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TEM Investigation of Implanted Ag Distribution in PyC and SiC after Annealing

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Containment of fission products (FP) within the TRISO fuel particle is critical to the success of the Very High Temperature Reactor (VHTR). In these particles, the SiC layer provides both structural support and is the primary diffusion barrier for FPs. While the behavior of the FPs, e.g. Ag, is of interest, it appears to be able to penetrate through the SiC and PyC layer in the TRISO fuel most easily [1, 2]. An understanding of the behavior transport mechanisms of FPs through the TRISO layers is needed to determine how it occurs so that mitigation measures can be devised in both normal and off-normal operating conditions. This work is aimed to determine the diffusivity behavior of key FP, e.g. Ag, through SiC and PyC both thermally, under irradiation, and under stress using FP introduction techniques.

The experimental approach in this study is to fully cap a PyC/SiC multi-layer on a SiC substrate to ensure retention of Ag introduced by ion implantation during high temperature annealing studies, to help determining the transport mechanisms of Ag as one of the key FPs through PyC and SiC by thermal diffusion. A PyC layer was deposited onto SiC, serves as a host layer, then FP Ag was implanted with a 400kV ion implanter into the PyC layer to a concentration of 1 at% at the peak, and then a Si/C coating layer was deposited onto all exposed sides of the PyC. The concentration profile of as-implanted Ag in PyC has been calculated by the SRIM code and overlaid in the TEM images of Figures 1 and 2. The sample was then annealed at 1100°C for 10 hrs to allow redistribution of the implanted Ag. Cross-sectional specimens for TEM/STEM observations were prepared via the focused ion beam (FIB) lift out technique, to examine the depth distribution of FP Ag throughout the PyC/SiC couple, and to detect any precipitation of Ag and the location of these precipitates in the sample.

Bright field (BF) TEM and high angle annular dark field (HAADF) STEM images were taken with a 200-kV JEM-2010F TEM/STEM electron microscope. Figure 1(a) shows the whole multi-layer cross-section for sample 1. It is clear that Ag nanoparticles accumulated at the implantation peak depth in the PyC layer. Figure 1(b), (c) and (d) show HAADF STEM images of selected area from Figure 1 (a). The HAADF STEM image shows the presence of Ag nanoparticles at the interface between Si/C coating and PyC layers by a strong Z-dependent image contrasts (Z: Ag = 47, Si = 14, C = 6), but not on the interface between PyC coating layer and the lower SiC substrate. The multi-layers' electron diffraction patterns were shown in Figure 1(e), (f), (g) and (h) from the corresponding areas of sample 1. It is very interesting that the PyC coating layer is separated into two layers due to irradiation-induced restructuring of the PyC layer with the irradiated regions showing a much high level of texture. Figure 2(a) shows the whole multi-layer cross-section for sample 2. In this sample, Ag naoparticles were not observed in the peak Ag range in PyC layer, due to the small thickness of the layer and all implanted Ag in the PyC has apparently migrated to the upper or lower interfaces. Figure 2(b) and (c) are HAADF STEM images of selected area from Figure 2(a). It can be clearly seen that the presence of Ag nanoparticles on both two interfaces, one between Si/C coating and the PyC layer, and the other between PyC coating layer and the SiC substrate. It should be noted that all Ag nanoparticles are actually in either the Si/C coating or the SiC substrate side of the interface with none in the PyC layer. The multi-layers' electron diffraction patterns were shown in Figure 2(d), (e) and (f) from the responding area of sample 2. Behavior of other implanted FPs in the simulated TRISO layers is also under investigation.

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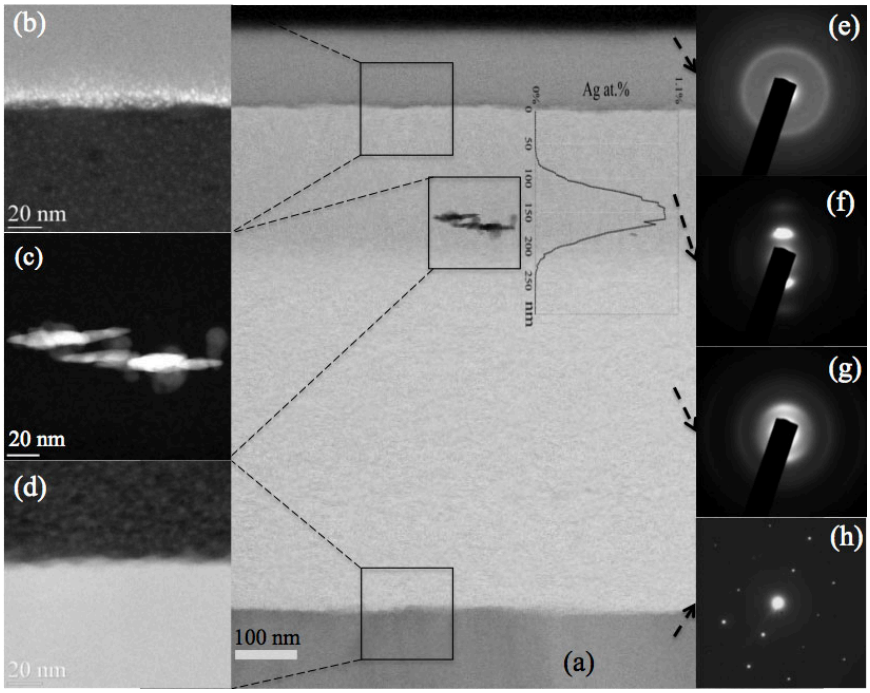

Figure 1. (a) Cross-sectional TEM bright field image, (b), (c) and (d) HAADF STEM images from selected area, (e), (f) (g) and (h) selected area electron diffraction (SAED) from responding area of sample 1.

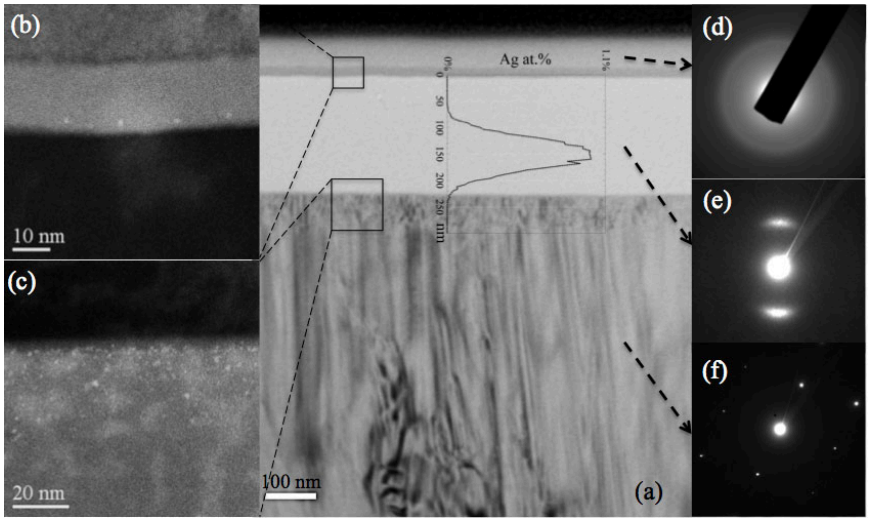

Figure 2. (a) Cross-sectional TEM bright field image, (b) and (c) HAADF STEM images from selected area, (d), (e) and (f) selected area electron diffraction (SAED) from responding area of sample 2.

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