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The Effect of High Temperature Annealing on the Grain Characteristics of a Thin Chemical Vapor Deposition Silicon Carbide Layer

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The unique combination of thermo-mechanical and physiochemical properties of silicon carbide (SiC) provides interest and opportunity for its use in nuclear applications. One of the applications of SiC is as a very thin layer in the TRi-ISOtropic (TRISO) coated fuel particles for high temperature gas reactors (HTGRs). This SiC layer, produced by chemical vapor deposition (CVD), is designed to withstand the pressures of fission and transmutation product gases in a high temperature, radiation environment. Various researchers have demonstrated that macroscopic properties can be affected by changes in the distribution of grain boundary plane orientations and misorientations [1 - 3]. Additionally, various researchers have attributed the release behavior of Ag through the SiC layer as a grain boundary diffusion phenomenon [4 - 6]; further highlighting the importance of understanding the actual grain characteristics of the SiC layer. Both historic HTGR fission product release studies and recent experiments at Idaho National Laboratory (INL) [7] have shown that the release of Ag-110m is strongly temperature dependent. Although the maximum normal operating fuel temperature of a HTGR design is in the range of 1000-1250°C, the temperature may reach 1600°C under postulated accident conditions. The aim of this specific study is therefore to determine the magnitude of temperature dependence on SiC grain characteristics, expanding upon initial studies by Van Rooyen *et al*, [8; 9].

Two coated particles batches, designated D and E, were chosen due to the different SiC coating thickness (39 µm and 32 µm) and CVD deposition temperatures (1450ºC and 1510ºC). The SiC CVD deposition rate used is approximately 0.23µm/min for both batches. Three electron back scattered diffraction (EBSD) data sets from batch E are described in this paper: an unannealed reference sample, a sample annealed at 1600°C and a sample annealed at 2000°C. Due to the lengthy sample preparation, only two EBSD datasets are available from batch D: the unannealed reference sample and a sample annealed at 2000°C. The EBSD analysis was executed using an EDAX TSL Hikari electron backscattered detector coupled to a FEI Quanta 3D FEG scanning electron microscope.

The effect of the annealing temperature on grain boundary characteristics of batches D and E is shown in Figure 1(a). Although a slight increase of high angle boundaries are observed with increased annealing temperature for batch E (black arrow), no significant trend is observed in the coincidence site lattice (CSL) boundaries with temperature. Because recent work by Lopez-Honorato *et al.* [5] showed that high angle boundaries do enhance diffusion of Ag, it is speculated that the increase in high angle boundaries upon annealing of batch E will result in increased Ag release at 1600ºC. The behavior of batch D (white arrow) is significantly different from those of batch E, by a significant higher fraction of high angle boundaries in the initial reference sample which decreased to approximately 50% after annealing at 2000ºC. Similar to batch E, no changes with annealing temperature is observed in the batch D CSL fractions.

In Figure 1(b), a slight decrease of Σ3 grain boundaries for batch E is observed with increased annealing temperature. Khalil *et al,* [10] found by formation energy calculations that a strong segregation of Ag to the Σ3 grain boundaries are implied. Batch E showed about double the fraction of Σ3 CSL compared to those of batch D in the reference samples. It is therefore implied that batch E will initially have a lower diffusion of species compared with coated particles of batch D. It is speculated that the lower SiC deposition temperature of batch D is the reason for this behavior.

Good correlation is achieved between the EBSD and previously determined average grain sizes using the lineal intercept method average grain sizes as reported by Van Rooyen *et al*, [8]. These values showed an increase in average grain size with increased annealing temperature. The results obtained from the two methods for Batch D did not show the same behavior and remains under investigation. Although interesting, it was found in previous studies by Van Rooyen *et al*, [9] that the average grain size may not be the critical parameter to control for Ag transport behavioral prediction. Final conclusions on the grain characteristic trends with temperature need to be considered once a larger sample set is investigated. Additionally, these preliminary predictions of Ag transport will need to be confirmed in actual irradiated SiC studies.

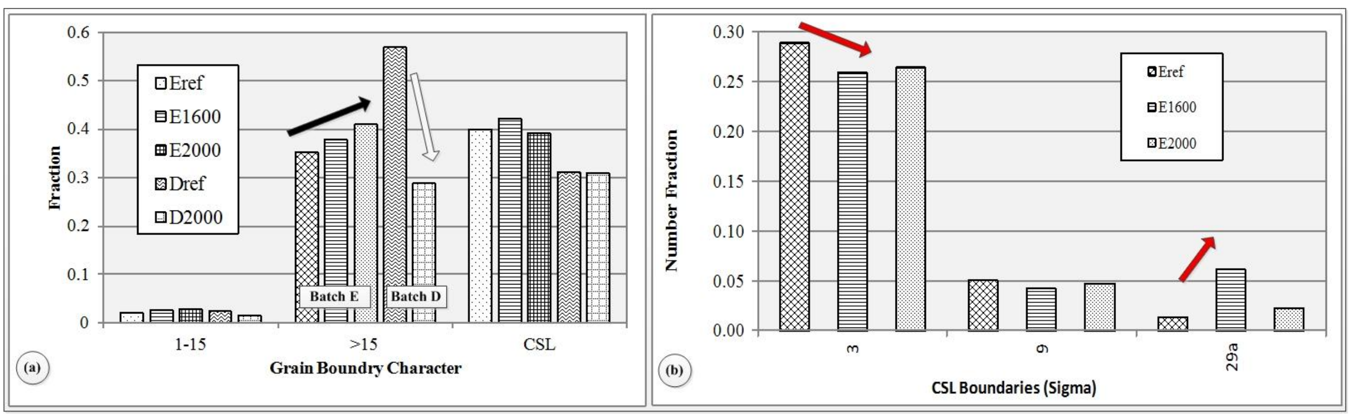

Figure 1. Effect of annealing temperature on (a) grain boundary character and of batches D and E and (b) CSL boundaries of batch E.

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