

## Characterization of Stresses in the SiC Layer of TRISO Coated Nuclear Fuels using Raman Spectroscopy and EBSD

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Tri-isotropic (TRISO) coated fuel particles are being developed for use in high temperature gas-cooled reactors. TRISO coated particles consist of an oxide kernel ~500  $\mu\text{m}$  in diameter surrounded by four layers: a buffer, which consists of low density carbon; an inner pyrolytic carbon (IPyC) layer; silicon carbide (SiC); and an outer pyrolytic carbon (OPyC) layer [1]. A backscattered SEM image of a TRISO particle cross section is shown in Fig. 1. The SiC layer is of particular interest as a pressure vessel for containing fission products within each particle. A full understanding of the SiC mechanical and structural properties in as-deposited and irradiated is essential in order to qualify this material for high burnup applications. In this work, Raman spectroscopy and electron backscattered diffraction (EBSD) were used to characterize the residual strain of the SiC layer in as-deposited and after indentation states.

Raman spectroscopy was performed on the SiC layer using a confocal  $\mu$ -Raman system. A characteristic Raman spectrum of the SiC layer is shown in Fig. 1. The peaks at  $796\text{ cm}^{-1}$  and  $972\text{ cm}^{-1}$  correspond to  $\beta$ -SiC [2]. Using a combination of Raman spectroscopy and FIB-prepared EBSD surfaces, the relative compressive and tensile strains in the SiC lattice were quantified by measuring the shift of the characteristic peaks for SiC or through variations in the calculated d-spacings of the SiC. Close analysis of the Raman spectra indicated that only the cubic polymorph of as-deposited SiC ( $\beta$ -SiC) was present. This polymorph is desired in nuclear applications because of resistance to irradiation swelling [3]. The residual strain in the SiC layer, which results from the CVD process is related to deposition temperature and carrier gas, and may contribute to the mechanical failure of the layer, and thus the release of fission products. As-deposited residual strains were significantly in tension, as determined by Raman and EBSD. These residual strains may contribute to premature failure of the SiC and subsequent release of fission products.

Further mechanical response of the SiC layer was probed by placing indentations over a range of lengths scales. Quantification of the Raman shift in the x, y, and z directions as well as EBSD patterns around the indents illustrated changes in strain from a tensile state to either a compressive state beneath the indent or a completely relaxed state on the edges of the indent. EBSD also illustrated crack propagation from the indents via transgranular mechanisms as shown in Fig. 2. Future investigations of post-irradiated samples are planned and will be compared directly with the as-deposited states.

### References

- [1] D. A. Petti, et al., INEEL / EXT-02-00300 (2002).
- [2] A.S. Nakashima and H. Harima, *Phys. Stat. Sol.* 162, (1997) 39.
- [3] L. Snead, et al., *J. Nucl. Matl.* 371 (2007) 329.

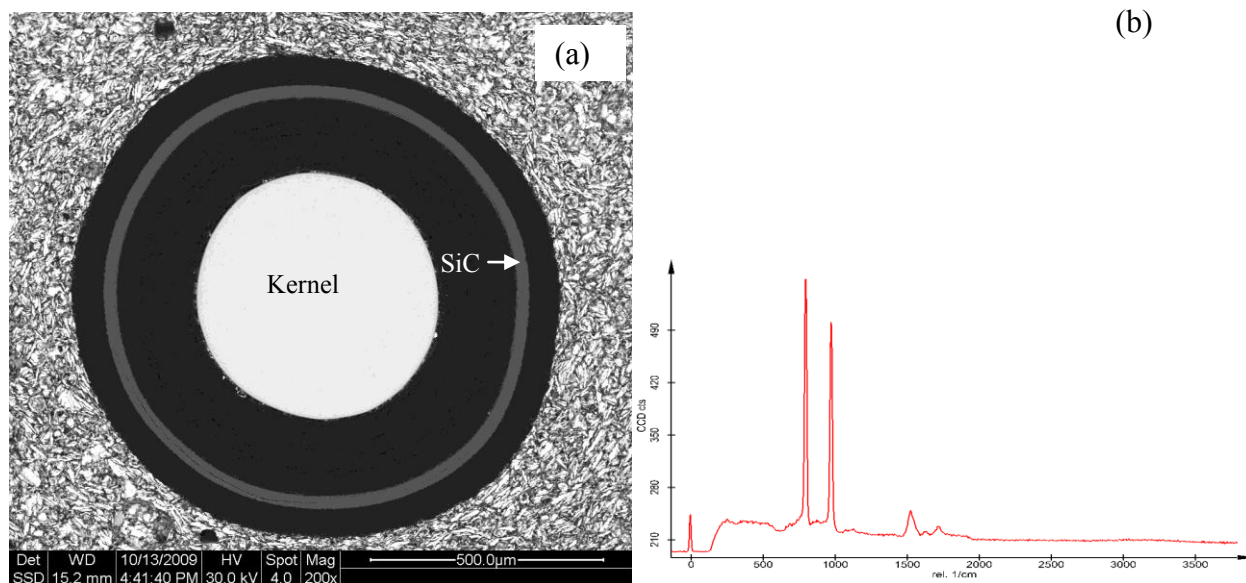


Fig. 1. Backscattered scanning electron image of a TRISO coated fuel cross-section and illustrating the SiC layer. A representative Raman spectrum is also shown. The TO and LO bands for  $\beta$ -SiC are at  $796\text{ cm}^{-1}$  and  $972\text{ cm}^{-1}$  wave number.

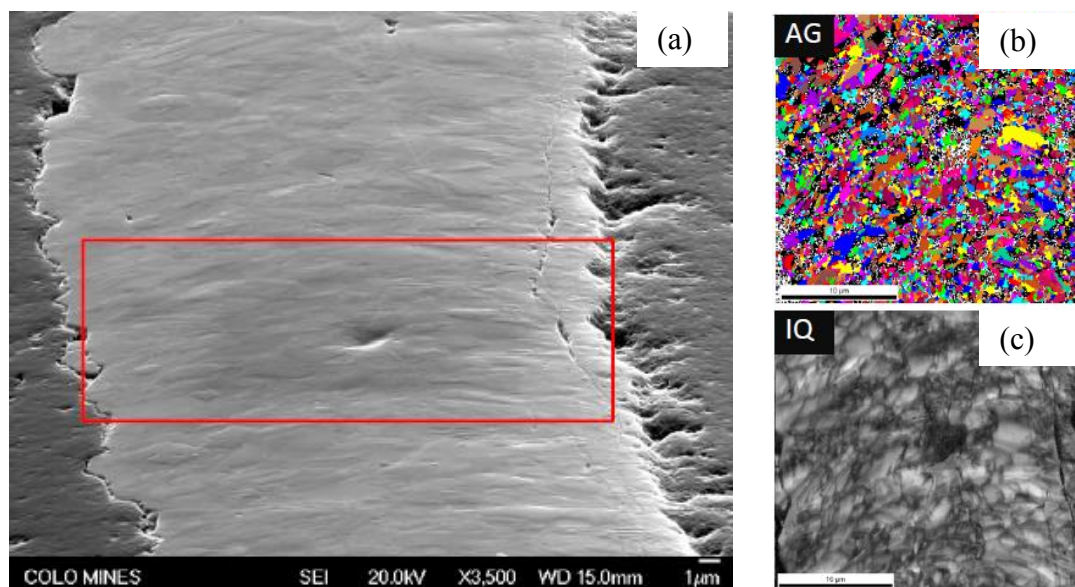


Fig. 2. (a) SEM image of the SiC layer after microindention at the location where EBSD and Raman analyses was performed. (b) Individual grain map of the outlined region in (a) showing the  $\beta$ -SiC grains. (c) Image quality map overlaid with a grain boundary map showing the transgranular crack propagation mode.