Nanoscale sculpting of ferromagnetic structures by electron beam ablation

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Abstract. We report an electron beam (e-beam) fabrication method to produce sub-5 nm structures, e.g. nanohole-arrays, nanojunctions and nanotips inside a TEM. The method is demonstrated using ferromagnetic nickel cross-sectional TEM specimens as well as electrochemically etched nickel tips. Different e-beam shapes and electron guns are compared, including point versus line focus and field-emission versus LaB$_6$ guns. As an extension of this 2-D patterning of nanostructures, a 3-D nanofabrication technique has been introduced using a high tilt tomographic holder. An electrochemically etched nickel tip of initially 18 nm in radius is sculpted down to sub-5 nm diameter. In some cases, mostly in 3-D nanofabrication, a protrusion during hole drilling was observed and confirmed by EELS as pure nickel.

1. Introduction, motivation and experimental details
Standard electron beam lithography and focused ion beam fabrication methods are widely used in semiconductor industries to produce nanostructures with a smallest dimension of ~20 nm. On the other hand, scanning probe techniques are used to realize structures on planar surfaces with atomic level precision. Bridging the gap in length-scale of 1-20 nm between these two groups of techniques is still challenging and searching for new fabrication methods is vital. TEM/STEM-based electron beam nanofabrication, known since the early 1980s, has recently been rediscovered as promising approach for nanostructuring of semiconductors, insulators and most recently metal nanostructures [1-6]. We explore advancements of this e-beam structuring technique in various aspects, concentrating on e-beam shape, influence of electron gun type, and sculpting from various viewing directions.

Two test samples have been prepared: A nickel thin film was sputtered on Si substrate and subsequently turned into an electron transparent ribbon prepared by the standard cross-sectional TEM sample preparation method [7]. The other sample is an electrochemically etched nanotip fabricated from nickel microwires using a modified SPM tip making method as detailed elsewhere [8]. The FEGTEM used is a JEM 2010F at 200kV, which provides the finest beam focusing, while the LaB$_6$ technology is represented by a JEM 3010 at 300kV, which provides the highest beam currents.
2. Results and discussions

2.1. FEGTEM nanofabrication with various beam shapes

Tuning the beam shape via condenser astigmatism and defocus into, e.g. point, line, or slightly underfocused probes with a sharp central maximum (Figure 1) was found to be an effective method to produce differently shaped nanostructures in the FEGTEM. Figure 1a shows two parallel arrays of holes sculpted on a TEM cross-sectional nickel specimen using point focused probe in order to explore the effect of increasing probe diameters. Figure 1b shows experiments to explore astigmatism: a slightly underfocused probe with a sharp central maximum with different diameters is seen to produce triangular shaped holes due to residual three-fold condenser astigmatism. Next to it, a parallel line focused probe is applied, which generates a line perforating the nickel film in a fine sequence of holes, presumably revealing thickness fluctuations. Finally figure 1c shows an array of 3x4 holes of ~4 nm in diameter and separated by ~12 nm, sculpted using a point focused probe.

![Figure 1](image1.png)

Figure 1: (a-c) Nanopattern sculpting on a nickel cross-sectional TEM specimen using various probe shapes (JEOL JEM 2010F at 200kV). [A] Point focused, [B] Slightly underfocused, [C] Line focused.

2.2. LaB₆ TEM nanofabrication of nanoconstrictions

LaB₆ gun e-beam sculpting was implemented mainly for nanojunction fabrication. Figure 2a shows a hole drilled with 10 nm radius indicating the focusing capability of a thermionic gun using the same type of cross-sectional nickel TEM specimen as for figure 1. Iterating the hole drilling by moving the beam from the centre of the Ni film towards both the laterally connected Si substrate and the specimen hole sides leads to the sculpting of a 2 nm wide nanojunction as shown in figure 2b. The junction thereby not only gets laterally constricted but also thinned, down to possible fracture, as the rather broad Gaussian beam profile of the probe is leading to delocalised ablation. The e-beam irradiation by the highly intense LaB₆ focused probe is also found to change the microstructure of the thin film (Figure 2c). Nano-sized grains of around ~7 nm in diameter were observed along the edge of the hole, indicating beam-induced irradiative quasi-melting, likely far below the nickel melting temperature.

![Figure 2](image2.png)

Figure 2: (a) Holey TEM specimen with a 10 nm radius hole in a Ni film. (b) A nanojunction of 2 nm created by iteratively drilling through the Ni film towards both the Si substrate and the specimen hole sides. (c) Microstructure change in the Ni film near the hole edge due to beam-induced irradiative quasi-melting.

The fabrication of free-standing nanostructures in this way is a method in early stages of development. To evaluate the potential, we explore the comparison to the more established method of patterning of metal thin films in plan-view geometry, deposited on TEM support films, using commercial Raith 150 electron beam lithography system. Some preliminary results are shown in figure 3 for comparison. Particularly attractive seems a combination of both techniques using holey support films which would combine the benefits of ultrafine nanostructuring with the ease of contacting the nanoconstrictions to external circuits.
Figure 2: Nanohole, nanojunction sculpting and structural change studies (JEOL JEM 3010 at 300 kV).

Figure 3: Nickel nanostructures with (a) a 100x50x10 nm$^3$ constriction and (b) with a gap of ~20 nm, fabricated by conventional e-beam lithography on a Si$_3$N$_4$ TEM support grid.

2.3. **Tomographic nanofabrication**

Some nanostructures are intrinsically three-dimensional in design and cannot be fabricated with the planar methods of 2.1/2.2. However, usage of a high tilt tomographic holder [9] provides a solution as demonstrated for sharpening of a nickel nanotip, which after etching would still be too large in tip radius for many applications, such as nanobridge or breakjunction formation experiments.

Figure 4: (a) Initial nanotip of 18 nm radius. (b,c) Nanotip sharpened in two mutually perpendicular directions.

The tip (Figure 4a) with initially 18 nm radius was mounted along the rotation axis of the goniometer, perpendicular to the e-beam. First a hole was drilled (arrow 1, Figure 4b) using a point focused probe
for testing purpose. A cut with a line focused probe at the very end of the tip yielded an apparent \(\sim 3\) nm radius tip (arrow 2, Figure 4b), however in this viewing direction only. The tip was then rotated by 90° for further cuttings from both sides (Figure 4c) to achieve a final radius of the tip in all viewing direction of \(\sim 3\) nm. The mounting of the tip on a tomography holder for the 3-D nanofabrication allows the combination with tomographic reconstruction via a second tilt series.

In some cases, during the hole drilling on nickel nanotips, protrusions were observed, confined one-sided to the e-beam exit point only. To decide between the two possibilities mentioned in the literature, (i) materials redeposition versus (ii) carbon contamination, a test tip was perforated by several point focused e-beams (Yellow circles, Figure 5a). After rotation by 45° the protrusions at the exits of the holes (Arrow, Figure 5b) became visible and accessible for EELS. They were confirmed as pure Ni, via Ni M-edge (and L-edge, not shown) and the absence of any C-K-edge (Figure 5c).

3. Conclusions

2-D and 3-D e-beam nanofabrication were successfully tested and found to be very promising techniques to realize the 1-20 nm structures that are not easily attainable by other fabrication methods known so far. Both type of guns have been proven to provide advantages, the FEGTEM by producing finer beams of different beam shapes and the LaB\(_6\) guns with higher total beam current.

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