measured in our experiments, in which we were not concerned with the problem of attaining limiting pressures, attained 0.7 Mbar (spectrum 5 in Fig. 2). It was obtained in the chamber with a transparent rounded cone ($r = 0.2$ mm); see Fig. 1a. After the experiment, a craterlike imprint was discovered on the surface of the carbonado with ϕ 100–120 μm. A maximum pressure of 0.4 Mbar was attained in the chamber illustrated in Fig. 1b (spectrum 4 in Fig. 2). When this pressure was attained, two weak concentric cracks, shaped like rounded hexagons with a diameter of 250 and 400 μm, appeared on the (111) working surface of the natural diamond (in this case, the diameter of the pressure zone was ~200 μm).

The pressure distribution with different applied loads was determined for both types of chambers. Examples of the measured pressure distributions are presented in

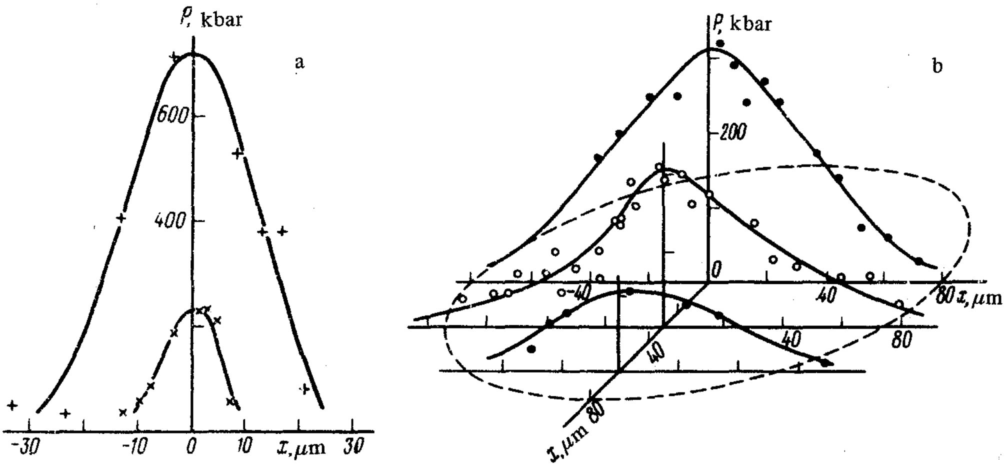

FIG. 3. Pressure distribution in the chamber. (a) Central cross section of the chamber; two different loads. (b) Cross Sections perpendicular to the x axis; one of the loads; the dashed line indicates the region of pressure with a diameter of 160 μm.

Fig. 3. As can be seen from the figure, the size of the region where the pressure differs from atmospheric pressure is small; for example, its diameter is 50 μm with a pressure of 0.7 Mbar at the center for the chamber in Fig. 1a. We note that the measured pressure distribution differs from the distribution following from Hertz's equation.

There naturally arise questions as to the conditions under which the ruby grains are located in the pressure region and does the capacitor paper play the role of the medium transmitting the pressure, i.e., do we have, in our case, a quasihydrostatic pressure chamber?

We shall note the facts that support this assertion. The appearance of the spectra obtained by us (Fig. 2) is virtually identical to the appearance of the spectra in Ref. 6, where the ruby grains were located in a quasihydrostatic plastic metallic medium (similar experiments served as a foundation for constructing the ruby scale in the megabar range). Thus we can hope that the conditions under which the ruby grains are located in our experiments are not worse than those in Ref. 6.

Another fact is the nonmonotonic nature of the pressure distribution in the chamber (Fig. 3) and the absence of significant deviations of the experimental points, which apparently indicates the absence of microcontacts, where a very large local pressure develops and microvolumes with low pressure.

Under the conditions of our experiment, even with small loads, the contact between the plunging dies occurs along a spherical surface. On the other hand, the nature of the pressure distribution differs from that predicted by the solution of Hertz's problem and is qualitatively close to that calculated in Ref. 7, where for similar systems the conditions required for the optimum stressed state are attained in order to obtain the maximum pressure at the center.

Close to the optimum conditions can be attained by using a lining, which can lead to a qualitative change in the distribution compared to Hertz's solution. The effect of the lining is confirmed also by the similar pressure distribution obtained with plunging dies having a different form.[5]

Thus, in this work, 1) we performed experiments in an optical chamber with the rounded cone-flat plane geometry and measured the luminescence spectrum of ruby specimens with dimensions $\sim 1\ \mu$m; 2) we measured for the first time the magnitude and distribution of the pressure in a chamber of this type.

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