GARNET ANVIL CELL (GAC) FOR HYDROTHERMAL STUDIES TO 6.0 GPA AND 1200 °C

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We have set up a high pressure cell with transparent garnet anvils for the study of hydrothermal mineral reactions and the deformation of rocks/minerals under visual observation in the pressure range up to 6.0 GPa and to temperatures of 1200 °C. Its main advantages are the higher temperature that can be achieved under nearly isothermal conditions and the much easier handling of garnet anvils in comparism with a diamond anvil cell. The new cell has been used for optical microscope observations and for deformation experiments in a special shear cell with one rotable anvil.

1 Introduction

Diamond anvil cells (DAC) have been used in a wide variety of high-pressure studies both at room and high temperature. Under external heating, oxidation of diamonds and the metal heaters require protection of the inner cell assembly by an inert or reducing gas atmosphere. Graphitization of diamond sets in above 900 - 1000 °C with increasing intensity. Furthermore, the strength of necessary supporting material (e.g. tungsten carbide) is considerably reduced at temperatures above 800 °C, thus limiting the reachable pressure in an externally heated DAC at very high temperatures to at least 2.5 GPa [1]. Instead of diamond, we used cubic garnet single crystals (YAG) as transparent anvils in a high-pressure cell. Among the various materials of the garnet structure, $Y_3Al_5O_{12}$ (YAG) has the highest melting point ($T_m = 2273$ K) and hence is likely to be the most resistant to plastic deformation at high temperatures. Garnets in general appear to have a creep strength higher by a factor of 3-10 than other oxides, compared at the same homologous temperatures T/T_m [2]. From these findings, we expect that a garnet anvil cell (GAC) can operate even up to 1500 °C under external heating, i.e. under nearly hydrothermal conditions compared to laser-heated cells. We have performed experiments with a garnet anvil cell up to 800 °C without any special precautions, and up to 1200 °C under protection gas. The reachable pressure depends on the size and shape of the active anvil face, with coned (diamond-like) garnet cylinders with 5 mm outer diameter and a 500 mm anvil face we were able to get 6.0 GPa without any problems. With a gasket-free preparation, we used a special cell with shear mechanism to study the brittle-ductile transition in calcite by deforming small calcite cylinders (2 mm diameter and 30 mm thickness) in the GAC under visual control. [3]

2 The Garnet Anvil Cell (GAC)

The cell body was designed for the study of high pressure phase transitions under shear deformation up to 100 GPa confining pressure at ambient temperatures and is described in detail in the literature [4]. In short, it consists of a lever-arm pressure loading mechanism pressing two opposing anvils together, where the lower anvil can be rotated using a suitable external lever rod, see Figure 1. With diamond anvils, the setup was succesfully used to determine the equilibrium phase boundary of a range of solid-solid phase transitions at low or room temperatures [5].

For present purposes, the inner part of the cell consisting of the two anvils combined with a re-

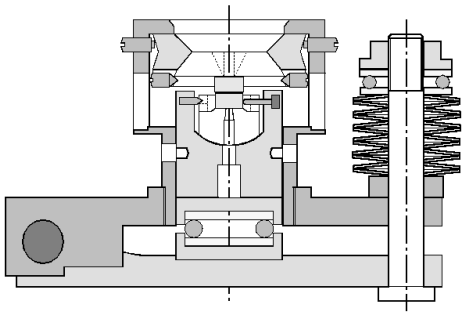

Figure 1: Side view of the shear cell with garnet anvils. The inner assembly is shown schematically only.

sistance heating, was completely re-designed: Instead of diamonds with tungsten carbide support, we use temperature resistive garnet single crystals machined to small cylinders of 5 mm in diameter and with a spherical upper surface to build the anvils.

A small compression face up to 2.5 mm in diameter is formed by creating a polished face on top of the upper surface. The smaller the diameter d_{cf} of the compression face, the more effectively it is supported by the mass around it. The back side of the anvils is a plane circle with slightly bevelled edges. The maximum pressure that can be reached in the central part of the sample is given with [3,6]

$$p_{max}(\text{in GPa}) = \frac{12.5}{[d_{cf}(\text{in mm})]^2} \quad . \qquad (1)$$

We estimate that a pressure of up to 2 GPa can be reached for $d_{cf} \cong 2.5$ mm. Using garnet anvils with smaller upper face (0.5 mm diameter), we were able to reach 6.0 GPa at room temperature without any problems.

Commercially available mantled heater wires (0.5 mm in diameter) wound around the anvils deliver heat mainly by direct thermal conduction. This geometry requires the least input of power and the lowest heater temperature in order to achieve a desired sample temperature [1]. The parallel alignment of both garnet anvils must be guarantied in order to prevent their destruction during shearing under an external axial load. Under applied torque during the deformation experiments, the cubic symmetry of the garnet anvils is slightly disturbed, and a weak cross shadow appears under crossed nicols superimposed to the sample image. Taking a time series of digitized snapshot images, it is possible to compensate this effect using suitable numerical routines.

3 Temperature and pressure distribution

As a consequence of the inner geometry of the cell, the temperature gradients over a sample sandwiched between the anvils remain negligibly small. This was checked by heating homogeneous discs of metals with a low melting point. This reveals no significant spatial dependency of melting onset under visual detection up to 330 °C and atmospheric pressure (melt transitions of Sn, Bi and Pb at 231,9 °C, 271,4 °C and 327,5 °C, respectively).

Under uniaxial loading, the stresses generated between the two Bridgman anvils obey the general relationship

$$\frac{d\sigma_r}{dr} + \frac{\sigma_r - \sigma_\theta}{r} + \frac{2f\sigma_z}{h} = 0, \qquad (2)$$

with the radial stress σ_r, tangential stress σ_θ, and axial stress component σ_z depending on radius r. h is the thickness of the sample disc and f is the coefficient of friction between the sapphire anvil and the sample surfaces [7]. Its solution for a cylinder with radius R under the external loading force F is given either by

$$\sigma_r(r) = 2f\sigma_z(R-r)/3h, \qquad (3a)$$

$$\sigma_\theta(r) = 2f\sigma_z(R+r)/3h, \qquad (3b)$$

$$\sigma_z = F/\pi R^2, \qquad (3c)$$

for completely elastic deformations, or alternatively by

$$\sigma_r(r) = \sigma_\theta(r) = \sigma_z(r) - \sigma_0$$
$$= \sigma_0[\exp(2f(R-r)/h) - 1] \qquad (4)$$

for a cylinder deformed in the fully plastic regime, where σ_0 is the yield stress according to the Tresca yield criterion [7]. The radial pressure distribution inside the sample chamber is given by

$$p(r) = \frac{1}{3}[\sigma_r(r) + \sigma_\theta(r) + \sigma_z] \qquad (5)$$

and is either independent on radius r in the elastic limit according to eq. (3), or decreases exponentially with r in the flow region, in dependence of the friction coefficient f and the aspect ratio R/h, as shown in eq. (4).

It can be shown, that, for uniaxial loading, the central part of a sample between rigid cylindrical anvils remains at nearly hydrostatic conditions, separated from a surrounding ring showing plastic flow [8]. In this sense, the outer plastic ring of the sample acts as a gasket for its inner parts.

In addition to uniaxial loading, we are able to apply independent shear forces by rotating the lower anvil. The shear stresses produced in the sample disc are, under boundary conditions of no-slip, a linearly increasing function of r in the central (nearly elastic) region. The total shear stress experienced by the sample is therefore gradually increasing from zero at the centre and reaches its maximum near to yield stress at the periphery ring. The displacement field at the surface of the sample may be reconstructed either by image processing (subtraction of subsequent digitized images), or by means of strain markers distributed over the sample. In both cases, the accuracy is mainly limited by the spatial resolution of the digitizing device (video camera or scanner).

4 Discussion

Using a standard gasket preparation, the GAC cell operates like a regular diamond anvil cell. It can therefore be used for in situ x-ray diffraction experiments as well as for optical spectroscopy, since garnet is transparent for the respective wavelength spectra.

However, the garnet structure gives rise to many Raman- and IR-active lattice modes, which is clearly disadvantageous for optical in situ studies and a drawback of the cell in comparism to a diamond anvil cell. On the other hand, deformation experiments using this technique are relatively simple and inexpensive. Furthermore, combining deformation experiments with ultrasonic measurements using a recently developed technique[9] seems to be a particularly interesting application of this cell. In-situ information on the time dependency of specific subprocesses could lead to a better understanding of the underlying kinetic mechanisms at the microscopic scale. The work on this topic is in current progress.

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The usage of garnets instead of diamonds in a high pressure cell is a suggestion of Shun Karato.

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