SYNCHROTRON X-RAY DIFFRACTION STUDY OF THE COMET WILD 2 PARTICLE (C2054,0,35,4) RETURNED BY THE NASA STARDUST MISSION. K. Hagiya[1], K. Ohsumi[2], T. Mikouchi[3], M. E. Zolensky[4], [1]Graduate School of Life Science, University of Hyogo, Koto, Kamigori, Hyogo 678-1297, JAPAN, [2]Inst. of Materials Structure Science, High Energy Accelerator Research Organization, Oho, Tsukuba, Ibaraki 305-0801, JAPAN, [3]Department of Earth and Planetary Science, University of Tokyo, Hongo, Bunkyo-ku, Tokyo 113-0033, JAPAN, [4]KT, NASA Johnson Space Center, Houston, TX 77058, USA, E-mail: hagiya@sci.u-hyogo.ac.jp.

Experimental Methods: One of the Comet Wild 2 particles (C2054,0,35,4) returned by the NASA Stardust Mission was analyzed by microarea diffraction equipment system. Since the size of the sample is only about 10 μm in diameter (Fig. 1), the Gandorophy method was first applied to this particle at SPring-8 by T. Nakamura of Kyushu University. From the diffraction pattern, it was made clear that there are olivine and pyroxene in this particle [1, 2]. Our purpose of the diffraction experiment is to perform the structure refinement of olivine and pyroxene including site occupancies. The equipment system 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 [3]. The incident beam is limited to 1.6 μm in diameter by a micropinhole set just upstream of the sample. It is essential to apply microbeam for obtaining diffracted intensities with high signal to noise ratios. The Laue photograph was successfully taken on an imaging plate by 30 minutes exposure (Fig. 2).

Analysis: All Laue spots are well indexed by one grain of olivine and two grains of orthopyroxene (Fig. 2). The numbers of observed Laue spots of olivine, the first pyroxene and that of second is 224, 261 and 32, respectively. The axial ratios (a/b and c/b) of each domain were refined based on the positions of Laue spots (Table 1). The average values of the site occupancies [Fe/(Fe+Mg)] of two crystallographic independent sites (M1 and M2) were estimated as 20.2% (a/b) and 7.3% (c/b) according to the Vegard's law shown in Fig. 3. 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 by 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 was 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 successfully converged with the residual factors (R) of 0.0032 for 168 Laue spots of olivine and of 0.0012 for 182 spots of orthopyroxene. The final parameters of olivine and pyroxene are listed in Table 2 and Table 3, respectively. The average value of the refined site occupancies of two sites (M1 and M2) of olivine is compatible with the results obtained from axial ratios. The second orthopyroxene could not be refined because of the small number of observed Laue spots. As the result, the chemical formulae of olivine and orthopyroxene are determined to be $(Mg_{0.89}Fe_{0.11})_2SiO_4$ and $(Mg_{0.90}Fe_{0.10})SiO_3$, respectively. Because chemical analysis of olivine and pyroxene using SEM-EDS or TEM-EDS could cause beam overlap with the surrounding phases (in this case, Fe sulfide) which gives slightly different composition, independent analysis using diffraction technique is important.

References: [1] Brownlee D. E. et al. (2006) *Science, 314,* 1711-1716. [2] Zolensky M. E. et al. (2006) *Science, 314,* 1735-1739. [3] Ohsumi K. et al. (1995) *Rev. Sci. Instrum.*, 66(2), 1448-1450. [4] Ohsumi K. et al. (1991) *J. Appl. Crystallogr.*, 24, 340-348.

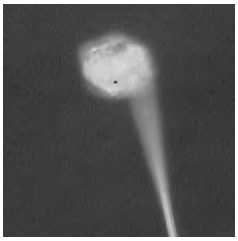

Fig. 1. Photograph of the sample (C2054, 0,35,4). The sample with about 10 μm in diameter was attached to the end of a thin glass fiber. The position of irradiated microarea shown in dot was confirmed by an optical microscope.

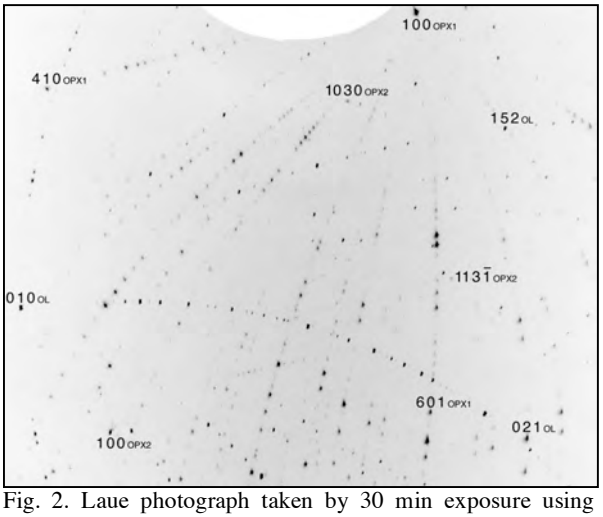

Fig. 2. Laue photograph taken by 30 min exposure using polychromatic X-ray microbeam of synchrotron radiation operated at energy of 2.5GeV. Several Laue spots of olivine and two orthopyroxenes with their indices are shown by subscripts OL, OPX1 and OPX2, respectively.

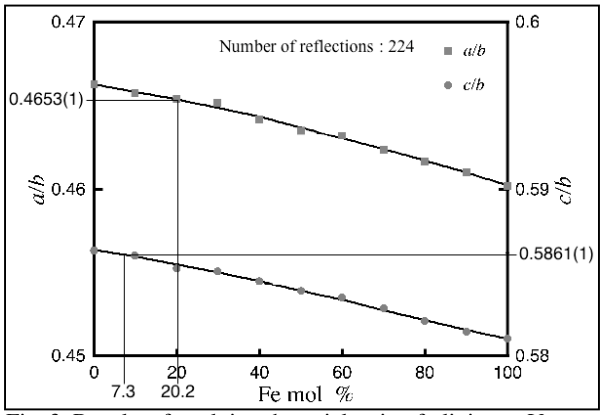

Fig. 3. Results of applying the axial ratio of olivine to Vegard's low. The Fe contents are estimated to 20.2 and 7.3% from the values *a*/*b* and *c*/*b*, respectively.

Table 1. The axial ratio obtained from Laue pattern fitting.

	olivine	orthopyroxene1	orthopyroxene2
a/*b*	0.4653(1)	2.0719(3)	2.0673(12)
c/*b*	0.5861(1)	0.5900(2)	0.5885(4)
No. spots	224	261	32

Table 2. Refined structural parameters of olivine.

	$\frac{Fe}{Fe+Mg}$	*x*	*y*	*z*	*B*
M1	0.10(4)	0	0	0	0.67(12)
M2	0.11(4)	0.989(2)	0.2778(1)	1/4	0.44(11)
Si		0.429(1)	0.0943(1)	1/4	0.49(5)
O1		0.768(3)	0.0918(4)	1/4	0.82(8)
O2		0.228(3)	0.4477(4)	1/4	0.56(9)
O3		0.284(3)	0.1630(3)	0.034(1)	0.77(6)

R = 0.0032 (168 reflections)

Table 3. Refined structural parameters of orthopyroxene.

	$\frac{Fe}{Fe+Mg}$	*x*	*y*	*z*	*B*
M1	0.02(6)	0.3758(2)	0.654(2)	0.868(1)	0.6(2)
M2	0.19(9)	0.3779(2)	0.4834(9)	0.361(2)	0.8(2)
Si1		0.2715(1)	0.3428(10)	0.049(1)	0.53(7)
Si2		0.4737(1)	0.3398(12)	0.798(1)	0.52(7)
O1		0.1828(4)	0.342(3)	0.035(2)	0.56(10)
O2		0.3107(5)	0.503(2)	0.047(4)	1.0(2)
O3		0.3034(4)	0.227(2)	0.172(3)	0.78(11)
O4		0.5636(4)	0.340(4)	0.807(3)	0.75(12)
O5		0.4349(9)	0.488(3)	0.694(6)	1.5(2)
O6		0.4469(4)	0.195(2)	0.593(3)	0.93(12)

R = 0.0012 (182 reflections)