Metal Etching with the Bosch Process

- Project started in 2011
- Goal was to fabricate photonic crystal (PhC) in bulk Ta
- Thermophotovoltaic applications

V. Stelmakh *et al.*, "Sputtered Tantalum Photonic Crystal Coatings for High-Temperature Energy Conversion Applications," in *IEEE Transactions on Nanotechnology*, vol. 15, no. 2, pp. 303-309 (2016).

Rinnerbauer, V. ; Lausecker, E. ; Schaeffler, F. ; Reininger, P.; Strasser, G. ;Geil, R. D.; Joannopoulos, J. D. ; Soljacic, M; Celanovic, I. "Nanoimprinted superlattice metallic photonic crystal as ultraselective solar absorber", Optica, 2(8) (2015).

Rinnerbauer, V; Lenert, A; Bierman, DM; Yeng, YX; Chan, WR; Geil, R. D.; Senkevich, JJ; Joannopoulos, JD; Wang, EN; Soljacic, M; Celanovic, I. "Metallic Photonic Crystal Absorber-Emitter for Efficient Spectral Control in High-Temperature Solar Thermophotovoltaics", Adv. Energy Mat. 4(12) (2014).

V. Rinnerbauer, S. Ndao, Y. X. Yeng, J. J. Senkevich, K. F. Jensen, J. D. Joannopoulos, M. Soljacic, I. Celanovic, and R. D. Geil, "Large-area fabrication of high aspect ratio tantalum photonic crystals for high-temperature selective emitters" J. Vac. Sci. Technol. B 31, 011802 (2013).

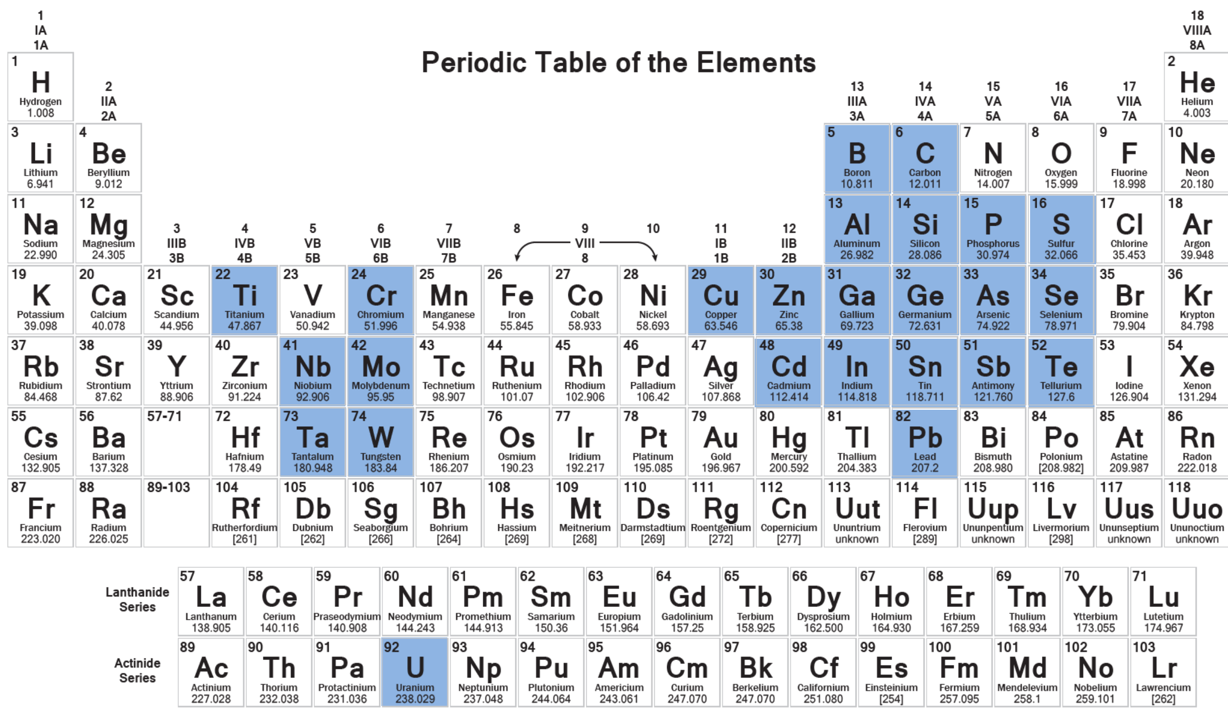
V. Stelmakh, V. Rinnerbauer, R. D. Geil, P.R. Aimone, J. J. Senkevich, J. D. Joannopoulos, M. Soljai, Ivan Celanovic, "High-temperature tantalum tungsten alloy photonic crystals: Stability, optical properties, and fabrication", Appl. Phys, Lett. 103, 123903 (2013).

Alcatel AMS100 DRIE

Specs:

- Standard Bosch process
- Wafer sizes: 4”, 6” and pieces mounted on handle wafer
- Power (Source): 3000 W
- Power (Substrate holder): 300 W RF/500 W LF (50 kHz – 460 kHz)
- Gases: SF_6, C_4F_8, Ar, O_2, CH_4, He
- Temperature ranges: -10 °C to 30 ° C
- Clamping: Mechanical

Project started with a question:
Do you think you can etch Ta in your DRIE?



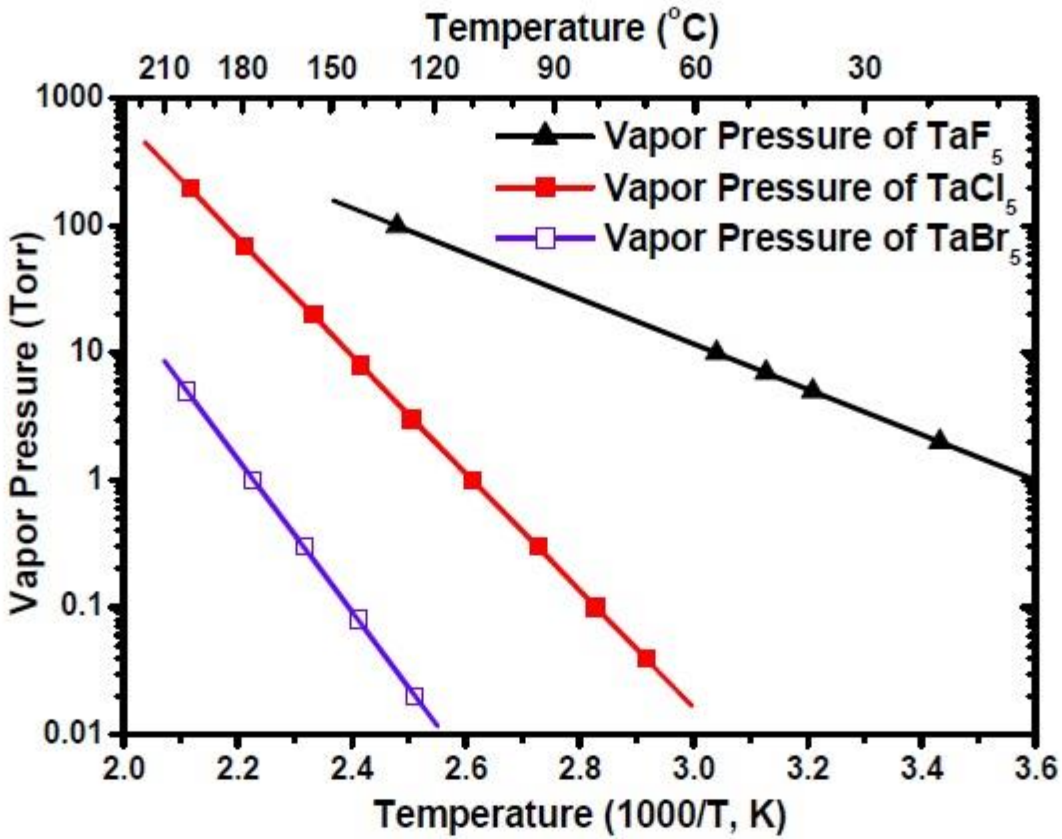
RIE etch workshop with john Coburn

Etchable materials that form volatile reaction products near room temp.

Etchants: CF_4, SF_6, Cl_2, HBr, O_2, H_2, CH_4

Etchable material + Available etchant = Maybe I can etch it

Vapor pressure of Ta halides



TaF_5 significantly more volatile than $TaCl_5$: ~2 Torr at 20 C

T. Kodas and M. Hampden-Smith, The Chemistry of Metal CVD, VCH Publishers Inc., New York, 1994, p. 388.

Notes about the Ta substrates

- 0.75” - 2” diameter Ta pieces lapped and polished to ~ 1nm rms roughness
- Fabrication steps*:
  - Interference lithography
  - Definition of cavity by isotropic plasma ashing
  - Pattern transfer to Cr hard mask (wet and dry etching approaches)
  - Final DRIE of Ta substrate, desired results:
    - Cavity diameter 0.5 – 1.0 um
    - Aspect ratio > 2
- Ta pieces bonded to 6” Al carrier wafer with thermal paste
- Preparing Ta substrates was very time consuming: little room for optimization
- Hard to determine etch rate with out FIB milling
- Improved process through small iterations

* V. Rinnerbauer, S. Ndao, Y. X. Yeng, J. J. Senkevich, K. F. Jensen, J. D. Joannopoulos, M. Soljacic, I. Celanovic, and R. D. Geil, “Large-area fabrication of high aspect ratio tantalum photonic crystals for high-temperature selective emitters” J. Vac. Sci. Technol. B 31, 011802 (2013).

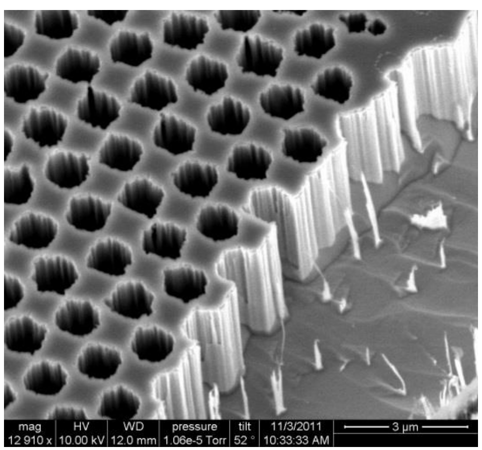
Let’s just see what happens

Bosh etch process based on our “Si Low roughness” process:

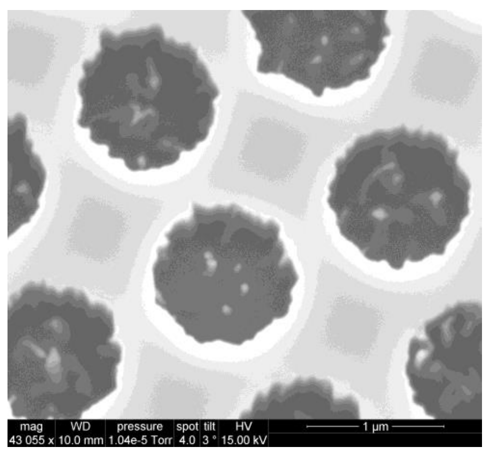
Source	SH bias	SF6/C4F8	SF6/C4F8	Valve/press	Temp
1200 W	75 W LF/10%	200/100 sccm	3/1 s	100%/~5mTorr	20 °C

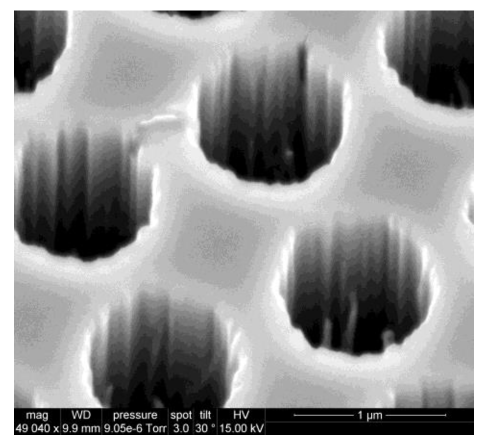
Observations:

- Etch rate: 0.6 um/min (determined with AFM)
- Wet etching Cr is a bad idea
- Grass inside the holes
- But at least we can etch Ta! and profile is nearly vertical!



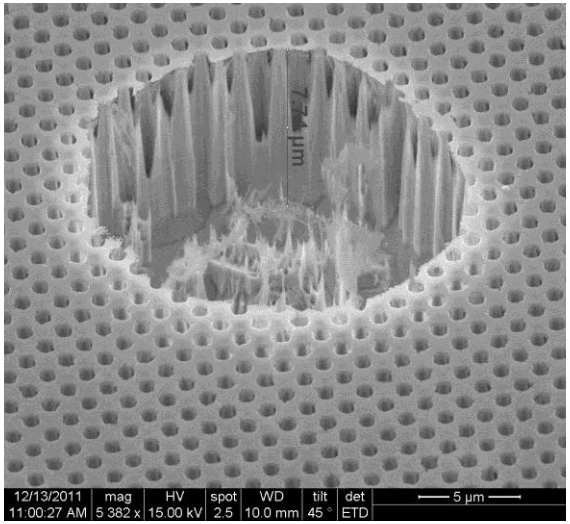
Wet etched Cr hard mask



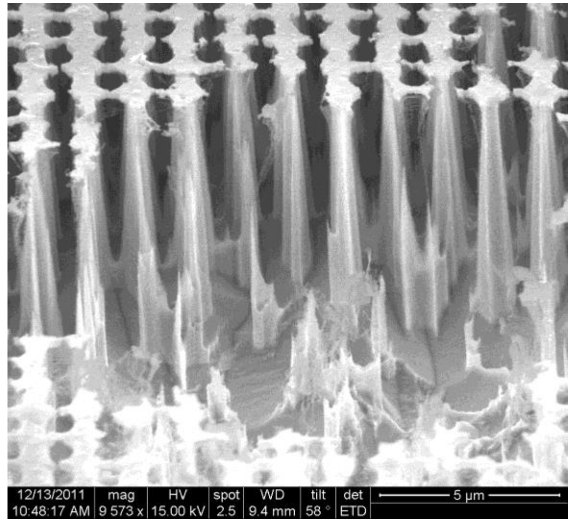


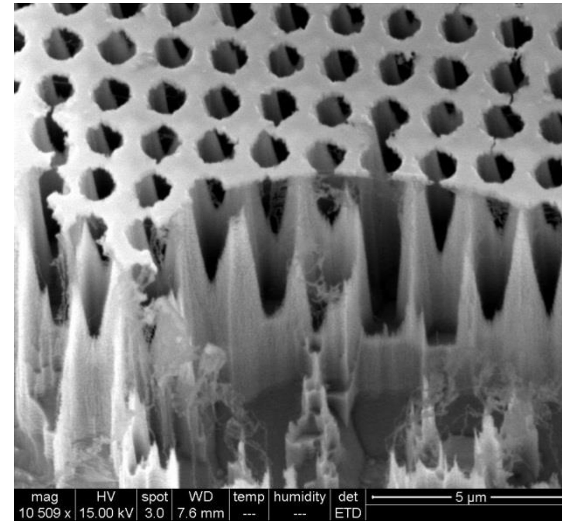
Cr hard mask on Ta etched for 5 mins

Ta Bosch etching appears to work, so let's etch a whole bunch of samples…



6 min etch, 7.7 um deep





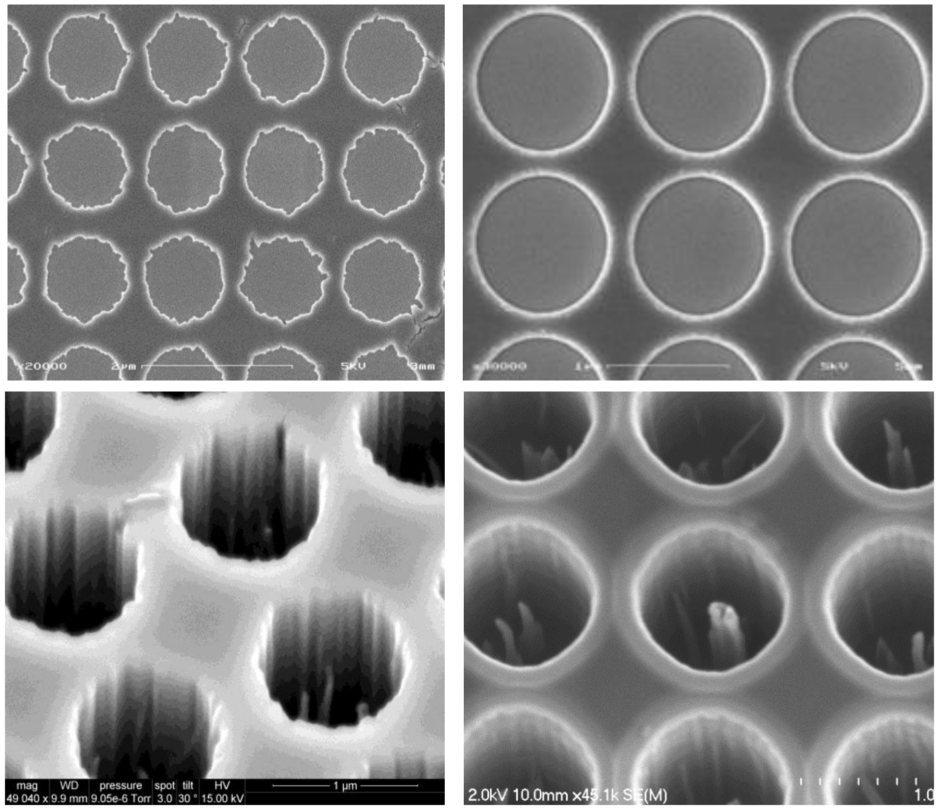
I destroyed six samples but learned two important things:

- Application of thermal grease is not trivial
- Increasing substrate temperature significantly increases Ta etch rate (1.3 um/min)

Source	SH bias	SF6/C4F8	SF6/C4F8	Valve/press	Temp
1200 W	75 W LF/10%	200/100 sccm	3/1 s	100%/~5mTorr	20 °C

Wet to dry etching of Cr hard mask

Cr wet etch | Cr dry etch

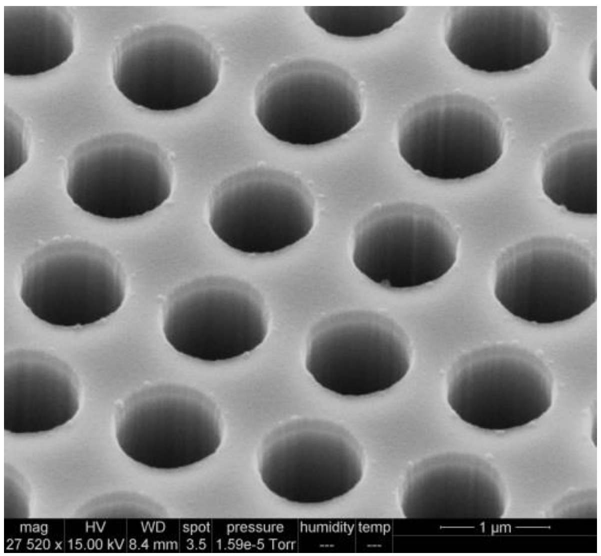


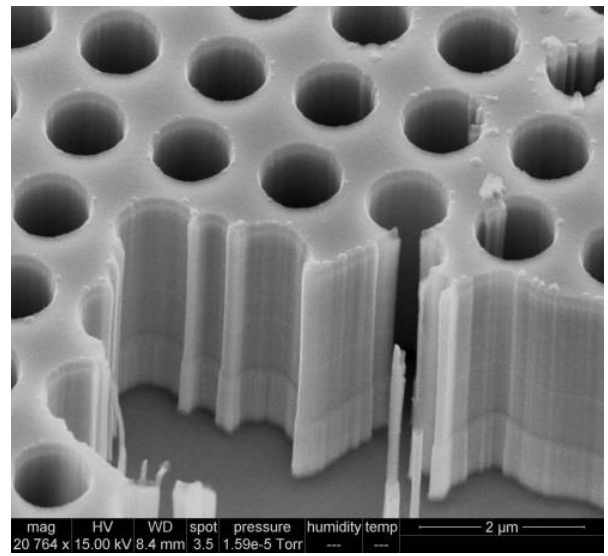
Dry etching Cr clearly the way to go but need to get rid of the grass

Source	SH bias	SF6/C4F8	SF6/C4F8	Valve/press	Temp
1200 W	75 W LF/10%	200/100 sccm	**3/2 s**	100%/~5mTorr	20 °C

Increased passivation time

Source	SH bias	SF6/C4F8	SF6/C4F8	Valve/press	Temp
1200 W	75 W LF/10%	200/100 sccm	**3/2 s**	100%/~5mTorr	20 °C

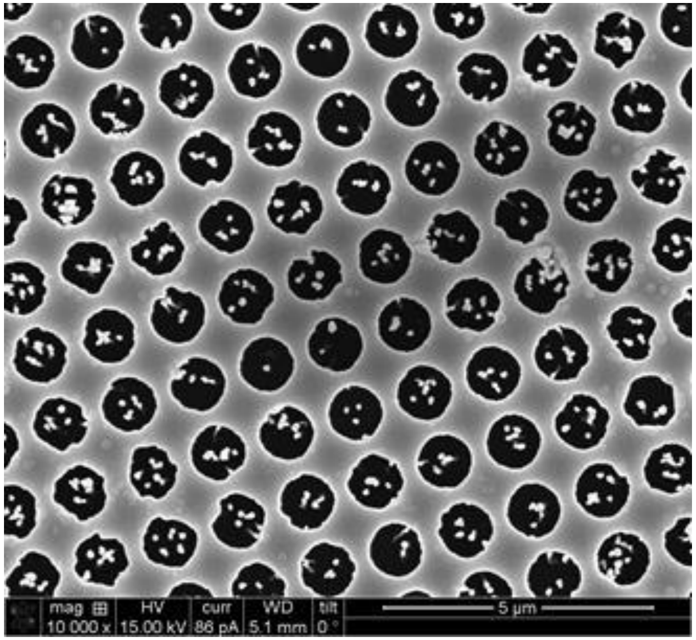


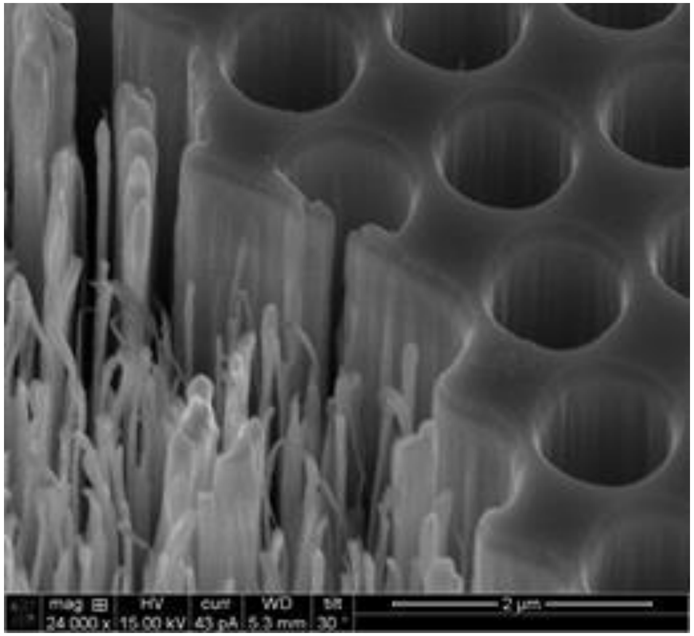


- Increased passivation time from 1.0 to 2.0
- Etch intervals with cooling step
- Etch rate: ~0.57 um/min (not significantly slower than 3/1s)
- Cr mask

Same process, different results

Source	SH bias	SF6/C4F8	SF6/C4F8	Valve/press	Temp
1200 W	75 W LF/10%	200/100 sccm	3/2 s	100%/~5mTorr	20 °C

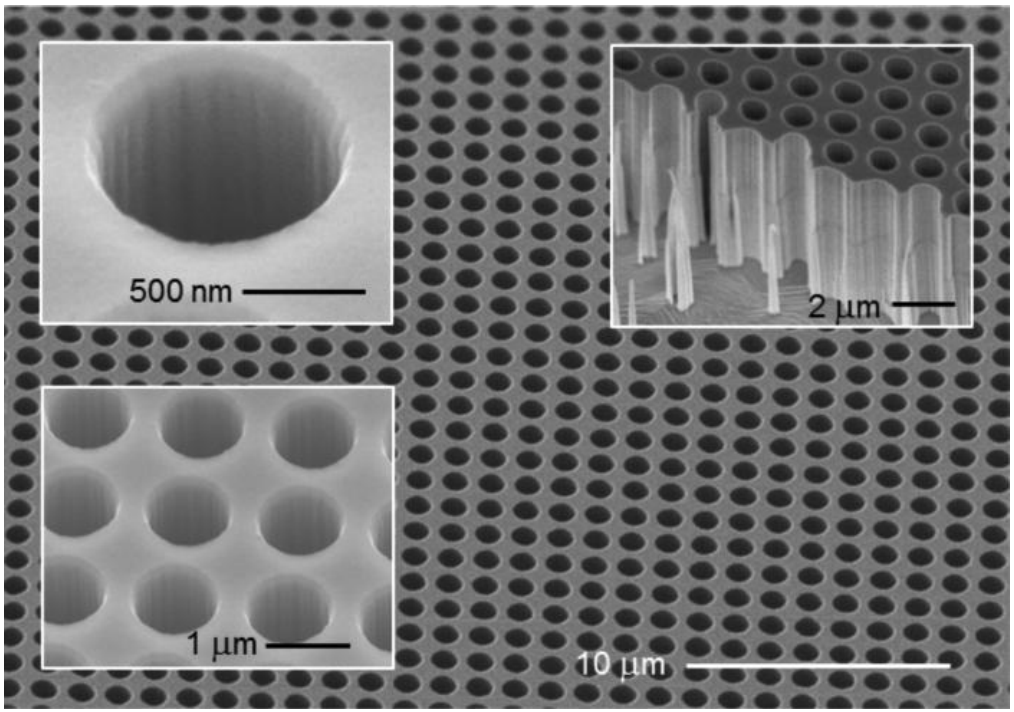




- Significant grass
- Maybe passivation time is too long?
- Possibly also an issue with prior processing (RIE, lithography)

Reduced passivation time

Source	SH bias	SF6/C4F8	SF6/C4F8	Valve/press	Temp
1200 W	75 W LF/10%	200/100 sccm	**3/1.5** s	100%/~5mTorr	20 °C



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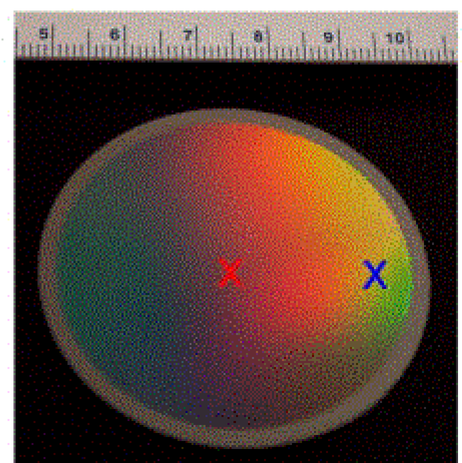
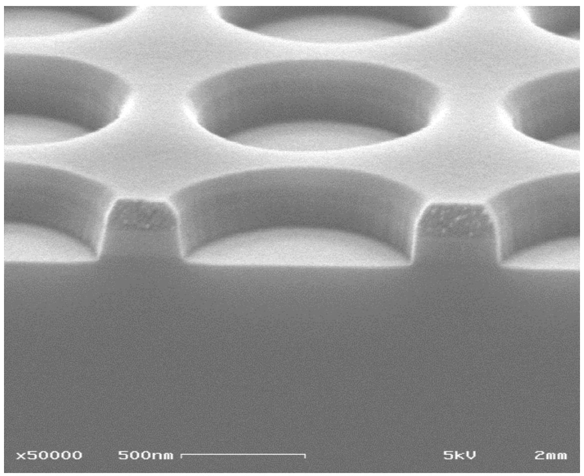
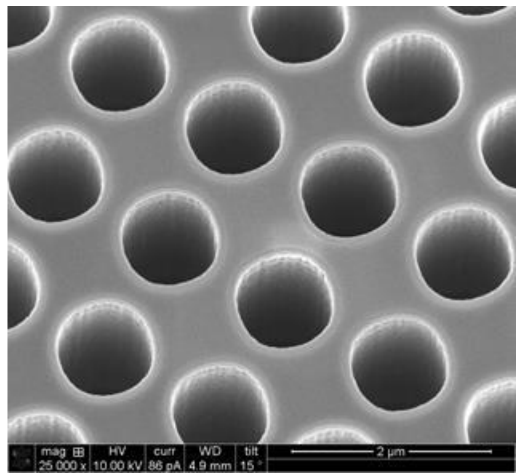
- 2” diameter Ta substrate
- Cr hard mask
- 3/1.5s passivation time

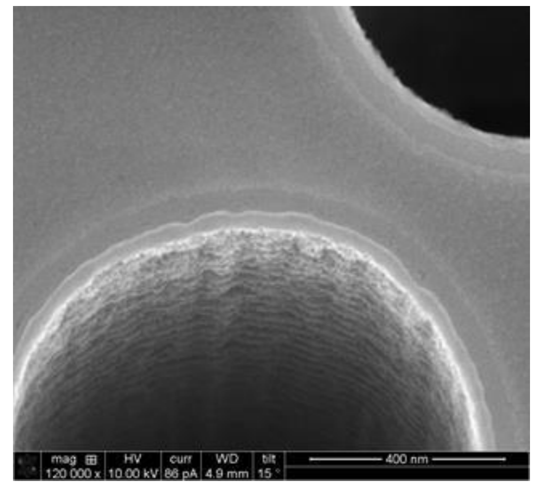


Attempts to reduce grass

- Reduce passivation time, but not too much: 1.5 s
- Implement SiO2 hard mask to address possible resputtering of Cr





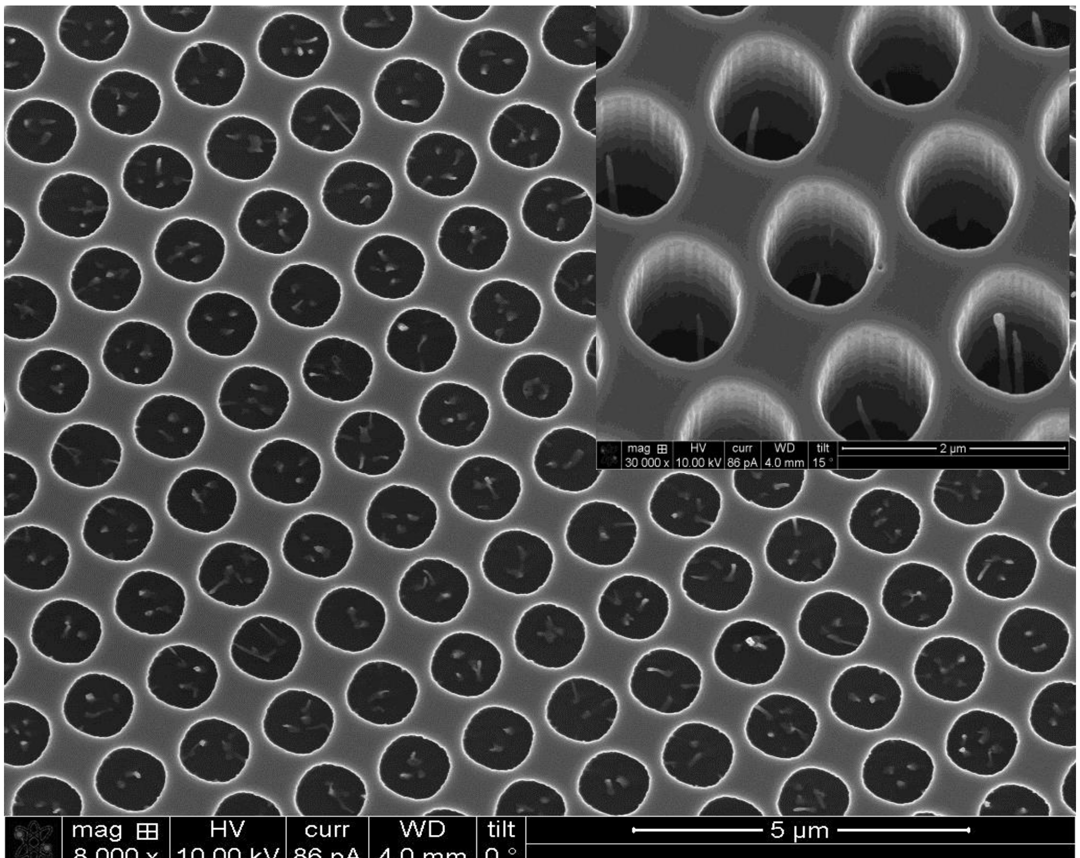


- ~100 nm SiO2 mask (but up to 250 nm)
- Selectivity: ~25:1

Source	SH bias	SF6/C4F8	SF6/C4F8	Valve/press	Temp
1200 W	75 W LF/10%	200/100 sccm	3/1.5 s	100%/~5mTorr	20 °C

Switched to SiO2 mask and reduced passivation time, but still some grass

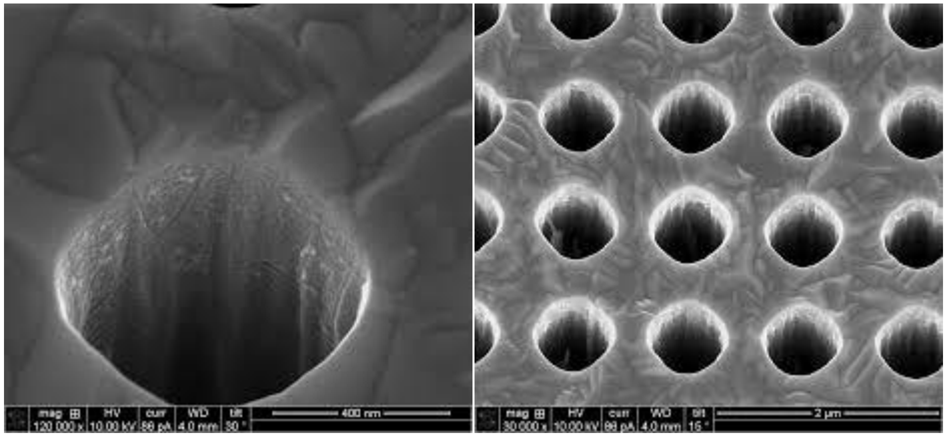
Source	SH bias	SF6/C4F8	SF6/C4F8	Valve/press	Temp
1200 W	75 W LF/10%	200/100 sccm	**3/1.5 s**	100%/~5mTorr	20 °C



5 min etch

Variations in the PhC

Thick (30 um) sputtered Ta



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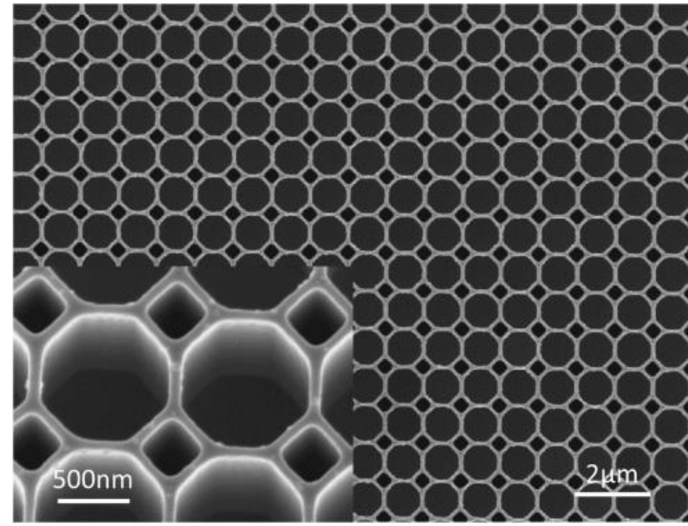
Patterning with UV-NIL process



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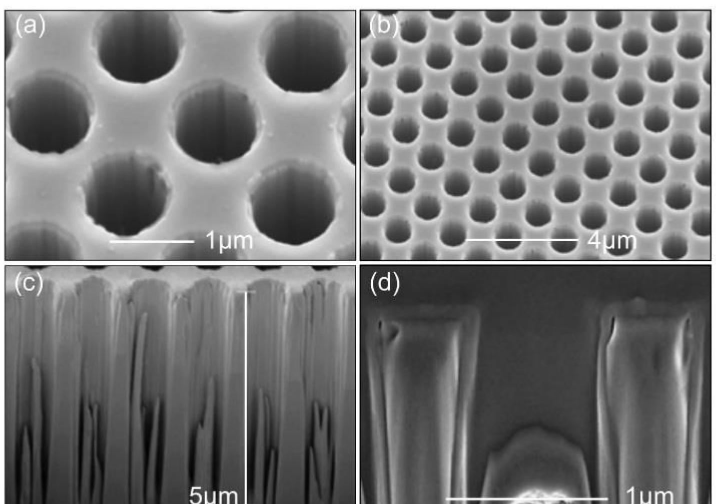
TaW alloys



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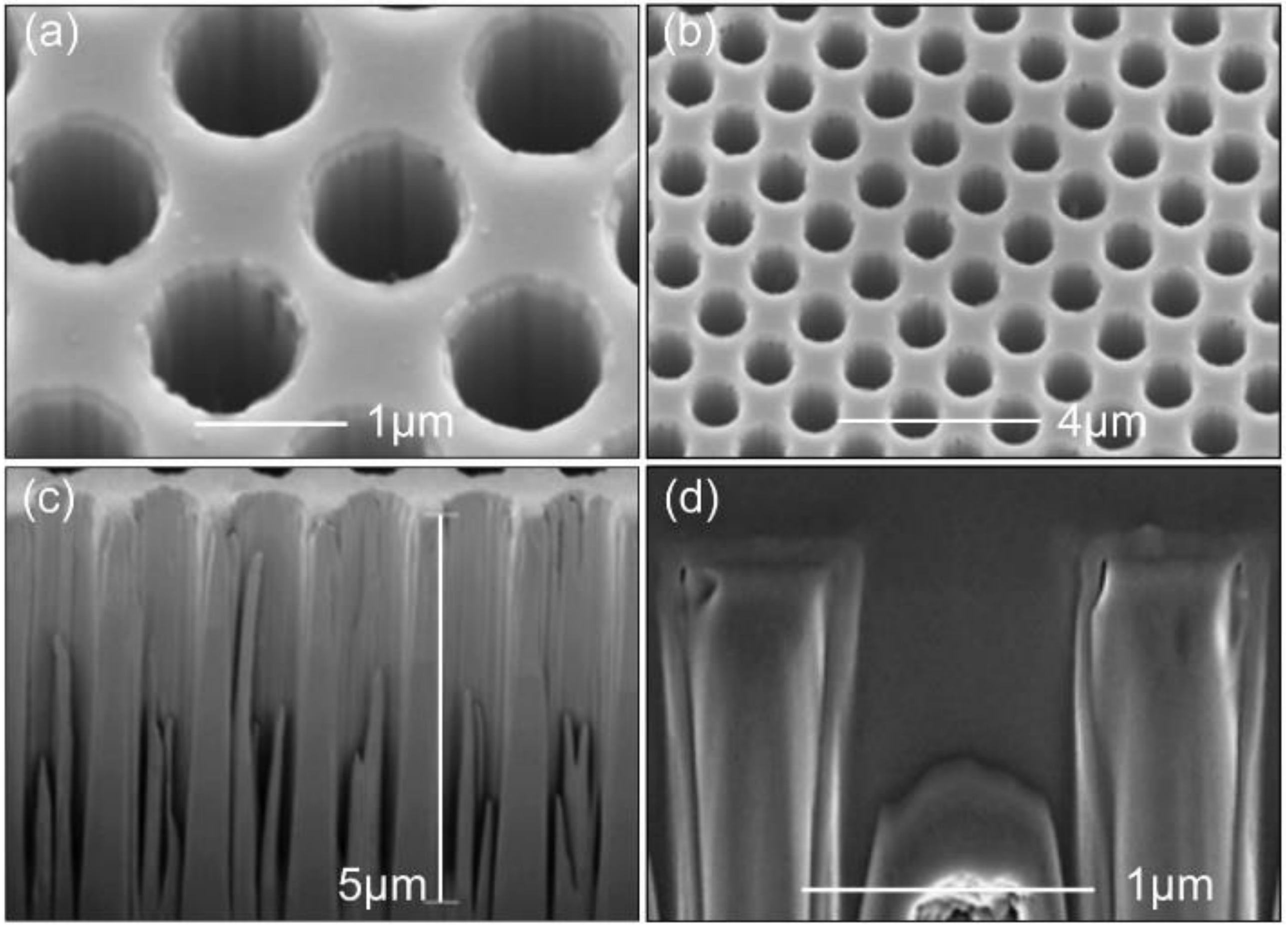
FIB cross section of TaW PhC



Influence of grass had minimal effect on performance of emission spectrum

Where we stand

Source	SH bias	SF6/C4F8	SF6/C4F8	Valve/press	Temp
1200 W	75 W LF/10%	200/100 sccm	3/1.5 s	100%/~5mTorr	20 °C

- SiO_2 hard mask
- Aspect ratio as high as 8:1
- Etch rate > 0.6 um/min
- In retrospect, some sort of DOE would have been good
- Chamber conditioning a likely suspect for inconsistent etch results
- After talking during breakfast with NNCI etch people: Improve substrate mounting (different bonding material?).
  - Boron-nitide is notorious for redeposition
  - Try fomblin oil, crystal bond, PMMA…