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DIFFRACTION PROFILE ANALYSIS OF OLIVINES IN THIN SECTIONS OF METEORITES BY THE MICRO-REGION LAUE METHOD USING SYNCHROTRON RADIATION

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Abstract: The lattice distortion of minerals included in a thin section of meteorites was measured by a newly developed X-ray technique for micro-region analysis (8 μm diameter) using synchrotron radiation. This technique using the Laue method with an imaging plate (IP) as a two-dimensional detector, as well as a micro-pinhole, was applied to olivine grains in two L6 chondrites (Y-7304 and Y-7305). The orientation matrix calculated from one end of the full width at half maximum (FWHM) along the elongated diffraction profiles is different from that based on the other end of the FWHM. Rotation about a certain axis equates one matrix to the other. The rotation angles for the Y-7304 and Y-7305 olivines were calculated to be 0.83 and 0.15 degrees, respectively, and are compatible with the FWHM of diffraction profiles. The results of the present X-ray study correspond quantitatively to the wavy extinction observed through an optical microscope. This method will be applied to analyze the lattice distortion of minerals caused by shock effects in meteorites.

1. Introduction

The micrometer-region of a sample in a thin section has been observed by an optical microscope (OM) for identification of minute mineral inclusions and for texture analysis, and by an electron probe micro analyzer (EPMA) for chemical composition. Also, a structure image and its mutual relation to other minerals have been observed by transmission electron microscopy (TEM). TEM has played an important role in the field of material science as well as earth sciences. However, TEM has not provided quantitative crystallographic information on these crystals. Meanwhile, on a single crystal more than 100 μm in size, many precise structural parameters such as the coordinates of atoms, thermal vibrations, and site occupancies have been obtained by X-ray diffraction, and accumulated as a data base.

Without removing a small part of a sample from a thin section, it is essential to refine the structure of minerals in the thin section and to analyze the crystallographic relationship between these minerals in almost the same region as the limit of OM or EPMA. For quantitative analysis of the distortion imposed on minerals which may be induced by shock effects, it is also important to analyze X-ray diffraction profiles of minerals in a thin section or a limited micro-region of crystals by single crystal X-ray diffraction rather than by microscopic observation.

A system for this purpose has been developed, based on that used to analyze a submicrometer-sized single crystal attached to a very fine glass fiber, by making use of polychromatic synchrotron radiation (SR) with the Laue method at beamline 4B (BL-4B) of the Photon Factory (PF), KEK (OHSUMI *et al.*, 1991; OHSUMI *et al.*, 1992).

The aim of this report is to describe the successful application of this system to profile analyses of olivines in thin sections of ordinary chondrites.

2. Equipment and Computer Software

It is important to reduce the background to as low a level as possible in obtaining the diffraction profile from a micro specimen. The diffraction equipment is placed in a vacuum chamber to avoid air scattering. The micro-region in a thin section should be kept stationary with respect to the incident X-ray beam while data collection is carried out, otherwise the micro-region that is irradiated by the X-ray beam changes. Because of the long time needed for collection of diffracted intensities, and the diffraction geometry required for a stationary thin section, the Laue method was employed with an imaging plate (IP; Fuji Co., Ltd.), which is a two-dimensional detector coated by storage phosphers.

Taking into account the limited space around the BL-4B, we produced a micro-beam at the sample position by using a micro-pinhole set just after the optical system which was installed in the beamline and the diffraction apparatus (OHSUMI

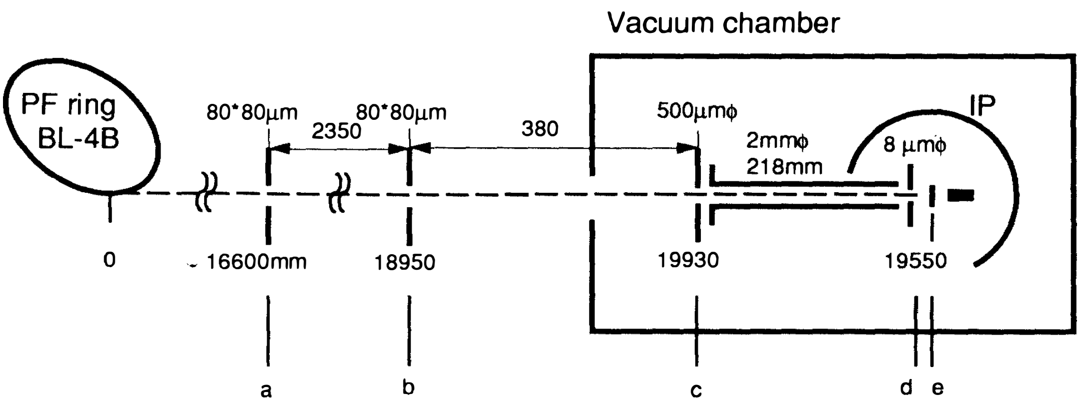

Fig. 1. A schematic drawing of the layout of an optical system and a diffraction equipment. The PF ring is a storage ring for SR experiments. Letters a, b and c denote first and second slit and third pinhole, respectively. A micro-pinhole is shown as d. Sample of thin section and imaging plate are indicated by e and IP respectively. The figures below the horizontal dotted line are distances from the source point.

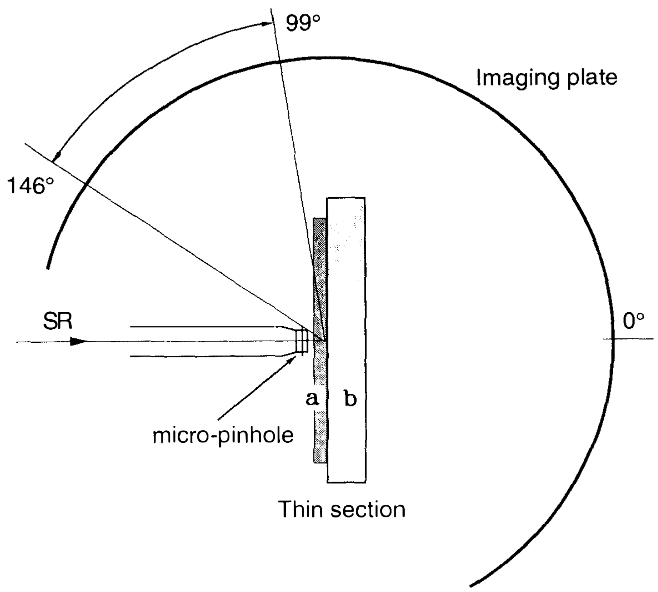

Fig. 2. A diagram showing the relation among the thin section, micro-pinhole and detector. a and b indicate sample and glass, respectively.

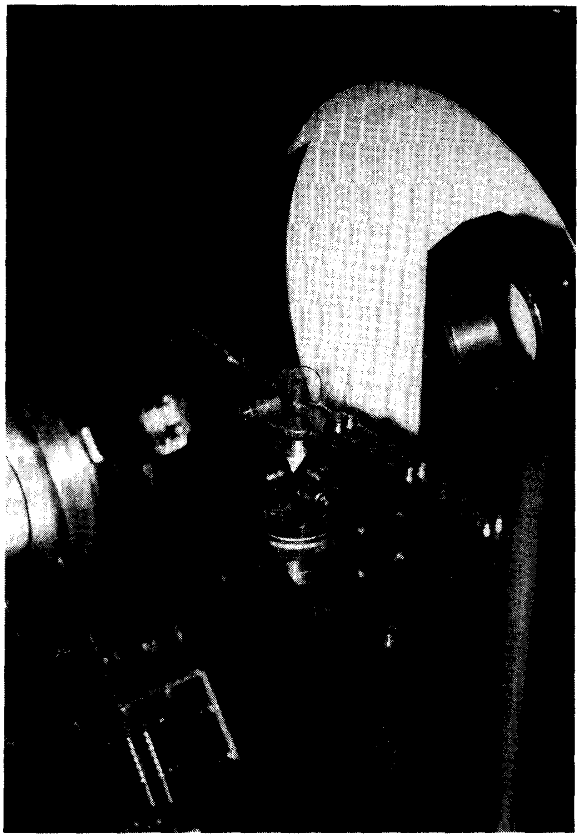

Fig. 3. A photograph showing an experimental setup of a thin section, micro-pinhole and imaging plate (IP).

et al., 1991). Two micro-pinholes with diameters of 5 and 8 μm were prepared. The micro-region in a thin section is observed and adjusted to the SR beam position by OM with a CCD (charge coupled device) mounted on the diffraction equipment.

The Laue pattern is recorded on an IP from −60 to 165 degrees in the two-theta range with a cylindrical camera radius of 100 mm. However, the Laue pattern in the range of −60 to 99 degrees cannot be used for analysis because the diffraction pattern in this area is attenuated by the glass slide of the thin section and also disturbed by scattering and diffraction from the edge of the micro-pinhole. A schematic diagram of the experimental layout is shown in Figs. 1 and 2, and a photograph in Fig. 3.

After taking a Laue pattern, the IP moves to the readout position and is read by the system using a laser. This procedure is carried out in the vacuum chamber, and the Laue pattern is shown on a monitor outside the X-ray shield hutch.

A computer software system which was developed for analysis of sub-micrometer-sized single crystals (HAGIYA *et al.*, 1994) has been revised and used for data reduction of Laue patterns obtained using polychromatic SR. The orientation of the sample with respect to the coordinate system of the equipment is calculated based on the positions of Laue spots on the IP using indices and cell dimensions which can be obtained even by the Laue method, by a proposed method (CARR *et al.*, 1993). If there is more than one domain of the same species in a micro-region, the relative orientations of these domains are calculated in terms of the rotation angles which bring one orientation to others about certain axes.

3. Experimental

After routine alignment of a fine SR beam ($80\times80\,\mu$m) to the diffraction apparatus, a micro-pinhole of 8 μm diameter set on a goniometer head was placed just after an X-ray collimator, and aligned to the incident fine SR beam by observing the signal from an ion chamber. The uniformity of the beam at the sample position was confirmed by an image recorded on a film (Radcolor film, Nitto Elec. Indust. Co., Ltd.). The actual beam size was measured using the signal from an ion chamber, a slit on the front side was scanned stepwise in the vertical direction (Fig. 4). This measurement yielded a beam diameter of about 8 μm.

Two L6 ordinary chondrites, Yamato(Y)-7304 and -7305 with different shock-induced textures (YANAI and KOJIMA, 1987), were selected for the present profile analysis. Two Laue patterns of olivines included in these L6 chondrites were taken with 25 minute exposures, with the storage ring current at around 250 mA and a ring energy of 2.5 GeV. The diffraction equipment was evacuated to 2×10^{-2} Torr during the experiment.

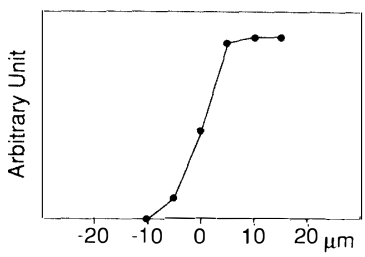

Fig. 4. Intensities of incident SR beam measured at 140 mm down stream from the sample position by scanning an ion chamber with a slit. From this figure the FWHM of the incident SR beam size at the sample position was determined as 8 μm in diameter.

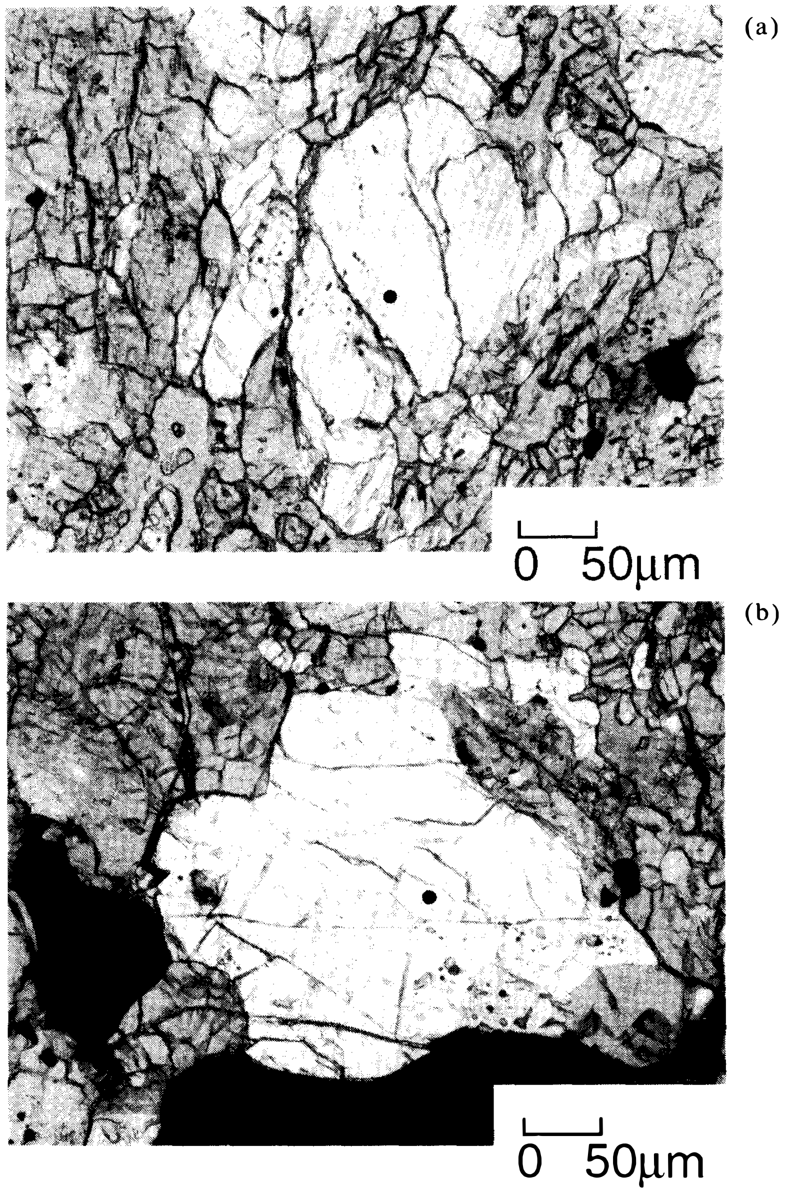

Fig. 5. Micro-regions with a diameter of 8 μm on both samples irradiated by a micro-SR beam are shown in dots in photographs, (a) Y-7304, (b) Y-7305. An identification of olivines and the positions of irradiated micro-regions were confirmed by an optical microscope attached to the diffraction equipment.

The irradiated micro-regions (8 μm) of both olivine grains are shown in Fig. 5. Typical Laue spots obtained from olivines are shown in the contour maps in Fig. 6.

4. Results and Discussion

The Laue spots recorded on the IP were indexed by comparing observed and calculated interplane angles of the crystal planes using a computer program. The orientation of the olivine grains was then determined for each sample and represented in the form of an orientation matrix. The orientation was then refined by a

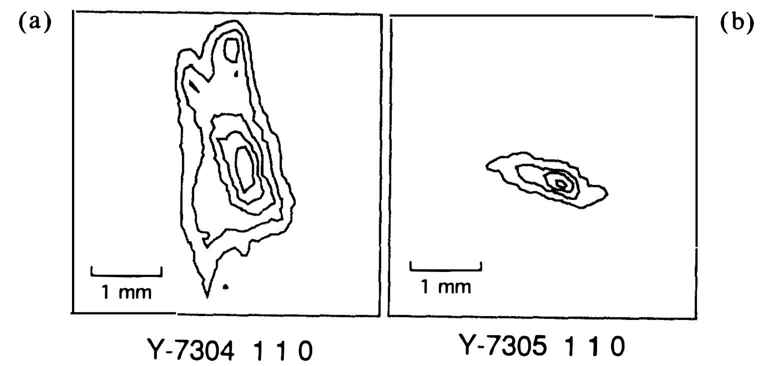

Fig. 6. Diffraction profiles recorded on IP in contour plots, horizontal and vertical lines indicate the x- and y-axis of the IP respectively. A scale bar indicates 1 mm on the IP. (a) Y-7304, (b) Y-7305.

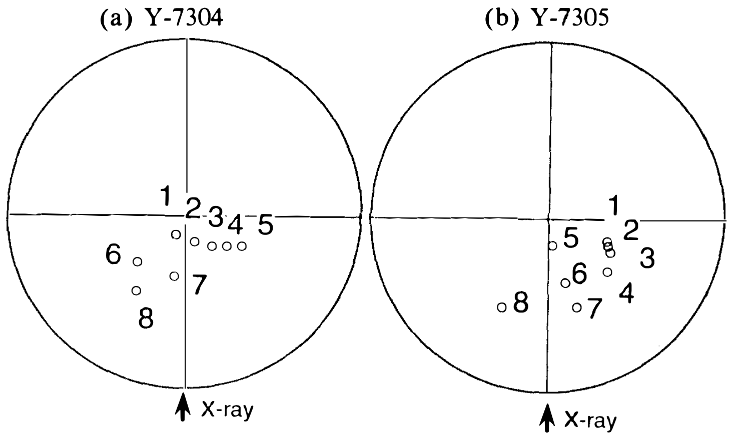

Fig. 7. Distribution of Laue spots used for profile analyses shown in stereo projection, (a) Y-7304, (b) Y-7305. Locations of diffraction spots shown in Figs. 6a and b are indicated as 2 and 4 in these figures revpectively.

least-squares method using eight indexed Laue spots with sample position and camera radius as parameters. At this stage, the center of the diffraction profiles was used for the positions of Laue spots. The distributions of observed Laue diffraction spots in reciprocal spaces with respect to the incident SR beam are shown in stereo projection in Fig. 7 for each olivine. It is seen from this figure that both orientation matrices are obtained based on almost the same ratio of whole reciprocal spaces, and then they are compatible with each other.

Elongation of the diffraction profiles observed on the IP may be considered to be caused by distortion of the crystal lattice. In order to evaluate the degree of lattice distortion, consider each elongated diffraction profile to consist of two non-distorted lattices, one of which gives a diffraction spot on one end of the full width at half maximum (FWHM) along the elongation, and another on the other end. The orientation matrices calculated at both ends of the FWHM of the profile are different from each other. Let's consider a rotation operation about some axis in order to equate one matrix with the other. For this procedure, each orientation matrix was refined using eight Laue spots shown in Table 1. The rotation angles for

Table 1. FWHMs of Laue diffraction with indices.

Y-7304						Y-7305					
No.	h k l	FWHM (deg.)	No.	h k l	FWHM (deg.)	No.	h k l	FWHM (deg.)	No.	h k l	FWHM (deg.)
1	3 2 0	1.4	5	1 2 0	1.2	1	5 3 0	0.19	5	2 1 1	0.31
2	1 1 0	1.4	6	3 1 1	1.4	2	3 2 0	0.25	6	5 5 2	0.25
3	3 4 0	1.3	7	4 5 1	1.0	3	4 3 0	0.30	7	3 4 1	0.30
4	3 5 0	1.3	8	2 1 1	1.5	4	1 1 0	0.25	8	3 5 3	0.19

Y-7304 and -7305 olivines are then calculated to be 0.83 and 0.15 degrees respectively.

FWHMs used for obtaining orientation matrices are shown in Table 1. Depending on the diffraction geometry, the operation angles (θ) make at most double of these angles (2θ) in the FWHMs shown in Table 1. FWHMs in Table 1 are within these values (2θ) for both olivines. This fact indicates that the analyses of both orientation matrices and FWHMs of diffraction profiles were done adequately and are compatible with each other.

A wavy extinction effect of the same olivine in Y-7304 was observed under OM. In the $30\times30\,\mu$m area around the micro-area ($8\,\mu$mϕ) which was irradiated by the incident SR beam, the extinction effect occurring at the $8\,\mu$m area of the grain moved to the next when the stage was rotated by about 0.5 degrees. On the other hand, the wavy extinction was not observed in Y-7305. On this scale, however, it is difficult to measure the exact extinction angle by visual observation through OM. According to the scheme of DODD and JAROSEWICH (1979), both olivines in Y-7304 and -7305 were classified into d and b respectively.

Both the FWHMs of the diffraction profiles determined by the X-ray diffraction method and the distortion angles of the lattices are compatible with the angles measured by OM. The results of the present study correspond to the angle divergences of extinction whole over the olivine domains.

For quantitative evaluation of the wavy extinction effect of minerals in a thin section, the present X-ray diffraction method was proved to be successful and provided the degree of extinction in terms of angle of lattice distortion. In order to do quantitative studies of the induced shock effects in minerals, this method should be combined with a study to clarify the relation between degree of shock effect retained in the minerals and that of the imposed shock. The study to evaluate the shock effect provides the shock histries of meteorites on their parent bodies or in the space.

The Laue method with SR is further expected to apply to the micro-regions of thin sections of meteorites for identification of trace minerals, for refinement of the crystal structures and for determination of the mutual orientation of the included minerals such as lamerae.

Acknowledgments

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