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A new shear apparatus for experimental deformation of rocks under the microscope

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Abstract—A new experimental technique for the study of deformation processes in rocks in transmitted light under the microscope uses a modified sapphire anvil cell in which one of the sapphire anvils can be rotated and thus permit the deformation of a thin solid sample disc. The deformation can be observed during the experiment through the anvils using a polarizing microscope stage. The technique is applicable up to 450°C and up to 2 GPa confining pressure in the central part of the sample.

INTRODUCTION

One of the aims of earth scientists is to gain a deeper insight into the physics of deformation mechanisms in rocks in order to be able to extrapolate constitutive equations to inaccessible conditions of strain rate and time (typically 10^{-15} s^{-1} and 1 Ma for geologic processes), and to understand how the microstructures observed in rocks may reflect natural p–T conditions of deformation. For this reason numerous high-pressure deformation devices have been developed (see e.g. Hobbs & Heard 1986) which allows deformation under controlled conditions. Among them, the probably most accurate device is the Paterson gas-medium apparatus (Paterson 1990).

One drawback of these experimental techniques, however, is the inaccessibility of microstructural data during the deformation process. Analysis of the sample is only possible by studying the 'post-mortem' case, not the *in situ* dynamic conditions. This is a serious limitation, since it is well known that ductile deformation processes in minerals operate far from thermodynamic equilibrium, and a single microstructure could be the result of a range of different deformation histories. Furthermore, the fabric may be modified after deformation during cooling and unloading.

In this paper we propose an alternative technique to circumvent this drawback. It is closely related to the Hajeck press for deformation of materials in thin section under the petrographic microscope (Means & Xia 1981, Jessell 1986), but is not restricted to the investigation of soft analogue materials instead of natural rocks. The main idea is to use a high-pressure device of the diamond-anvil cell type, but with large sapphire anvils, and to provide the possibility to generate controlled shear stresses inside the sample by applying an external torque to the anvils. The evolving deformation is monitored in transmitted light under the polarizing microscope, and typical time constants for several characteristic microprocesses may be calculated from digitized pictures using conventional image processing methods.

THE SAPPHIRE ANVIL CELL

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 (Barabanov *et al.* 1987). It consists of a lever-arm pressure loading mechanism pressing two opposing anvils together, where the lower anvil can be rotated using as suitable external lever rod (Fig. 1a). With diamond anvils, the set up was successfully used to determine the equilibrium phase boundary of a range of solid-solid phase transitions at low or room temperature (see e.g. Blank & Zerr 1992, Blank & Buga 1993).

For present purposes, the inner part of the cell consisting of the two anvils combined with a resistance heating, was completely re-designed: instead of diamonds we use temperature resistant sapphire spheres of 6 mm in diameter to build the anvils. A small compression face up to 2.5 mm in diameter is formed by creating a polished face on the spherical sapphire single crystals. The smaller the diameter d_{cf} of the compression face, the more effectively it is supported by the mass around it. Another flat face, 2.5 mm in diameter is formed on the opposite side of the anvils, and is used as an optical window for the observations. The maximum pressure that can be reached in the central part of the sample is according to

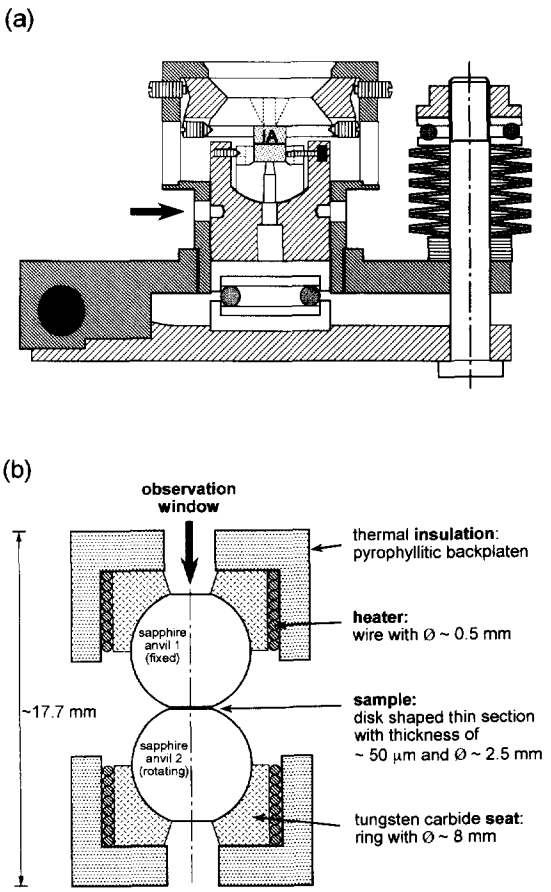

Fig. 1. (a) Side view of the sapphire shear cell. The lever-arm construction for the generation of axial load (tightening of a nut on the threaded end of an axis with spring washers) is seen on the bottom/right-hand side of the cell. The inner piston may be rotated using an external metal rod through the window of the cell body near the centre (arrow); IA=inner assembly. (b) Schematic drawing of the inner assembly of the cell with heater, thermal insulation, and mounted mineral thin section (φ = diameter).

the empirical relationship of Dunstan & Spain (1989) given by

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

We estimate that a pressure of up to 2 GPa can be reached for $d_{\text{cf}} \cong 2.5$ mm. Using a similar set up with slightly larger sapphire balls, Takano & Wakatsuki (1991) were able to reach 12.6 GPa at room temperature. The anvils are set in tungsten carbide seats. Commercially available mantled heater wires (0.5 mm in diameter) wound around the tungsten carbide seats deliver heat mainly by direct thermal conduction to the seats and then to the sapphire anvils. This geometry requires at least input of power and the lowest heater temperature in order to achieve a desired sample temperature (Bassett *et al.* 1993). A scheme of the inner assembly is shown in Fig. 1(b).

Sample preparation is similar to the first stage of the frequently used methods producing specimens for transmission electron microscope analysis (TEM): a rock cylinder of 2.5 mm in diameter is cut from the bulk material using a diamond coring tool. For materials which are particularly susceptible to cracking, this step could be performed using ultrasonic cutting techniques. Then the obtained rock cylinders are sawn into discs of about 200–300 μm thickness. The resulting cylinders are subsequently ground to their final thickness of 30–50 μm and polished to optical quality on both surfaces. Finally, the polycrystalline minidisc obtained is mounted on top of the lower sapphire anvil and loaded into the high-pressure cell. The parallel alignment of both sapphire anvils must be checked carefully in order to prevent their destruction during shearing under an external axial load. Both sapphire spheres are oriented with the *c*-axis perpendicular to their polished faces. As a result, we can make use of crossed polarizers for the examination of the mounted rock sample. Under applied torque, the crystal symmetry of the sapphire anvils is slightly disturbed, and a weak cross shadow may appear superimposed to the sample image. Taking a time series of digitized snapshot images during an experimental run, we are able to compensate this effect using suitable numerical routines.

TEMPERATURE AND PRESSURE DISTRIBUTION

As a consequence of the inner geometry of the cell, the temperature gradients over the cylindrical sample 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). It is assumed that the temperature is still homogeneously distributed over the sample volume at the maximum value of about 450°C during deformation runs performed with natural rocks (calcite), at least within the range of ±10°C.

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

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

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 (Jackson & Waxman 1963). Its solution for a cylinder with radius R under the external loading force F is given either by

$$\sigma_r(r) = \frac{2f\sigma_z}{3h}(R - r),$$

$$\sigma_\theta(r) = \frac{2f\sigma_z}{3h}(R + r), \quad (3)$$

$$\sigma_z = \frac{F}{\pi R^2}$$

for completely elastic deformations, or alternatively by

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

for a cylinder deformed in the fully plastic regime, where σ_0 is the yield stress according to the Tresca yield criterion (Jackson & Waxman 1963).

The radial pressure distribution inside the sample chamber is given by

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

and is either independent on radius r in the elastic limit according to equation (3), or decreases exponentially with r in the flow region, in dependence of the friction coefficient f and the aspect ration R/h, as shown in equation (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 (Prins 1984). 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 (Maugin 1992). 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).

DISCUSSION

Deformation experiments using this technique are relatively simple and inexpensive. This technique could also be applied in the research on the development of microstructures in recrystallizating materials and materials undergoing phase changes. In both cases, *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. Generally, all observable changes in microstructure of a polycrystalline disc (crack growth and healing, dynamic recrystallization, grain boundary migration, sub-grain formation and rotation, etc.) are closely related to a change in the mechanical properties of the studied material, and hence of particular interest to the study of the brittle-ductile transition of rocks. Furthermore, the *in situ* observation of deformation processes in high-pressure minerals (e.g. aragonite) seems to be a promising application of the presented cell. The technique needs, of course further development, especially a provision for producing controlled stresses inside the sample. There are also questions about the relevance of observations made on thin sections compared to the behaviour in the bulk material. However, the method is suitable for the investigation of the kinetic time constants of microprocesses in the neighbourhood of grain boundaries or for the visual detection of qualitative changes of deformation mechanisms under varying thermodynamic conditions. The method could also be developed as an interesting supplementary tool in high pressure rock deformation experiments.

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