

3D shapes of olivine negative crystals as fluid inclusions in a mantle xenolith from Pinatubo volcano

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Olivine is one of the most common minerals in space environments as well as in the Earth. The crystal shapes of olivine may reflect their formation conditions and the anisotropy may affect various physicochemical processes. Crystal shapes are classified into two categories; growth and equilibrium forms. Growth form is reflected by its growth condition while equilibrium form is the form where the sum of the products of surface free energy and surface area is minimum. The surface energies of forsterite, Mg end-member of olivine, were obtained by lattice dynamics [1] and *ab initio* calculation [2] at 0 K in vacuum, and the equilibrium forms were shown.

Last year, we examined the 3D shapes of olivine negative crystals in equilibrated chondrites (LL5-6) [3]. The results show that the degree of annealing can be estimated from the negative crystal shapes and {100} planes, which have high surface energy, develop even on highly annealed negative crystals. However, this is not consistent with the equilibrium form and possibility of absorption of molecules such as H₂O on the surface was discussed. In this study, we observed olivine negative crystals, which are fluid inclusions, in a mantle xenolith to compare with the meteorite samples and to estimate the thermal history of the negative crystals with the mantle origin.

The sample is a harzburgite xenolith in pumice erupted in 1991 by Mt. Pinatubo. Abundant fluid inclusions are present in olivine (Mg#:91-92) grains of the xenolith [4]. Two cylinder-shaped samples 20-30 μm in size containing fluid inclusions were extracted from a polished thin section (#P-3) by using FIB (FEI Quanta 200 3DS). Then, the samples were imaged using imaging microtomography system at beamline BL47XU, SPring-8, Japan with the effective spatial resolution of ~150 nm to obtain their 3D structures. The crystallographic orientations of host olivine crystals were determined with an FE-SEM/EBSD (JEOL 7001F/HKL CHANNEL5). The crystal planes of negative crystals were determined from the CT images together with the EBSD results using the same method as [3].

A void with facets, or negative crystal, ~15 μm in size was present in each sample. Both negative crystals did not have any fluid because a part of the wall of the negative crystal was chipped off during FIB micro-sampling. We observed some evaporation residue, probably NaCl, inside the negative crystals in the CT images. The small chipped portions were manually extrapolated to obtain the whole shapes of the negative crystals. Both negative crystals have well developed {100} planes with high surface free energy as well as {010} planes with low surface free energy.

The above feature is similar to the meteorite samples. In contrast, {001} planes with low surface free energy poorly develop unlike the meteorite samples. It is expected that absorption of water molecules on the surface affects the surface energy. However, relative relation between the surface energies of {100}, {010} and {001} planes is not largely changed according to [1].

The difference in the development of the {001} planes between the Pinatubo sample and meteorite samples is possibly due to the presence of fluid and vapor respectively, in contact with the negative crystal surfaces, or merely fluid and vacuum. The edge of an equilibrium form becomes rounded at high temperatures due to the effect of the entropy of edge free energy. The edges of the Pinatubo samples are sharper than the meteorite samples. The negative crystal shape in the meteorite samples may not be a

strict equilibrium form [3]. We may expect such entropy effect even in the negative crystals that do not have a strict equilibrium form. If this is the case, annealing temperature of the Pinatubo samples is lower than the meteorite samples (1100K).

[1] de Leeuw et al. (2000) *Phys. Chem. Minerals*, 27: 332. [2] Bruno et al. (2014) *JCP*, 118:2498. [3] Nakamura et al. (2016) *JpGU*, PPS12-17 (abstract). [4] Kawamoto et al. (2013) *PNAS*, 110: 9663.

Keywords: Olivine, negative crystal, xenolith