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SIMS Detector on FIB/SEM DualBeam Microscopes for Material Science Applications

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For material science samples, micro-chemical measurement by energy dispersive x-ray microanalysis (EDX) with a DualBeam FIB/SEM microscope is a standard technique. However, this technique for detection of hydrogen is impossible, and the detection of lithium requires well defined measurement conditions and is very challenging [1]. Secondary Ion Mass Spectrometry (SIMS) is a materials chemical analysis technique relies on collection, separation of ions according to their mass-to-charge ratio (*m/z*) from detected secondary ions. The secondary ions are one of emitted signals from solid material surfaces by primary ion beam bombardment in a FIB/SEM DualBeam microscope.

Add-on a SIMS detector to a DualBeam FIB/SEM microscope to collect secondary ions from a FIB/SEM microscope allows focused ion beam to be used not only for major functions of sample preparations and nano-fabrications, but also for collecting sample chemical information by ion beam. The integrated SIMS to a FIB/SEM DualBeam microscope gives a far better depth (z) resolution for chemical analysis because ion beam interaction volume is in general orders of magnitude smaller than a common SEM/EDX technique at the same beam energy. FIB/SEM/SIMS also delivers a better lateral (x,y) resolution comparing to dedicated SIMS systems because the focused ion beam has a smaller probe size.

In this paper, we give an overview of an integrated ToF SIMS to FIB/SEM DualBeam for materials science applications, focusing on SIMS for nano-analysis and to detect light, low concentration elements. First, we give a short overview and discussion of the pros and cons of current mass analyzers FIB/SEM microscopes. Results presented in this paper were mainly acquired from a gallium FIB DualBeam with a compact ToF SIMS detector added on to it [2,3], although plasma FIB Xe+ beam is also mentioned.

High resolution nano SIMS results from a sample of mixed organic/inorganic nanoparticles and a sample with a low content of rubidium segregated in Cu(In,Ga)Se2 (CIGS) grain boundaries [4] are presented. SIMS signal enhancement in post-processing is described. SIMS images from nanoparticles and from a sample of WC with a binder material cobalt proved that a Gallium FIB/ToF SIMS is capable of doing nano SIMS analysis down to about 20 -30nm.

ToF SIMS results clearly show a distinguished advantage for the measurement of light element, in this case, lithium in an Al-Li alloy sample. By using FIB TEM specimen preparation as a link, we have tried to bridge microscopic chemical analysis techniques between FIB SEM SIMS measurements and TEM STEM EDS analysis in Al-Li sample. The TEM specimen was prepared as Gallium-free with a Plasma Xe^+ FIB from the same location where SIMS was performed. Sample correlations between different microscopes relies on SEM BSE images.

In conclusion, integrated ToF SIMS in a DualBeam SEM/FIB system allows the ion beam to be used not only for milling and deposition but also as a beam for chemical analysis. Light mass detection capability and good detection efficiencies makes the SIMS technique powerful for materials science research. FIB/SEM/SIMS gives better correlation between electron imaging techniques comparing to dedicated SIMS. Combining SIMS measurements in FIB/SEM microscope; linked with site-specific planar view TEM specimen preparation, and EDX analysis by HR TEM/STEM, provides near a complete sample chemical information for materials science research.

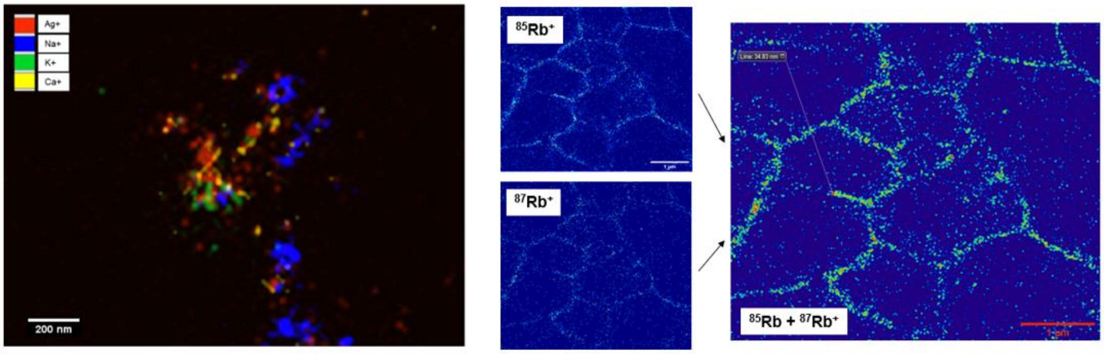

Figure 1. High resolution ToF SIMS images. (a) a color mixed ToF SIMS image of 107Ag+ and 109Ag+, 23Na+, 39K+, 40Ca+ from nanoparticles on silicon surface. (b) a low content heavy alkali element rubidium segregation in Cu(In,Ga)Se2 solar cell sample.

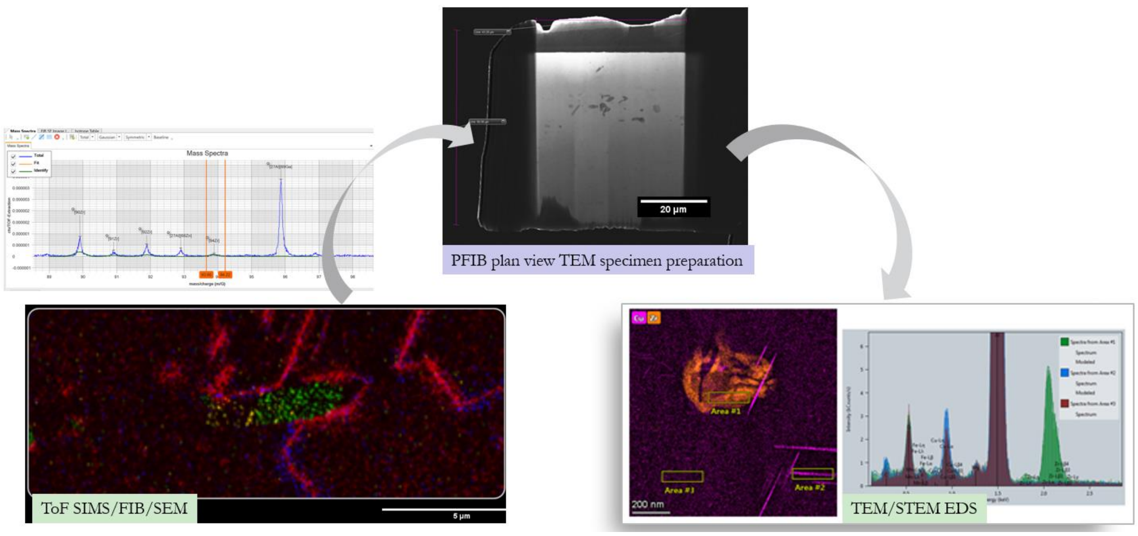

Figure 2. A combined Al-Li alloy sample chemical micro-/nano- measurements by ToF SIMS/FIB/SEM and STEM EDS by a TEM. The bridge of the analysis is a PFIB prepared gallium-free TEM specimen. The SIMS image shows lithium segregations at grain boundaries. Zirconium isotopes mass peaks are shown in the mass spectrum but was unable to form a SIMS image because of small isolated particles. TEM STEM EDS proved that the Zirconium particles in the alloy also proved that the Lithium-rich phase in needle shape.

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