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Identifying Chemical Disordering in Irradiated SiC Fiber-Reinforced SiC Matrix Composites with High-Throughput Correlative Microscopy

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Continuous silicon carbide (SiC) fiber-reinforced, SiC matrix (SiC_f/SiC_m) composites have exceptional properties. They can withstand extreme conditions serving as an advantageous nuclear grade structural component such as superior high temperature strength, low neutron absorption, low activation, good chemical and dimensional stability, and decent radiation tolerance for fusion applications. This work aims to apply a systematic microscopy/spectroscopy characterization to elucidate the radiation-induced evolution of microstructure and microchemistry of these composites by the combination of multiple microscopy characterization techniques (also known as correlative microscopy), including Raman spectroscopy, scanning electron microscopy (SEM), high resolution transmission electron microscopy (HRTEM), scanning TEM Energy-dispersive X-ray spectroscopy (STEM-EDS), and transmission Kikuchi Diffraction (TKD). Correlative microscopy plays an important role in deciphering the fundamental mechanisms of materials degradation in extreme environments.

This work focused on characterization of irradiated SiC/SiC composites, which were neutron-irradiated at ~300°C up to ~44 displacements per atom (dpa) in the High Flux Isotope Reactor (HFIR) at Oak Ridge National Laboratory (ORNL). The post irradiation examination was performed in the Low Activation Materials Design and Analysis Laboratory (LAMDA) at ORNL. The room-temperature micro-Raman scattering measurements were conducted on both as-recieved and irradiated SiC/SiC composites using a confocal LabRAM HR Evolution, Horiba Scientific Raman spectroscope. A Tescan MIRA3 GMH equipped with an electron backscatter diffraction (EBSD) system (Oxford Instruments) was used for SEM imaging and TKD. A Thermo Fisher Scientific Versa 3D dual-beam SEM/focused ion beam (FIB) was used to extract TEM lift-out lamella from specimens before and after irradiation for TEM and TKD characterization. A Thermo Fisher Scientific Talos F200X FEG-STEM was used for STEM-EDS mapping for chemical composition. A self-written machine-learning (ML) code was applied to analyze the STEM-EDS data cube as a high-throughput analytical microscopy tool.

Raman spectroscopy has shown the Si-Si peak fitted by a Lorentzian function with sharp peak at full width half maximum (FWHM) of 10 cm^{-1}. Based on phonon confinement model, the peak width suggests coherent domain size of 5-10 nm (Kanemitsu et al., 1993). The line position and shape suggest Si atoms are in a form of nanocrystalline instead of amorphous having a peak position at 480-475cm^{-1} (Yogi et al., 2018). Since Si-Si peak was not found by ion irradiation study (Huguet-Garcia et al., 2014), we also applied another microscopy method to reveal such disordering of Si atoms at a higher magnification using TEM. The ML processed STEM-EDS maps on Si (Figure 1 and Figure 2) inside random single grains denoised the artifacts and low-counting background signals. Therefore, the analytical analysis suggests that generation of homonuclear bonds is more significant in the fiber than the matrix (Figure 2(b)) [4].

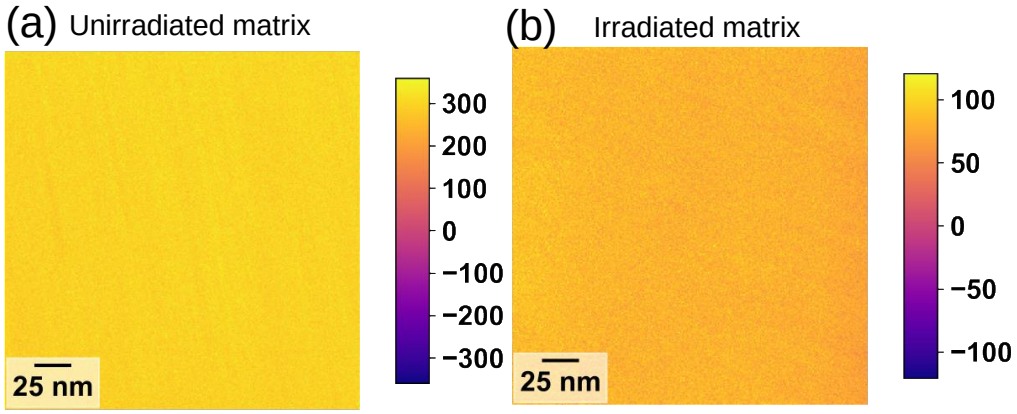

Figure 1. ML processed Si mapping of the (a) unirradiated and (b) irradiated CVI SiC matrix, respectively. The SiC/SiC composite is under neutron irradiation with a dose 44 dpa and temperature ~300°C.

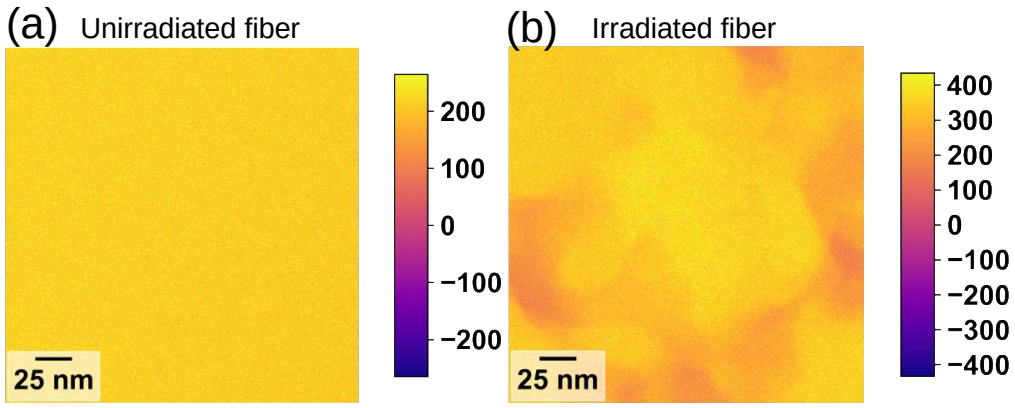

Figure 2. ML processed Si mapping of the (a) unirradiated and (b) irradiated SiC fiber core, respectively. The SiC/SiC composite is under neutron irradiation with a dose 44 dpa and temperature ~300°C.

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https://pubs.acs.org/doi/abs/10.1021/acs.analchem.8b01352 (Accessed February 24, 2022).
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