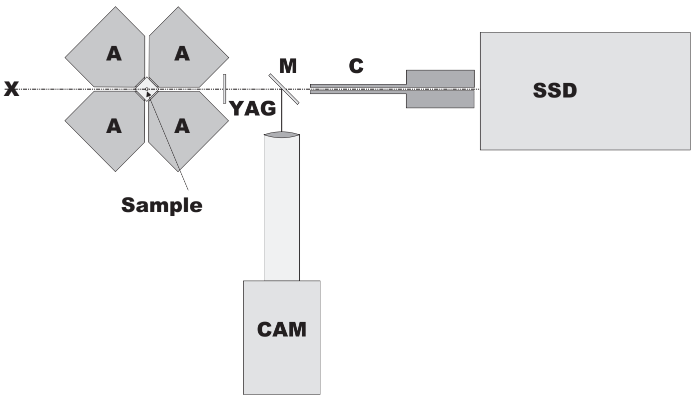
Use of X-ray Imaging Techniques at High Pressure and Temperature for Strain Measurements

Michael Vaughan, Jiuhua Chen, Li Li, Donald Weidner, and Baosheng Li
Center for High Pressure Research, State University of New York, Stony Brook, NY, 11794-2100, USA

We have developed a method for measuring the macroscopic strain in a polycrystalline or single crystal sample under high pressure and temperature using an x-ray "shadowing" method. The 1 mm^3 sample is contained inside a cell assembly similar to that use for a DIA-type cube anvil high-pressure apparatus. Transmitted x-rays are incident on a fluorescent crystal; the resulting image is viewed by a CCD camera and stored on videotape. By varying the geometry of the anvils and/or the cell contents, we can alter the stress field. These experiments have been carried out at about 10 GPa at temperatures up to 1100°C. [synchrotron x-ray imaging strain rheology]



1. Description

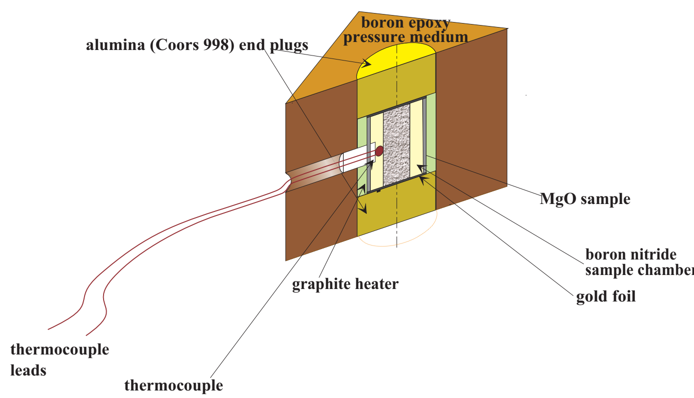
The above diagram is the system in plan view – i.e. looking down from the top. The system uses x-rays from a synchrotron source for two purposes: conventional energy-dispersive diffraction in a vertical diffraction plane, and imaging. The x-rays (**X**) pass between the four anvils (**A**) and through the sample. The sample is a one-mm diameter vertical cylinder about one mm long. The transmitted x-rays impinge on a fluorescent YAG crystal, where an image of the sample is generated. The visible light generated by the YAG is reflected by the mirror (**M**) into a CCD camera (**CAM**). X-rays diffracted from the sample pass over the YAG-mirror assembly through the collimator (**C**) into a solid-state detector (**SSD**).

2. Experimental Procedure

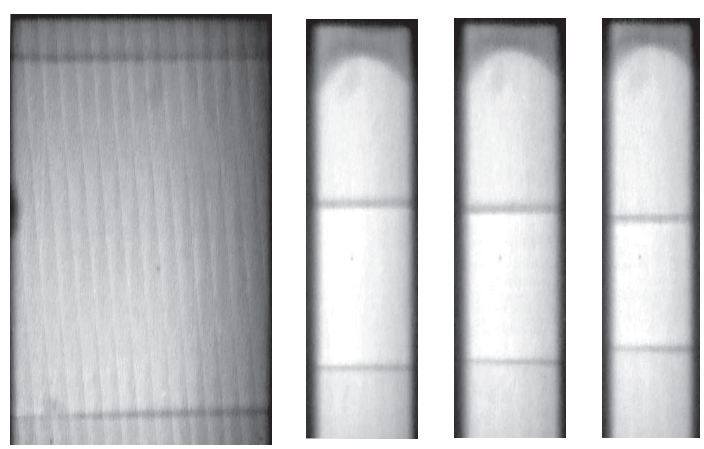
Experiments using this setup have been carried out at the superconducting wiggler beamline (X17) at the NSLS (National Synchrotron Light Source, Brookhaven National Laboratory, New York). These experiments used white radiation and a DIA-type high-pressure apparatus. In the standard DIA apparatus, we try to minimize deviatoric stress; for these experiments we modify this in various ways.

3. Strain Rate Measurements

In the example shown below, a hard "piston" of alumina is placed above and below the sample to greatly increase the deviatoric stress. Above and below the MgO sample are placed gold foil. Below, we see a cutaway diagram of the cell assembly.



The gold foil can be easily seen near the top and bottom of the left-hand (room P, T) image, below.

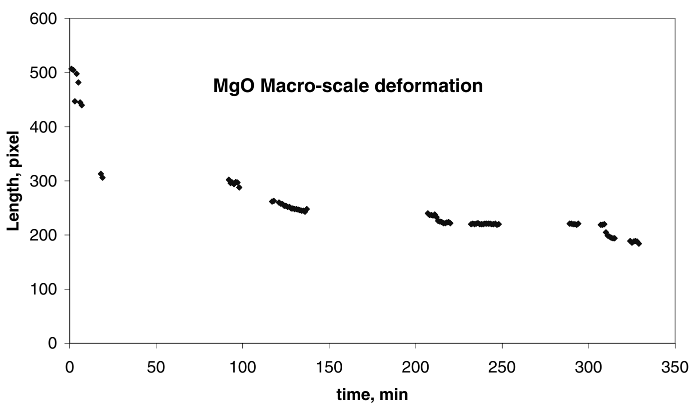


Room P Room T	10 GPa 25°C	10 GPa 600°C	10 GPa 1100°C

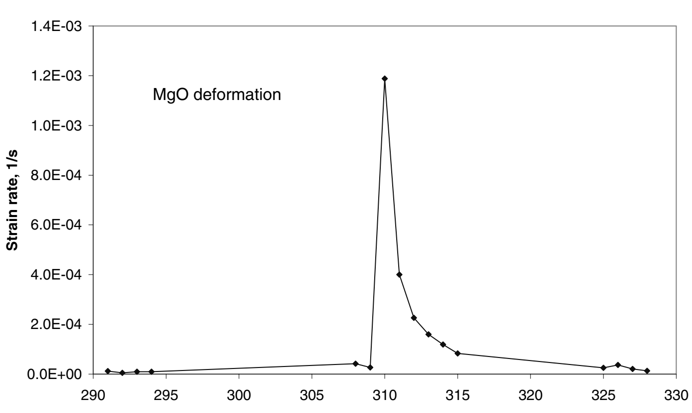
During the experiment, we increased the pressure at room temperature and observed a 53% shortening of the sample. We then stopped increasing the pressure, and increased the temperature to 600°C. The sample continued to shorten another 8%. We finally increased the temperature to 1100°C, observing an additional 16% shortening. Obviously, this shortening must be accompanied by a corresponding increase in diameter. The sides of the image are blocked by the anvils, which continue to get closer as the pressure and temperature are increased.

To determine the pressure, we measure the unit cell volume of the MgO with diffraction, and use its equation of state to calculate the pressure. The temperature is measured using a thermocouple.

The entire process was recorded on video tape. Images taken each minute were captured and analyzed to determine the strain and strain rate. A program (JDK 1.2) was written to track the spacing between the gold strain markers as a function of time. By comparing two images taken at different times, the relatively small size change of the sample can be measured, even if it is smaller than one pixel. Using a least-squares method, the position change of the gold markers is located. These data were used to calculate the strain and strain rate as a function of time, as shown in the next two figures.



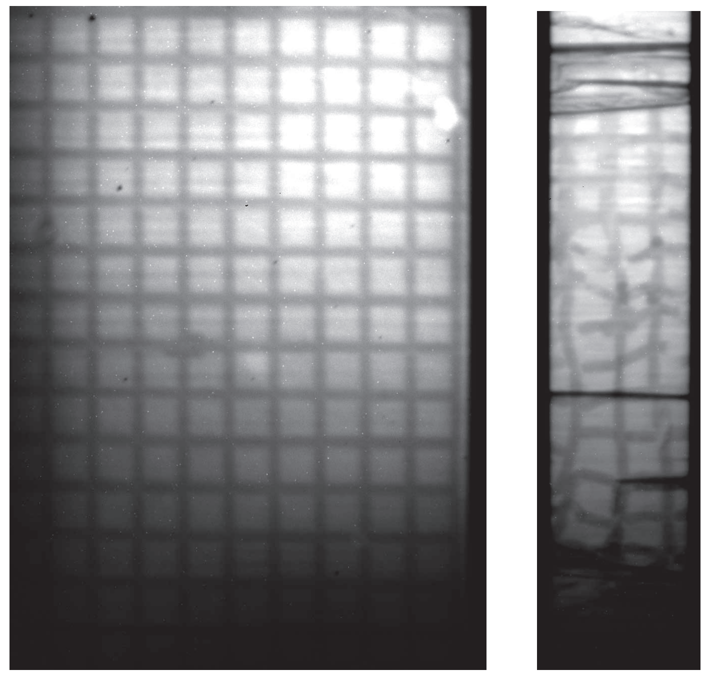
Length of the MgO sample as a function of time. One pixel is about 4 microns. Compression was completed by about 210 minutes. The first heating occurred at about 200 minutes to 600°C where it was maintained until the second heating at about 300 minutes to 1100°C. The compression phase was not uniform in time, but temperature changes occurred in less than one minute.



Strain rate *vs.* time for the MgO sample during and after the temperature change from 600°C to 1100°C. The temperature change was completed in less than one minute.

4. Distortion Measurements

In another type of experiment, a cylindrical sample of olivine was cut in half vertically and a gold TEM grid sandwiched between the two halves. Below are the resulting images before and after compression and heating. The distortion of the sample is mimicked by the distortion of the grid.



Room P	~6 GPa
Room T	358°C

If few fix some special points inside the grid, it is possible to study the distribution of the stress field inside the crystal. The grid method still needs further study to improve the resolution of images and stability of the cell assembly.

5. Summary

This x-ray imaging technique can be used to study macroscopic strain of polycrystalline or single crystal samples under high pressure and high temperature. It will be a helpful tool in the field of mineral science. It is also helpful in other studies such as tracking the length of a sample for ultrasonic interferometry, and determining the sample density using x-ray absorption.