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Challenges in FIB TEM Sample Preparation: Damage Issues and Solutions

Xiangli Zhong[1], Xiaorong Zhou[1], Sarah J Haigh[1], Philip J Withers[1], M. Grace Burke[2]

[1.] Department of Materials, University of Manchester
[2.] Oak Ridge National Laboratory
* Corresponding author: xl.zhong@manchester.ac.uk

Focused ion beam (FIB) is now a fairly common technique for transmission electron microscope (TEM) sample preparation [1]. However, challenges remain with respect to FIB processing that may result in unreliable TEM analysis [2][3]. These challenges include ion damage, enlargements in pre-existing cracks and/or holes, curtaining issues, and a high failure rate due to internal stress within the sample[4][5]. In particular, it is possible for the FIB preparation process to promote 'enlargements' of cracks and holes as well as damage to the original 'edges' of such features; such modifications to the original microstructural features can complicate precise TEM analysis. Curtaining issues are commonly regarded as a intrinsic issue related to material morphology and, thus, they have often been overlooked despite their significant effect on uniformity of the uniformity of the TEM specimen thickness. Also, Ga^+ enrichment on the surface of FIB-prepared TEM specimens of Al alloys is another well-known issue.

Strategies and methods to overcome these problems are required. A 'filling' workflow was therefore developed to tackle the hole/crack edge damage and curtaining issues. For Al alloys, a Xe^+ beam FIB was employed to prepare the TEM sample for comparison with the specimen prepared using conventional Ga+ FIB.

To minimize the hole enlargement/damage, readily available ion beam-induced redeposition can be used to fill and protect the edges of cracks and holes. Using the methods presented here the crack and hole edges are well-protected from the ion beam-tail damage. Curtaining was reduced due to the nature of the 'filling'. Guidelines have been developed for applications that can be applied broadly to most materials for FIB TEM sample preparation. **Fig. 1** shows a Ti-SiC composite TEM sample prepared using the 'fill' method. It was first milled using routine TEM sample preparation rough milling route as shown in **Fig.1a** and **Fig. 1b.** The region of interest (ROI) was the interface between the SiC fibre and Ti matrix where cracks had formed. A trench was then milled in front of the exposed ROI until the microstructure and cracks were completely covered by the redeposition material (**Fig. 1c**). The trench milling for generating redeposition materials was performed using rectangle raster scanning with a 25µs dwell time. The ion beam energy was 30KV with 47nA beam current to generate a high amount of redeposition material. The conventional *in situ* lift-out procedure was then performed followed by the final thinning process. The final thinning steps were carefully performed: by first thinning from the backside of covered surface, it was possible to retain the redeposited material for effective protection of hole and crack edges **(Fig. 1d).** The last steps of the final thinning process should be performed by alternatively cleaning both sides of the lamella to ensure that the redeposition materials are removed from the sample surface. Low voltage cleaning was then performed at 5keV and 2 keV to minimize the ion damage induced by 30 kV high energy ion beam. **Fig 1e-g** shows the thinned TEM lamella and the protected crack edge.

A hole in an exemplar Fe sample was protected by the redeposition material (**Fig.2**) using the procedure described above. SAED analysis revealed that the redeposition material, which protected the hole edge, exhibited an epitaxial relationship with the Fe specimen. The transition from polycrystalline materials to

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high content of elemental Fe with epitaxial relationship with the base material may be attributed to the kinetic energy of primary 30kV ions acting on the redeposition filling materials. The kinetic energy may also partially transform to thermal energy during the bombardment on the redeposition materials. The local thermal effects may anneal the material. The kinetic bombardment of the fill materials and the heat generated during the process may preferentially remove local Ga whilst annealing the filling materials, thus promoted the epitaxial relationship.

Another method to fill the holes and cracks *in situ* during the TEM sample preparation process is to use electron beam-induced deposition. However, this may induce foreign material at the hole/crack edge so that attention is needed to ensure structural integrity of the thin lamella under the high beam current.

The issue of Ga^+ ion beam damage and its effect on the analysis of Al alloys was assessed using Ga^+ and Xe^+ FIBs. Fig. 3 shows the comparison of Al alloy AA7108-T6 TEM samples prepared using Ga^+ FIB (Fig. 3 (b) or Xe^+ FIB (Fig. 3 (d), with Ga enrichment clearly evident at the phase boundary. The precipitate free zones (PFZs) shown in Fig. 3 were due to the presence of a coarse $MgZn_2$ precipitate and associated depletion of solutes. In the evaluation of commercially pure Al, the STEM-EDXS analyses confirmed Ga enrichment of the grain boundary occurred due to FIB preparation. However, no Xe was detected for the Xe^+-prepared sample. It was noted that the ion beam-induced amorphization and dislocations were similar to both types of FIB-prepared samples. We conclude Xe+ pFIB is the preferred tool for Al TEM sample preparation[3].

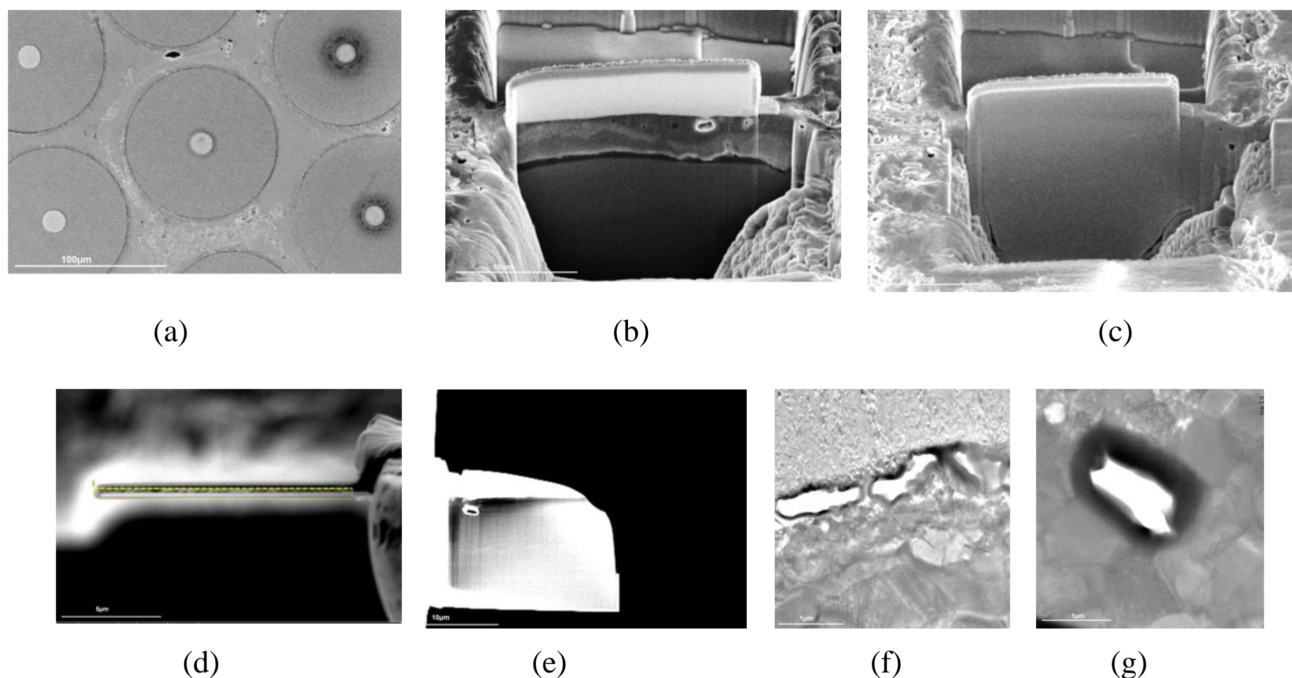

Fig. 1. Sequential steps for the *in situ* fill method using redeposition material for a Ti/SiC sample: (a) plan-view image of the Ti/SiC; (b) SE image of the Ti/SiC cross-section showing voids and cracks formed during a HIP process; (c) SE image of the same region but with the voids and cracks "filled-in" by redeposited material generated from the cut in front; (d) progressive thinning ends at redeposited side of the sample, (e) image of the TEM lamella, (f) low magnification TEM image of holes that were protected by the redeposition material, which has a darker contrast, (e) TEM image of the cracks at the ROI was protected by the redeposition material, which exhibited a darker contrast.

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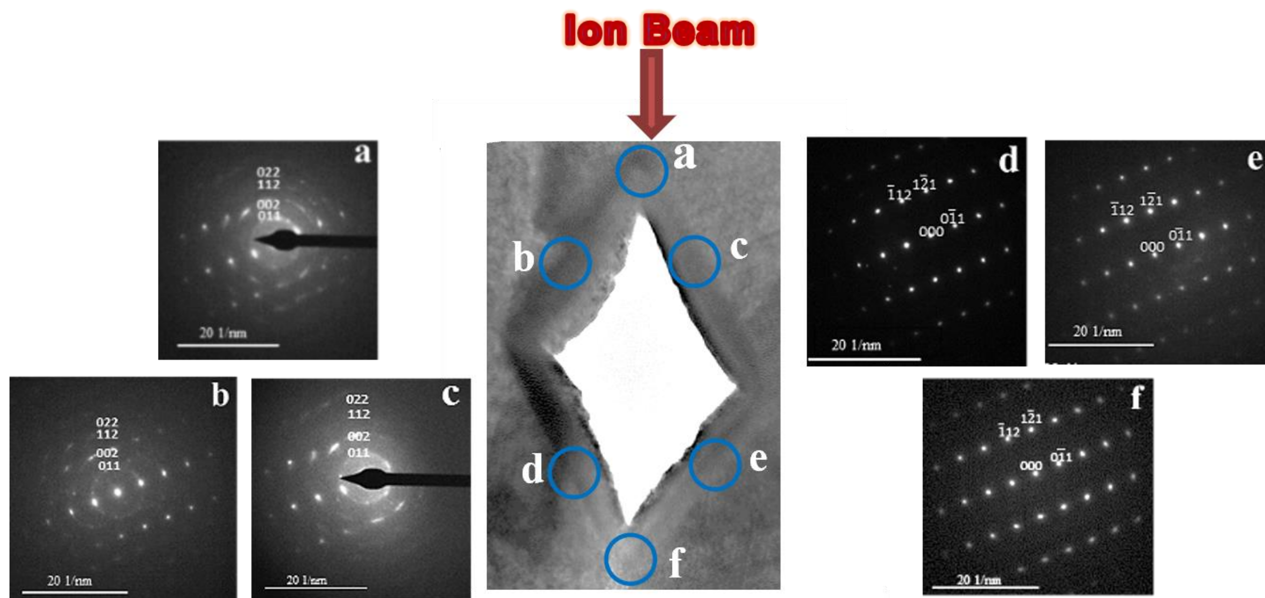

Fig. 2 SADPs acquired at various locations of the interface between *in situ* fill material and the matrix showed that the edge of the hole was protected. Note that the redeposition material exhibited a epitaxial relationship with the base metal.

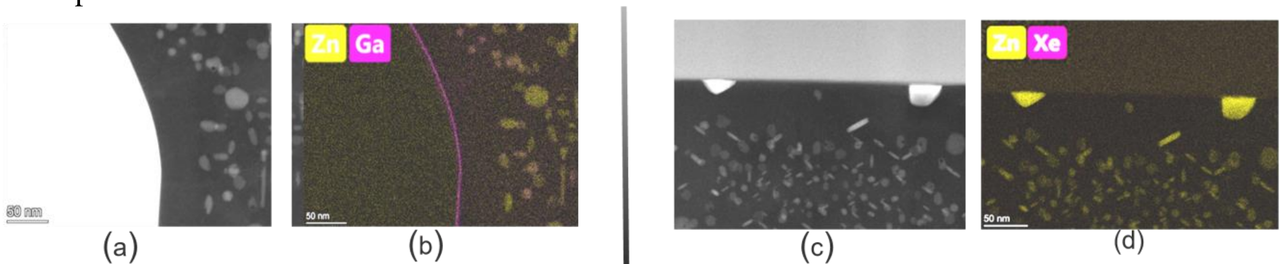

Fig. 3 Comparison of Ga^+ and Xe^+-prepared Al alloy AA7108-T6 (a)-(d) and commercial Al (e)-(j). (a) ADF STEM image and (b) corresponding Zn and Ga STEM-EDXS maps obtained from the Ga+-prepared sample showing Ga+ enrichment along the phase boundary; (c)-(d) STEM-EDXS elemental maps obtained from the Xe+-prepared sample showing no enrichment of Xe at the phase boundary.

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