Japan Geoscience Union Meeting 2015
(May 24th - 28th at Makuhari, Chiba, Japan)

SCG58-10 Room:203 Time:May 25 15:45-16:00

Phase transition of sillimanite with Al/Si-disordering at high temperature

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Naturally occurring polymorphs of Al_2SiO_5 (andalusite, kyanite, sillimanite) have assumed a special significance for geologists because of their value as indicators of the pressures (P) and temperatures (T) experienced by metamorphic rocks. Especially, sillimanite may have more geological information in its crystal structure or microstructure. For example, it has been indicated experimentally that sillimanite show the structures like anti-phase boundaries (APB) and/or enrich to Al releasing SiO_2-rich melt at high temperature (Holland & Carpenter, 1986). Miyake et al. (2008) showed sillimanite in Napier complex has APB-like structure and fine mullite inclusion (which is more Al-rich than sillimanite, $Al_2[Al_{2+2x}Si_{2-2x}]O_{10-x}$). In addition, Greenwood (1972) suggested the phase of completely Al/Si-disordered sillimanite as another phase from sillimanite. And, Fischer et al. (2014) found new phase different from sillimanite, mullite, and "completely Al/Si-disordered sillimanite" . These phase also can be valuable geological indicator, but their behaviors are not clear. They need very high resolution for analysis to be detected and classified. Like this, phase relation of sillimanite at high T (and high P) is still so confused. In this study, synchrotron powder X-ray diffraction (XRD) and transmission electron microscope (TEM) experiments were carried out on samples of sillimanite heat-treated in various conditions (P, T and duration time) in order to clarify behavior of sillimanite at high temperature.

We heated sillimanite ($Al_{2.00}Si_{0.99}Fe_{0.01}O_5$) crystals in Rundvagshetta, Lutzow-Holm, Antarctica.sillimanite in the range of 0.8-2.5GPa, 1000-1500 ℃ for 10-1751 hours and then quenched. Experimental products (46 samples) were examined by Synchrotron powder XRD experiment at BL-4B_2 in photon factory, KEK and observed TEM (JEOL JEM-2100F) from the viewpoint of l=odd reflections (which distinct with Al/Si-disordering) and chemical composition.

As a result of XRD experiments, the diffraction patterns of mullite were observed in many samples. Moreover, in four samples heated at 1300-1400 ℃, 1GPa, the peaks of unknown phase were observed in addition to that of sillimanite and mullite. This phase have an intermediate feature between peaks of sillimanite and mullite. (So, we will call it "intermediate-phase" .) As a result of TEM observation of these four samples, grains of the intermediate-phase did not give l = odd reflections, which are characteristic of sillimanite, but distinct with Al/Si-disordering. By chemical analysis (TEM-EDS), the intermediate-phase was slightly Al-rich more than sillimanite but less than mullite. So, It was revealed that the intermediate-phase has the structure which has disordered distribution of Al/Si on the tetrahedral sites, with releasing very little SiO_2 from Al_2SiO_5. This phase can be stable at high temperature and 1GPa. The intermediate-phase is probably not same as "completely Al/Si-disordered sillimanite" by Greenwood (1972) etc., but may be same as the new phase founded by Fischer et al. (2014). The sillimanite with APB and fine mullite inclusion by Miyake et al. (2008) can be explained as that sillimanite transformed from intermediate phase with mullite. APB was made by Al/Si-ordering when intermediate phase transformed to sillimanite.

References:

[1]Holland & Carpenter (1986) Nature, 320, 151-153
[2]Miyake et al. (2008) JAMS Annual Meeting Abstract
[3]Greenwood (1972) THE Geological Society of America, 132, 553-571
[4]Fischer et al. (2014) IMA General Meeting Abstract, 21, 335

Keywords: sillimanite, mullite, Al/Si-disordering, synchrotron X-ray experiment