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FIB Milling strategies for TEM Sample Preparation of Spheroidal Powder Particles

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There has been a dramatic surge of activity in metal additive manufacturing (AM), in which metal powders are fused or otherwise bonded to make near-net shape components. In many of these processes, gas atomized metal powder is used as the feedstock. Such powders can exhibit very different microstructures from the bulk alloy, and while these differences are largely eliminated in processes that melt the powder, the microstructural features in the powders can be retained in the final product in such processes as gas dynamic cold spraying (CS). As such, there is now significant interest in studying the microstructures of the atomized alloy powders. Conventional focused ion beam (FIB) methods for TEM specimen preparation are optimized for the production of lamellae from bulk flat samples: a platinum cap is deposited on the region of interest (ROI); trenches are milled out either side of the ROI; the resultant lamella is transferred to a TEM support grid; and then final thinning to electron transparency is performed [1].

Even for bulk samples, complications can arise with Al alloys due to the effects of extended exposure to Ga^{+} ions [2]. When working with spherical powder particles, there are additional complications due to the sample geometry. To illustrate this, we present here data obtained from gas atomized Al6061 powder, which is suitable for CS applications. The powder is mounted using colloidal graphite, and milling was performed using a dual-beam FEI Helios NanoLab 460F1 Ga^{+} FIB-SEM. In all cases, sequential e-beam and ion-beam Pt deposition was necessary to get a sufficiently thick protective layer on the curved particle surface. Figures 1A-1C show the problems that can arise when attempting to obtain a FIB-cut cross section using conventional approaches. There is a very small contact area between the lamella and the graphite support in the latter stages of milling, and the lamella becomes unstable increasing the probability of ion beam damage. To overcome this, we have developed a “circle” milling technique in which semi-circular trenches are milled leaving a ring of metal to support the lamella during the latter stages of milling. This is shown in Figures 1D-1F. For efficient milling, the semi-circular trenches are produced using a high ion current (9.3nA), and there is a ≈3μm exclusion zone around the 2μm Pt strap to prevent the beam from damaging the ROI under these conditions. For the example shown, the rough-cut milling took about 6 minutes. The supported lamella is thinned further using successively lower ion currents, and is then transferred to a TEM grid for final milling as usual. Examples of STEM data from one such sample are shown in Figure 2. The HAADF image shows that a sample of uniform thickness has been obtained. The X-ray maps reveal the distribution of the alloying elements (Mg, Si and Fe) in the cell boundaries within the powder particle, and they also show that the Ga contamination is confined to the protective Pt layer.

References:

[1] LA Gianuzzi in “Introduction to Focused Ion Beams”, ed. LA Gianuzzi (Springer, USA) pp. 213
[2] KA Unocic, MJ Mills, GS Daehn, *J. Micros.*, **240** (2010), pp. 227-238
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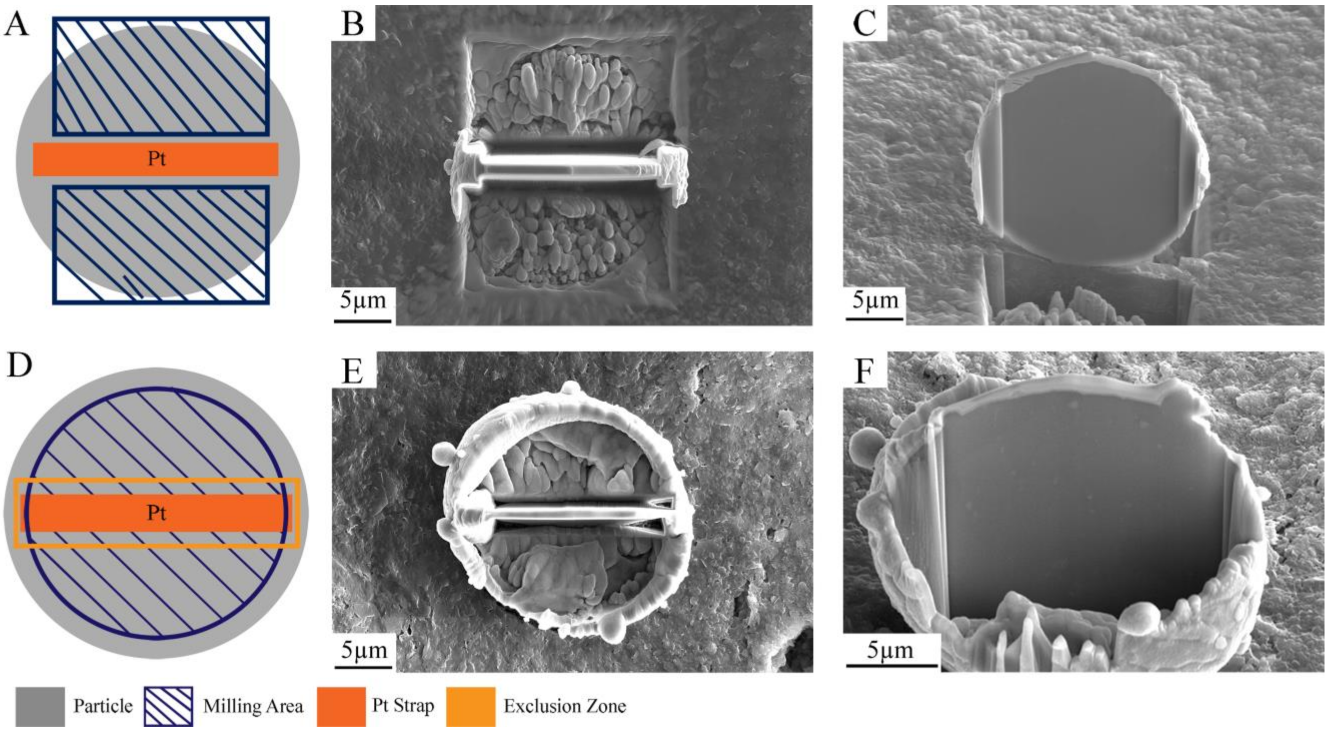

Figure 1. Schematic diagrams and secondary electron SEM images of powder particles FIB milled using: A-C) Conventional rectangular trench approaches. D-F) The “circle milling” approach. C) and F) were obtained by tilting the samples in B) and E) by 52˚ to better reveal the geometry of the lamellae.

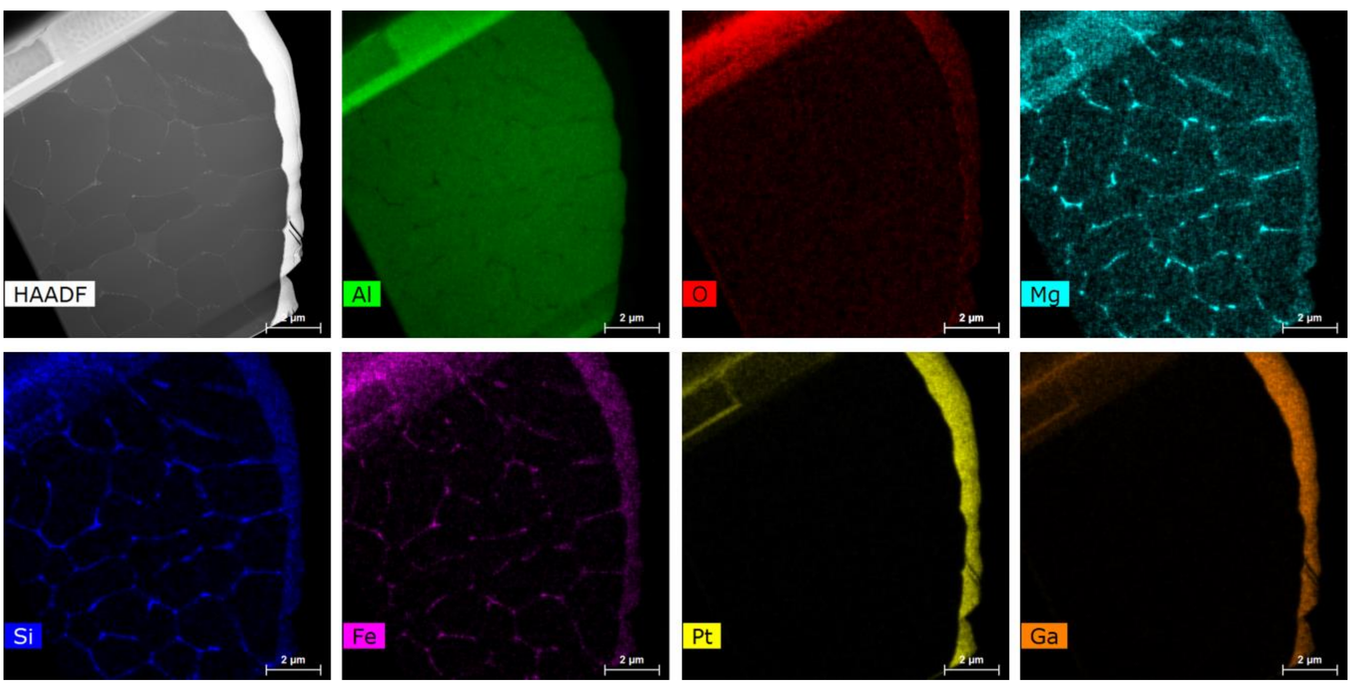

Figure 2. STEM data obtained from a FIB-cut cross-section through an as-atomized Al6061 particle produced using the “circle milling” approach shown in Figures 1D-1F above.

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