CHARACTERIZATION OF TINY FRAGMENTS OF STONY METEORITES BY X-RAY DIFFRACTION USING GANDOLFI ATTACHMENT.

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Introduction: The canonical X-ray diffraction method is traditional but powerful to characterize crystals [1, 2]. However, the application to extraterrestrial materials has been much limited, compared to other common analytical techniques (e.g., μRaman, EPMA, FE-SEM, TEM, and SIMS). This may be partly because the facilities of synchrotron radiation is used to such analyses. However, recent advances on X-ray diffractometer (e.g., optics, detector, and goniometer) make possible to characterize much more conveniently, for example, powder diffraction pattern is obtained from the in-plane rotation of polished thin section [3-5]. This method enables to determine the chemical group of ordinary chondrites and petrologic type to a certain extent [3] and the metamorphic sequence for the CO3 chondrites in detail [4, 5]. In the present study, we characterized a tiny grain of ordinary chondrites for a new nondestructive classification using the Gandolfi attachment combined with the focusing optics producing intense X-ray.

Measurement condition: The X-ray diffractometer (RIGAKU, SmartLab) was used for the present study, installed at NIPR in 2014. Cu $K\alpha_1$ (λ=0.15406 nm) X-ray was used. X-ray tube voltage was 40 kV, and tube current was 30 mA. One-dimensional solid-state detector (D/teX Ultra 250) was used for the X-ray counting. The scan speed of the goniometer was normally 0.2 º/min and ten repeated analyses were summed. The polycapillary focusing optics (CBO-f unit) [6] through multi-layer film mirror for incident X-ray was used to obtain intense X-ray beam (~0.4 mm). The Gandolfi attachment was used for the goniometer head, and obtained the powder diffraction pattern, by the revolution rate at 20 rpm and the rotation rate at 89 rpm. The centering of the sample for rotation was adjusted using the designated optical microscope. The incident beam position was fixed and the diffracted beam was scanned in the range of 2θ = 28-50º. The measuring time was then ~20 hr per one sample.

Samples: Antarctic ordinary chondrites were mainly analyzed (3 H3, 5 H4, 3 H5, 2 H6, and 1 LL5 at this moment). In order to prepare the sample, the tungsten carbide needle (the tip diameter 5 μm) was used (MICROSUPPORT). The samples of 0.1-0.3 mm in size were taken out from a tiny interior fragment of Antarctic meteorites (normally a few mm in size) in the polypropylene capsule. The sub mm sample was attached using thermal wax with main ingredient of ethylene glycol (PMC adhesive wax, MARUTO INSTRUMENT) on the needle glass (the tipe diameter ~10 μm), divided from the glass rod of 1mm in diameter (G-1000, NARISHIGE) into two using cigarette lighter. The identical method was applied to synthetic forsterite in order to correct the 2θ values.

Analytical method: The diffractions of olivine and low-Ca pyroxenes were mainly focused and the method has been reported [3, 4]. The mean compositions of olivines were determined from the 2θ of the olivine 130 peak [7] and olivine 130 correlates with the chemical group for EOCs. Combining the common peak at 2θ = ~31º of two polymorphs for low-Ca pyroxene (Opx and Cpx), they were distinguished from the peak of Opx 511 at 31.6º since Cpx and high-Ca pyroxene are absent from both peaks. The peak position and FWHM were analyzed using the RIGAKU Data Analysis software (PDXL).

Results and discussion: Although the peak integrated intensities of the powder diffractions obtained from the present measuring method are much weaker than those from the bulk measurement using the polished thin section [3, 4], the fayalite number of olivines was obtained from the peak 130 using the equation by [7] and the correction of the 2θ values of the synthetic forsterite [8] gave the nearly consistent values within each chemical group of the chondrites. The peak of the orthopyroxene 511 shows for the equilibrated chondrites but some samples are lacking. The apparent lack of the peak would be because the weak peak is overlapped with the nearest peak (olivine 130) due to the large FWHM. The measured rounded grain of the H3 chondrite (Y-791113) would be a chondrule consisting of low-Ca clinopyroxene but absent from olivine.

Implication: The present method could be applied to limited and tiny samples such as return sample from space and unmelted micrometeorites, and specific textures of chondrules, matrices, CAIs, AOAs, shock veins and so on extracted from the sections. On the other hand, the size more than ~1 mm is not suitable for the present measurement method but this is suitable for the in-plane rotation method [3].

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