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High Speed TEM Sample Preparation by Xe FIB.

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Preparation of Transmission Electron Microscope (TEM) samples by Focused Ion Beam (FIB) milling is one of the most precise techniques now routinely used for example in failure analysis or material science. These TEM samples are commonly prepared using Ga FIB technology, starting more than 20 years ago [1-3]. Presently FIB columns are commonly combined with the Scanning Electron Microscopy (SEM) technique for observation during the preparation process and for the enhanced navigation, end-pointing and analytical capabilities. This FIB-SEM combination makes TEM sample preparation process much easier than before. However, when TEM sample size increases to several tens of µm, the preparation time increases considerably due to low Ga FIB milling speed. This preparation time drops the throughput of the method and limits the size of Ga-FIB-prepared samples to 5-10 µm. The new Xenon plasma FIB tool equipped by ECR plasma ion source (i-FIB) offers more than 50x higher milling speed, while still having sharp current distribution well adapted for sub-100 nm thickness samples preparation required for high-quality TEM images. TEM sample preparation obtained with a single-beam plasma FIB instrument has already been shown [4]. Nevertheless, the combination of the high resolution SEM and the high current plasma FIB [5, 6] offers more chance to speed up the preparation of extremely large TEM samples. This lecture will present the first results of the large TEM lamella preparation by ECR plasma FIB (Orsay Physics) which currently equips the SEM-FIB TESCAN FERA3 instrument. The different steps of extra-large TEM lamella preparation will be detailed (deposition of protective layer, rough milling, polishing and TEM imaging of the prepared sample).

The main challenge for TEM sample preparation using Xe plasma FIB seems to be the curtaining effect reduction. This can be achieved thanks to the wide range of FIB currents and optimized sample orientation. We are considering here dimensions of about at least 100 µm by 50 µm whereas for standard Ga FIB it is common to prepare samples about 20 µm by 10 µm for larger ones. The processing time is similar in both cases (Xe or Ga beam) but the removed volume can be considerably bigger in the case of large TEM preparation by Xe beam. This kind of very large TEM lamella is not conceivable in a reasonable time by Ga FIB. Due to the properties of Xe beam the rough milling step is a really fast process, but the polishing method is more complicated because of the increased spot size when compared to Ga beam at the same low current. However, after the preparation using 30 keV Xe beam only, we have obtained atomic resolution in TEM at 300 keV. It looks like due to the smaller ion range of Xe^+ compared to Ga^+ in the sample [4] we can get better surface quality with Xe^+ ions. Not only use of the noble gas will avoid Ga contamination but also we are expecting even better results with a final polishing at lower beam energy.

This presentation will show how to prepare very large TEM lamella (fig 1) with very good quality (fig 2) on which high resolution TEM imaging is possible (fig 2).
Moreover, there is the obvious need to automatically prepare lamellas from many sample sites. By using high current Xe plasma FIB milling, it would bring a significant time savings. This goal can be achieved with software like TESCAN AutoSlicer for automated multi-site cross sectioning and TEM sample

preparation. This method will be described in detail and the possible combination with Ga FIB milling and its influence on the preparation time will be discussed.

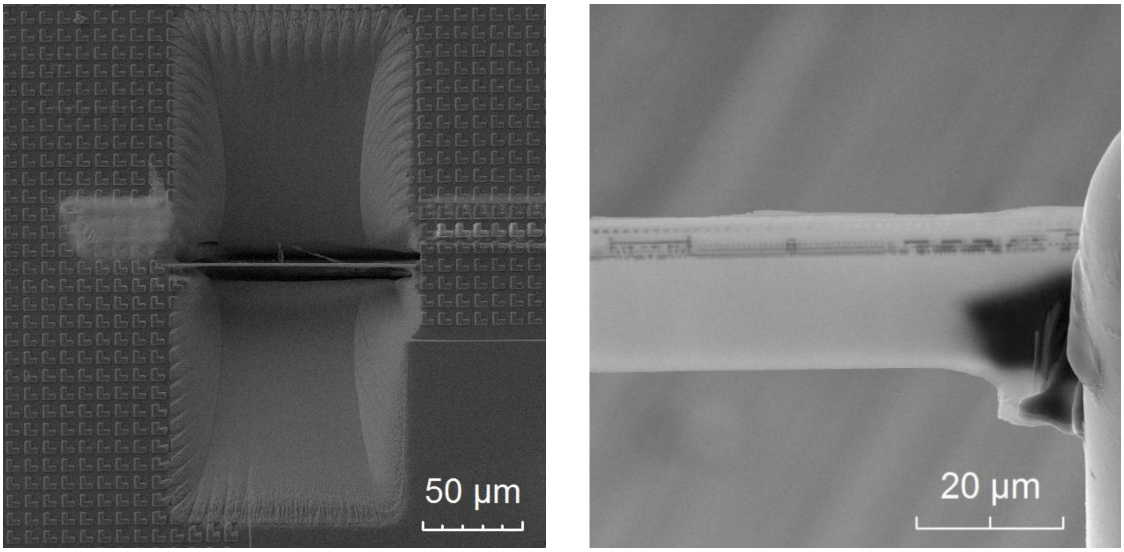

Figure 1. SEM images of a very large TEM lamella (125 µm long). The lamella has been prepared using 30 keV Xe beam.

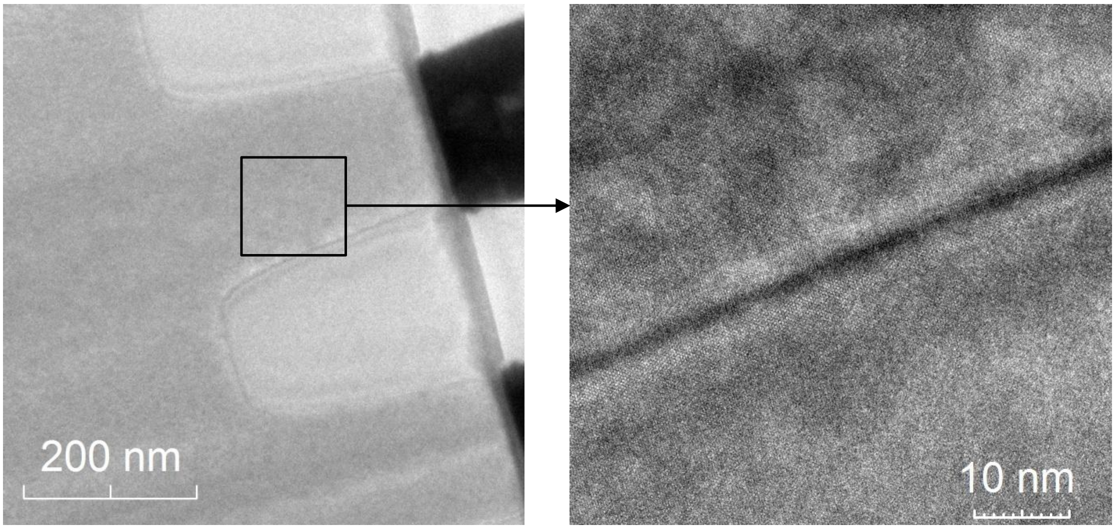

Figure 2. TEM and High Resolution TEM images obtained at beam energy 300 keV.

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