Sublimation growth of 3C-SiC in induction heating of preceramic Si-C nanoparticles

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Cubic 3C-SiC is unique compared to hexagonal polytypes of SiC, because of its isotropic physical properties, higher electron mobility and lower density of the near interface traps in e.g. $SiO_2/3C-SiC$ system. Furthermore, 3C-SiC is the only polytype that grows on silicon and is shown to be a suitable substrate for the growth of graphene.

Poor availability of good quality bulk 3C-SiC seeds and substrates hinder the development of 3C-SiC technology. Cubic silicon carbide occurs spontaneously in the range of 1500-2000 °C, in both a solution and a chemical vapour deposition (CVD) route (Chaussende *et al*, 2008). However, stabilization of 3C-SiC nucleation is a challenge, because several parameters influence the polytype nucleation and growth (e.g. temperature, Si/C ratio, supersaturation, vapour composition, substrate properties). Because 3C-SiC is a metastable form, a solid-state 3C to 6H transition is usually encountered at temperatures above 2000 °C.

In this work sublimation growth of 3C-SiC in controlled induction heating of previously reported preceramic silicon-carbon nanoparticles (Miettinen *et al* 2011) under argon flow at atmospheric pressure was studied. A wide range of induction temperatures from 1900 °C up to 2600 °C was used to allow 3C-SiC nucleation to occur, and to follow up solid-state transition from 3C- to 6H-SiC at temperatures above 2000 °C.

An X-Ray diffraction (XRD) analysis was done after induction heating of the precursors. Additionally, thermal gravimetric analysis (TGA) was conducted to study thermal stability of the formed SiC crystals and to remove free carbon. Scanning electron microscopy (SEM) and dispersive Raman spectroscopy analyses were performed both after induction heating and after TGA treatment.

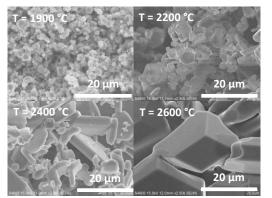


Figure 1.The SEM micrographs of TGA treated SiC crystals formed at different induction temperatures

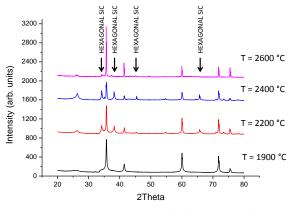


Figure 2. The XRD patterns of SiC crystals formed at different induction temperatures

SEM micrographs (Fig. 2) prove that large crystals were grown during the process. Crystal size increased with increasing induction temperature. At the highest induction temperature complete dissociation of the precursor silicon-carbon particles occurred and long SiC rods were grown on the exit of the crucible, while material left in the crucible was carbon.

XRD analysis reveal that SiC crystals consisted of several polytypes. Peaks related to cubic 3C-SiC (PDF 04-002-9070, ICDD) and hexagonal 6H-SiC (PDF 04-010-5698, ICDD) crystal structures matched with the sample spectrum in all the cases (Fig. 2). However, the spectra contained regions that could not be interpreted entirely with the 3C and 6H-SiC reference patterns. Consequently, it was obvious that also higher order hexagonal (e.g. 69R-SiC and 84R-SiC) polytypes existed in our crystals, except at induction temperature of 2600 °C, where only peaks of 3C- and 6H-SiC were detected.

As a conclusion, induction heating of synthetic Si-C nanoparticles at temperatures between 1900 and 2600 °C led to growth of large polycrystals of SiC. 3C-SiC was dominant at the lowest (~74 w-%) and at the highest (~91 w-%) induction temperature, where complete dissociation of the precursor silicon-carbon particles occurred. Solid-state transition from 3C- to 6H-SiC started below 2000 °C and was almost completed at induction temperature of 2400 °C. Lower induction temperature and longer growth period is needed for growth of larger 3C-SiC crystals.

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Chaussende, D. et al (2008) J. Crystal Growth **310**,976-981. Miettinen, M. et al (2011) J. Nanopart. Res. **13**,4631-4645.