American Mineralogist, Volume 94, pages 64–73, 2009

Dehydration dynamics of barrerite: An in situ synchrotron XRPD study

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Abstract

The thermally induced structural modifications of the natural zeolite barrerite [$Na_{16}Al_{16}Si_{56}O_{144}{\cdot}52H_2O$, a = 13.6239(4) Å, b = 18.2033(5) Å, c = 17.8317(7) Å, V = 4422.3(3) Å^3, space group *Amma*, framework type STI] were studied in a temperature-resolved X-ray powder diffraction experiment, using synchrotron radiation, in the temperature range 339–973 K. In the initial stage of heating, up to 508 K, barrerite Phase A (space group *Amma*) is stable, the unit-cell volume decreases by about 4% and a water release of about 66% is observed. Between 521 and 598 K, a phase transition to the collapsed so-called barrerite Phase B (space group *Amma*) is observed. During the transition, the rotation of the 4^25^4 secondary building units causes a large decrease in cell volume and deformation of the channel system. Phase B, at 611 K, shows the statistical breaking of T-O-T bridges in the 4-rings and the migration of the involved tetrahedral atoms to new "face-sharing" tetrahedra, with a consequent reduction of the free volume of the channels parallel to [100]. The new structure is stable up to 741 K and the total volume decrease is about 16%. A new phase appears from 754 K with cell parameters similar to those reported for the highly deformed barrerite Phase D and is stable up to 910 K, which is the temperature at which the total volume decrease is 22.5%. The material does not undergo amorphization up to the highest temperature investigated.

Keywords: Zeolite, barrerite, dehydration, X-ray powder diffraction, synchrotron radiation, crystal structure