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A Comparison of Ga FIB and Xe-Plasma FIB of Complex Al Alloys

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Remarkable advances have been made in dual-beam focused ion beam – scanning electron microscopy (FIB-SEM) technologies over the last 20 years. Such FIB-SEMs can now be used to perform a wide variety of microstructural characterization and microfabrication experiments. The incorporation of X-ray spectrometry, electron back-scattered diffraction (EBSD) and *in situ* lift out capabilities into dual-beam FIB-SEMs make them one of the most powerful instruments for the analysis of microstructure in materials. The vast majority of these instruments are equipped with Ga liquid metal ion sources, and the latest generation Ga FIB columns are extremely powerful and versatile. The main drawbacks of Ga FIB are the limits on the maximum material removal rate, issues with amorphization for certain types of sample, and the possibility of chemical reactions between Ga and the specimen. Such issues cause particular problems for metallurgical samples, because some metals interact strongly with Ga and the important processes often occur on length scales that are not accessible by Ga FIB. More recently, dual-beam FIB-SEMs with inductively-coupled Xe plasma ion sources have been developed. Such plasma FIB (PFIB) columns give much higher material removal rates, and the Xe ions produce very different types of damage, which makes PFIB instruments much more suitable for certain types of sample [1,2].

In our work, we have investigated the relative advantages of Ga FIB and Xe PFIB technologies for various types of samples. Results on ceramic coatings with hierarchical porosity are reported elsewhere [3], and here we consider complex Al alloy systems. It has been shown previously by Unocic *et al.* that Ga FIB milling of Al alloys can lead to Ga accumulation at surfaces, grain boundaries, and interphase boundaries [4]. The sample used for this study was a 150 µm thick cold-sprayed coating of an Al-Cr-Mn-Co-Zr alloy on an Al 6061 alloy substrate. The complex alloy used for the coating exhibits a hard, quasi-crystal reinforced microstructure [5]. The microstructures of the coating and substrate were investigated using two FEI Helios NanoLab FIB-SEMs: a 460F1 Ga FIB and a Xe PFIB.

The most obvious difference between the two FIB columns is the milling rate achievable. To illustrate this, trenches were cut into the coating, and the trench walls were examined to reveal the microstructure in cross-section. Examples of secondary electron SEM images obtained from trenches cut using the Ga FIB and the PFIB are shown in Figures 1a and b, respectively. The trenches have approximately the same dimensions (17 µm wide and 10 µm deep) and were produced using the highest ion beam currents that did not result in excessive damage. The milling times for the two trenches were 26 and 4 minutes for the Ga FIB and PFIB, respectively. While the curtaining on the PFIB cuts was more pronounced, the near-surface damage was far less than that for Ga FIB. Following Burnett *et al.* [2] we evaluated this damage using EBSD on sections cut through the Al 6061 substrate. Examples of the data are shown in Figure 2. In each case, the upper figure is an overlay of the image quality and inverse pole figure maps, while the lower image is the corresponding grain identification map. Although the size of the areas collected are quite different, the percentage of patterns indexed and the confidence index (CI) are significantly higher for PFIB than for Ga FIB, indicating that PFIB gives much less near-surface crystal damage [6].

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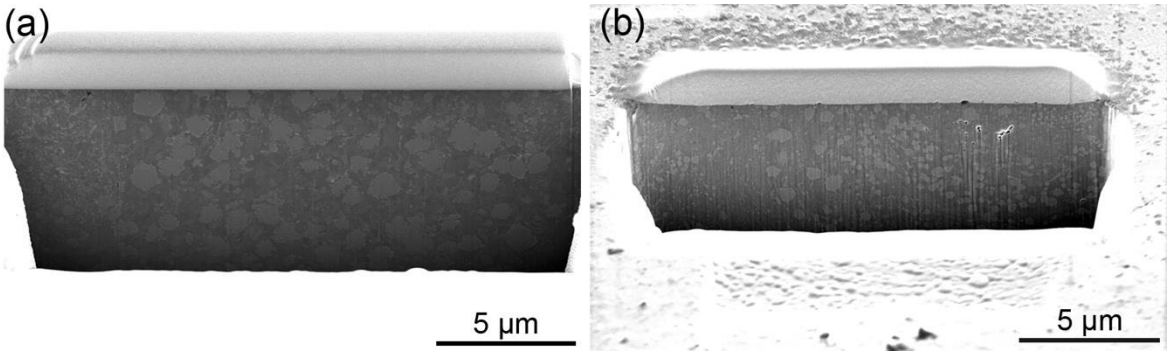

Figure 1. FIB-SEM Cross sections: (a) Ga FIB, and (b) PFIB

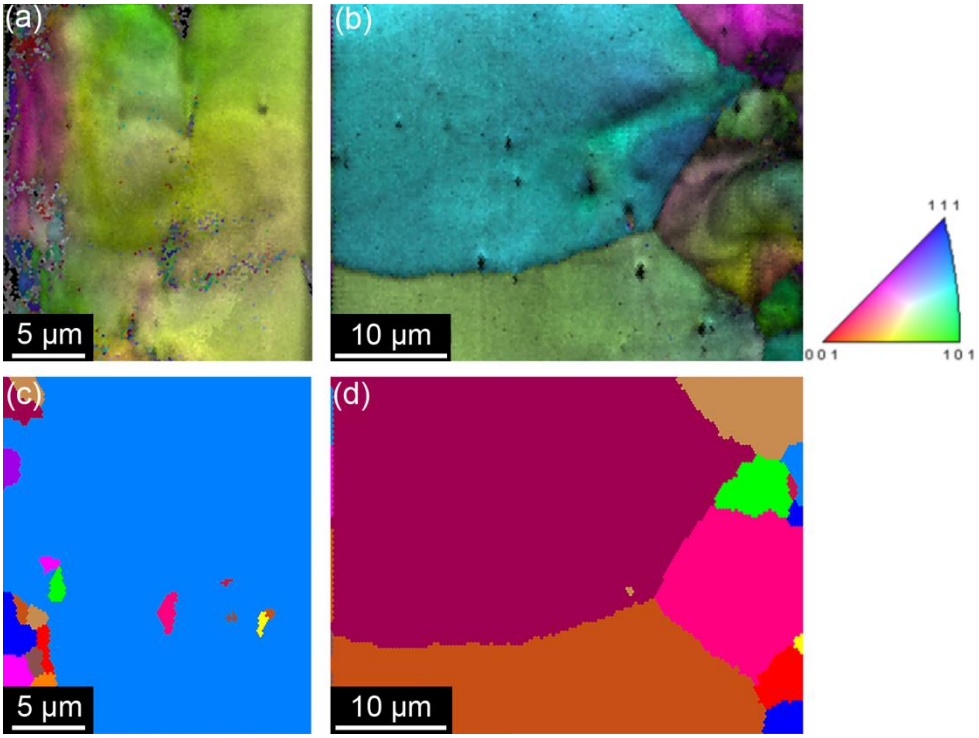

Figure 2. EBSD IPF-IQ Maps and grain identification maps: Ga FIB (a,c), PFIB (b,d). For GaFIB the index % and CI are 94.6 & 77.1%, whereas for PFIB they are 99.9 & 95.3%, respectively.

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