High-pressure in-situ X-ray diffraction study in topaz-OH system

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In last year's meeting, we have shown that topaz-OH ($Al_2SiO_4(OH)_2$, topaz I hereafter) has an another high pressure phase (denoted topaz II hereafter), and results of NMR and Raman spectroscopic characteristics of this new phase were given (K105-010). The study revealed that we need further phase relation study in Al_2O_3 -SiO₂-H₂O system. Also there is an issue in this system that a phase boundary defined by topaz-OH + stishovite = phase egg, has been reported to have about 2GPa difference between two quench experiments (Schmidt et al.,1998 and Ono, 1999). Here we report our recent study of in-situ high pressure X-ray diffraction experiments of topaz-OH using synchrtorn radiation.

We used high pressure X-ray diffraction system installed in BL04B1, SPring-8. In order to generate high pressure, Kawai-type double-stage multi-anvil press was used. In this experiment, we devised a new heater material. Since graphite transforms to diamond above 10GPa, it can not be used above 1000 °C. So we used BN-composite EC heater, which is a well sintered composite made of hBN and TiB₂ mixture, and is commercially available from Denka Co. This new heater worked fine at 10 - 13GPa and up to 1300 °C. Because of its low X-ray absorption, X-ray diffraction and radiographic images can be observed through the heater. Starting sample was a mixture of Al₂O₃, Al(OH)₃, SiO₂, and was put into ether Mo or graphite capsule.

The results are summarized below.1) Above10GPa and below 700 °C, we observed an assemblage of diaspore(AlOOH) + stishovite is stable. 2) At 11GPa, and above 700 °C, the assenblage of diaspore + stishovite is replaced with topaz I. These results are consistent with Schmidt et al. (1998) based on quench experiments. 3) At 11.5 -12.5 GPa and above 900 °C, diaspore + stishovite is replaced with phase egg. This temperature is about 200 °C higher than the boundary for phase egg formation reaction from diaspore plus stishovite of Schmidt et al. (1998), but it might be due to slugish egg formation reaction. 4) Diaspore disappears at above 1000-1100 °C. This would correspond to diaspore dehydration reaction. 5) Topaz I was stable at 1100 °C, and 11.5 to 13.5 GPa. No transition to topaz II nor breakdown was observed. 6) We still see phase egg, for example, at 1100 °C and 11-12GPa. This is outside of phase egg stable region defined by Schmidt et al. (1998), but still inside of it defined by Ono (1999). As topaz and egg can coexsist, observation of 5) does not conflict with this phase egg observation. This would suggest that the phase boundary of topaz-OH plus stishovite to phase egg, determined by Ono (1999) will be more close to our in-situ observation. However, we need to determine reverse reaction, before final conclusion. 7) At 11.5GPa and 1200°C, topaz I breaks down to kyanite-including assemblage, and this is consistent with Ono (1999). 8) We quenched samples from 13.5GPa&1100°C, 12.0GPa&1200°C and 12.5GPa&1250°C, and they were checked by micro-Raman spectroscopy. All were mostly topaz I.

So far we have not found stable region of topaz-OH II yet. We will continue this study and determine phase boundaries in this system using in-situ observation technique.

Ono, S. (1999) Contrib. Mineral. Petrol., 137, 83-89. Schmidt, M.W., et al. (1998) Am. Mineral., 83, 881-888.