3.5 HIGH DOSE NEUTRON IRRADIATION OF HI-NICALON TYPE S SILICON CARBIDE COMPOSITES, PART 1: MICROSTRUCTURAL EVALUATIONS - A. G. Perez-Bergquist, T. Nozawa, C. Shih, K. J. Leonard, L. L. Snead, Y. Katoh (Oak Ridge National Laboratory)

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ABSTRACT

Over the past decade, significant progress has been made in the development of silicon carbide (SiC) composites, composed of near-stoichiometric SiC fibers embedded in a crystalline SiC matrix, to the point that such materials can now be considered nuclear grade. Recent neutron irradiation studies of Hi-Nicalon Type S SiC composites showed excellent radiation response at damage levels of 30-40 dpa at temperatures of 300-800°C. However, more recent studies of these same fiber composites irradiated to damage levels of >70 dpa at similar temperatures showed a marked decrease in ultimate flexural strength, particularly at 300°C. Here, electron microscopy is used to analyze the microstructural evolution of these irradiated composites in order to investigate the cause of the degradation. While minimal changes were observed in Hi-Nicalon Type S SiC composites irradiated at 800°C, substantial microstructural evolution is observed in those irradiated at 300°C. Specifically, carbonaceous particles in the fibers grew by 25% compared to the virgin case, and severe cracking occurred at interphase layers.

HIGHLIGHTS

- Hi-Nicalon Type S SiC fiber composites were neutron irradiated to >70 dpa at 300, 500, and 800°C.
- The composites irradiated at low temperature showed substantial degradation in ultimate flexural strength. Strengths dropped from 335 MPa in the unirradiated state to 205 MPa at 800°C, 200 MPa at 500°C, and 95 MPa at 300°C. Similarly, proportional limit stress dropped to 128 MPa at 500°C and 91 MPa at 300°C.
- Fibers exhibited no change in phase as measured by electron diffraction, and size of SiC grains within the fibers was consistent before and after irradiation.
- Fibers were found to contain excess free carbon in small pockets evenly distributed throughout. These carbonaceous pockets were found to increase in size with irradiation at low temperature, from 14.8 nm in diameter in the unirradiated condition to 18.5 nm in diameter after 300°C irradiation. Pockets did not grow after 800°C irradiation.
- EELS analysis showed a slight shift in the carbon spectrum between the unirradiated, 300, 500, and 800°C samples, indicating a possible phase shift in the carbon located in the amorphous pockets.
- Interphase layers were not noticeably changed after 800°C irradiation, but layers at 300°C exhibited severe cracking and degradation. SiC layers were partially amorphized and showed a clear drop in thickness. PyC layers had changed from turbostratic to amorphous and had grown at the expense of the SiC layers.

Matrix regions surrounding the SiC fibers appeared unchanged in all irradiated samples, with no cracking, porosity, phase changes, or severe microstructural changes or accumulation of defect structures.

REFERENCE

[1] A. G. Perez-Bergquist, T. Nozawa, C. Shih, K. J. Leonard, L. L. Snead, Y. Katoh, High Dose Neutron Irradiation of Hi-Nicalon Type S Silicon Carbide Composites, Part 1: Microstructural Evaluations. J. Nucl. Mater., In Revisions.