

Secondary Ion Mass Spectrometry in the TEM: Isotope Specific High Resolution Correlative Imaging.

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In the context of an ever increasing complexity and size reduction of devices in materials science research, there is a pressing need for accurate characterization at high-spatial resolution and high-chemical sensitivity. Transmission Electron Microscopy (TEM) can offer sub-Å spatial information combined with electron spectroscopies yielding chemical and electronic information. However, the analytical TEM retains some limitations that make it unsuitable for applications requiring distinguishing isotopes, detecting trace elements below 0.1 at.% concentration or characterization of very light elements such as hydrogen, lithium or boron [1,2]. By contrast, Secondary Ion Mass Spectrometry (SIMS) has superior sensitivity (ppm range) and is capable of distinguishing isotopes and detecting all elements (including low-Z elements), but suffers from an inherent fundamental limitation in spatial resolution [3]. By correlating TEM with SIMS in-situ, it is possible to overcome their individual limitations and obtain information at high-resolution and high-sensitivity simultaneously.

In order to push the analytical limits of the TEM by adding SIMS capabilities, a dedicated, compact, high-performance mass spectrometer was developed in-house. The mass spectrometer, together with a Ga⁺ focused ion beam (FIB) column have been fitted in a modified FEI Tecnai F20 pole piece. A special sample holder which can be biased to high-potential (± 5 kV) was also developed to create an electric field above the specimen for an optimized collection of the secondary ions. This unique prototype [4,5] dedicated to parallel ion-electron spectrometry (PIES) combines all the conventional TEM information, scanning TEM, energy dispersive X-ray spectroscopy (EDX) and mass spectrometry, therefore allowing for correlative TEM imaging and ion-electron spectrometry (Fig. 1). The main advantage of the in-situ approach is its capability to analyse the same area of interest of any sample without need of transferring the sample from one instrument to another one, which would result in a number of artefacts ranging from surface contamination to issues with localizing exactly the same ROIs. Moreover, the in-situ correlative approach allows fast and multiple interlacing between the different imaging and analysis modes.

In this work we sum up the characteristics and advantages of such an instrument, focusing on the case studies of materials containing very low atomic number elements, namely H, Li or B, with, in addition, various isotopes. These elements are known to be extremely difficult or impossible to study by conventional EDX. Here we demonstrate the ability of the PIES instrument to map these elements (Fig. 2) and study the best approach for correlating TEM and SIMS results. Moreover, we show how the information in the EDX spectrum can be correlated with SIMS for quantification of trace elements.

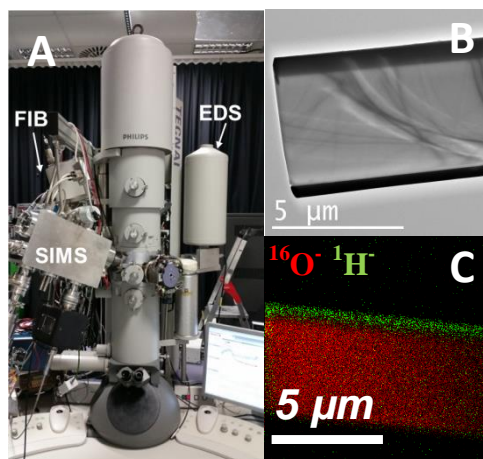


Figure 1: A) photo of the TEM-SIMS prototype instrument. B) TEM image of a FIB lamella with corresponding $^{16}\text{O}^-$ and $^1\text{H}^-$ maps (C).

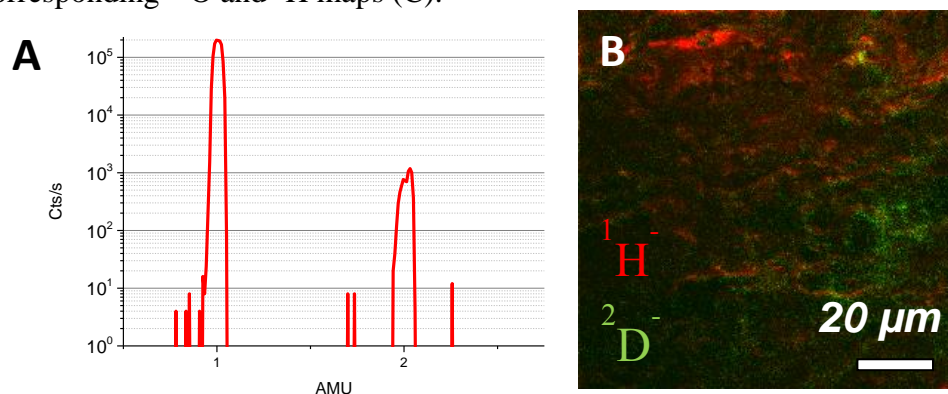


Figure 2: A) Mass spectrum for masses 1 and 2. B) Combination of $^1\text{H}^-$ and $^2\text{D}^-$ maps in red and green respectively from the surface of a Si wafer etched in 0.1M NaOD solution.

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