Low-Energy Focused Ion Beam Milling Provides Reduced Damage During TEM Sample Preparation

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Introduction

The combined focused ion beam (FIB) and scanning electron microscope (SEM), known as the DualBeam, is well-known for its unique ability to produce site-specific thin samples starting from bulk and then attaching the section to a transmission electron microscope (TEM) grid, all *in-situ*. It has been reported that producing a thin sample using a 30 kV gallium FIB creates surface damage several tens of nanometers deep. However, recent DualBeam technology improvements now enable the FIB to produce thin samples with a thickness well below 50 nanometers and deliver a tightly focused ion beam at an energy of 2 kV and below, which dramatically reduces the damage depth to as low as 1 to 2 nanometers in typical materials, such as silicon.

One example of a challenging application being addressed with this capability is the failure analysis of LaB_6 emitters. LaB_6 emitters are widely used in electron microscopy as high-brightness electron sources, owing to their favorable properties for thermionic emission: low work function and high melting point. The emitter consists of an LaB_6 single crystal with a carefully shaped tip (Figure 1).

During its lifetime, an LaB_6 emitter can undergo different types of failure. Some failures can be related to a source in manufacturing (mechanical failure, defect in the crystal, etc), others to its operation (for instance, overcurrent, which may lead to mechanical failure), or to its environment. In the latter case, bad vacuum will cause gas molecules in the gun to be ionized by collisions with the electron beam. The positive ions thus formed are accelerated towards the emitter and may sputter away the cathode material.

Sample Preparation of LaB_6 on the DualBeam

Characterizing the LaB_6 emitter by means of high resolution TEM (HR-TEM) requires from one to several thin samples to be prepared, which must meet certain criteria: the preparation of each sample should be site-specific and deliver lamellas with a thickness well below 50 nm and of highest possible quality.

The work described here was performed on a Helios NanoLab DualBeam (FEI Company, Hillsboro, OR). Its high-stability platform, sub-2 kV FIB mode, and integrated *in-situ* preparation capabilities are useful for preparing high-quality thin sections.

Two sites of interest are identified on the LaB_6 emitter (Figure 2a): the apex, exposed to heavy particle bombardment during its lifetime, and the significantly less-exposed emitter side. From each site, after protecting the sample surface using beam-induced deposition of platinum, a thin sample was prepared using different pre-thinning techniques. Finally, both samples were thinned using increasingly lower FIB energies.

The final step consisted of a 2-kV FIB polishing step. Most of the protective platinum was removed, and some areas near the actual sample surface showed very high electron transparency (Figure 3). Although assessing the sample quality is premature at this point, this work already demonstrates the flexibility offered by DualBeams for very thin sample preparation, as well as their site specificity and end-pointing capabilities. Both samples were then transferred to the TEM, without further preparation.

HR-TEM Microscopy Study on LaB_6

High-resolution TEM is an excellent method to study the structure of materials at the atomic level. In combination with spherical aberration (C_s) correction, HR-TEM images directly visualize the atomic order of crystals, and determination of the atomic positions in the image is usually straightforward. The most limiting factor for the determination of atomic positions in C_s-corrected images is the thickness of the sample in the electron beam direction.

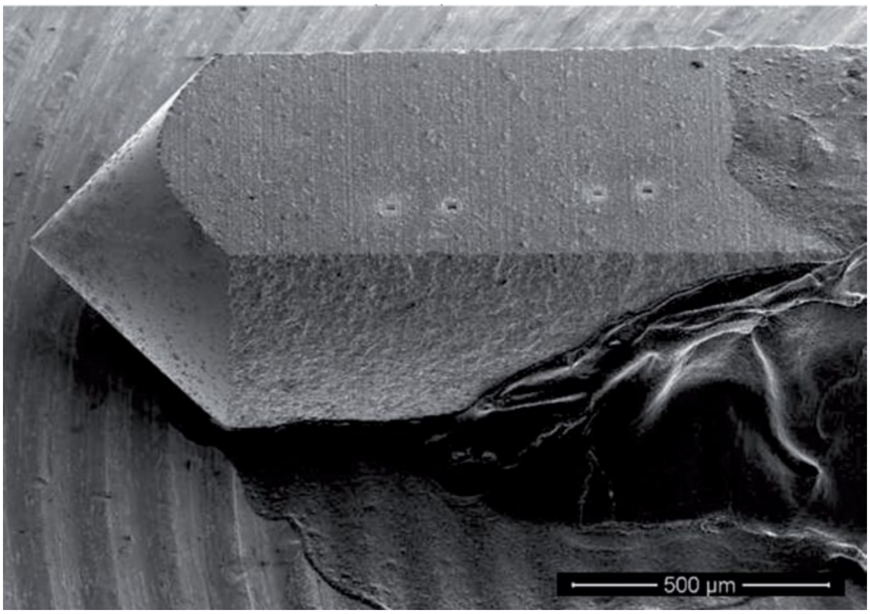

Figure 1: LaB_6 emitter overview, SEM image.

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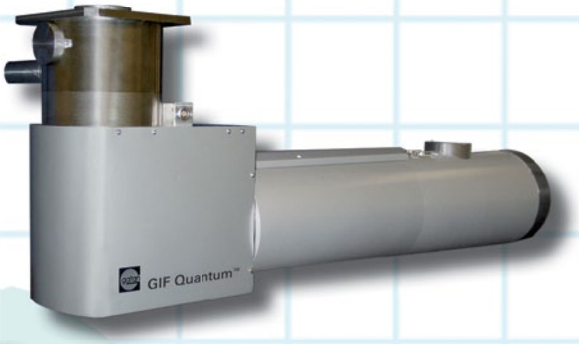
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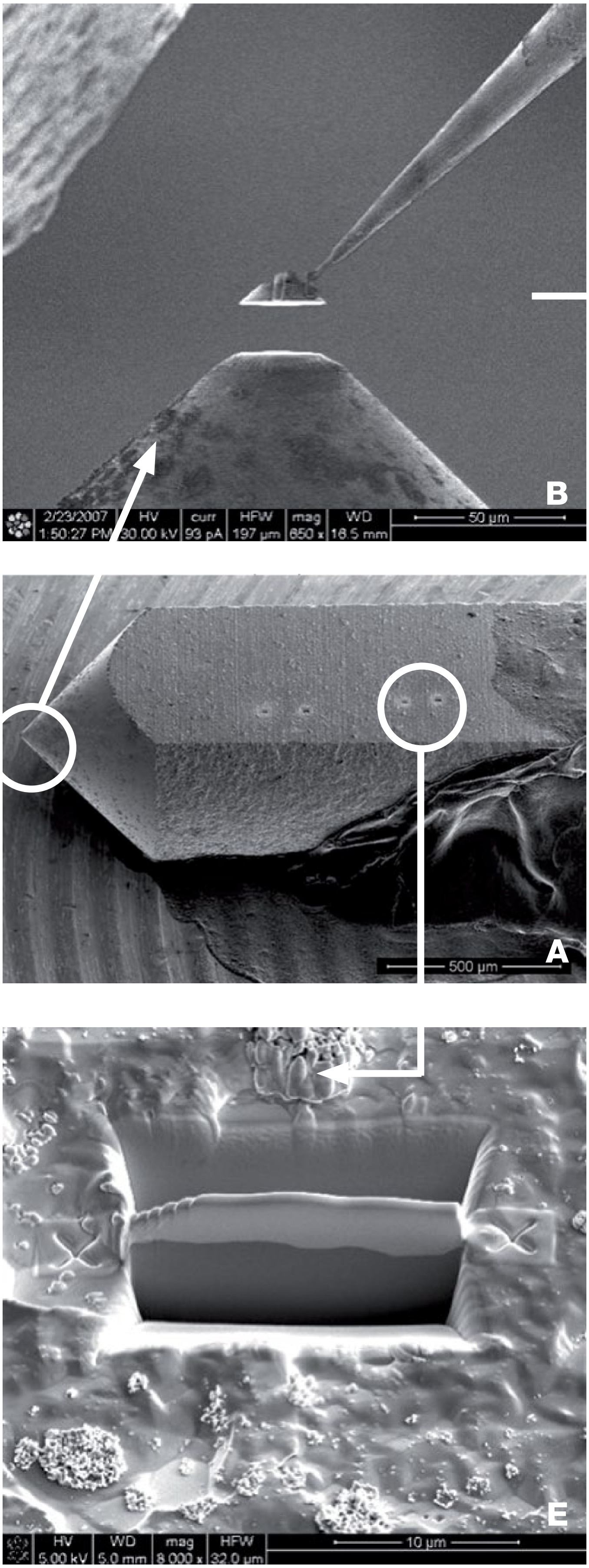

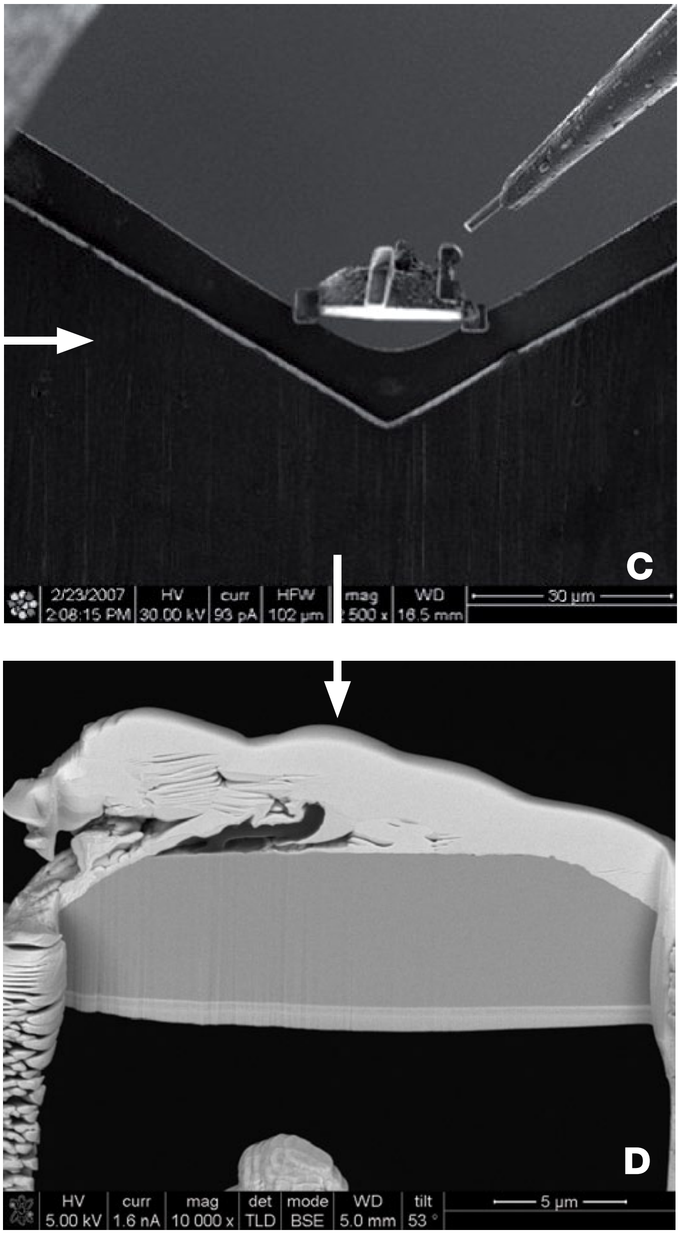

Figure 2: Starting from the bulk LaB_6 emitter (a), manual preparation on the apex by chunk lift-out (b, c) and subsequent FIB pre-thinning (d), and by automated preparation on the side (e).

In thick samples, multiple scattering processes of the transmitted electrons occur that can destroy the clear relationship between the dark contrast in the image and the atomic position. This makes a direct interpretation of the image impossible, and the information about the real atomic positions can only be recovered by time-consuming image simulation. Therefore, an excellent sample preparation that produces a quality thin specimen is required to obtain the best results in atomic resolution TEM.

The HR-TEM images here were acquired at 300 kV on a Titan 80-300 (FEI Company, Hillsboro, OR) equipped with a spherical aberration corrector for the TEM mode. The cubic structure model of the LaB_6 is plotted on the first HR-TEM image (Figure 4). Spherical aberration correction enables the

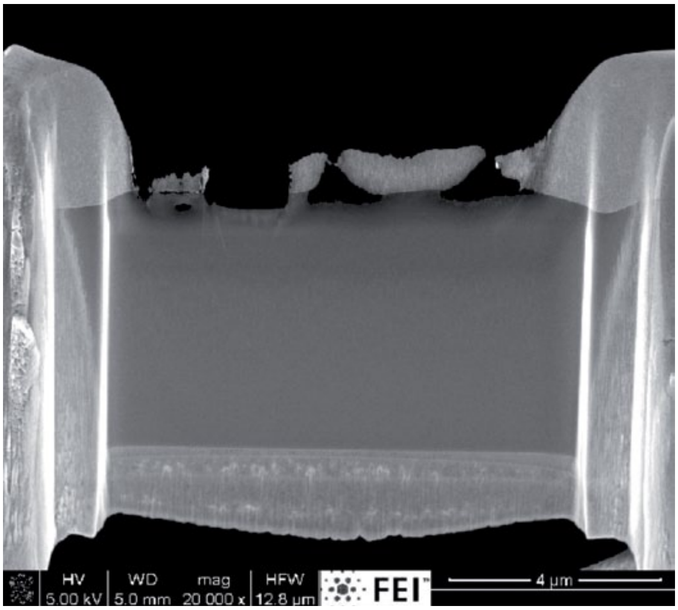

Figure 3: Final thinned sample ready for HR-TEM.

atomic structure on the thin lamella to be seen directly in the HR-TEM images.

Because of the extreme thinness of the sample, it behaves like a "weak phase object," and the atoms are imaged in dark contrast. The clearly apparent positions of the boron octahedron and the lanthanum atoms demonstrate the power of C_s-corrected TEM and DualBeam sample preparation to visualize atomic structure. The power spectrum of the image in the upper-left corner demonstrates that information below 100 pm is transferred in the image.

To quantify the lamella thickness, some image simulation with variation in focus and sample thickness was calculated (Figure 5). In the simulation, an area is marked indicating the region of focus and thickness where the experimental image best fits the simulation. The result gives an estimated local thickness below 10nm for the LaB_6 lamella.

The complex electron wave exiting the sample contains information about the material in both its amplitude and its phase. Any single image contains only the intensity distribution (amplitude) and cannot reveal all the information potentially available from HR-TEM analysis. However, focus series reconstruction, which uses a series of images acquired at different focus values, can recover the phase information and reconstruct the full complex form of the exit wave. An example of the phase part of the LaB_6 image is shown in Figure 6. From the complex wave, residual aberrations can be compensated to improve the image quality even further or to access information about the occupancy of single columns in the beam direction.

A sample of the tip region of the thermal emitter was obtained using the site-specific sample preparation capability of a DualBeam and subsequently imaged with C_s-corrected HR-TEM. In the TEM images, small rectangular areas (approx. 2 x 2 nm) are visible where the crystalline structure of the LaB_6 is destroyed (Figure 7). This gives a new insight into the aging process of a thermal emitter tip in an electron microscope.

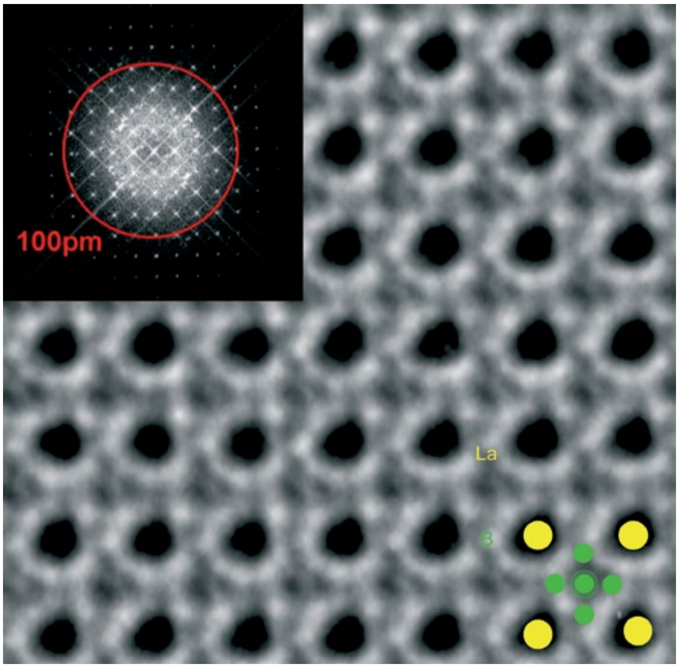

Figure 4: C_s-corrected HR-TEM image of LaB_6 with model of the cubic structure (green = boron, yellow = lanthanum) and power spectrum of the image.

Conclusion

This case study on the thermal electron emitter material of LaB_6 demonstrates some significant advances in the preparation of thin sections for high-resolution S/TEM (scanning/transmission electron microscopy) and the progress that has been made in atomic resolution imaging using a

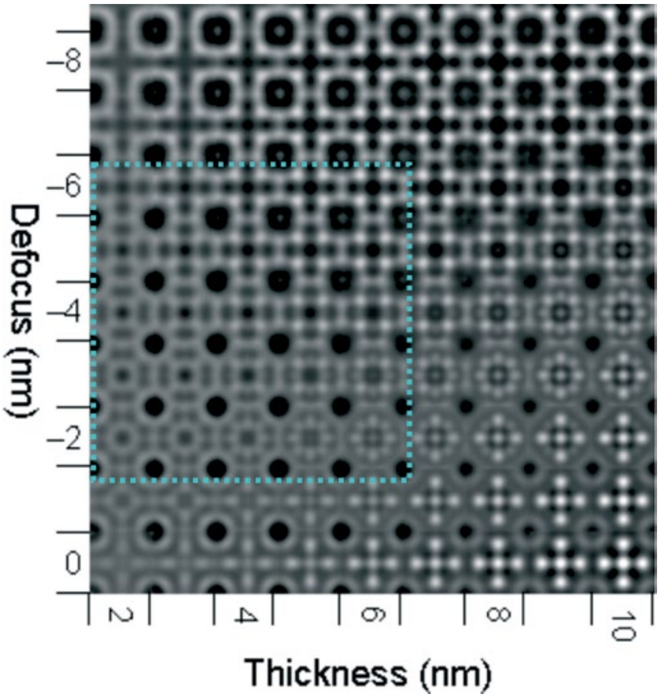

Figure 5: Image simulation of the HR-TEM image depending on the focus of the objective lens and thickness of the LaB_6 crystal. The dotted region closely matches the experimental image in Figure 3.

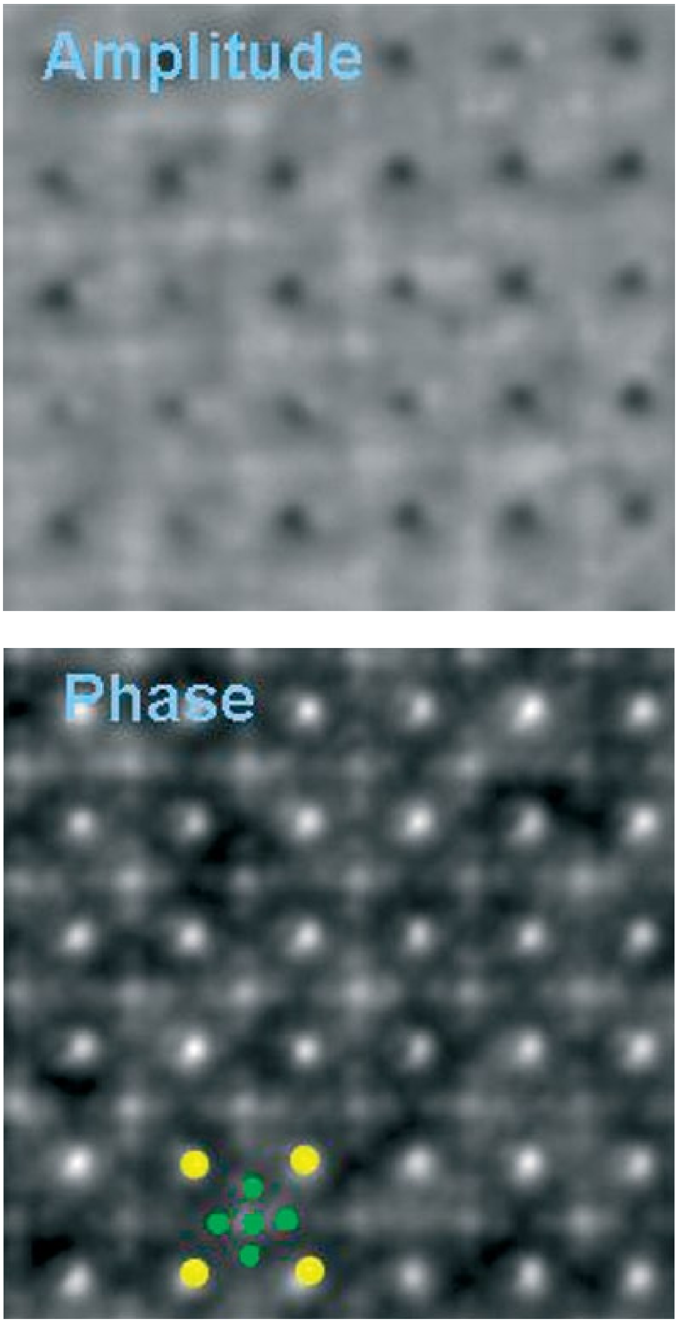

Figure 6: Focus series reconstruction of the amplitude and the phase of the complex exit wave with TrueImage from a series of 20 images.

spherical aberration corrected TEM. The high precision of FIB milling and the ability to remove surface damage by milling at low energies provide the superior sample quality required for atomic-level resolution. The combination of DualBeam sample preparation and high-resolution TEM provides a reliable method for solving many current challenges in nanotechnology—a discipline that is focused on better understanding of materials at the atomic level. In this specific example, insight into the atomic structure of the LaB_6 emitter can help to identify the causes of premature failures and ultimately extend the useful life of these costly consumables.

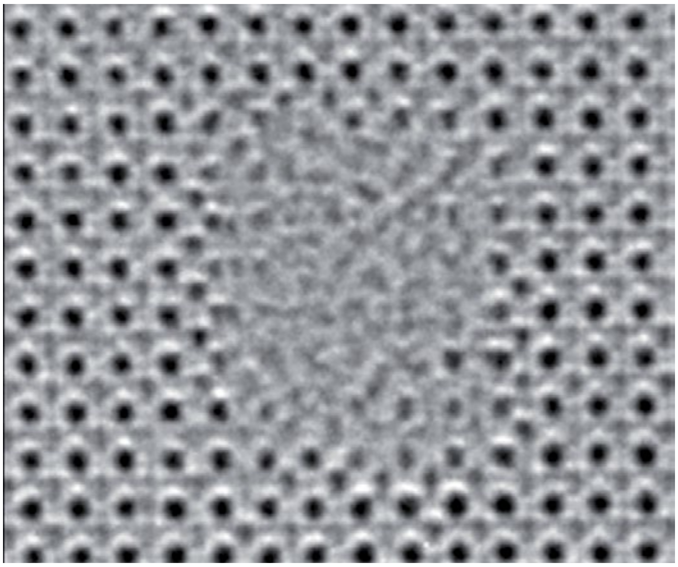

Figure 7: Cs-corrected HR-TEM image on the area of the emitter tip.

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