INL/JOU-15-36781
Accepted Manuscript

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March 2017

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Prepared for the U.S. Department of Energy
Office of Nuclear Energy
Under DOE Idaho Operations Office
Contract DE-AC07-05ID14517

Sample Preparation Techniques for Grain Boundary Characterization of Annealed TRISO-Coated Particles

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Manuscript has 12 pages, not including the abstract or references.
12 Figures
0 Tables

ABSTRACT

Crystallographic information about chemical vapor deposition layers of silicon carbide (SiC) is essential to understanding layer performance, especially when the layers are in non-planar geometries such as spherical. Electron back scatter diffraction analysis of spherical SiC layers was performed using a different approach to sampling via a focused ion beam milling technique to avoid the negative impacts of traditional sample polishing and to address the need for very small samples of irradiated materials for analysis. Mechanical and chemical grinding and polishing of sample surfaces can introduce lattice strains and result in unequal removal of SiC and the surrounding layers of different material due to the hardness differences of these materials. The nature of layer interfaces is thought to play a key role in the performance of SiC; therefore, analysis of representative samples at these interfacial areas is crucial. In work reported here, a focused ion beam was employed in a novel manner to prepare a more representative sample for electron back scatter diffraction analysis from tristructural isotropic layers that are free of effects introduced by mechanical and chemical preparation methods. In addition, the difficulty of handling neutron-irradiated microscopic samples (such as those analyzed in this work) has been simplified with pre-tilted mounting stages. This study showed that while the average grain size of samples may be similar, the grain boundary characteristics can differ significantly. Furthermore, it was found that low-angle grain boundaries comprise 25% in the focused ion beam-prepared sample compared to only 1 to 2% in the polished sample from the same particle. From this study, it was determined that characterization results from the focused ion beam-prepared samples provide more repeatable results, as the effects of sample preparation are eliminated.

Keywords: silicon carbide, focused ion beam, grain boundary character

1.0 INTRODUCTION

A significant challenge for next-generation, high-temperature nuclear reactor designs is the availability of new materials compatible with extreme conditions. Developing this type of material is the focus of work presented here. Silicon carbide (SiC) has extraordinary physiochemical properties, including chemical stability, thermal and radiation resistance, high resistance to oxidation, high thermal conductivity, and high mechanical strength. Dimensional stability under high-temperature (i.e., maximum linear expansion of 0.7 % at 250°C) and irradiation conditions further make SiC a material of interest. Of greatest significance for the nuclear industry, SiC serves as the main barrier to fission product release in the high-temperature gas reactor design. Fission product migration, particularly silver (Ag) migration, through the SiC layer in tristructural isotropic (TRISO)-coated fuel (see Figure 1) has been internationally investigated for more than 40 years [1, 2], but existing modeling efforts fail to fully describe the measured Ag release [3]. One of several migration mechanisms studied is via SiC grain boundaries. A greater understanding of the nature of SiC grain boundaries is essential to understanding and preventing fission product migration in this mode.

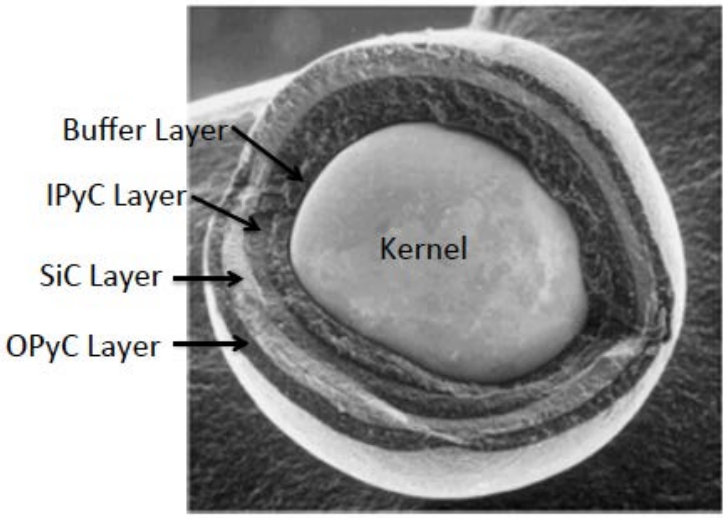


Figure 1. SEM image of a TRISO-coated particle showing the kernel at center, surrounded by three types of layers: a porous carbon buffer surrounding the kernel and pyrolitic carbon layers (IPyC and OPyC) sandwich layers around the SiC layer.

Electron back scatter diffraction (EBSD) is a practical characterization technique for obtaining crystallographic information, including crystal type, orientation, grain boundary characteristics, grain size distribution, and texture. These measurements are obtained from small areas using a scanning electron microscope (SEM). The ability of this technique to perform scanning over a wide range of magnifications makes it possible to investigate the microstructure down to nano levels when needed [4]. Data from this type of analysis provide details about high-angle grain boundary distribution, which can be used to test the hypothesis of Ag transport along grain boundaries [4, 5, 6, 7].

Because Si and C are fairly light elements, the signal generated for EBSD collection is weak. However, unirradiated SiC materials examined to date possessed fairly well preserved microstructures and were measureable. However, because a crystal structure is stressed mechanically by heat treatment or by neutron exposure, the Kukuchi patterns (i.e., lines) will become weak. As the material approaches an amorphous state, the lines may not exist at all. This behavior provides an additional challenge for collection of EBSD data on irradiated SiC layers.

Reliable EBSD analysis of a polycrystalline material (such as SiC), whether it is irradiated or not irradiated, requires a smooth surface [7]. Typically, sample preparation methods for analysis of the SiC layer consist of mechanical grinding of a sample TRISO-coated particle that is embedded in epoxy resin down to a hemisphere cross section. Subsequent fine polishing can be accomplished by various methods [7, 8, 9, 10], including that reported by Tan et al. [10], who used diamond paste, alpha alumina, and colloidal silica solutions in a given order with good results. Nevertheless, in many instances, TRISO particle researchers have encountered difficulties when mechanically and chemically grinding and polishing sample surfaces, because these methods can introduce lattice strains and result in unequal removal of the SiC and surrounding pyrocarbon layers. This phenomenon is illustrated in the micrograph of a traditionally polished TRISO particle in Figure 2.

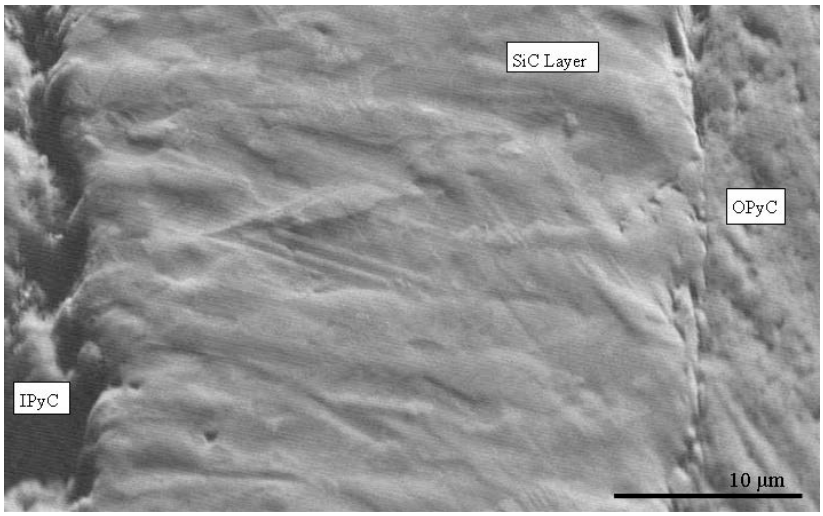


Figure 2. SEM image (3300×) of the sample Eref SiC layer with rounded edges at the IPyC and OPyC layer boundaries after traditional polishing (adapted from [9]).

Focused ion beam (FIB) milling can sputter materials of differing hardness in a highly localized manner with a beam probe approximately 5 nm in diameter. Consequently, FIB cross sectioning through materials of differing hardness has a much better outcome than that from mechanical methods [11]. Kirchhofer et al. [8] used a FIB to raster across the surface of a section of a mounted, coated TRISO particle that previously had been mechanically polished in preparation for analysis. The FIB removed the rounded edges between the different layers, creating a smooth surface suitable for EBSD analysis. However, it is unclear if the depth of material removal by FIB would result in removal of all material mechanically affected by the polishing/grinding process.

To address the problems caused by mechanical polishing, van Rooyen et al. [12] used the FIB to mill into the particle and extract a sample from within the TRISO-coated layers, well beneath (at least 35 µm from the cross-sectioned surface) any damage from material grinding This difference in FIB application and relative EBSD sample location is visualized in Figure 3. Additionally, this sampling technique facilitates EBSD analysis of neutron-irradiated SiC layers, for which very small samples are required. It is necessary to prepare ever smaller irradiated samples to accommodate activity and dose limits in characterization facilities.

In general, handling very small samples is difficult for analysis. Mounting and proper alignment are difficult without the aid of a high-powered microscope. For example, Helary et al. [13] reported an awkward ex situ transfer of a cross-sectioned particle from epoxy to the SEM sample stage, where it was necessary for alignment of the cross-sectioned face o be exactly parallel to the sample stage. The FIB removal of a small section of the TRISO layers by van Rooyen et al. [12]

addresses the need for very small samples of irradiated material, which are also much more easily aligned than whole particles. In addition, the small sample allows for successive FIB removal of layers to create of three-dimensional images. Such successive milling and imaging is a very cumbersome process with a whole particle.

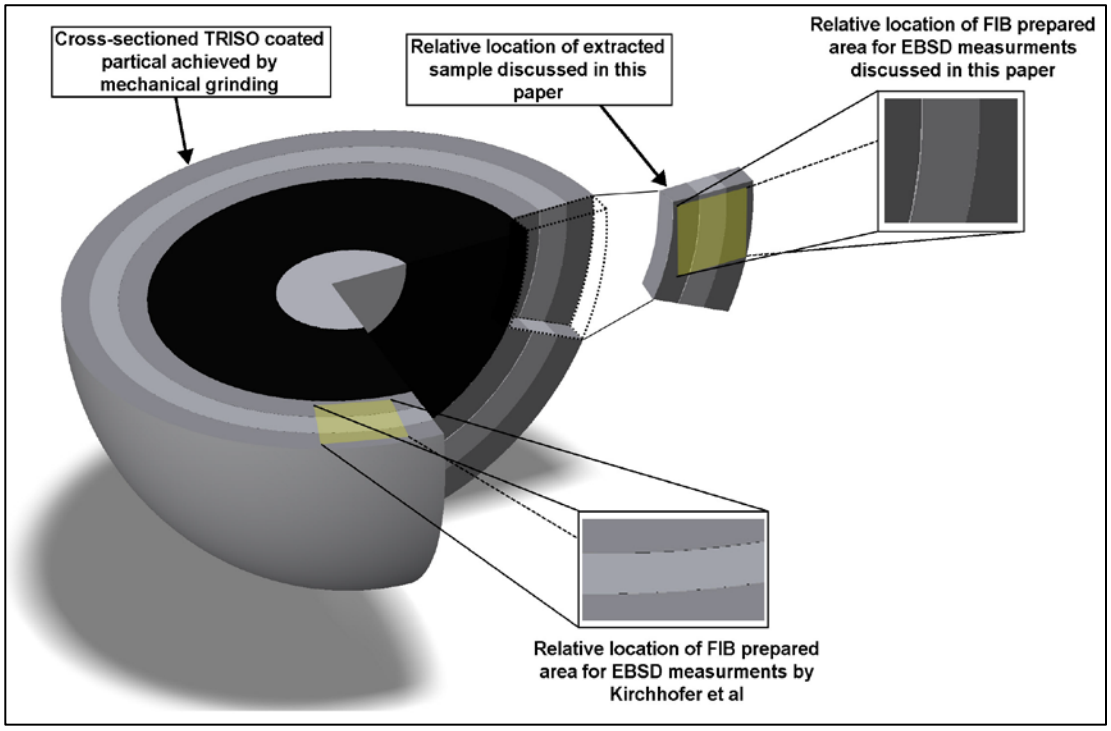


Figure 3. Illustration denoting a cross-sectioned, TRISO-coated fuel particle and the relative locations of samples removed via FIB for EBSD analysis by Kirchhofer et al. [8] and van Rooyen et al. [12].

The primary objective of the work presented in this paper was demonstration of the specific FIB technique used for removal of a small, but representative, sample for an out-of-pile separate effects study. Results of EBSD analysis of samples prepared by both traditional polishing and by the FIB milling and polishing technique are compared. General use of FIB lift-out techniques to prepare EBSD samples is well established [14-18]; however, the authors have demonstrated use of FIB to access and extract a representative sample from within a material that was exposed for analysis by mechanical and chemical polishing. While mechanical and chemical polishing are necessary steps, the result is a material unsuitable for EBSD analysis due to rounded edges between layers of differing hardness and due to damage of the crystal structure. When FIB milling is used well below the surface of the material to be analyzed, a sample free of the effects of chemical and mechanical polishing is produced for EBSD analysis. Such samples will also be amenable to additional analyses, such as 3-dimensional rendering of grain boundary characteristics via successive FIB milling and EBSD characterization.

2.0 MATERIAL AND METHODS

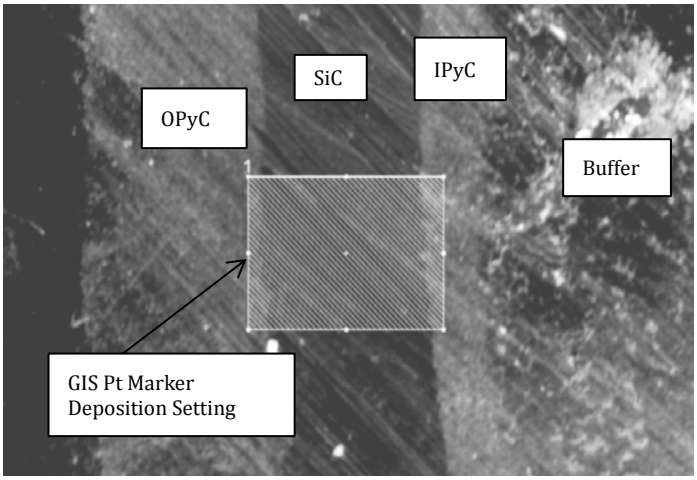
2.1 Sample Description

Coated particles used in developing the modified FIB sample preparation method have non-fuel kernels (composed of ZrO_2) surrounded consecutively by layers of porous carbon buffer, inner pyrolitic carbon (IPyC), SiC, and outer pyrolitic carbon (OPyC). The SiC layer was applied via chemical vapor deposition at 1510°C at a deposition rate of 0.24 micrometers/minute in the Advanced Coating Facility at the South African Nuclear Energy Corporation. The completed and coated particles were suspended in an epoxy resin and mechanically thinned to a hemisphere by using a Buehler-Beta grinder polisher and exposing the various layers of coating. Finally, it was polished using a 0.05-µm colloidal silica suspension [9]. The samples were further mounted in an epoxy resin for transport and analyses.

2.2 Sample Preparation Technique

The sample extraction method starts with a mounted cross-sectioned hemispherical fuel particle placed in the FIB/SEM system. The surrogate fuel samples used in this project were prepared and analyzed in an FEI Quanta 3D SEM-FIB instrument used for high-resolution imaging and milling of specimens. It is also equipped with platinum and carbon gas injection systems for deposition specimen marking and probe welding, as well as an OMNI Probe for in-situ lift-out of samples from a given specimen.

The gas injection system is used to deposit a platinum marker on a random portion of the exposed SiC ring. This marker is used as a guide to mill the surrounding material away to expose a surface segment of the SiC layer between the IPyC and the OPyC layers that was not exposed to the potential grinding damage in the original cross-section preparation. The segment of interest for EBSD analysis is the side face of the wedge formed by cutting into the surface on either side of the platinum rectangle (see Figure 4).



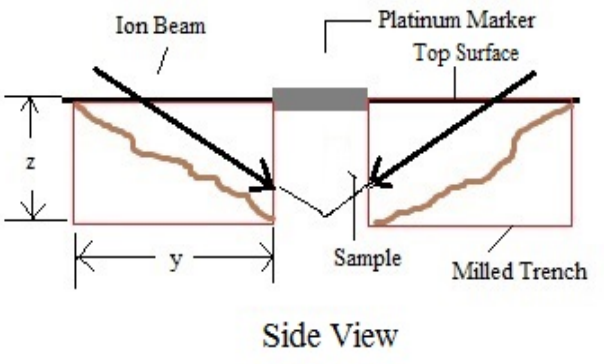


Figure 4. (top) SEM image of the dimension settings for a gas injection system-deposited platinum marker used in preparation for ion beam milling segment extraction of the sample SiC layer, and (bottom) FIB sample extraction step sketch. The electron beam, not shown in the image, is normally oriented to the sample surface.

To reach the desired 35-μm depth from the top face of the cross-sectioned particle, wedge-shaped trenches are excavated from the material, starting just above and below the platinum marker, while the stage is tilted at 52 degrees. At this angle (i.e., 52 degrees from the electron beam), the ion beam is normal to the sample surface, with beam coincidence at the sample eucentric height of about 10 mm. The tilt is executed to make room for the FIB to cut the bottom sample surface on both sides at a 52-degree angle while the sample is in the upright position. The volume to be milled is programmed into the system as a box. If a depth of 35 μm is desired, the upper box surface rectangle would need to be drawn with sufficient dimensions to compensate for redeposition of milled material that is not carried away in the vacuum system. When the dimensions of the upper rectangular layer of the trench are established using patterning tools, the ion beam is ready to raster across the chosen rectangle to sputter material as it progresses.

Debris build-up is a common occurrence in deep trenches and is sometimes termed re-deposition curtain. The *x*-dimension of the trench is set by visual inspection. The *y*-dimension in this instance was given a dimension value of one and a half times the desired depth. Figure 5 illustrates the beginning phase of milling and the completed trenches for a SiC layer sample extraction of a TRISO particle.

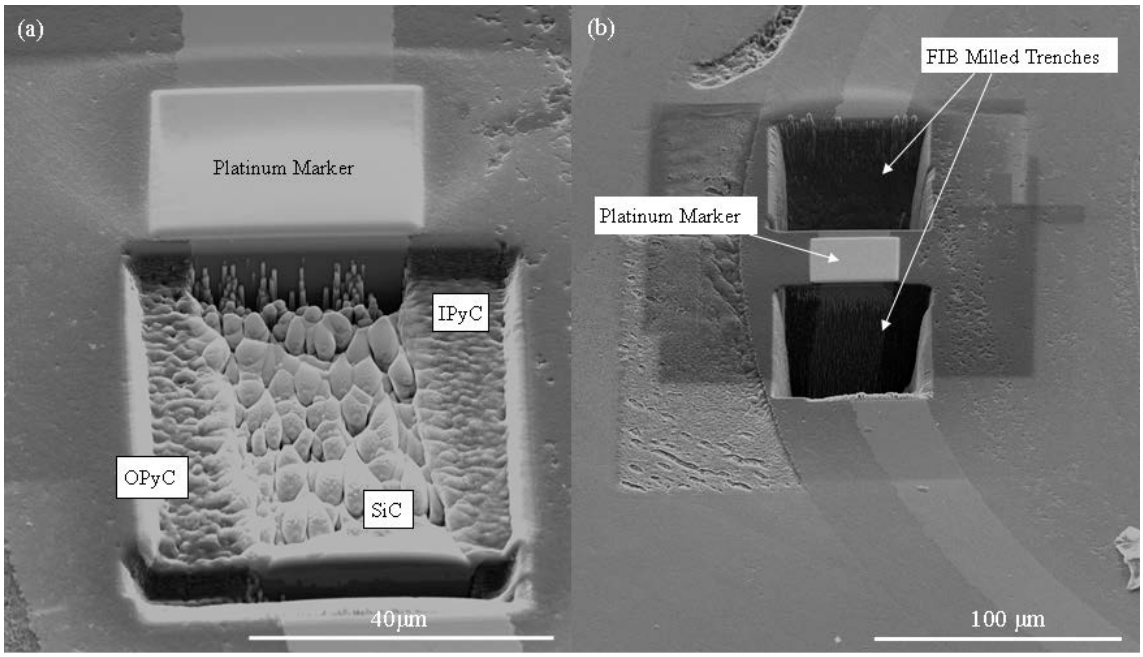


Figure 5. SEM images (a) at the beginning stage of ion beam bulk milling for sample extraction of an SiC layer from a TRISO-coated particle and (b) at completion of bulk-milled trenches above and below the platinum marker.

Note the difference in milling morphology between the surrounding pyrocarbon layers and the SiC layer in Figure 5(a). The SiC layer grains appear to have a more spherical shape compared to the long columnar grains along the radial direction that is typical of the chemical vapor deposition-coating process. Also, the SiC grains do not appear to increase in size from the IPyC layer to the OPyC layer.

Figure 6(a) shows the resulting debris formation in the bulk milled trenches and the redeposition of material onto the surface of interest. The specimen platform is tilted to 52 degrees to achieve a 90-degree angle between the ion beam and specimen surface during bulk milling. To clean the debris off the face of interest, the platform is tilted an additional 2 to 54 degrees. A small rectangle is drawn near the edge of the platinum marker with a patterning tool and the ion beam is employed to sputter material to the required depth within the chosen area. This procedure exposes the SiC layer (see Figure 6(b)).

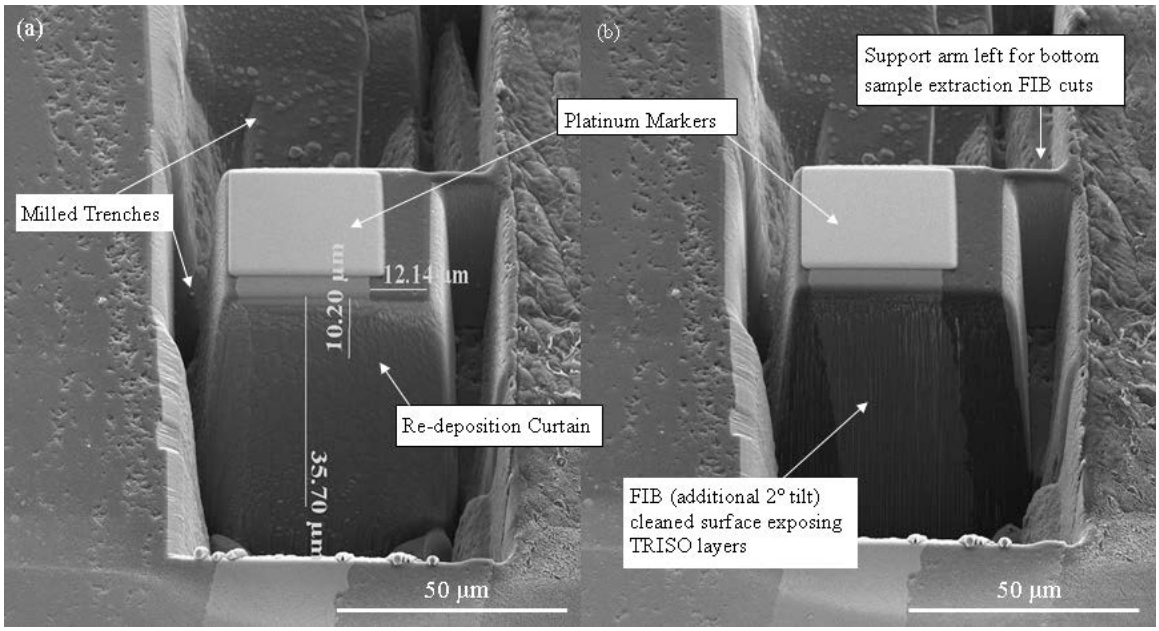


Figure 6. SEM images of TRISO-coated particle SiC layer sample (a) after bulk-milled trenches and side cuts and (b) after ion beam milling of a debris curtain from the surface of interest at a specimen tilt angle of 54 degrees from the vertical.

As shown in Figure 6, a small arm of material is left on one side to stabilize the specimen during cutting of the bottom portion for final sample separation from the specimen.

For cutting, the stage is tilted back to the upright position. This will allow the ion beam, which is at an angle of 52 degrees with respect to the vertical, to slice segments at the bottom of the sample in preparation for removal. Figure 4 shows a sketch illustrating this step. The ion beam consecutively passes unhindered through each milled, wedge-shaped trench to form a V-shaped cut into the bottom of the sample when viewed from the side. The sample is rotated 180 degrees between the two ion beam cuts. The arm on one side continues to hold the sample in place while an OMNI probe needle used for lift-out is welded to the sample using the platinum deposition feature.

EBSD analysis requires that the normal surface of evaluation be tilted 70 degrees with respect to the vertical. The stage control will not tilt the full 70 degrees. Attaching the sample to a grid clamped into place by a 45-degree pre-tilted mount accommodates this requirement. The next step welds the extracted sample onto a copper half grid that contains a series of labeled posts for attaching FIB lift-outs. The stage is then tilted 7 degrees. This adjustment, plus the pre-tilt of 45 degrees, puts the sample in position for FIB polishing of the sample surface (i.e., close to parallel with the ion beam). A series of gradually descending currents (30 nA, 15 nA, 7 nA, 1 nA, 0.3 nA, 0.1 nA, and 48 pA at 5 keV) was used to achieve a gradual reduction in hills and valleys on the sample surface for a smooth finish. The sample surface before and after FIB polishing is shown in Figure 8. Sample extraction, polishing, and EBSD scanning were performed in the same chamber.

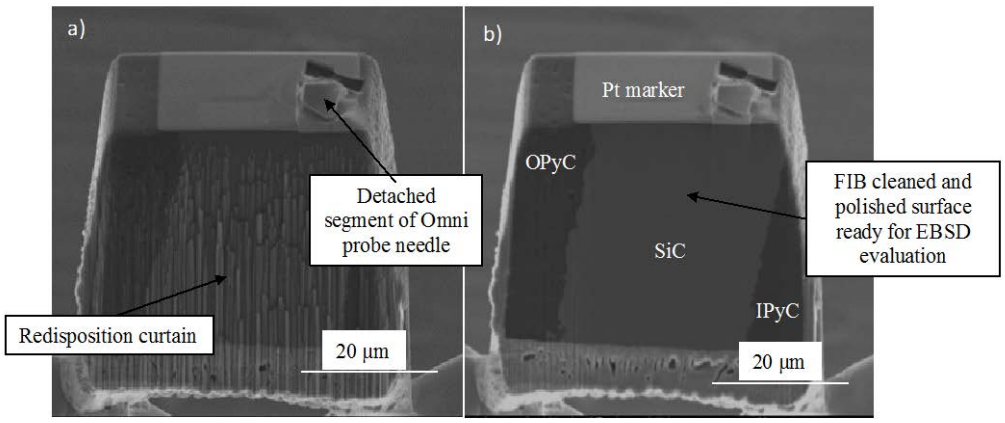


Figure 7. SEM images of the sample surface (a) before FIB polishing and (b) after FIB polishing

2.3 EBSD Data Collection Parameters

For all samples prepared via traditional polishing, grains at the IPyC-SiC and OPyC-SiC boundaries were excluded from the EBSD analyses due to lack of clarity from rounded edges.

A fourth data set was obtained from a sample prepared with the FIB method described above. Boundary grains were included in the analysis.

The traditionally polished samples were analyzed using an FEI Quanta 650 FEG FIB-SEM and the FIB polished samples were analyzed with an FEI Quanta 3D FEG FIB-SEM. Both instruments were equipped with a TSL Hikari EBSD system. All EBSD scans were performed using a hexagonal grid pattern to maximize the number of data points collected within the scanned area. The area for the EBSD analysis was selected with an initial step size of 50 nm for the first measurement and then standardized at 0.1 µm for the mapping chosen for all subsequent analyses. The scanned area was approximately 13 µm x 36 µm for the traditionally polished samples and was 45.70 µm x 31.70 µm for the FIB-polished samples. The accelerating voltage was 20 kV. All data were cleaned via grain Confidence Index (CI) standardization and neighbor orientation.

3.0 RESULTS AND DISCUSSION

The results for the EBSD analysis of the SiC layer samples of the same particle batch (Eref) are presented. Three sets of data were obtained from samples prepared via traditional polishing. Two of these three data sets were from different locations in the same particle (Eref 1.1 and 1.2) and were compared to results from a second particle (Eref2) to investigate the extent that measurements at one location in one particle of a batch were representative of batch properties.

Figures 8 and 9 contain grain size, character, and orientation data for the SiC layer of TRISO-coated particles from the same batch (Eref) prepared by traditional polishing (Eref 1.1, 1.2 and 2) or FIB (Eref3FIB).

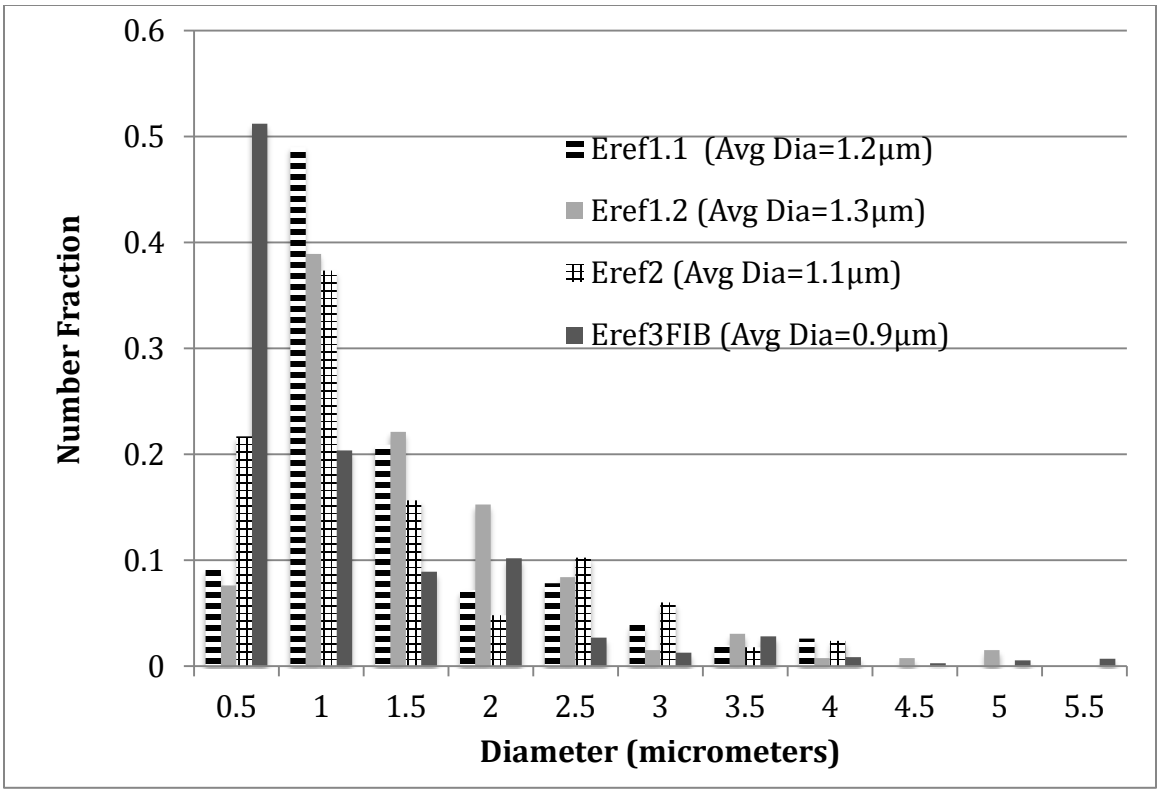


Figure 8. SiC layer grain size (i.e., diameter) distribution via EBSD analysis of coated particles Eref1, Locations 1 and 2 and Eref2, prepared with traditional polishing, and ErefFIB, prepared via FIB.s

While three sets of polished sample diameter data did not allow for a conclusive determination, it was clear that these data do not rule out the possibility that limited particle measurements can represent the properties of a batch. The average SiC grain diameter according to these data was 1.2 µm, with a spread of only ±0.1 µm. In contrast, the average diameter determined via analysis of the FIB-prepared sample was 0.90 µm, which was lower than the three-measurement average by 25%. The reason for the lower average diameter value may be inclusion of edge (i.e., boundary) grain sizes. These grains appear to be smaller than those toward the center of the SiC layer (see Figure 9d.)

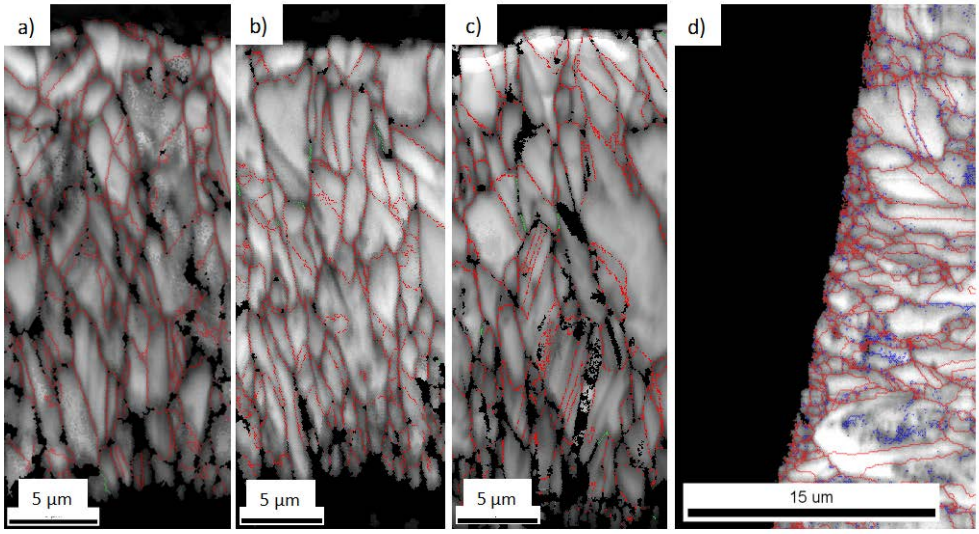


Figure 9. EBSD images of SiC layer grains in samples (a) Eref1, Location 1, (b) Eref1, Location 2, (c) Eref2, all prepared with traditional polishing, and (d) ErefFIB, prepared via FIB.

In the EBSD images of traditionally polished samples (Figure 10a through 10c), the pyrocarbon layers would appear at the top and bottom of the figure; however, because SiC layer edge grains were excluded from the analysis, the dark areas at the top and bottom are merely an artifact of the analysis rather than an indication of the start of a pyrocarbon layer. In contrast, the transition between SiC and pyrocarbon layer (from right to left) and the SiC layer edge grains are clear in the FIB sample (see Figure 9d). While the orientation of the polished and FIB samples is different, it should be noted that the SiC layers are theoretically isotropic.

Grain boundary character distributions for each sample are presented in Figures 10 and 11. A statistically significant difference is noted in the SiC grain characteristics determined via analysis of the polished samples (Figure 10) and those determined from the FIB-prepared sample (Figure 11). Most significant is the difference in the relative quantity of low angle grain boundaries, which comprise 25% in the FIB-prepared sample (Figure 11) and only 1-2% in the polished sample (Figure 10). This difference is almost certainly due to the exclusion of layer edge grains in the polished sample analysis. The SiC grains at the pyrocarbon boundaries are notably smaller than those at the layer interior, which is clearly visible in Figure 10d.

For all samples, the percentage of Coincidence Site Lattice (CSL) grain boundaries was the single largest category. For the polished samples, the CSL category was a majority (i.e., ranging from 52 to 72%); however, it was only 40% for the FIB-prepared sample. Exclusion of layer edge grains from the polished sample analysis may have biased the grain boundary character data to a relatively larger average percentage of CSL boundaries. The spread of 52 to 72% of CSL grain boundaries in the polished samples may be attributed to an insufficient or non-homogenous polishing technique, and is not necessarily a variation in coated particle batch parameters. Future analysis and interpretation will include binning the data as a function of distance from the interface.

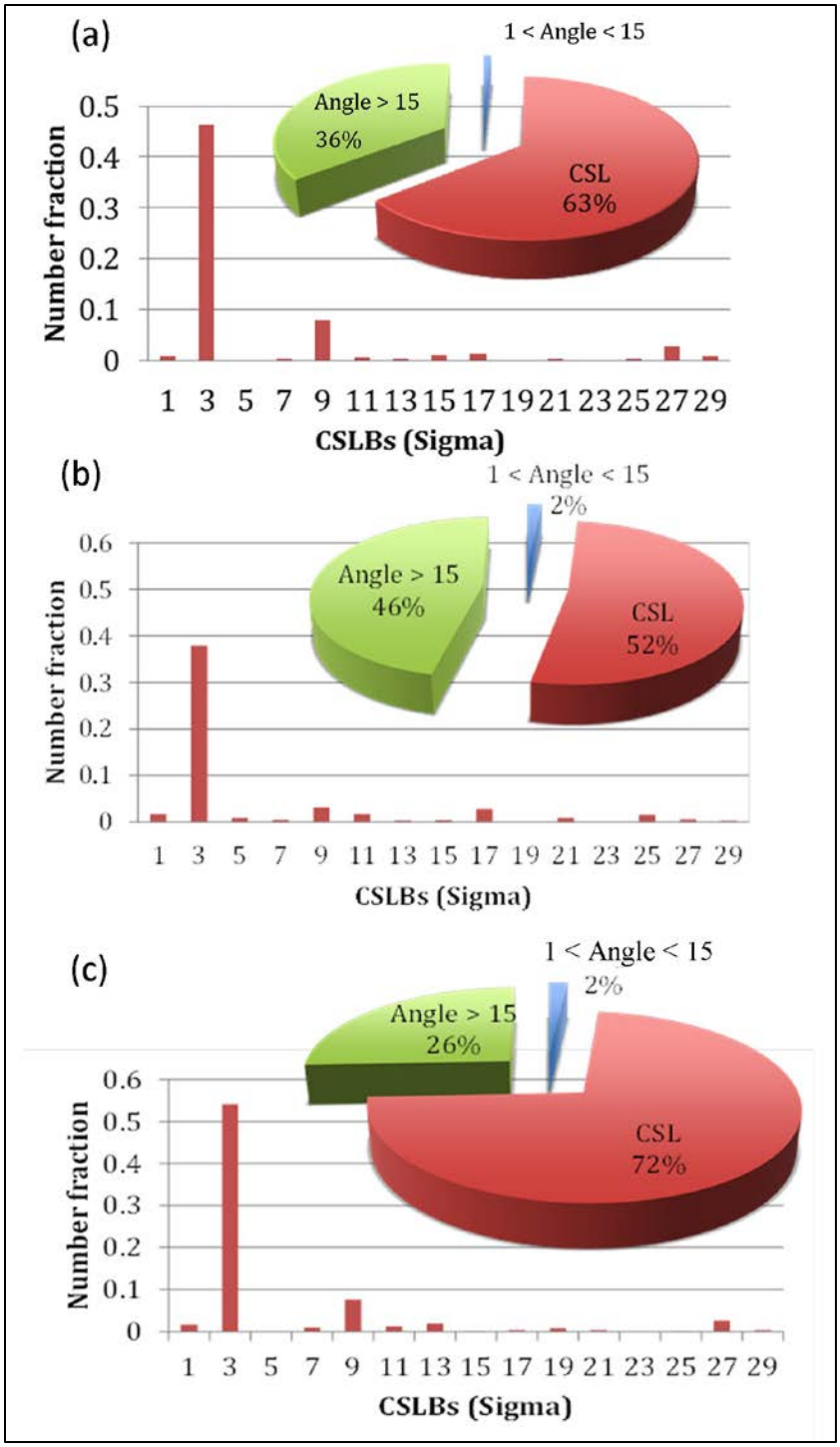


Figure 10. SiC layer grain boundary character distribution via EBSD analysis of (a) coated particle Eref1, Location 1, prepared with traditional polishing, (b) coated particle Eref1, Location 2, prepared with traditional polishing, and (c) coated particle Eref2, prepared with traditional polishing.

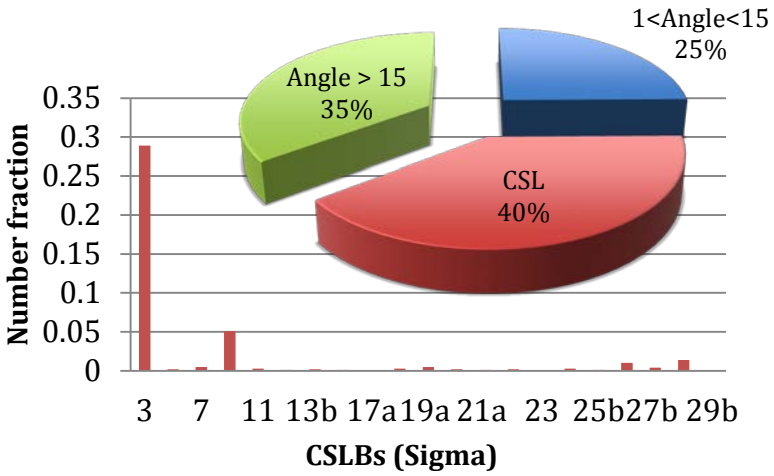


Figure 11. SiC layer grain boundary character distribution via EBSD analysis of the coated particle ErefFIB that was prepared via the FIB method.

Figure 12 shows the pole figures for each SiC analysis data set. In these figures, the scale represents multiples of a random distribution in that sample. While the results indicate none of the samples is particularly textured, there is a notable difference between, the relative intensities of grain orientations for the polished samples (maximum range 11 to 13) and those for the FIB-prepared sample (maximum = 4). This implies that a larger percentage of grains in the polished samples have a particular orientation and that the FIB–prepared sample is less textured than the polished samples. Again, this difference may be due to including layer edge grains in the FIB sample. The edge grains in Figure 9d are not only smaller than grains deeper in the SiC layer, but also less consistently oriented. A dominant SiC grain orientation is undesirable because grain boundary migration of fission product species would be facilitated.

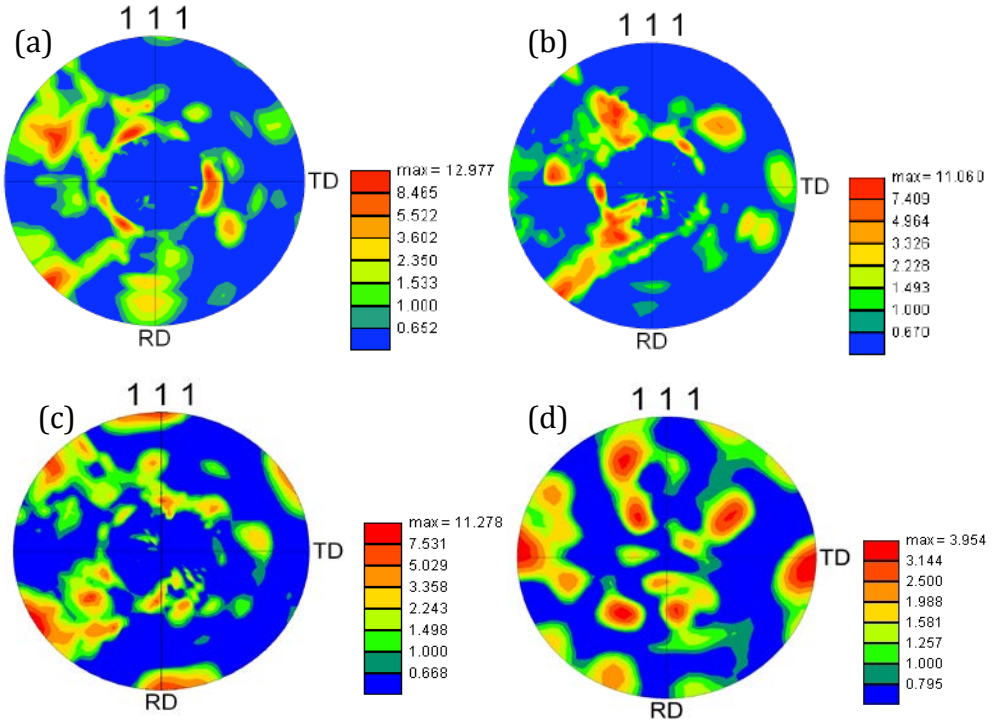


Figure 12. SiC layer crystal orientation in the 111-direction via EBSD analysis of coated particle (a) Eref1, Location 1, (b) Eref1, Location 2, and (c) Eref2, prepared with traditional polishing and (d) ErefFIB, prepared via FIB method. (TD = tangent to the surface of the particle in the plane of the cross-section and RD = normal to the plane of the cross section.)

4.0 SUMMARY AND CONCLUSIONS

A method of representative sample milling and polishing for damaged and dissimilar layers using FIB extraction has been demonstrated and shows promise for materials containing components with different hardness and for future application to neutron damaged materials. The material edge rounding and damage that occurs from traditional sample polishing can be avoided, resulting in more complete and accurate sampling.

In addition to demonstrating the effects the different sample preparation techniques had on the quality of characterization results, comparative grain character analyses of TRISO particles from the same production batch are reported. Three of the data sets were obtained from traditionally polished SiC layers, with the edges rounded due to the polishing process. As such the edge grains were not included in the final data analysis. In contrast, the FIB-milled, polished, and extracted sample had pristine edges and all edge grains were included. In addition, the small size of the extracted sample makes possible analyses on neutron-irradiated material, which is the fate of the TRISO particles in this study. Small samples can be difficult to align properly for EBSD analysis; therefore, pre-tilted mounts allow for reliable and easier alignment of the SiC layer samples, resulting in viable analyses.

While more data are required to make a conclusive statement about the cause of differences in the resulting data, it would seem that including the edge grains had a significant impact on the average grain characteristics.

This study showed that the average grain sizes of samples can be similar while the grain boundary characteristics differ significantly. Furthermore, it was found that low-angle grain boundaries comprise 25% in the FIB-prepared sample versus only 1 to 2% in the polished sample from same

particle. From this study, it was determined that the results of characterizing FIB prepared samples provide more repeatable results because the effects of sample preparation are eliminated.

ACKNOWLEDGEMENTS

This work was sponsored by the U.S. Department of Energy, Office of Nuclear Energy, under U.S. Department of Energy Idaho Operations Office Contract DE-AC07-05ID14517, as part of the Very High-Temperature Reactor Development Program and as part of an Advanced Test Reactor Nuclear Science User Facilities Experiment. James I. Cole is thanked for his review of this paper.

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