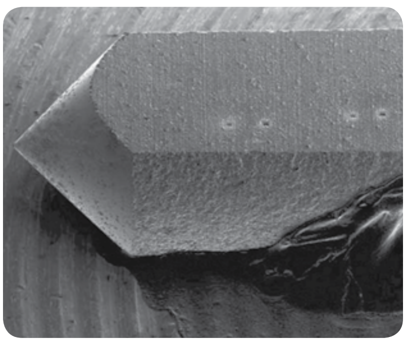
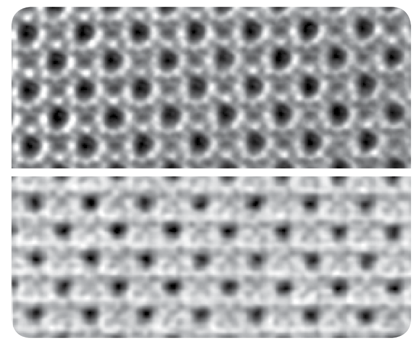
Using DualBeam to prepare the highest quality samples for atomic scale HR-S/TEM

LaB_6 emitter failure case study



↑ LaB_6 emitter overview, SEM image.

↑ Final thinned sample ready for HR-S/TEM.



↑ Comparison between Cs-corrected HR-TEM (top) and Cs-corrected annular bright field STEM (ABF) imaging (bottom).

The LaB_6 emitter case study

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.

During its lifetime, an LaB_6 emitter can undergo different types of failure. Some can be related to the source 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.

This study offers an unprecedented characterization of a worn-out LaB_6 emitter down to the atomic level.

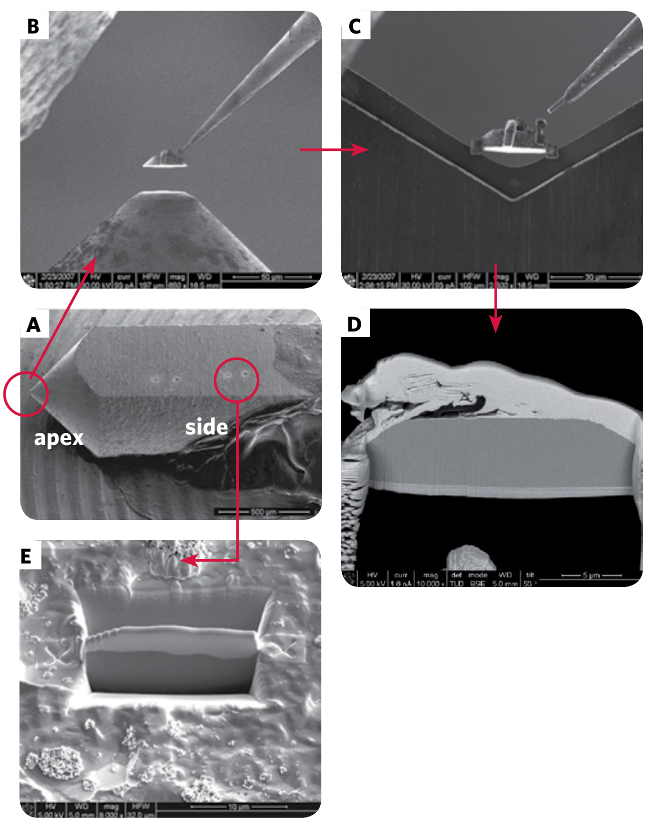
KEY BENEFITS
Increase your throughput by minimizing the preparation steps needed for a site-specific S/TEM sample
Maximize your imaging quality by Cs-corrected S/TEM imaging in combination with DualBeam™ sample preparation of ultra thin TEM lamellas
Minimize your time to result with fast automated or ultra precise manual sample preparation on demanding materials
Increase your thin sample preparation success rate and flexibility by *in situ* lift-out
Increase your imaging resolution by Cs-corrected S/TEM on Titan™ Themis
Maximize the information about your material on the atomic level using quantitative HR-TEM with focus series reconstruction on Titan Themis and TrueImage Atlas

Site-specific thin sample preparation for HR-TEM

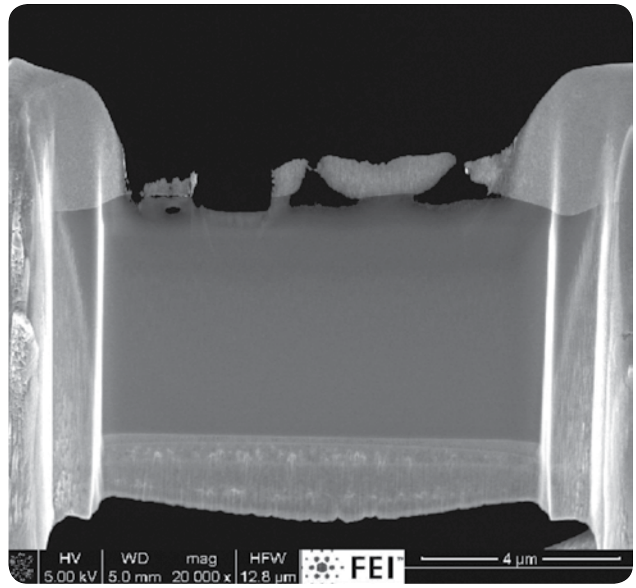
Characterizing the LaB_6 emitter by means of high resolution transmission electron microscopy (HR-TEM) requires from one to several thin samples to be prepared, which must meet certain criteria: the preparation of each sample should be site specific and deliver lamellas with a thickness well below 50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

DualBeams are renowned today for their unique ability to deliver site specific thin samples starting from bulk, and also to weld them to a TEM grid, all *in situ*. It has often been reported that producing a thin sample using a 30 keV gallium focused ion beam (FIB) creates damage several tens of nanometers deep. However, the recent DualBeams have introduced low energy FIB polishing (2 keV and below), which has dramatically reduced the damage depth down to 1 to 2 nm in typical materials such as silicon.

For the LaB_6 study, the Helios NanoLab™ DualBeam offered the best match for outstanding thin sample preparation for HR-TEM, owing to its high stability platform, high performance sub-2 keV FIB mode and integrated *in situ* preparation capabilities.



↑ **Figure 2A.** Starting from the bulk LaB_6 emitter (A), a FIB cut is used to separate the apex chunk for lift-out (B) and transfer to TEM grid (C) for further thinning (D); for the side sample, FIB automation was used for pre-thinning (E).



↑ **Figure 2B.** Final thinned sample ready for HR-S/TEM.

Preparing outstanding quality LaB_6 thin samples using Helios NanoLab

Two sites of interest are identified on the LaB_6 emitter (**Figure 2A, A**): the apex, exposed to heavy particle bombardment during its lifetime, and the significantly less exposed side. From each site, after protecting the sample surface using beam induced deposition of platinum, a thin sample is prepared using different pre-thinning techniques:

- **Apex:** because of its geometry, no automated pre-thinning is possible there. To optimize the position and orientation of the final lamella, a whole apex chunk is cut by the FIB (**B**), transferred and welded onto the TEM grid (**C**) and pre-thinned using high keV FIB (**D**)
- **Side:** the side sample is directly pre-thinned on the emitter using FEI's automated AutoTEM™ tool (**E**), and then transferred and welded onto the TEM grid

Finally, both samples are thinned using increasingly lower FIB energies. The final step consisted of a 2 keV FIB polishing step (**Figure 2B, F**). Most of the protective platinum is removed, and some areas near the actual sample surface show very high electron transparency.

While 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 are now transferred to the Titan Themis, without any specific post-preparation.

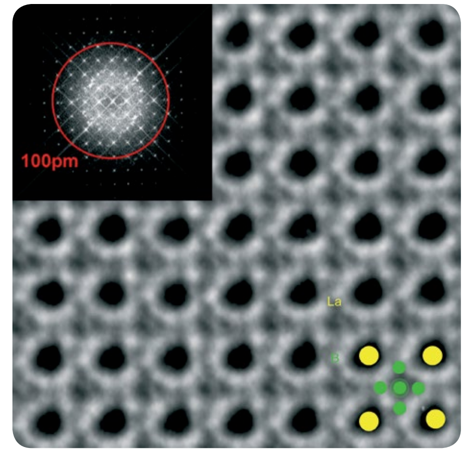
HR-TEM microscopy study on LaB_6

HR-TEM is an excellent method for study of the structure of materials at the atomic level. In combination with spherical aberration (Cs) correction, HR-TEM images provide direct visualization of the atomic order of crystals and determining atomic positions in the image is straightforward. The most limiting factor for this straightforward determination of the atomic position in the Cs-corrected images is the thickness of the sample in the electron beam direction. In thick samples, multiple scattering processes of the transmitted electrons occur, which after a material dependent sample thickness destroy the clear relationship between the dark contrast in the image and the atomic position (for more details please see dynamic image theory). This makes a direct interpretation of the image impossible and the information of the real atomic positions can only be recovered by time consuming image simulation. Therefore, excellent sample preparation is required to obtain the best results in atomic resolution transmission electron microscopy. The HR-TEM images were acquired on a Titan equipped with a spherical aberration corrector for the TEM mode at 300 kV.

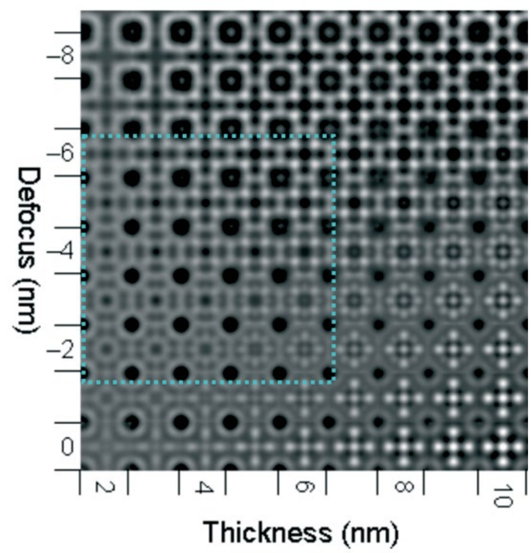
The cubic structure model of the LaB_6 is plotted on the first HR-TEM image (**Figure 3**). The HR-TEM images acquired with spherical aberration correction enables the atomic structure on the thin lamella to be seen directly. Due to the extreme thinness of the sample it behaves like a 'weak phase object' and the atoms are imaged in dark contrast. The position of the boron octaheder and the lanthanum atoms in the cubic structure is clearly and directly visualized, which proves the power of a Cs-corrected Titan Themis in combination with a superb sample preparation tool. The power spectrum of the image in the upper left corner proves that information below 100 pm is transferred in the image. To quantify the lamella thickness, some image simulation with a variation in focus and the thickness of the sample has been calculated (**Figure 4**). In the simulation, an area is marked indicating the region of focus and thickness where the experimental image fits nicely to the simulation. The result gives an estimate of 2 to 6 nm thickness for the LaB_6 lamella cut by the Helios NanoLab 50 series DualBeam. This proves the excellent quality of the sample preparation achieved on this tool.

This case study on the thermal electron emitter material of LaB_6 demonstrates impressively the progress made in FIB and DualBeam to support preparation of the highest quality S/TEM samples using the Helios NanoLab; and the progress in atomic resolution imaging using a spherical aberration corrected Titan S/TEM. The combination of these tools provides the best solution for solving challenges in nanotechnology, which require understanding of the materials on the atomic level.

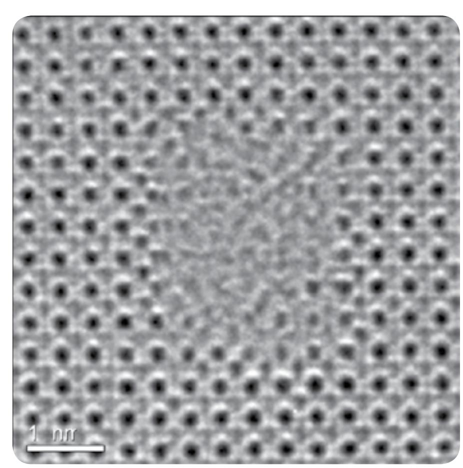
Because of the site specific sample preparation capability of a DualBeam, a sample of the tip region of the thermal emitter can be obtained. When looking at the HR-TEM images, small rectangular areas of approximately 2x2 nm can be found where the crystalline structure of the LaB_6 is destroyed (**Figure 5**). This gives a new insight into the aging process of a thermal emitter tip in an electron microscope and will be discussed elsewhere.



↑ **Figure 3.** Cs-corrected HR-TEM image of LaB_6 with model of the structure and power spectrum of the image.



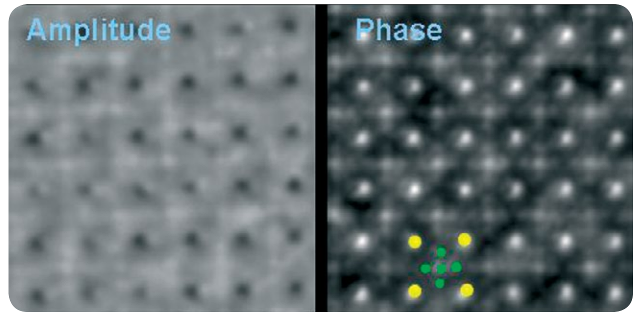
↑ **Figure 4.** Image simulation of the HR-TEM image depending on the focus of the objective lens and thickness of the LaB_6 crystal.



↑ **Figure 5.** Cs-corrected HR-TEM image on the area of the l emitter tip.

The electron wave created by the sample is a complex wave containing amplitude and phase information of the material. When taking only single images, valuable information about the material is lost and only the intensity distribution of this complex wave is obtained.

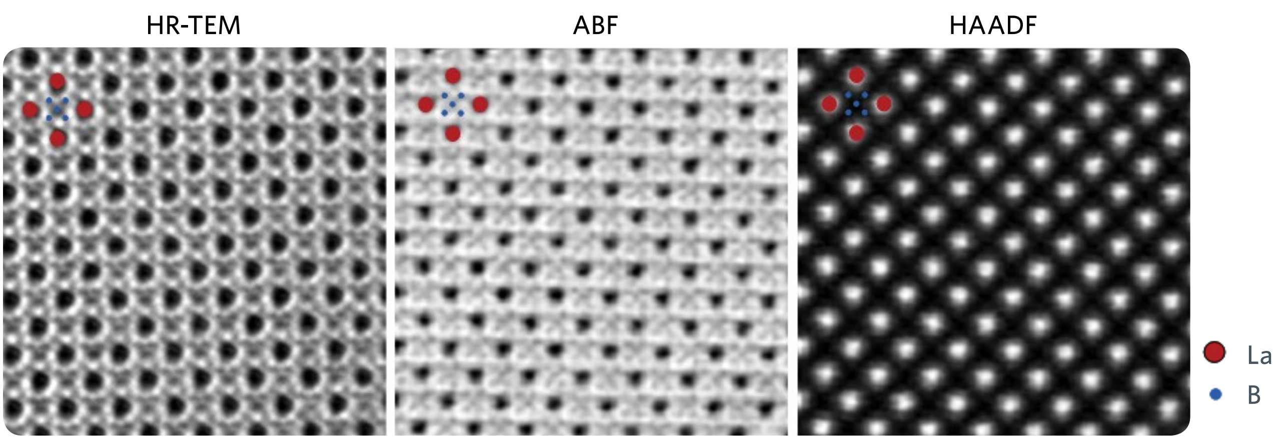
By applying a method of focus series reconstruction (FEI's TrueImage™ software) using a series of images in different focus, the complex wave of the sample is obtained. An example on the LaB_6 is shown on the left side (**Figure 6**). From the complex wave, residual aberrations can be compensated to improve the image quality even further or to access information about the occupancy of single columns in the beam direction.



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

HR-STEM microscopy study on LaB_6

In order to prove the quality of the sample preparation by the Helios NanoLab DualBeam, HR-STEM images were acquired on the LaB_6 cut using a Cs-corrected Titan S/TEM. The images are compared to the HR-TEM results in **Figure 7**. A clean surface is required to obtain the best HR-STEM images, so that the small beam created by the Cs-corrector is not broadened by an amorphous surface layer. Such an amorphous layer dampens the contrast significantly, which is created by the crystalline part of the specimen. The contrast of light elements like boron is quite small due to their weak scattering power and therefore a clean surface is mandatory to obtain the best results in angular bright field imaging (ABF). The HR-STEM results are as good as the HR-TEM results and prove that the sample thickness and the cleanliness of the specimen is excellent.



↑ **Figure 7.** Comparison between Cs-corrected HR-TEM imaging, Cs-corrected annular bright field STEM (ABF) and HAADF STEM imaging on LaB_6 crystal in [100] projection at 300 kV acceleration voltage. The boron lattice can be visualized using HR-TEM and ABF imaging, while it is invisible in the HAADF image.

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