Microsc. Microanal. 8 (Suppl. 2), 2002
DOI 10.1017.S1431927602104764

Site-specific TEM Specimen Preparation of Grain Boundary Corrosion in Nickel-Based Alloys Using the FIB "Plan-View Lift-Out" Technique

M.W. Phaneuf and R. J. Patterson
Fibics Incorporated, 556 Booth St. Ottawa, Ontario, Canada K1A 0G1 (info@fibics.com)

Use of the focused ion beam (FIB) microscope allows site-specific specimen preparation for TEM analyses in a routine manner not realizable with any other technique. The ability of FIB to image in secondary electron mode with a high degree of orientation contrast and in secondary ion mode with a high sensitivity to the presence of elements such as oxygen makes FIB an ideal tool for the analysis of metallurgical systems [1].

Many significant advances have been made in the preparation of TEM specimens by FIB [see references contained in 2], of which our "Plan-View Lift-Out" (PVLO) is a minor modification [2]. Combining the imaging capability of the FIB (which can be extended to cover square millimeters of area by digitally stitching images together into large mosaics) with the PVLO technique allows the production of TEM specimens from site-specific features such as cracks or the terminus of corrosion selected from observations taken over large areas of material. These TEM specimens have a nearly "one-to-one correspondence" with FIB the image(s), making it easy to relate the small-area TEM information with the "big picture" of the specimen.

Figure 1 is a FIB secondary electron image (SEI) showing the terminus of grain boundary corrosion in a nickel-based alloy. This specimen has been metallographically mounted and polished prior to FIB imaging. The corroded region appears black as it is an insulator, which charges positively when imaged with the positive Ga^+ ion beam, trapping the secondary electrons emitted when the beam strikes the specimen. Figure 2 shows the same region, this time as a FIB secondary ion image (SII), which shows the total positive secondary ion yield detected at each point. The presence of oxygen in the corrosion product leads to an enhanced signal due to enhanced secondary ion yield [1].

To prepare a PVLO sample, a thin layer of tungsten is deposited over the region of interest (ROI), which can be up to ~25 μm x ~25 μm in size. This tungsten protects the original surface during subsequent FIB imaging and milling and its complete removal during final thinning helps mark that one has reached the original surface and hopefully removed any surface artifacts left from polishing (no tungsten should be detected in the ROI on the final specimen).

To remove the ROI from the bulk, an "inverted barn" shape is FIB milled out of the surrounding material (Figures 4 & 5). The Lift-Out technique is the used to remove the "barn" and place it on a copper carrier [2] where it can be affixed using glue or FIB deposited tungsten (Figure 6). After milling away the portion of the carrier behind the specimen, the roof of the barn is milled away, as is the protective tungsten. Final thinning then proceeds to electron transparency (Figures 7 & 8). Note the correspondence between the target area shown in Figure 2 and the final specimen as viewed in Figure 7. This specimen is essentially uniformly thin over a 20 μm x 25 μm region.

References:
1. M.W. Phaneuf, *Micron*, 20, (1999) 277-288.
2. R. J. Patterson, D. Mayer, L. Weaver and M.W. Phaneuf, these proceedings

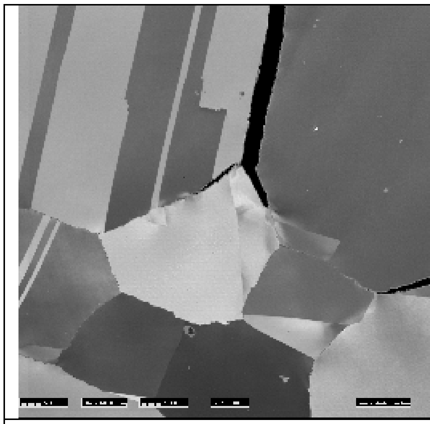


FIG. 1. FIB secondary electron image (SEI) of the terminus of grain boundary corrosion (which appears black as it is an insulator) in a Ni-based alloy. Field of view = 80µm.

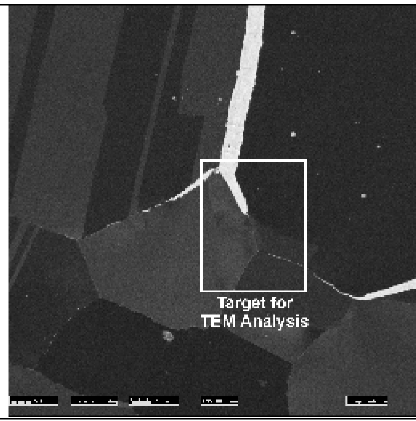


FIG. 2. FIB secondary ion image (SII) of the region shown in Figure 1. Oxygen enhanced ionization yield increases the signal from the corroded regions making them appear white.

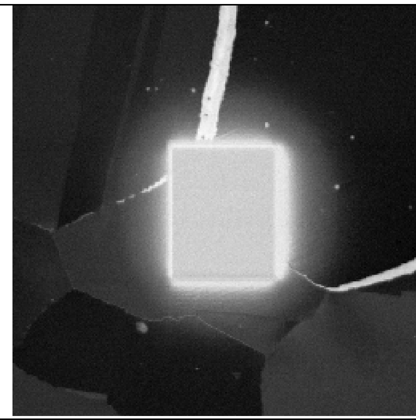
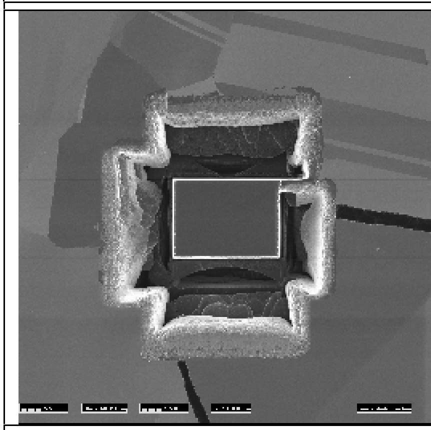
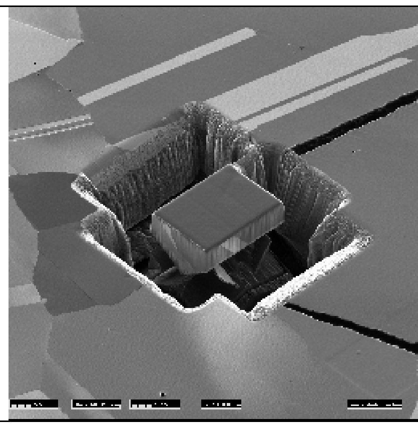


FIG. 3. FIB SII after a thin protective layer of FIB tungsten is deposited over the region of interest. This tungsten also serves as a marker of the original surface. FOV = 80 µm.





FIGs. 4 & 5. Top and off-axis views (FIB SEI) of the “inverted barn” milled free of the surrounding material. A small support (top right) holds the barn in place until final trimming just prior to lift-out. The region to be removed is 25µm x 20µm at its base which is still covered by a protective tungsten coating.

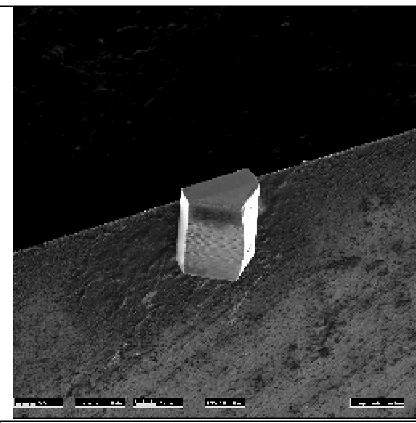


FIG. 6. FIB SEI of the “barn” glued down on the carrier, ready for thinning to electron transparency.

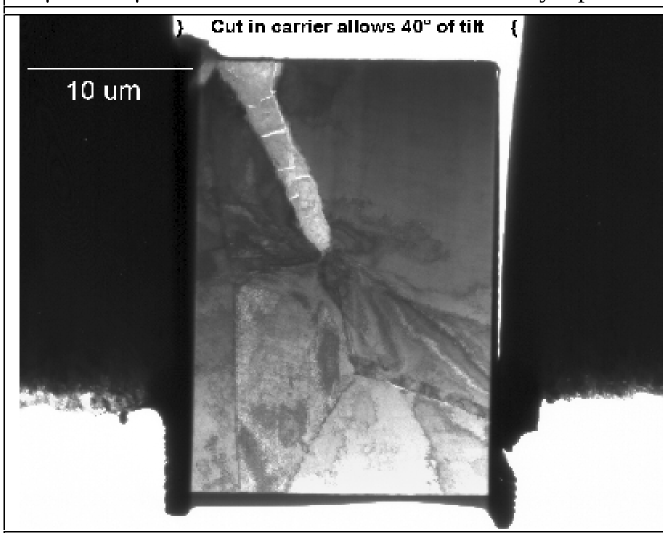


FIG. 7. TEM BF image (originally 1,500X) of the final specimen mounted on the carrier. Compare to FIGs. 2 & 3.

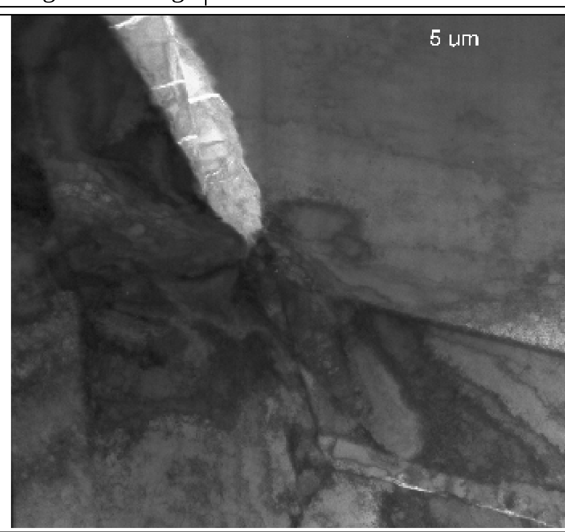


FIG. 8. TEM BF image (originally 3,810X) of the tip of the corrosion and the unattacked portion of the g.b.

https://doi.org/10.1017/S1431927602104764 Published online by Cambridge University Press