[MS18-P09] High-pressure synthesis, crystal structure, and promt gamma activation analysis of B-mullites

Reinhard X. Fischer[a], Hanna Lührs[a], Stefan Söllradl[b,c], Jürgen Konzett[d], Harmut Schneider[a]

[a] *Fachbereich Geowissenschaften, Universität Bremen, Klagenfurter Straße, D-28359 Bremen, Germany*
[b] *Paul Scherrer Institute, Villigen & University of Bern, Switzerland*
[c] *FRM II, Forschungsneutronenquelle Heinz-Maier- Leibnitz,Garching, Germany*
[d] *Universität Innsbruck, Innsbruck, Austria*
E-mail: rfischer@uni-bremen.de

The chemical composition of the technically important ceramic material mullite is given by the solid-solution series $Al_2Al_{2+2x}Si_{2-2x}O_{10-x}$, $0.2 < x < 0.9$ [1]. Depending on the synthesis temperature and atmosphere the crystal strucuture of mullite is able to incorporate a large variety of cations [2, 3 and references therein]. B-doping of mullite results in significant changes of lattice parameters *b* and *c* as reported by several authors [4-6]. Furthermore the incorporation of B results in a strong reduction of the mean thermal expansion coefficient of 15% [5]. The crystal structure of B-mullite has recently been solved from neutron diffraction data of a series of sol-gel derived B-mullite compositions synthesized at 1200°C [7]. Here the results of the chemical analyses of B-mullites using prompt gamma activation analyses (PGAA) are presented as well as the crystal structure refinement of a high-pressure synthesized Bmullite with very high boron content (Al8Si2B2O19). The results of the chemical PGAA are in very good agreement with the results of the Rietveld refinements based on neutron diffraction data. It was found that at ambient pressure (1200°C) about 50% of the initial B2O3 in the gel enters the mullite structure. Another borosilicate phase with the composition $Al_8Si_2B_2O_{19}$ was synthesized at 10 kbar and 875°C. This phase was first described by [8] as a boron-bearing sillimanite derivative and later on as "boron-mullite" with traces of disordered boralsilite [9]. Here we present PGAA and XRD data corresponding to a Bmullite structure with a significantly higher B-content than observed before. For lattice parameters b and c and the inclination angle ω linear trends with increasing boron content are observed. The Rietveld refinements support the substitution mechanism known for B-mullites [7] involving the formation of BO3 units and oxygen vacancies. For $Al_8Si_2B_2O_{19}$ lattice parameter a is significantly smaller than expected for "normal" B-mullite, thus probably suggesting a second mechanism for B-incorporation.

References

[1] Fischer, R.X., et al. (1996) J Eur Ceram Soc, 16(2), 109.
[2] Schneider, H., in Mullite, H. Schneider and S. Komarneni, Editors. 2005, WILEY-VCH: Weinheim. p. 70- 93.
[3] Schneider, H., et al. (2008) J Eur Ceram Soc, 28(2), 329.
[4] Griesser, K.J., et al. (2008) Mineralogy and Petrology, 92(3-4), 309.
[5] Lührs, H., et al. (2012) Mater Res Bull, 47, 4031.
[6] Zhang, G.M., et al. (2010) J Eur Ceram Soc, 30(12), 2435.
[7] Lührs, H., et al. (2013) Z Kristallogr - Crystalline Materials. doi: 10.1524/zkri.2013.1595
[8] Werding, G., et al. (1992) Eur J Mineral, 4(1), 193.
[9] Grew, E.S., et al. (2008) Am Mineral, 93(2-3), 283.

Keywords: B-mullite, high-pressure synthesis, crystal structure