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# Post-FIB TEM Sample Preparation Using A Low Energy Argon Beam

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Despite the significant advances in the transmission electron microscope (TEM), such as application of aberration correction and monochromation [1], sample preparation is still one of the most critical steps determining the quality, and precision of the results. The important challenge here is to prepare samples that are thin enough for electron transparency, free from any surface damage and also have negligible surface roughness. The presence of any surface artifact would make both the interpretation of the image and the chemical analysis difficult or impossible.

Focused Ion Beam (FIB) milling is a widely used technique, which enables TEM samples to be prepared in plan-view and cross-section, and from specific, sub-micron semiconductor devices [2,3]. However, bombarding the specimen surface with energetic ions or neutral atoms has been found to cause surface damage or surface amorphization [4]. This damaged layer is important especially when metallic crystalline materials are considered since they are easily beam damaged due to their low cohesive energies (Al 3.5eV) [5]. Usual FIB Ga ion beam milling with an accelerating voltage of 30kV causes surface damage of about 30nm thickness and even the lower energy end-finish with a 5-10kV focused Ga beam leaves a damage layer of a 5-10nm thickness [4,6]. This surface artifact can be removed by the application of low energy (<2000V) and low angle (0-15$^{o}$) milling practices [7].

For this purpose, recently developed low energy focused Ar ion beam milling system is successfully applied to minimize the ion-induced artifacts appeared on various metallic alloys due to the FIB milling process. Figure 1 is the schematic of the system, with a 2kV Ar ion image showing the sample layout and the milling geometry. Milling is targeted similar to the FIB box milling where the 10-12μm size focused Ar ion beam is scanned in a selected area at incident angles between 15$^{o}$ and -10$^{o}$. Figure 2 shows how the amorphous layer of the Ti6Al4V FIB TEM sample (Fig.2.a.) is removed by consecutive Ar ion milling stages at 2kV(Fig.2.b.) and at 500V (Fig.2.c.), respectively. It is clearly seen that reducing the thickness of the amorphous layer significantly improved the quality of the high-resolution TEM (HRTEM) images. Also the diffuse background due to the amorphous scattering is reduced and spatial frequencies of the corresponding fast Fourier transforms (FFT) become sharper and more distinguishable (Fig.2). A second example is shown in the case of a Ni based superalloy in Figure 3, where Cr distribution in the alloy is critical for material performance under high-temperature environments. In these images fine γ' particles (dark regions) that are relatively lean in Cr compared to the γ matrix (light region) are imaged in the energy filtered TEM (EFTEM) mode using Cr-$L_{2,3}$ edge (575eV). The resolution of the fine γ' particles is significantly enhanced after the removal of the surface amorphous layer by using low energy milling (Fig. 3b.).

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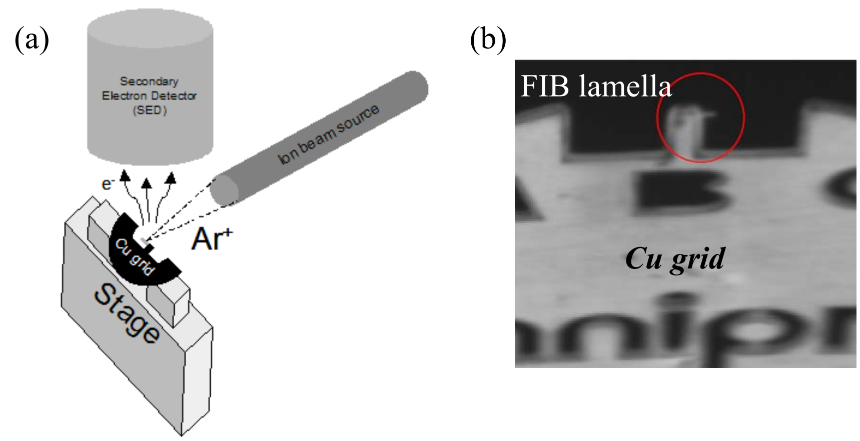


**Figure 1**. **a.** Sample configuration in the Ar ion milling system and **b.** 2kV Ar ion image showing the FIB lamella.

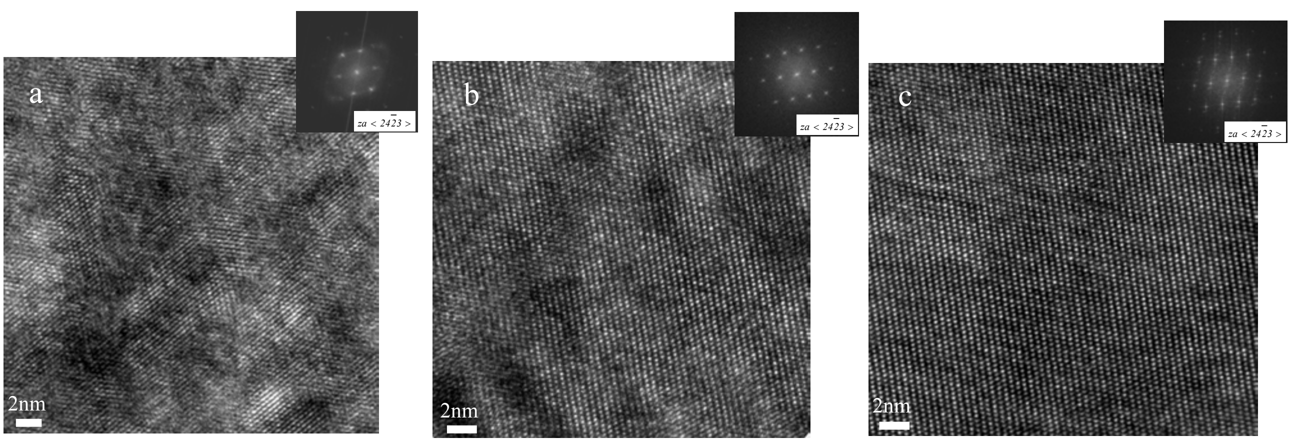


**Figure 2.** HRTEM images and the corresponding FFTs of the α-Ti in the Ti6Al4V alloy after **a.** 30kV Ga ion milling using FIB, **b.** 2kV Ar ion milling for 2hrs, and **c.** 500V Ar ion milling for 1hr.

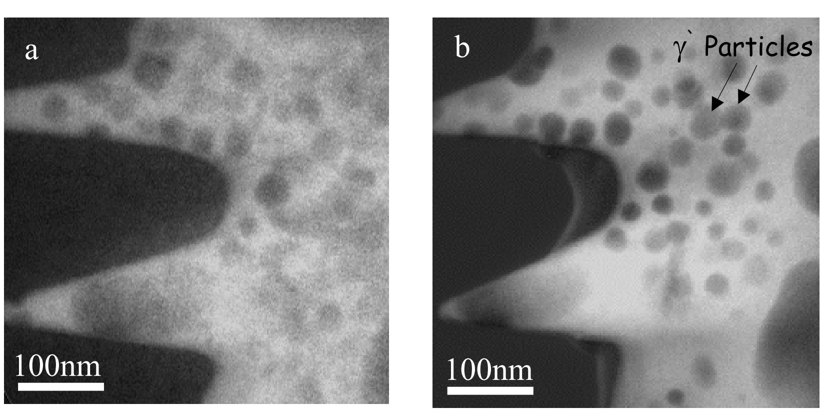


**Figure 3.** EFTEM Cr- $L_{23}$ edge (575eV) elemental map of Ni based superalloy (Renè 88DT) after **a.** 30kV Ga ion milling using FIB and **b.** 2kV Ar ion milling for 2hrs.