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Deformation-induced crystallographic-preferred orientation of hcp-iron: An experimental study using a deformation-DIA apparatus

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ABSTRACT

Shear and uniaxial deformation experiments on hexagonal close-packed iron (hcp-Fe) was conducted using a deformation-DIA apparatus at a pressure of 13–17 GPa and a temperature of 723 K to determine its deformation-induced crystallographic-preferred orientation (CPO). Development of the CPO in the deforming sample is determined *in-situ* based on two-dimensional X-ray diffraction using monochromatic synchrotron X-rays. In the shear deformation geometry, the $\langle 0001 \rangle$ and $\langle 11\bar{2}0 \rangle$ axes gradually align to be sub-parallel to the shear plane normal and shear direction, respectively, from the initial random texture. In the uniaxial compression and tensile geometry, the $\langle 0001 \rangle$ and $\langle 11\bar{2}0 \rangle$ axes, respectively, gradually align along the direction of the uniaxial deformation axis. These results suggest that basal slip $(0001)\langle 11\bar{2}0 \rangle$ is the dominant slip system in hcp-Fe under the studied deformation conditions. The P-wave anisotropy for a shear deformed sample was calculated using elastic constants at the inner core condition by recent *ab-initio* calculations. Strength of the calculated anisotropy was comparable to or higher than axisymmetric anisotropy in Earth's inner core.

1. Introduction

After the first proposals by Morelli et al. (1986) and Woodhouse et al. (1986), the presence of anisotropy and heterogeneity in Earth's inner core has been established based on seismic observations. The most general feature of the inner core is its axisymmetric anisotropy with respect to polar direction, in which a P-wave propagating along the polar direction is ~3% faster than that along the equatorial direction (e.g. Creager, 1992). Recent studies reveal more detailed structures of the inner core, showing that the top-most ~50−150 km is nearly isotropic, the inner-most inner core (radius of ~300–600 km) has distinct anisotropy, and the anisotropy in the western hemisphere is greater than that in the eastern hemisphere (for a review, see Souriau and Calvet, 2015). Many hypotheses have been proposed regarding the origin of the complex inner core anisotropy and heterogeneity. However, there is still no clear consensus, even for the most general features of axi-symmetric seismic anisotropy with respect to polar direction (e.g. Sumita and Bergman, 2007; Lasbleis and Deguen, 2015; Romanowicz et al., 2016).

Earth's inner core most likely consists of hexagonal close-packed iron (hcp-Fe) (e.g. Tateno et al., 2010), and the seismic anisotropy in the inner core is believed to be due to the crystallographic-preferred orientation (CPO) of hcp-Fe (e.g. Sumita and Bergmann, 2007). Plastic deformation of hcp-Fe (e.g. Wenk et al., 2000a) and anisotropic growth of hcp-Fe from liquid iron alloy (Bergman, 1997) are possible CPO formation processes in the inner core, and the various candidates for the driving force of the plastic deformation in the inner core include thermal convection (Jeanloz and Wenk, 1988; Wenk et al., 2000a), anisotropic growth of the inner core (Yoshida et al., 1996), Lorentz force (Karato, 1999; Buffet and Wenk, 2001), and Joule heating (Takehiro, 2010). These possible models of formation of the inner core anisotropy have been tested by computer simulations. However, no clear conclusion has been obtained due to the lack of accurate information regarding the physical properties of hcp-Fe, such as its dominant slip system and elastic constants under the inner core condi-

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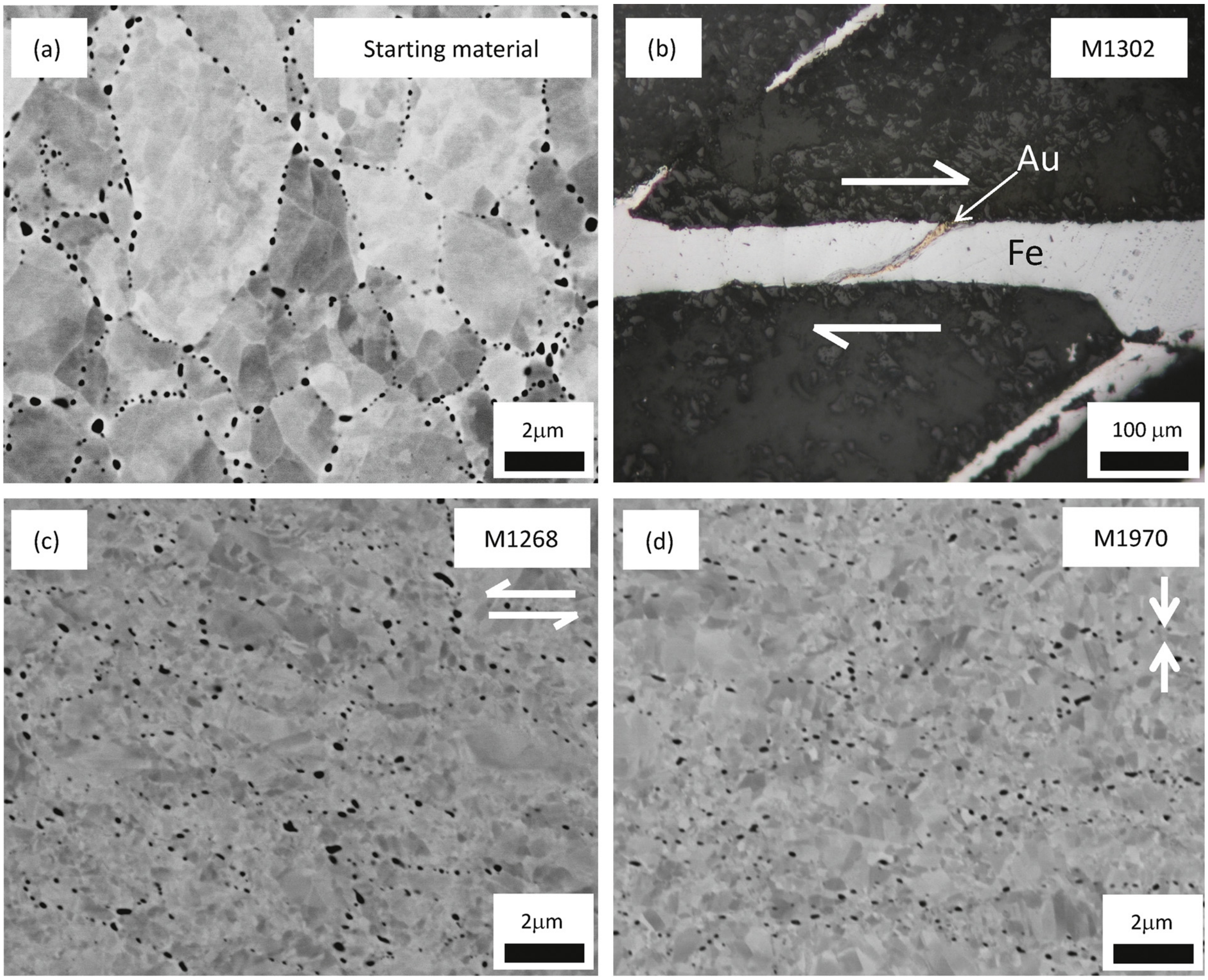

Fig. 1. (a) Back-scattered electron image (BEI) of the starting material, namely Fe. (b) Optical microscopic image and (c) BEI of samples recovered from shear deformation experiments of Fe (b, M1302; c, M1268). (d) BEI of the sample recovered from the uniaxial deformation experiment of Fe (M1970).

tions (e.g. Sumita and Bergman, 2007; Lasbleis and Deguen, 2015; Romanowicz et al., 2016).

It has been established that the dominant slip system in hcp metals generally depends on the axial ratio (c/a) through the switch of close-packed planes, where the principal slip systems are basal slip and prismatic slip for those with $c/a > 1.63$ (e.g. Zn, Cd) and <1.63 (e.g. Hf, Ti), respectively (e.g. Wang and Huang, 2003). However, the estimation of the dominant slip system in hcp-Fe is not straightforward because there are some exceptions to the above general relationship, and hcp-Fe has a c/a value close to 1.63. Thus, experimental investigations are necessary to understand the dominant slip system in hcp-Fe.

Many experimental studies have been carried out on deformation-induced CPO of hcp-Fe, and the operating slip system(s) has been explored (Wenk et al., 2000b; Merkel et al., 2004, 2012, 2013; Miyagi et al., 2008). In these previous studies, the CPO of hcp-Fe in the uniaxial deformation was measured using a diamond anvil cell (DAC) (Wenk et al., 2000b; Merkel et al., 2004, 2013; Miyagi et al., 2008) or a deformation-DIA apparatus (D-DIA) (Merkel et al., 2012), and was compared with simulated CPO patterns assuming strengths of possible slip systems (e.g. viscoplastic self-consistent (VPSC) simulation). These results consistently show that basal slip is the dominant slip system at room temperature, but the influence of temperature on the dominant slip system in hcp-Fe is not clear because of the successive phase transformations during deformation (Miyagi et al., 2008; Merkel et al., 2013) or relatively limited temperature in experiments (Merkel et al., 2012). Since the geometry of deformation has only been studied under uniaxial compression in previous studies, numerical modeling with some assumptions was necessary to interpret the active slip systems. On the other hand, deformation with other geometries, such as uniaxial tension and simple shear, can provide more insight for the understanding of slip system activity in hcp-Fe.

In this study, we conduct deformation experiments of hcp-Fe using a D-DIA with a variety of deformation geometries, simple shear, uniaxial compression, and uniaxial tension, at a pressure (P) of 13–17 GPa and a temperature (T) of 723 K; we measure developing CPOs *in situ* during deformation. In this study, we attempt to specify dominant slip systems directly from experimental CPOs by deformation with various geometries.

2. Experimental procedures

2.1. Sample preparation and cell assembly

The deformation experiments are conducted using a pre-sintered aggregate of iron. The polycrystalline aggregates are prepared by a sintering reagent of Fe sponge (99.9% purity, Wako Pure Chemical Industries) at pressure and temperature conditions of $\sim$1 GPa and 873 K, respectively, using the Kawai-type multi-anvil apparatus installed at the Geodynamics Research Center, Ehime University. This sintering conditions are within the stability field of the bcc structure of iron. Fig. 1a shows the microstructure of the sintered iron specimen. Black dots in the figure represent iron