SYNCHROTRON X-RAY DIFFRACTION STUDIES OF OLIVINE FROM COMET WILD 2

K. Ohsumi[1], K. Hagiya[2], T. Mikouchi[3], M. E. Zolensky[1]; [1]KT, NASA Johnson Space Center, Houston, TX 77058, USA; [2]Graduate School of Life Science, University of Hyogo, Koto, Kamigori, Hyogo 678-1297, JAPAN; [3]Department of Earth and Planetary Science, University of Tokyo, Hongo, Bunkyo-ku, Tokyo 113-0033, JAPAN (E-mail: kazumasa.ohsumi-1@nasa.gov)

Introduction and Experimental Methods: We have analyzed a collection of the Comet Wild 2 coma grains returned by the NASA Stardust Mission, using micro-area Laue diffraction equipment. The purpose of the diffraction experiment is to permit the structure refinement of olivine including site occupancies. In addition to the intrinsic importance of the olivine structures for revealing the thermal history of Wild 2 materials, we wish to test reports that olivine recovered after hypervelocity capture in silica aerogel has undergone a basic structural change due to capture heating [1].

The diffraction equipment placed at beam line BL-$4B_1$ of PF, KEK was developed with a micropinhole and an imaging plate (Fuji Co. Ltd.) using the Laue method combined with polychromatic X-ray of synchrotron radiation operated at energy of 2.5 GeV. The incident beam is limited to 1.6 μm in diameter by a micropinhole set just upstream of the sample [2, 3]. It is essential to apply a microbeam to obtain diffracted intensities with high signal to noise ratios. This equipment has been successfully applied to various extraterrestrial materials, including meteorites and interplanetary dust particles [4]. The Laue pattern of the sample C2067,1,111,4 (Fig. 1) was successfully taken on an imaging plate after a 120 minute exposure (Fig. 2).

Crystal Structure Analysis: All Laue spots are well indexed by the traditional cell parameters of olivine. The numbers of observed Laue spots number more than two hundred, which are sufficient to permit a full three dimensional crystal structure analyses for an orthorhombic phase. Structure refinement was carried out by the least-squares method minimizing the residual factor ($R = \Sigma[Io(hkl)\text{-}kIc(hkl)]^2/\Sigma Io(hkl)^2$) with the structural parameters based on the integrated intensities of Laue spots. Here $Io(hkl)$ and $Ic(hkl)$ are observed and calculated intensities of Laue spots, respectively, and k is the scale factor. Summation was made on the indices hkl's of all the observed Laue spots. It is necessary to apply the absorption correction to diffracted intensities in the case of Laue method with polychromatic X-ray, because there are many cases where the longer wave length of X-rays are emitted as the primary indexes of Laue spots in spite of the grain size being in the order of micrometer. However, the absorption correction could not be applied in the present case, since the size and shape of the grain emitted were not certain, and also the location of the grain in the particle was not known. Therefore the refinement was performed by using the intensities of Laue spots emitted with the wavelength shorter than 1.5 Å. The least-squares refinements were converged with the residual factors (R) of 0.0056 for 124 Laue spots. The final parameters of olivine are listed in Table 1 and include site occupancies [Fe/(Fe+Mg)] of two crystallographic independent sites (M1 and M2) as 0.72(2) and 0.69(2). The chemical formula of this olivine is determined to be $(Mg_{0.71}Fe_{0.29})_2SiO_4$. Because chemical analysis of complexly intergrown Wild 2 materials using SEM-EDS or TEM-EDS commonly entails cause beam overlap with the surrounding phases, yielding slightly erroneous compositions, independent analysis using X-ray diffraction techniques can yield the most accurate technique for compositional analysis, as they have done here.

Although this olivine is more Fe-rich than our previous sample of Wild 2 olivine (C2054,0,35,4) with the site occupancies of 0.905(4), 0.894(4), it is within the range of the observed Wild 2 olivine composition [5].

The structure refinement of both olivines were successfully carried out assuming the orthorhombic *Pbnm* cell parameters of a=4.788, b=10.338, c=6.035 Å. The Laue method using polychromatic X-ray by synchrotron radiation directly gives axial ratios, not exact cell parameters. However, the structure factors used in the least-squares refinement of the structure are calculated based on the exact cell parameters. Therefore, the cell parameters of Wild 2 olivine can not be significantly different from typical values of olivine. This result contrasts with reported results from Raman spectroscopy of olivine recovered from aerogel in hypervelocity lab simulations, which suggest quenched unit cell changes from capture heating and shock [1].

References: [1] Foster N.J. et al. (2007) *MAPS* **42**, A51; [2] Ohsumi K. et al. (1991) *J. Appl. Crystallogr.*, **24**, 340-348; [3] Ohsumi K. et al. (1995) *Rev. Sci. Instrum.*, **66**(2), 1448-1450; [4] Ivanov A.V., et al. (2000) *American Mineralogist* **85**,1082; [5] Zolensky M. et al. (2006) *Science* **314**, 1735-1739.

Fig. 1 Optical photograph of Wild 2 sample C2067,1,111,4 with the grain diameter being 15 μm. Irradiated area is indicated by the central white dot.

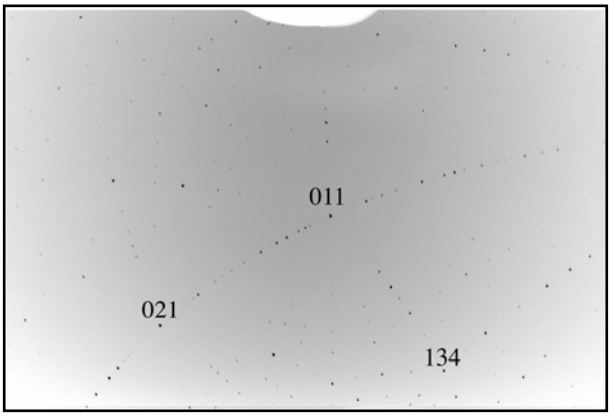

Fig. 2. A partially indexed Laue pattern of sample C2067,1,111,4 obtained by a 120 min. Laue exposure with a ring operation of 2.5 GeV at PF, KEK.

Table 1 Structural parameters of olivine determined by least-squares refinement.

	Mg site occupancy	*x*	*y*	*z*	*B*
(Mg, Fe)1	0.72(2)	0	0	0	0.45
(Mg, Fe)2	0.69(2)	0.983(3)	0.2785(2)	1/4	0.45
Si		0.428(2)	0.0954(3)	1/4	0.49
O1		0.760(6)	0.0905(11)	1/4	0.55
O2		0.229(6)	0.4468(10)	1/4	0.55
O3		0.289(4)	0.1639(4)	0.0321(8)	0.55