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TEM Specimen Preparation Using a Low Energy Ion Beam for Nuclear Metallic Materials

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Traditional TEM specimen preparation techniques such as electropolishing for nuclear metallic materials irradiated by either accelerated ions or neutrons are not optimum for producing TEM specimens for advanced (S)TEM analysis due to three main reasons: (1) the difficulty in preparing specimens from a specific site of interest, (2) lack of control in obtaining an ideal thickness at the electron-transparent region of interest, and (3) the time-consuming process to generate new electropolishing “recipes” for new nuclear alloys that are being developed for next-generation nuclear reactor systems. In order to overcome these issues, the Focused Ion Beam (FIB) technique is used to prepare TEM specimens from nuclear metallic materials. Such a technique is ideal for preparing TEM specimens from a specific region of interest in a radioactive nuclear material to study the microstructural evolution that resulted in materials properties degradation after exposure to high energy radiation. The radioactivity of the FIB lamellae will be significantly reduced and will ease the handling of such TEM specimens. Also, unlike the electropolishing technique, hundreds of FIB lamellae can be extracted from the same volume that is required to prepare one TEM specimen using electropolishing technique. This can significantly reduce the total experimental cost by reducing the volume of nuclear materials to be irradiated at either an ion beam facility or test nuclear reactor.

However, one fundamental drawback of using FIB to prepare TEM specimens from an irradiated nuclear metallic material is that FIB induces cascade damage into the sub-surfaces of TEM specimens during the milling/polishing process. Such damage is identical to the damage induced by high energy ion/neutron irradiation. Hence, the FIB-induced cascade damage will affect both quantitative and qualitative results obtained, especially for dislocation loops analyses from the irradiated nuclear metallic system and will indirectly underestimate the radiation resistivity of nuclear metallic material. In this study, a TEM specimen preparation technique using both FIB and low energy ion beams has been developed to reduce the FIB-induced damage in nuclear metallic materials. The metallic systems involved in this study are mainly BCC and FCC alloys. A 200 kV FEI Tecnai T20 analytical TEM with a LaB_6 cathode was used to characterize the defects induced by low energy ion beams in the BCC and FCC alloys. The significant improvement in quality of the TEM specimens prepared by using this newly developed technique, as shown in Figure 1, will be discussed in detailed.

References:

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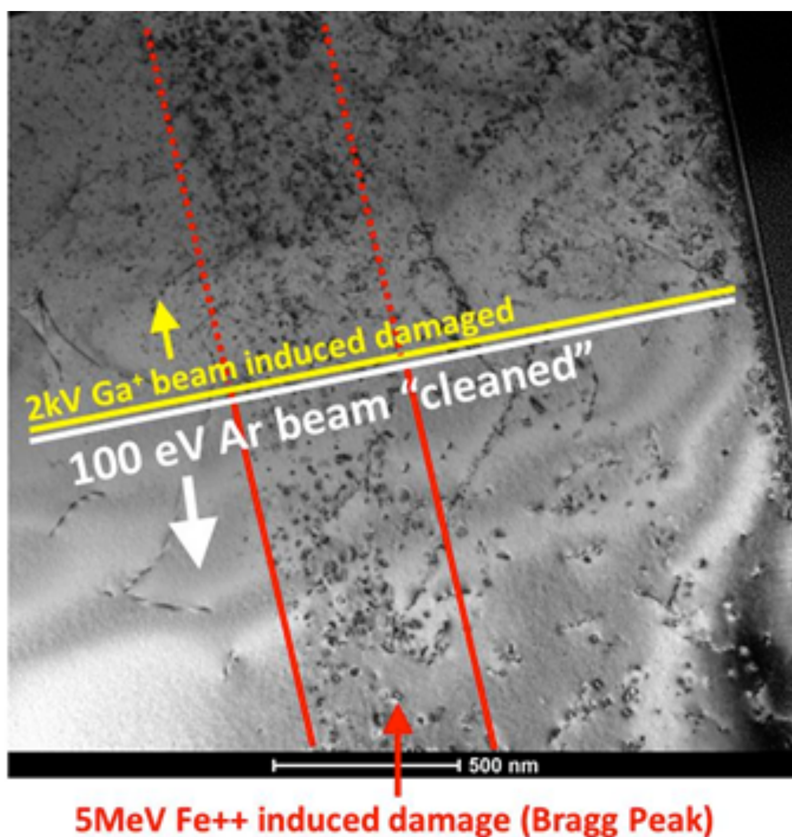

Figure 1 (Left). CTEM Kinematic Bright Field (KBF) micrograph imaged from an ion irradiated FCC – FeNiCr alloy, i.e. Alloy 800H, up to 1 dpa showing a significant improvement in specimen quality after post-Focused Ion Beam (FIB) ion polishing process, i.e. 100 V Ar ion-beam milling. KBF micrograph imaged both the high energy ion irradiated region and non-irradiated region. The Bragg peak damage induced by the high energy ion beam is shown by the red arrow. The upper region of the KBF micrograph, the region above the yellow solid line, corresponds to a region that received no post-FIB ion polish. The lower region of the KBF micrograph, the region below the white solid line, showed the region that had undergone post-FIB ion polishing. This KBF micrograph reveals the significant improvement of the quality of the TEM specimen after the post-FIB ion polishing.

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