

***SURFACE MORPHOLOGY CHANGES AND  
BLISTERING OF W EXPOSED TO HIGH  
FLUENCE D PLASMA***



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# Overview

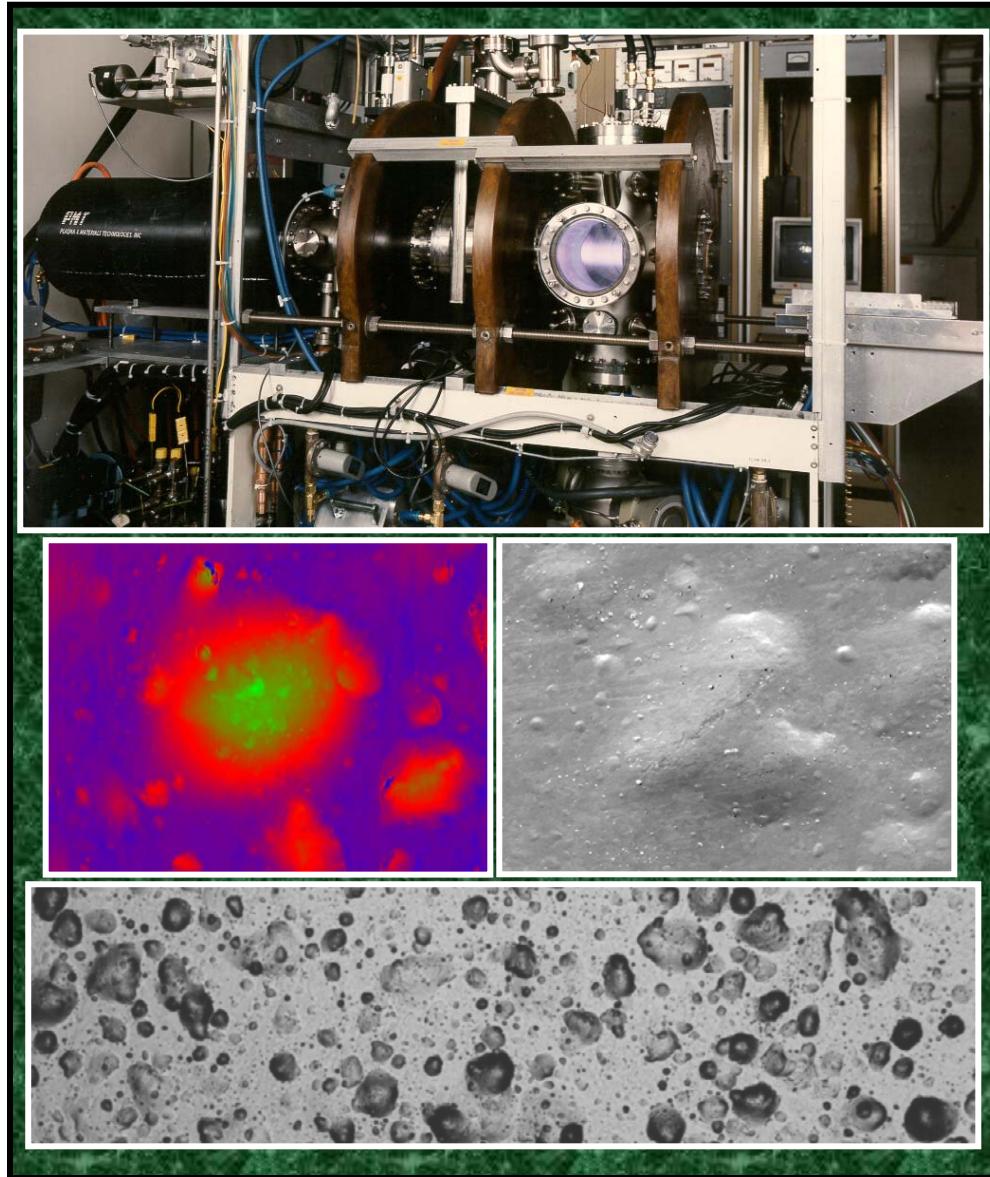
**Primary Motivation:** Studies of blister formation in W to evaluate potential impact on ITER operation.

**Tritium Plasma Experiment Overview:**

- Collaboration between Idaho National Laboratory and Sandia/CA
- Unique capability to expose samples to D+T plasmas.
- Use of tritium provides much greater sensitivity for retention and plasma driven permeation measurements.

**In this study:**

- We examine blister sizes for Plansee W at different sample temperatures.
- Surface diagnostics include SEM, optical microscopes, laser profilometry.



# Experimental Configuration

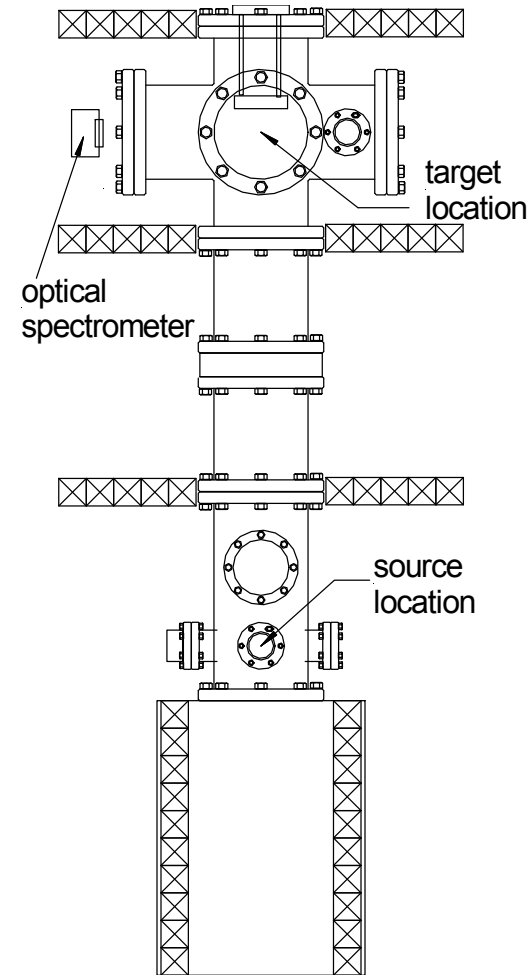
## Tritium Plasma Experiment (TPE) Overview

### Diagnostics:

- Langmuir probe measurements at locations near both the source and target ends of the plasma chamber.
- Optical spectrometer available at target end ( $\lambda=585-685$  nm.)
- Retention obtained by thermal desorption spectroscopy (TDS).

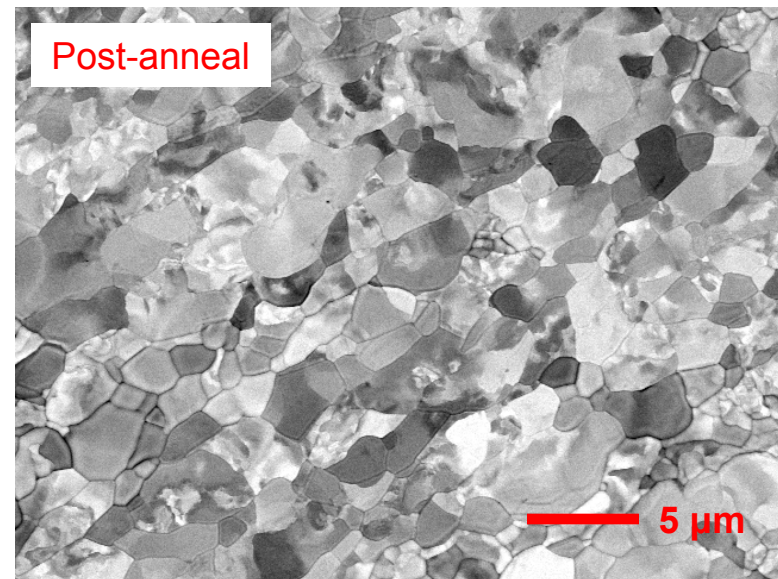
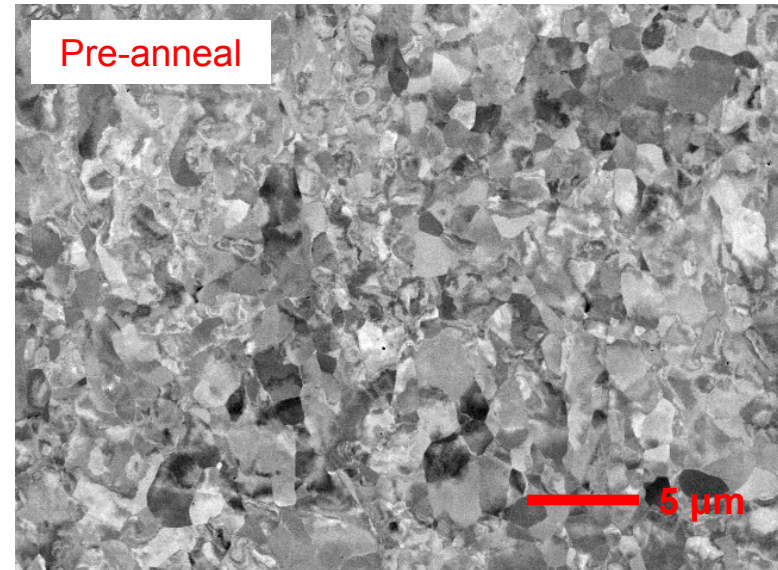
### Discharge Properties:

- Electron temperature = 8 - 15 eV
- Electron density =  $10^{16} - 10^{18} \text{ m}^{-3}$
- Ion Flux =  $10^{20} - 10^{22} \text{ m}^{-2}\text{s}^{-1}$
- Ion Fluence =  $10^{23} - 10^{26} \text{ m}^{-2}$
- Plasma column FWHM = 5 cm



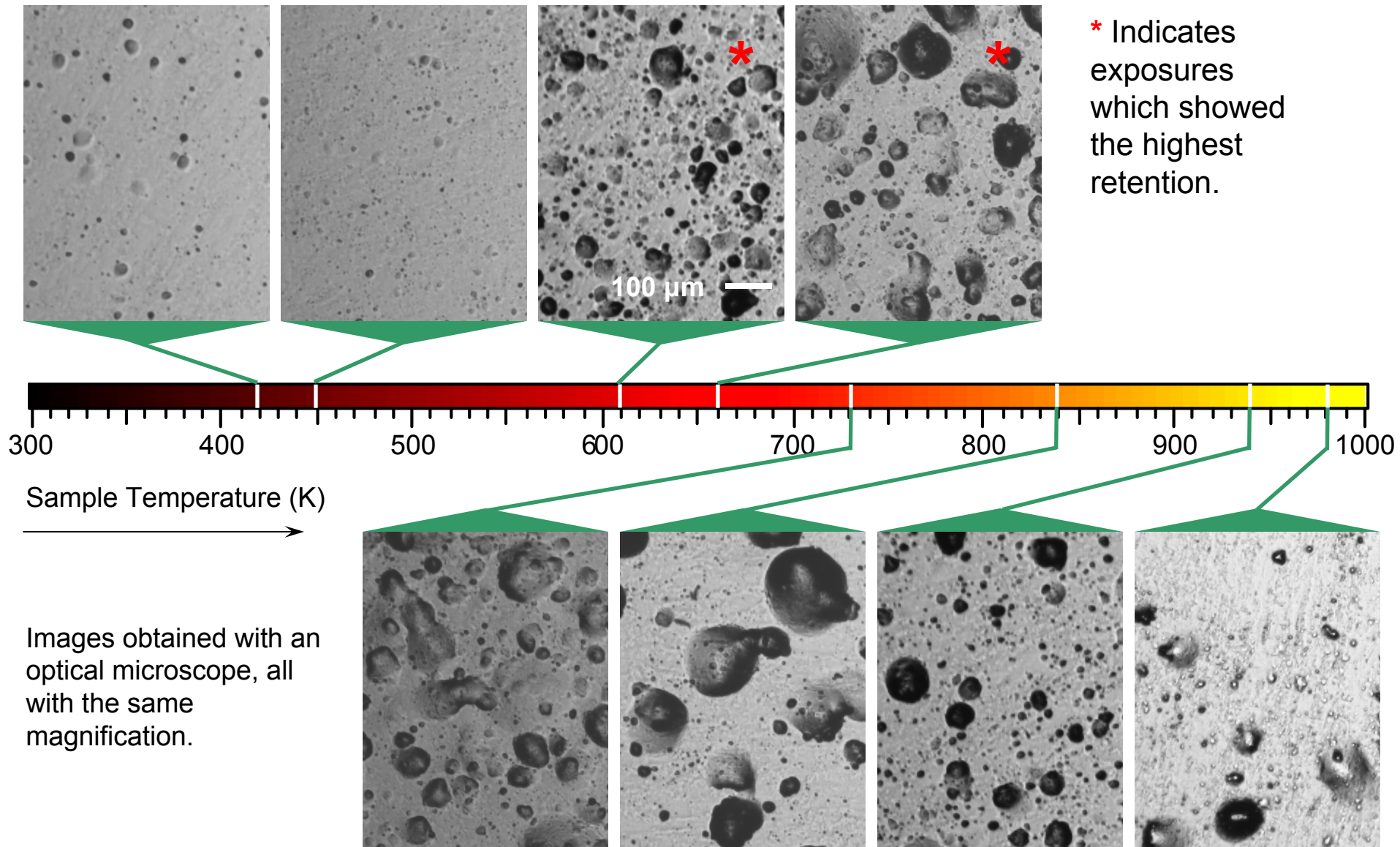
# Sample Preparation / Plasma Exposure Conditions

- Samples used in this study were manufactured by PLANSEE.
  - 99.9999 % purity by weight.
  - Hot rolled into 1 mm thick sheets.
  - Polished to a surface finish of 0.02 microns.
  - Annealed under UHV conditions for 1 hr prior to plasma exposure.
- Plasma bombardment conditions:
  - Ion energy,  $E=70$  eV
  - Flux,  $\Phi=1.1\times10^{22}$  D/m<sup>2</sup>-s
  - Exposure time,  $t=2$ hr
  - Fluence =  $10^{26}$  D/m<sup>2</sup>
  - Sample temperature controlled by modifying heat sink configuration.

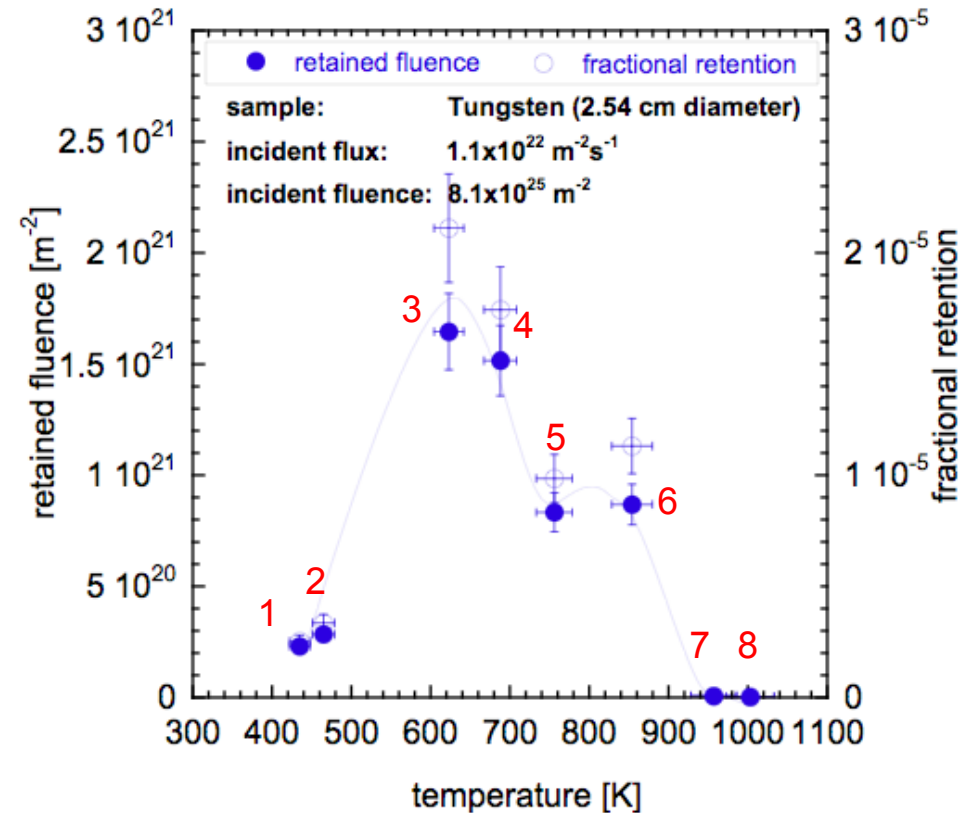
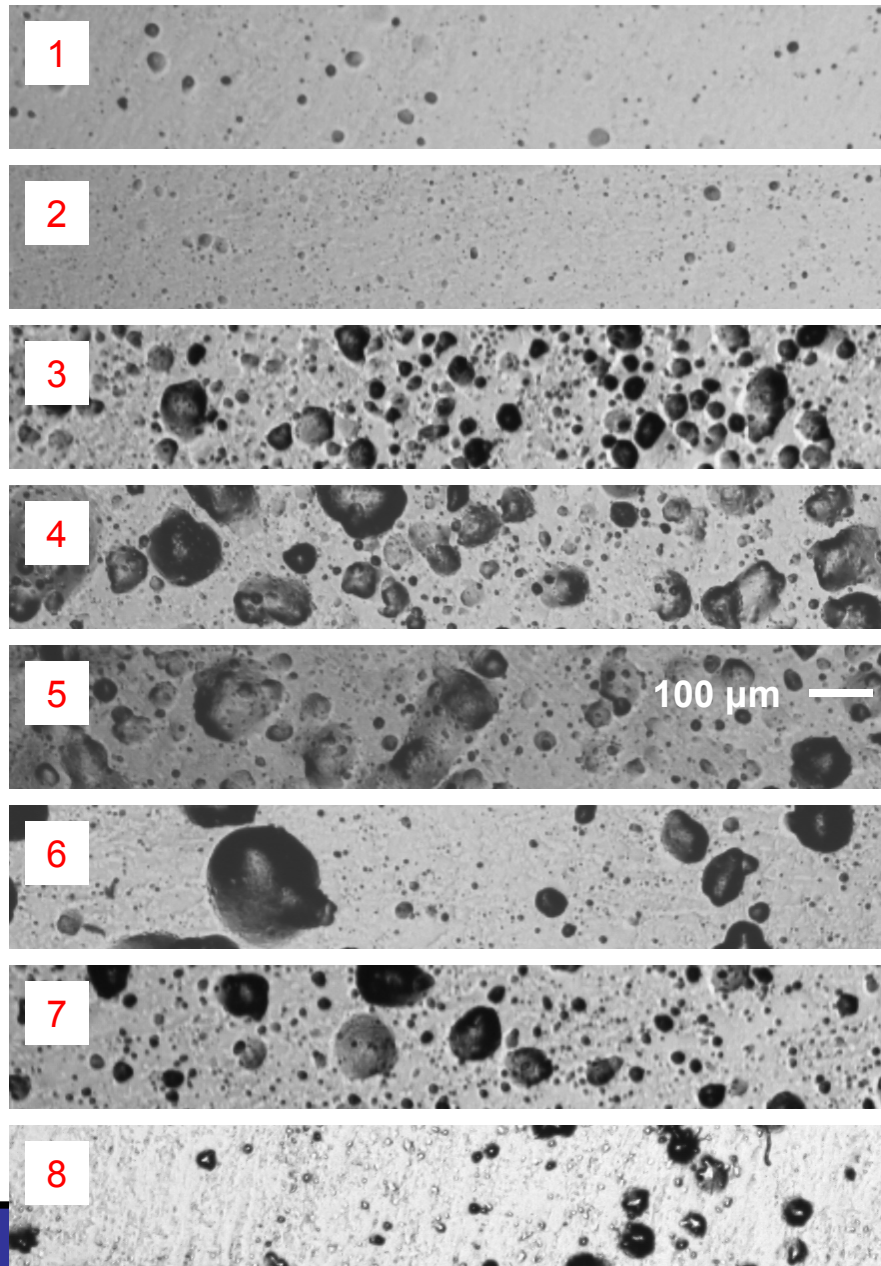




# Blister Size Variation with Sample Temperature



# Correlation with Retained Fluence



All images shown here were obtained using an optical microscope, under the same magnification.

# Detailed Blister Images

Optical Microscope

Pre-TDS

Post-TDS

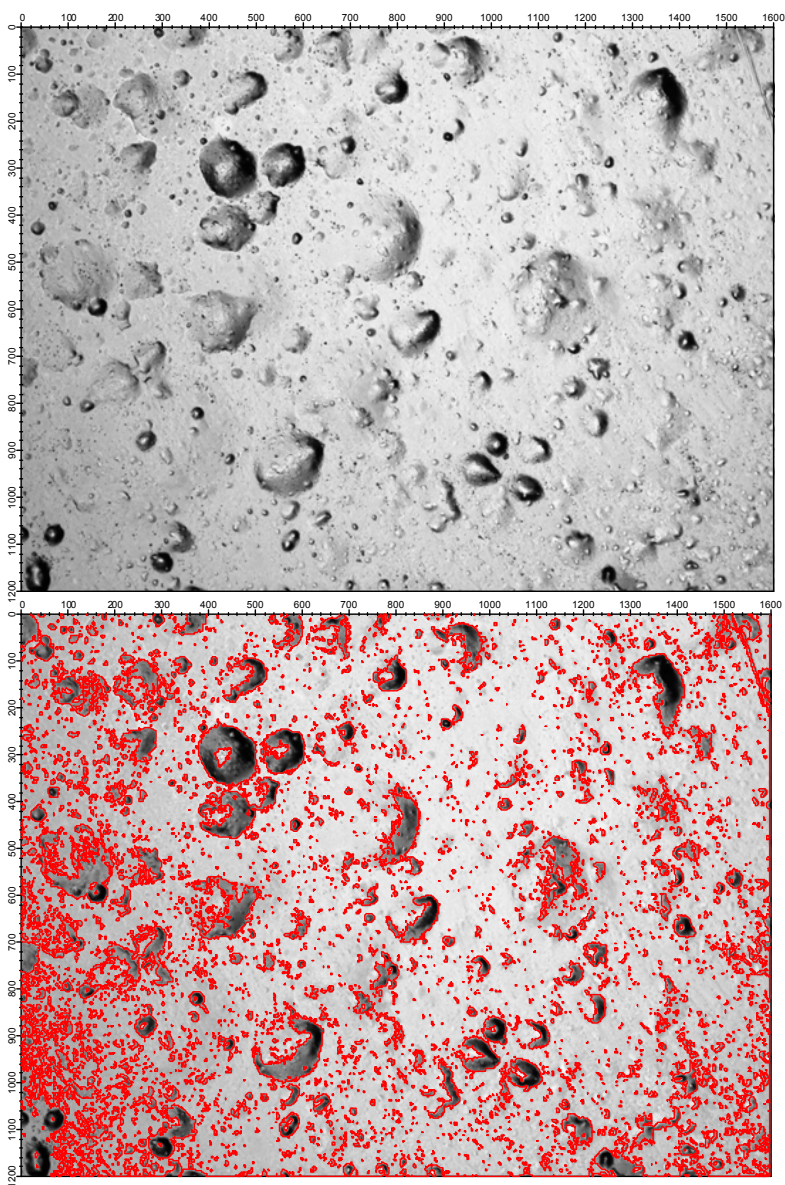
The blisters shown to the left were imaged before and after thermal desorption. There is no conclusive evidence that the blister caps have collapsed, or any other substantial differences emerging due to the desorption process.

SEM Images

Initial fracture of the blister caps appears to occur on the grain boundaries.



# Image Analysis: Blister Diameter Distribution



- **Difficulties with image analysis:**
  - SEM images do not have enough contrast for accurate blister size analysis.
  - Optical images offer better contrast. However, in many cases, light is not reflected from the surface in a consistent manner, making it difficult to discern blister boundaries with automated software (see images to the left.)
  - Alternative: manually outline the location of blister peaks. (Somewhat more subjective.)

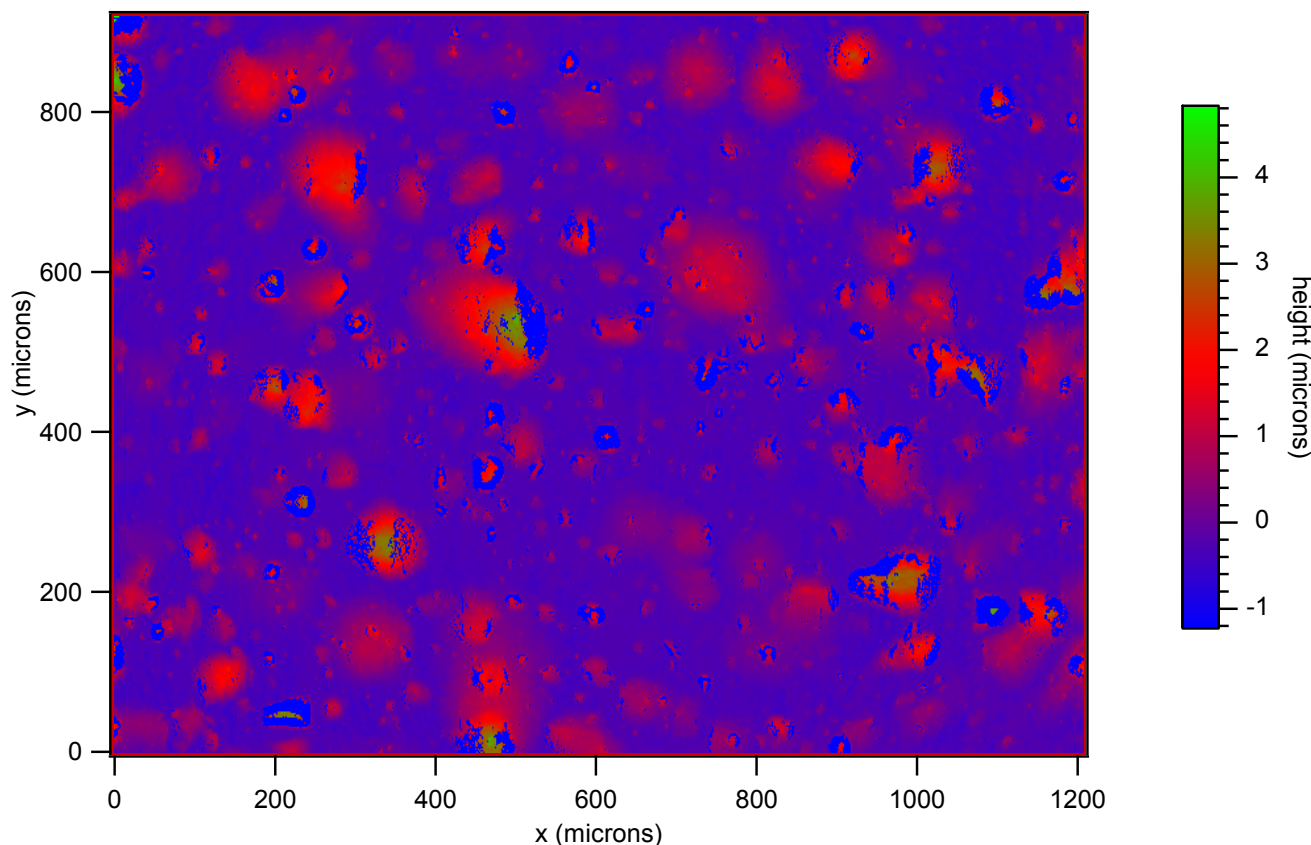


# Blister Height Measurements

- **Laser Profilometry:** A Veeco Wyko laser surface profiler was used to obtain 2-D surface height maps.
  - Light passes through a beam splitter. Reflected light from the sample recombines with the reference beam, producing interference fringes (provides height measurement.)
  - Vertical resolution: 3nm

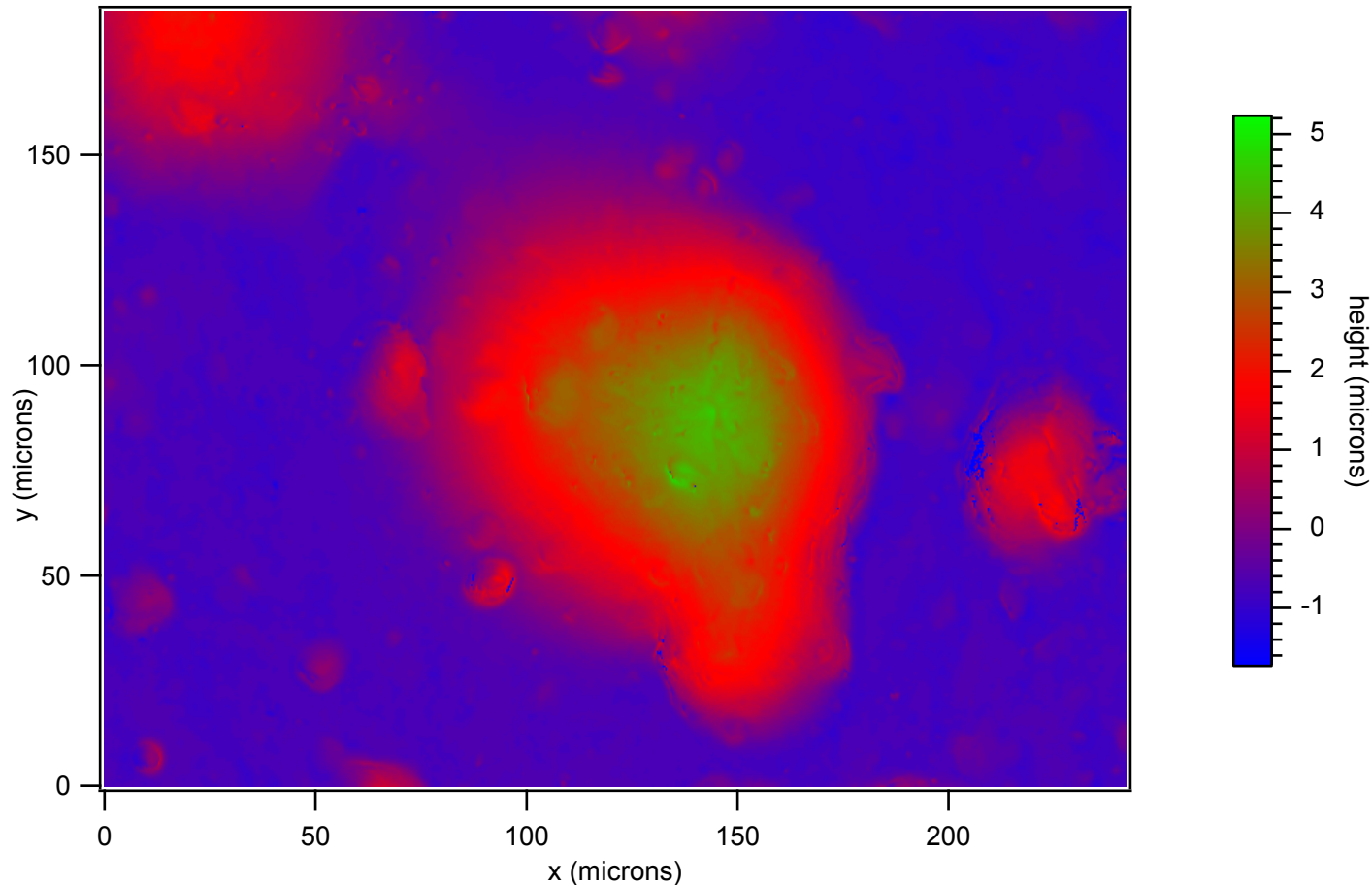
## Plot Data:

- W sample surface after exposure at 660 K.
- A wide range of blister heights is evident, up to 5 microns.
- Dark blue regions indicate areas where the surface height gradient was too steep to obtain an accurate measurement of the fringe pattern.

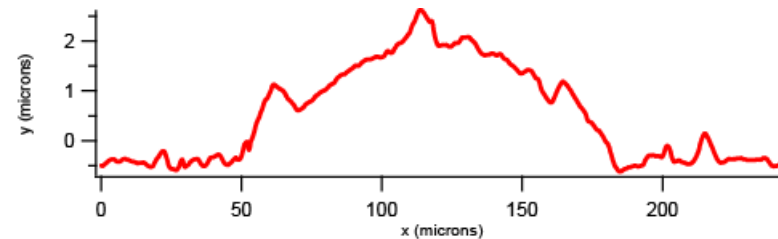
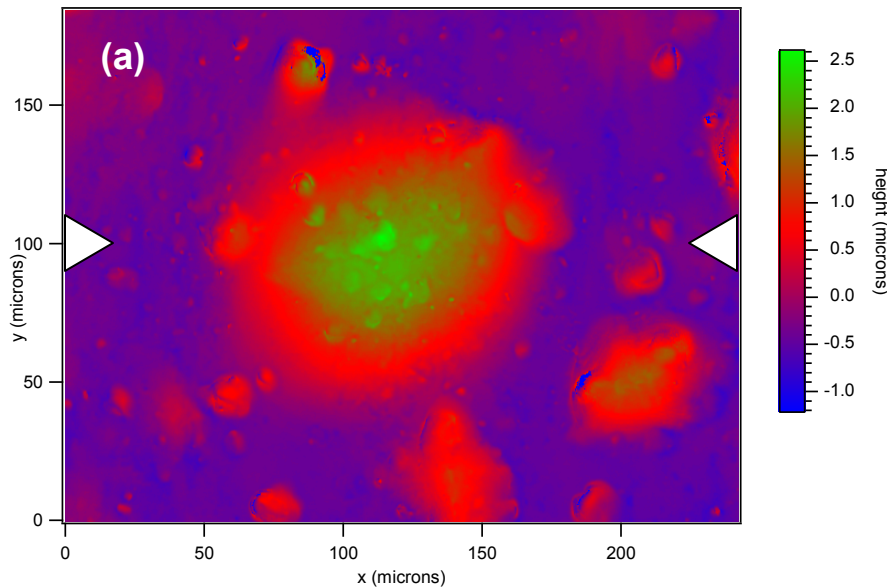


# Profilometry Results

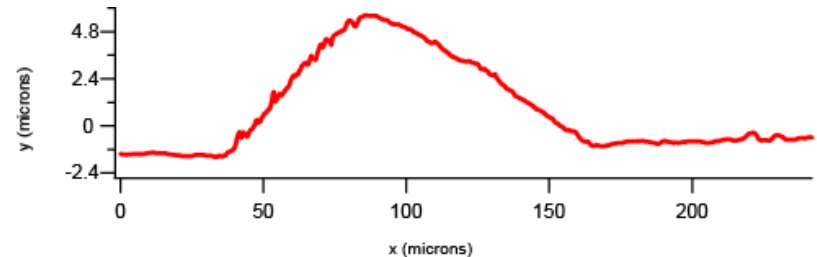
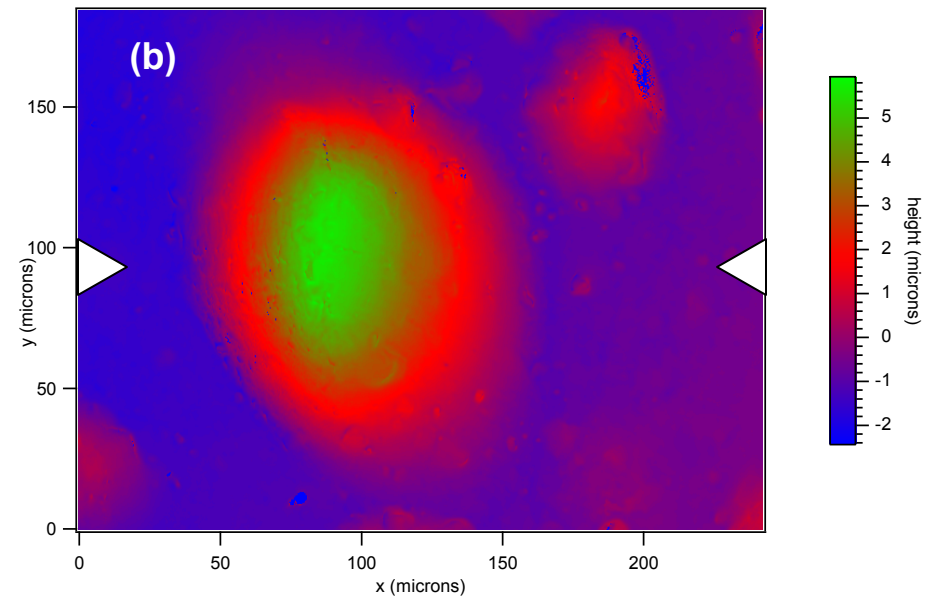
- **Profilometry Advantages:** In addition to detailed height information, laser profilometry has the potential to allow for much more effective analysis of blister sizes. It eliminates many of the difficulties associated with processing optical images of surfaces (where light may be reflected in different directions away from the imaging device.)



# Profilometry Results



Case (a) shows smaller blisters forming on the blister cap. Arrows indicate the location of horizontal profile shown below.



Case (b) shows one of the largest observed blisters on this sample (height of over 5 microns.)



# Concluding Remarks

- In this series of experiments, we see a strong dependence on blister size as a function of temperature.
  - $350\text{ K} \leq T \leq 450\text{ K} \rightarrow$  small blisters ( $< 10$  microns)
  - $600\text{ K} \leq T \leq 800\text{ K} \rightarrow$  high blister density, large blisters (up to  $\sim 100$  microns)
  - $T > 900\text{ K} \rightarrow$  decreasing blister density and size ( $\sim 50$  microns)
- Highest blister density seems to correspond well to conditions which produce high blister density.
- Several blisters were noticed to have ruptured (small cracks near grain boundaries.)
- Laser profilimetry techniques, if sufficiently refined, can provide a great deal of information regarding blister sizes (and allow for more unambiguous interpretation than optical images.)

# Acknowledgements

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