Silica Glass Micropillar Fabrication

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Affiliation: Materials Science and Engineering Department, Cornell University Primary Source of Research Funding: Corning Inc. Contact: spb14@cornell.edu, zwr6@cornell.edu Primary CNF Tools Used: AJA sputter deposition, Autostep i-line stepper, PT770 etcher, Oxford 100 etcher, Oxford 81 etcher

Abstract:

A method for the fabrication of a large number of silica glass micropillars has been developed. This method writes a pattern onto photoresist, which is used as a mask to etch a sputtered chromium film. The residual chromium is then used as a mask for a deep silica glass etch, resulting in the formation of pillars of various heights (5 to $12 \mu m$). An initial proof-of-concept fabrication lead to pillars that had unsatisfactory surface roughness, but nonetheless lead to very interesting compression results viewed with *in situ* SEM. Improvements to the fabrication method are underway and will lead to smoother sidewalls and taller pillars.

Summary of Research:

Although silicate glasses are brittle materials, in small volumes they can undergo extensive plastic deformation. The character of this deformation controls the induced stress and strain state of the contact, and ultimately dictates the fracture initiation and propagation of the bulk material. By far the most common experimental technique for the study of plasticity in ceramics is indentation, however interpretation of plasticity from indentation tests is obscured by the complex, mechanism dependent stress and strain state underneath the indentation. Micropillar compression tests are much less commonplace, but have the tremendous advantage of a well-defined stress and strain state [1], as well as the ability to observe and quantify the plastic deformation *in situ* through SEM.

The most common technique for micropillar fabrication is annular milling using a focused ion beam. While this technique is wide spread, it is extremely lowthroughput, is technically challenging in electrically insulating materials, and has been shown to induce structural damage in the pillars, potentially skewing any obtained mechanical data. In this project, a methodology for the simultaneous etching-based fabrication of a large number of silica glass micropillars (> 1 million) for compression testing is developed.

This method starts by using the AJA sputter deposition tool to apply a thick chromium film onto a standard 100 mm silica glass wafer. This process required significant optimization as significant stresses can develop within chromium depending on sputtering conditions and care has to be taken to avoid delamination of the film. Once the chromium has been sputtered, anti-reflective coating and thick (~ 3 μ m) i-line photoresist is spun on top of the chromium. The photoresist is then exposed on the AS200 stepper tool in order to pattern arrays of circles of unexposed photoresist with various diameter. The exposed photoresist is then developed, leaving unexposed cylinders of photoresist behind on the chromium film.

After etching away the anti-reflective coating of the Oxford 81 etcher, the photoresist pattern is used as a mask for a chromium etch on the PT770 etcher. The damaged photoresist is stripped in the Oxford 81 etcher, leaving patterned cylinders of chromium on the silica glass wafer. The wafer is then loaded into the Oxford 100 etcher where it undergoes a long fluorine based etch using the remaining chromium as a mask. After the etch is sufficiently deep, the remaining chromium is etched away in the PT770, leaving arrays of silica glass micropillars.

An initial fabrication yielded pillars of satisfactory height, but lacked smooth sidewalls which makes the determination of the stress state during compression more difficult. As shown in Figure 1, significant plastic deformation can be achieved in these *in situ* SEM micropillar compressions. Optimization of the process hopes to address the smoothness of the pillar sidewalls by ensuring more vertical sidewalls of the chromium mask.



Figure 1: (Background) An uncompressed silica glass micropillar from initial fabrication showing high sidewall roughness. (Foreground) A highly compressed silica glass micropillar with little to no fracture present. This level of ductility can be achieved by exposing the pillars to high electron beam fluxes during compression.

References:

 Fei, H., et al. (2012). "Evaluation of Micro-Pillar Compression Tests for Accurate Determination of Elastic-Plastic Constitutive Relations." Journal of Applied Mechanics 79(6): 061011-061011.

