CBED analysis of iridescent garnets from Tenkawa, Nara Prefecture, Japan

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Grossular ($Ca_3Al_2Si_3O_{12}$) - andradite ($Ca_3Fe^{3+}{}_2Si_3O_{12}$) garnet solid solutions (space group, Ia-3d), termed grandite series, often exhibit optical birefringence and iridescence. Iridescent garnet from Tenkawa, Nara Prefecture, Japan, has the fine lamellar texture (ca. 100-300 nm in thickness) with small differences in Al/Fe^{3+} ratio by transmission electron microscope (TEM) (e.g., Shimobayashi et al., 2005). Our previous studies (Chang et al., 2016, 2017) reported that extra reflections, 110, 200, 411 and so on, to break the symmetry of Ia-3d were detected in selected area electron diffraction (SAD) patterns from Al-rich fine lamellae and the crystal family of this Al-rich fine lamella is triclinic and lattice type is I-lattice and suggest the space group is I-1 or I1 in the convergent-beam electron diffraction (CBED) pattenrs. Chang et al. (2017) also reported that CBED patterns in the core of lamella and around the boundary between Al-rich and Fe-rich lamellae were different. In this study, we examine CBED by scanning TEM (STEM) to investigate the symmetry to investigate the symmetry of iridescent garnet, especially from the core of Fe-rich lamella to the core of Al-rich lamella, in more detail. And furthermore, chemical map by STEM-EDS were obtained from the same area as STEM-CBED analysis.
A thin section (100 μm thickness) cut parallel to the (001) face through the center of the crystal was prepared from an euhedral single crystal of iridescent garnet with well-developed rhombic dodecahedral {110} facets. TEM specimens with various crystal planes, (001), (110), and (111) were prepared from the thin section by using a focused ion beam technique (ThermoFischerScientific: Quanta 200 3DS, Helios G3 CX) and then TEM (JEOL, JEM-2100F; ThermoFischerScientific, Titan^3 60-300 Double Corrector) observation was carried out to investigate the symmetry and analysis of chemical composition.
CBED patterns in the core of Fe-rich lamella have the clear high order Laue zone (HOLZ) lines in (111), (110) and (001) TEM samples and mirror, 2, 3, 4-fold axes can be identified. These results show that the symmetry of Fe-rich lamella core has Ia-3d. In the core of Al-rich lamella, on the other hand, although HOLZ lines were observed in CBED patterns, no symmetryical elements, such mirror, 2-fold axis and so on, were identified. Thus the symmetry of Al-rich lamella is I(P)-1 or I(P)1. Around the boundary between Fe-rich and Al-rich lamellae, Al/Fe chemical components gradually change. Clear CBED patterns and HOLZ lines were not observed and no symmpetrical elements were identified exactly in all samples. In all TEM samples, however, the analysis of very weak and no-clear HOLZ line shows that mirror plane may exit normal to lamella boundary.

Reference
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