Crystallography

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High temperature transition of sillimanite, Al_2SiO_5, using synchrotron x-ray powder diffraction data

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Introduction

Sillimanite (Al_2SiO_5) and mullite ($Al_2[Al_{2+2x}Si_{2-2x}]O_{10-x}$) have been examined in order to decide the phase diagram in the binary system Al_2SiO_5-Al_2O_3, but it is not well constrained. For example, Holm & Kleppa (1966) suggested the transformation temperature of sillimanite → mullite + quartz is approximately 1130˚C by calorimetric experiment. However, Tomba et al. (1999) suggested that the temperature at which the X-ray diffraction pattern of mullite could be identified is above 1500˚C. Like this, there are discrepancies in experimental results about temperature at which sillimanite transforms to mullite. In this study, synchrotron X-ray diffraction (XRD) experiments were carried out in order to clarify the phase transition temperature from sillimanite to mullite. The samples used in this study are sillimanite crystals separated from the cordierite-bearing assemblages, Rundvågshetta, East Antarctica (sample name: RVH92011102A, provided from Prof. Kawasaki of Ehime University).

Experimental Procedure

Powder XRD experiment at atmospheric pressure was carried out with synchrotron X-ray in order to identify the diffraction pattern in high resolution. It was performed by using a multiple-detector system and a compact furnace (Toraya et al., 1996, Yashima et al., 2005, 2006) at the BL-4B2 beam line of the Photon Factory (PF), High Energy Accelerator Organization (KEK) in Tsukuba.

XRD experiments of the samples heating at various temperatures, from 1000 to 1530°C for more than 24 hours were experiments at room temperature and moreover *in-situ* XRD experiment at high temperature were carried out at various temperatures, from 1175 to 1400°C, for less than 24 hours.

Results and Discussion

The XRD pattern from the starting material has only the peaks from sillimanite (Fig. 1A). On the other hand, the XRD pattern from some samples showed the mullite peaks in addition to sillimanite peaks (Fig. 1B). The XRD patterns of the run products heated at 1470°C showed that mullite diffraction intensity depends not only on the temperature but also on the heating time. Moreover, the diffraction intensity of mullite in another series products at almost same heating time depended on the heating temperature. TTT (Time-Temperature-Transformation)-diagram for appearance of mullite XRD peaks was made in order to clarify relationship between time and temperature for the transformation from sillimanite to mullite from the all XRD experiments data. From TTT-diagram, the transformation temperature was estimated as 1120°C. This temperature is consistent with the data suggested by Holm & Kleppa (1966).

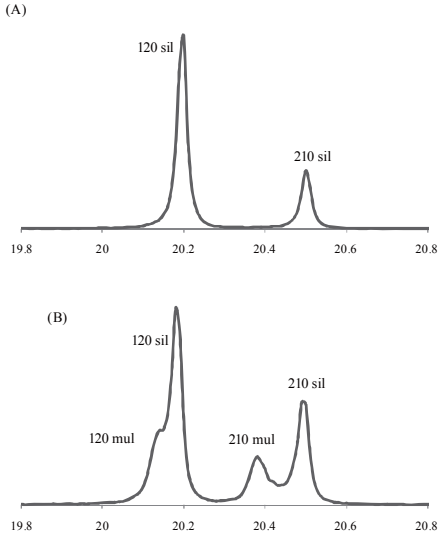

Fig.1. (A) Powder X-ray diffraction (XRD) patterns from starting material. (B) In-situ XRD pattern from the sample at 1470°C for 140.5 hours. It shows presence of mullite in addition to sillimanite.

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References

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