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Densification, microstructure and properties of liquidphase sintered silicon carbide materials

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1. INTRODUCTION

The formation of silicon carbide in the laboratory was first reported by Berzelius in 1824[1]. However, it was only after the invention of the electric smelting furnace by Cowles[2] in 1885, and the development of the Acheson process[3] in 1892, that silicon carbide's technical importance as a hard material was realised. Today, silicon carbide is one of the most widely used non-oxide ceramics with an annual world production of approximately 700 000 ton[4]. Because of its excellent high temperature strength, high hardness, good oxidation and thermal shock resistance, and low specific mass, it is commonly used as an abrasive and a raw material for making refractory materials such as firebricks and setter tiles. It is also commonly used in heating elements. In addition, silicon carbide is used as a silicon and carbon source for refining iron and steel.

Since 1885 different methods of sintering of silicon carbide materials have been developed. High porosity-silicon carbide types are (i) ceramic bonded silicon carbide, "CSiC", (ii) recrystallised silicon carbide, "RSiC", (iii) reaction-bonded silicon carbide. These types of silicon carbide materials normally contain up to 20% porosity and thus are not suitable for engineering applications. The low porosity (less than 2%) containing silicon carbide material types are (i) silicon infiltrated silicon carbide, "SiSiC", (ii) solid-state sintered silicon carbide, "SSiC", (iii) liquid phase-sintered silicon carbide, "LPSSiC"[4]. SSiC and LPSSiC materials are the most commonly used silicon carbide materials for engineering applications.

One of the main problems in the production of silicon carbide materials is in their densification, because of its strong covalent bonding character[5,6]. This means that silicon carbide materials cannot be fully densified without sintering additives and high sintering temperatures. For example, solid-state sintering of silicon carbide, using boron and carbon additives, used

to pressureless sinter silicon carbide since the early 1970's[7], will only achieve full densification (>98% of its theoretical density) at temperatures around 2100^{o}C. On the other hand, the densification of silicon carbide using liquid phase sintering additives, such as yttria, Y_2O_3 and alumina, Al_2O_3, introduced by Omori *et al.* in the early 1980's[8], requires temperatures lower than 2000^{o}C.

The advantage of LPSSiC materials is twofold. Firstly, the sintering additives allow densification at lower temperatures, which reduces grain growth. Secondly, the presence of residual intergranular phase in LPSSiC materials enhances the toughness of the material, which is the inherent problem in the other types of silicon carbide materials. As a result, LPSSiC materials have been found to have useful applications in industry as structural engineering ceramic materials.

However, a major problem associated with sintering of silicon carbide in the presence of oxide additives is the reaction between silicon carbide and the oxide. These reactions result in the formation of volatile components and consequent mass loss[9]. If not properly controlled, the resultant mass loss can significantly affect the final properties of the material. The sintering additives not only react with the silicon carbide, resulting in decomposition, but also react with each other and the silica, present in commercial silicon carbide powders. These reactions result in a wide range of possible phases which remain as the grain boundary component of the material. The understanding of the influence of these complex grain boundary phases on the densification and properties of LPSSiC is not very well established. Most of the research work in the literature[10-15] has been focused on a few parameters such as the polytype and grain size of the silicon carbide.

Understanding of the relationships between the microstructure and the electrical properties of LPSSiC materials is also very limited. Volz *et*

al.[16,17,18], Martin *et al.*[19, 20] and Makrlik *et al.*[21] have studied the electrical properties of dense LPSSiC with the Y_2O_3-Al_2O_3 sintering additives system. However, none of these groups did conductivity- temperature dependence measurements.

Additionally, ultra-high pressure sintering (pressures around 5GPa) of silicon carbide has only been reported by Gadzira *et al.*[22,23] and Gadzyra *et al.*[24]. Their work was limited to solid state sintered silicon carbide.

As a result of these shortfalls in the literature, attainment of tailor-made properties, that are suited to a particular application, of LPSSiC materials, and their process-control, may not be fully realized.

The aim of this research work was to establish relationships between densification of LPSSiC materials and their microstructures and to relate these microstructures to mechanical and electrical properties.

To achieve these objectives, different molar ratios of Y_2O_3:Al_2O_3 were sintered using three different densification techniques, hot pressing (HP) gas pressure sintering (GPS) and ultra-high pressure (UHP) sintering. Post-sintering heat treatments on the different materials was also carried out. The processed LPSSiC materials were analysed in terms of densification, microstructure, as well as mechanical and electrical properties.

Densification kinetics during hot pressing was measured. The density of all materials was measured using the Archimedes method, and the mass loss of the gas pressure sintered and post-sintering heat treated materials was also measured.

The microstructure of the LPSSiC was studied by XRD and the phase content was quantitatively determined using Rietveld refinement

calculations. The microstructure was further studied using optical light microscopy and a Field Emission Scanning Electron Microscope (FESEM). The FESEM images were used to quantify microstructural parameters, including mean SiC grain size and grain boundary content.

Mechanical properties which were measured are Vicker's hardness, indentation fracture toughness, four-point bending strength and elastic modulus. The mode of fracture of the indentation-generated cracks was studied to relate the microstructure of the materials to their measured fracture toughnesses.

Ac and dc electrical conductivity of LPSSiC materials was measured at room and elevated temperatures (up to 330^{o}C). Impedance spectroscopy techniques were used to separate the electrical properties of different phases/ groups of phases in the LPSSiC materials.

Increased understanding of microstructure-mechanical and microstructure - electrical properties relationships would be invaluable, since this would enable the better control of the processing of LPSSiC materials for particular applications.