High-Pressure Physics: The 1-Megabar Mark on the Ruby $R_I$ Static Pressure Scale

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Papers from the GEOPHYSICAL LABORATORY
Carnegie Institute of Washington
No. 1680
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Abstract. Ruby crystals were subjected to a static pressure greater than 1 megabar in a diamond-windowed pressure cell. The pressure was monitored continuously by observing the spectral shift of the sharp fluorescent \( R_1 \) ruby line excited with a cadmium-helium gas-diffusion laser beam. One megabar appears to be the highest pressure ever reported for a static experiment in which an internal calibration was employed.

Until recently, the limit to most high-pressure experimentation was approximately 300 kbar. That was the pressure to which internal calibration extended (for example, the volume equation of state of sodium chloride); it was also the pressure at which mechanical failure of apparatus usually occurred. In 1975 there were two reports of internally calibrated experiments at 500 kbar (1, 2), both of which employed extensions of the National Bureau of Standards (NBS) calibration (3) of pressure dependence of the wavelength of the \( R_1 \) ruby fluorescence line. The NBS calibration showed the spectral shift to be linear to 291 kbar, and required serious revision (a factor of 2 at 500 kbar) of previous fixed point scales (4), from which pressures had earlier been estimated (5).

Fig. 1. Simplified diagram of diamond pressure cell, after Mao and Bell (6). The two half-cylinders shown are of identical shape. The axis of the lower one is normal to the page; the axis of the upper one lies in the plane of the page. An upper half-cylinder of boron carbide is used for x-ray diffraction of the sample under pressure; it was replaced with a tungsten carbide half-cylinder for the experiments reported here. The upper portion of the outer cylinder is 3.2 mm in diameter. The work area of the diamonds (not drawn to scale) is \( 1.5 \times 10^{-3} \text{ cm}^2 \).

We report here experiments in which pressures of 1 Mbar were reached, as measured by a further extension of the new NBS scale. The data are reproducible and can be easily compared with other types of calibration. The estimated uncertainty in pressure is no greater than 10 percent. To the best of our knowledge, this is the highest static pressure ever reached in an experiment in which an internal calibration was employed.

The difference between external and internal calibration of pressure is fundamental. External procedures usually involve monitoring mechanical loading of high-pressure apparatus. However, the loading is not transmitted to an internal sample because the apparatus deforms, and as parts begin to yield it is not possible to determine the internal pressure.

In the experiments reported here, it was possible to monitor the sample being pressurized with a new diamond-windowed cell. The cell was designed for static experimentation in the megabar pressure range, which was inaccessible with previous apparatus. Ruby fluorescence in the cell was excited by a laser beam, and its wavelength was monitored continuously with a spectrometer linked to the pressure cell by a fiber optic bundle.

The improved diamond pressure cell used in the experiments has been described in detail by Mao and Bell (6) and is shown diagrammatically in Fig. 1. The apparatus
two single-crystal diamonds opposed as pressure anvils. A scissors-shaped lever-block assembly is spring-loaded to apply a mechanical advantage of 2. The diamonds are supported by half-cylinder seats of tungsten carbide with a zirconium shim (0.001 inch thick) placed between the low-pressure-bearing surfaces. The half-cylinders are adjusted to achieve and maintain excellent alignment of the diamonds (to better than one-half a Newton color fringe interference of the diamond faces) during an experiment. A sheet (0.010 inch thick) of work-hardened steel (7) is placed between the high-pressure diamond faces, and then a crystal of ruby is placed on the steel and pressed into it as the diamond anvils are squeezed together. Blue laser light (8), wavelength 441 nm, is used to excite fluorescence in the ruby. Spectrometer and detector systems are the same as previously described (6), except that the photomultiplier tube in the experiments was cooled to \(-50^\circ\text{C}\) to reduce dark noise (9).

Before the experiments were done we observed the sodium chloride B1–B2 (NaCl-CsCl structure types) transition at 291 kbar (1) and simultaneously measured the wavelength of the \(R_1\) ruby line. A single ruby fragment was monitored each time an experiment was done. The observed spectral shift of the \(R_1\) ruby line in a typical experiment and the corresponding pressures from a linear extension of the NBS scale are listed in Table 1. The intensity of the \(R_1\) ruby line appeared to diminish slightly as the pressure was increased to the megabar range. No sign of mechanical failure was observed in the diamonds, and with improved support it should be possible to increase the pressure to at least 1.5 Mbar.

<table>
<thead>
<tr>
<th>(\Delta \lambda (\text{\AA}))</th>
<th>(\text{Pressure (kbar)})</th>
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<tbody>
<tr>
<td>30</td>
<td>83</td>
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<tr>
<td>75</td>
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<td>370</td>
<td>1018</td>
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</table>

The capability of routinely experimenting at pressures in the megabar range has far-reaching applications. It will be possible to study insulator-metal transitions and numerous other proposed physical and chemical changes in materials at high pressures (10). The accessibility of this pressure range coupled with the high temperatures already reached (2) makes it possible to experiment directly at the conditions of the earth’s core.

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References and Notes
7. This material was kindly supplied by L. C. Ming, University of Rochester.
11. We wish to thank G. J. Piermarini and S. Block of the National Bureau of Standards and A. Van Valkenburg of the Geophysical Laboratory for useful suggestions and assistance in this project.

29 December 1975; revised 8 January 1976