Assessment of Oxygen Uptake in Ion Irradiated ZrC using Transmission Electron Microscopy Techniques

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Post-irradiation examination (PIE) of ZrC specimens irradiated with 10 MeV Au^{3+} at 800^o C was conducted using transmission electron microscopy (TEM) techniques. The microstructural changes were systematically investigated in samples irradiated to doses ranging from 0 to 30 dpa. The TEM specimens were prepared by FIB-lift out method and diffraction imaging techniques were used to investigate the nature of the radiation-induced defects. Figure 1 shows bright field images obtained along the (110) zone axis for samples irradiated to 0.5, 5 and 30 dpa. At low doses (0.5 dpa), "black dot" defects were observed in the microstructure. Increasing the dose resulted in the formation of dislocation loops at 5 dpa and entangled dislocation networks at 30 dpa. Based on these results, complemented with XRD and Raman measurements, it was possible to unveil the underpinning mechanisms driving the radiation response of ZrC at high temperatures. This is an important contribution to the current state of art of the behavior of ultra-high temperature ceramic materials in extreme environments such those encountered in gas-cooled nuclear reactors. The TEM obtained data was processed and submitted in a manuscript titled **"The irradiation response of ZrC ceramics at 800^o C"** to the Journal of the European Ceramic Society.

In addition to the defects observed in the irradiated ZrC samples, SEM/TEM analysis of the specimens showed that concurrent oxidation occurred during ion implantation at 800^o C. A secondary electron image of the top surface of one samples, showing the characteristic ZrO_2 nodules that were formed in the surface, is shown in Figure. 2. Morphological and microstructural changes in the nanometric oxide nodules were investigated as a function of dose using HRTEM and EELS compositional analysis. It was found that radiation shifted the size distribution of the oxide nodules to lower sizes and induced the formation of faceted cavities at high doses (Figure 3). Differences in the crystalline structure of the oxides were also noted for samples irradiated at different doses. All these results were compiled in a manuscript titled "**Early stage oxidation of ZrC_x under ion-irradiation at elevated temperatures"** that will be submitted to the Journal of Corrosion Science.

Finally, during the TEM analysis it was noted that prolong e-beam exposure induced microstructural changes in the specimen. A series of e-beam irradiation were conducted at the TEM to investigate the nature of these changes and their underlying driving mechanism. The result are being putting together in a manuscript that will be submitted to the Journal of Nuclear Materials.

Figures

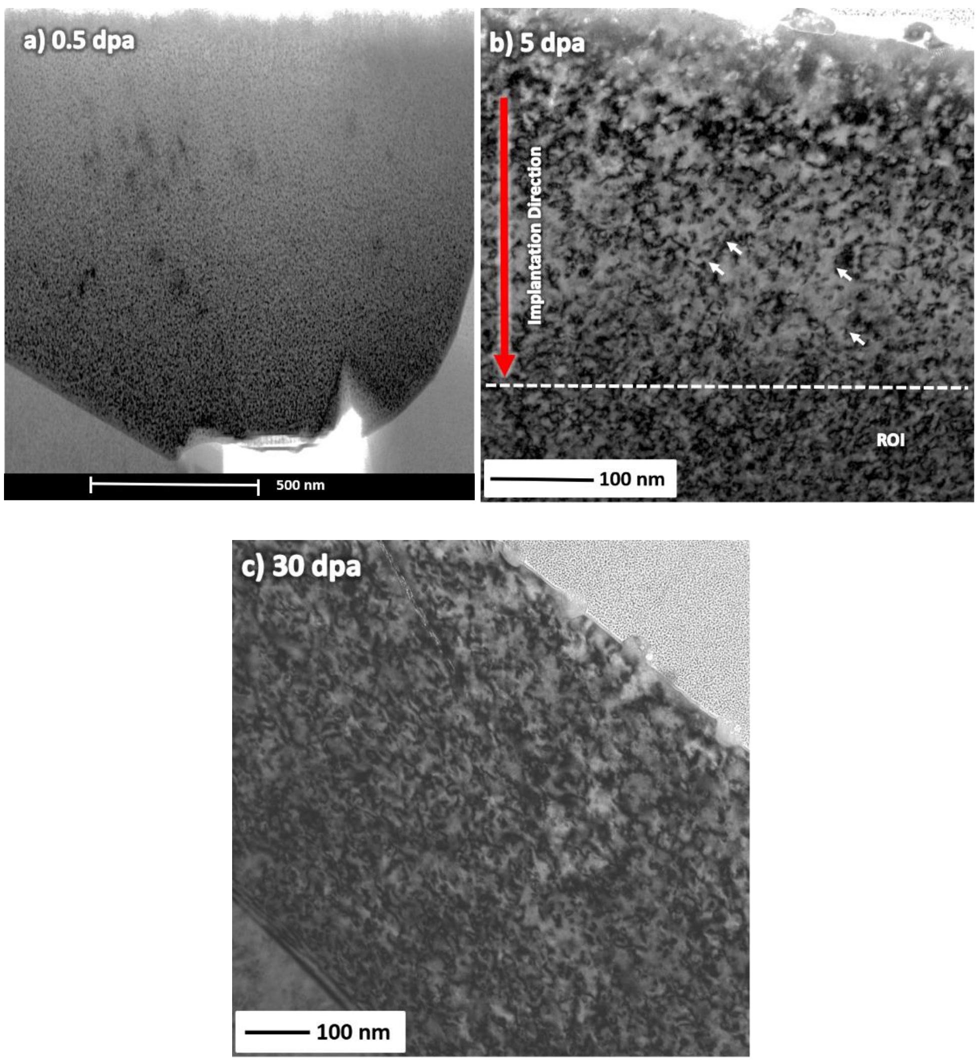


Figure 1. Bright Field Images of Irradiated Microstructure for ZrC specimens irradiated at 0.5, 5 and 30 dpa.

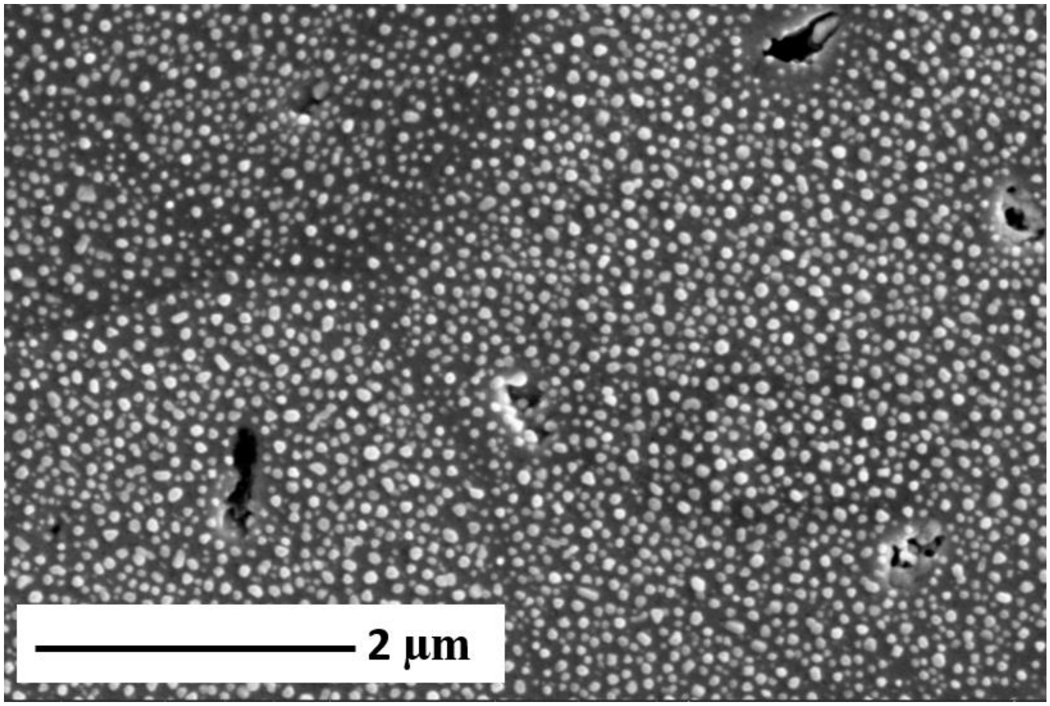
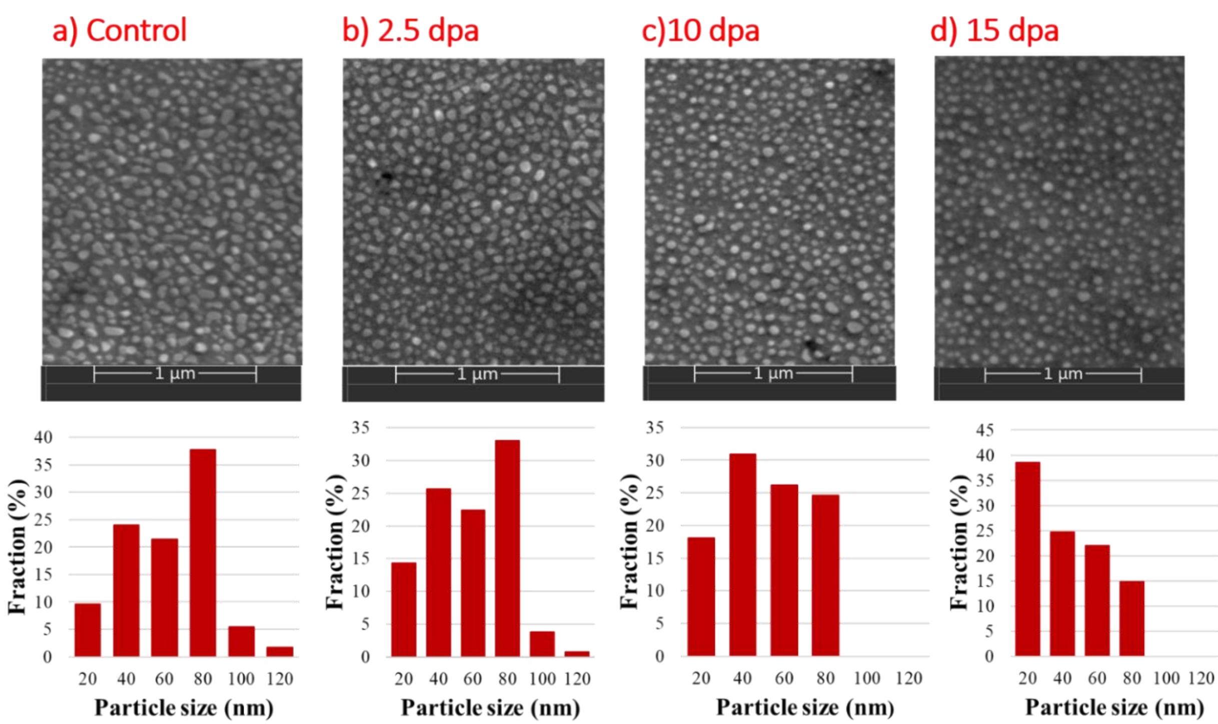


Figure 2. SEM plain view of the top surface of an irradiated ZrC sample showing oxide nodules.



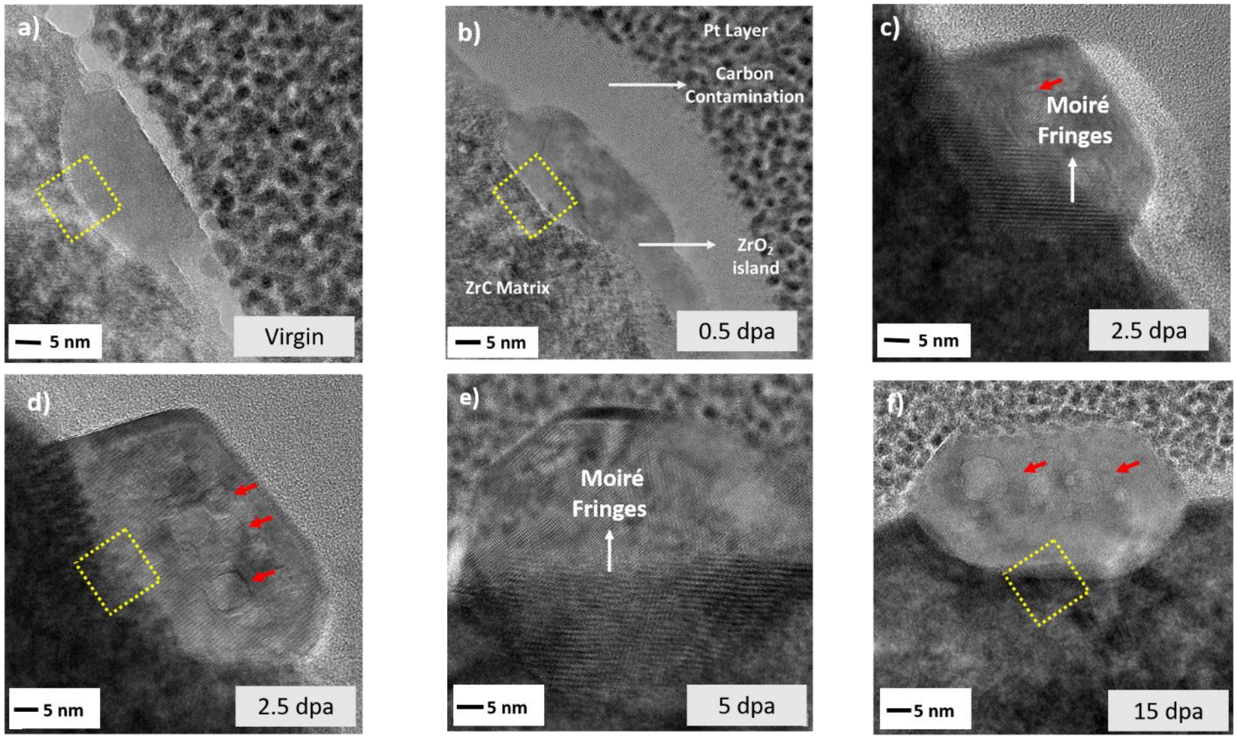


Figure 3. Morphological changes in the oxide nodules as a function of dose.