

Investigation of Plastic Yielding in Sub-micron Diameter Nickel Pillars



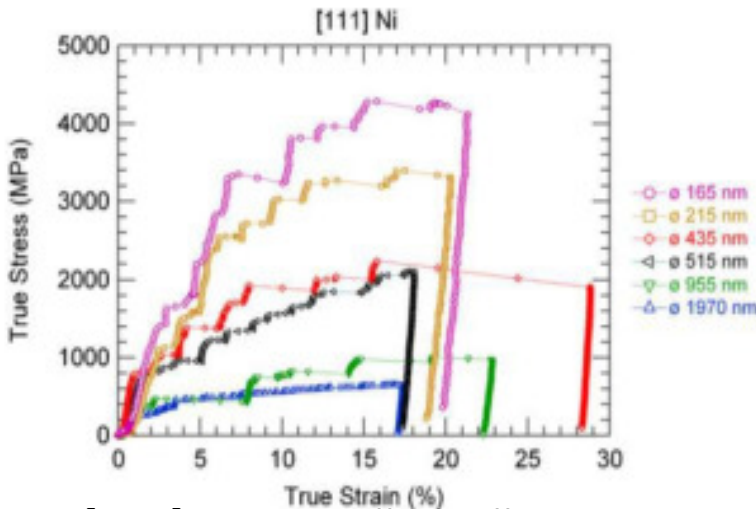
Matthew Sedore

Brad J. Diak

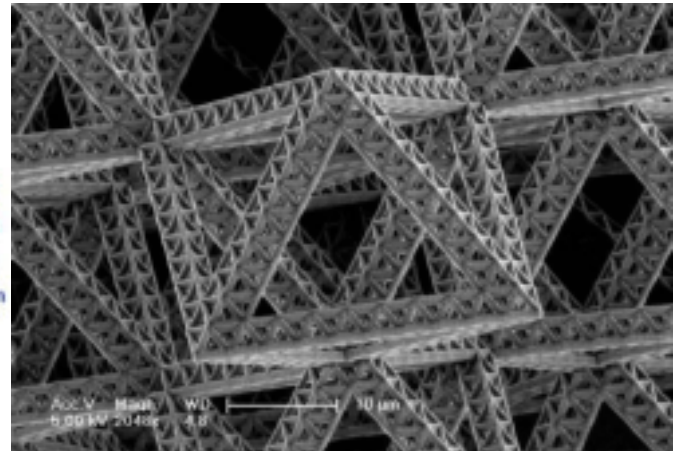
**Dept. Mechanical and Materials Engineering
Queen's University, Kingston, Canada**

Motivation

- Small specimen strength approaching theoretical limits.
- Continuing discussion on whether fabrication method contributes to the strengthening



[111] Ni FIB-milled pillars
[Frick et al, 2008]



Fractal titanium-nitride nanotrus
(Julia Greer, Caltech 2014)

Objectives

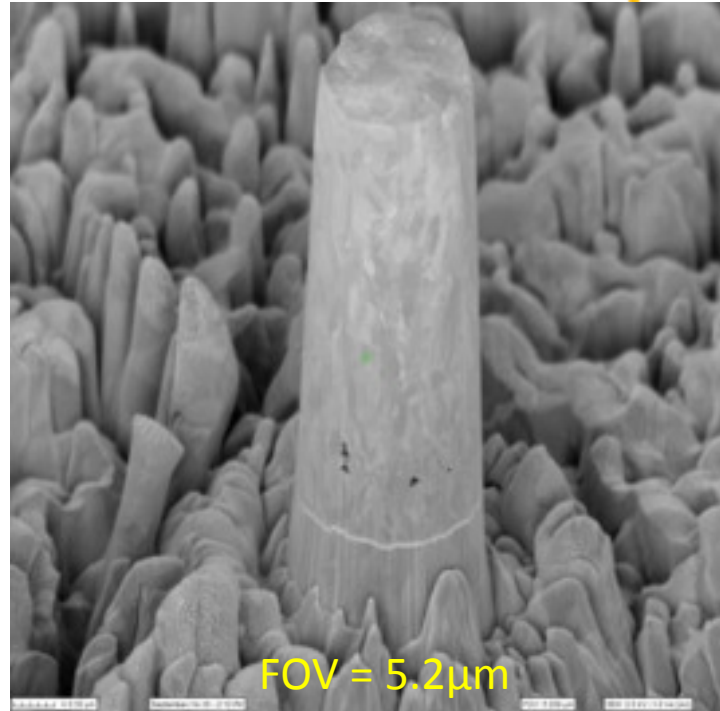


- Fabricate micro/nanopillars using lithography with electroplating as well as FIB milling techniques.
- Mechanically compress pillars to measure deformation behaviour.
- Compare the various methodologies to revise fundamental strengthening theories and determine the validity of current practices in the literature.

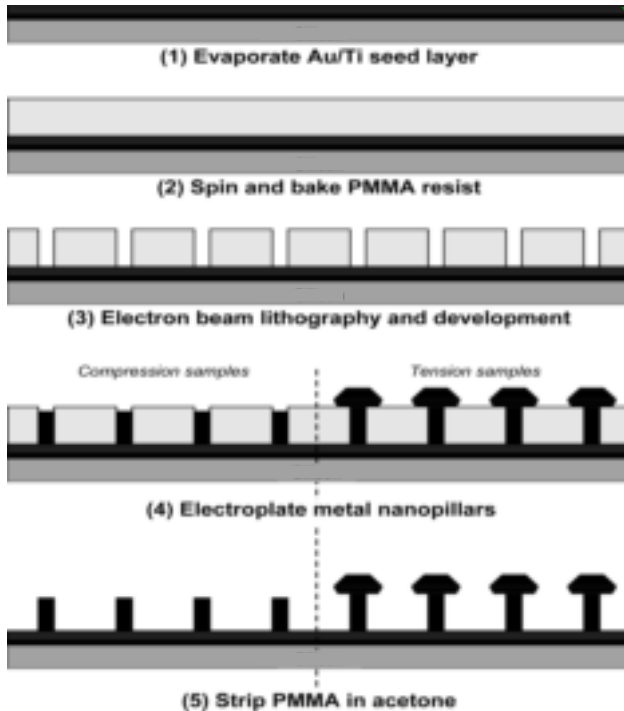
Experimental Procedure: Guidelines for pillars



- Aspect ratios between 1:2 and 1:6 (diameter : height).
- Diameter $< 3\mu\text{m}$
- Electroplating into patterned template using Watts bath for high purity nickel.
- FIB mill pillars from electroplated nickel.
- Anneal to remove ion damage to pillars.



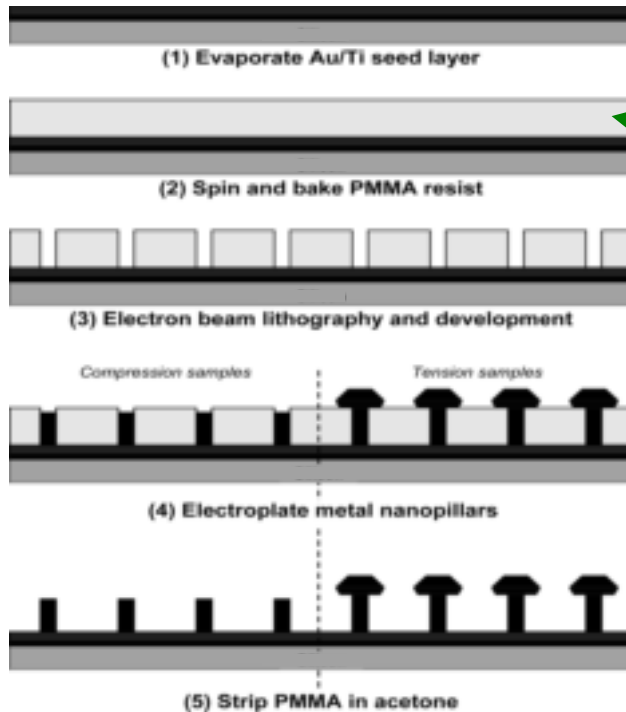
Experimental Procedure: Fabrication Methods



Hummer VI-A gold sputtering device from Anatech LTD

Schematic representation of FIB-less process to make nanopillars (Burek and Greer 2010).

Experimental Procedure: Fabrication Methods

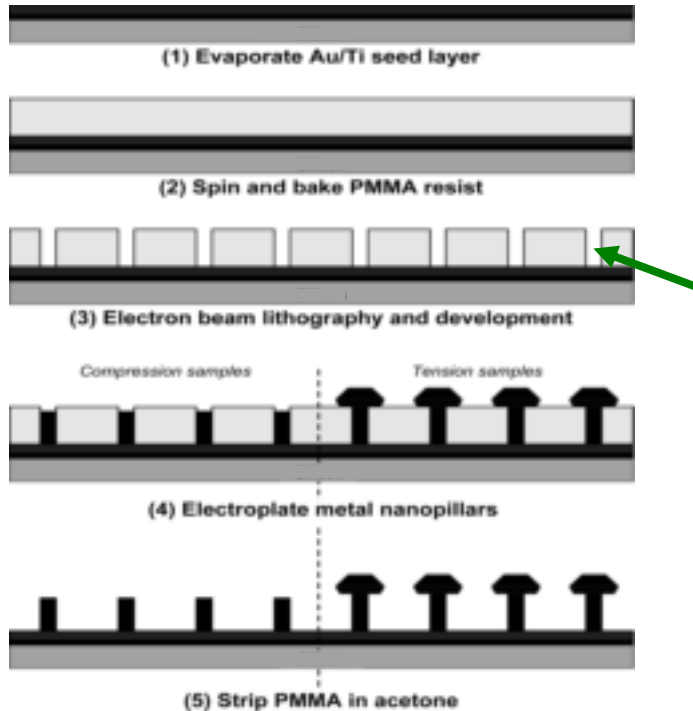


Schematic representation of FIB-less process to make nanopillars (Burek and Greer 2010).



Laurel spin coater at NanoFabrication Kingston (NFK)

Experimental Procedure: Fabrication Methods

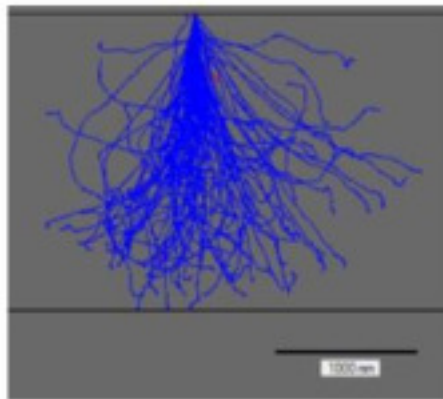


Schematic representation of FIB-less process to make nanopillars (Burek and Greer 2010).

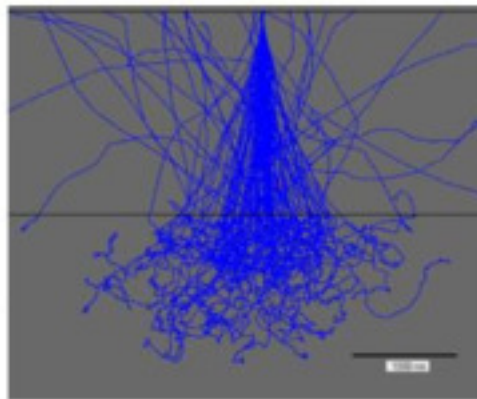


Raith SEM/Lithography at NFK

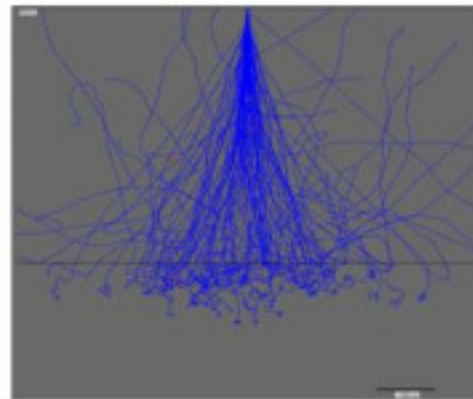
Monte Carlo Simulations (Raith SEM/Lithography)



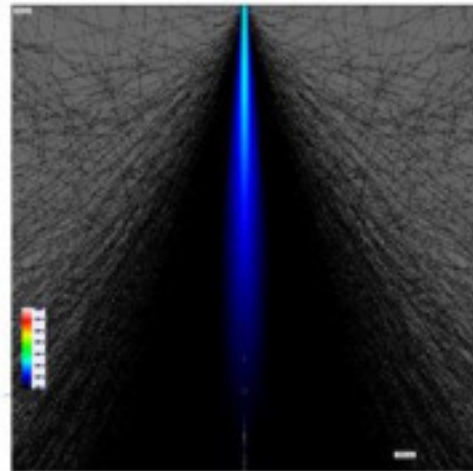
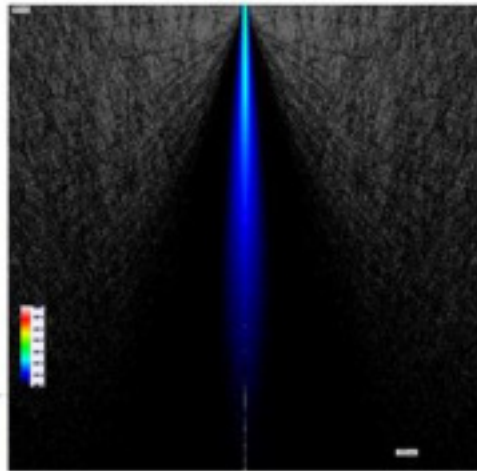
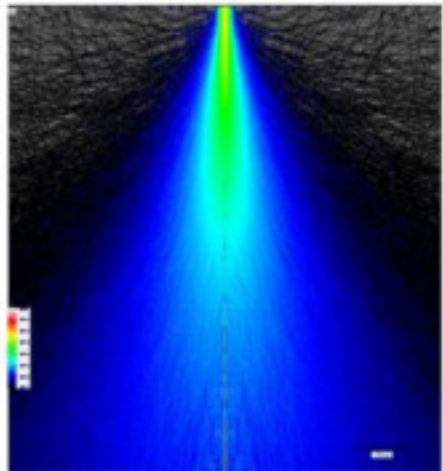
2um PMMA 10keV



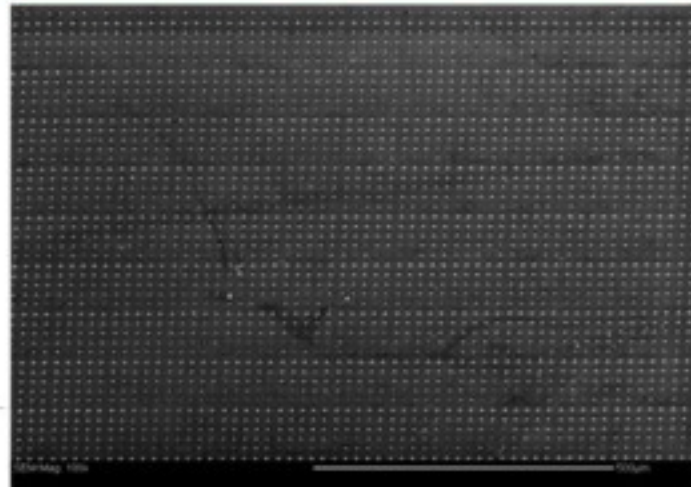
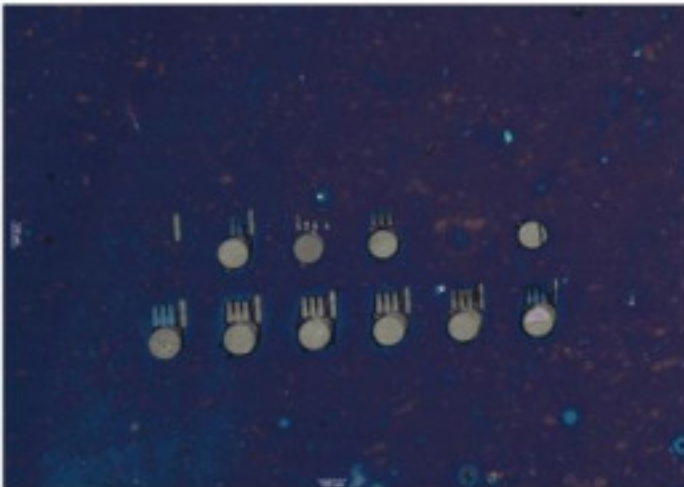
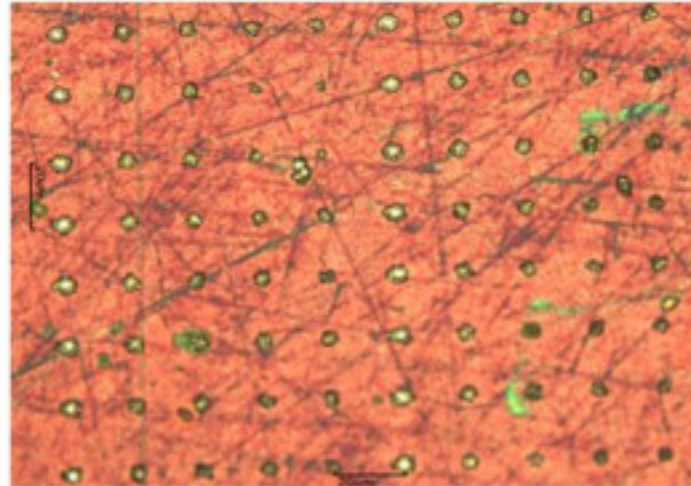
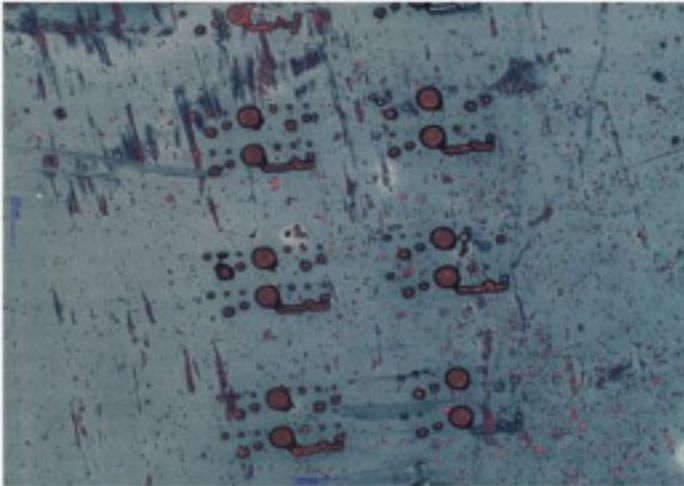
2um PMMA 30keV



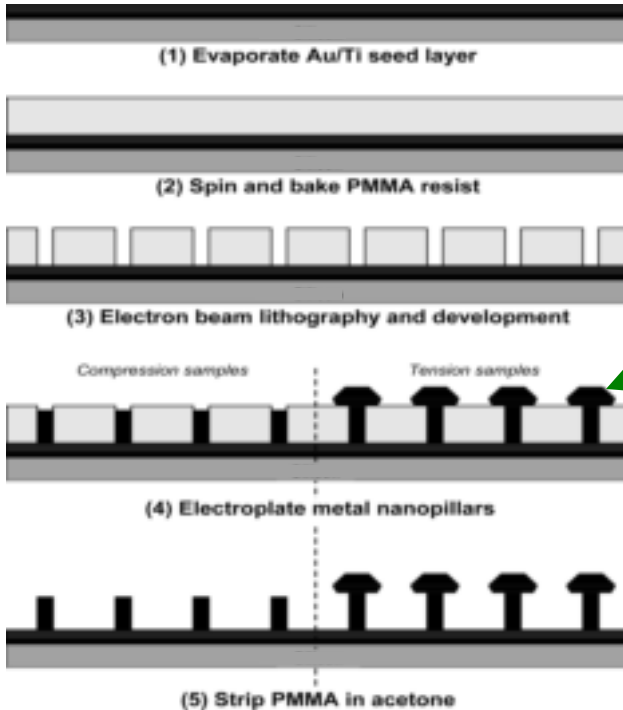
5um PMMA 30keV



Maskless Photolithography and Raith EBL



Experimental Procedure: Fabrication Methods



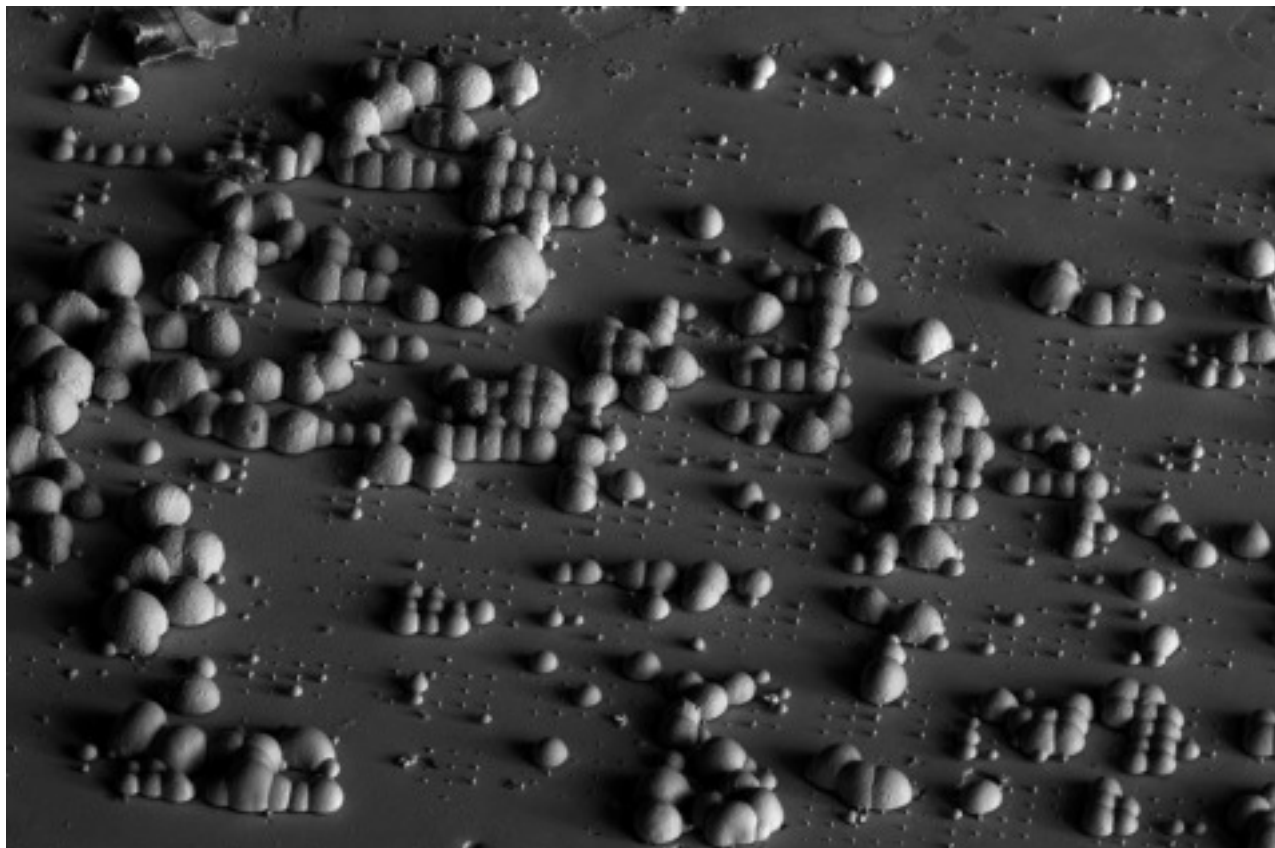
Schematic representation of FIB-less process to make nanopillars (Burek and Greer 2010).



Electroplater from International Micro Industries

Ni Electroplating Conditions	
Current Density	525 mA/cm ²
Flow	1000 rpm
Temperature	55 Celsius
Anode	Platinun-Ti mesh
Cathode	Au on annealed Cu

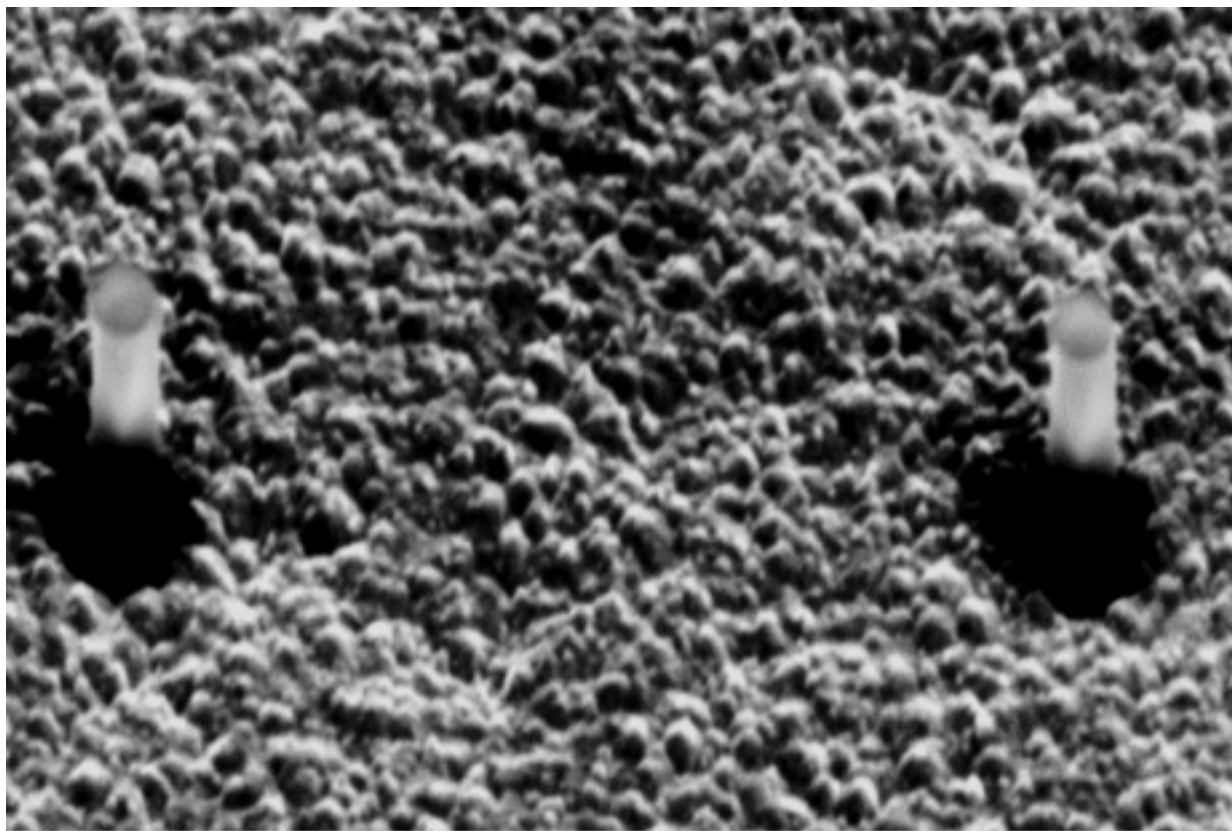
Results: Fabricated Pillars (EBL, Gold, no Plasma)



	7/15/2016	dwell	HV	HPW	pressure	400 μm
	10:43:23 AM	45 μs	18.00 kV	1.04 mm	4.38e-4 Pa	



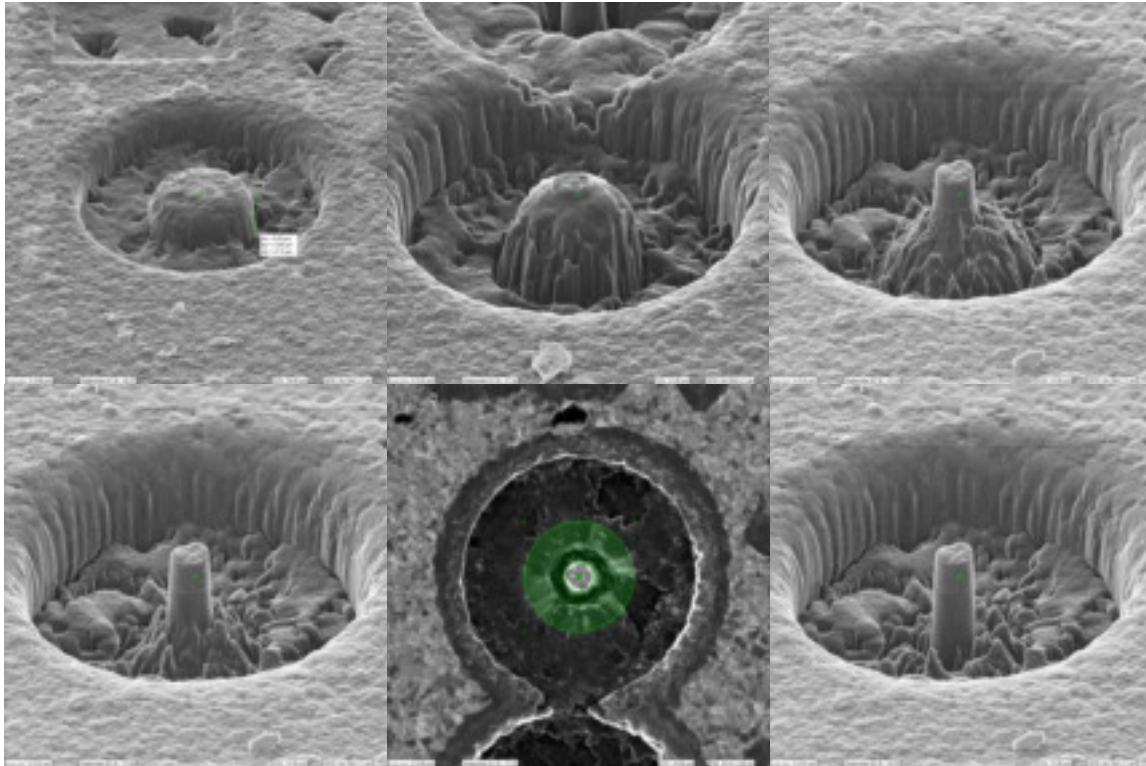
Results: Fabricated Pillars (EBL, no Gold, Plasma)



	10/17/2016 4:53:48 PM	dwell 45 μ s	HV 10.00 kV	HFV 25.9 μ m	mag 8 000 x	WD 10.3 mm	 5 μ m Nova NanoSEM
--	--------------------------	---------------------	----------------	---------------------	----------------	---------------	-------------------------------



Experimental Procedure: Focused Ion Beam

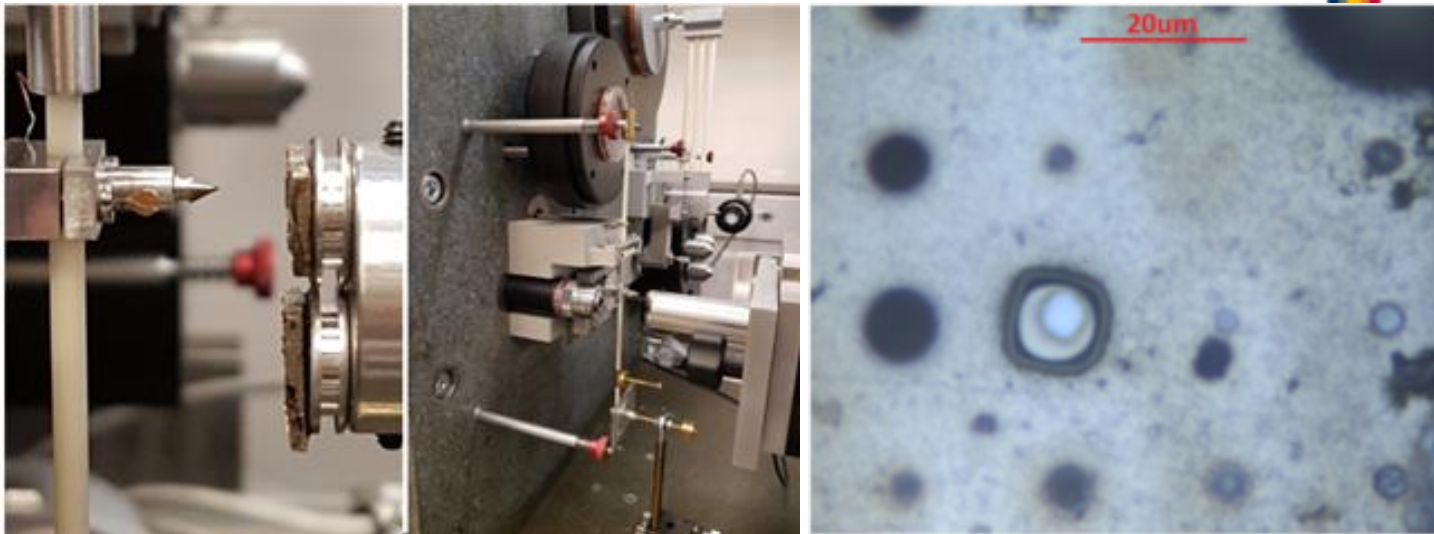


FIB Milling Conditions (FIBICS Inc.)

Milling Step	Rough	Shape	Polish
Voltage	30keV	30keV	30keV
Beam Current	7nA	100pA	100pA



Mechanical Testing



- Nano-test Vantage nano-indenter from MicroMaterials at Reactor Materials Testing Lab (RMTL)
- Slowest loading rate of $1.5 \mu\text{N/s}$ after some trial and error. Loading rates of 1.5 to $30 \mu\text{N/s}$ were used in this work.

Acknowledgements



- Professor Rob Knobel and Graham Gibson (lithography and clean room training)
- Jason Wang and Pooyan Changizian (nano-indentation), Curtis McDonald and Jessica Hiscocks (characterization of pillars)
- Charles Cooney (sample prep and mechanical help)
- Funding courtesy of NSERC Discovery Grant