

of Mg or Al between the tetrahedral and octahedral sites is almost constant against Ga content in the $\text{MgAl}_{2-x}\text{Ga}_x\text{O}_4$ solid solution. A compositional variable of the Ga/(Mg+Ga) ratio in the octahedral site is not influenced by the occupancy of Al. The occupancy of Al is independent of the occupancy of Ga, though it depends on the occupancy of Mg according to thermal history. The local Al-O bond length in the tetrahedral site is 0.15 Å longer than the expected bond length. The nature that Al in spinel structure occupies mainly the octahedral site arises from the character of Al itself.

Keywords: spinel, crystal structure, NMR spectroscopy

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Soft synthesis and crystallographic characterization of calcium magnesium mixed carbonates

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It seems to be generally accepted that smithsonite, magnesite, siderite, as well as mixed carbonates like dolomite (MgCaCO_3) or huntite ($\text{Mg}_{0.75}\text{Ca}_{0.25}\text{CO}_3$) have been formed in the nature under hydrothermal conditions. Rao et al. developed a general soft synthesis procedure for obtaining anhydrous carbonates by precipitation from solution at normal pressure. They had success in the synthesis of smithsonite and siderite, but failed in the magnesite synthesis. This finding questions that the hydrothermal synthesis were the only way of genesis of some of these minerals in nature. The scope of this work is to apply the Rao et al. method to the synthesis of double carbonates of general formula $\text{Mg}_x\text{Ca}_{1-x}\text{CO}_3$. The compounds obtained have been characterized by X-ray fluorescence, atomic absorption analysis, TG and X-ray powder diffraction. The results obtained clearly demonstrate that anhydrous double calcium magnesium carbonate minerals can be obtained by soft synthesis for x composition ranging from 0 to 0.7. Hydroxysalts instead of anhydrous salts are obtained for larger values of x. The crystallographic parameters of the anhydrous compounds have been calculated and it has been shown that the volume of the cell accomplishes with the Vegard law.

Keywords: alkaline-earth double carbonates, lattice parameters

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Synchrotron X-ray diffraction studies of two olivines from the comet Wild 2

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Introduction and Experimental Methods: We have analyzed a collection of the Comet Wild 2 coma grains returned by the NASA Stardust Mission. This is the first solid sample return mission since

Apollo 17. 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 [Foster N.J. et al. (2007) MAPS 42, A51]. The diffraction equipment placed at beam line BL4B1 of the Photon Factory, KEK was developed for microdiffraction studies of materials. [Ohsumi K. et al. (1991) J. Appl. Cryst., 24,340 & (1995) Rev. Sci. Instrum., 66(2),1448]. This equipment has been successfully applied to various extraterrestrial materials [Ivanov A. V. et al. (2000) Amer. Min. 85,1082]. Two Laue patterns of the samples (C2054,0,35,4 and C2067,1,111,4) were taken on an IP after 90 and 120 minutes exposures respectively. Structure refinements and Results: All Laue spots of both patterns are indexed by the traditional cell parameters of olivine. Structure refinements were carried out by a least-squares method minimizing the residual factor(R) based on the integrated intensities of Laue spots. The results of the several cycles of least-squares refinements including site occupancies of both samples lead the chemical formula as $(\text{Mg}_{0.89}\text{Fe}_{0.11})_2\text{SiO}_4$ for C2054 and $(\text{Mg}_{0.71}\text{Fe}_{0.29})_2\text{SiO}_4$ for C2067. Success of the refinement assuming the traditional cell parameters implies that the cell parameters of Wild 2 olivine cannot be significantly different from its typical values.

Keywords: synchrotron X-ray diffraction, microcrystallography, mineralogy and crystallography using X-ray diffract

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Olivine from highly oxidized scoria and lava of Kasayama volcano, Hagi, Japan

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Oxidation state and distribution of Fe in olivine, $M2M1ZO_4$, in the andesitic scoria and lava oxidized at high temperature were investigated using $\text{Fe}_{L\beta}/\text{Fe}_{L\alpha}$ -intensity ratio, X-ray Rietveld and ⁵⁷Fe Mössbauer methods. Olivine samples were collected from the reddish black and black scorias, and the reddish black and black Opx-andesites in Kasayama volcano, Hagi, Yamaguchi Prefecture. The Fo contents of olivine in reddish scoria exceeded 91 mol%, while those of reddish black and black scorias are 83-85 and 79-81mol%, respectively. The $\text{Fe}^{2+}:\text{Fe}^{3+}$ ratios in olivine of reddish black and black scoria determined using $\text{Fe}_{L\beta}/\text{Fe}_{L\alpha}$ -intensity ratio are $\text{Fe}^{2+}:\text{Fe}^{3+}=92(3)-93(3):8(3)-7(3)$ and $\text{Fe}^{2+}:\text{Fe}^{3+}=93(3)-98(3):7(3)-1(3)$, respectively. On the other hand, the Fo contents of olivine in reddish black and black lava were 91-99, 79-80 and 73-80, respectively. The $\text{Fe}^{2+}:\text{Fe}^{3+}$ ratios of olivine in reddish black and black lava are $92(3)-99(3):8(3)-1(3)$ and $93(3)-99(3):7(3)-1(3)$, respectively. Mg and Fe occupancies at M1 and M2 in olivine refined by the X-ray Rietveld method ($R_{wp}=4.89$, $R_e=3.79$, $S=1.29$) are $\text{Mg}(M1)=0.835$ (7), $\text{Fe}(M1)=0.165$ (7), $\text{Mg}(M2)=0.830$ (6), $\text{Fe}(M2)=0.170$ (6). The ⁵⁷Fe Mössbauer spectrum of olivine in the reddish black scoria consists of three doublets which are assigned to $\text{Fe}^{2+}(M1,M2)$ and $\text{Fe}^{3+}(M2)$ in olivine and $\text{Fe}^{3+}(M2)$ in laihunite. The $\text{Fe}^{2+}(M1,M2):\text{Fe}^{3+}(M2)$ ratios determined by ⁵⁷Fe Mössbauer method for olivine in the black scoria is $\text{Fe}^{2+}:\text{Fe}^{3+}=69:31$, and the resulting chemical formula of the olivine is $[\text{M}1(\text{Mg}_{0.835}\text{Fe}^{2+}_{0.165})\text{M}2(\text{Mg}^{2+}_{0.830}\text{Fe}^{2+}_{0.053}\text{Fe}^{3+}_{0.117})]_{\Sigma 2.000}\text{Si}_{1.000}\text{O}_4$, although the amount of