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LOW COST SYNTHESIS OF SILICON-BASED CERAMIC POWDERS FROM NA, K AND CS GEOPOLYMER

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Abstract

Geopolymer carbon precursors (1:1:4.5:12+18C) for carbothermal reduction and nitridation were prepared by the following route. NaGP, KGP and CsGP resins were prepared by mixing metakaolin ($\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$) in NaOH, KOH, or CsOH alkaline solutions, respectively. The viscous and homogenous slurries was poured into a Teflon mold to obtain bar samples at ambient temperature and cured in a constant 50 °C temperature/humidity oven for 24 h. GP bars were powdered by following additional drying in an open air furnace at 300 °C for 1 h. To obtain GP18C precursors, the powdered pure GPs were ball milled with 18 moles of carbon at room temperature using cylindrical zirconia balls in a plastic grinding jar for 5 h, with a ball to powder wt % ratio of 5:1, respectively. The carbothermal reduction and nitridation process of the GP18C precursors were carried out in an atmosphere controlled, tube furnace at temperatures of 1400°, 1500° and 1600 °C for 2 hours using high purity nitrogen (99.99%) under dynamic conditions. XRD, Rietveld refinement and SEM-EDS analyses were made to determine transformation and morphology of all the products after carbothermal reduction and nitridation. Different types of nitride analogues of GPs were examined in the products depending on the alkaline conditions of the GPs.

Sialon-type ceramic powders were synthesized from geopolymer carbon precursors (1:1:4.5:12+9C) prepared different alkaline conditions by carbothermal reduction and nitridation. NaOH, KOH, or CsOH alkaline solutions were used to make NaGP, KGP and CsGP resins by mixing metakaolin ($\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$), with water glass, respectively. NaGP9C, KGP9C and CsGP9C resins were prepared by mixing 9 moles of carbon nano-powder with GP resins. The viscous and homogenous slurries was poured into a Teflon mold to obtain bar samples at ambient temperature and cured in a humidity controlled, constant temperature oven at 50 °C for 24 h. The GP9C compacts were powdered by following additional drying in an open air furnace at 300 °C for 1 h. Subsequently, the GP9C powders were planetary milled with a ball to powder wt % ratio of 5:1 for 10 min to increase their reactivity. As a result, the GP9C precursors were carbothermally reacted in an atmosphere-controlled tube furnace at temperatures of 1400°, 1500° and 1600 °C for 2 hours with high purity nitrogen (99.99%) under dynamic conditions. XRD, Rietveld refinement and SEM-EDS analyses were made to determine transformation and morphology of all the products after carbothermal reduction and nitridation. Depend on the alkaline conditions of the GPs, phase pure or multiple-type sialon compounds were examined.