

An *in situ* Method for Preserving Buried Voids and Cracks During TEM Sample Preparation using FIB

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The focused ion beam (FIB) technique has become a widely-employed method for transmission electron microscope (TEM) sample preparation due to its site-specific and versatile nature [1-2]. Advantages, disadvantages and methods/strategies on overcoming difficulties related to this technique has been extensively studied over past 15+years [3]. Nevertheless, commonly-observed crack and hole enlargements or edge damage during the final thinning step are often left unattended due to the experimental difficulty in eliminating these problems, although it is critical for precise TEM microstructural analysis. We propose a method called “*in situ* filling” to overcome this drawback of FIB sample preparation for TEM analysis. With this method, the crack and hole edges are well-protected from the damage and enlargement during FIB thinning. This method and its strategy can be applied broadly to most of materials using FIB for TEM sample preparation.

A coarse-grained Fe alloys was chosen for the feasibility study, and two ‘real-life’ materials were used to test this proposed new method. The two test samples were hot isostatically pressed (HIP) processed Ti-SiC composite and an excimer laser surface-treated B₄C-reinforced Al-SiC composite.

Two pairs of identical regular-shaped holes were created in order to simulate the effect of the Ga ion beam damage on the hole edge and assess the effectiveness of the protection method by using re-deposition material for *in situ* “filling”. The conventional TEM sample rough-cutting operation was first performed, then pairs of holes were cut by importing a bitmap file designed for this purpose, as shown in Fig. 4a. The holes were subsequently covered with re-deposition material by cutting an extra trench in front of the lamella (Fig. 4b). After these two holes were covered, another pair of holes was cut using the same bitmap file; the cutting parameters are shown in Fig. 1c. Final thinning was performed using the “standard” thinning process. The edges of the covered holes were clearly retained as shown in Figs. 1d – 1f.

Similarly, the Ti-SiC and laser-treated Al-B₄C samples were first cut using the standard TEM sample preparation rough-cutting route in order to expose the area of interest. A trench was then cut in front of the rough-cut surface until the microstructure and holes were completely covered with the re-deposition materials. Electron beam secondary electron (SE) imaging was used to judge the extent of the coverage. When the SE image appeared gray and the microstructures were not visible, the structure was fully covered with re-deposited material. The standard *in situ* lift-out procedure was then performed. Although the thinning parameters can be the same as the normal thinning procedure and depends on the specific materials of interest, particular attention was paid to the final thinning steps. Starting the thinning from the backside of covered surface would be beneficial for retaining the covered re-deposition filling materials to effectively protect the hole and crack edges. The last steps of the final thinning operation should be performed by alternatively cleaning both sides of the lamella. [4]

References

[1]. L. Lechne, J. Biskupek, U. Kaiser, *Microsc Microanal*, **18**, (2012) p. 379–384.

[2]. M.W. Phaneuf, *Micron* **30** (3), (1999) p. 277–288.

[3] L.A. Giannuzzi & F.A. Stevie, *Micron*, **30**, (1999) p. 197–204.

[4] The authors thank Mr. Y. Fan and Dr. Z Liu for providing the composite specimens for evaluation.

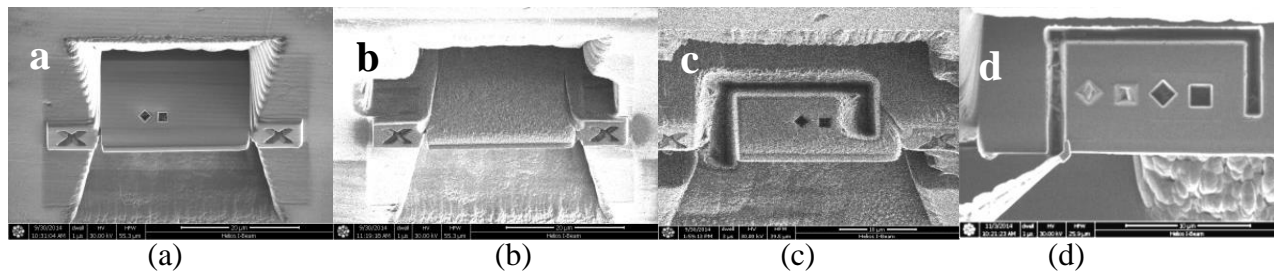


Fig. 1. Cutting with- and without protection using re-deposition material: (a) a pair of holes cut using bitmap file (left holes); (b) holes covered by re-deposition material; (c) another identical pair of holes cut at this stage (right holes), (d) lift-out for final thinning. Note the larger size of the holes cut prior to final thinning compared to the “protected” (filled) holes.

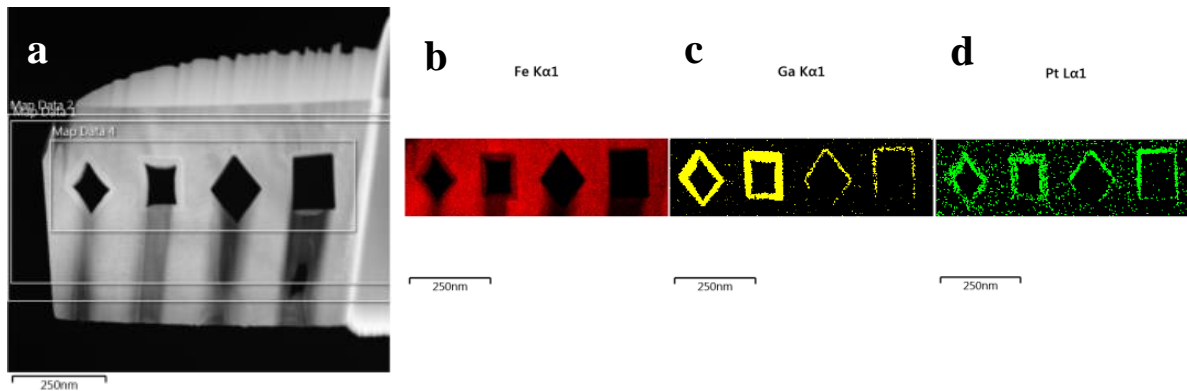


Fig. 2 Analytical TEM study of the *in situ* filling method on the Fe sample: (a) STEM image of the lamella; (b-d) Fe, Ga and Pt XED spectrum images showing the protected edges of the filled holes (left) respectively.

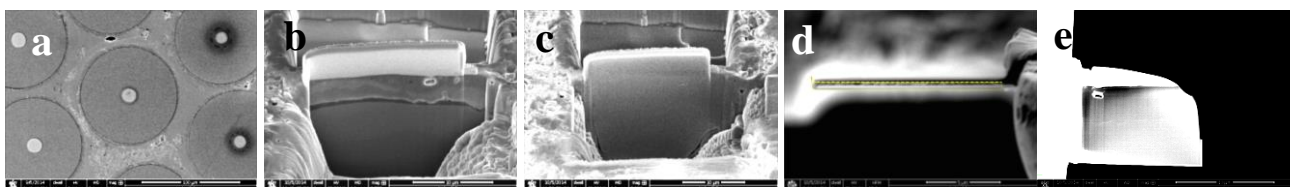


Fig. 3. *In situ* filling method for Ti/SiC sample; (a) plan-view of the Ti/SiC sample; (b) cross-section of the Ti/SiC showing voids and cracks formed during HIP process; (c) voids and cracks were filled using re-deposited materials from a cut in front of the lamella; (d) progressive thinning ends at re-deposited filling side of the sample; (e) final thinned lamella.

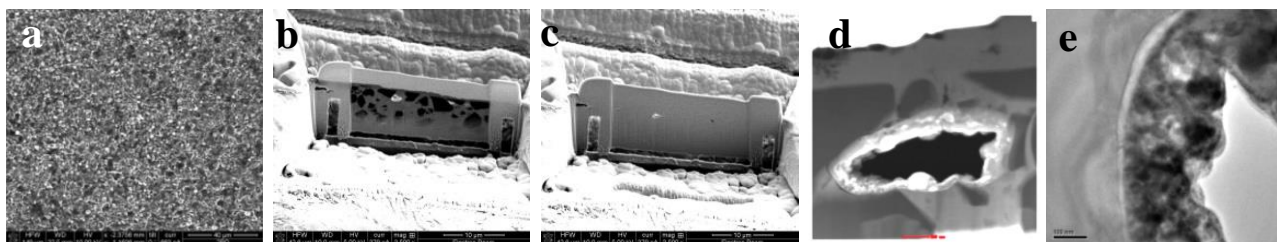


Fig. 4. Use of re-deposition material to coat the Al/B₄C voids: (a) Laser-treated surface; (b) initial re-deposition material is visible; (c) lamella face is fully covered with re-deposition material; (d)-(e) HAADF STEM images show edges of the void are well-preserved by the re-deposited material.