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STENCIL MASK METHODOLOGY FOR THE PARALLELIZED PRODUCTION OF MICROSCALE MECHANICAL TEST SAMPLES (PREPRINT)

Paul A. Shade and Michael D. Uchic
Metals Branch
Structural Materials Division

Sang-Lan Kim and Robert Wheeler
UES, Inc.

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14. ABSTRACT
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Stencil Mask Methodology for the Parallelized Production of Microscale Mechanical Test Samples

Paul A. Shade[1], Sang-Lan Kim[2], Robert Wheeler[2], Michael D. Uchic[1]
[1]Air Force Research Laboratory, Materials & Manufacturing Directorate, AFRL/RXLM, Wright-Patterson AFB, OH 45433
[2]UES, Inc., 4401 Dayton-Xenia Rd., Dayton, OH 45432

ABSTRACT

A new methodology to parallelize the production of micromechanical test samples from bulk materials is reported. This methodology has been developed to produce samples with typical gage dimensions on the order of 20 to 200 μm, and also to reduce the unit-cost-per-sample compared to conventional focused ion beam (FIB) fabrication methods. The fabrication technique uses standard microelectronic process methods such as photolithography and deep-reactive ion etching (DRIE) to create high aspect ratio patterned templates—stencil masks—from a silicon wafer. In the present work, the stencil mask pattern is a linear row of tensile samples, where one grip of each sample is integrally attached to the bulk substrate. Once fabricated, the stencil mask is placed on top of a pre-thinned substrate, and the pattern and substrate are co-sputtered using a broad ion beam milling system, which ultimately results in the transfer of the mask pattern into the substrate. The methodology is demonstrated using a Si stencil mask and a polycrystalline Ni foil to manufacture an array of metallic micro-tension samples.

I. INTRODUCTION

Over the past decade, there has been considerable progress in the development of new mechanical testing methods to characterize the properties of materials at the micro- and nano-scale.[1-4] One common application of these new testing methodologies is the measurement of mechanical properties of structures that are physically small in scale, such as the strength of nanowhiskers[5] and MEMS devices.[6] Another common application is the use of small sample testing to gain insight into plastic deformation processes through systematic alteration of the sample dimensions in order to help isolate selected aspects of material behavior. Examples of these studies include the exploration of size-scale strengthening effects,[7-9] the quantitative measurement and analysis of dislocation avalanches (i.e., strain bursts),[10] and the measurement of local property variations in engineering alloys.[11]

For the latter application, focused ion beam (FIB) milling has been widely used to fabricate small samples from bulk crystals for micro-compression,[2] micro-tension,[12] and micro-bending studies.[13] This process can be applied to a wide range of inorganic materials and is relatively low damage, where the damage layer associated with 30 kV Ga^{+} ions typically extends

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from 20-50 nm in depth.[14] Importantly, ion beam milling methods enable the extraction of microsamples from modern engineering materials in bulk form that possess complex microstructures (e.g. polycrystalline alloys that are composed of multiple phases), whereas it is difficult or impossible to use microelectronic processing methods to directly synthesize microsamples with the same chemistry and distribution of microstructural features. However, commercial liquid-metal ion source FIB microscopes are optimized for milling of features with micron and sub-micron dimensions. Typical FIB sputtering rates (for 30 kV Ga^+ ions) range from 0.1 to 2 μm^3 nA^{-1} s^{-1}, and the maximum beam current for most FIB microscopes is approximately 50 nA. Thus, this fabrication route is not conducive to fabricating statistical quantities of samples when the sample dimensions range from 20 to 200 μm in scale, and as a result, this sample size range for mechanical property measurements has been relatively unexplored. Note that this size range can be currently accessed with other microfabrication methods such as micro-EDM machining[2] or ultrafast laser machining, but these processes typically have a much wider zone of affected or altered material at the sample surface compared with ion milling. The damage layer from these coarser-scale machining methods can usually be removed via subsequent FIB milling [2], resulting in a two-step sample preparation method that requires significantly less fabrication time compared to using only FIB milling.

The present study describes a new sample preparation methodology, using ion beam sputtering for material removal, which provides a parallelized production process to enable statistical measurements of microscale mechanical properties. The methodology is inspired by an ion sputtering process developed by Hauffe and co-workers in the mid-1990's, termed slope-cutting,[15] which uses a physical mask with a well-defined edge, such as a knife-edge blade used for machine tooling, and a broad ion beam source to prepare a cross-section surface. The physical mask protects one portion of the sample while the edge defines the location of a cross-section surface that is created when the remainder of the sample is eroded by the ion beam. Slope-cutting maintains some of the advantages of FIB cross-section preparation, such as the ability to cleanly section multiphase materials while retaining features such as porosity, and the ability to apply the method to a wide variety of materials. Yet the dramatic increase in total ion current from 50 nA to hundreds of μA or higher (albeit focused over a much larger area) results in a much shorter time to sputter mesoscopic volumes of material. In addition, the equipment used to perform a slope cutting experiment is simpler and usually much less expensive than a FIB microscope.

Instead of using a simple rectilinear mask as in slope cutting, the methodology described herein uses microelectronics processing techniques to create physical masks with complex two-dimensional shapes, i.e., stencil masks. In the next section we briefly describe the experimental methodology, and in the application section we present a demonstration of the method to produce a micro-tensile sample array in a polycrystalline Ni foil.

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II. STENCIL MASK METHODOLOGY FOR PRODUCING MICROMECHANICAL TEST SAMPLES

The stencil mask methodology consists of three primary tasks. The first task is to create a freestanding stencil mask, which defines the pattern that is to be transferred to the substrate. The second step is to ensure that the substrate is in a suitable form for creating a three-dimensional test sample via ion sputtering, where the region of interest is a thinned section with uniform thickness on the order of 20 to 100 μm. The final step is to use the stencil mask in conjunction with broad ion beam milling to transfer the pattern into the substrate. Next, we describe each of these steps in some detail.

One of the critical stencil mask characteristics for the present application is the creation of high aspect ratio structures, where the thickness of the mask is significantly greater than the dimensions of key micron-scale lateral features within the pattern and also thicker than the substrate itself, in order to allow for complete sputtering through the substrate prior to erosion of the mask pattern. For the present study, we have selected a relatively inexpensive fabrication route which uses standard microelectronic processing techniques to create mask structures from single crystal Si wafers. We have developed various stencil mask designs using computer-aided design (CAD) software (SolidWorks DWGEditor) to create an open-format CAD file (DXF format) that contains the layout of the mask structures. For this study, 200 stencil masks were fabricated from a single 100 mm diameter Si wafer, where each mask is approximately 5 x 10 mm in size and contains between 10 to 20 micromechanical test structures on one edge of the mask. The mask design was then patterned in Cr via laser-based lithography onto a 125 x 125 x 2.3 mm soda lime glass substrate to create a photomask (Photo Sciences, Inc). A 7-to-8 μm thick layer of positive photoresist was deposited onto a 200 μm thick, 100 mm diameter Si wafer, exposed to ultraviolet light through the photomask and then developed, to create the original design specified in the CAD file in the form of a protective resist on the silicon wafer. The unprotected areas of the wafer were then etched through the full wafer thickness via deep reactive ion etching (DRIE) using the Bosch process, which consists of alternating cycles of plasma etching of the substrate and deposition of an etch-resistive polymer which acts to minimize lateral etching, thus allowing for the creation of high aspect ratio structures with nearly vertical sidewalls.[16] The photoresist deposition and DRIE procedures were conducted by C. Ellis of Auburn University.

The second step of the stencil mask methodology is to ensure that the substrate material is in the form of a thin sheet. For this particular study, we have obtained commercially-available metallic foils in order to minimize the experimental effort of producing substrates with the desired starting product form. Mechanical polishing methods can be used to carefully thin many inorganic materials to this size range, although in our experience there are some practical difficulties in successfully polishing foils when the foil thickness is reduced to less than 100 μm (e.g. sample handling and ensuring polishing uniformity). A potential alternate method for preparing tens-of-microns thick lamellae from bulk samples with areal

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dimensions greater than 1 mm^2 is the use of slope cutting, although we have not experimentally demonstrated this processing route at the present time.

Once the stencil mask and the substrate are prepared, the mask is clamped to the top of the substrate and the structure is placed in a vacuum chamber that contains a broad ion beam source. For the experimental data shown in the following section, a commercially-produced broad ion beam milling system, a Gatan Precision Etching and Coating System (PECS), is used to sputter the mask and substrate using relatively low energy (1-10 kV) Ar^+ ions with beam currents that range from 100 to 500 μA and an approximate full width half maximum spot size of 5 mm. An important consideration in the design and use of the stencil mask is the relative sputter erosion rate of the mask compared to the substrate materials. There are tabulated data of sputtering yield of elements for selected ion species and energies,[14] which can be useful in estimating the relative thickness of the mask required for a particular substrate material and substrate thickness. The mask does not erode in a uniform fashion, but rather develops facets in the top surface during milling, as sputtering yields are dependent on the surface inclination relative to the path of the accelerated ions.[14] The development of a faceted surface at the top of the mask results in faster erosion of the mask around its outer edge, and therefore the thickness of the mask and substrate must be optimized to ensure that the mask profile at the mask-substrate interface remains unchanged when the ion milling process is complete.

III. APPLICATION

In the following section, we demonstrate the application of the stencil mask methodology to create an array of micro-tensile samples in a polycrystalline metal. The substrate material used in this study is a 99.0 % purity annealed Ni foil which was purchased commercially from Goodfellow. The foil was obtained in both 25 and 50 μm thicknesses and has an approximate average grain size of 10 μm.

An example of the Si stencil mask pattern employed for these experiments is shown in Fig. 1. One aspect in the design of the stencil masks that was essential in obtaining high aspect-ratio masks with nearly vertical sidewalls was to include a channel that defined the outer edge of the tensile sample pattern in the mask. The final mask design employed a fixed channel width of 50 μm for the 200 μm thick wafer, which is denoted by the gray arrow in Fig. 1B. An initial design of the stencil mask pattern did not include this feature, and instead contained a relatively large open region that surrounded the tensile sample area and allowed for lateral etching during DRIE processing. This initial geometry resulted in an unacceptable pronounced vertical taper of the tensile mask pattern, where the tensile structures at the top of the mask were substantially narrower compared to the bottom of the mask, as shown in Fig. 1E. Note that the outer border of the channel, also labeled in Fig. 1B, was mechanically removed prior to stencil mask milling (Figs. 1C, 1D).

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Prior to placing a sample in the PECS, the mask and substrate were clamped together using a Pelco SEM Pin Mount (32 mm dia.). The pin mount has two Cu clips as shown in Fig. 2A, which are used to clamp the mask and substrate in a fixed position throughout the milling process. We found, however, that the pin stub could not be removed from the PECS without the mask moving relative to the substrate, and thus had to rely on an internal viewing port on the PECS to monitor milling progress. Importantly, the use of clamps eliminated the need to use glues or adhesives to fix the position of the stencil mask relative to the substrate. In our experience, many glues or adhesives were unsuitable for this application without the use of active substrate cooling, as these substances produced copious amounts of redeposited material due to thermal heating of the stencil mask and substrate during ion milling. Note that the pin mount was modified by drilling an approximately 5 mm diameter thru-hole in the stub, so that the region of interest for both the mask and foil were positioned over this thru hole. This hole substantially reduced the amount of redeposited material from the aluminum holder. The amount of redeposited material was further minimized by suspending an aperture with a rectangular thru-hole above the SEM pin mount (Fig. 2B), which reduced the amount of ion beam exposure to the mask and substrate in regions away from the tensile sample pattern.

Images from various stages of the fabrication process are shown in Fig. 3. Fig. 3A shows an SEM image of a stencil mask suspended over a Ni foil substrate prior to milling. Fig. 3B is an SEM image of tensile samples fabricated from a 25 μm thick Ni foil substrate after broad beam milling at an accelerating voltage of 6 kV for 36 hours and subsequently removing the stencil mask. One can clearly see the transfer of the stencil mask pattern into the substrate. One of these tensile samples was examined in cross-section using a FIB microscope, as can be seen in Fig. 3C. The corners at the top surface of the samples are somewhat rounded, and there is a small amount of taper to the sample sidewalls, which is typically less than 5 degrees. There is a relatively small amount of redeposited material on the sample surface. Note that the milling progress was carefully monitored through an in-situ viewport so that the process could be stopped as soon the substrate was completely milled through, thus minimizing redeposition and further rounding of the substrate surface.

Lastly, we have heuristically identified some selected issues with the stencil mask milling process, which are as follows. One is the minimization of redeposition onto the sample-of-interest, either from the stencil mask, the apparatus to hold the mask-substrate assembly together, or from the use of low melting temperature materials such as glues or adhesives. When the process conditions are not optimized, a significant amount of material can be redeposited onto the sample surface, as can be seen in Fig. 3D, which shows a cross-section cut from an early stencil mask experiment. Another issue is the taper in the geometry of the sample. This could be corrected by biased tilting of the sample during the milling process to compensate for the non-orthogonal geometry that typically arises, however, that may impose more rounding of the corners at the top surface. A FIB-based cleanup routine may be required

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to ensure a perfect final geometry. A final issue is the increase in substrate temperature due to the broad beam milling process. Figure 4 shows the results of ex-situ thermocouple measurements of the substrate temperature upon removal from the milling system, which provides a lower bound for the temperature rise under different operating conditions. These temperatures may be significant for certain materials, and thus the use of an in-situ cooling stage throughout the broad beam milling process may be required.

IV. SUMMARY

This study presents a new pathway for the parallel production of micromechanical test samples or other 2½-D structures from a wide range of materials using broad ion beam milling and protective stencil masks made from microelectronic processing methods. Compared with conventional FIB-based fabrication, the methodology provides a fast and relatively low cost processing route to manufacture an array of test structures with dimensions that range from 20 to 200 μm in scale. The methodology has been successfully demonstrated using stencil masks made from Si wafers, and pattern transfer to a Ni foil was demonstrated using a commercial broad ion beam milling system.

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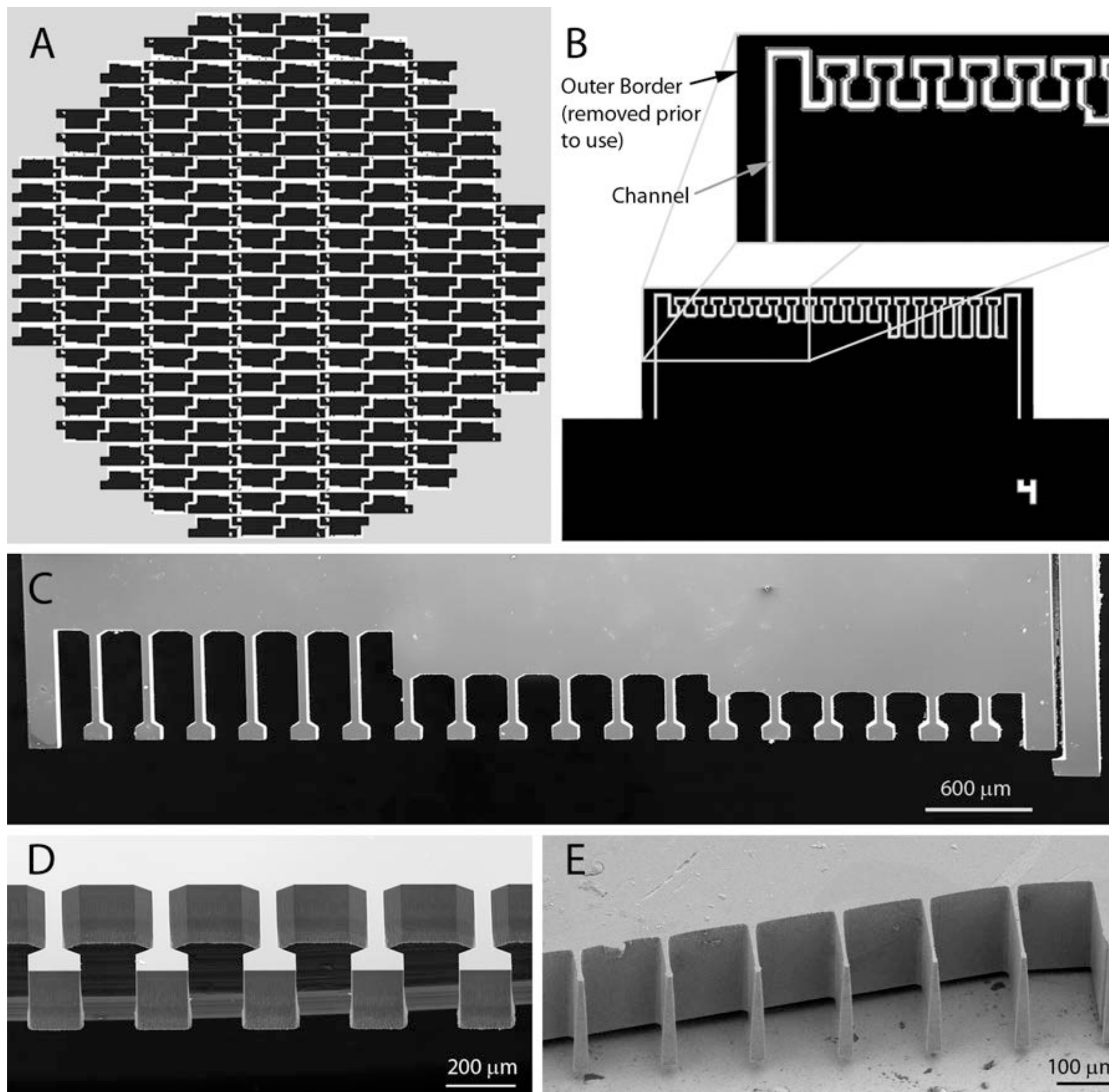

Figure 1. (A) CAD layout of 200 stencil masks on a 100 mm diameter wafer. (B) Schematic of a single stencil mask, with a pattern consisting of a linear row of 18 tensile samples with three different gage lengths. The inset shows a close up view of the tensile pattern layout, where the 50 μm wide channel (denoted by a gray arrow) provides nearly vertical sidewalls from DRIE processing. (C) SEM image of the stencil mask shown in (B), with the outer border removed from the mask. (D) SEM image taken at a stage tilt of 52 degrees, highlighting the nearly vertical sidewalls of the stencil mask. (E) SEM image of a stencil mask where the layout design did not include a channel, which shows a more pronounced through-thickness taper from DRIE processing.

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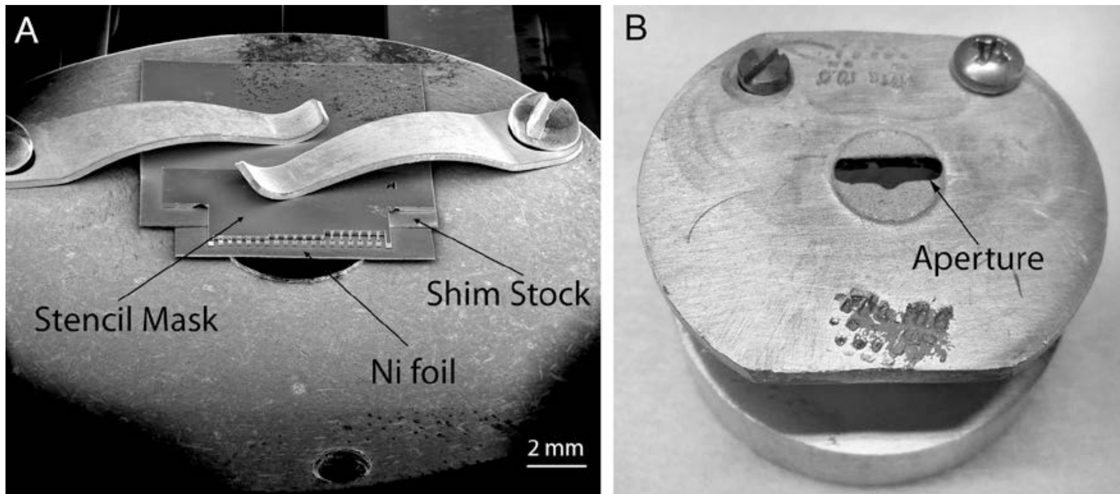

Figure 2. (A) SEM image of the mask and substrate on a Pelco SEM pin mount, showing the positioning of the region of interest over the 5 mm thru hole (image taken at a stage tilt of 52 degrees). (B) Optical image of a 2 mm x 5 mm rectangular aperture positioned above the mask and substrate shown in (A).

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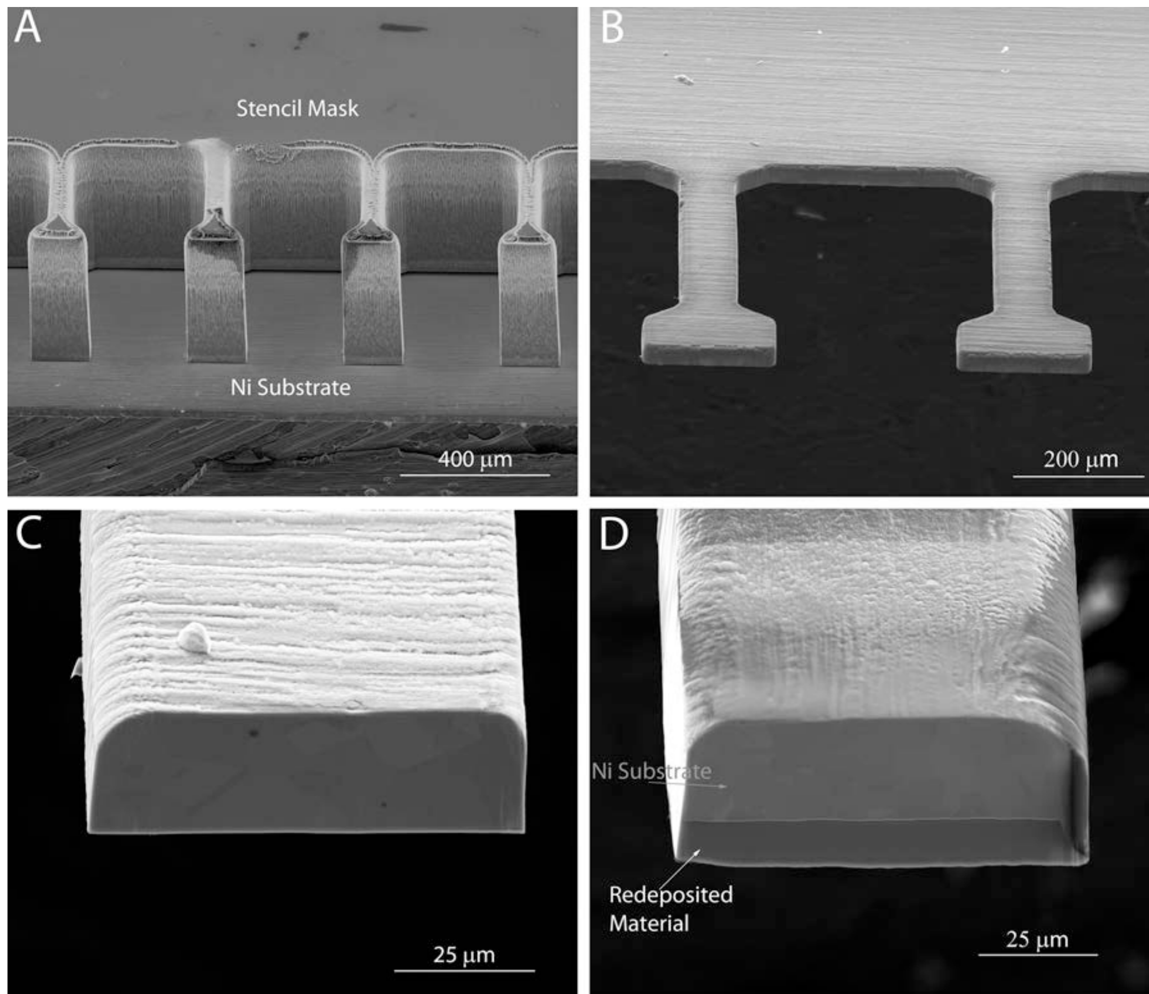

Figure 3. (A) SEM image showing a stencil mask suspended over a 25 μm thick Ni foil substrate prior to broad beam milling. (B) SEM image of tensile samples fabricated from a 25 μm thick Ni foil substrate after broad beam milling with a suspended stencil mask as in (A) at an accelerating voltage of 6 kV for 36 hours and subsequently removing the stencil mask. (C) SEM image of a cross-section cut from one of the tensile samples in (B), which shows there is relatively little redeposited material on the surface. This image also demonstrates rounding at the top surface and a slight taper of the sample sidewalls. (D) SEM image of a cross-section cut from a set of tensile samples fabricated under non-optimized operating conditions, which shows a significant amount of redeposited material on the surface in contrast to (C).

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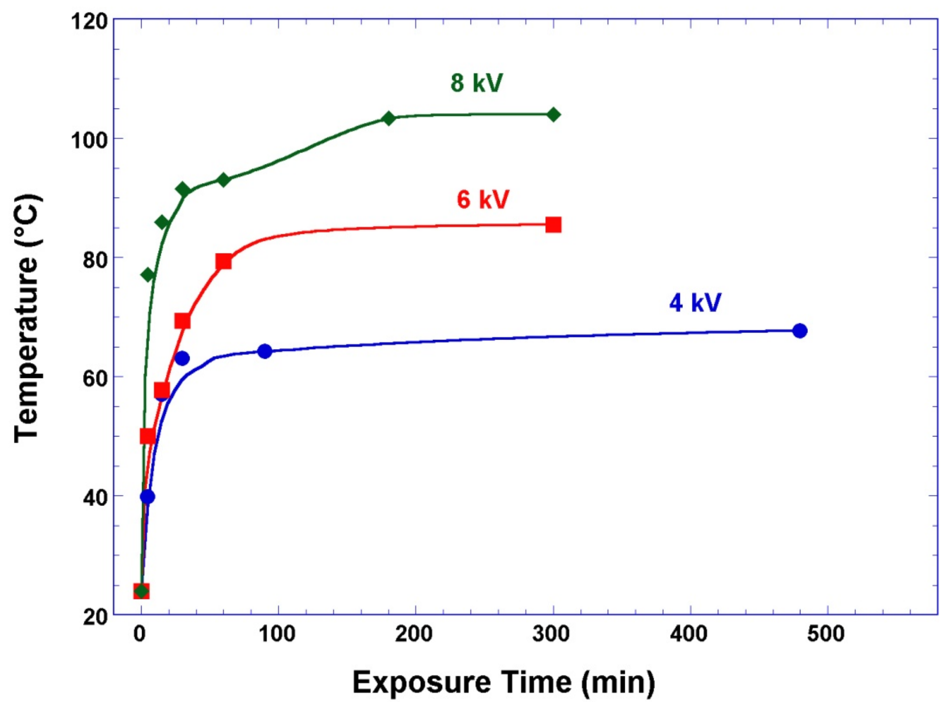

Figure 4. Plot of the change in specimen temperature for a Ni foil clamped to the Pelco sample stage as a function of both milling voltage and total exposure time for the Gatan PECS. Temperature was measured ex-situ, after removing the sample holder from the vacuum chamber of the ion milling system, using a Type K thermocouple that was placed in direct contact with the sample.

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