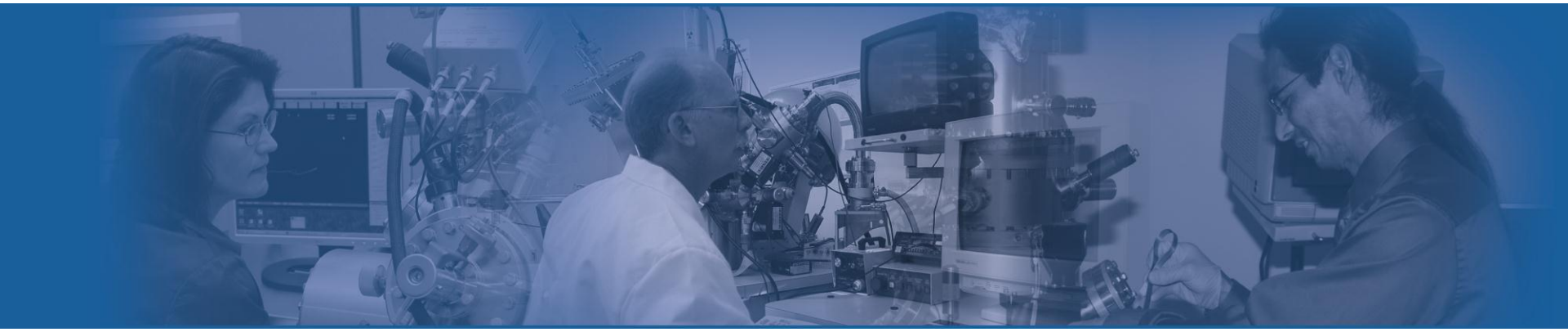

Characterization of a Low-Flow Flat Cell using the Nu Astrum GDMS

Glyn Churchill, DeAnn Barnhart, Karol Putyera

EW-GDS
September 3-4, 2012
Kingston, UK

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Outline

- Introduction
- Glow discharge instruments at EAG
- The Nu Astrum
- Pin and flat cells
- Flat cell and sample holder contribution
- Operating parameters
- Abundance sensitivity
- Flat cell sputtered surface roughness
- Flat cell crater shape

- Summary

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Introduction

- The Nu Instruments Astrum GDMS has been commercially available for 2 years
- The flat cell for the instrument has recently been introduced

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Evans Analytical Group GDMS Instruments

- Reduced Flow GD Source:
  - VG9000 by Thermo-Fisher (13)
  - Astrum by Nu Instruments (2)

- Fast-Flow GD Source:
  - Element GD by Thermo-Fisher (4)

From 2005

From 2010

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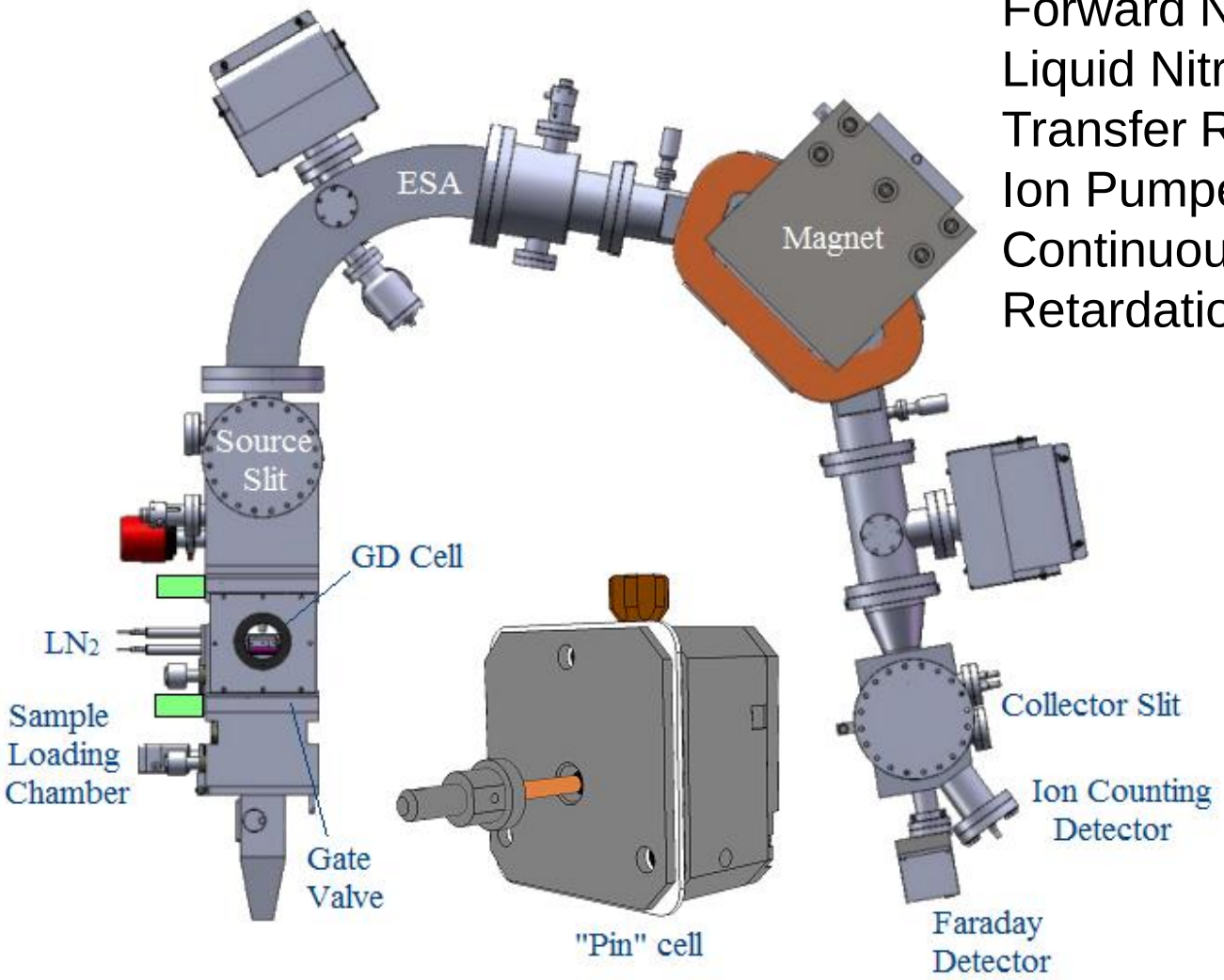
Schematic Diagram of the Nu Astrum



Forward Nier-Johnson Geometry
Liquid Nitrogen Cooled Sample Transfer Region Pumping
Ion Pumped Analyser
Continuously Variable Collector Slit
Retardation Filter

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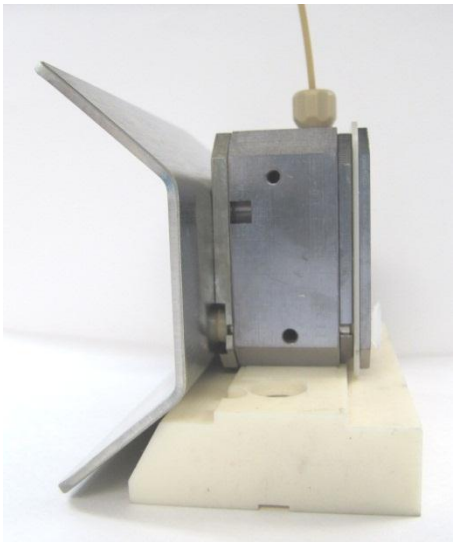
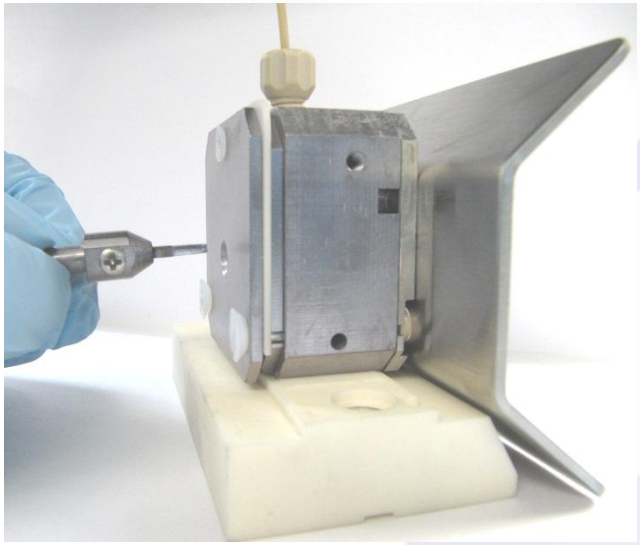
Nu Astrum Pin Cell





Standard pin size
2 x 2 x 22 mm

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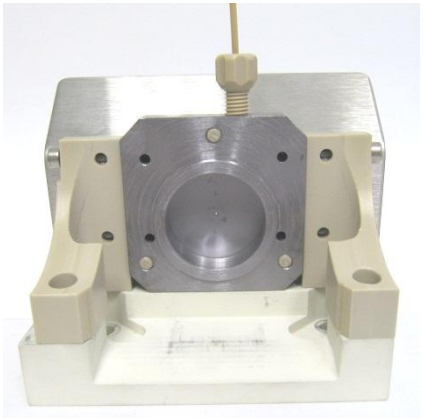
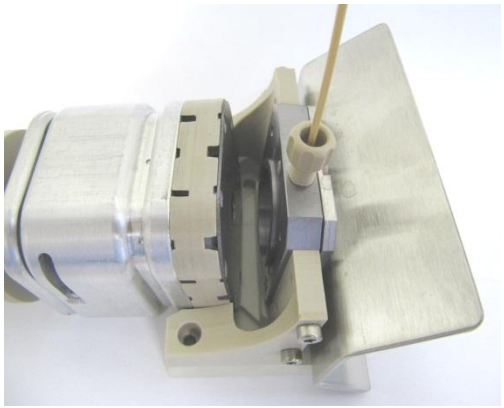
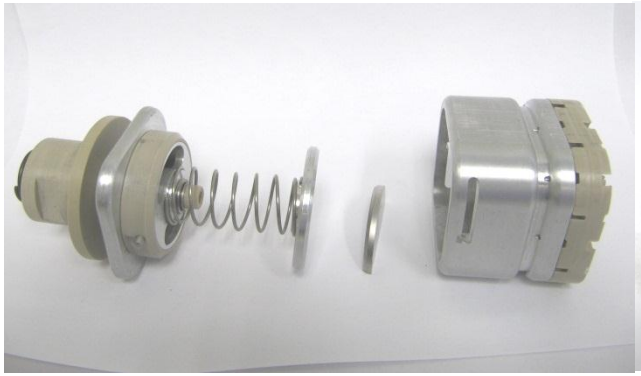
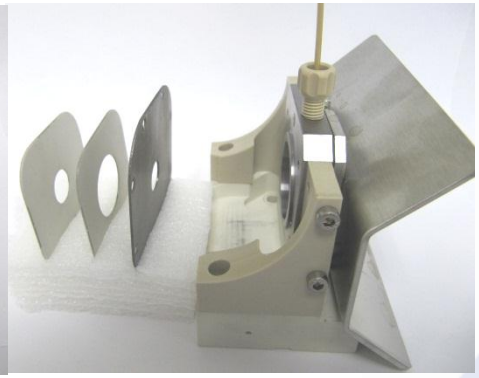
Nu Astrum Flat Cell









Minimum sample size 12 mm diameter
Maximum sample size 42 mm diameter or nearly 42 x 42 mm square (Corners cut off)

Maximum height around 20 mm

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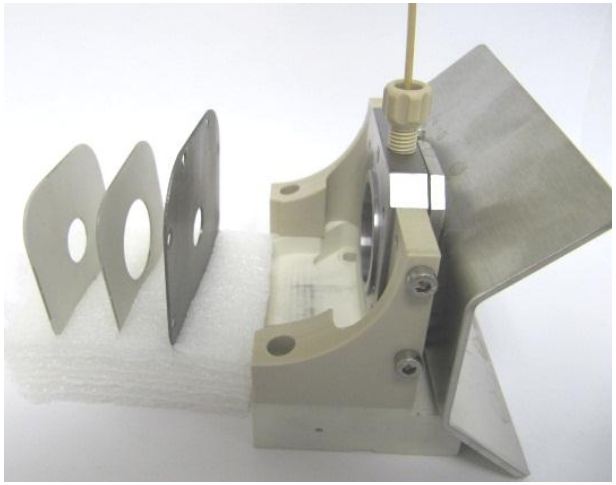
Critical Astrum Flat Cell Materials



• Main cell body	Tantalum
• Cell exit slit	Tantalum foil
• Anode front plate	10 mm diameter tantalum plate
• Insulators	11 mm diameter aluminum nitride (can be made out of aluminum oxide or sapphire)

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Signal contribution from the cell

- There are two main sources of unwanted contribution from the cell assembly:

1) The aluminum nitride insulators

- Aluminum was measured at approximately 1.5 ppmw

2) The tantalum anode front plate, cell body and exit slit

- Tantalum was measured at approximately 0.3 ppmw

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Flat Cell Typical Operating Parameters

- Glow voltage 1000 V
- Glow current 2 mA
- Glow discharge power 2 W
- Exit aperture 0.3 mm diameter 0.07 mm^2
- Argon flow rate 0.1 to 0.2 sccm
- Source pressure 1 to 2 x 10^{-5} mbar
- Transfer chamber pressure ~ 5 x 10^{-8} mbar
- Detector chamber pressure ~ 3 x 10^{-9} mbar
- Sample temperature < -10 °C (2 hrs @ 2 W)
- Anode to cathode distance 1.28 mm

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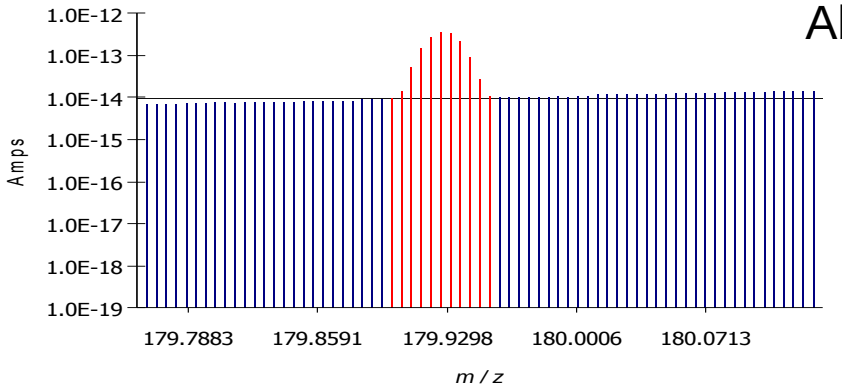
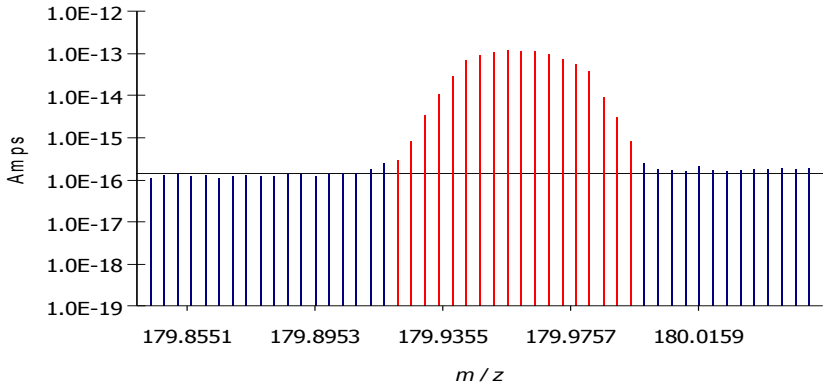
Abundance sensitivity for tantalum

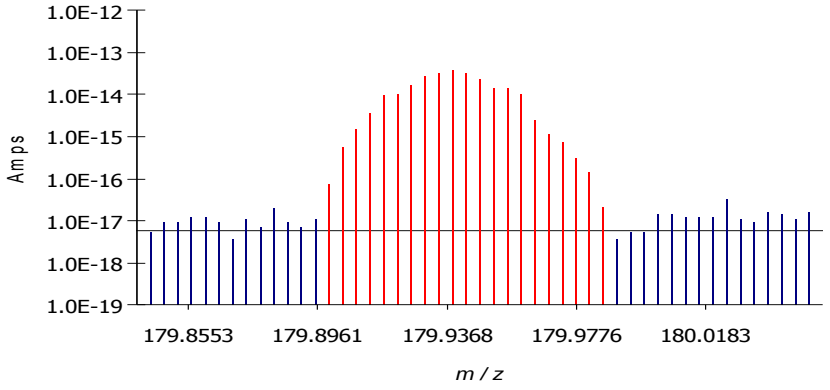
Abundance tail from the ^{181}Ta at the ^{180}Ta peak



VG9000 pin cell
Abundance sensitivity < 5 ppm



Astrum pin cell
Abundance sensitivity < 0.2 ppm



Astrum flat cell
Abundance sensitivity < 0.05 ppm

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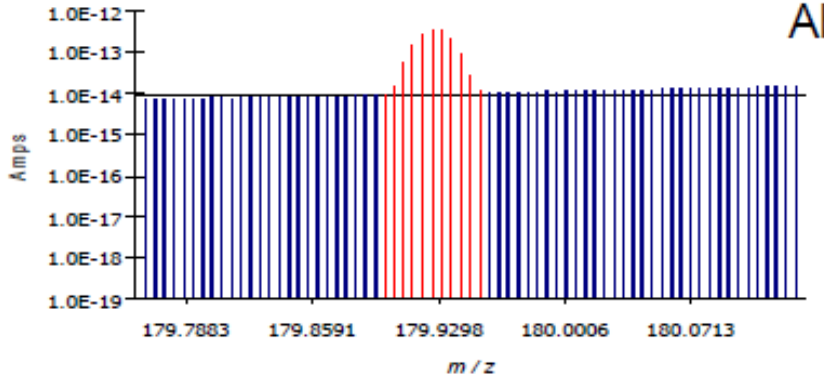
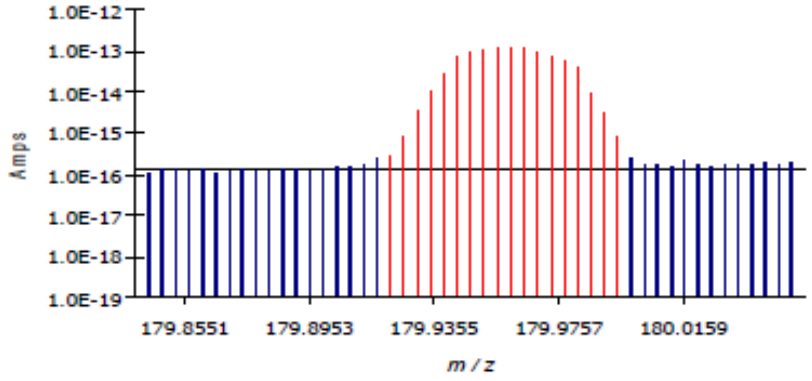
Abundance sensitivity for tantalum

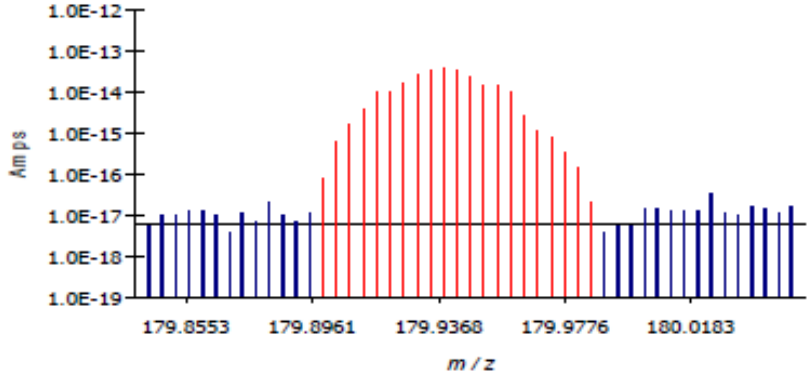


Abundance tail from the ^{181}Ta at the ^{180}Ta peak

VG9000 pin cell
Abundance sensitivity < 5 ppm



Astrum pin cell
Abundance sensitivity < 0.2 ppm



Astrum flat cell
Abundance sensitivity < 0.05 ppm

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Sample Temperature

- Titanium thermal conductivity = 21.9 $Wm^{-1}K^{-1}$
- Copper thermal conductivity = 400 $Wm^{-1}K^{-1}$
- Titanium thermal conductivity is nearly 20 times poorer

- For titanium the temperature differential across a 22 x 2 x 2 mm pin at 3.5 W would be 879 K

- For a well polished flat sample the temperature the cooling is significantly improved

D.R. Lide, (Ed.) in *Chemical Rubber Company handbook of chemistry and physics*, CRC Press, Boca Raton, Florida, USA, 79th edition, 1998

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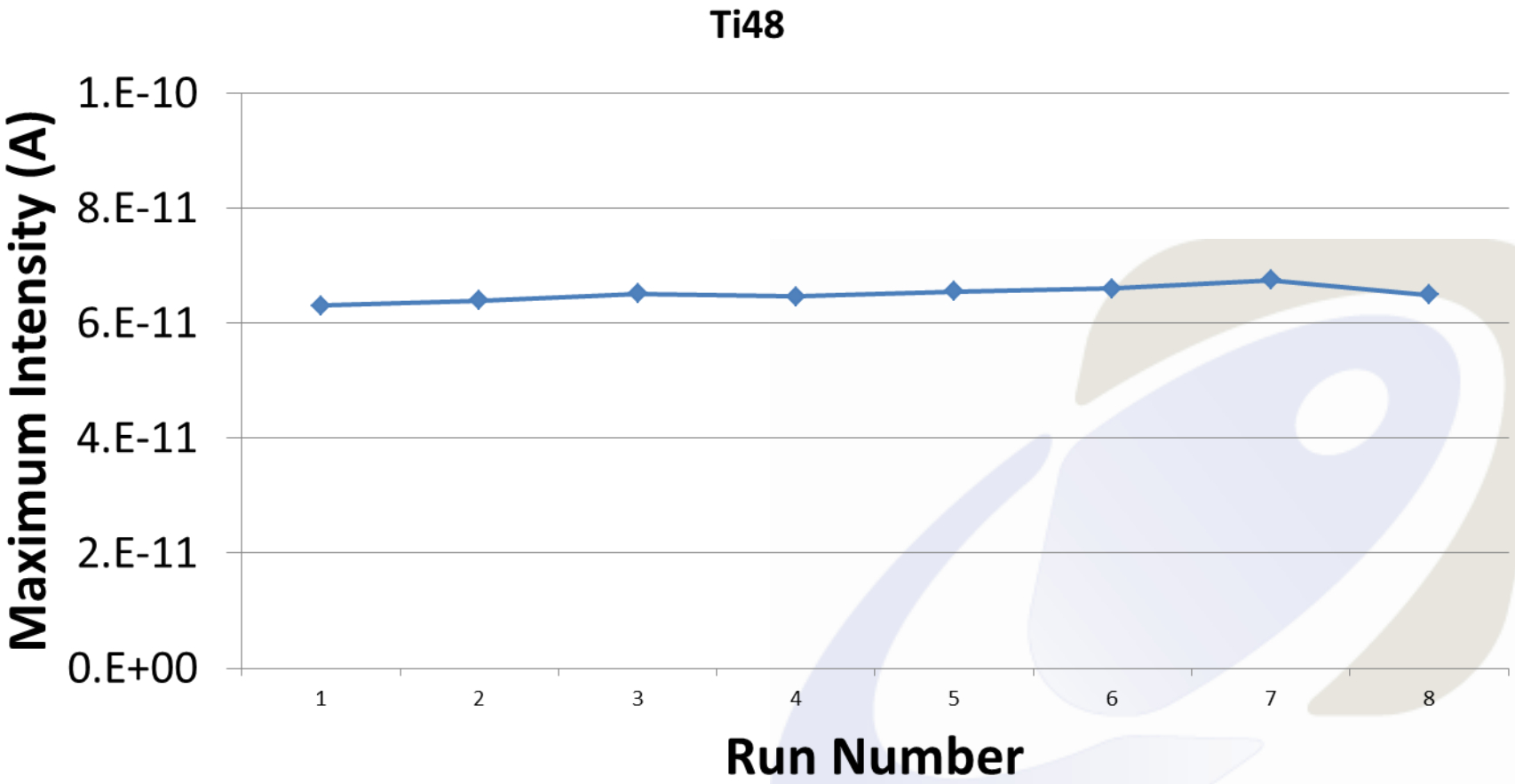
Maximum intensity change over 2 hours

< 2% SD over 2 Hours



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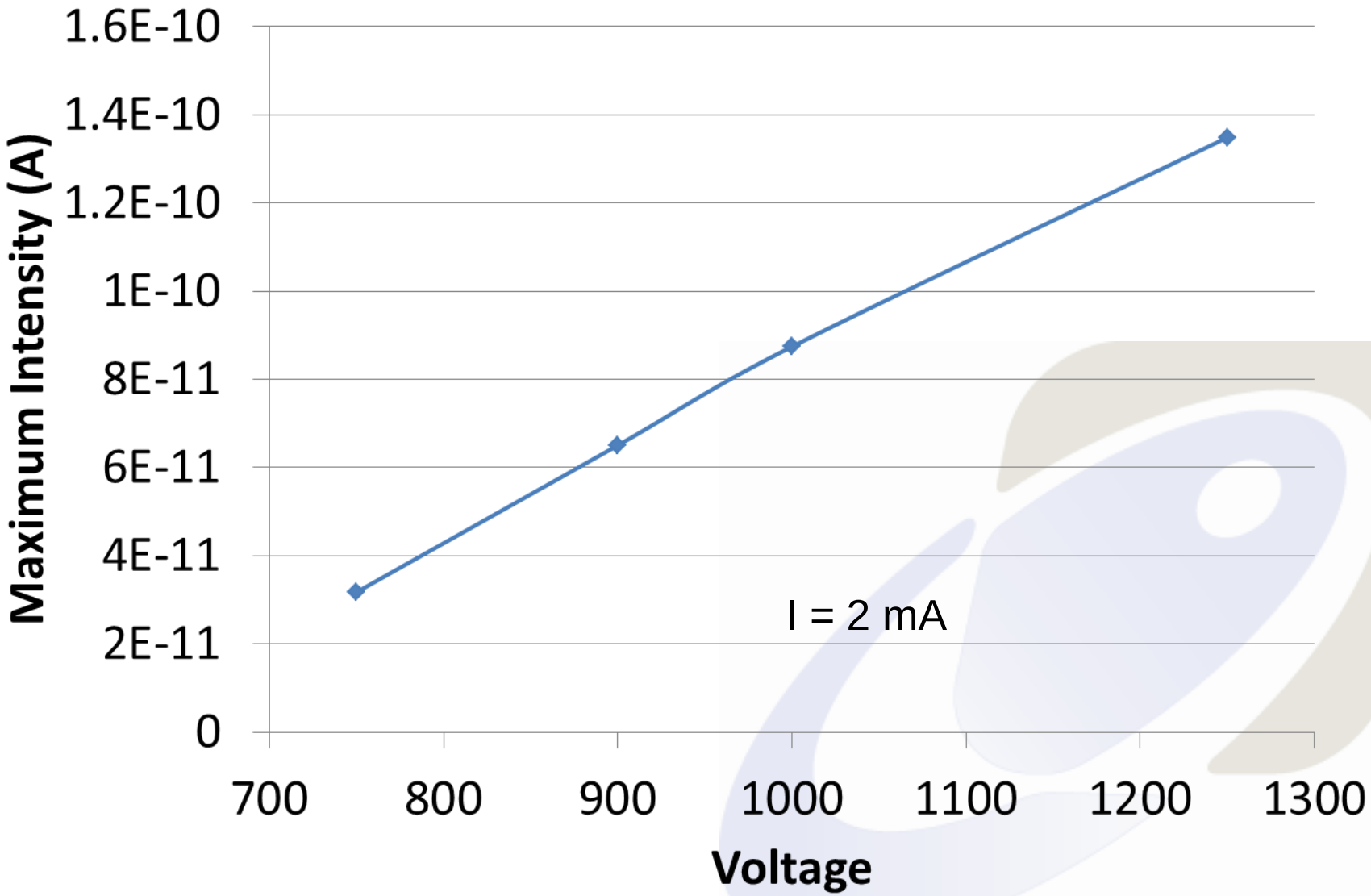
Signal Intensity vs. Voltage in Titanium



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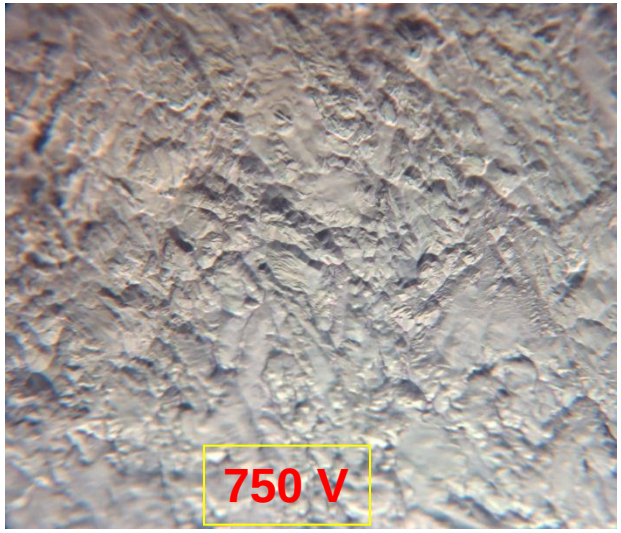
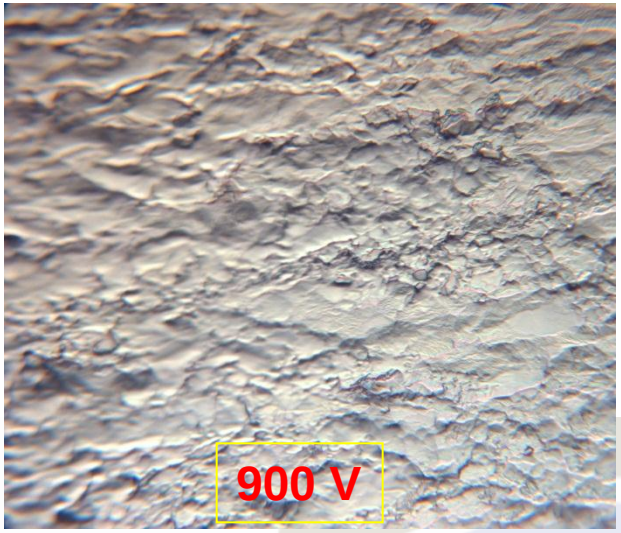
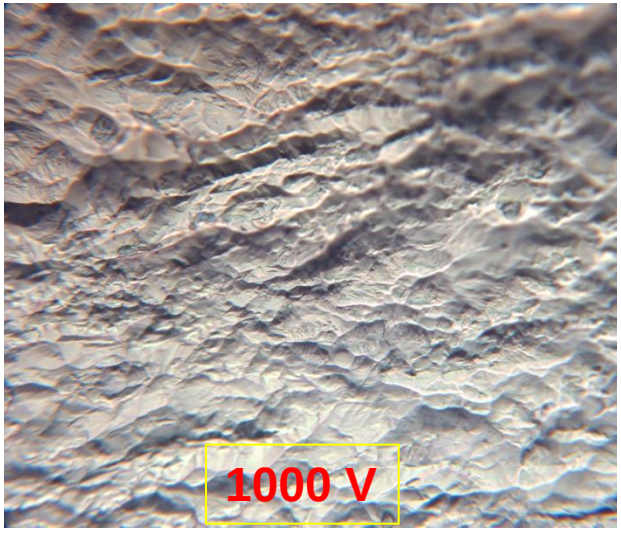
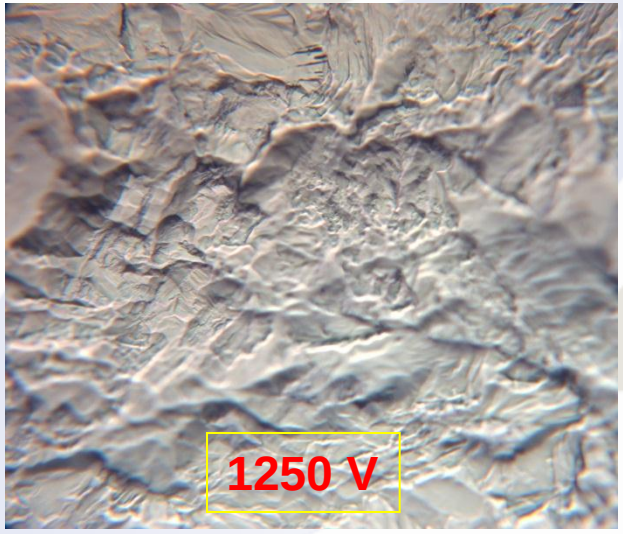
Atomized Surface Microstructures
Two hours plasma sputtering – 2 mA – Increasing GD Voltages

Optical Microscopy Images at 500x









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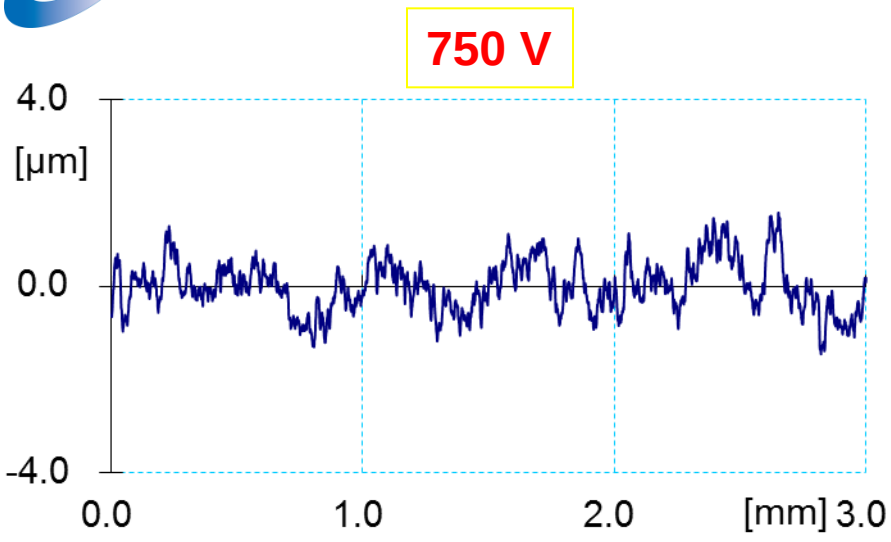
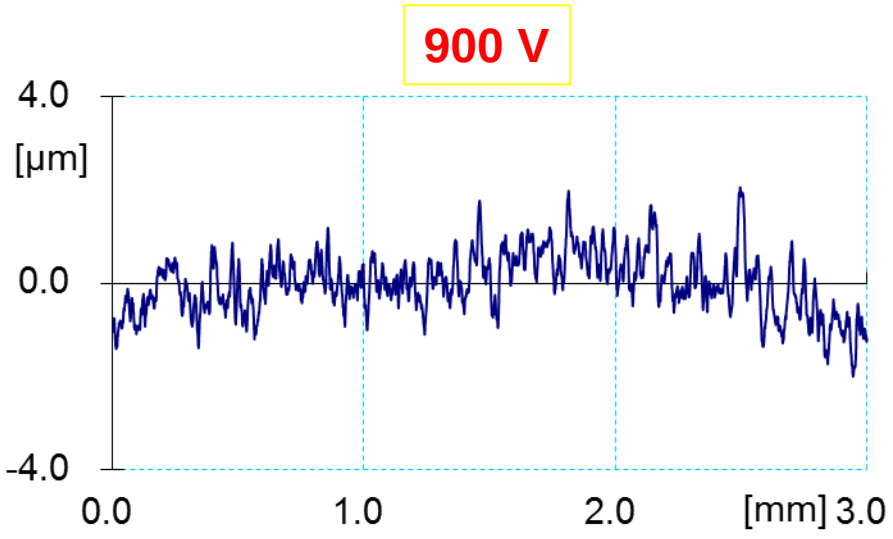
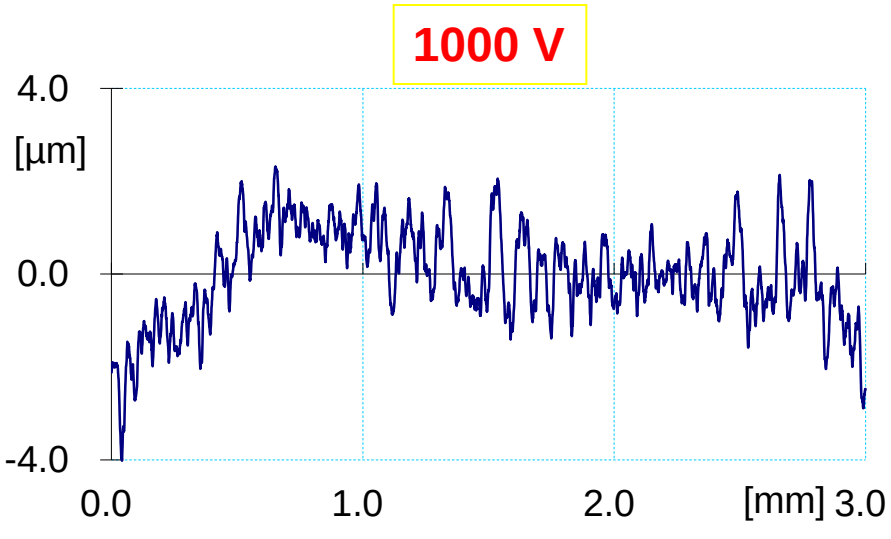
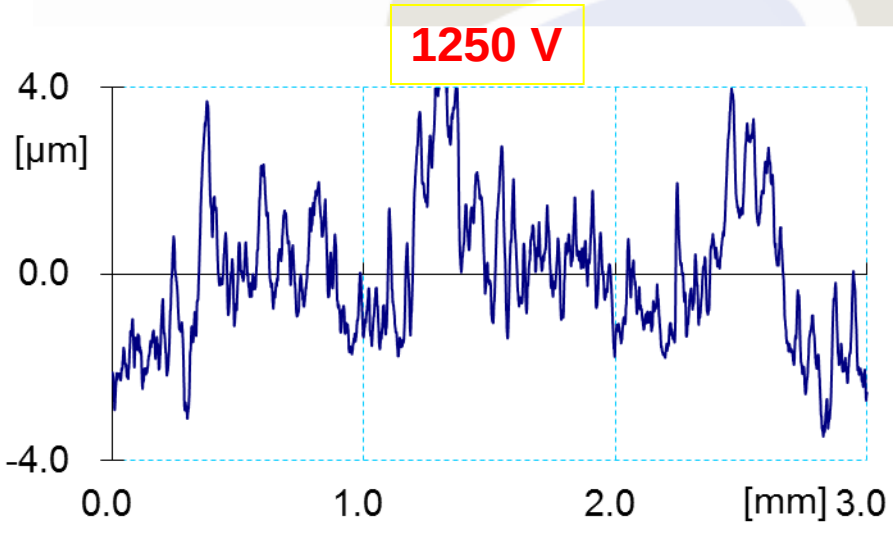
Atomized Surface Roughness Development
P Profiles









Two hours plasma sputtering – 2 mA – Increasing GD Voltages

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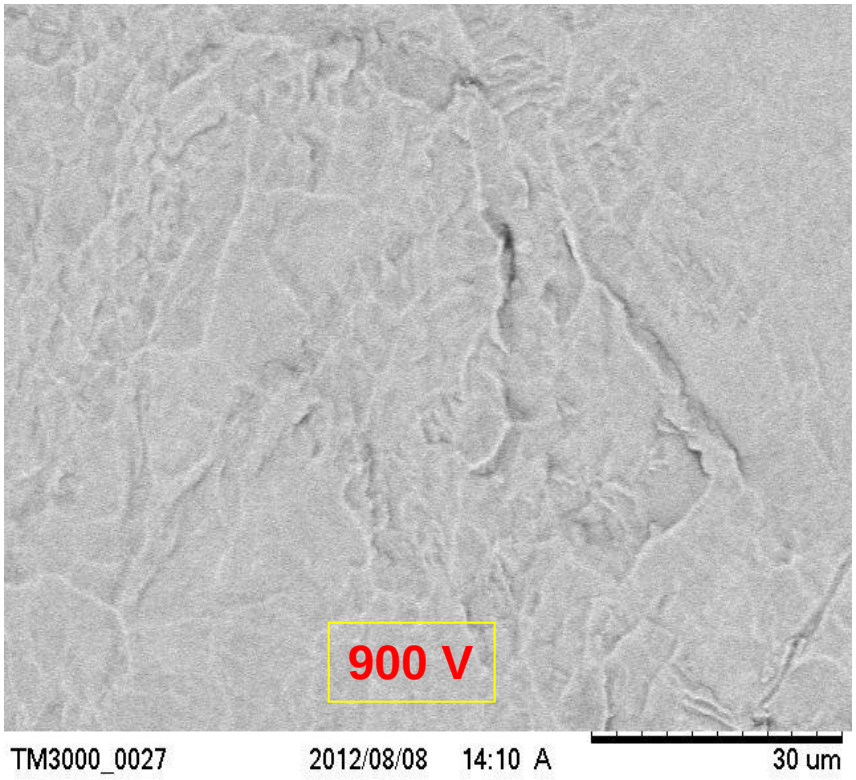
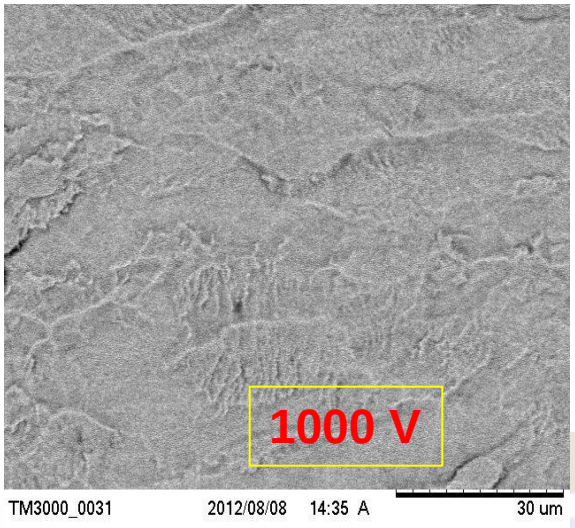
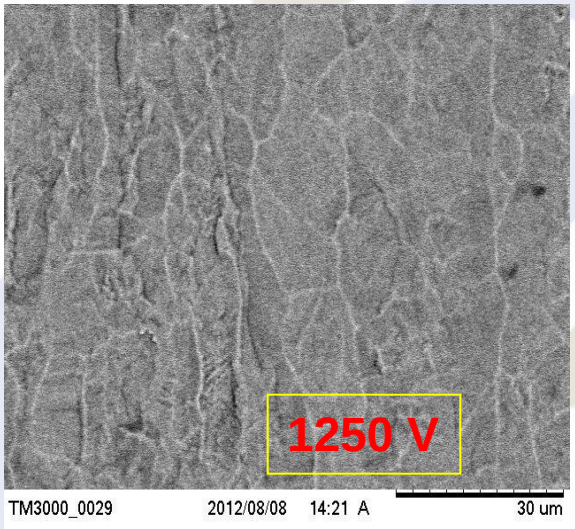
Plasma Etching

Two hours plasma sputtering – 2 mA – Increasing GD Voltages

SEM – COMP Mode Images







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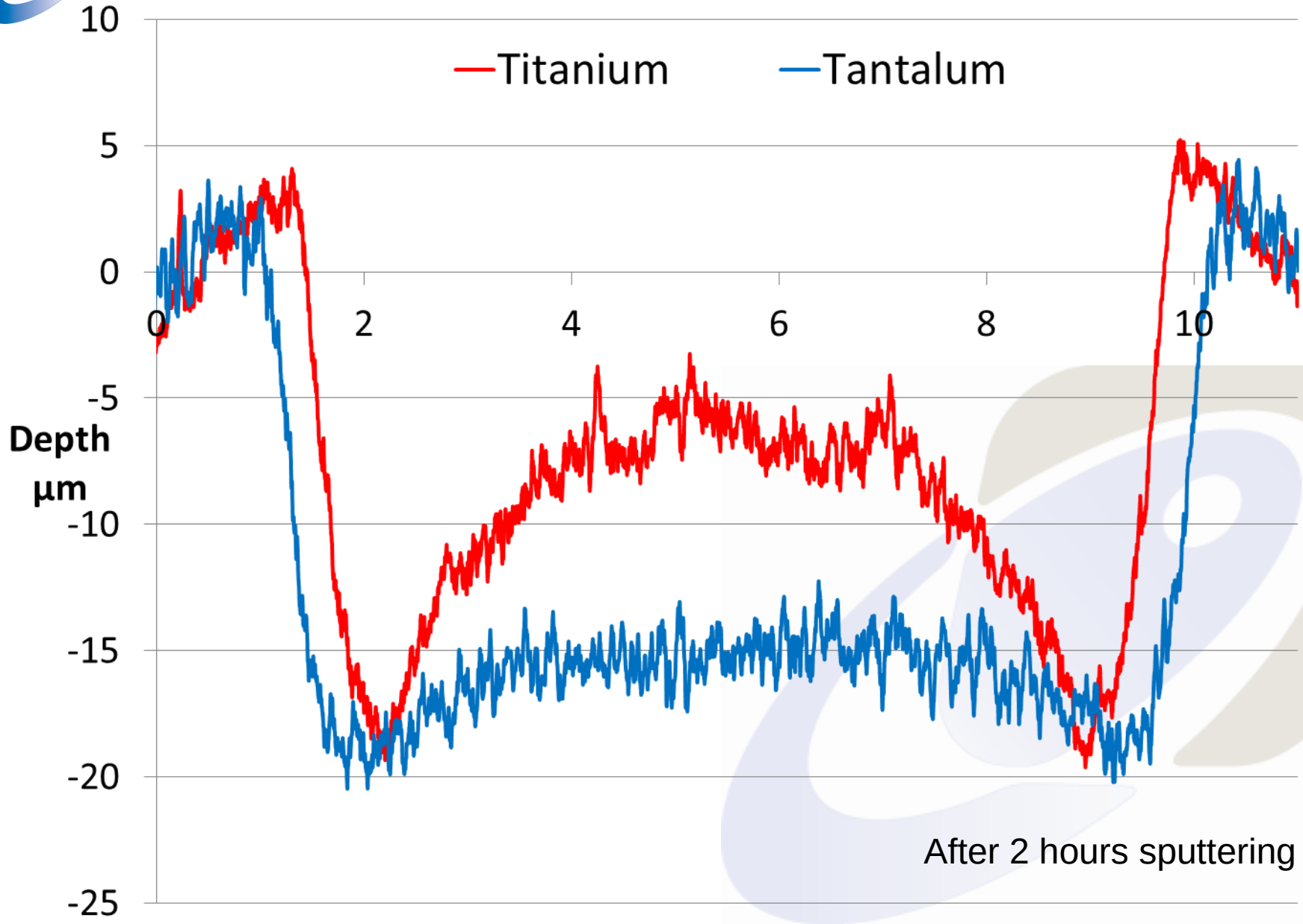
Tantalum vs. titanium crater profile at 1000 V, 2 mA



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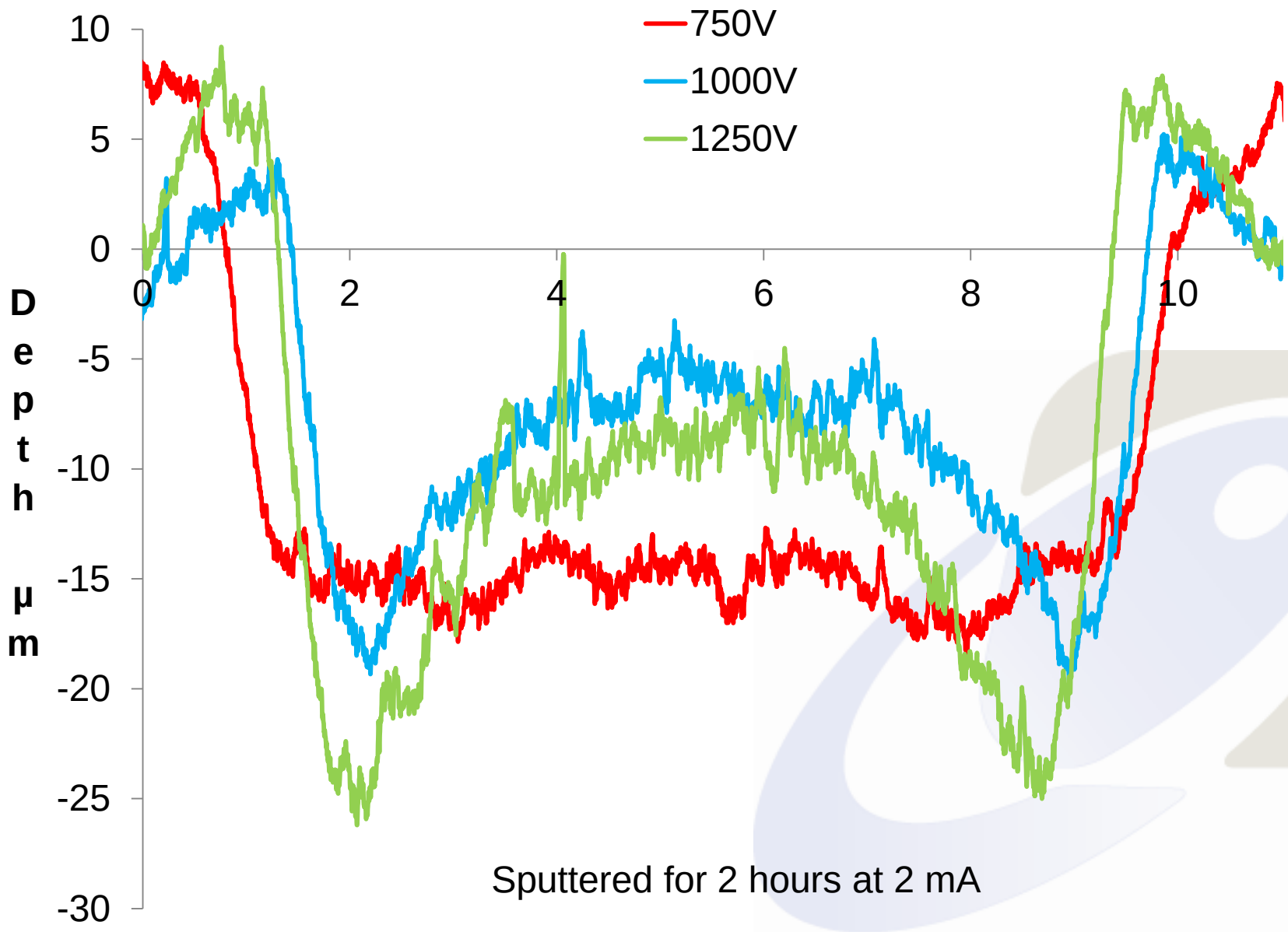
Crater profiles 750 V to 1250 V



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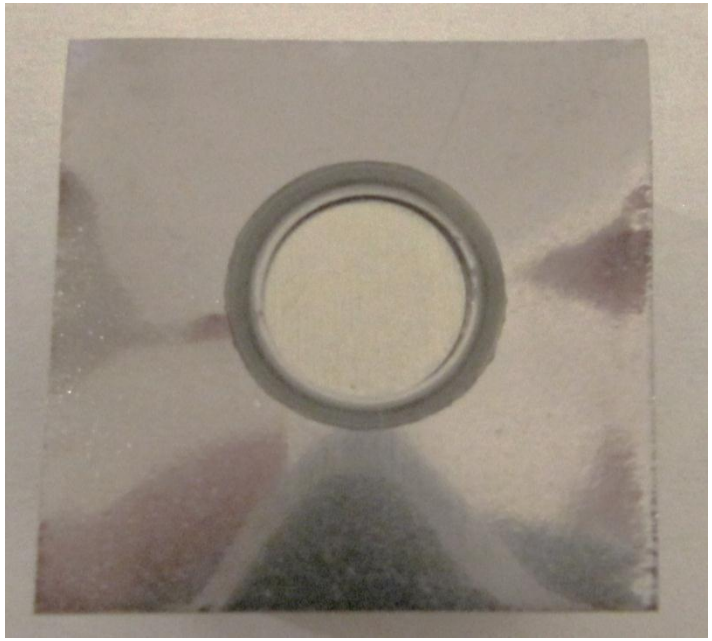
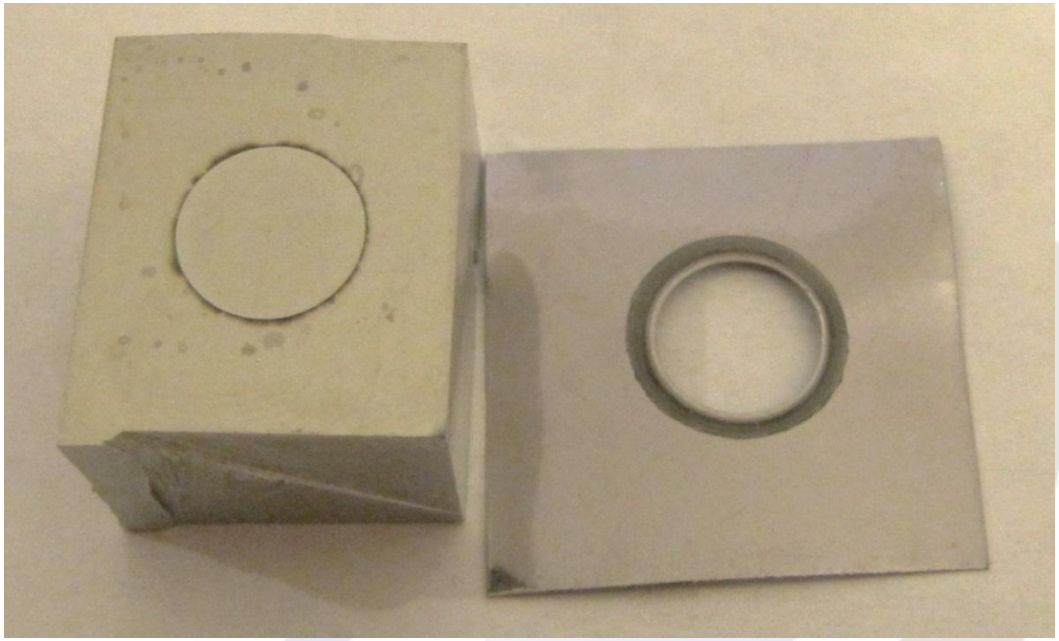
Cathode Mask

- Titanium with a 8 mm tantalum foil mask used to improve the crater sides.





Raith A, Hutton R.C, and Huneke J.C. JAAS,1993, 8, 867.

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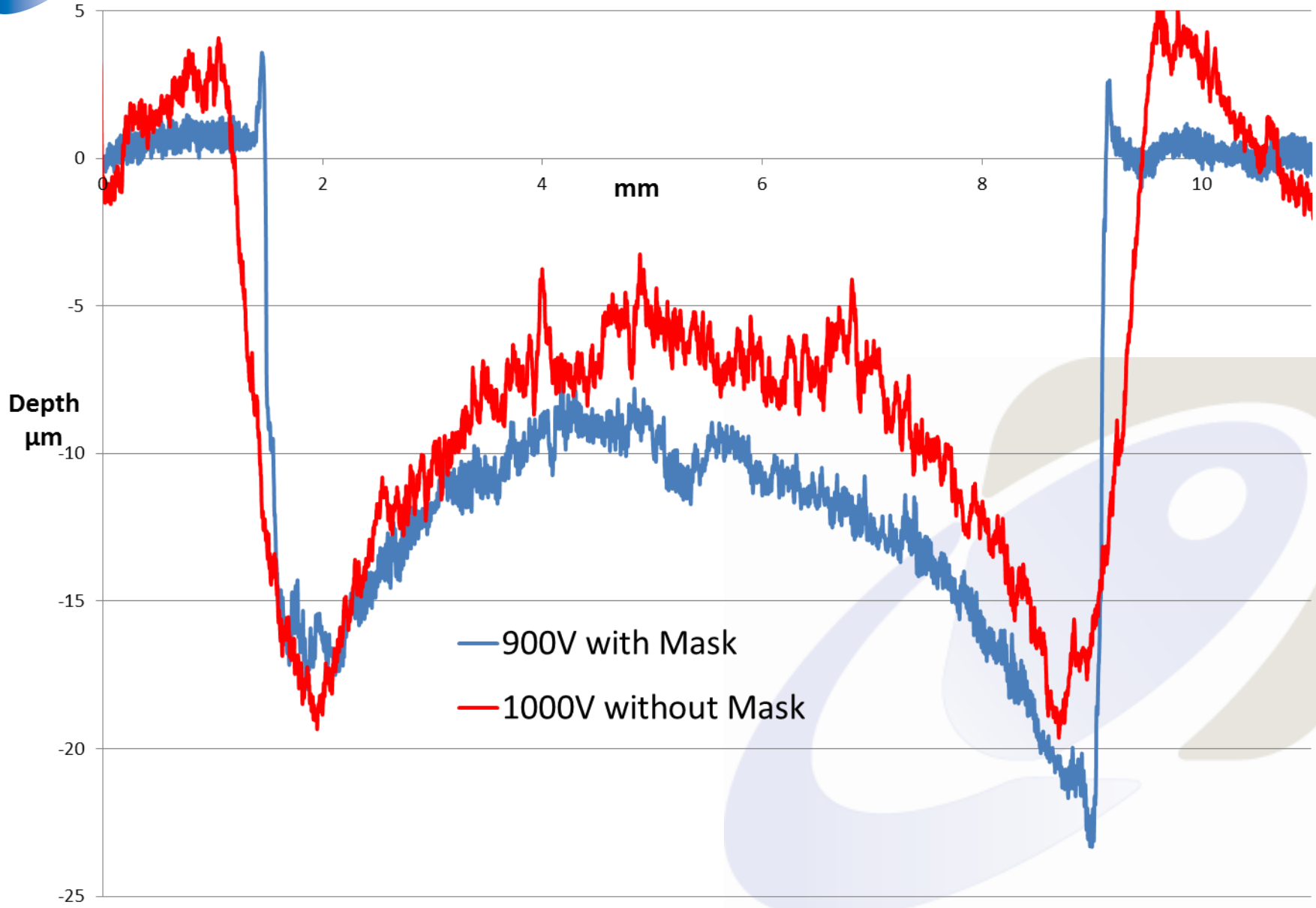
4N5 Titanium with and without a 8 mm Tantalum mask



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Tantalum cathode mask contribution

- The tantalum contributes approximately 12 % of the titanium signal therefore a very clean mask needs to be used in order that the mask does not significantly contribute to the sample impurities

- Without the mask there is approximately a 0.3 ppmw contribution from the anode front plate

- A smaller difference between the anode front plate and the mask would reduce this ratio at the expense of the crater wall

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Summary

- The Astrum and VG9000 flat cells are similar in design and performance
- The improved abundance sensitivity and resolution of the Astrum has many advantages over the VG9000
- New acquisition electronics coupled with the fast laminated magnet should give a large improvement in depth resolution over the VG9000
- The improved cooling for the flat cell may reduce preferential removal of certain elements
- Techniques for improving crater shape like the anode mask can be used with this cell

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We would like to thank:

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And also Edward Steers and Sohail Mushtaq from London Metropolitan University

Thank You!

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