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Macroscopic Stress Measurements by X-ray Diffractions

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Introduction: Understanding dynamic phenomena of the Earth's mantle, such as deep-focus earthquakes and mantle convection, requires detailed knowledge of rheological properties of the Earth's materials at the mantle pressure and temperature conditions. Numerous efforts have been made to gain the knowledge by using a traditional (stress-strain gauge) high-pressure deformation device [Karato et al., 1986; Kohlstedt et al., 1995; Mackwell et al., 1985; Paterson et al., 1982]. In addition to the uncertainty of friction correction in the device, increasing the confining pressure (< 3GPa) has been a major difficulty in advancing the technique. A higher confining pressure is achieved in a modified deformation experiment by recovering a strain-mark for strain measurements[Karato and Rubie, 1997]. However, it leaves the stress unknown. Marriage of high-pressure in-situ x-ray diffraction and strain/size measurement by diffraction peak width analysis [Weidner, 1998] has opened a new window to study the rheological properties of minerals at high pressure and temperature by measuring microscopic stress. We recently explored the possibility of measuring macroscopic stress through x-ray diffraction along different diffraction vectors.

Methods and Materials: materials in a stress field will strain in response to the stress following the Hooke's law:

$$\varepsilon_{ij} = \sum_{k=1}^{3}\sum_{l=1}^{3} s_{ijkl}\sigma_{kl}$$

where ε_{ij} is the strain tensor (ε_{ij} represents distortion in the plane containing the *i* and *j* coordinate axes), s_{ijkl} the elastic compliance tensor and σ_{kl} the stress. The position of an x-ray diffraction peak records the spacing of the specific lattice planes that are oriented parallel to the diffraction vector, which bisects the incident and scattered vectors. The macroscopic strain for a particular diffraction vector is given by

$$\varepsilon_{11}(hkl) = \delta d_{hkl}/d_{hkl}$$

with the direction *1* parallel to the diffraction vector. Under deviatoric macroscopic stress, the spacing of the specific lattice planes varies in response to the stress field. For example, a simple uniaxial stress will contract the lattice planes that are oriented perpendicular to the loading axis and expand those parallel to the axis. Therefore, the powder diffraction Debye rings will become distorted from ideal circles (Figure 1). Varying the orientation of the diffraction vector in all possible directions, we can map out the entire stress field. However, geometric restrictions of the x-ray optics and high-pressure apparatus often severely limit the orientations for x-ray access to the sample. For a stress field with cylindrical symmetry (*i.e.* $\sigma_1 \neq \sigma_2 = \sigma_3$, in Figure 1), the minimum number of orientations to sample the stress field is two. In the case of energy dispersive diffraction, the two detectors should be placed at different ψ angles (with a few exceptions).

Results: Proof-of-concept experiments have been carried out in both energy dispersive mode and angle dispersive mode. In energy dispersive mode, a multi-elements solid state detector (SSD) is used. Diffraction patterns are simultaneously collected along the horizontal and vertical vectors. In angle dispersive mode, full diffraction Debye rings are collected by using a two-dimensional detector (CCD/IP). Difference in d-spacings between horizontal and vertical diffraction data are observed, which can be used to derived the macroscopic stress in the sample.

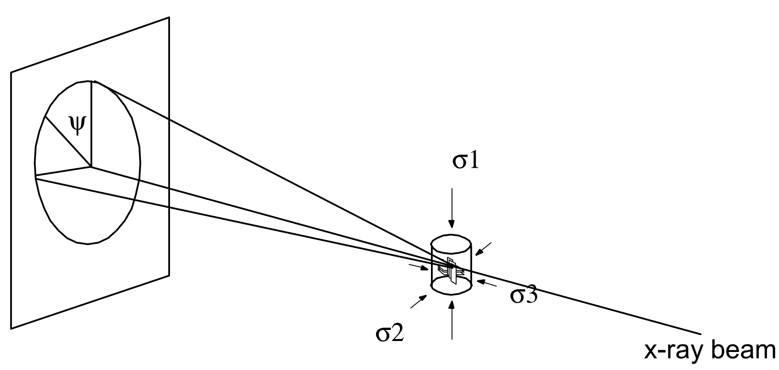

Figure 1. Illustration of Debye rings for diffracted x-rays. A macroscopic stress field will distort the circles.