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Low Energy 500 eV Focused Argon Ion Beam Provided by Multi-Ions Species Plasma FIB for Material Science Sample Preparations

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Multi-Ions Species (Xe^+, O^+, Ar^+, N^+) Plasma Focused Ion Beam (MIS-PFIB) microscope opens wider applications for sample preparation to conventional Gallium-Focused Ion Beam (G-FIB) microscopy world. The plasma ion source is a RF plasma generator utilizing external antenna inductively coupled plasma (ICP) source [1], and inside the source is an empty chamber, from where high angular intensity ion beam is extracted. Subsequent ion beam is then demagnified, collimated, scanned and focused on the sample. Switching between type of ions takes less than 10 mins automatically.

Xe^+ ion beam has being greatly accepted by FIB microscopy community providing the advantages of not only for high sputtering yield, high current milling at 30 keV for a large 3D volume data acquisition and for correlated microscopy applications [2] , but also for a much less ion implantation and amorphization of gallium free TEM specimen preparation [3]. Reactive oxygen ion beam dramatically improves sample milling quality for carbon-carbon bonding hard or soft materials by smoothing the cut-face very effectively during sputtering [4]. Low energy broad Ar^+ ion beam for sample preparation have been widely used for material science sample preparation for decades but it is not integrated with FIB-SEM DualBeam microscope in general.

In this paper, we focus on applying of a low energy to 500 eV Ar^+ focused ion beam provided by a MIS-PFIB/SEM DualBeam microscope for sample preparations, i.e., bulk sample surface cleaning for a built-in monochromated SEM BSE imaging in nano-scales and Ar^+ ion beam low energy TEM specimen final milling with built-in retractable STEM for real-time monitoring (RTM).

The materials used for Ar^+ ion beam bulk sample preparation is Al-Li-Cu-Mg alloy. T_1 (Al_2CuLi) phase is the most prominent precipitates in artificially aged Al-Li-Cu-Mg alloy with a Li content at about 1.4 - 1.5 wt.% when the Cu: Mg ratio is higher than 3:1. To observe T_1 phase distributions, it has been required to prepare a TEM specimen and observed it by a TEM/STEM. However, in this report, we observed T_1 phases located both at grain boundary and inside the grain in BSE signal by a monochromated SEM at 3 keV. This is greatly assisted by the 500 eV Ar^+ ion beam cleaned sample surface in-situ in the MIS-PFIB/SEM microscope chamber. Al-Li alloys surface is very sensitivity to surface oxidation, Ar^+ ion beam cleaning process removed not only the surface oxide contaminations but also carbon induced by electron beam scan, allowing epeated image acquisitions at the same location. The results are shown in figure 1 and 2. SEM images were taken automatically in monochromated beam condition with tiling and stitching for the whole Ar^+ cleaned area. The technique used for Ar^+ ion beam cleaning was deliberately set the beam to about 300 µm defocus and scanned pixel overlapping to 98% with the pixel dwell time as short as 50 – 100 ns. Such an approach for the sample cleaning results in not only removed surface

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contamination (oxidations and deposited carbon), but also smoothed the sample surface further even the beam is under the perpendicular incident to the sample surface.

TEM results acquired from 500 eV final cleaned CuO/AL interface TEM specimen are shown in figure 3 and 4. TEM specimens preparation could be optimized by using different ions at different sample preparation steps by switching types of ions freely, for example, Xe$^+$ beam protection layer can be deposited at 12 keV by MultiChem C+Pt gas mixing, and O$^+$ ion can be switched for rough milling such as diamond, then Xe$^+$ milling for cleaning and finally switch to low keV Ar$^+$ for the last step cleaning . All process can be carried out by one system, Hydra PFIB. PFIB can prepare TEM specimen successfully with the key factors of a proper protection cap layer and the use of STEM for RTM. Ar$^+$ ion beam at low kVs (5 keV to 500 eV) provides a much gentle milling allows the process in a controlled way.

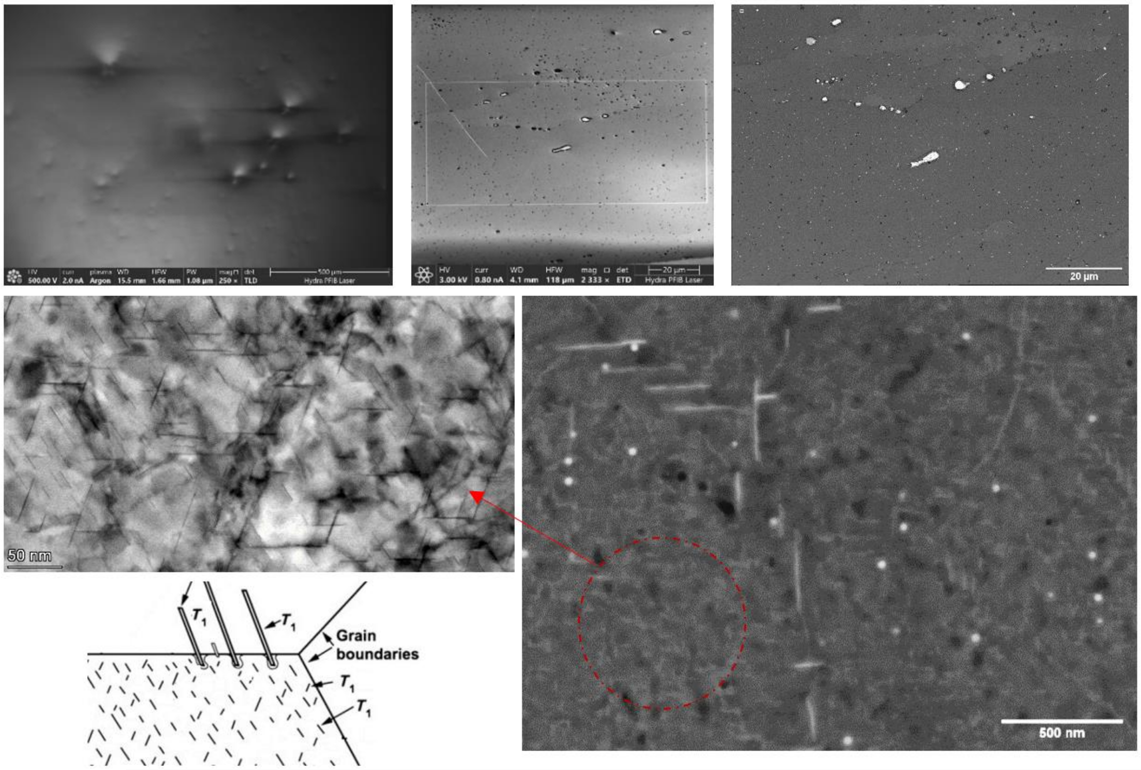

Figure 1. (Top left) 500 eV Ar ion beam image acquired from a contaminated Al-Li alloy surface. (Top center) SEM secondary image after 500 eV Ar ion beam clean for 30 minutes. The rectangle marker, 100 µm by 85 µm, was milled by Ar ion beam at 30 kV at 60 pA, the line width is ~ 150 nm. (Top right) A tile and stitched 3 keV SEM BSE images acquired by 437 tiles with a pixel size of 2.44nm/pixel from 500 eV cleaned Al-Li alloy surface, the large white phases and smaller white dots are the intermetallic phases including Al7Cu2(Fe,Mn) and Al20Cu2Mn3 . (bottom left) T1 phase in a Al-Li alloy is visible not only the large plate-shape at grain boundary but also small needle-shape inside the grain, the width of the large T1-plate is about 12 - 15 nm measured by line scan FWHM. The small white dot phase is Al20Cu2Mn3 dispersions. (Bottom right upper) TEM/STEM BF image from a plan view specimen prepared by low kVs Ar+ ion milling illustrating the T1 phase inside a grain. (bottom right lower) a diagram modified from reference [5] illustrates T1 at G.B and inside grain. The STEM image was acquired by Thermo Scientific Talos F200X TEM at 200 kV.

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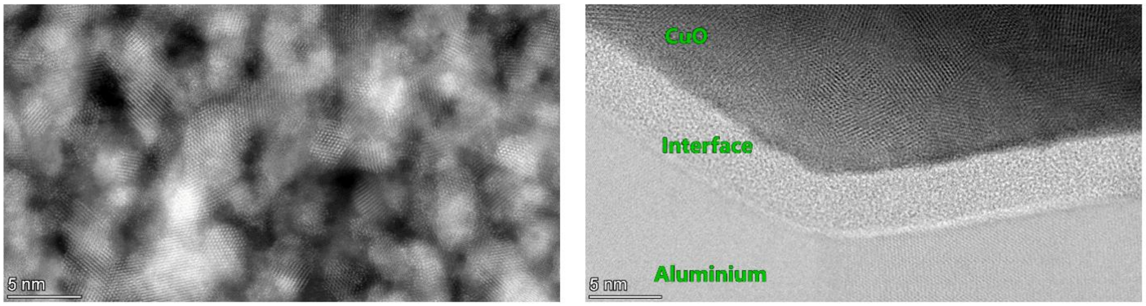

Figure 2. (left) STEM HAADF image shows Pt lattice image in protection cap layer deposited by 12keV Xe+ ion beam with MultiChem GIS gas mixing Pt+C. (right) STEM BF image shows the interface between CuO and Aluminum. The specimen is gallium free with a final Ar+ ion beam "washed" at 500 eV while under SEM/STEM RTM. Images were acquired from Thermo Scientific Spectra 300 TEM at 300 kV, image pixel size: 19 pm/pixel, Beam convergent angle: 30.0 mrad, current: 100 pA

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