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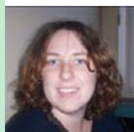
Microscale Bioactive Patterning: A novel surface modification of poly(dimethylsiloxane)



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In the fabrication of microfluidics and biomedical devices, poly(dimethylsiloxane) (PDMS) has become an increasingly popular material as it is transparent, elastic, durable, inert and biocompatible. Its use is, however, limited by the siloxane's low surface energy and extreme hydrophobicity which hinder cell adhesion. Our group has developed a novel surface treatment that increases the hydrophilicity of the polymer's surface in a patterned fashion providing a means to both promote and confine cell adhesion.

A thin layer of aluminum is deposited onto the surface of PDMS, using the Edwards Auto500 sputter deposition system, in the presences of an argon plasma. The aluminum layer can be etched away exposing an oxygen rich layer of PDMS. The modified PDMS layer allows for the growth of fibroblast, epithelial and myoblast cells to confluence due to its hydrophilicity. We can in turn pattern the surface treatment the subsequent cell growth by depositing the metal film through a stencil mask. These patterned substrates permit the study of cell-cell interactions, cell motility and cellular responses to various spatial and geometric perturbations, while the surface modification itself will extend the use of PDMS in microfluidic and biomedical devices.

Further, these patterns were integrated into microfluidic channels formed by soft lithography. SU8 negative photoresist was exposed to UV light through a photomask to create a master. PDMS pre-polymer was then poured over the master and cured, resulting in the formation of channels. Aluminum dots were then aligned manually using an optical microscope.

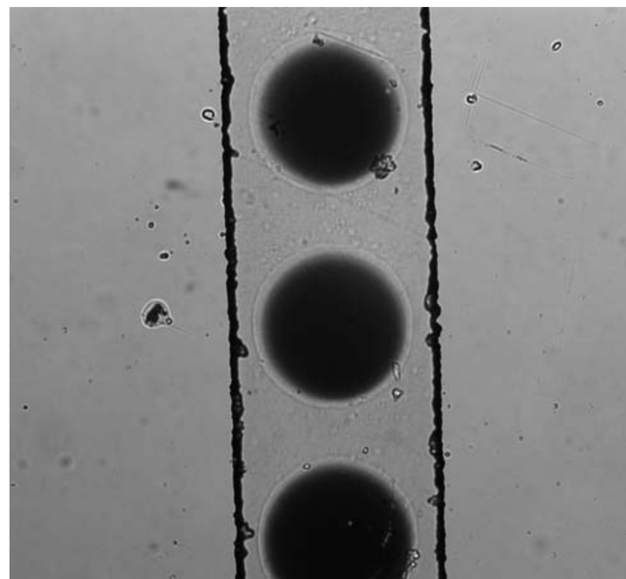

Figure 1: 180 mm Aluminum dots inside of a microfluidic channel.

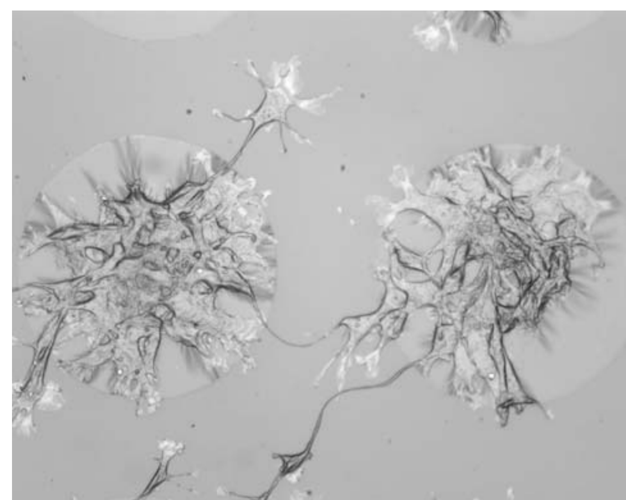

Figure 2: Optical micrograph of patterned C2C12 cells on modified PDMS.

*NanoUser **Jessica McLachlan** is the recipient of the Xerox Canada Award in the Macromolecular Science and Engineering Division at the Canadian Society for Chemistry Conference held this past summer in Winnipeg, Manitoba for her poster entitled "Microscale Bioactive Patterning: A Novel Surface Modification of Poly(dimethylsiloxane)". Congratulations Jessica!*

Characterization of the Plastic Zone Beneath Nano-indentations in Gold

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Introduction:

It is now well established that small volumes of metal require higher stress to plastically deform compared to their bulk counterparts[1-2] however there is still ambiguity regarding the mechanisms responsible for this "size effect" of material flow stress. There are two main schools of thought explaining this phenomenon; namely, the Strain-Gradient Plasticity (SGP) theory and the Dislocation-starvation theory[3-4]. Bhakhri & Klassen[5] and others[6] have studied the kinetics of deformation in the "size effect" range and have concluded that despite the clear size-effect of the material flow stress, the actual plastic deformation process is controlled by the same type of dislocation-dislocation interactions irrespective of the volume of the material being deformed.

Investigations of the size-effect of plastic deformation are, of necessity, performed using the nanoindentation hardness testing technique. Despite this, few studies have been reported of the actual dislocation structure and crystallographic misorientation around, and beneath, nanoindentations[7-8]. In most of the reported works, Electron Back Scattered Diffraction (EBSD) has been used to characterize the misorientations and microstructural evolution in indentation plastic zone. Investigations reporting images of the actual dislocation sub-structures in indentation plastic volume are relatively few[9].

Here, we present the preliminary results from Transmission Electron Microscopy (TEM) of the plastic zone around a nanoindentation in a pure gold specimen. Focused Ion Beam (FIB) micromachining was used to prepare an electron transparent cross-section of the indentation. The FIB technique allowed us to extract TEM specimens directly from the region of the nanoindentation (Figure 1(a)). This would have been impossible using conventional TEM foil preparation methods.

Nanoindentation and TEM Sample Preparation:

A series of Constant-force nanoidentation creep tests were performed at room temperature to on the (001) Au specimen using a nanoindentation tester fabricated by Micro Materials Ltd. (Wrexham, UK). A pyramidal, Berkovich, diamond Indenter was loaded, along [001] surface normal, at a constant loading rate until a pre-specified indentation depth of 400 nm was reached. The indentation load was then held constant for one hour.

The LEO 1540XB FIB/SEM-station in the University of Western Ontario Nanofabrication Lab was used to perform the FIB milling and polishing of the TEM specimens from beneath several of the indentations (Figure 1(a)). The indentation was coated with a platinum layer to protect the surface from ion damage during the FIB milling.

A cross-section through the indentation was prepared by using a FIB milling current of 1 nA at 30keV ion energy (Figure 1(b)). The lamella was then plucked from the substrate using the insitu nanomanipulator. Finally, the sample was thinned and polished with focused ion beam of 50pA current. The detailed specimen fabrication procedure is outlined elsewhere[10].

TEM Results & Discussion:

The TEM investigation of the indentation plastic zone was carried out using at the Brockhouse Institute of Materials Research (McMaster University) using a PHILIPS CM12 electron microscope at 120 KeV. Figure 2(a) shows a low magnification micrograph of the indentation cross-section. Figure 2(b) shows the dislocation structure beneath the tip of the indentation. Dislocation cell formation is clearly evident. The cell walls contain a very high dislocation density, however the dislocation density in the interior of the cells is relatively low. Figure 2(c) shows the lower dislocation

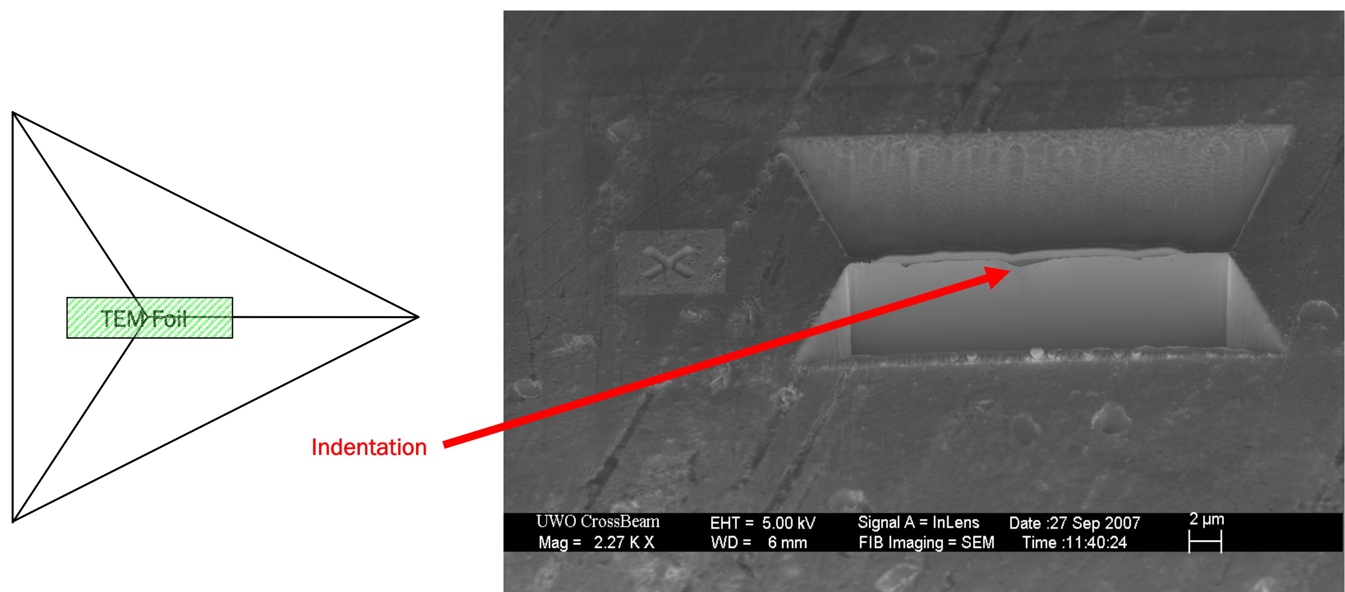

Figure 1: (a) Schematic of TEM foil cross-section through the indentation. (b) SEM micrograph of milled lamella around the indent site.

density in the less highly strained region further from tip of the indentation. Well defined dislocation cells are not evident in this region.

These observations of decreasing dislocation density away from the indentation tip (but still beneath a flank of the indentation) agree with what has been reported concerning the plastic strain around an indentation[11]. The area directly beneath the indenter tip is the most the highly stressed region and the well-developed dislocation cells that we observe in this region indicate that the indented gold is in the advanced (Stage-II/Stage-III) stages of work-hardening.

Currently, TEM analysis of “uncrept” specimens, indented to a depth of 400 nm and then immediately unloaded, is in progress. Results from this sample will be compared to the observations reported here to compare the extent of dislocation recovery that occurs when indented gold is allowed to creep for one-hour at room temperature. Future work will include the TEM analysis and comparison of evolved microstructures from the specimens made at different indentation depths and at elevated temperatures.

Acknowledgement:

The authors wish to acknowledge Dr. Todd W. Simpson of The Nanofabrication Laoboratory at The University of Western Ontario for fabricating the TEM specimen used in this study and Mr. F. Pearson at the Brockhouse Institute for assisting with the TEM.

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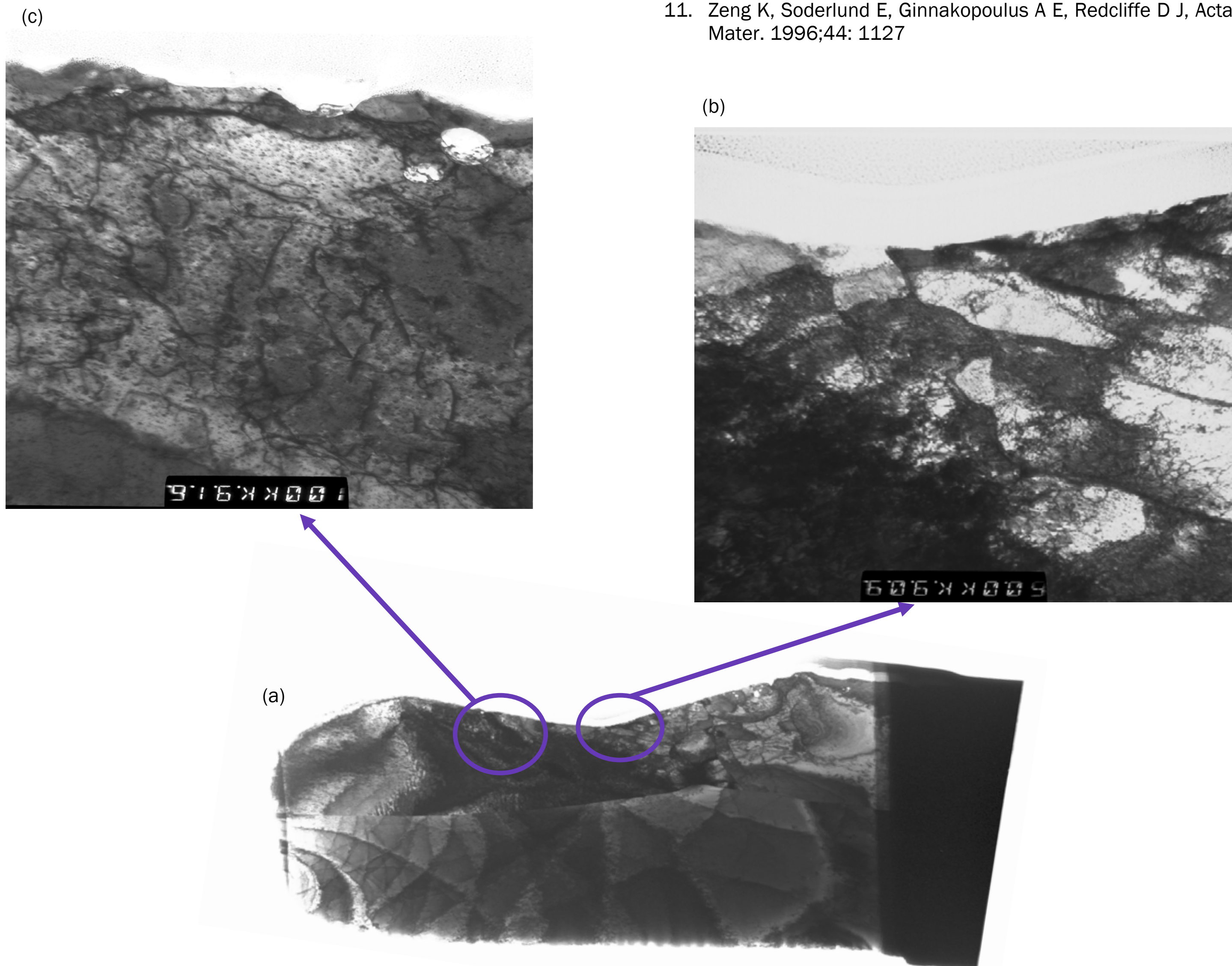

Figure 2: TEM micrographs of the indented Au specimen; (a) Low-magnification specimen profile, (b) Heavily dislocated microstructure beneath the indenter tip (c) Less heavily deformed microstructure in the region away from the indentation tip.

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Anisotropic Etching of Silicon by Potassium Hydroxide Wet Etch

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The most common method of wet etching silicon utilizes a hot potassium hydroxide solution. It is an inexpensive process and is used in preference to a dry etch method when deep or lengthy through wafer etching is required. The process is used in the manufacture of items such as pressure sensors, jet printer heads, microfluidic devices and TEM grids.

The etching is highly anisotropic; the <100> and <110> crystalline planes are etched much faster than the <111> plane. The resultant etched recess has a flat bottomed pyramidal shape with <111> sidewalls (54.7 degrees from the <100> plane). The etch rate is dependant on the KOH concentration and the etchant temperature. There are several websites where one can calculate the etch rates of silicon, silicon nitride and silicon oxide for a given KOH concentration and temperature. The calculations are based on a paper published in 1990 (H. Seidel et al J. Electrochemical Soc. Vol.117, p.3612).

The best masking material is silicon nitride because it has a negligible etch rate in KOH. Silicon dioxide etches slowly in KOH (about 1nm/minute) so it can only be used as a mask for shallow recesses. Photoresist is unsuitable as a mask but is used to pattern the oxide or nitride mask. Silicon nitride deposition is available in the Nanofab on the STS 310 PECVD tool.

The etch equipment is very simple and consists of a temperature controlled bath, a beaker of etchant, a stirrer, a thermometer, a sample holder and a stop watch. We have used 20% KOH solutions to etch silicon at 80°C with a resultant etch rate of approximately 1.2 micron per minute. The process has been used by an industrial customer for processing devices. The equipment will take a four inch silicon wafer; smaller pieces can be mounted on a double side polished nitride coated silicon wafer, which are available in the Nanofab.

Some traditional mounting materials have been found unsuitable for mounting small pieces because they are attacked by KOH allowing the underside of the piece to be etched or released from the mount. We now provide a new mounting material, WaferBond™, which is impervious to KOH. It is easy to use because it comes as a liquid ready to be spun on the nitride coated silicon wafer. After etching, the sample can be removed by heat or by dissolving the WaferBond™ in a proprietary solvent.

Interested?

Contact Rick Glew about wet etching of Silicon.

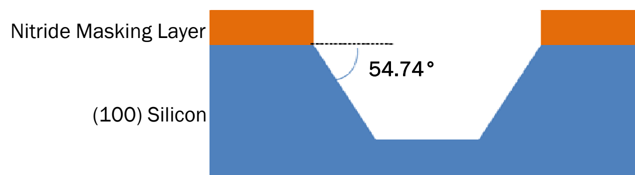

Figure 1: Diagram of etch profile on (100) silicon.

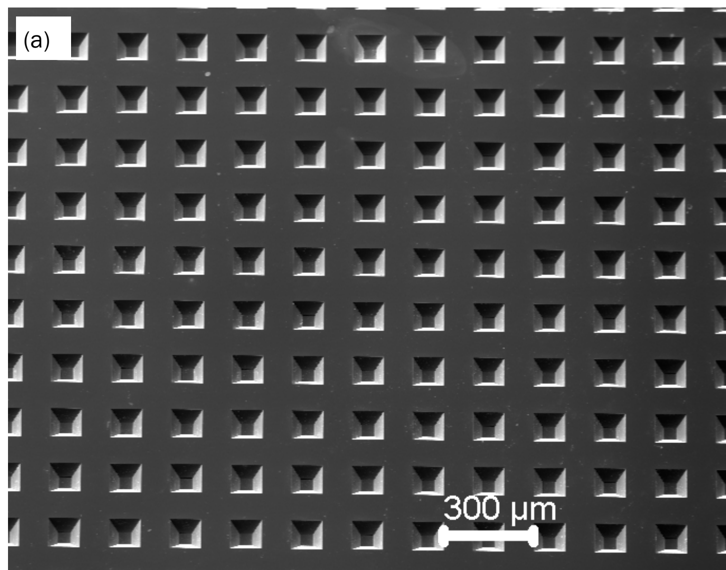

Figure 2a: (left) KOH etched 100 micron square recesses in (100) silicon.

Figure 2b: (below) A magnified 100 micron etched square recess.