

Synthesis and characterization of lithium silicates powders

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Lithium-based ceramics, such as Li_2O , LiAlO_2 , Li_4SiO_4 , Li_2SiO_3 , Li_2TiO_3 and Li_2ZrO_3 , have long been recognized as promising tritium breeding-materials for D-T fusion reactor blankets. Among these candidate materials, lithium orthosilicate (Li_4SiO_4) and lithium metasilicate (Li_2SiO_3) are recommended by many ITER research teams as the first selection for the solid tritium breeder. Li_4SiO_4 has even been selected as the breeder material for the helium-cooled solid breeder test blanket module (HCSB TBM) in China and EU. In present study, the processes of solid-state reaction between amorphous silica and Li_2CO_3 powders was studied by thermogravimetry analysis–differential scanning calorimetry (TGA/DSC); the lithium silicate powders were synthesized at 700, 800 and 900 °C with Li:Si molar ratios of 0.5, 1, 2 and 4, respectively, using solid-state reaction method. The as-prepared lithium silicates were characterized by X-ray diffractometry (XRD) and scanning electron microscopy (SEM). The results show that the phase composition and morphology of the as-prepared samples change with the different synthesis conditions. At low temperature of 700 °C, all samples contain the amorphous silica, and the major crystalline phase is Li_2SiO_3 with different microstructure for Li/Si ratio of 0.5, 1 and 2. As for Li/Si = 4, 98% purity of Li_4SiO_4 can be obtained at 700 °C. At high temperature of 900 °C, the significant sinterization effect will occur in all samples and Li_4SiO_4 will even decompose. The results also show that pure Li_4SiO_4 can be synthesized by calcining at 800 °C for 4 h, and its' solid-state reaction synthesis may be divided into two steps:

(1) 515–565 °C: $\text{Li}_2\text{CO}_3 + \text{SiO}_2 \rightarrow \text{Li}_2\text{SiO}_3 + \text{CO}_2$;

(2) 565–754 °C: $\text{Li}_2\text{CO}_3 + \text{SiO}_2 \rightarrow \text{Li}_2\text{SiO}_3 + \text{CO}_2$ and then $\text{Li}_2\text{SiO}_3 + \text{Li}_2\text{CO}_3 \rightarrow \text{Li}_4\text{SiO}_4 + \text{CO}_2$.

While Li/Si = 2, 99% purity of and pure Li_2SiO_3 can be obtained at 800 and 900 °C, respectively.

Keywords: Test blanket module; Tritium breeder; Lithium silicates; Solid-state reaction; X-ray diffractometer; Scanning electron microscope