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Review

# Studies of mineral properties at mantle condition using Deformation multi-anvil apparatus

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**Abstract**

This paper reports recent studies of the rheological and viscoelastic properties of minerals at mantle pressure and temperature using Deformation multi-anvil apparatus (D-DIA). Stress–strain–time relations were measured using synchrotron X-ray radiation to determine these properties. Rheological properties of San Carlos olivine were measured at pressure up to 10 GPa and indicated that the effect of pressure on the viscosity of olivine is much smaller than previous reported. The unique capability of synchrotron X-ray can resolve the stress heterogeneity within a polycrystalline material as well as within a multi-phase mixture and elucidate the stress–strain distribution in an aggregate. Anelasticity measurements were carried out using olivine as the sample at mantle pressure and temperatures. The results showed that grain boundary activity rather than pressure dominate the relaxation processes. The aim of this paper is to illustrate the methods using new tools for high pressure research.


*Keywords:* High pressure; Synchrotron X-ray; Multi-anvil; Plasticity; Anelasticity

## 1. Introduction

In order to understand the structure and dynamics of the Earth, one has to understand the various mechanical properties of the rocks of which the Earth is made. Two typical mechanical properties are rheological and viscoelastic properties. The frontier has been defined by the bringing together of new tools to study these properties at high pressure and temperature.

The flow of solid mantle is responsible for many phenomena on the Earth such as plate tectonics, deep Earthquake and mantle convection. It motivates the understanding of the rheological properties of minerals at elevated pressure and temperature. Small strain postglacial rebound data were used to infer flow properties of the mantle but are limited to ~1000 km [1,2] and cannot account for the large strain mantle convection [3]. The geoid data resolve radial viscosity variation in the deep mantle [4] but lack uniqueness [5,6]. Laboratory studies can provide information about the flow mechanism and flow law of the materials at mantle conditions. Thus, high-pressure rheological measurements are very important.

Viscoelasticity relates to all minerals since they all have an anelastic component, defined by a quality factor $Q^{-1}$. One manifestation of viscoelasticity in the Earth is seismic attenuation and dispersion. Interpretation of seismic attenuation in the earth's mantle is key to the understanding of present-day global dynamics, as attenuation depends strongly on temperature, therefore provides complementary information to that given by elastic velocities [7]. Anelasticity defines the time-dependent stress–strain response of a material system [8]. Using the notation of Nowick and Berry [9], relaxed and unrelaxed elastic modulus, referring to elastic properties at zero and infinite

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frequency, can be significantly different due to anelastic relaxation. The relaxation process can be caused by numerous physical mechanisms [10]. Common mechanisms are the reversible movement of the point defects in the lattice, dislocations and grain boundaries. Attenuation can also be caused by phase transitions. In a pressure–temperature region where the high-pressure phase and low-pressure phase coexist, a small perturbation of pressure can induce some of the low-pressure phase to transform to the high-pressure phase and results in softening of the bulk modulus [11]. Attenuation can also result from domain wall motion in ferroic materials such as perovskites. Being close to the phase boundary introduces phenomena such as soft vibrational modes, which can cause some shear velocity to become very low or even to vanish [12], or high attenuation of acoustic waves as domain wall boundaries become very mobile [13], specifically at long time scales (mHz–Hz). These effects could produce significant signals on the seismic record if present in the Earth. Furthermore, dispersion, the frequency dependence of acoustic velocity, bridges the laboratory data measured at high frequencies (MHz–GHz) to the seismic waves at lower frequencies (Hz–mHz).

Both rheological and viscoelastic studies are among the most challenging of the experimental fields that strive to inform us about the Earth. These time-dependent properties, mainly governed by the behaviors of the defects, do not solely depend on the thermodynamic state of the material. Not only pressure, temperature and chemical potentials affect these properties, but also the history of the sample. Deformation history produces samples with a grain size, texture and dislocation density. Successes have come in the past five years that have enabled the measurement of time-dependent properties using synchrotron X-rays to define the strain and stress in the sample in a multi-anvil high-pressure system.

A series of breakthroughs have enabled this new technology. They include

(1) Use of a DIA – a cubic multi-anvil high-pressure device – in conjunction with a synchrotron source that enables X-ray analysis of the sample [14].
(2) Development of D-DIA for deformation experiments [15,16].
(3) Analysis of stress using X-ray diffraction [17].
(4) Analysis of strain from X-ray images [18,19].
(5) Use of X-ray transparent anvils in the multi-anvil system in order to obtain the necessary diffraction data for stress analysis [20,21].
(6) The understanding of the effect of plasticity on X-ray stress measurements [22,23].
(7) Implementation of conical slits to allow white energy-dispersive or two-dimensional detectors to allow monochromatic angle-dispersive measurements [20–22].

In the traditional deformation experiment, the average differential stress is derived from force per unit area with appropriate corrections for friction between the point of measurement and the sample. The strain is derived from the displacement of the pistons acting on the sample with appropriate corrections for length changes in the parts other than the sample, then the relative displacement of the two ends of the sample is defined. At pressure higher than 3 GPa, sample size becomes very small and friction is too large, these protocols are very difficult to implement. The strategy changes with the aid of an intense X-ray source. Stress is monitored by X-ray diffraction, which defines the elastic changes in lattice spacing. Strain rate is defined by X-ray radiographic images defining sample length with time. In this new measurement protocol, the stress and strain are measured in the sample directly, with no need for corrections for friction or length changes in soft parts of the cell assembly.

The concept of previous $Q$ studies at low pressure (<200 MPa) [24–29] is to force a sinusoidal strain onto an assembly consisting of the sample and a reference material that are mechanically in series. The phase delay of the sample strain relative to that of the standard and the amplitude ratio of the sample to the standard strain are then used in the interpretation. Then $Q^{-1}$ is the tangent of the phase lag, and $G(\omega)$ is the amplitude ratio times the elastic modulus of the reference material. The high-pressure $Q$ measurement has a similar concept as the above. The difference between the high–low pressure experiments is the method of measuring strain. The strains are measured from X-ray radiographic images.

## 2. Methods

### 2.1. General description

The experiments were performed using the D-DIA, located at the National Synchrotron Light Source (NSLS, United States) X17B2 beam line. As shown in Fig. 1, samples were placed in a boron nitride capsule that insolated them from a graphite furnace. The furnace, surrounded by an alumina sleeve, was inserted inside an amorphous boron and epoxy (or mullite) pressure medium. Differential stress was produced in the specimens with hard alumina pistons placed on both ends of the specimens. Samples were separated from each other or from the alumina pistons by metal foils (such as Ni, Re, Pt, Au) that were used as strain markers for measuring the total strain (elastic and plastic strain) of the specimens. A W3%Re–W25%Re thermocouple can be placed in one of the alumina pistons touching one end of the samples or placed from the side entry touching the side of the sample. A solid-state multi-detector (energy-dispersive spectrometer, EDS) together with a conical slit was used for elastic strain measurement (Fig. 2). In order to properly collect the X-ray beams diffracted by the samples at a $2\theta$ angle of 6.5°, cubic boron nitride anvils and sintered diamond anvils, which are transparent to X-rays, were used.

During the experiment, the top and bottom rams as well as the main ram in the D-DIA (Fig. 3) are driven at a specific speed (oil flux) to maintain both constant pressure and