Focused Ion Beam Preparation of Low Melting Point Metals: Lessons Learned from Pb/Sn Solders

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Focused ion beam (FIB) tools, typically utilizing Ga sources have been powerful tools for preparation of samples for transmission electron microscopy (TEM) [1]. The advent of Xe plasma sources [2] provides an important alternative to Ga with a chemically inert beam, with a much larger source size/profile, which has been utilized for TEM sample preparation [3, 4]. Recently, mitigating sample-heating effects with the use of cryo-stages has shown promise for minimizing local heating effects in Pb-solder [5] and In-bump bonds [6] for SEM cross-sections. For TEM samples, the potential for beam heating is likely worse due to the more limited heat-dissipation geometry of a nearly free-standing foil.

In this paper we show comparisons between room-temperature (RT) Ga- and Xe-plasma FIB samples of eutectic Pb-Sn solder to Au. Solder joints are ubiquitous in the microelectronics industry [7] and intermetallic compounds form readily during the soldering process and aging and annealing. In this work we utilized a Thermo Scientific Helios G3 Ga-FIB and a Thermo Scientific Helios G4 Xe Plasma FIB (PFIB). The specimen consisted of a Pb-Sn eutectic solder to Au. Figure 1 shows high-angle annular dark-field (HAADF) STEM images of specimens prepared by Ga- and Xe-plasma FIBs. The Pb-Sn phase boundary has clearly been attacked by the Ga likely due to a low melting binary or ternary phase/and or localized ion-beam heating. Unfortunately, no Ga-Sn-Pb ternary phase diagram exists but the Sn-Ga phase diagram has a 21°C eutectic. Both Ga and Xe -FIB samples show Kirkendall voids at the Au-intermetallic interface. In contrast, the PFIB sample as seen in Fib 1b. still mostly retains the Pb-Sn phase boundaries. Both specimens show comparable Au-Sn intermetallic formation, seen in orange in the X-ray map overlays in Figure 2. It is likely that the ion-beam heating profiles between the Ga and Xe were significantly different resulting in less localized heating with Xe. In the case of In [6], artifacts (voiding intermetallic formation) were seen both for RT Ga and Xe cross-sectioning. Additional experiments will determine the effect of sample temperature and potential beam-solid chemical interactions [8].



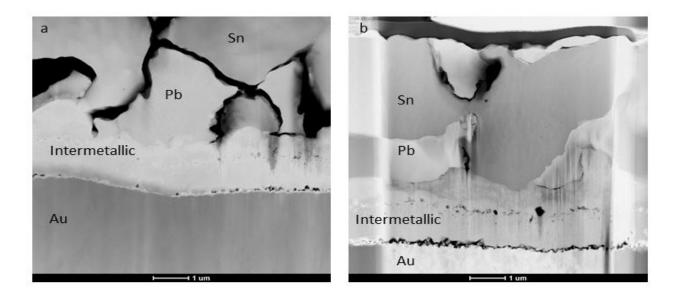


Figure 1. HAADF-STEM images of a solder sample prepared with a. Ga-FIB sample and b. Xe-plasma FIB. The Pb-Sn phase boundaries are mostly preserved in the Xe-plasma FIB sample albeit with more specimen curtaining.

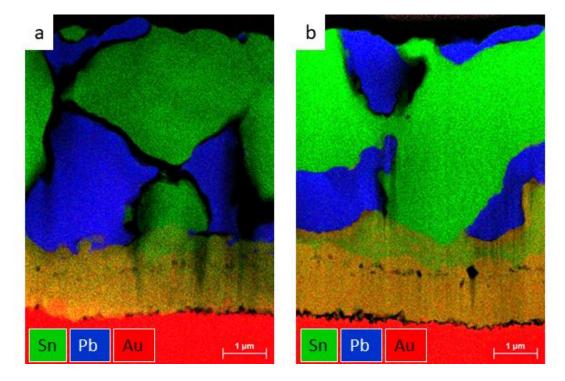


Figure 2. Color overlays of X-ray maps from a. Ga-FIB and b. Xe-plasma FIB samples.

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- [8] Sandia National Laboratories is a multimission laboratory managed and operated by National Technology & Engineering Solutions of Sandia, LLC, a wholly owned subsidiary of Honeywell International Inc., for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-NA0003525. This paper describes objective technical results and analysis. Any subjective views or opinions that might be expressed in the paper do not necessarily represent the views of the U.S. Department of Energy or the United States Government.