

INDIRECT PRESSURE MEASUREMENTS IN DIAMOND CELL UP TO 1 MEGABAR

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Résumé - On décrit une méthode de mesure indirecte de la pression en cellule diamant. Des jauges de contrainte mesurent la force appliquée aux enclumes qui a été étalonnée en fonction de la pression dans l'échantillon par la méthode de fluorescence du rubis.

Abstract - An indirect pressure measurement method for the diamond anvil cell is described. Strain gauges measuring the force applied to the anvils, are calibrated against the actual pressure in the sample, measured by the ruby fluorescence technique.

Diamond anvil cells (DAC) associated with laser heating are of particular interest in geophysical studies, since they can reproduce pressure and temperature conditions of the earth mantle. The classical determination of the pressure in a DAC consists in measuring the wavelength shift of ruby fluorescence lines under pressure. This shift has been calibrated by X-rays methods up to 1 MBar [1]. Unfortunately the ruby fluorescence method may sometimes be inconvenient since the ruby chips may take part in mineral reactions at high temperature, as it was shown by Madon [2]: a sample of synthetic $(\text{Mg}_{0.5}\text{Fe}_{0.5})_2\text{SiO}_4$ olivine powder was compressed with small ruby chips in a diamond cell to 240 kbar and laser heated to 1300°C. Examination of the recovered sample by transmission electron microscopy revealed the presence of $\approx 30\%$ vol. of an aluminous ferro-magnesian garnet produced by the reaction between olivine and ruby. Such reactions are clearly inconvenient for the study of phase transformations. There is therefore a need for a method of pressure determination that does not interfere with the reaction under investigation. We have developed an indirect method for measuring pressure, using strain gauges.

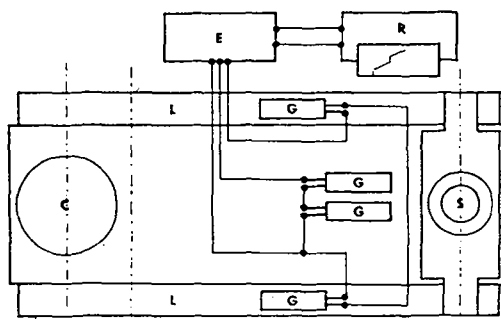
Calibration procedure and results

Fig. 1 - Strain gauges Bridge
G : gauges, L, levers,
C : cylinder, S : screw,
E : extensometer,
R : recorder.

The DAC we use is of the type described by Mao and Bell [3]. The anvils are 0.3 carat brilliant-cut diamonds with 16 facets and a flat table 0,5mm in diameter. Two strain gauges in series, glued on the load transmitting members (fig. 1) measure their mean elastic deformation, a linear function of the load applied by the screw. Two other strain gauges, glued on the body of the cell, equilibrate the temperature

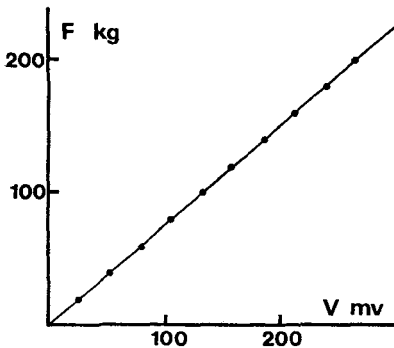


Fig. 2 - Force calibration

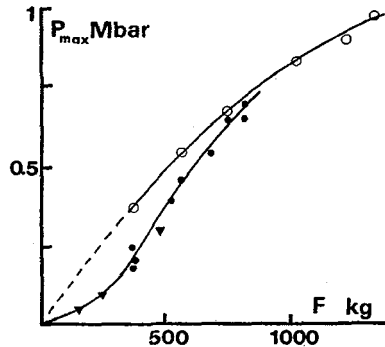


Fig. 3 - Pressure calibration in Ni gasket
 Open circles : 30µm diam. hole, ruby fluo.
 Closed circles : 300µm diam. hole, ruby fluo.
 Triangles : 300 µm diam. hole, phase transitions : CuCl 44,2 kbar, AgI 100 kbar, NaCl 300 kbar.

variations of the active gauges. This half-bridge is connected to a Philips type 9308 extensometer bridge with two internal resistances. The system works as a wheatstone bridge. The output voltage of the bridge depends on the gauges and extensometer used. It has been calibrated against the applied load using an Instron compression machine, directly applying the force to the levers (fig. 2) The cell is designed so that the axial force on the diamonds is equal to 5 times the load applied by the screw). The applied load was in turn calibrated as a function of the maximum hydrostatic pressure between the diamonds. Pressure calibrations were made using well known phase transitions (CuCl, AgI, NaCl) as well as the ruby fluorescence technique at room temperature (fig. 3). For the ruby fluorescence technique the following equation, given by Mao and Bell [1], was used :

$$P_{\text{Mbar}} = 3,808 \left[\left(\frac{\lambda_0 + \Delta\lambda}{\lambda_0} \right)^5 - 1 \right]$$

where $\lambda_0 = 694 \text{ nm}$ is the wave length of the fluorescence line, at room pressure (and $\Delta\lambda$ is the wave length shift. It can be seen fig. 3 that the relation between pressure and force depends on the initial geometry of the gasket. The method described here is therefore successful only if specific calibration conditions are used for experiments. Below 250 kbar, with a 300 µm diameter hole (usual experimental conditions), the obtained pressure for a given force is strongly dependant on the compressibility of the uncompacted powder sample. In this domain it is difficult to obtain reproducible calibration results. It can be noted that at very high pressure the two curves fig. 3 tend towards a common limit and the calibration becomes almost independant on the initial experimental conditions.

The accuracy of the pressure measurements by strain gauges is limited by the accuracy of the ruby technique. At very high pressure, with a solid medium, the ruby lines become very broad and an accurate determination of the peaks is not possible. The values of pressure are obtained with an accuracy not better than 10 %.

Strain gauges, carefully used, can therefore give continuous pressure measurements, as accurate as the ruby fluorescence technique, up to 1 Mbar, even during laser heating.

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