

FABRICATION OF HIGHLY DENSE AND FINE-GRAINED POLYCRYSTALLINE ANORTHITE BY VACUUM SINTERING

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Introduction: In laboratory measurements of physical and chemical properties of the earth's lower crust, highly dense polycrystalline aggregates of major constituent minerals such as anorthite ($\text{CaAl}_2\text{Si}_2\text{O}_8$) are required. For precise measurements of these properties, the aggregates should have (1) high density, (2) fine grain size (to escape from cracking during quenching), (3) homogeneous microstructure, (4) controlled sample shape and size, and (5) controlled chemistry. We have developed a technique to synthesize anorthite aggregates which meet the above conditions.

Experimental: We used nano-sized powders of SiO_2 , Al_2O_3 , CaCO_3 and $\text{Mg}(\text{OH})_2$ as starting material to synthesize single phase aggregates of anorthite and, two phase composite of anorthite + diopside and anorthite + quartz. Those powders were mixed to provide a resulting mole ratio of CaO, Al_2O_3 , SiO_2 , and MgO to obtain desired mineral assemblies. Densified aggregates were obtained through the following stages: calcination, forming and sintering. The final materials were characterized with X-ray powder diffraction (XRD) and secondary electron microscopy (SEM).

Results and Discussion:

Anorthite: Calcination was conducted under temperature of 670-970°C with fixed duration of 30-180 min. Coalescence of the powder was observed > 820°C, which was revealed by SEM. XRD result indicates that decarboxylation of anorthite solid state reaction completes at > 770°C for 90 min. For sintering, a temperature of 1210-1260°C with controlled time can provide essentially full dense aggregates of anorthite with an average grain size of 1.7 μm and porosity of 0.2 vol%.

Two-phase materials (anorthite + diopside): Diopside grains of 10 vol% was introduced to anorthite aggregates. Calcination at 770°C for 30 min and sintering at 1200°C for 50h were found to be the best conditions so far to synthesize highly dense aggregates. Average grain sizes of 1.6 μm and 0.8 μm were detected for anorthite and diopside grains, respectively. Density of 99.6% was achieved.

References: S. Koizumi et al., Phys. Chem. Miner. 37, 505-518 (2010)

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