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Observation of FIB induced damage in Fe-Cr alloy

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In general, ion irradiated materials have a narrow damage layer with a varying ion induced defect profile. For example, when Fe ions with energy of 8MeV are introduced to a ferrous alloy, the depth of a damage layer is approximately 2μ m from the surface. Various micro-structural defects such as interstitial/vacancy clusters, dislocation loops and voids exist in the narrower damage layer. To understand their role on the change of mechanical behavior of an irradiated material, it is necessary to produce a cross section view TEM (Transmission Electron Microscope) specimen using a FIB (Focused Ion Beam) instrument. However, it has been known that a TEM specimen suffers from severe bombardment of Ga ions with high energy during FIB processing and FIB-induced damage is produced on its surface [1-3]. The FIB-induced damage makes analysis of ion irradiation induced defects difficult. In this work, FIB-induced damage in metal material, Fe-Cr alloy, was investigated using TEM to enhance application of FIB on defect analysis in ion irradiated material.

The Fe-Cr alloy used for this work is a base for low activated ferritic-martensitic steels which are now considered as one of the candidate structural materials for advanced nuclear reactors. It was irradiated by Fe ions with 8MeV at 400℃ using Tandem ion accelerator. The FIB used for the preparation of a TEM specimen of the ion irradiated Fe-Cr alloy was Nova 200 made by FEI. The energy of Ga ion was 30keV. The final thinning was performed down to 150nm thick. We selected a FIB lamella with higher damage among several FIB lamellae. Figure 1(a) shows TEM micrographs of FIB lamella that was lifted out on grid after FIB processing. The TEM micrograph had very complicated contrasts by FIB-induced damage. Also unknown products were observed above pt layer that had been deposited during FIB processing for the protection of the surface as shown in Figure 1(b). From High resolution TEM (HRTEM) analysis in Figure 1(c), they were identified as complex products that consisted of a Fe crystal grain with body centered crystal structure (BCC) and a Fe amorphous phase. Energy dispersive spectrum (EDS) analysis as presented in Table 1 indicated that there contained Ga (30 at%) and Cr (3.4 at%) in both the Fe grain and the Fe amorphous phase. They would be also deposited on the surface of the FIB lamella during the FIB processing. Figure 2(a) shows TEM micrographs of FIB lamella after additional Ar ion milling with a low energy below 1keV. Complicated contrasts in figure 1(a) disappeared significantly but ripple shaped contrasts still remained after the Ar ion milling. The ripple shaped contrast would be due to surface roughening by Ga ion bombardment during FIB processing. Because Ar ion milling can lead to surface roughening with specific milling conditions [4], the energy of Ar ions was constrained to lower below 1keV and incident angle was controlled below 10 degree for the prevention of surface roughening by Ar ion milling. By means of repeated Ar ion milling at low energy, the FIB lamella was damage-free or had the least damage as shown in Figure 2(b). In Figure 2(c) and 2(d), black dot contrasts that were considered as Fe ion irradiation induced defects were clearly observed at a depth of about 1.5μ m.

In summary, FIB induced damage in Fe-Cr alloy contains not only redeposited products such as Fe crystal grains and amorphous phases but also surface roughening on the FIB lamella. It is possible to make a damage-free TEM specimen through Ar ion milling at low energy.

References

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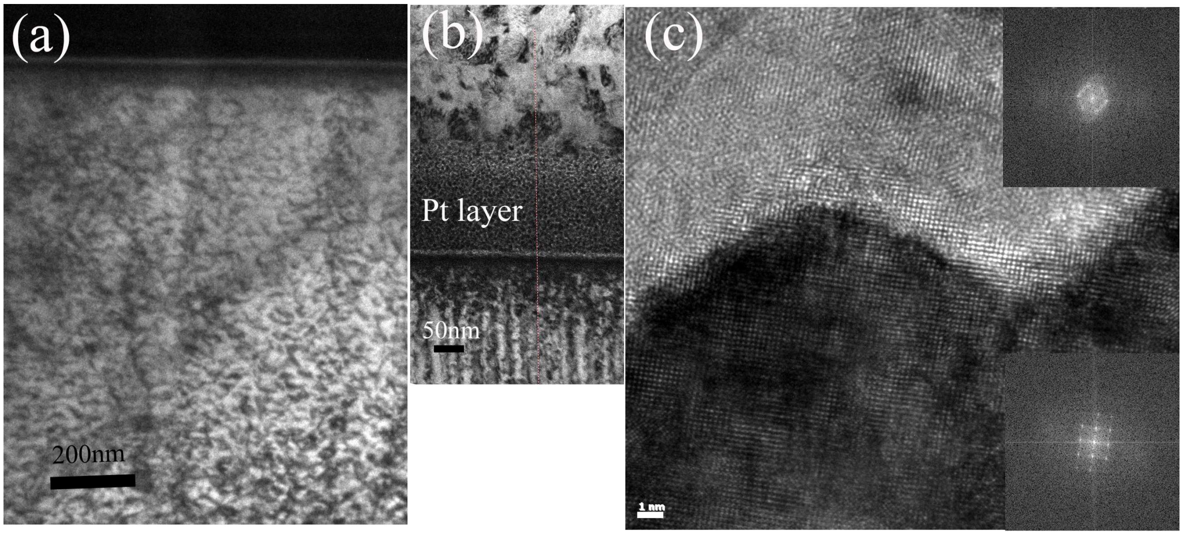

FIG. 1. TEM image of the FIB lamella in Fe ion irradiated Fe-Cr alloy after FIB processing (a) and an enlarged TEM image near pt layer in the FIB lamella (b). HRTEM images and FFT images revealed that unknown products above pt layer were BCC Fe (B=[001]) and amorphous phase (c).

TABLE 1. EDS quantitative results of the redeposited product formed above the pt layer

	Fe	**Cr**	**Ga**
at%	66.1 / ±1.24	3.4 / ±0.20	30.5 / ±1.12

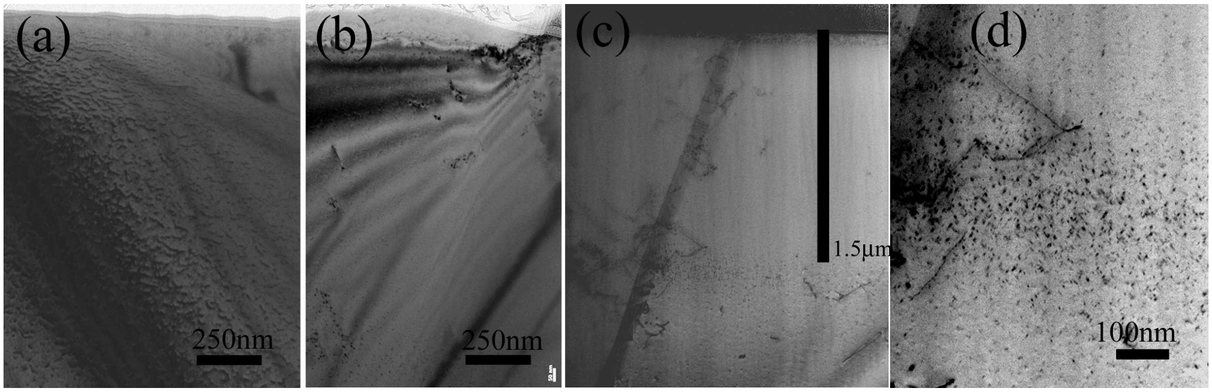

FIG. 2. TEM images of the FIB lamella (a) after Ar ion milling and (b-d) repeated Ar ion milling. Figure 2(c) and 2(d) show black dot contrasts were clearly observed at a depth of about 1.5μ m without FIB-induced damage.