

Deciphering Liquid Metal Embrittlement and Altered FIB Damage Microstructures on Aluminum

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Focused ion beam milling has advanced site-specific TEM and atom probe tomography (APT) sample preparation for analysis of grain boundaries, precipitates, or other features of interest, as well as allowing for 3D cross-sectioning at high rates to allow for depth profile investigations [1-4, 8]. With the rise of in-situ TEM mechanical testing, there is a need to fabricate nano-specimens for testing. Focused Ion Beams (FIBs) are most commonly used for micro-machining and patterning and used for prepared specimens via nanomilling and nanomanipulation) [2]. However, there are issues with liquid metal (LM)-based ion sources in certain materials. FIB milling can amorphize materials introducing a damage layer and can result in Ga implantation contamination in certain materials. This has been observed in grain and phase boundaries of certain materials, such as semiconductors and some metals [8, 9]. Nanomilling of specimens is necessary for thinning of material for TEM-transparency (~100nm thick). Milling at these dimensions and angles induces damage, some of which can be protected/mitigated, some of which cannot. Trace concentrations of impurities at grain boundaries can impact grain growth and mechanical properties. Gallium diffusion in Al occurs with Ga as a liquid at room temperature, and can diffuse rapidly to existing defects, primarily grain boundaries [4]. Grain boundaries and grain boundary character (composition, orientation) greatly influence mechanical properties [10]. Aluminum (Al) has been shown to be embrittled by gallium (Ga) implantation and inclusion in the matrix, as liquid Ga penetrates the matrix [4]. In addition to ion implantation, ion irradiation can cause the development of knock-on damage, such as defects (loops and black dot damage) and amorphization, both of which occur on the nanometer scale but contribute to overall bulk material properties [1-3, 5]. In addition, phase changes can occur in other multi-component material systems [6].

New FIB technology has been developed, incorporating LM ion sources other than Ga [11, 12]. These include Au and Si, as well as gas-based sources, such as Ne, Ar, and Xe [13]. The LM sources using Au and Si are typically used to carry out controlled, implants in sample materials for nanopatterning and nanofabrication. TEM prep is an extension of those tools and systems. The impacts of different types of liquid metal-based ion sources on resulting microstructures has not been well-characterized. This work will identify the effects of different liquid metals on the Al microstructure to advance understanding of Al sample preparation and understand the liquid metal-induced embrittlement of Al. We will expand upon the current understanding of FIB-based damage in materials and investigate novel liquid metal FIB systems and their impact on the Al microstructure to minimize detrimental impacts on Al specimens. Through examining a new path for testing of liquid metal embrittlement-sensitive samples, we show other methods are useful and should be used to prepare specimens.

Al TEM samples prepared via Ga-FIB, Au-FIB, Si-FIB, and mechanical polishing and analyzed using TEM and STEM techniques using a JEOL 2100 TEM, FEI Tecnai 200, and FEI STEM. Au and Si-FIB prepared specimens were mounted for TEM analysis using the ex-situ lift-out method, ensuring that no Ga implantation occurs. Analysis of the defect types, size, and distribution depending on the sample preparation method will be presented. Chemical elemental mapping of FIB-prepared samples will examine segregation of liquid metal FIB ions to Al defects to investigate the possibility of limited diffusion of

Au/Si within the Al matrix, as compared to Ga. Comparison Al TEM samples will also be prepared by non-ion beam techniques, namely dimpling and Tripod polishing to look at native state TEM samples, and be able to better compare the damage that occurs using ion beam based techniques.

Initial results show that even will low kV (5 and 2 kV) Ga⁺ cleaning following lamella lift-out, dislocation loops and Ga-induced black dot damage populate the Al microstructure. However, similar defects are observed in the Au and Si-prepared samples. Clear differences in the ion bombardment damage are not observed. Future chemical mapping with STEM-EDS will explore the composition of existing and irradiation-induced defects in the Al [14].

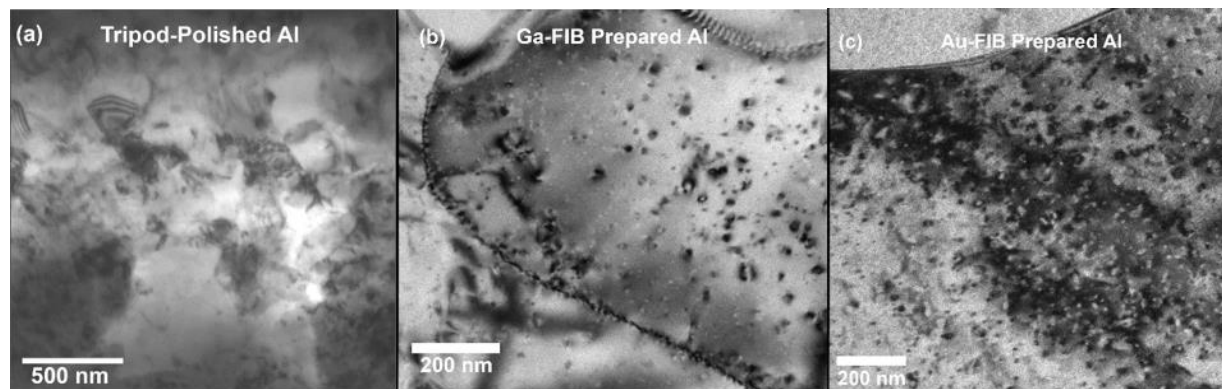


Figure 1. Bright field TEM micrograph of Al specimens showing damage dependent on the sample preparation method.

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