Experimental report

03/05/2017

Proposal: 5-11-418 **Council:** 4/2016

Title: A windows into the Earth s mantle:On the crystal structure of hydrous wadsleyite (beta-Mg2SiO4)

Research area: Other...

This proposal is a new proposal

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Samples: beta-Mg2SiO4

Instrument	Requested days	Allocated days	From	To
D9	14	7	07/06/2016	14/06/2016

Abstract:

Wadsleyite (beta-Mg2SiO4) is the high-pressure polymorph of the most common olivine mineral, and it is able to store water as hydroxyl group (OH-) in Earth s mantle transition zone. The crystal structure of wadsleyite was solved and refined in the space group Imma with a=5.8, b=11.9, c=8.4 Å. The presence of H (up to 3.3 wt% H2O) in synthetic wadsleyite was confirmed by IR spectroscopy, SIMS, calculated or experimental electrostatic potentials, difference-Fourier analysis of the charge density from single-crystal X-ray diffraction data, as well as 1H and 17O NMR. Based on the fact that we were able to grow crystals of hydrous wadsleyite of approx. 1 mm3, the aim of the present study is a re-investigation of the crystal structure and crystal chemistry of a (hydrous) wadsleyite at ambient conditions and at low-T (20 K) by means of single-crystal neutron diffraction, in order to define: the location of the proton site(s) and the real topological configuration of the OH-group(s), along with the anisotropic displacement parameters of the H-site(s).

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A windows into the Earth's mantle: On the crystal structure of hydrous wadsleyite (β-Mg_2SiO_4)

The neutron diffraction experiment was conducted on a synthetic crystal of wadsleyite of approximately 1 mm^3, whose chemical analysis (by WDS-EMPA and SIMS) and structural data at room condition (by single-crystal X-ray diffraction) were already available. Infrared spectra showed the presence of hydroxyl group. One data collections (*i.e.*, at room temperature) was performed at a short wavelength using the four-circle diffractometer available at D9 (approx. 0.84 A). The diffraction patterns was successfully indexed with an orthorhombic lattice, with: a = 5.70, b = 11.44, c = 8.26 A. The reflection conditions agreed with the space group: *Imma*. Intensity data were collected up to 2-theta max = 66.14°, with 970 reflections, whose 355 unique with R(int) = 0.0605. The structure refinement was successfully conducted starting the structure model of Sano-Furukawa et al. (2011), without any proton site. The Fig. 1 shows a clinographic view of the wadsleyite structure based on this structure refinement. The refined converged with R_1(F) = 0.0631, 250 reflections with $Fo > 4sig(Fo)$ and 44 refined parameters. A careful inspection of the difference-Fourier map of the nuclear density did not allow to locate unambiguously the proton site.

Fig. 1. Clinoigraphic view of the wadsleyite structure based on the structure refinement of this study.