Abstract No. chen0534

In Situ Stress and Strain Measurements at High Pressure and Temperature

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Beamline(s): X17B1

Introduction: Strain rate and stress are two key variables in rheological studies. Experimental data of these two variables are crucial in deriving the flow law that governs the material rheological behavior[1]. Rheologcial property of minerals is very important for understanding the earth dynamic phenomena such as mantle convection and deep earthquakes. Strain and stress measurements in previous studies are based on estimation through piston displacement and external load. Complexity and uncertainty are introduced in modelling the friction and piston deformation. Here we report a system of direct measurements of strain and stress of sample at high pressure and temperature.

Methods and Materials: The strain is measured by correlating sample length marks in recorded x-ray radiographs. The stress is measured by simultaneously collecting diffraction patterns of the sample in two perpendicular diffraction planes (e.g. defined by principal directions). Differential stress in the sample is derived from the different lattice strains along different principal directions. The measured stress is accurate to about bars and strain about 10^{-4}. High pressures are generated in the 'T-cup' 6-8 double-stage apparatus. Sintered cBN cubes are used as second stage anvils which allow x-ray access for multi-diffraction detection (Figure 1).

Results: Rheological properties of fayalite have been studied by using this system. Powdered sample is packed in a cylindrical chamber between two dense corundum hard pistons. Cold compression generates significant differential stress (4GPa) in the sample. Heating to 700°C totally releases the differential stress with 10% plastic strain. Further compression regains differential stress up to 2 GPa. A significant weakening of the sample is observed during phase transition from olivine to spinel structure. About 1.5 GPa differential stress is generated in the transformed sample by further compression. Figure 2 illustrates the stress and pressure history of the fayalite sample during the fourth heating cycle. The sample is completely transformed to spinel. The initial compression raised the differential stress to about 0.8 GPa. Temperature further increases the stress. This is because of the thermal expansion of the sample and corundum plugs. The furnace is also cylindrical. We see at 400 °C that the stress increases with time at constant temperature. We attribute this to the relaxation of the cell. Complex frictional forces in the cell define the ultimate stress that is delivered to the sample. A further increase in temperature is accompanied with decreases in stress and increases in strain rate. Here the sample is flowing. And with this data we can derive some of the flow properties of this material. In this particular experiment, the sample length changed by about 5-10% during each cycle.

Conclusions: This technique provides means to directly measure both strains and differential stresses in a sample, and therefore avoids uncertainties of instrumental corrections and assumptions in a traditional device for high-pressure rheology measurements. It opens a new window for studying rheological properties of materials under high pressures and temperatures.

Acknowledgments: This study was supported by the US National Science Foundation

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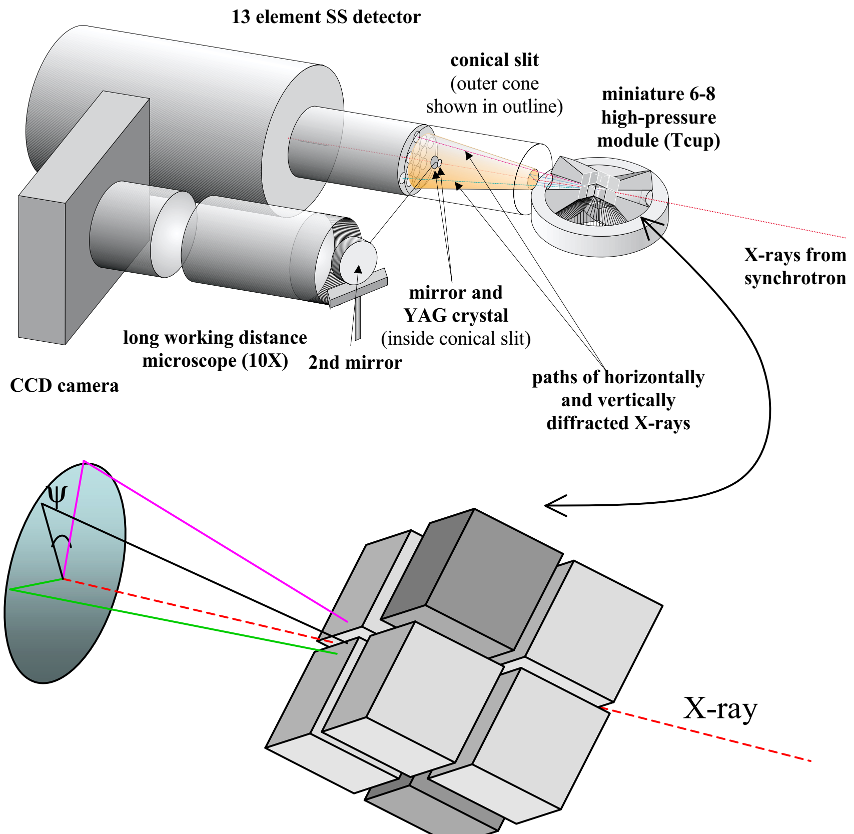

Figure 1. Detection optics with conical slit assembly. The x-rays pass through the sample in the Tcup, and the 2-theta angle is fixed by the conical slit. An energy dispersive detector array is placed behind the conical slit and samples the diffracted x-rays at specific values of ψ. Also illustrated is the image optics. A YAG crystal which converts x-rays to optical light is magnified by the long working distance microscope and recorded by the CCD camera. The YAG crystal is embedded in the conical slit assembly.

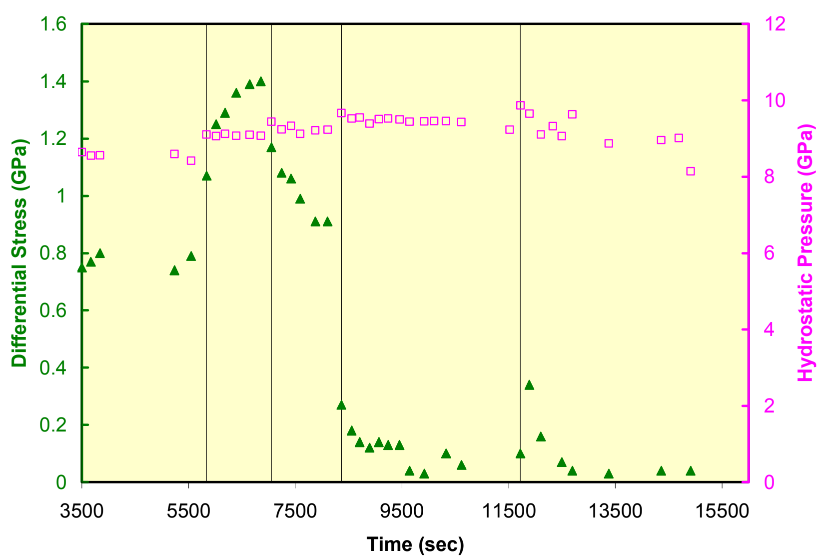

Figure 2. Stress and pressure history of a fayalite-spinel sample during the fourth heating cycle. At the end of the third cycle, the differential stress was zero. Further compression elevated the differential stress to 0.8 GPa at the beginning of this figure.