Title:
Investigation on Low Temperature Synthesis of β-SiC by using MCM-48 / Polyacrylamide Nanocomposite as Precursor and Magnesiothermic Reduction Process

Abstract:
In this study first, MCM-48 silicate mesoporous was synthesized. Then in conditions different from concentration of acrylamide monomer and with different ratios of carbon to silica, MCM-48 / polyacrylamide nanocomposite was prepared by in situ polymerization method. Next in argon atmosphere at 700°C, samples were carbonized with the heating rate of 5°C/min and the retention time of 3 hours at the maximum temperature. After carbonization, the samples prepared in polymerization conditions of high concentration of acrylamide monomer were put under heat treatment at the temperature ranges of 650-700°C and 800-850°C along with coarse grained magnesium (~2 mm). Moreover after carbonization, the samples prepared in polymerization conditions of low concentration of acrylamide monomer were put under heat treatment along with coarse grained magnesium (~2 mm) at the temperature ranges of 550-600°C, 650-700°C. And following it, the samples prepared in the same polymerization conditions were also put under heat treatment at the temperature range of 550-600°C along with fine-grained magnesium >0.1 mm. All samples in argon atmosphere were put under heat treatment with the heating rate of 5°C/min and the retention time of 6 hours at the maximum temperature and finally under acid washing operations in order to strip undesirable phases. The physicochemical properties and microstructures of the nanocomposite precursor and SiC obtained at low temperature were characterized by various techniques such as X-Ray Diffraction (XRD), Transmission Electron Microscopy (TEM), X-ray map, Differential Scanning Calorimetry (DSC), Thermogravimetric Analysis (TGA) and N2 adsorption–desorption isotherm. XRD results indicate formation of β-SiC phase in the temperature range of 800-850°C (high concentration of acrylamide monomer-coarse grained magnesium), in the temperature range of 650-700°C (low concentration of acrylamide monomer-coarse grained magnesium) and in the temperature range of 550-600°C (low concentration of acrylamide monomer-fine-grained magnesium). TEM microstructures and BET studies show that in these three temperature ranges, the synthesized SiC powder is in the form of mesoporous and with very high specific surface in which the reduction is also seen with increase in synthesis temperature. The exothermic peak in DSC graph shows self-combustion process of magnesiothermic synthesis of silicon carbide. The results also revealed in addition to formation of silicon carbide phase, carbon can also contribute to silica phase reduction. The results showed that the grain size of magnesium, polymerization conditions and the content carbon amount affect the initial temperature of reaction of SiC formation.

Keywords:
SiC, nanocomposite, mesoporous, magnesiothermic reduction, in situ polymerization