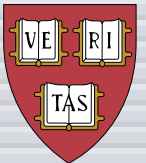
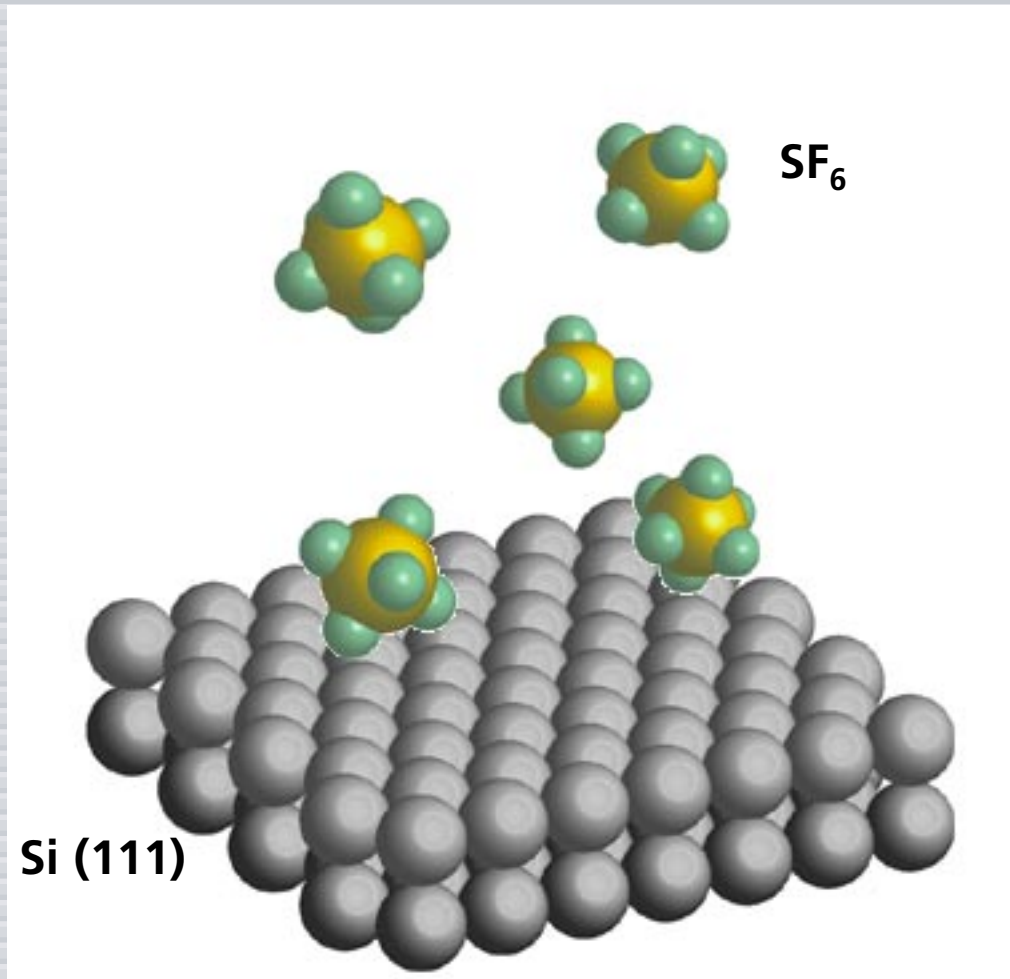


# **Femtosecond laser-assisted microstructuring of silicon surfaces for novel detector, sensing, and display technologies**

**Jim Carey  
Tsing-Hua Her  
Mike Sheehy  
Claudia Wu  
Rebecca Younkin  
Catherine Crouch  
Meng Yan Shen  
Li Zhao**

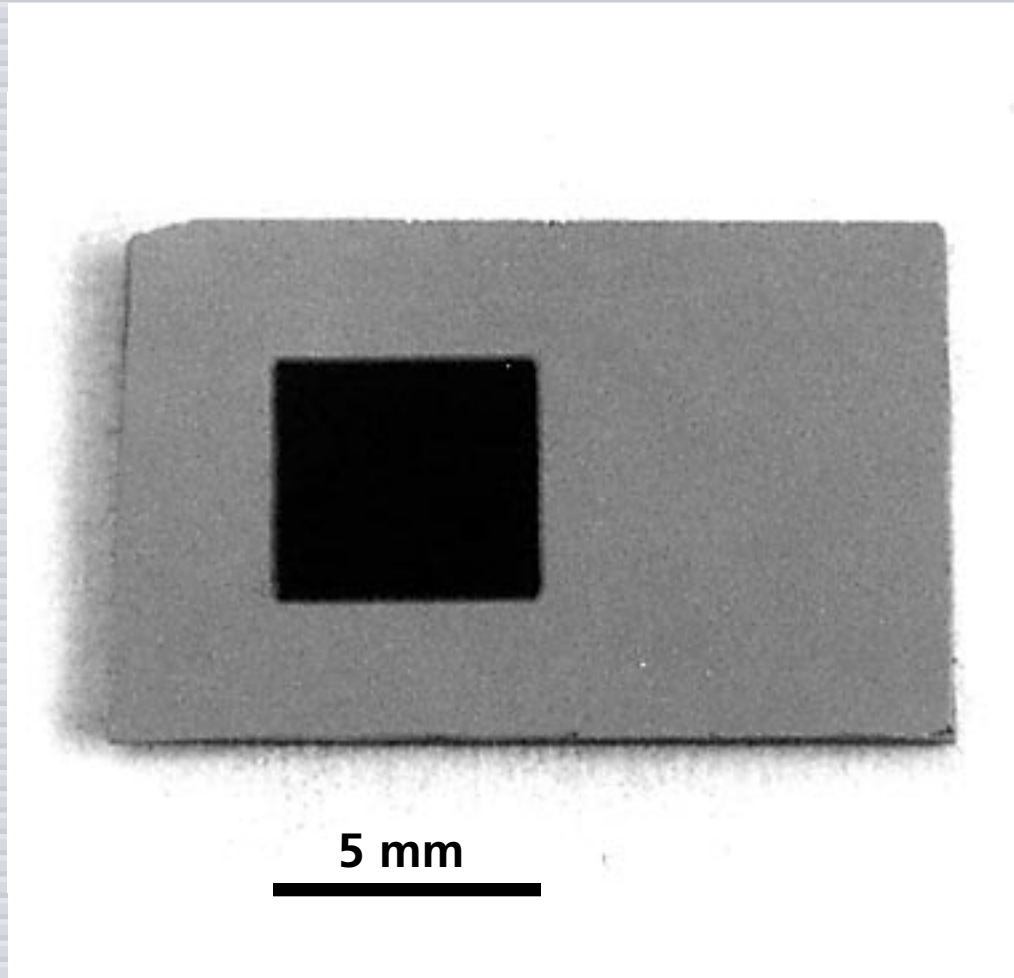


# Introduction



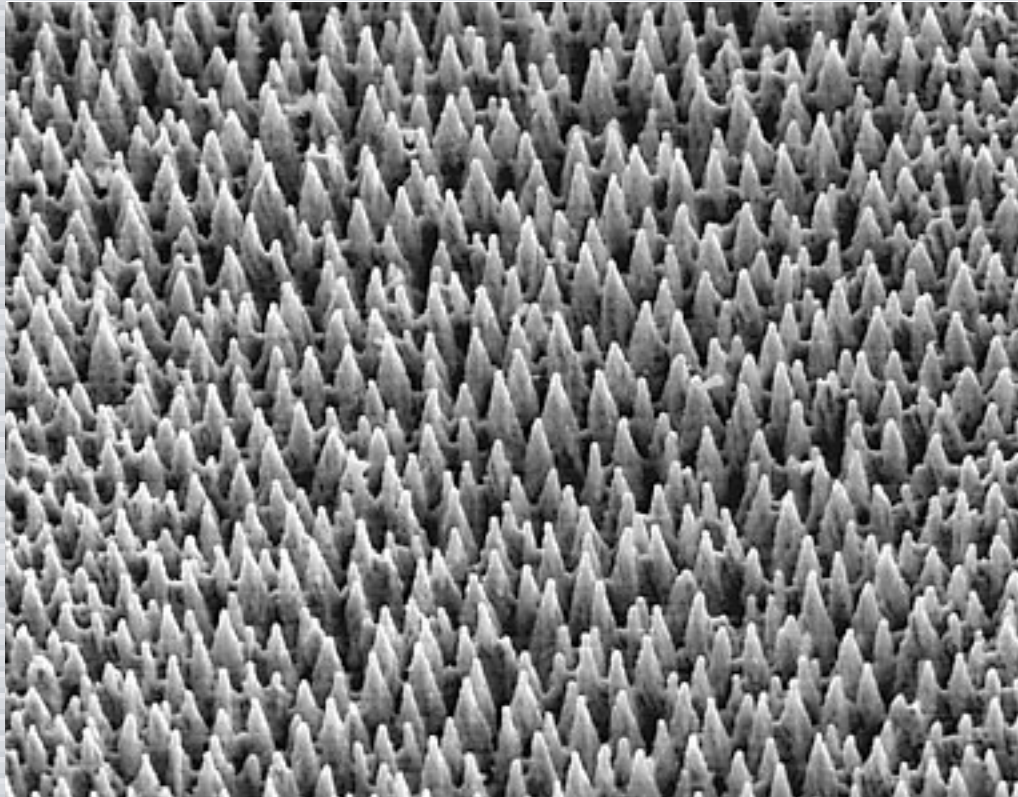
irradiate with 100-fs 10 kJ/m<sup>2</sup> pulses

# *Introduction*



**"black silicon"**

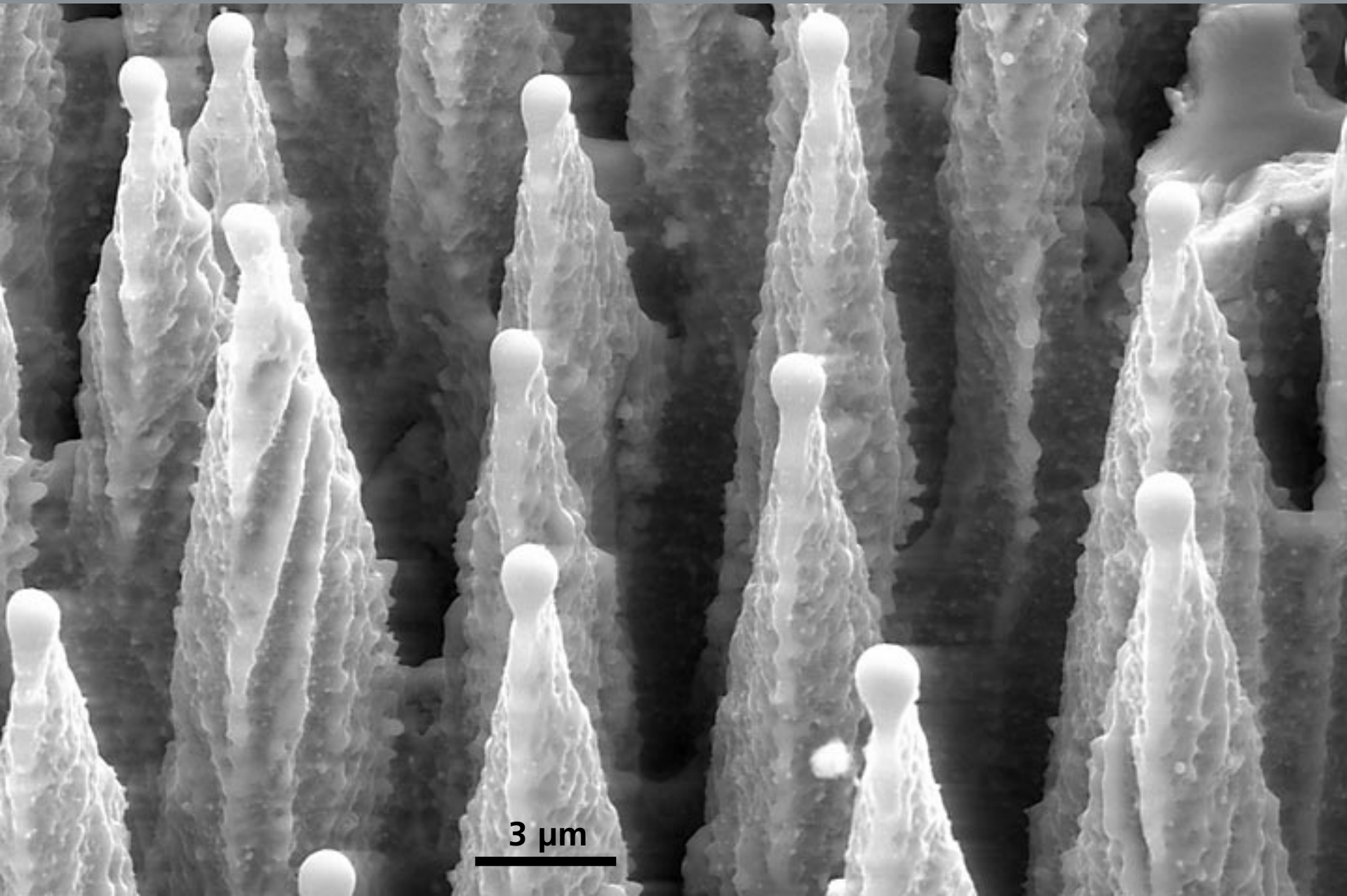
# *Introduction*



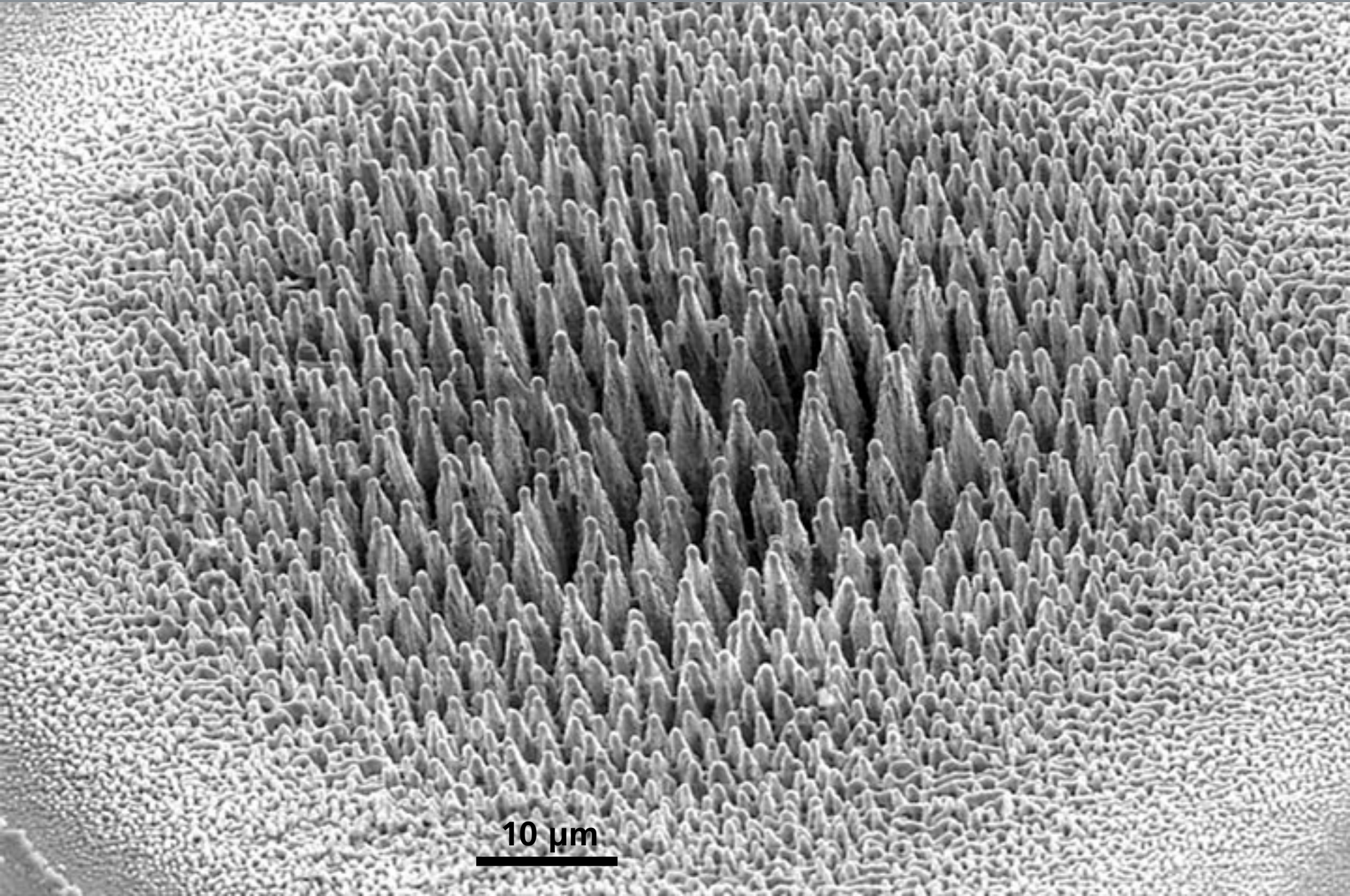
**20  $\mu\text{m}$**



# *Introduction*



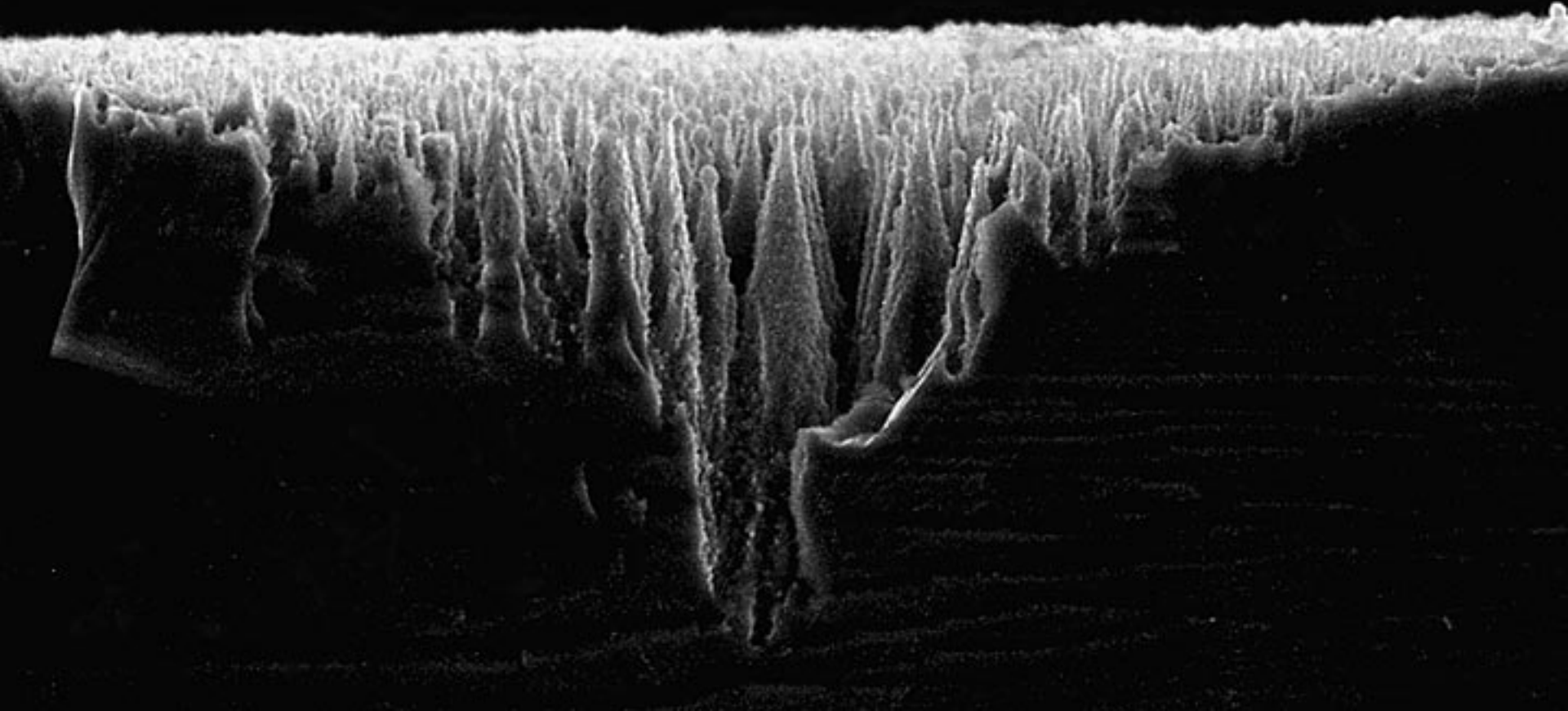
# *Introduction*



10  $\mu\text{m}$



# *Introduction*



# *Introduction*

# *Introduction*

- ▶ **maskless etching process**
- ▶ **self-organized, tall, sharp structures**
- ▶ **nanoscale structure on spikes**

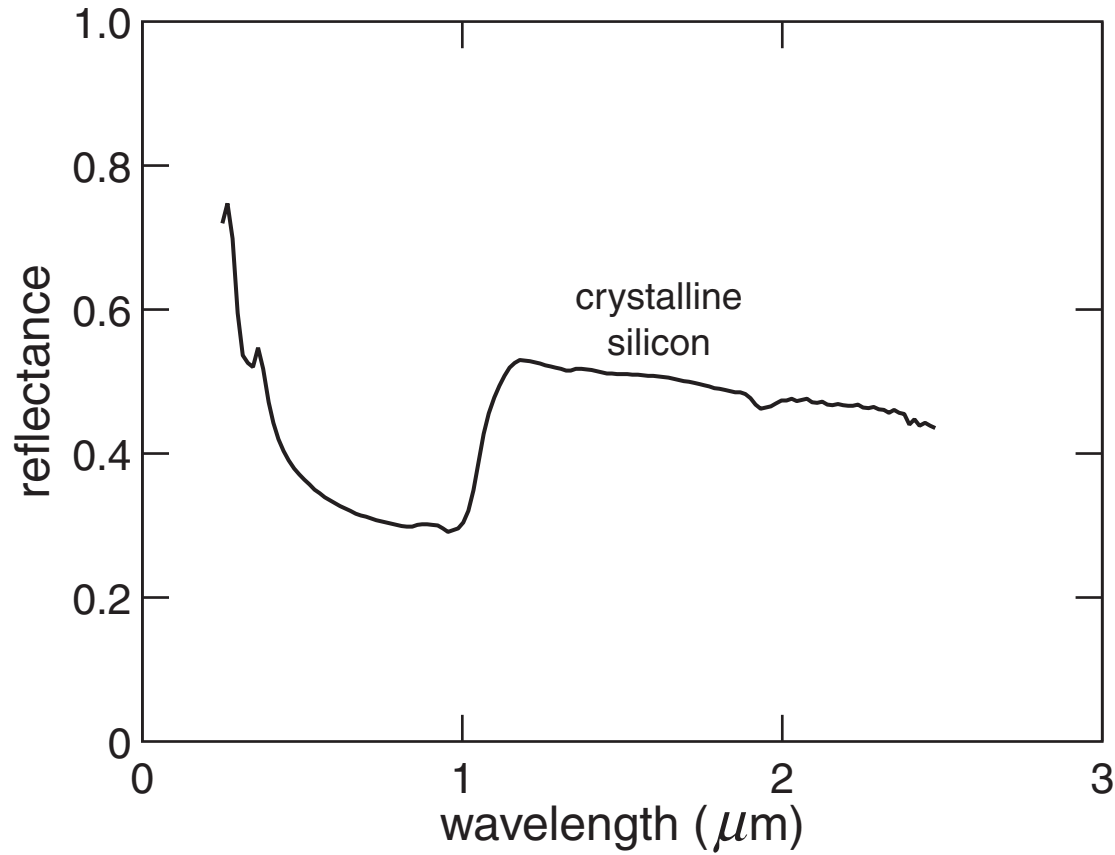


# Outline

- ▶ **Properties**
- ▶ **Structural and chemical analysis**
- ▶ **Outlook**

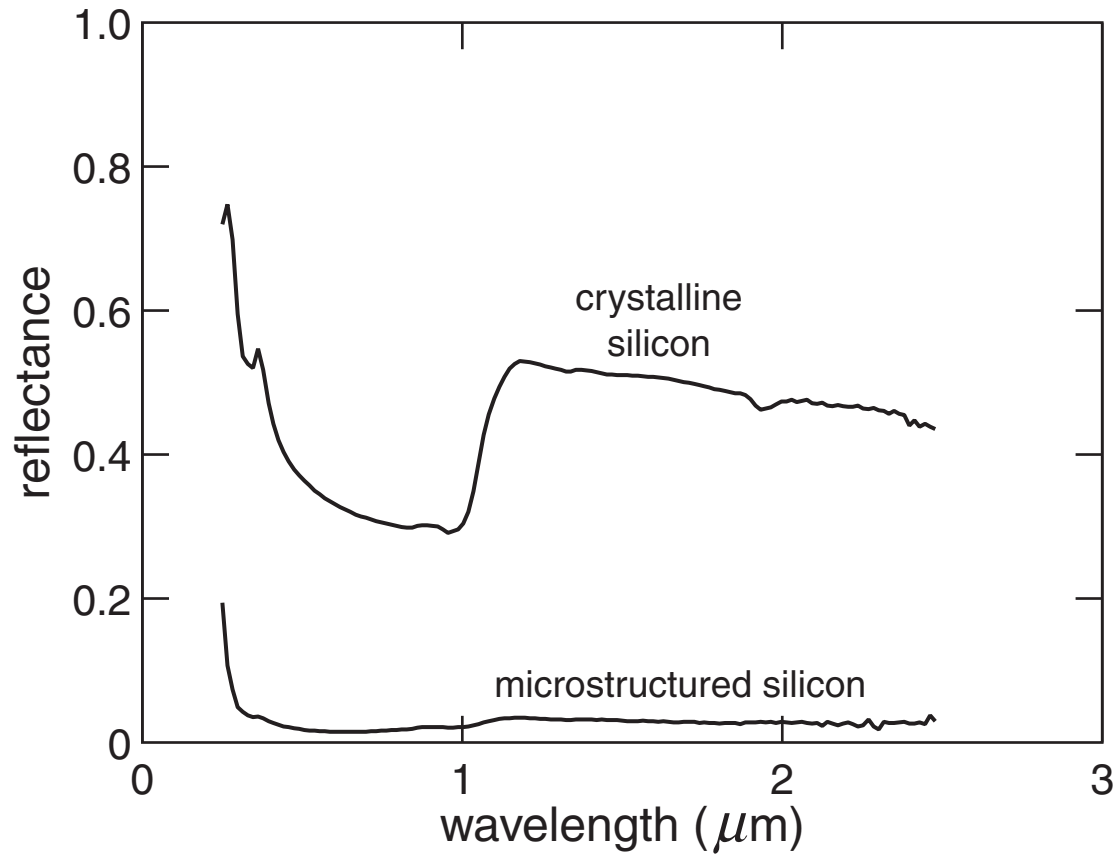
# *Properties*

## reflectance (integrating sphere)



# Properties

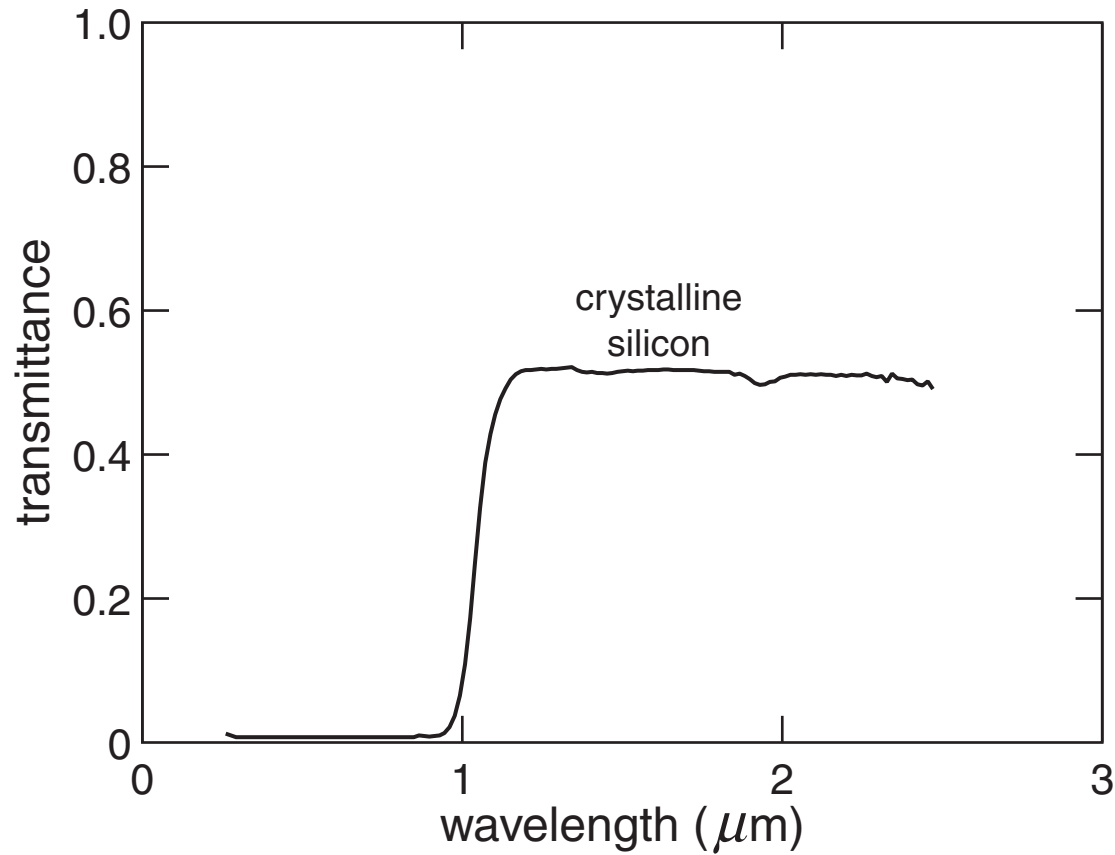
## reflectance (integrating sphere)





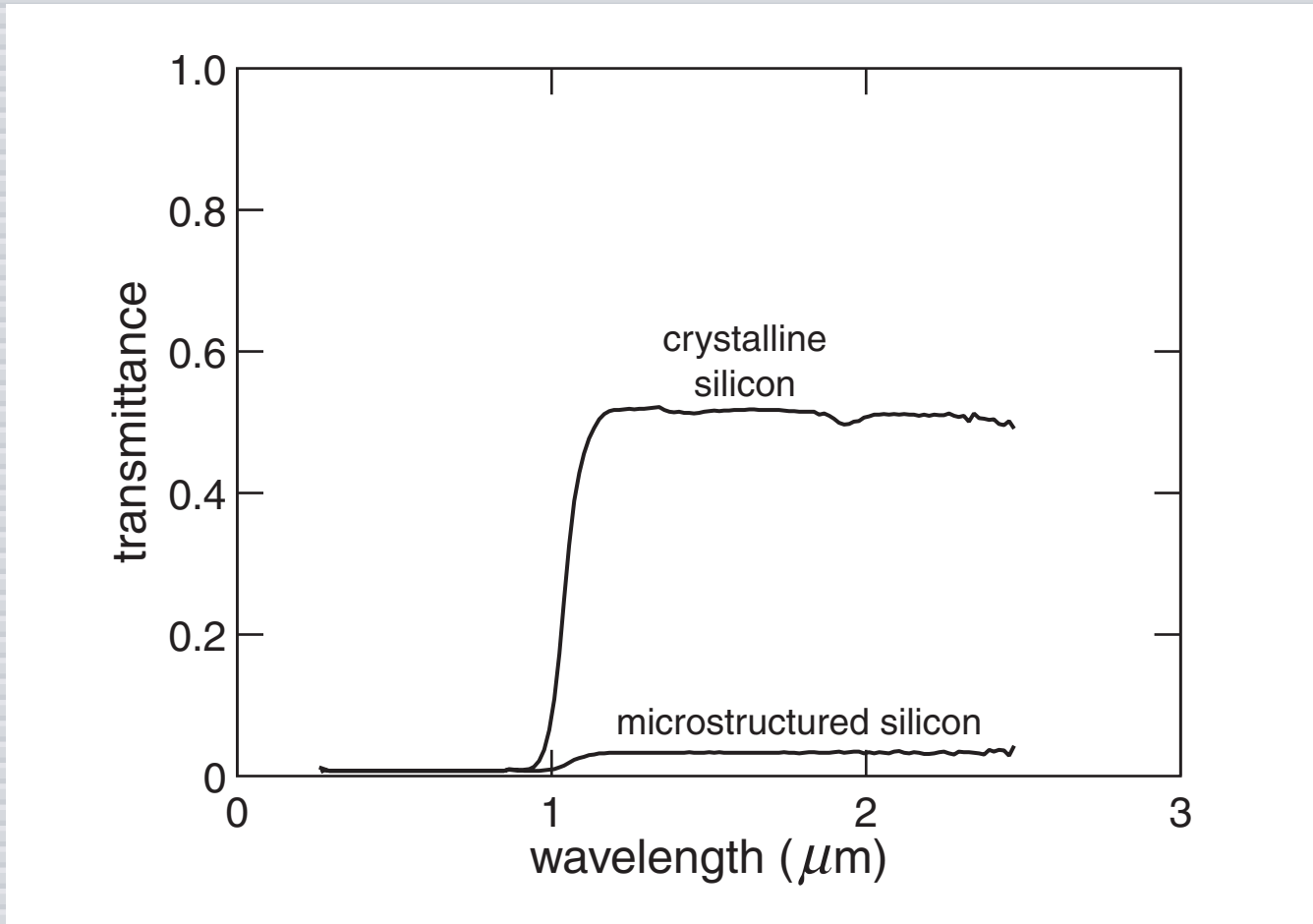
# Properties

## transmittance (integrating sphere)



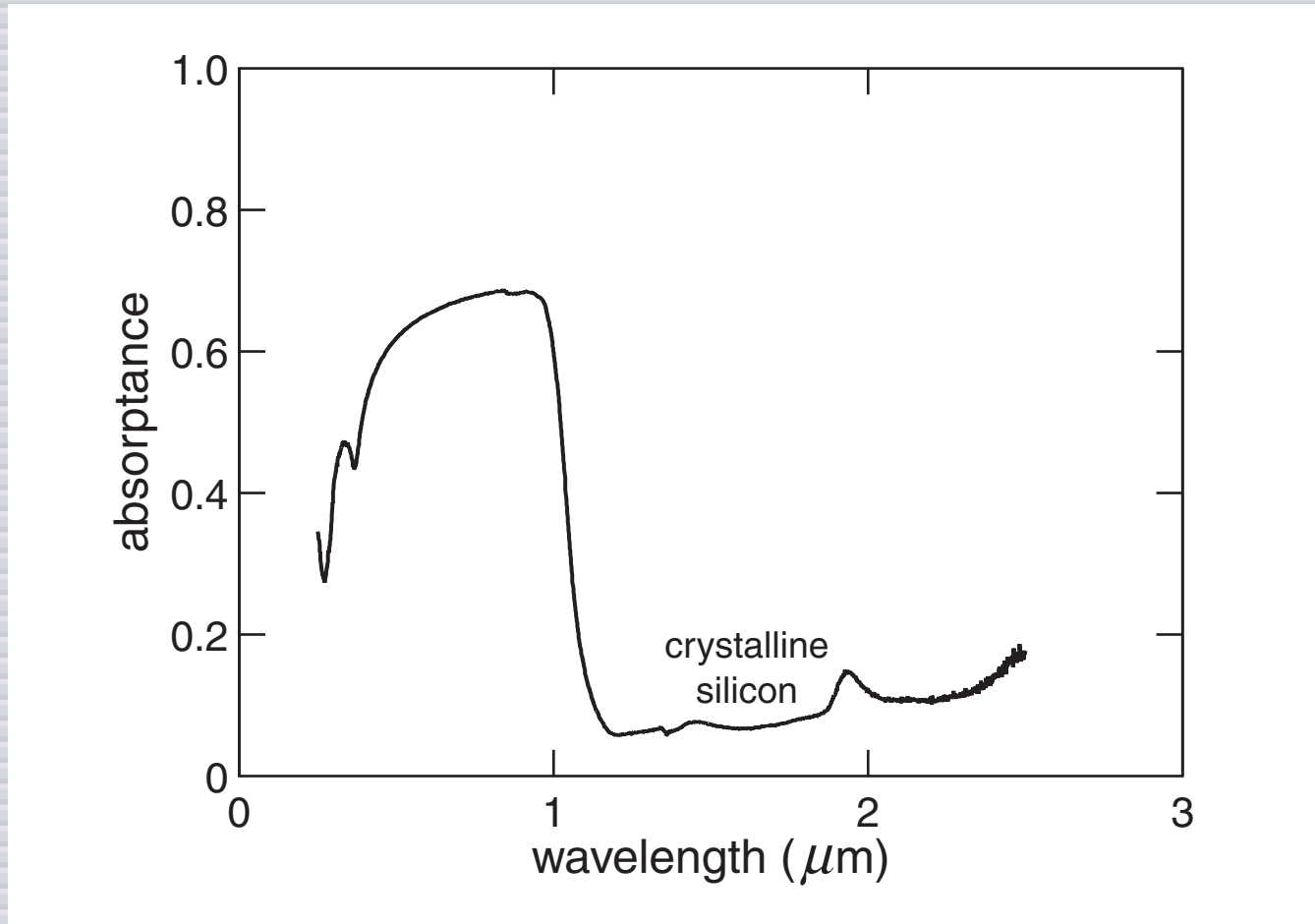
# Properties

## transmittance (integrating sphere)



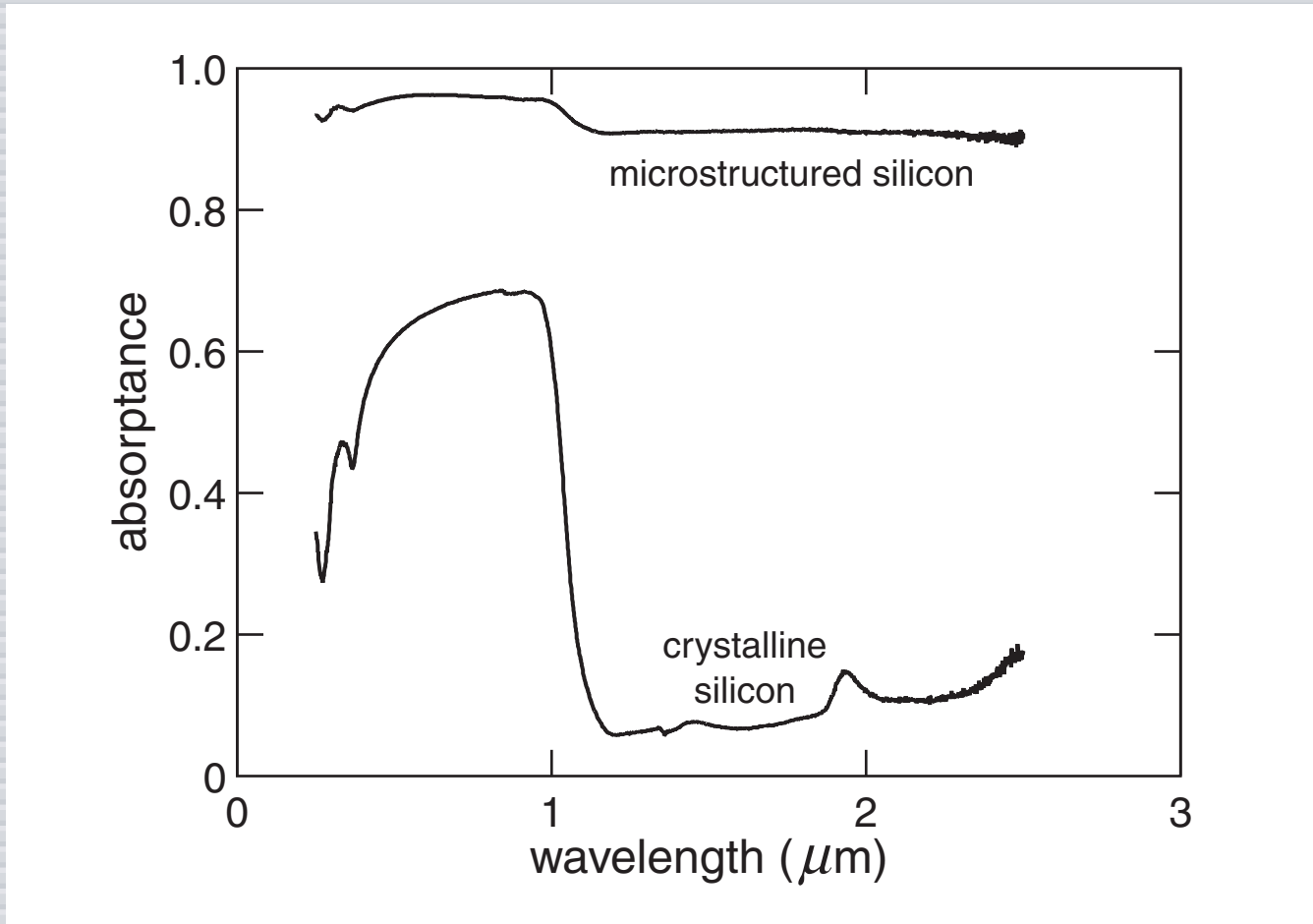
# Properties

**absorptance ( $1 - R - T$ )**



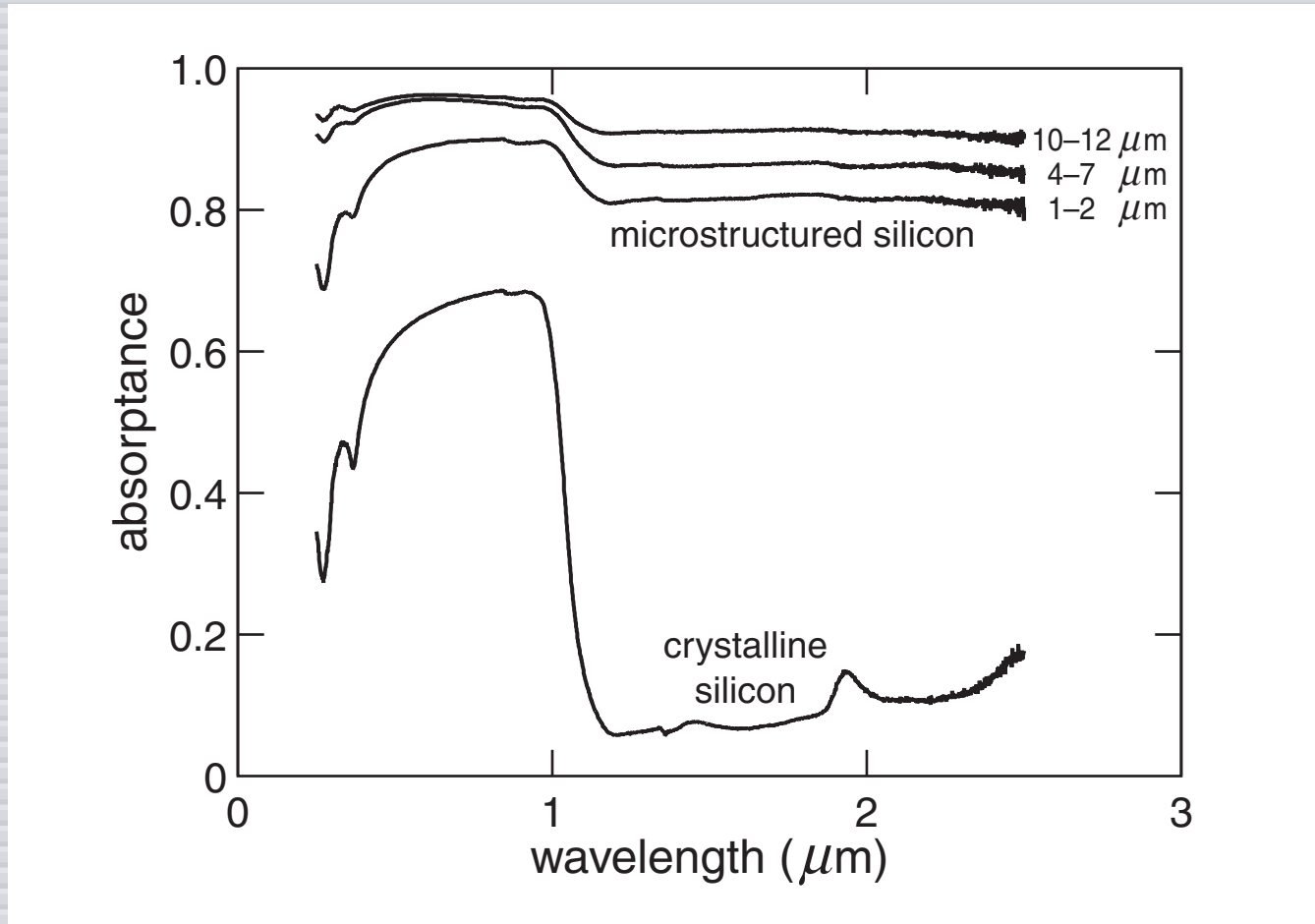
# Properties

absorptance ( $1 - R - T$ )



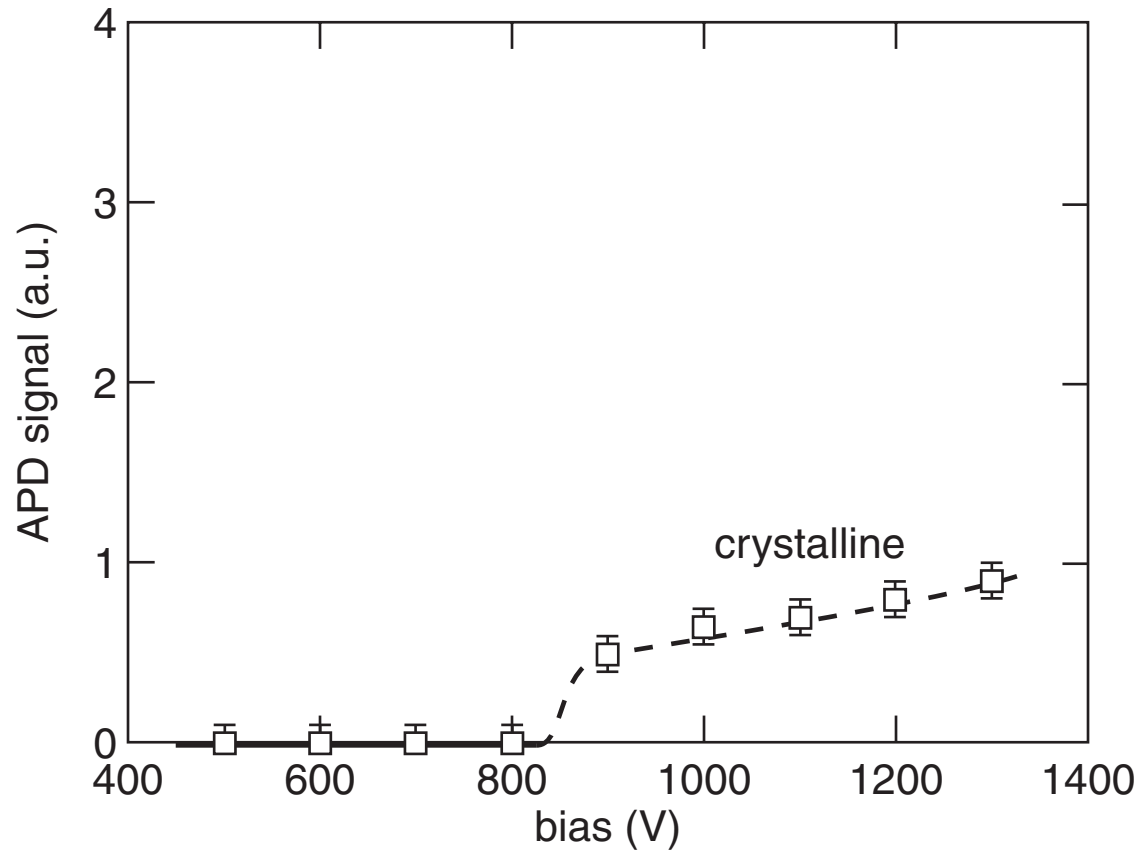
# Properties

absorptance ( $1 - R - T$ )



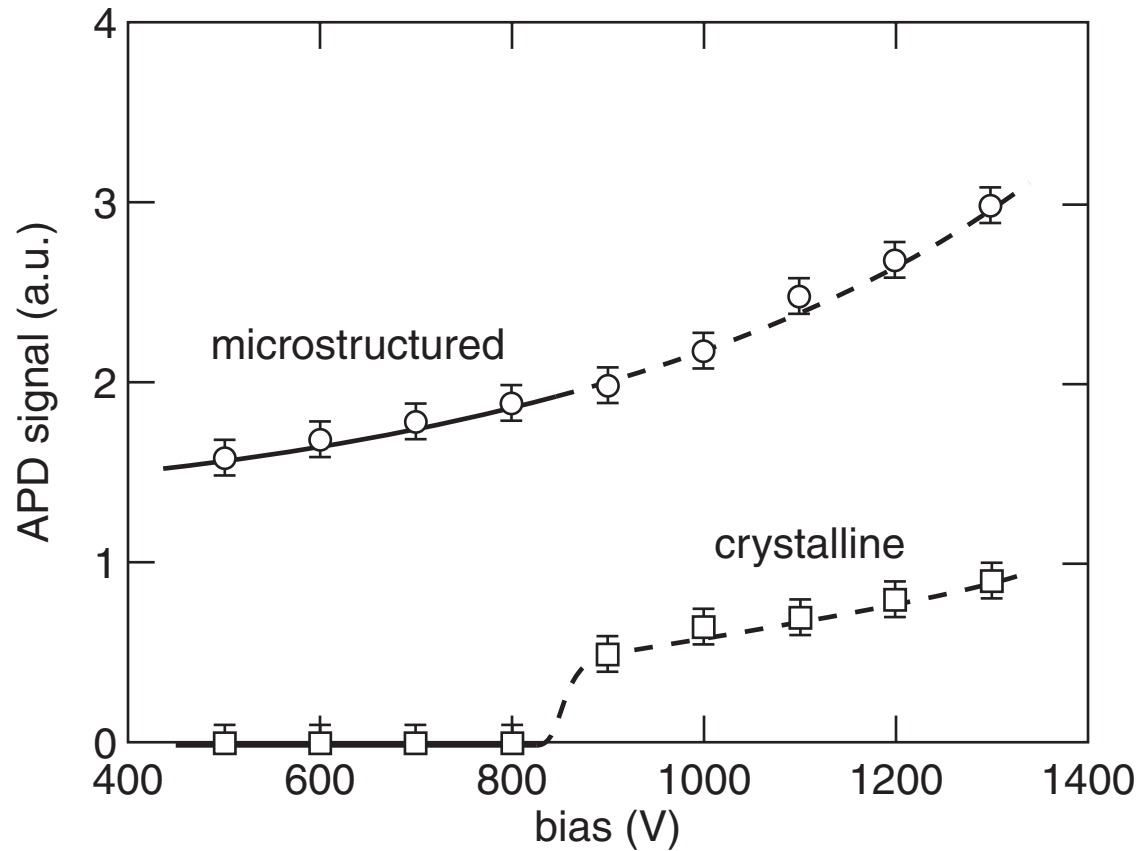
# Properties

## avalanche photodiode response at $1.3 \mu\text{m}$



# Properties

## avalanche photodiode response at 1.3 $\mu\text{m}$



# *Properties*

## **Points to keep in mind:**

- ▶ **near unity absorption**
- ▶ **sub-band gap absorption**
- ▶ **IR photoelectron generation**



# *Properties*

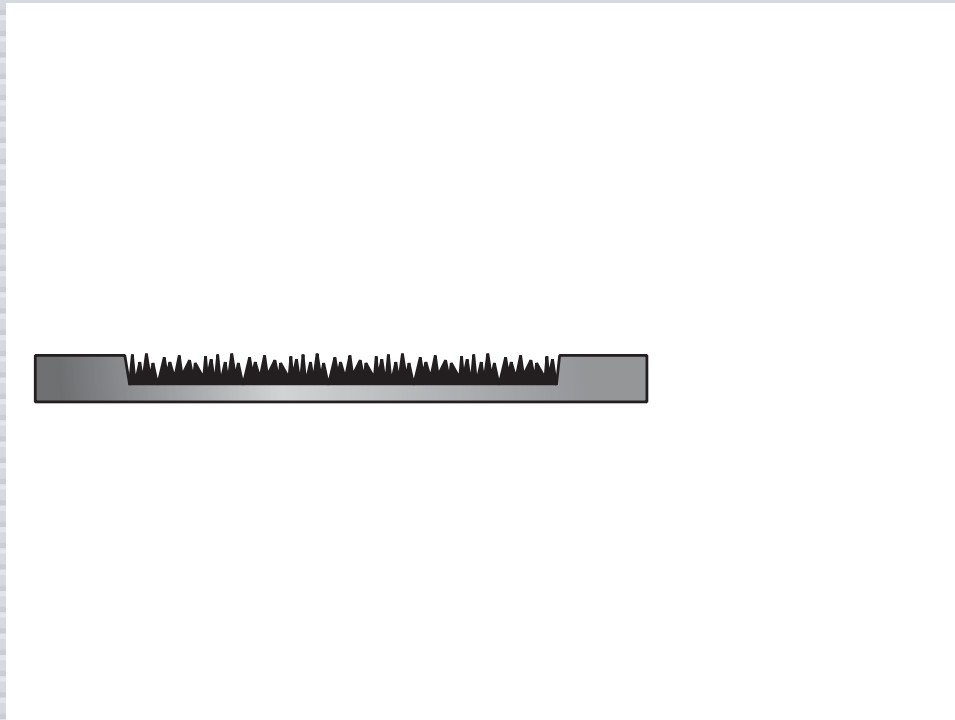
**Points to keep in mind:**

- ▶ **near unity absorption**
- ▶ **sub-band gap absorption**
- ▶ **IR photoelectron generation**

**can spikes be used as field emitters?**

# *Properties*

## **field emission setup**



# *Properties*

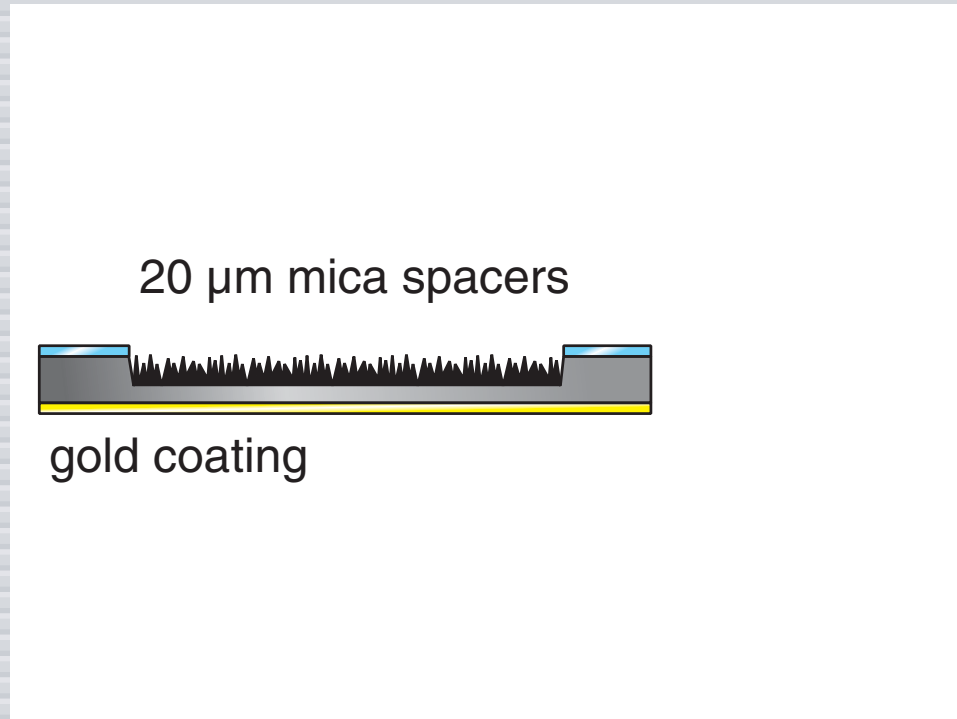
## **field emission setup**



gold coating

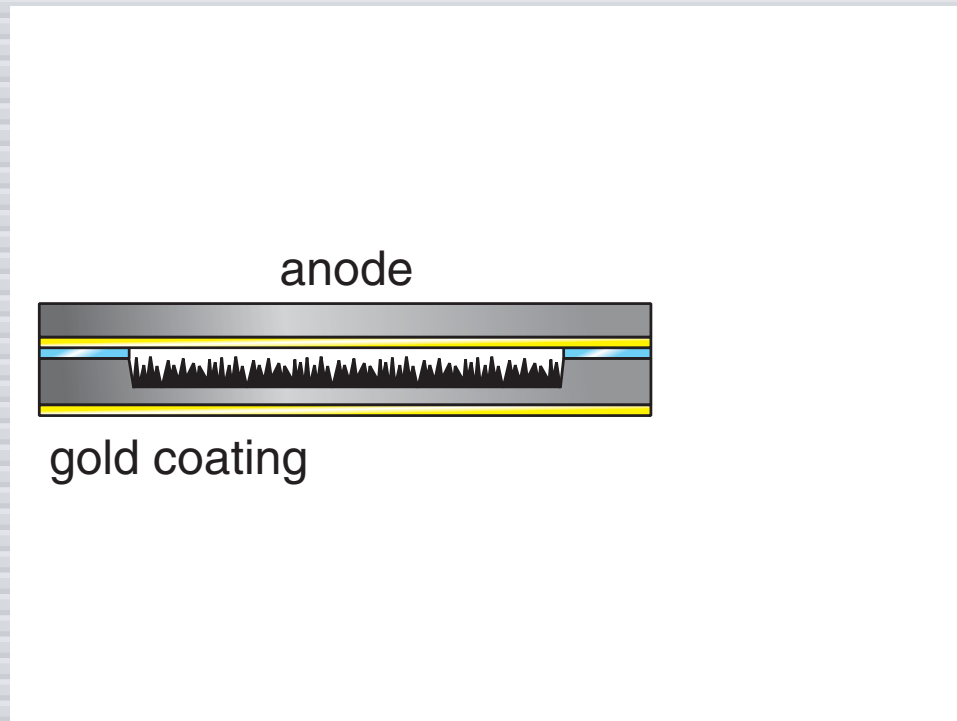
# *Properties*

## field emission setup



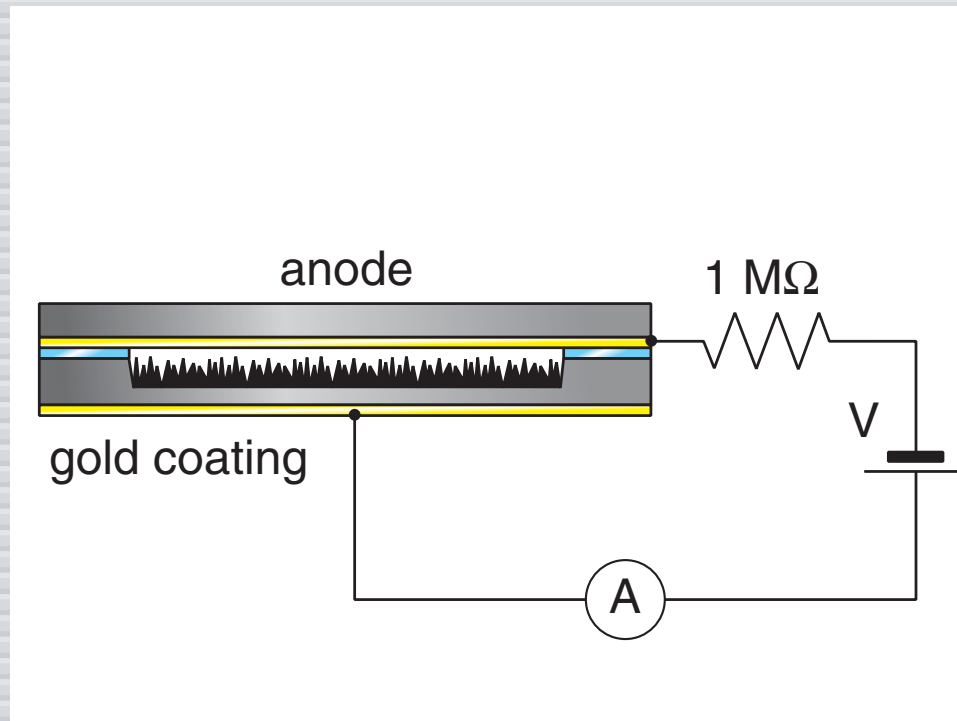
# *Properties*

## field emission setup

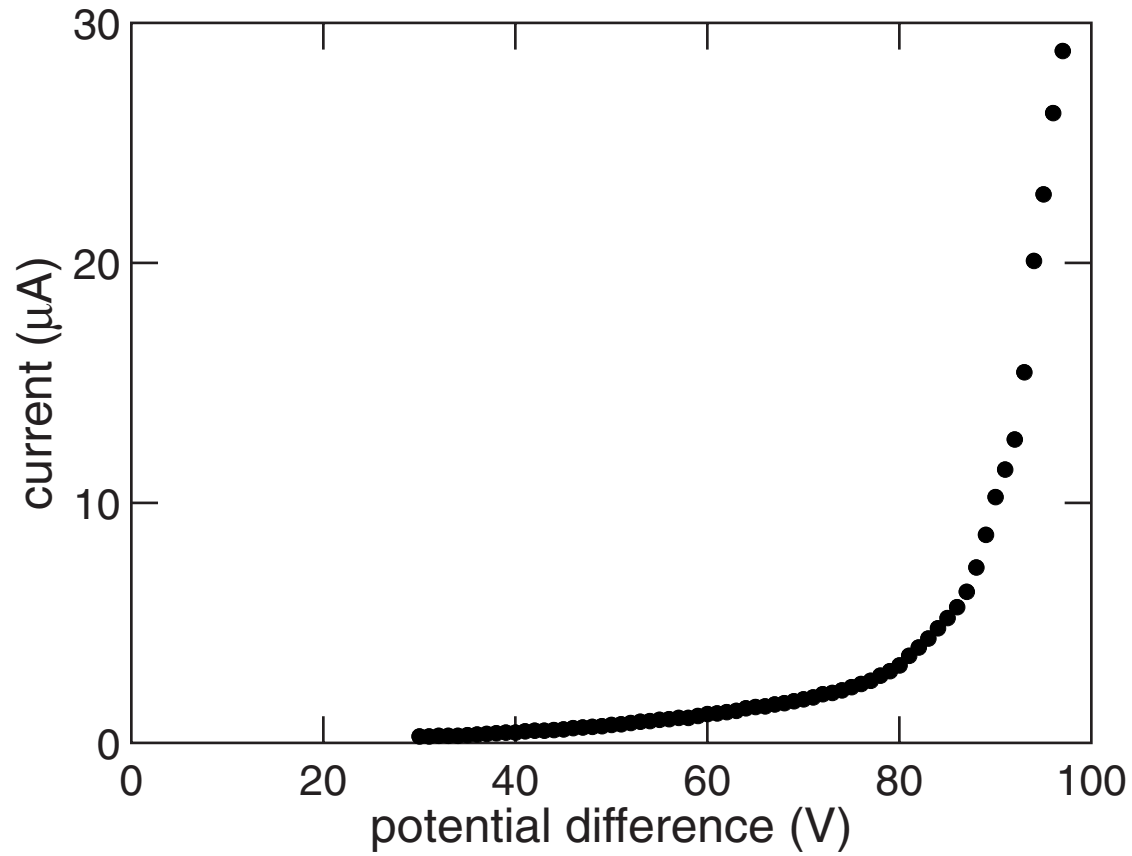


# *Properties*

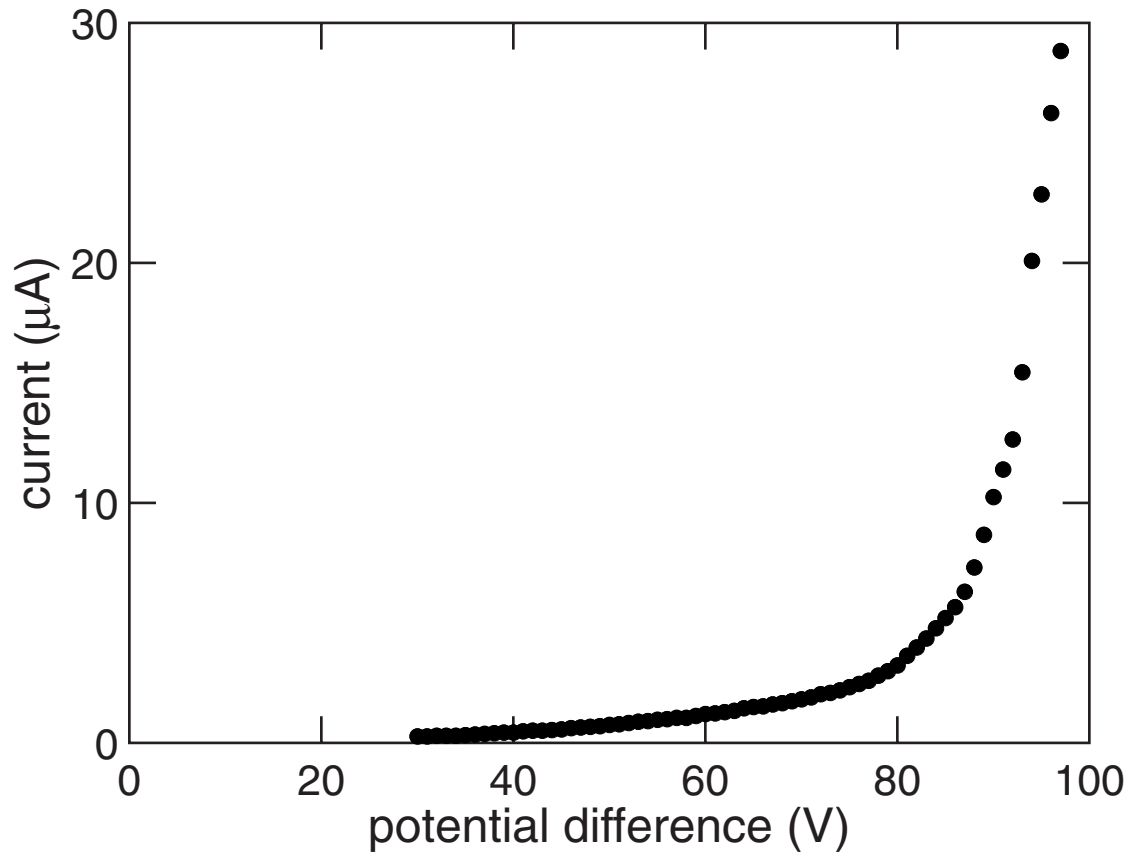
## field emission setup



# Properties



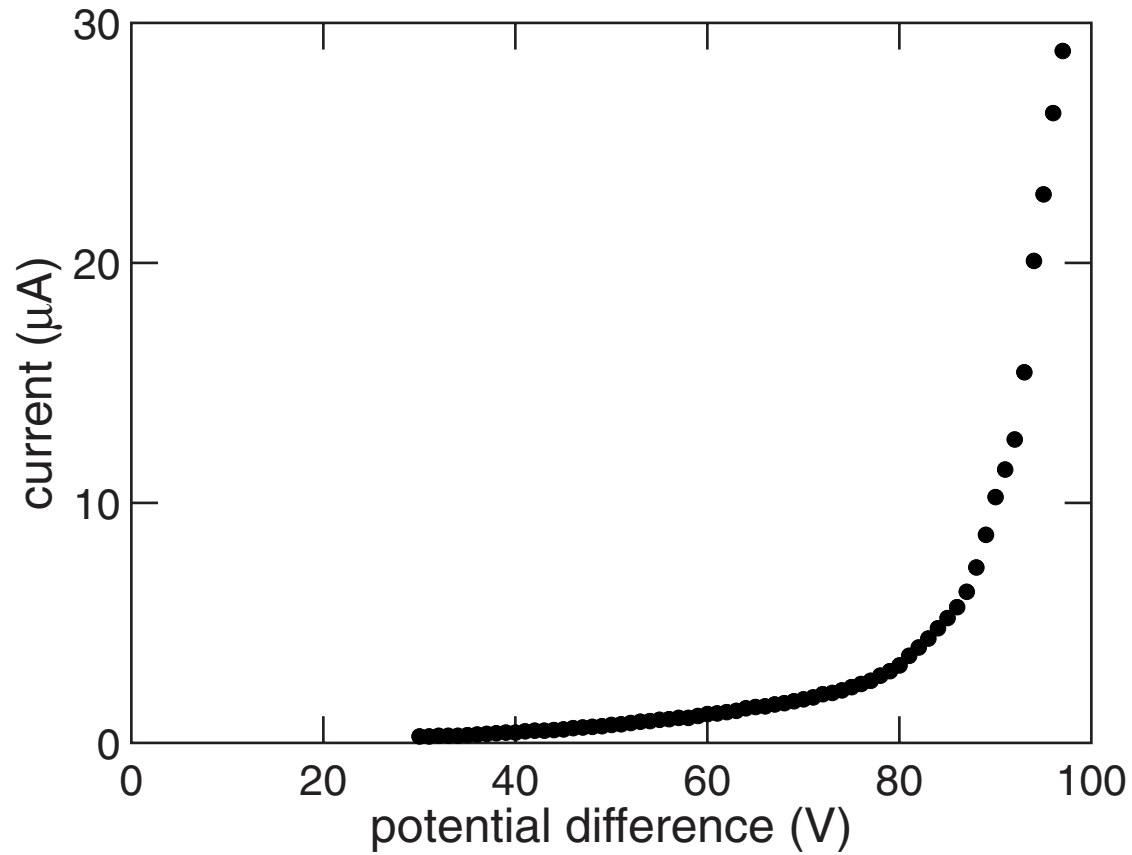
# Properties



**turn-on field ( $1 \mu\text{A}/\text{cm}^2$ ):  $1.2 \text{ V}/\mu\text{m}$**

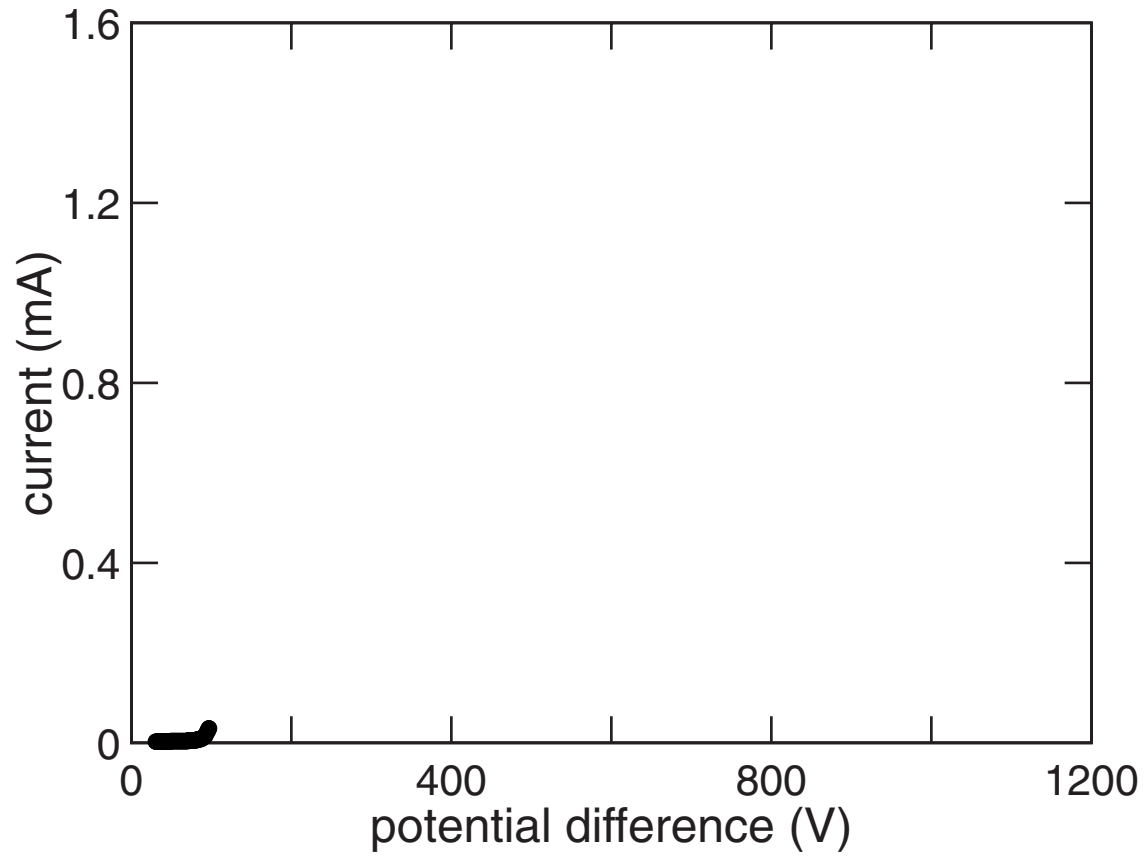


# Properties

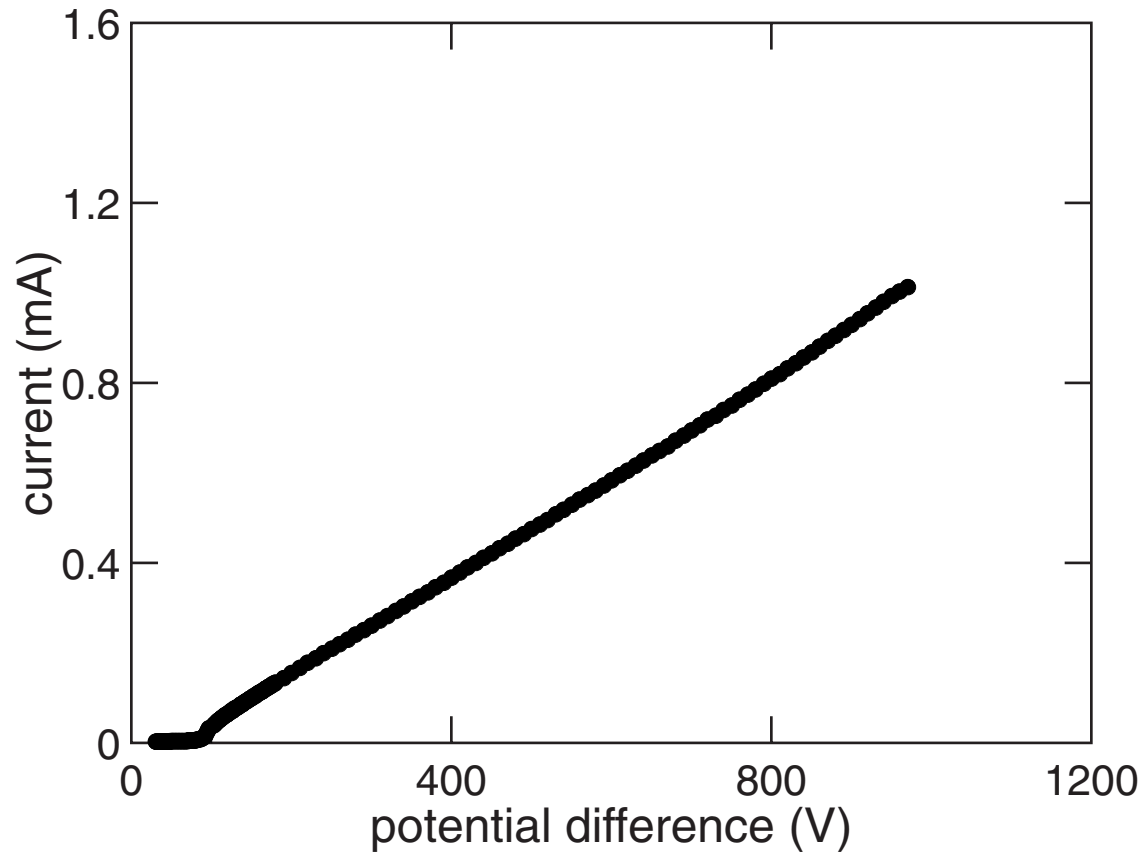


**threshold field ( $10 \mu\text{A}/\text{cm}^2$ ):  $2.1 \text{ V}/\mu\text{m}$**

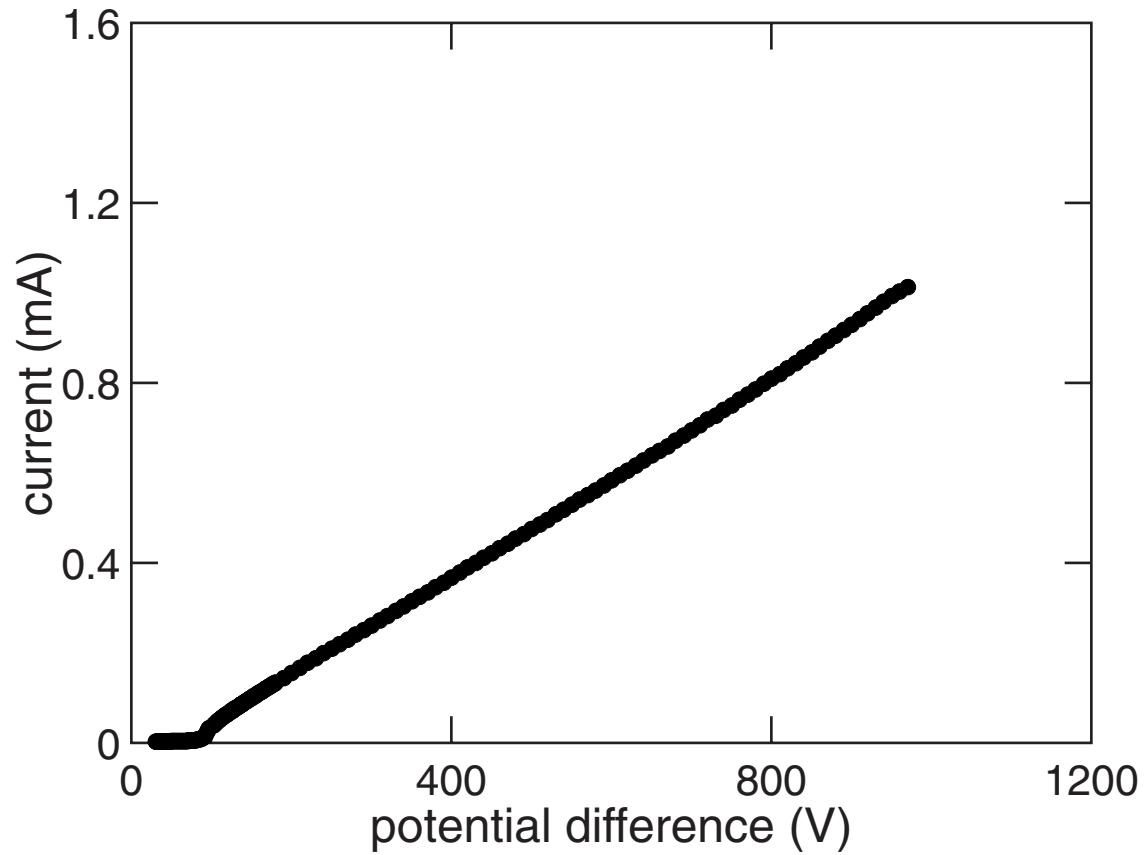
# Properties



# Properties



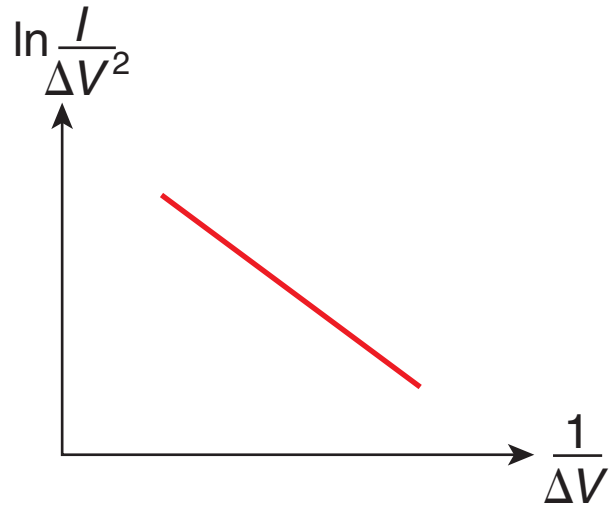
# Properties



**maximum current: 20 mA (4 mm<sup>2</sup> sample)**

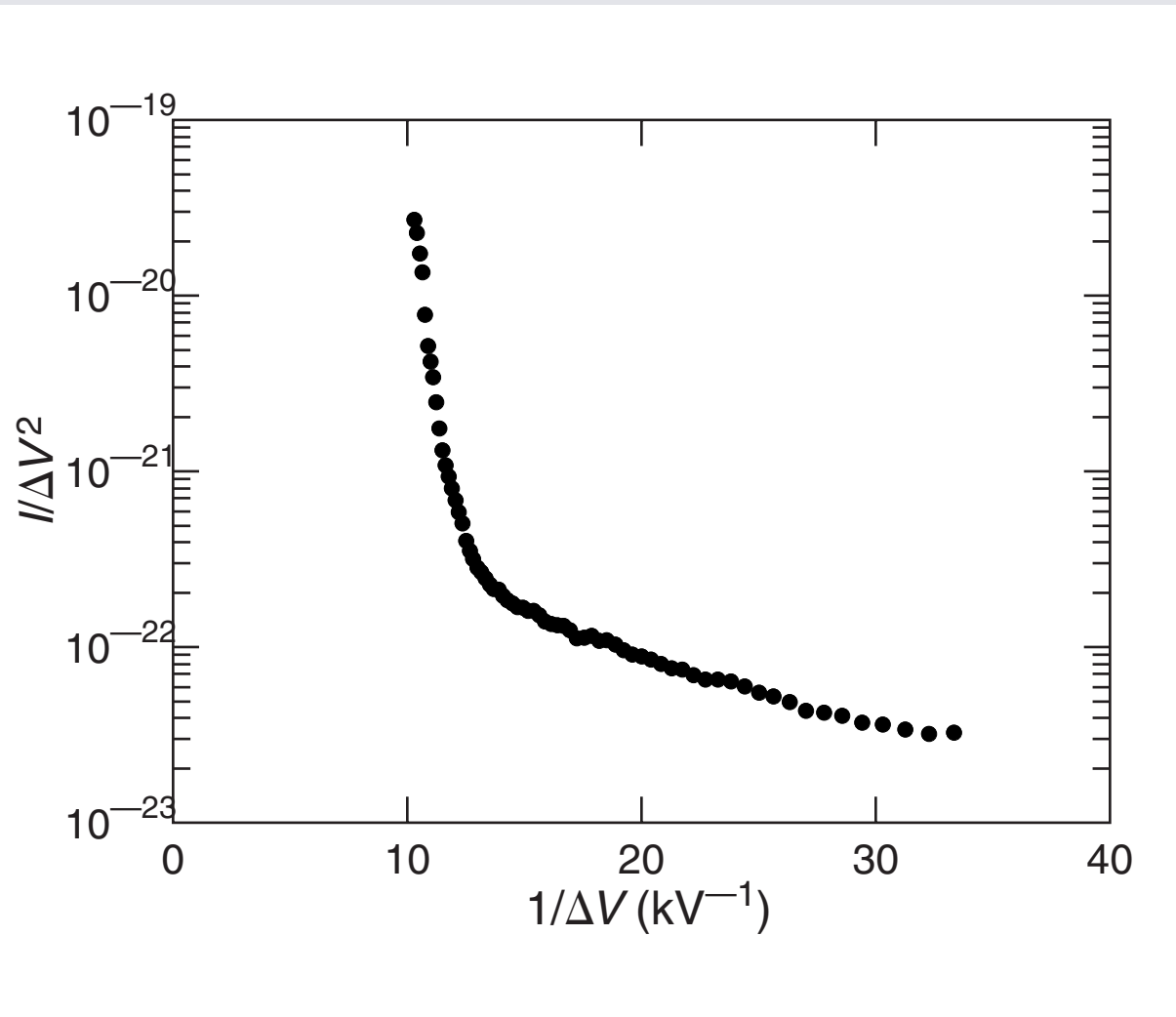
# Properties

$$\ln \frac{I}{\Delta V^2} = \ln a - b \frac{1}{\Delta V}$$

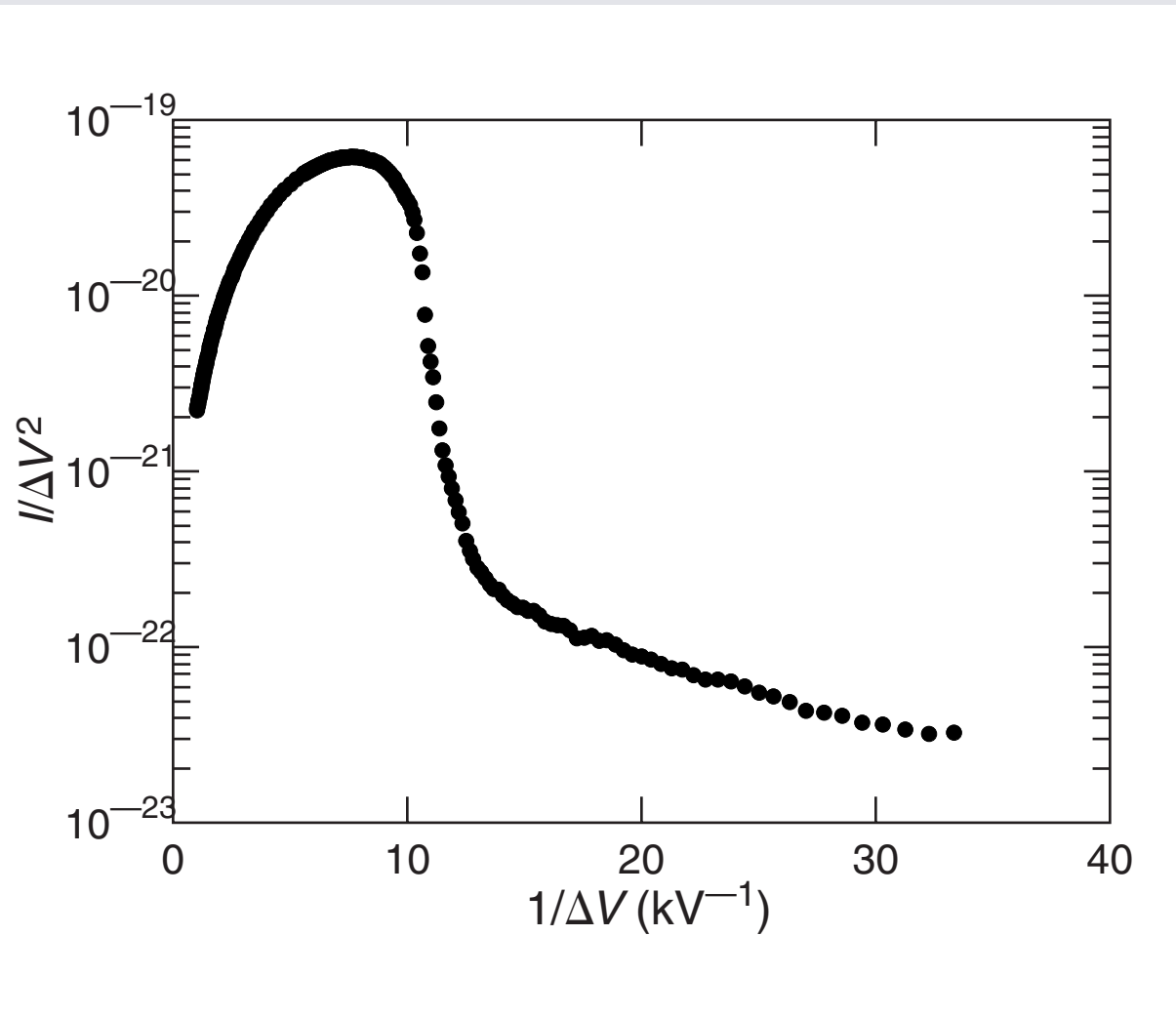


R.H. Fowler and L. Nordheim, *Proc. R. Soc. Lond. A* (1928)

# Properties



# Properties



# *Properties*

## **Points to keep in mind:**

- ▶ **near unity absorption**
- ▶ **sub-band gap absorption**
- ▶ **IR photoelectron generation**
- ▶ **high field emission at low fields**



# Outline

- ▶ Properties
- ▶ **Structural and chemical analysis**
- ▶ Outlook

## *Structural and chemical analysis*

- ▶ **What causes these properties?**
- ▶ **Other gases?**

## **Ion channeling and electron backscattering:**

- ▶ **spikes retain crystalline order**
- ▶ **high density of defects**

## **Secondary ion mass spectrometry:**

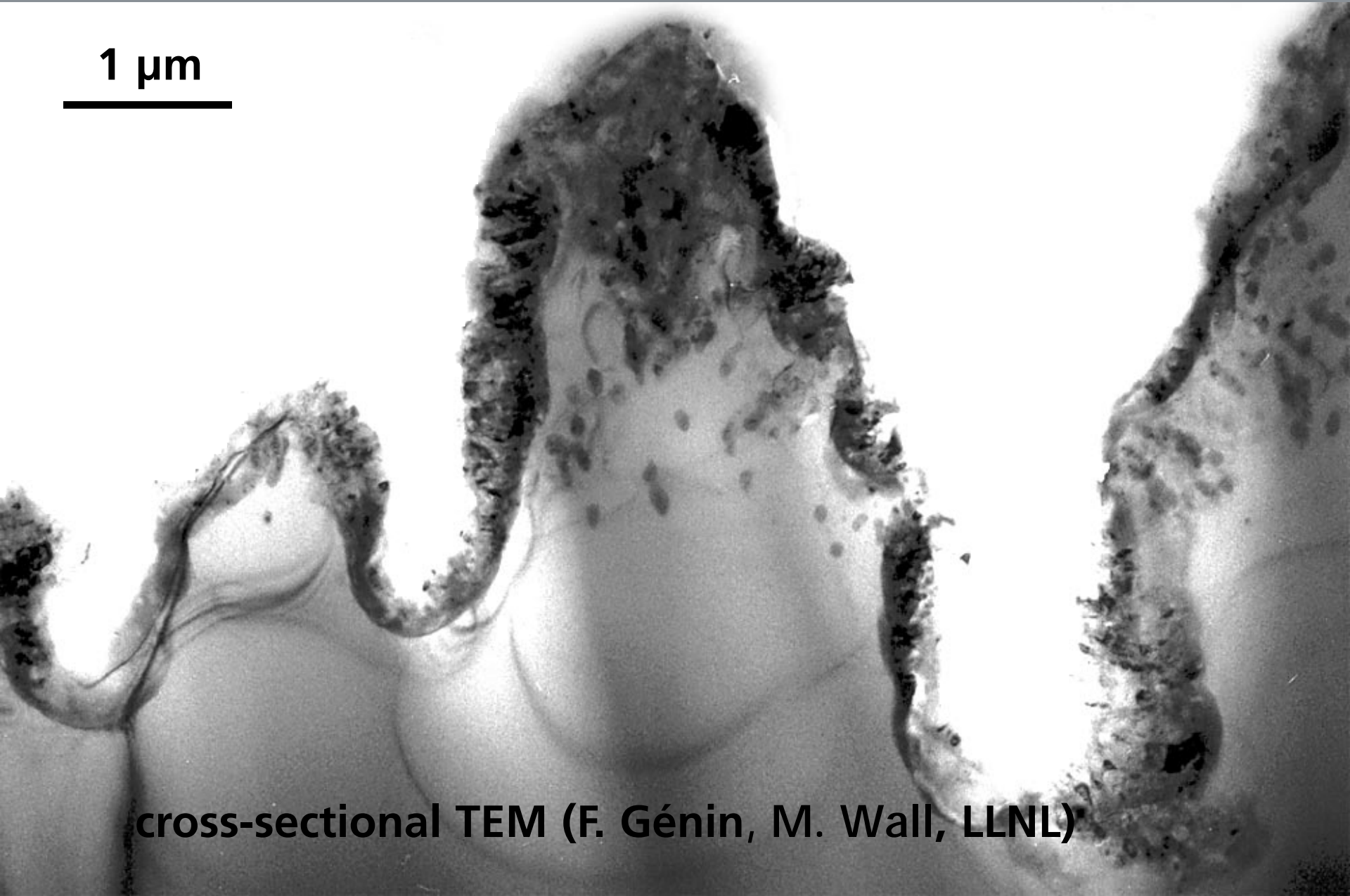
- ▶  **$10^{20} \text{ cm}^{-3}$  sulfur**
- ▶  **$10^{17} \text{ cm}^{-3}$  fluorine**



*Structural and chemical analysis*

**1  $\mu\text{m}$**

---



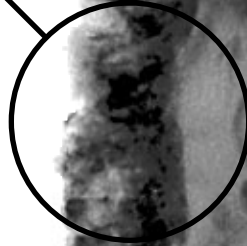
**cross-sectional TEM (F. Génin, M. Wall, LLNL)**

# *Structural and chemical analysis*

**1  $\mu\text{m}$**

---

**porous "fuzz"**



**cross-sectional TEM (F. Génin, M. Wall, LLNL)**

# *Structural and chemical analysis*

**1  $\mu\text{m}$**



**nanocrystallites**

**cross-sectional TEM (F. Génin, M. Wall, LLNL)**



# *Structural and chemical analysis*

**1  $\mu\text{m}$**



**crystalline Si**



**cross-sectional TEM (F. Génin, M. Wall, LLNL)**



# *Structural and chemical analysis*

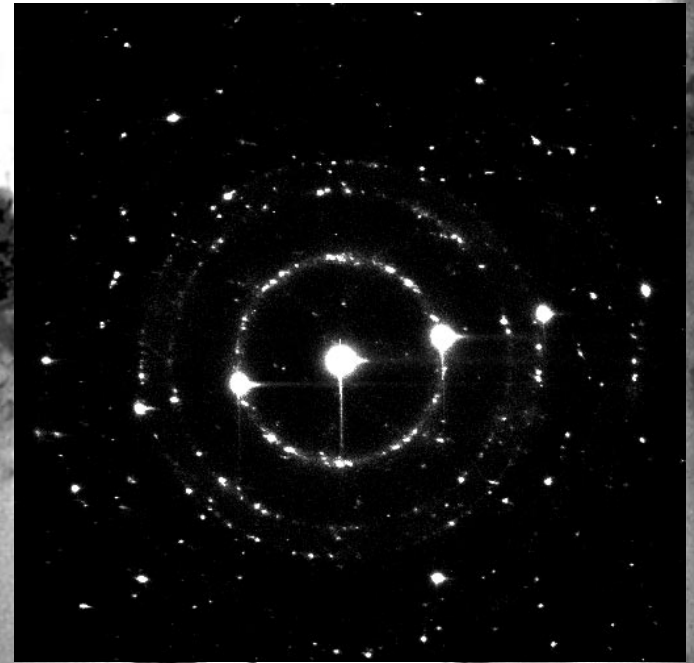
**1  $\mu\text{m}$**

The image is a transmission electron micrograph (TEM) showing a biological specimen, possibly a cross-section of a plant stem or root. The specimen has a central, lighter-colored region surrounded by darker, more textured layers. A scale bar in the top left corner indicates a length of 1 micrometer. A yellow dot is placed on the central region of the specimen. An inset in the top right corner shows the electron diffraction pattern corresponding to the area marked by the yellow dot. The diffraction pattern consists of a central bright spot surrounded by a grid of smaller, dimmer spots, indicating a crystalline structure.

**electron diffraction (F. Génin, M. Wall, LLNL)**

# *Structural and chemical analysis*

**1  $\mu\text{m}$**

A transmission electron micrograph (TEM) showing a biological specimen, possibly a cross-section of a plant stem or root. The specimen exhibits a central, elongated, and somewhat irregular structure with a darker, more textured interior. A yellow dot is placed on the upper part of this central structure. To the left of the central structure, there is a scale bar labeled "1 μm". The overall image is in grayscale, with varying shades of gray representing different densities and structures within the specimen.

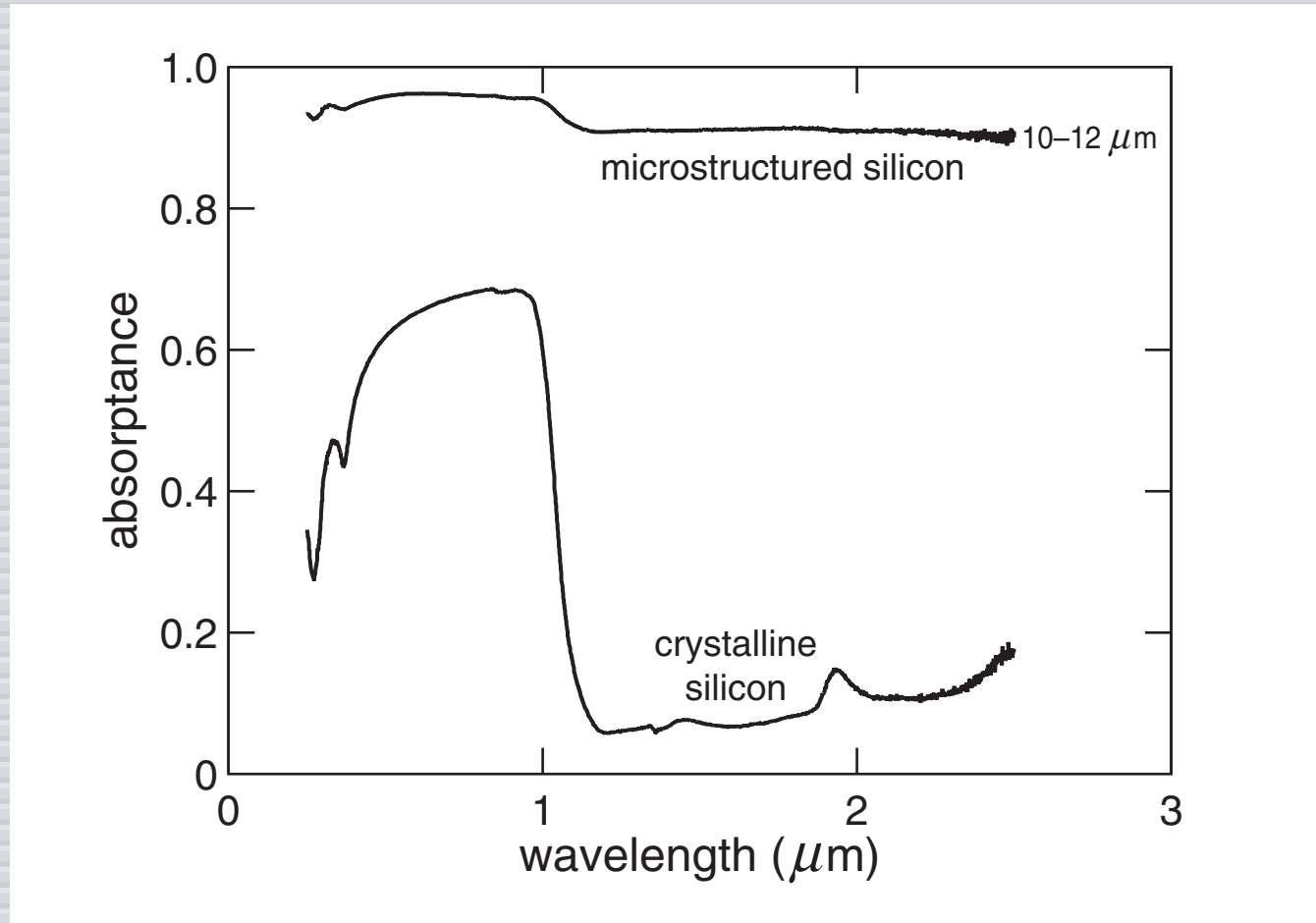
**electron diffraction (F. Génin, M. Wall, LLNL)**

**cross-sectional TEM:**

- ▶ **core of spikes: undisturbed Si**
- ▶ **surface layer: disordered Si, impurities, nanocrystallites and pores**

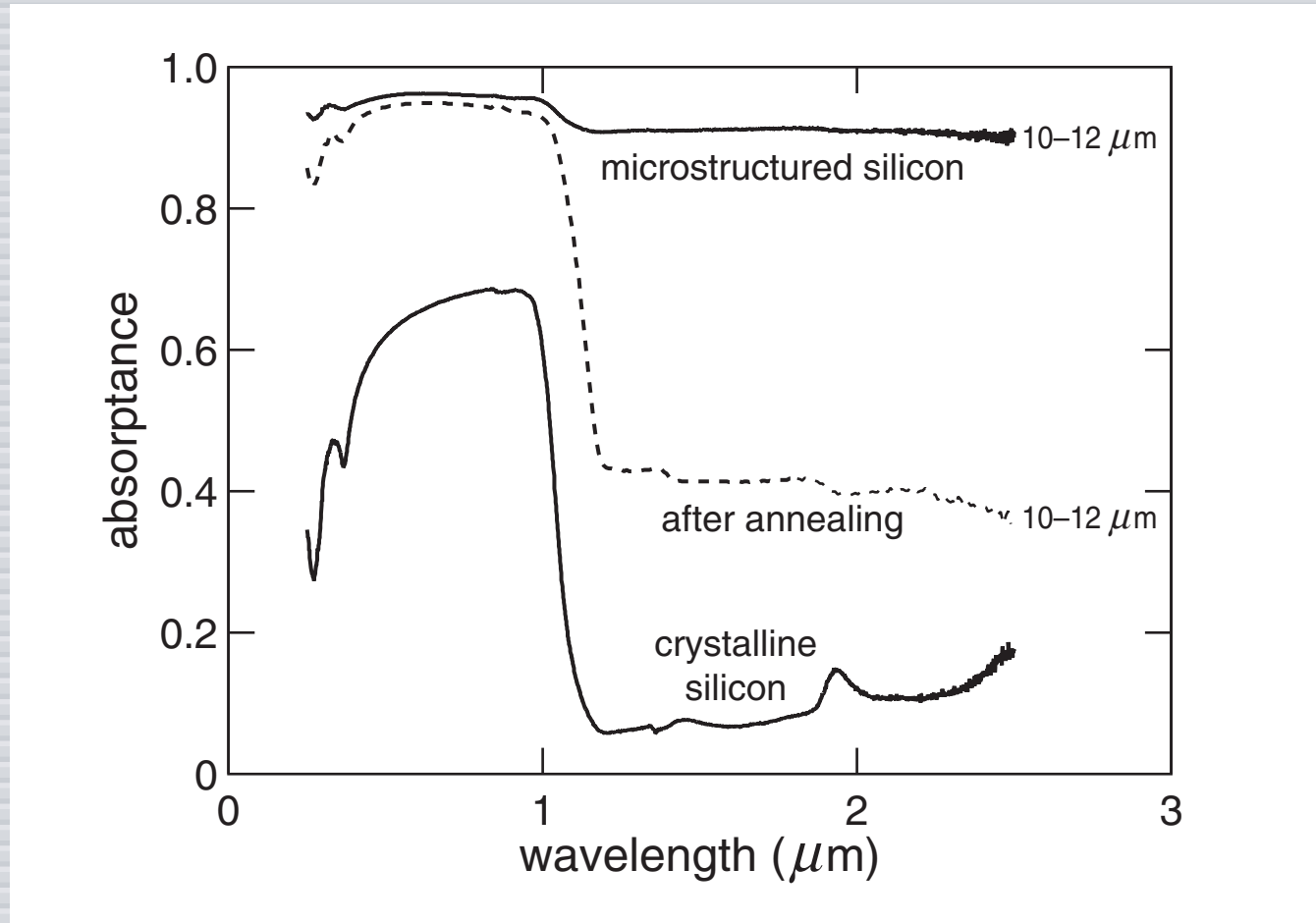
# Structural and chemical analysis

anneal 4 hours at 1200 K



# Structural and chemical analysis

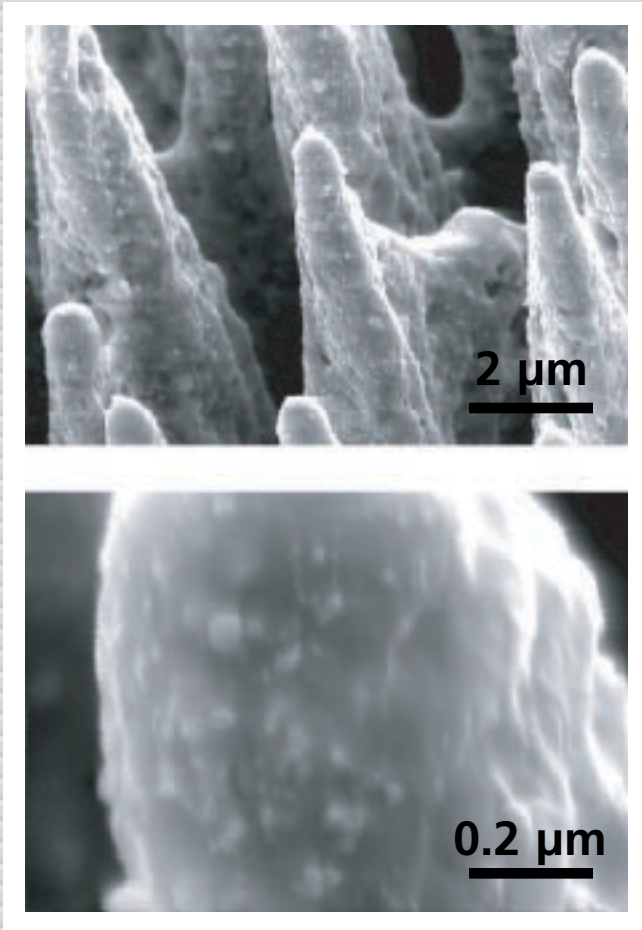
anneal 4 hours at 1200 K





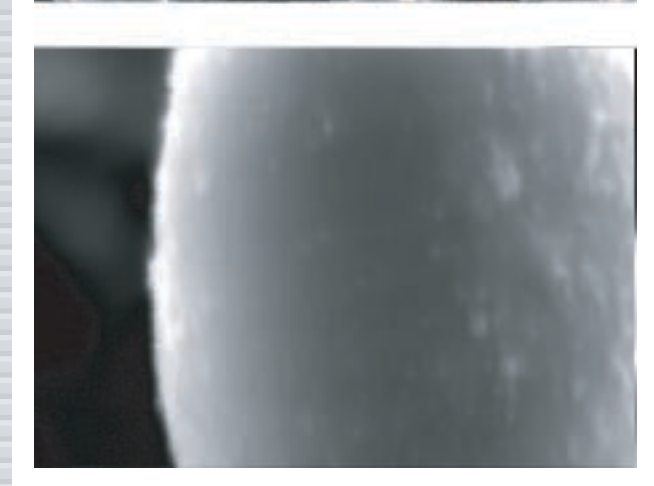
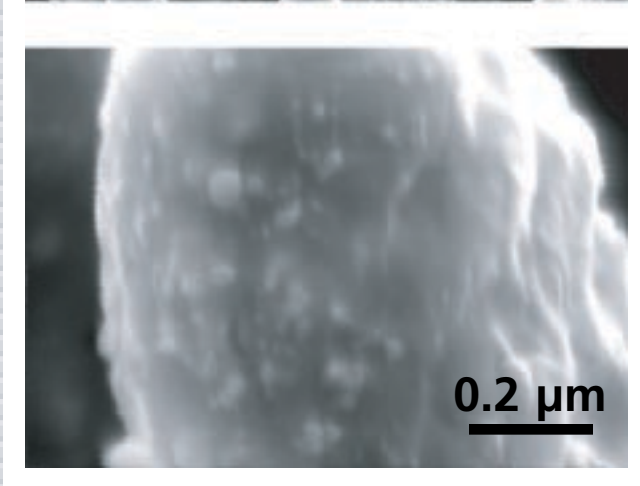
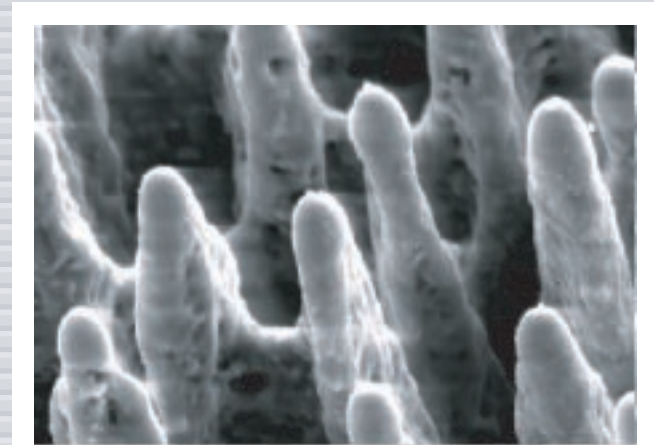
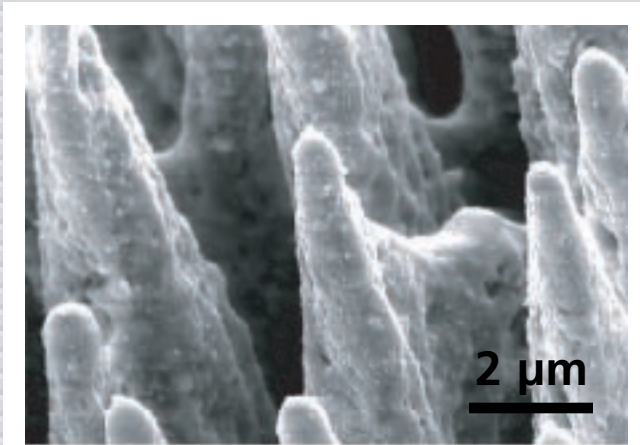
# *Structural and chemical analysis*

**anneal 4 hours at 1200 K**



# *Structural and chemical analysis*

**anneal 4 hours at 1200 K**



# *Structural and chemical analysis*

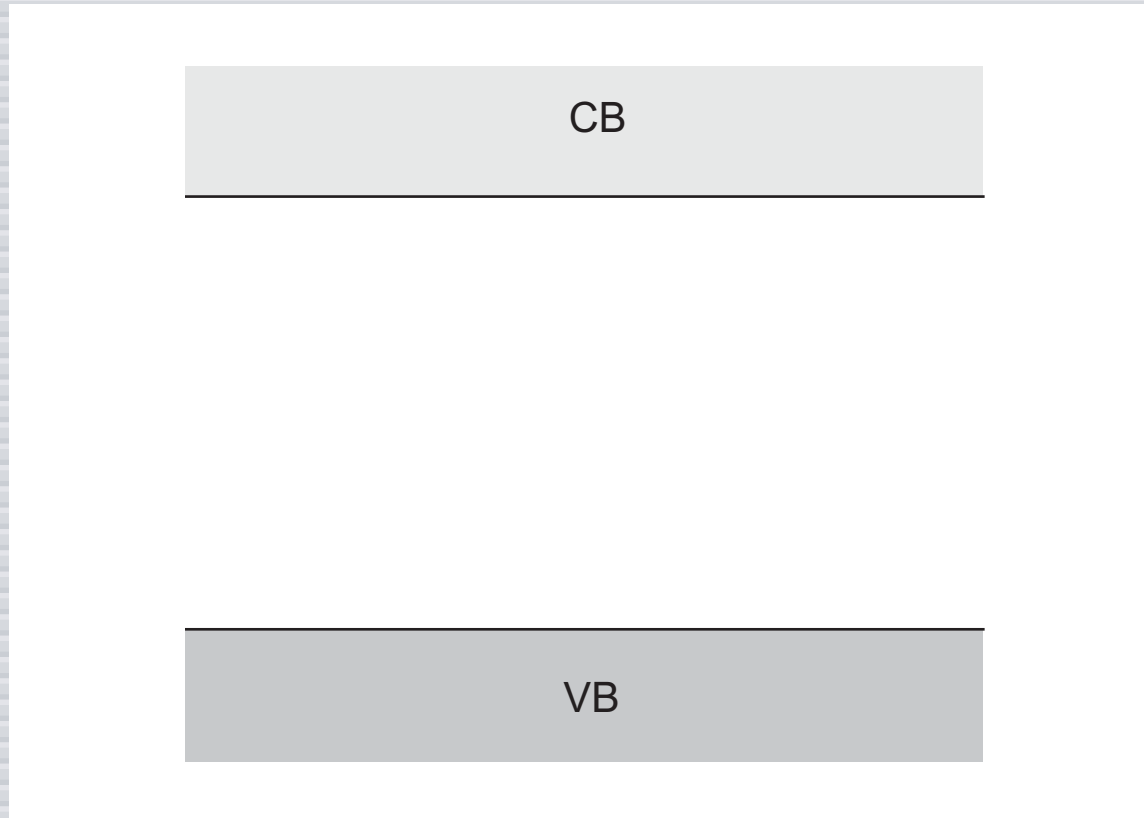
## **Effects of annealing:**

- ▶ **IR absorption: reduced twofold**
- ▶ **SEM: fewer surface nanostructures**
- ▶ **SIMS: sulfur content reduced twofold**



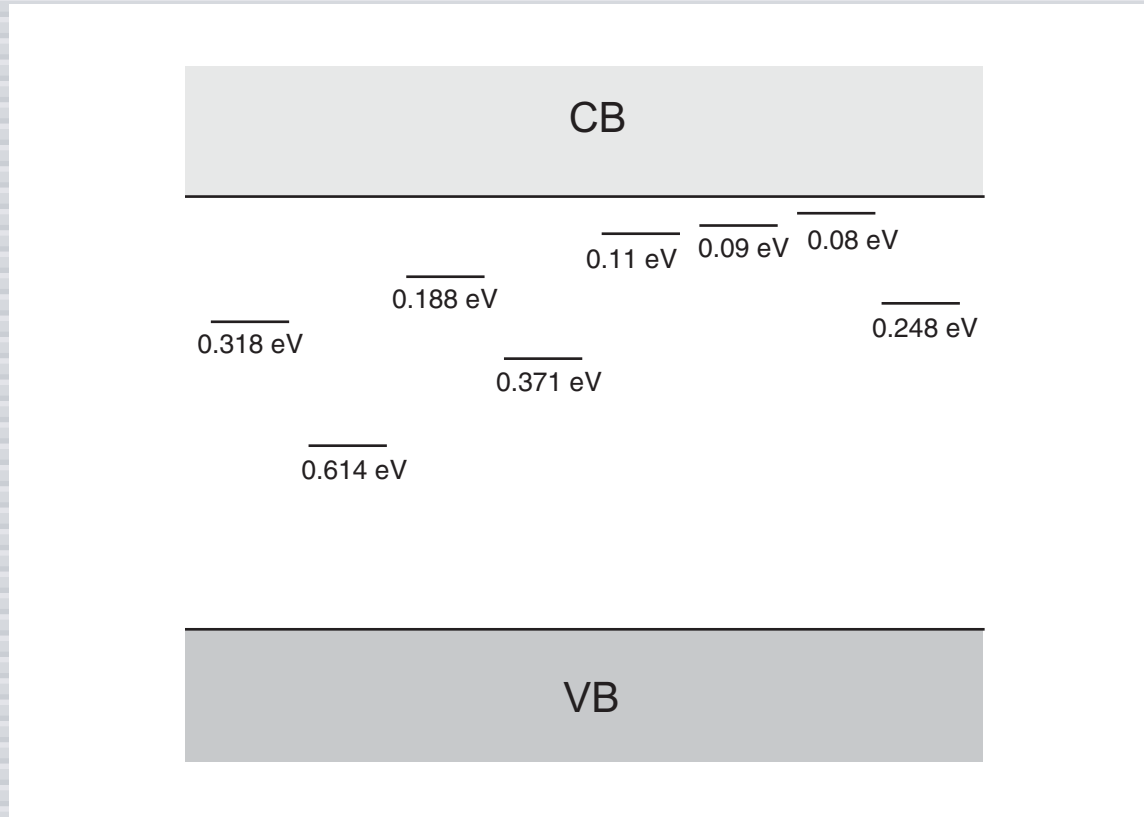
# *Structural and chemical analysis*

**sulfur introduces states in the gap**



# Structural and chemical analysis

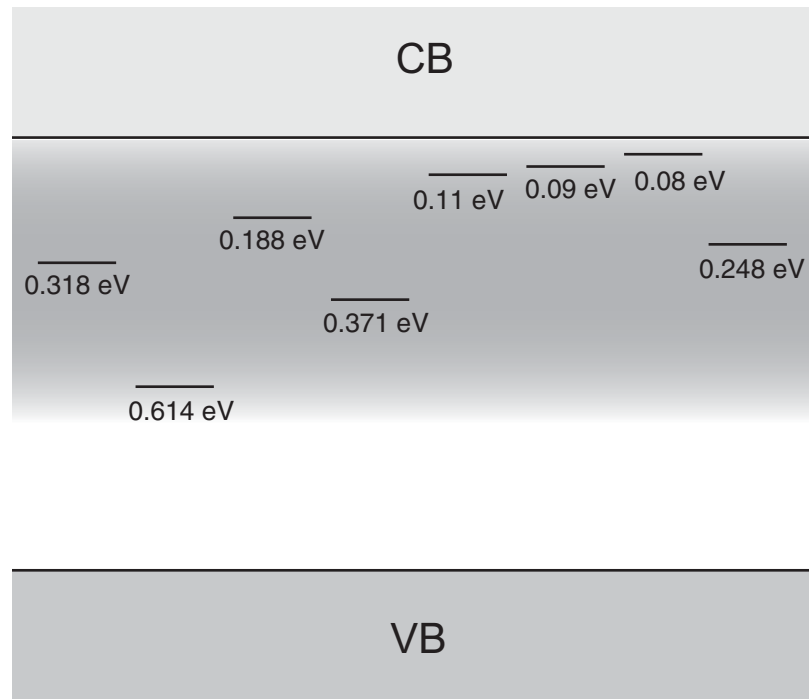
## sulfur introduces states in the gap



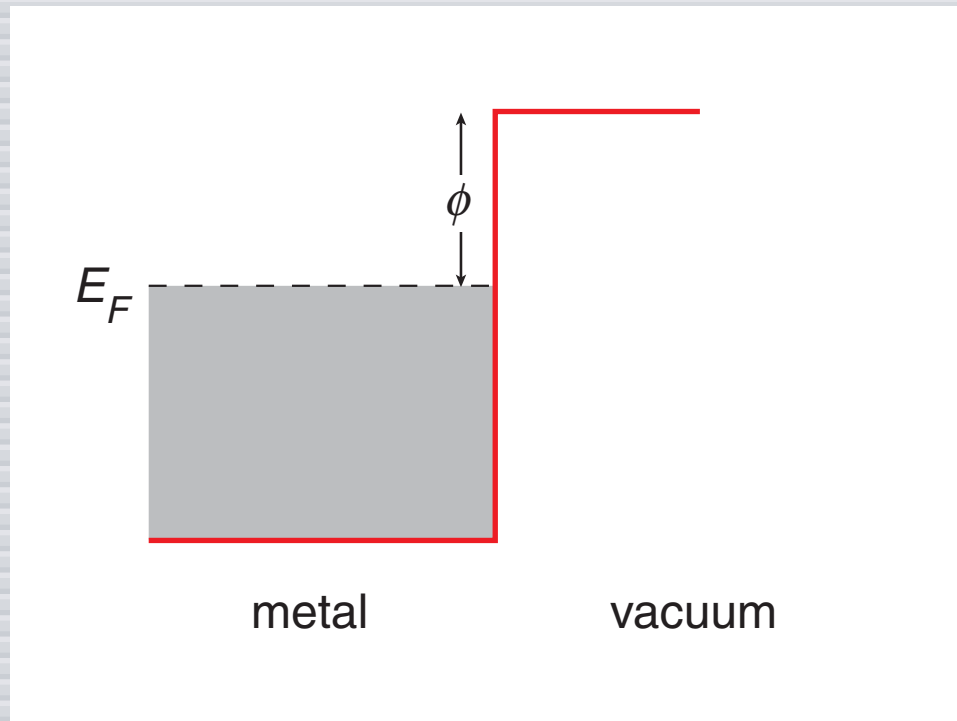
Janzén, et al., *Phys. Rev. B* **29**,1907 (1984)

# Structural and chemical analysis

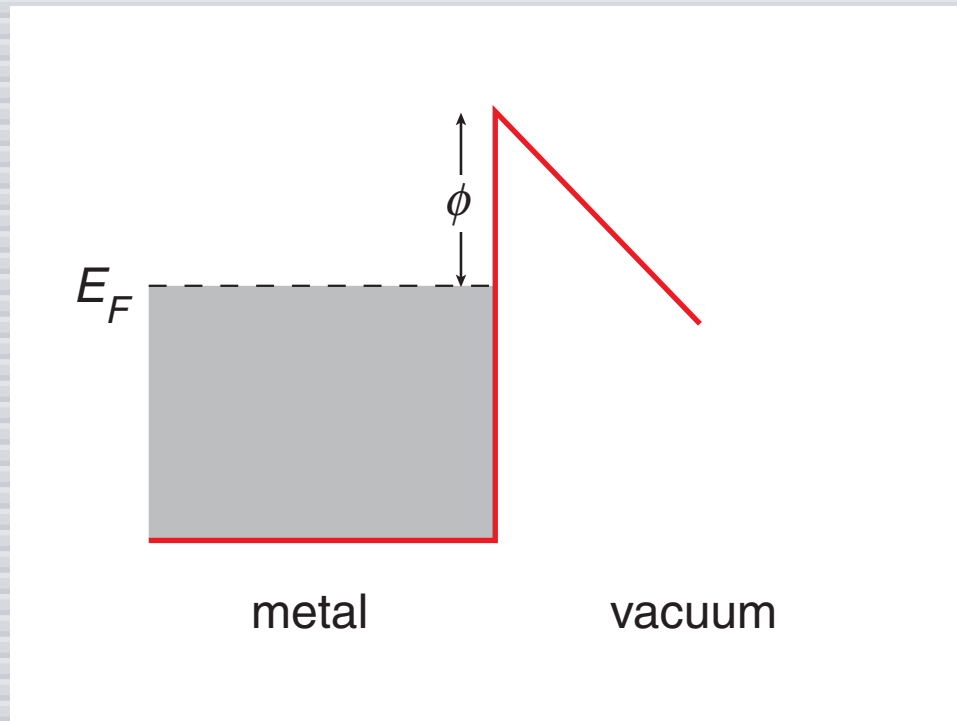
**states broaden into a band**



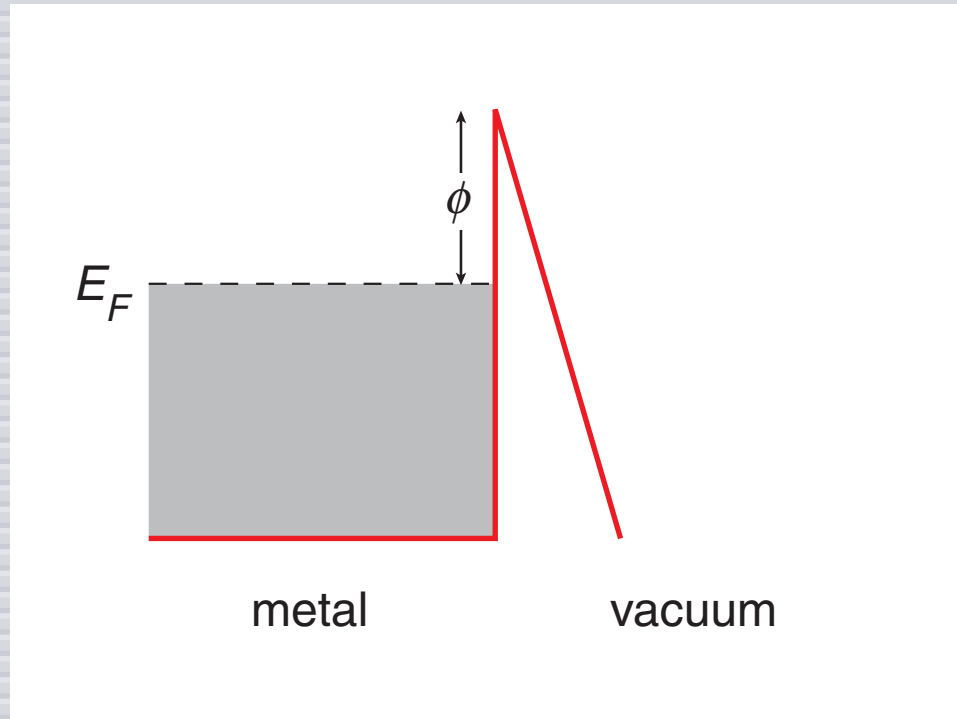
# Structural and chemical analysis



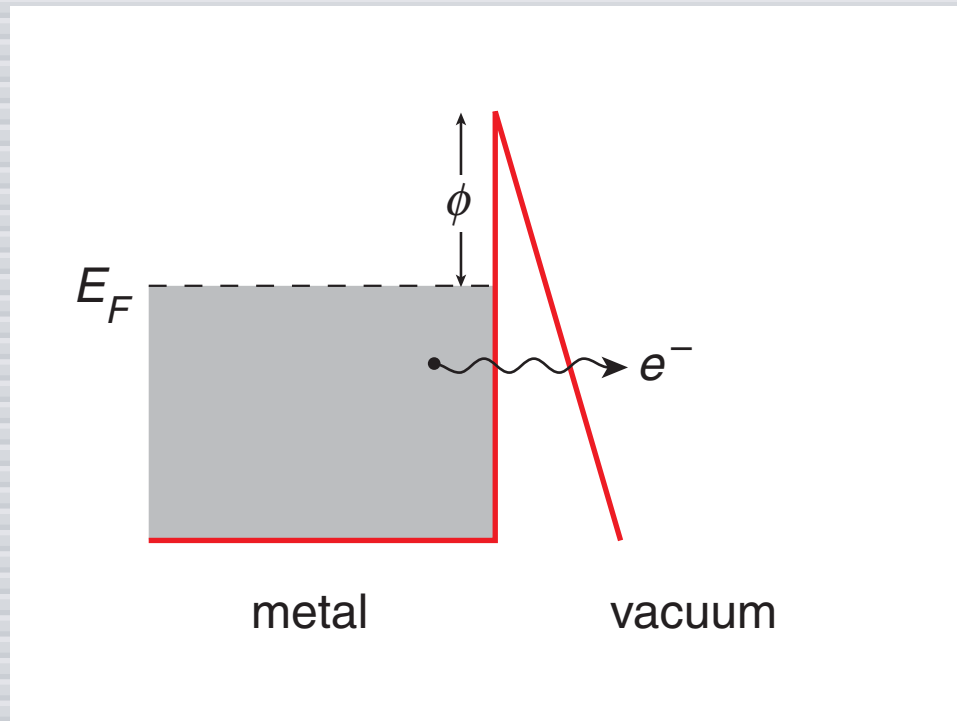
# Structural and chemical analysis



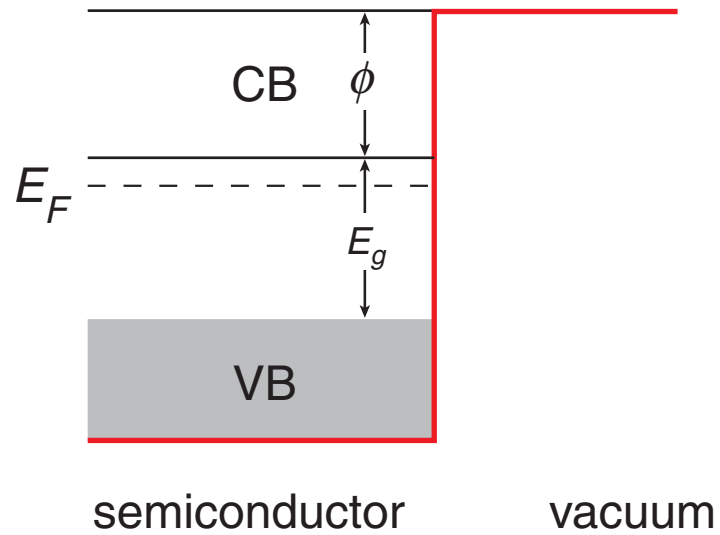
# Structural and chemical analysis



# Structural and chemical analysis

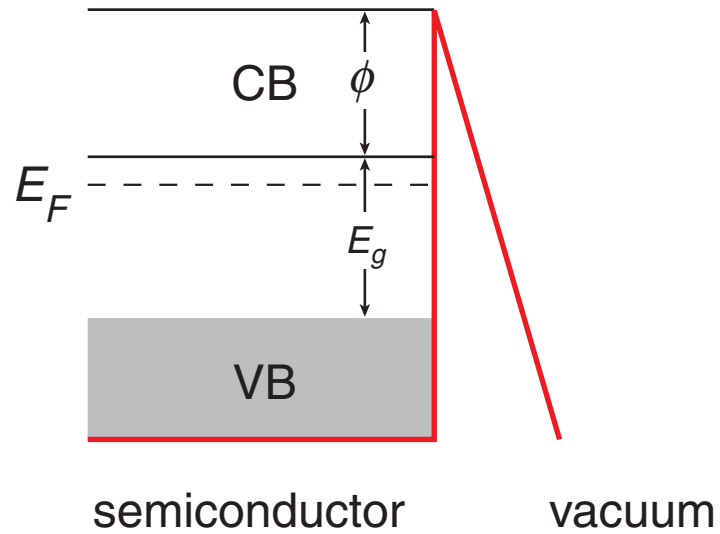


# Structural and chemical analysis

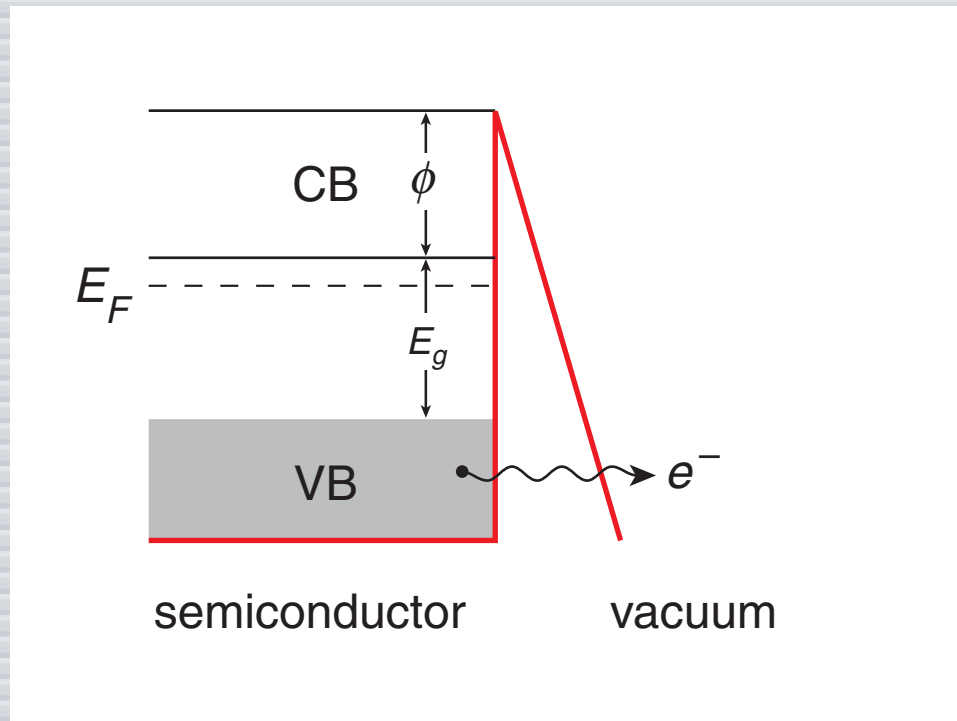




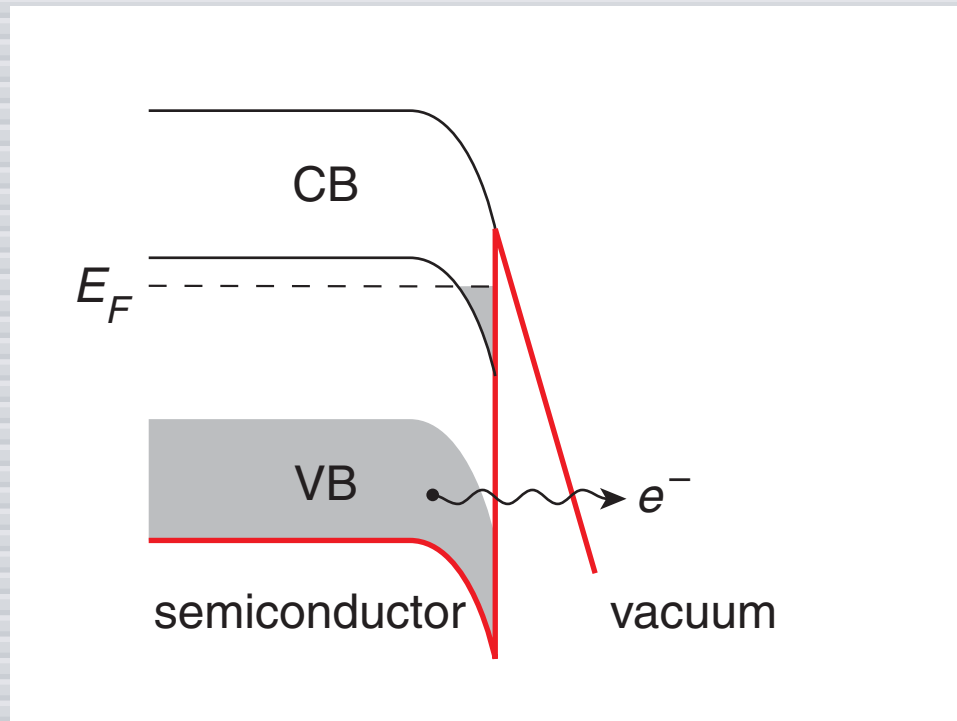
# Structural and chemical analysis



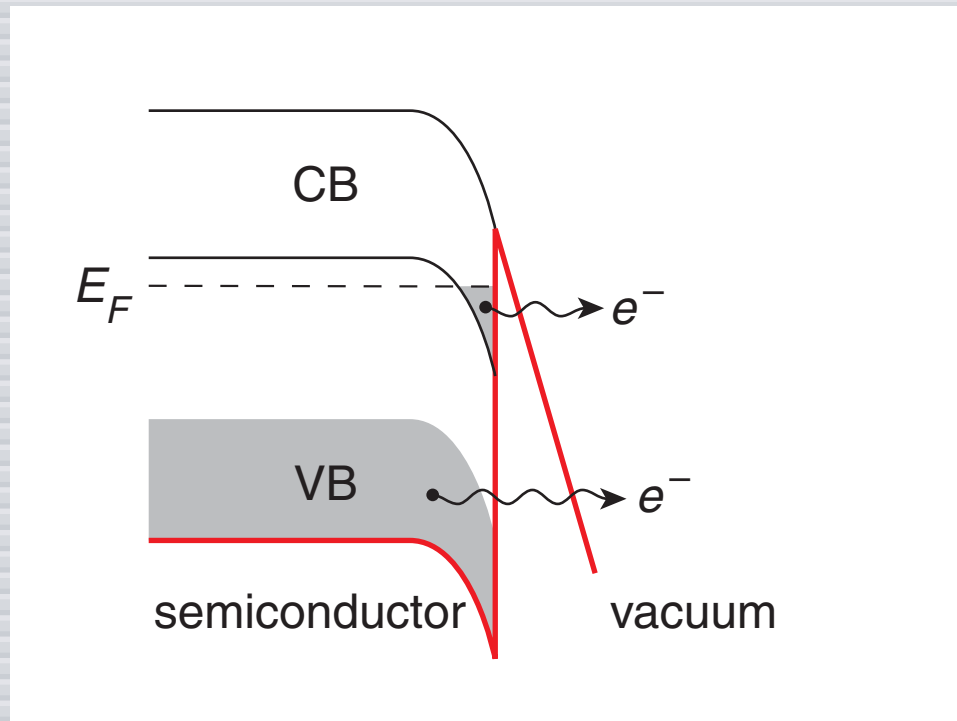
# Structural and chemical analysis



# Structural and chemical analysis

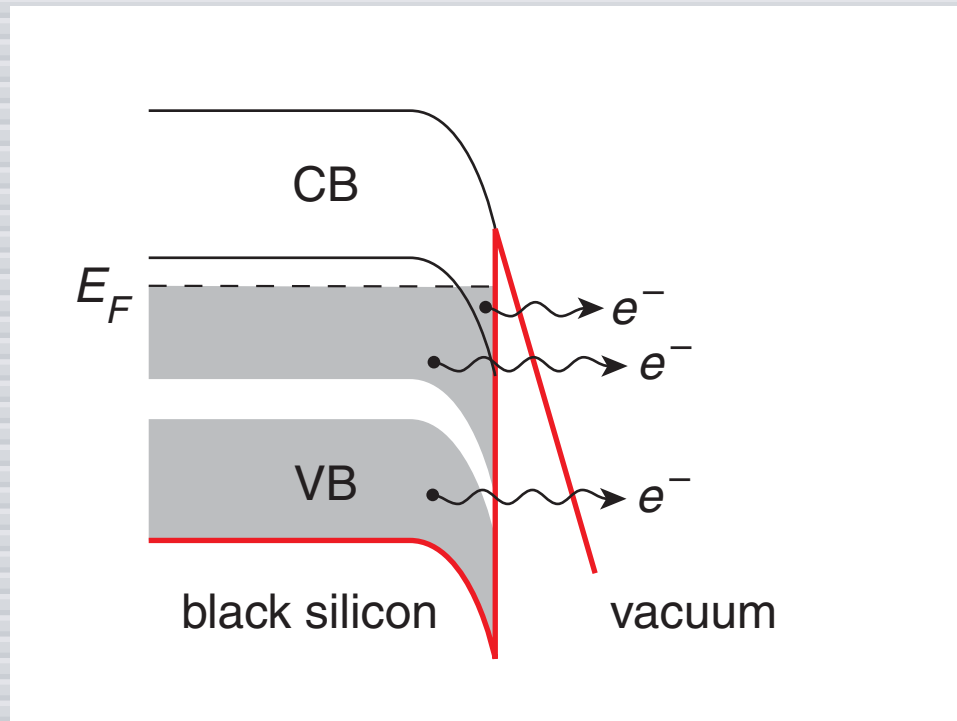


# Structural and chemical analysis



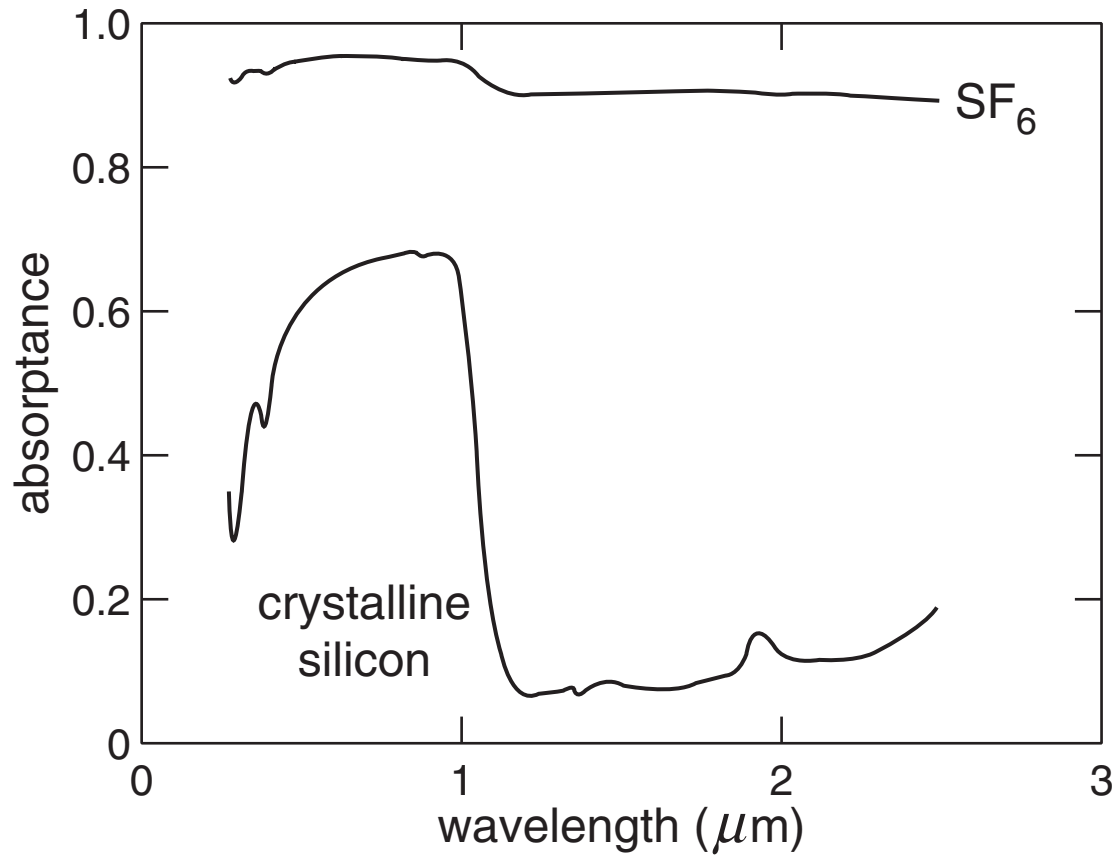
# *Structural and chemical analysis*

**sulfur band provides additional electrons**



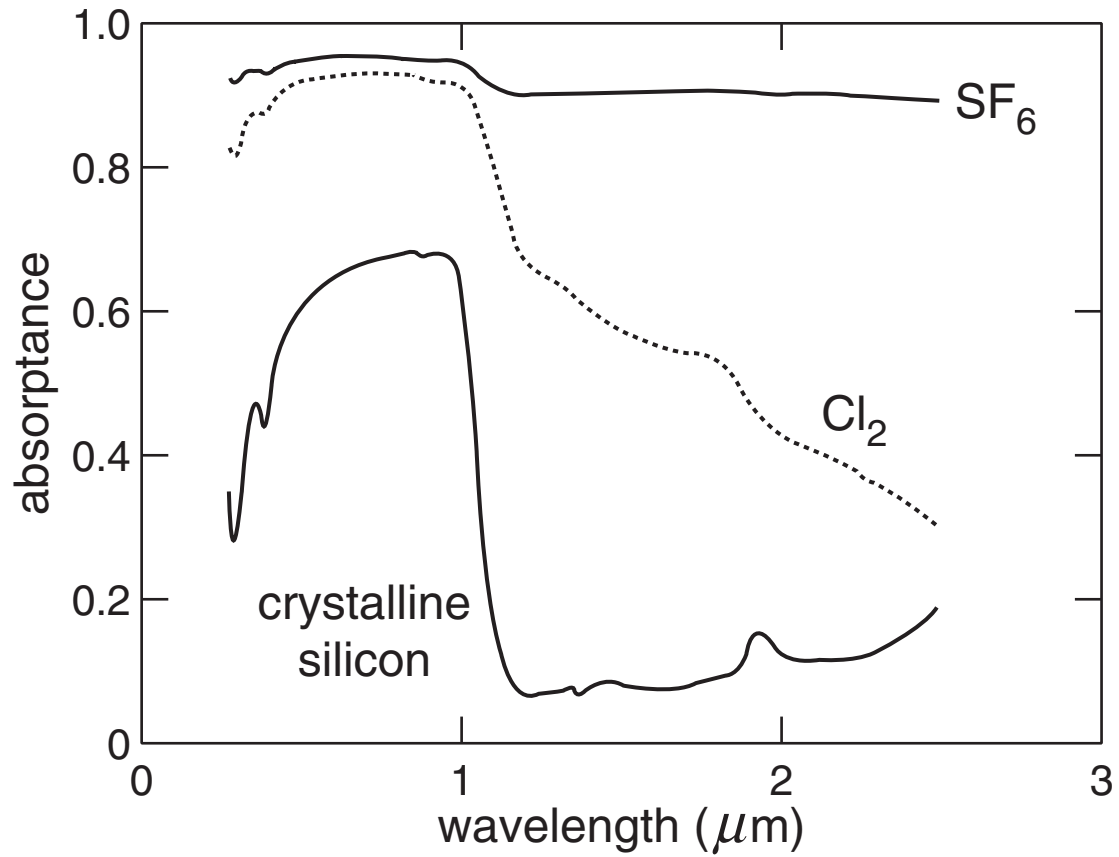
# *Structural and chemical analysis*

## **effect of ambient gas on absorptance**



# *Structural and chemical analysis*

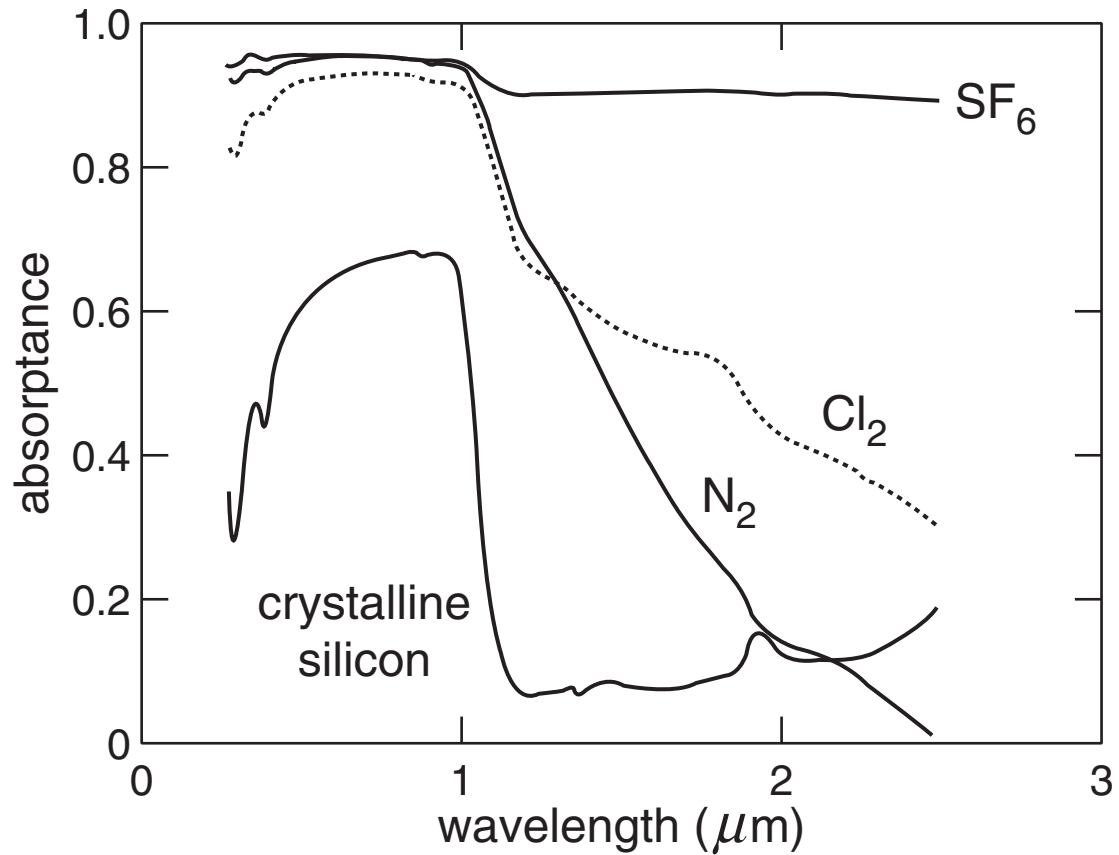
## **effect of ambient gas on absorptance**





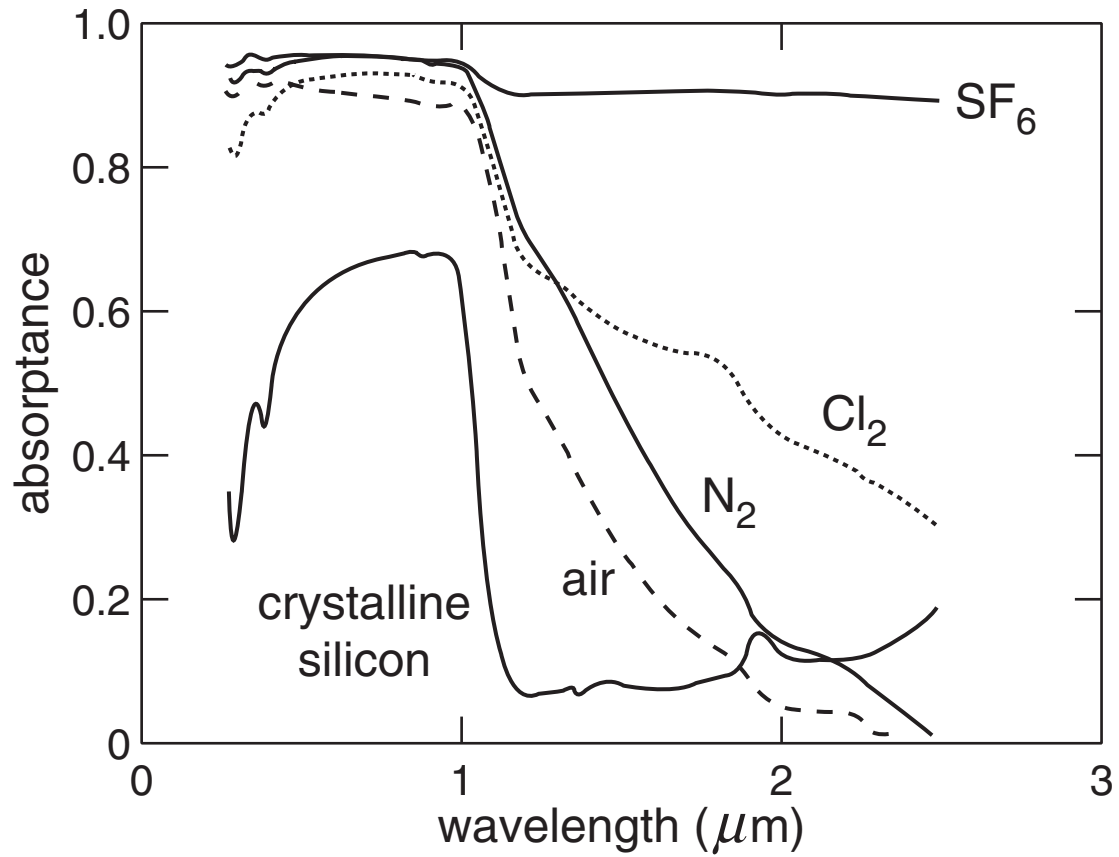
# Structural and chemical analysis

## effect of ambient gas on absorptance



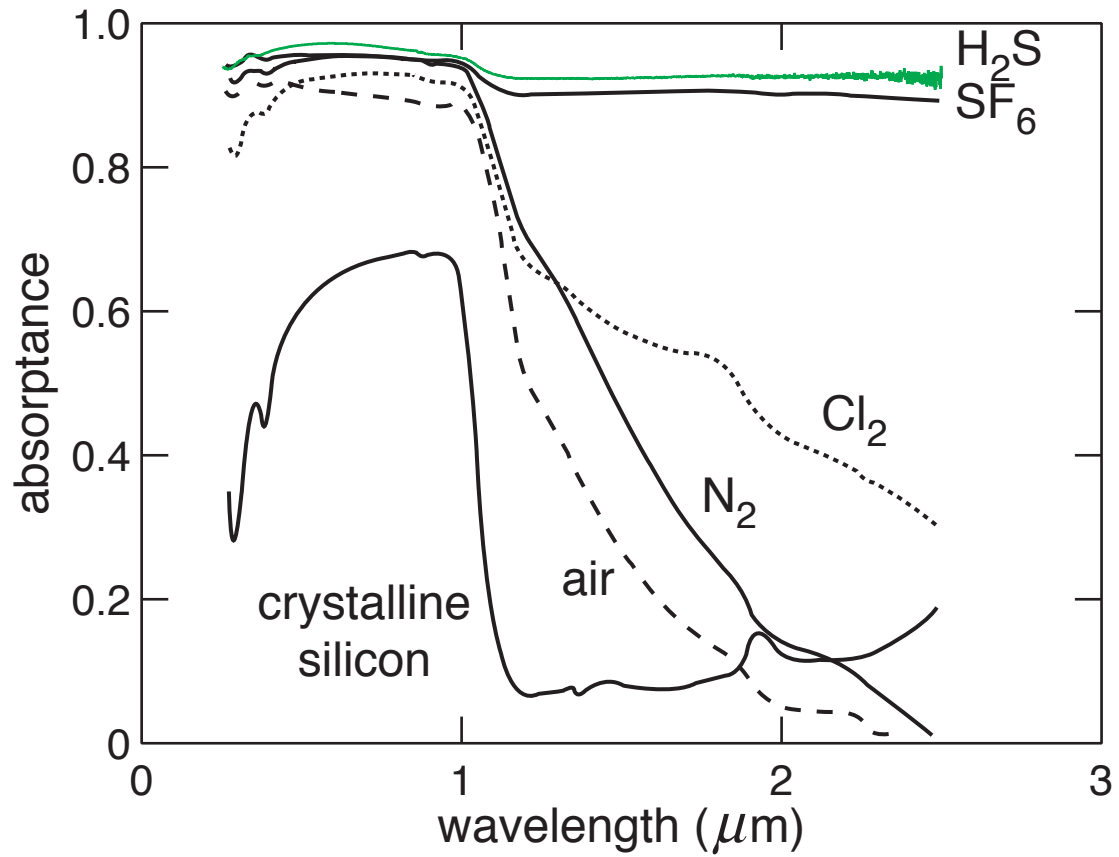
# Structural and chemical analysis

## effect of ambient gas on absorptance



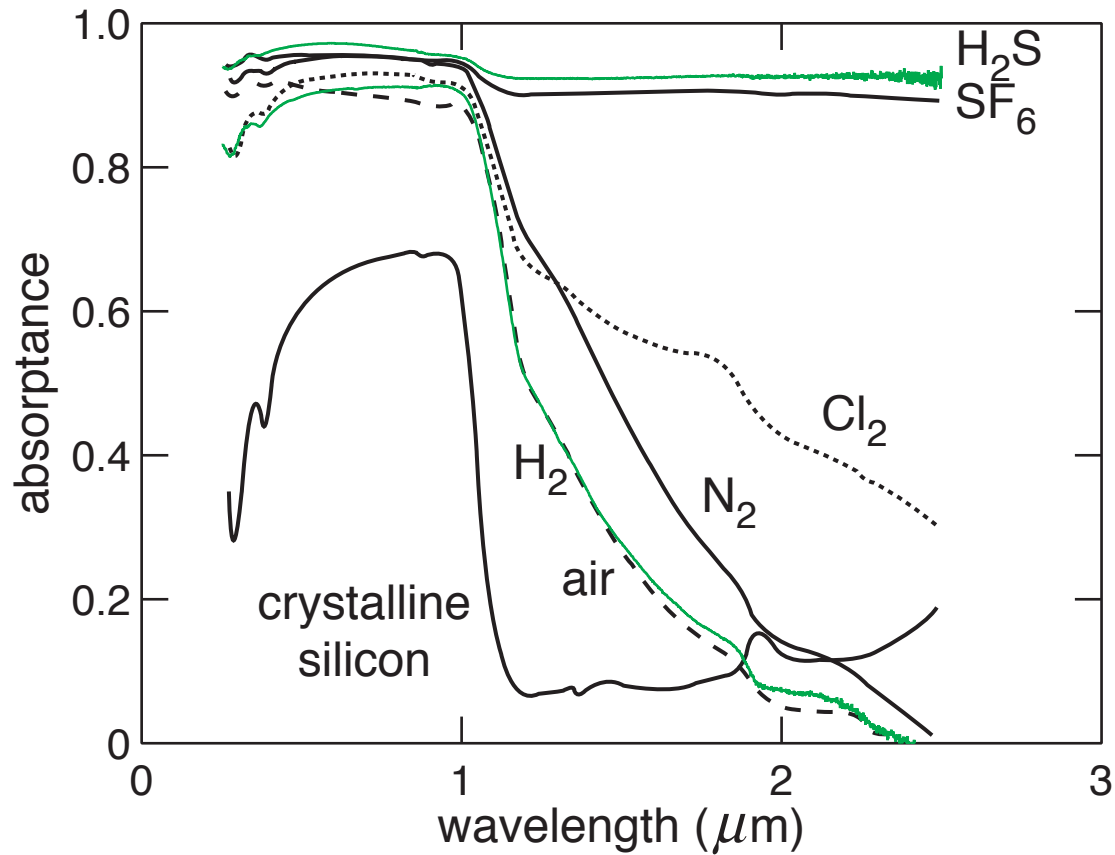
# Structural and chemical analysis

## effect of ambient gas on absorptance



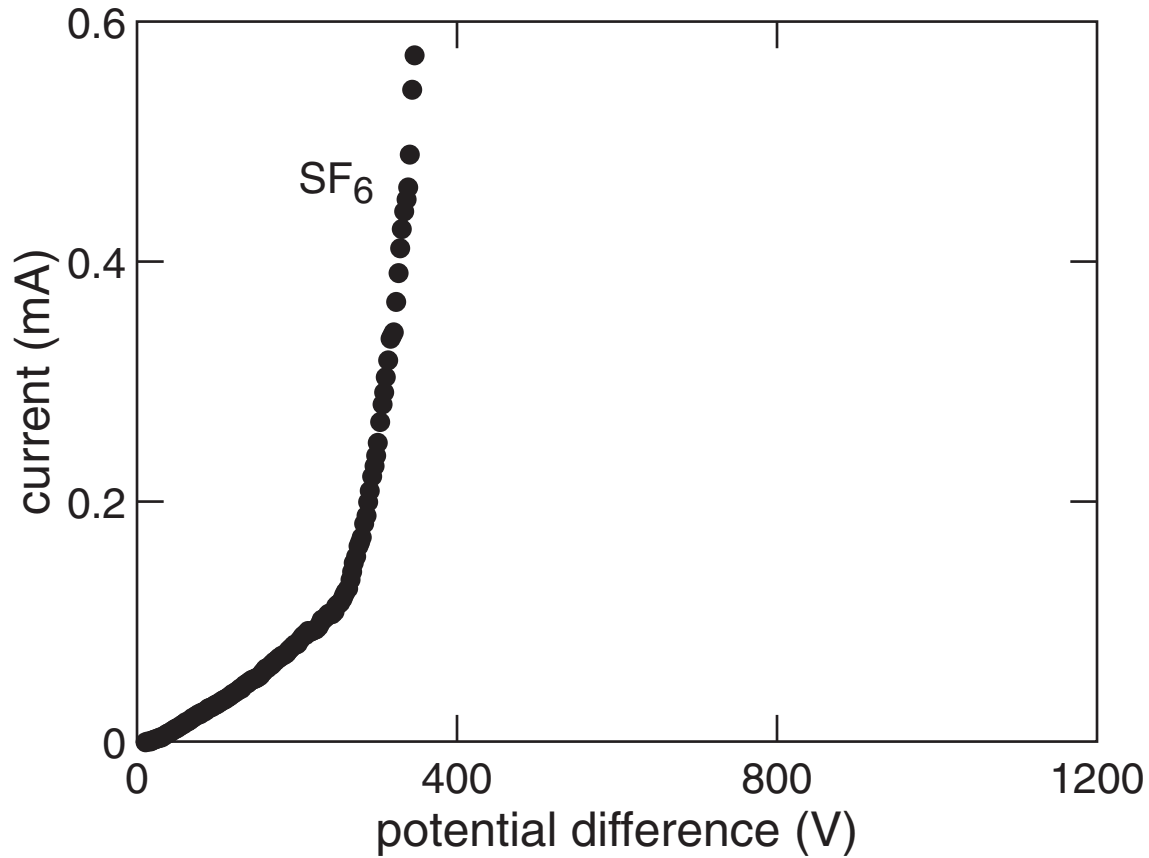
# Structural and chemical analysis

## effect of ambient gas on absorptance



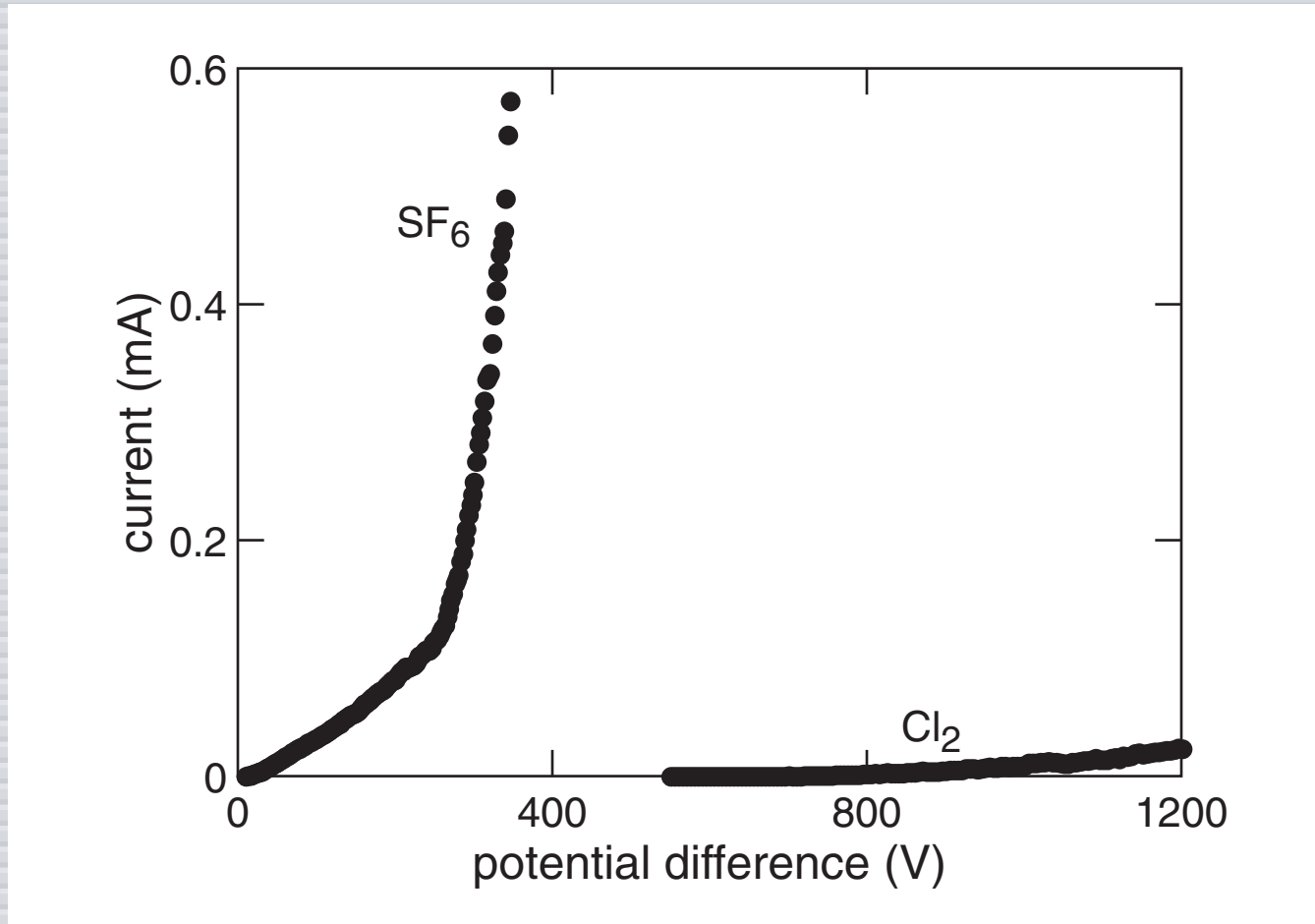
# *Structural and chemical analysis*

## **effect of ambient gas on field emission**



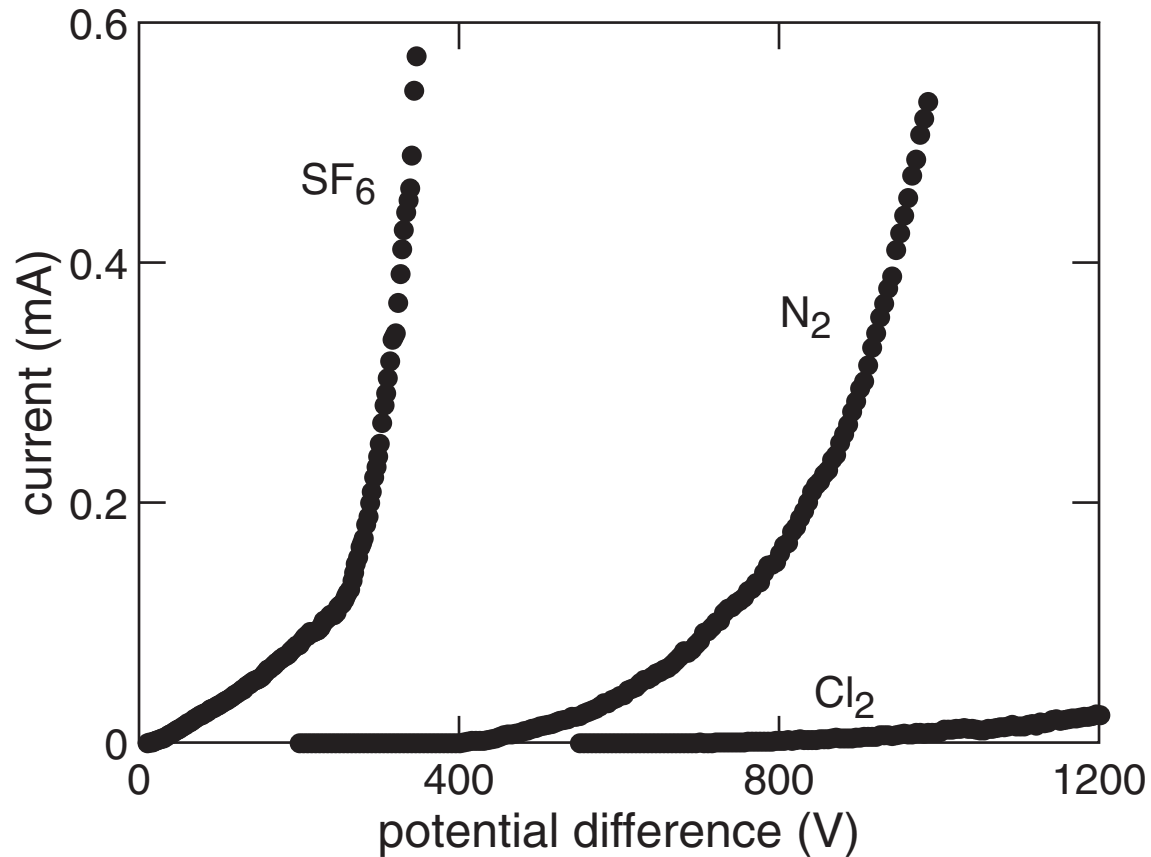
# *Structural and chemical analysis*

## effect of ambient gas on field emission



# *Structural and chemical analysis*

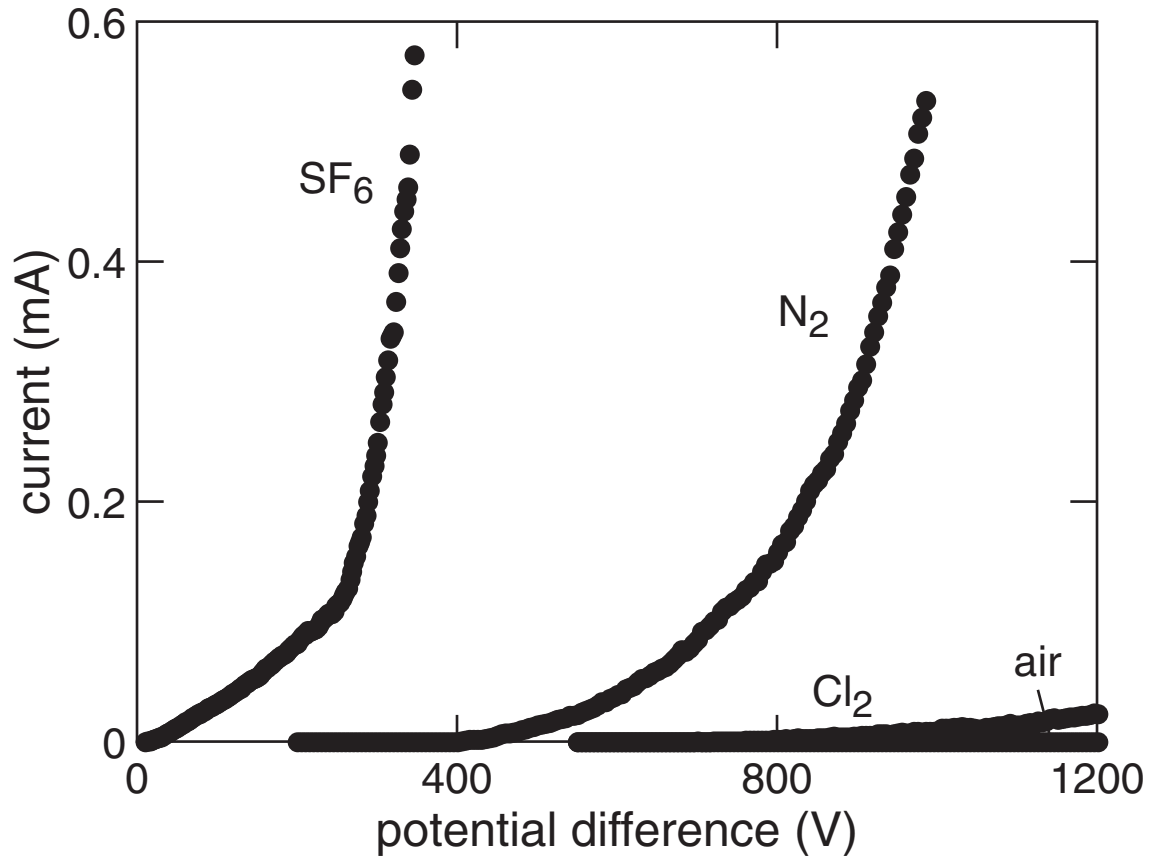
## effect of ambient gas on field emission

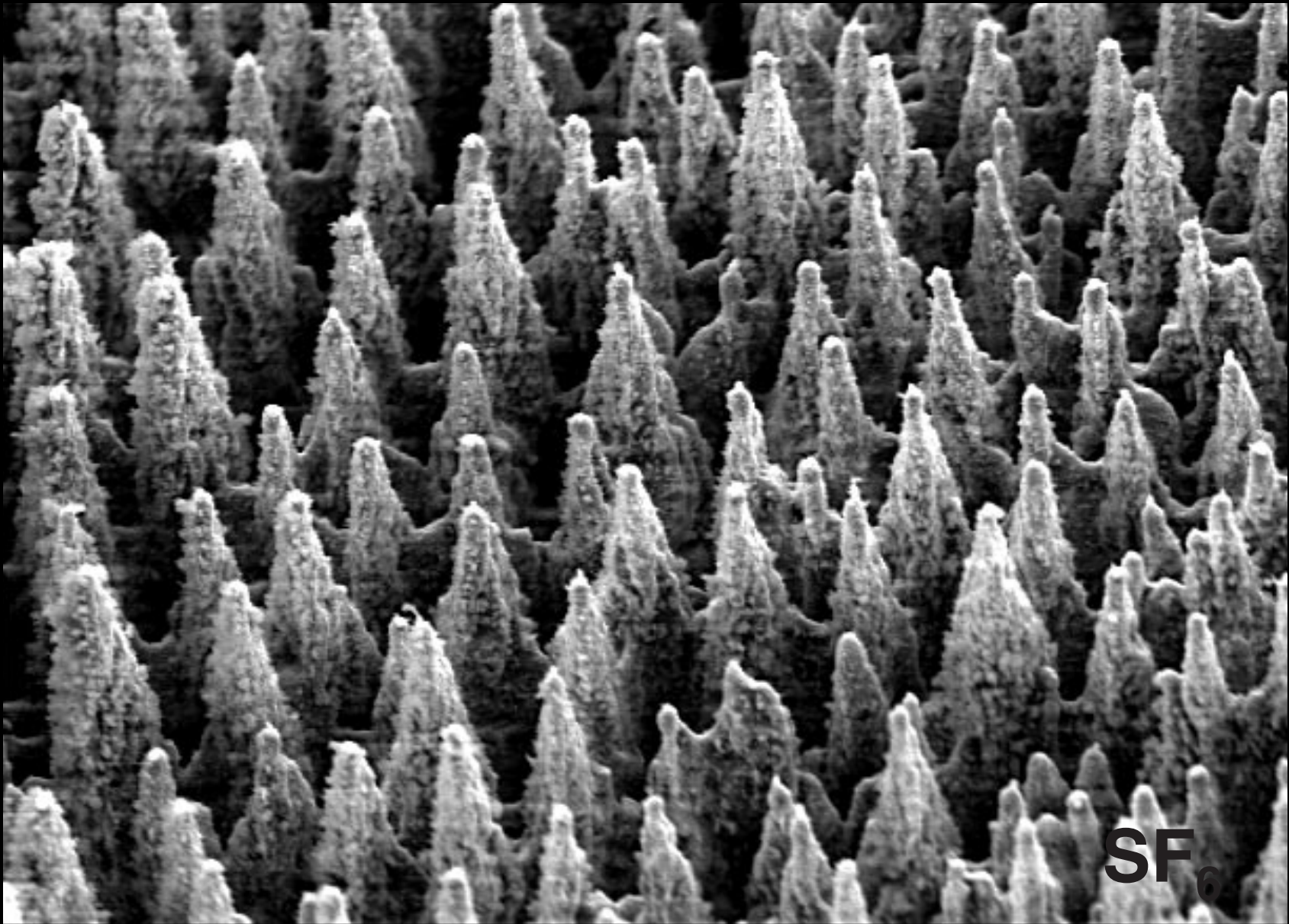




# Structural and chemical analysis

## effect of ambient gas on field emission



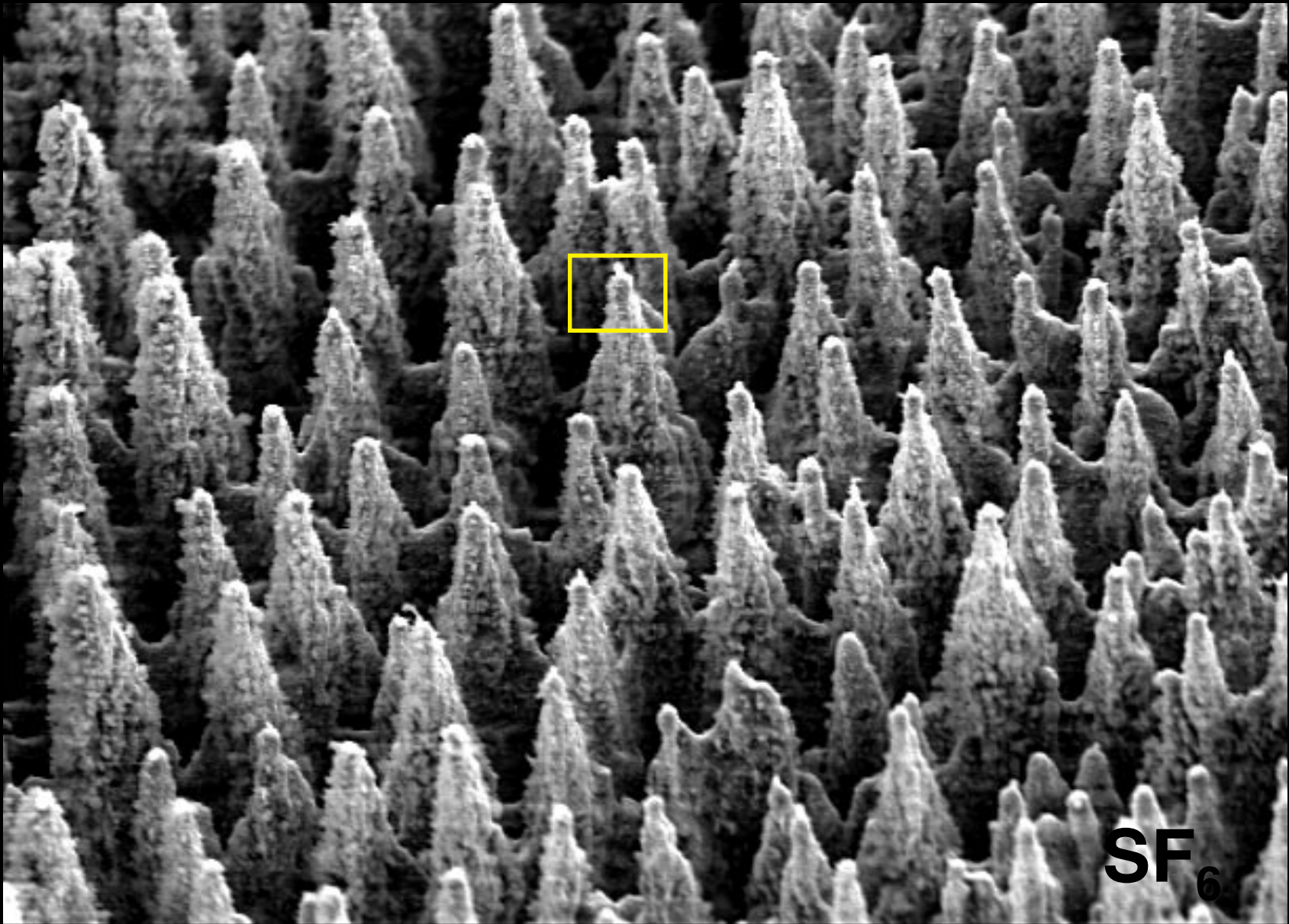


x3000  
#240

10  $\mu$ m  
SF6

5kV

14mm



**SF<sub>6</sub>**

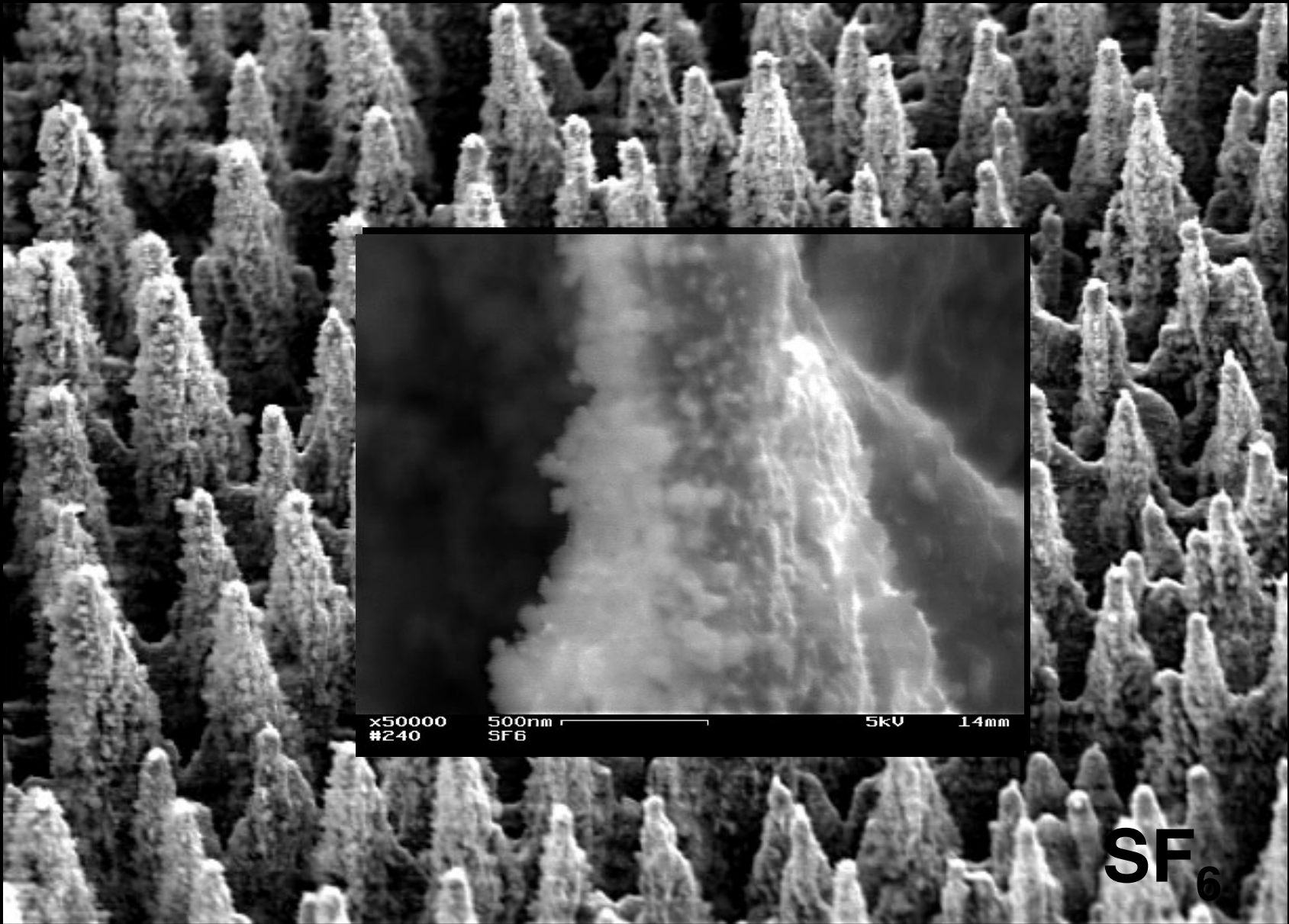
x3000  
#240

10 μm  
SF6

5kV

14mm

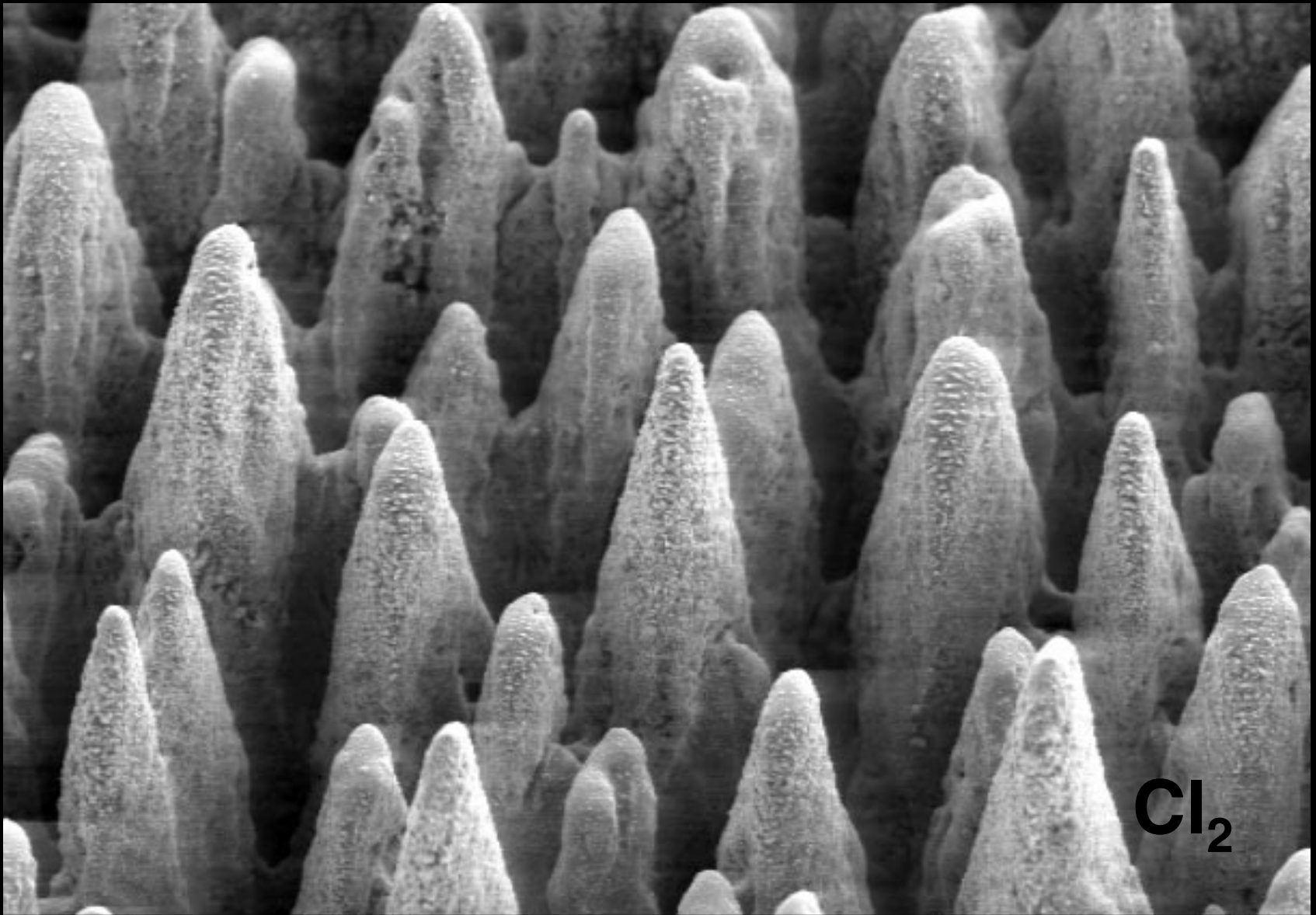




x50000 500nm 5kV 14mm  
#240 SF6

SF<sub>6</sub>

x3000 10µm 5kV 14mm  
#240 SF6

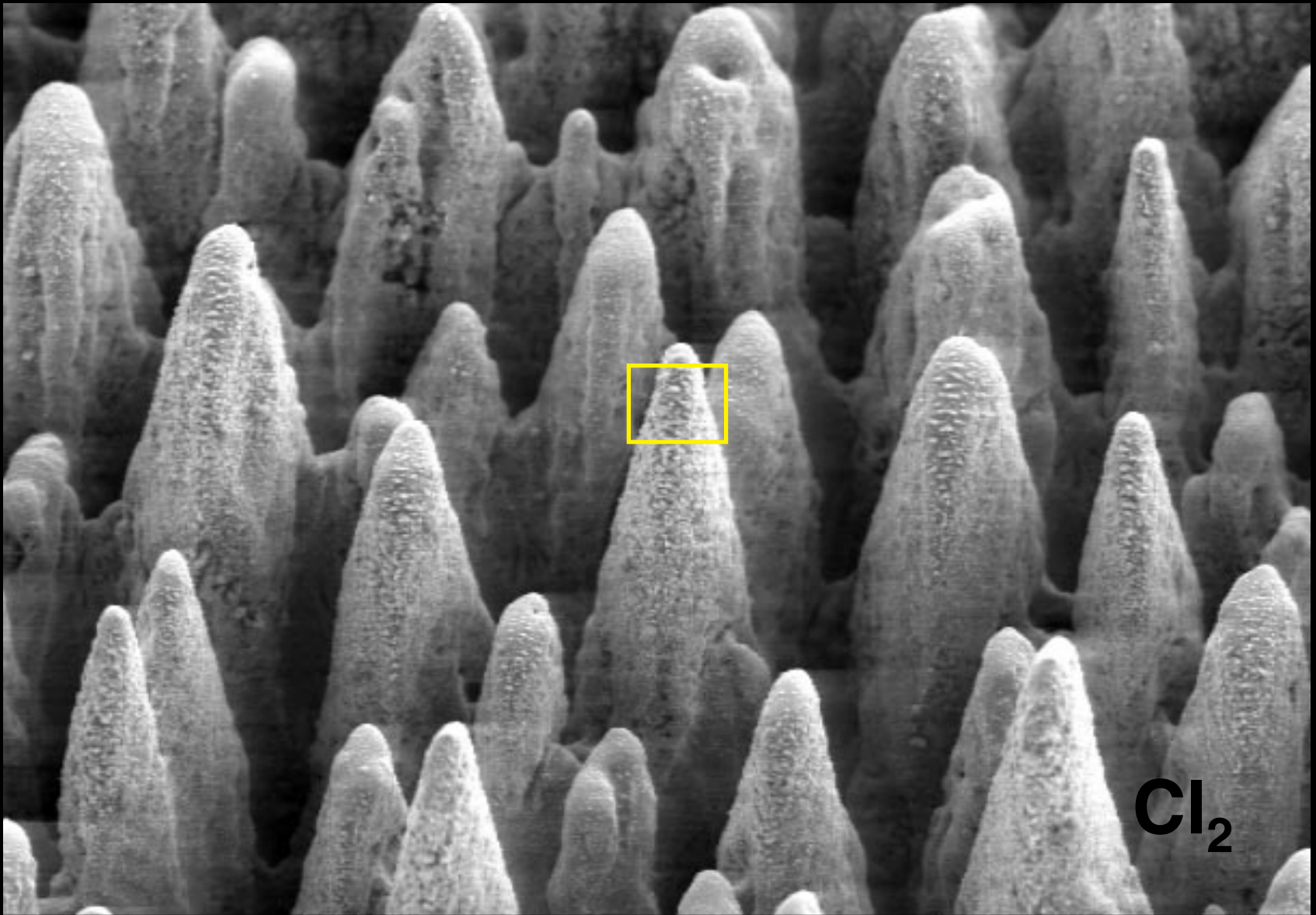


Cl<sub>2</sub>

x3000  
#34  
512 x 480

10 μm  
10/18 Cl2 #3

4.00kV  
11/6/00  
CL2#3-1.TIF  
12mm

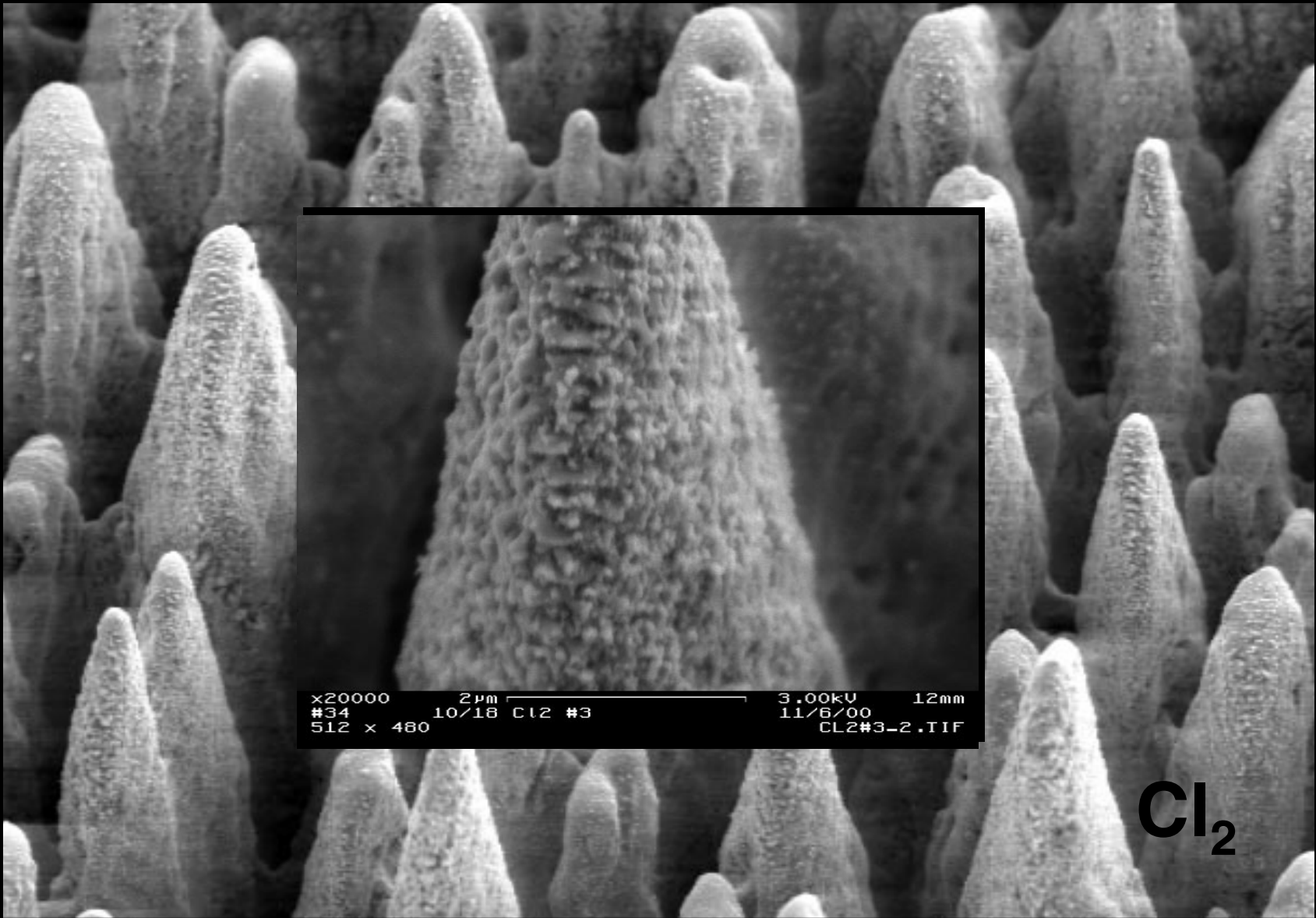


Cl<sub>2</sub>

x3000  
#34  
512 x 480

10 μm  
10/18 Cl2 #3

4.00kV  
11/6/00  
CL2#3-1.TIF  
12mm

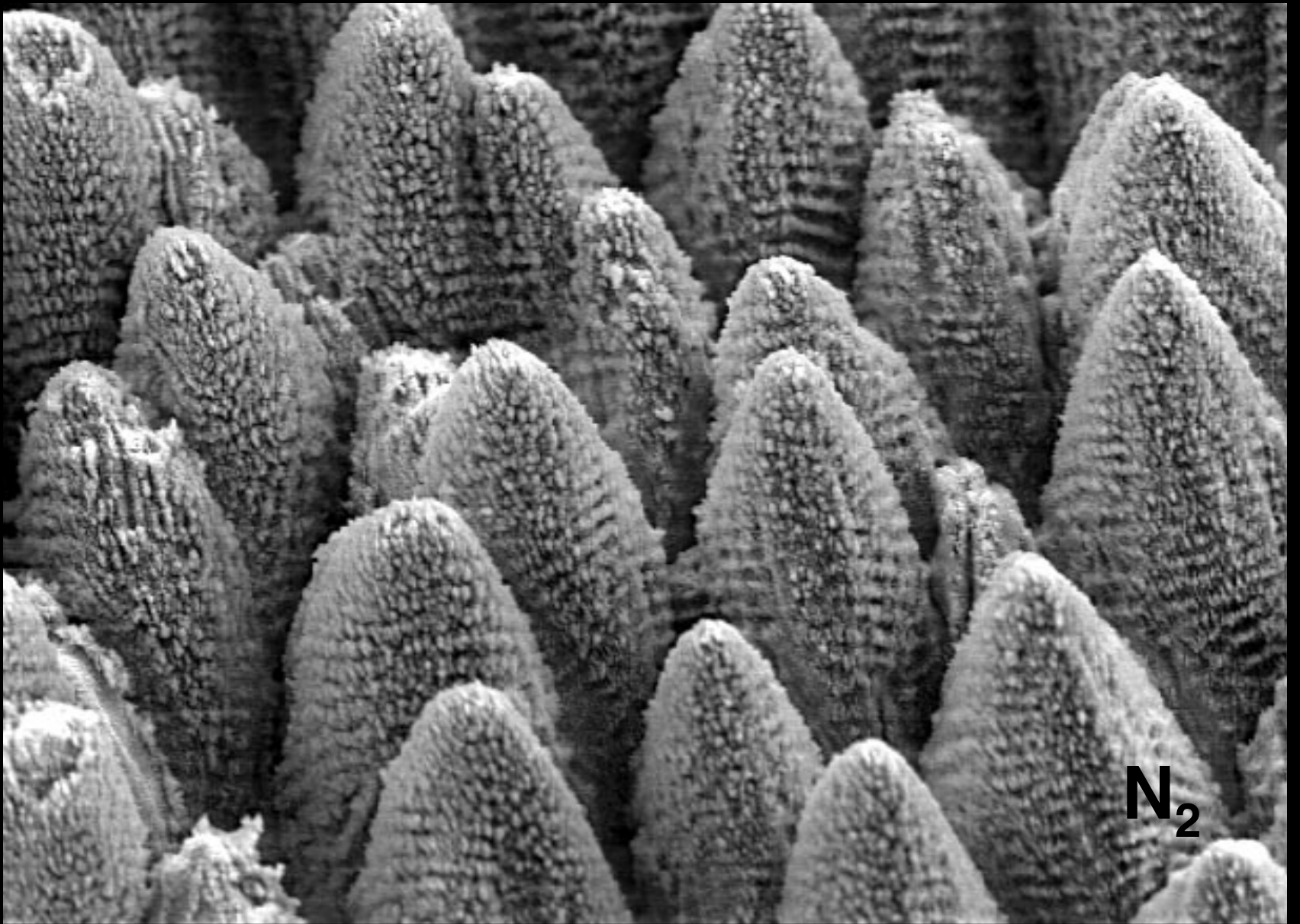


x20000 2 μm 3.00kV 12mm  
#34 10/18 Cl2 #3 11/6/00  
512 x 480 CL2#3-2.TIF

Cl<sub>2</sub>

x3000 10 μm 4.00kV 12mm  
#34 10/18 Cl2 #3 11/6/00  
512 x 480 CL2#3-1.TIF





$N_2$

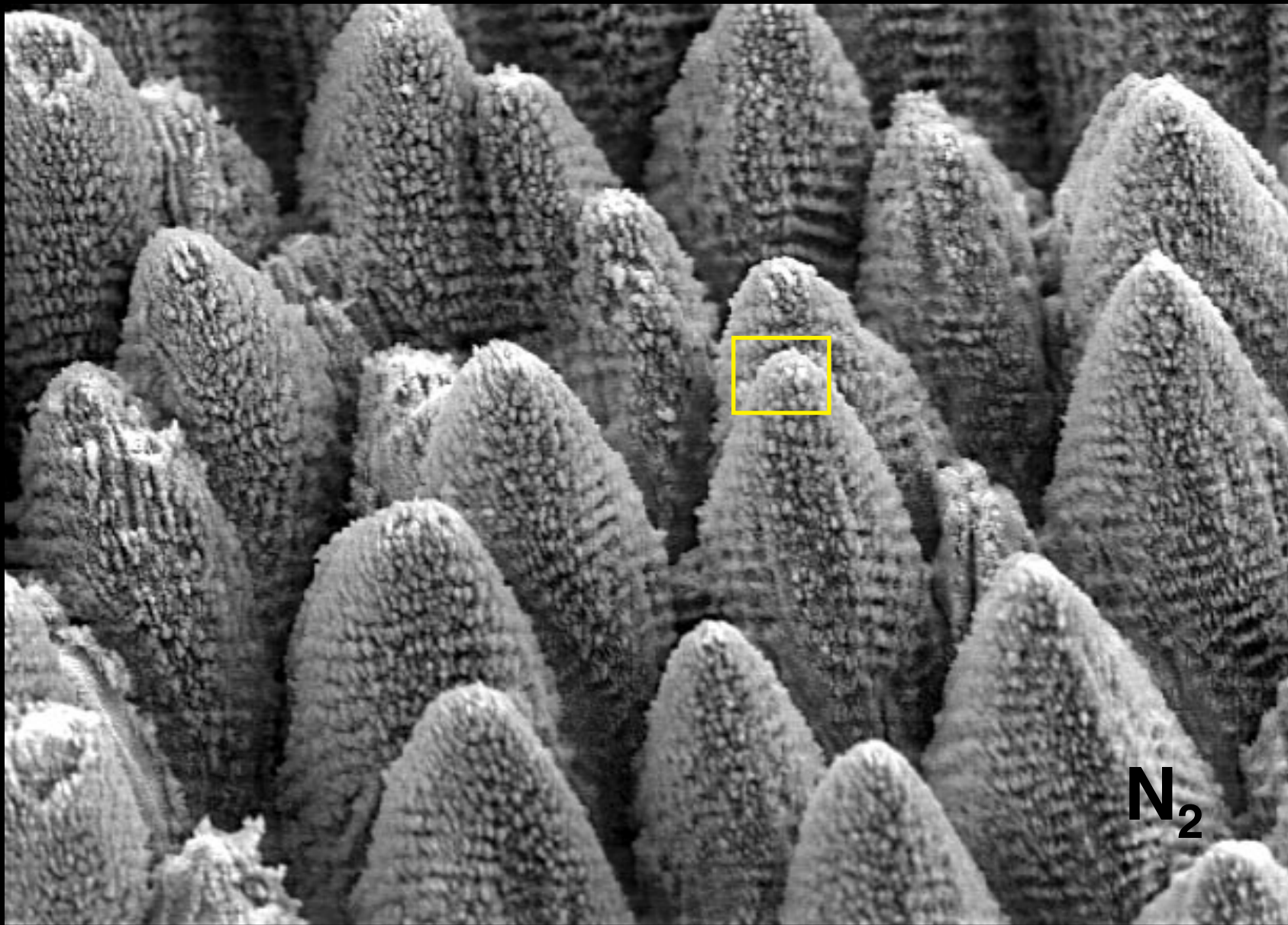
x3000  
#240

10  $\mu$ m

5kV

14mm





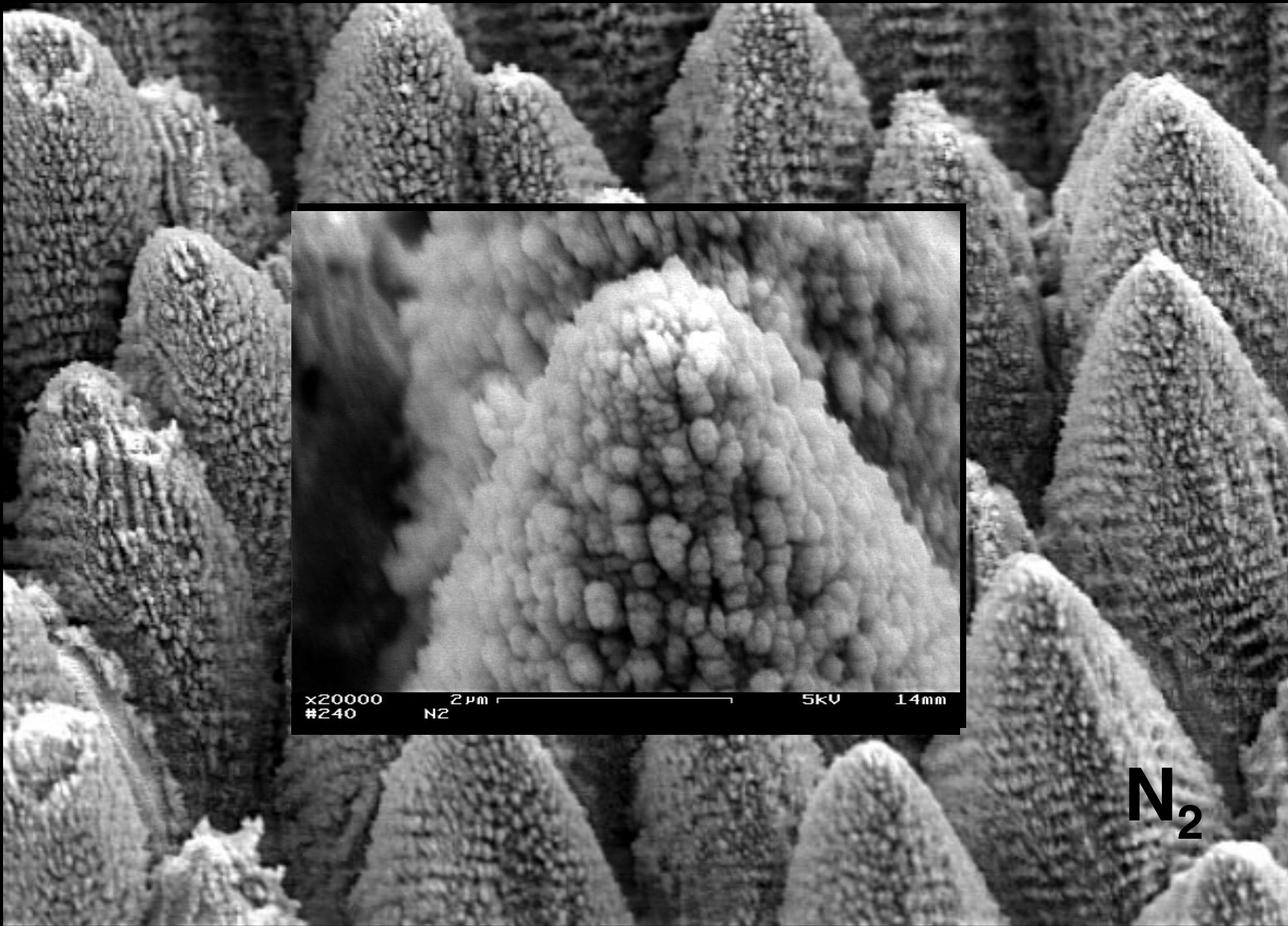
$N_2$

x3000  
#240

10  $\mu$ m

5kV

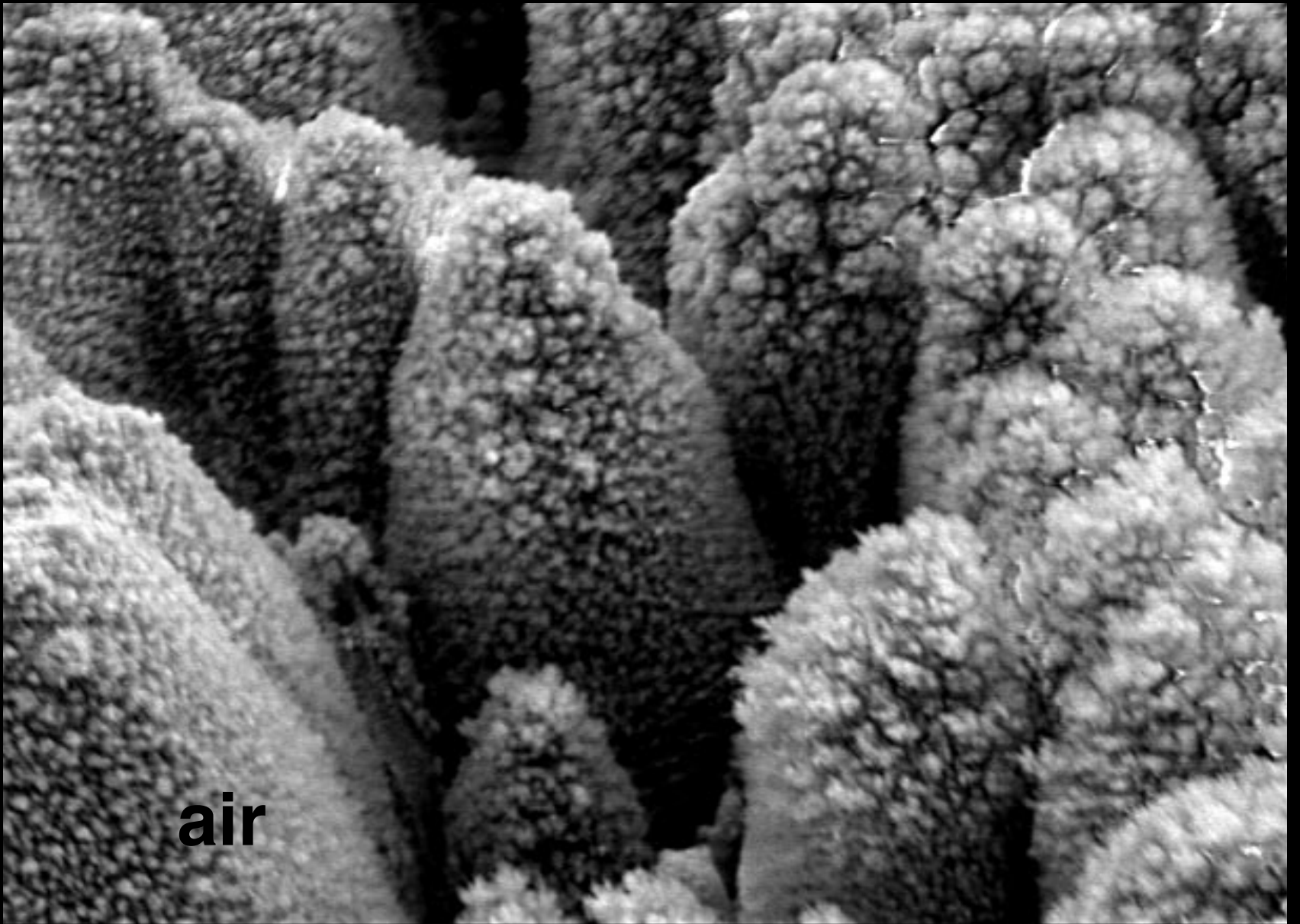
14mm



x20000 2 μm 5kV 14mm  
#240 N2

N<sub>2</sub>

x3000 10 μm 5kV 14mm  
#240 N2



air

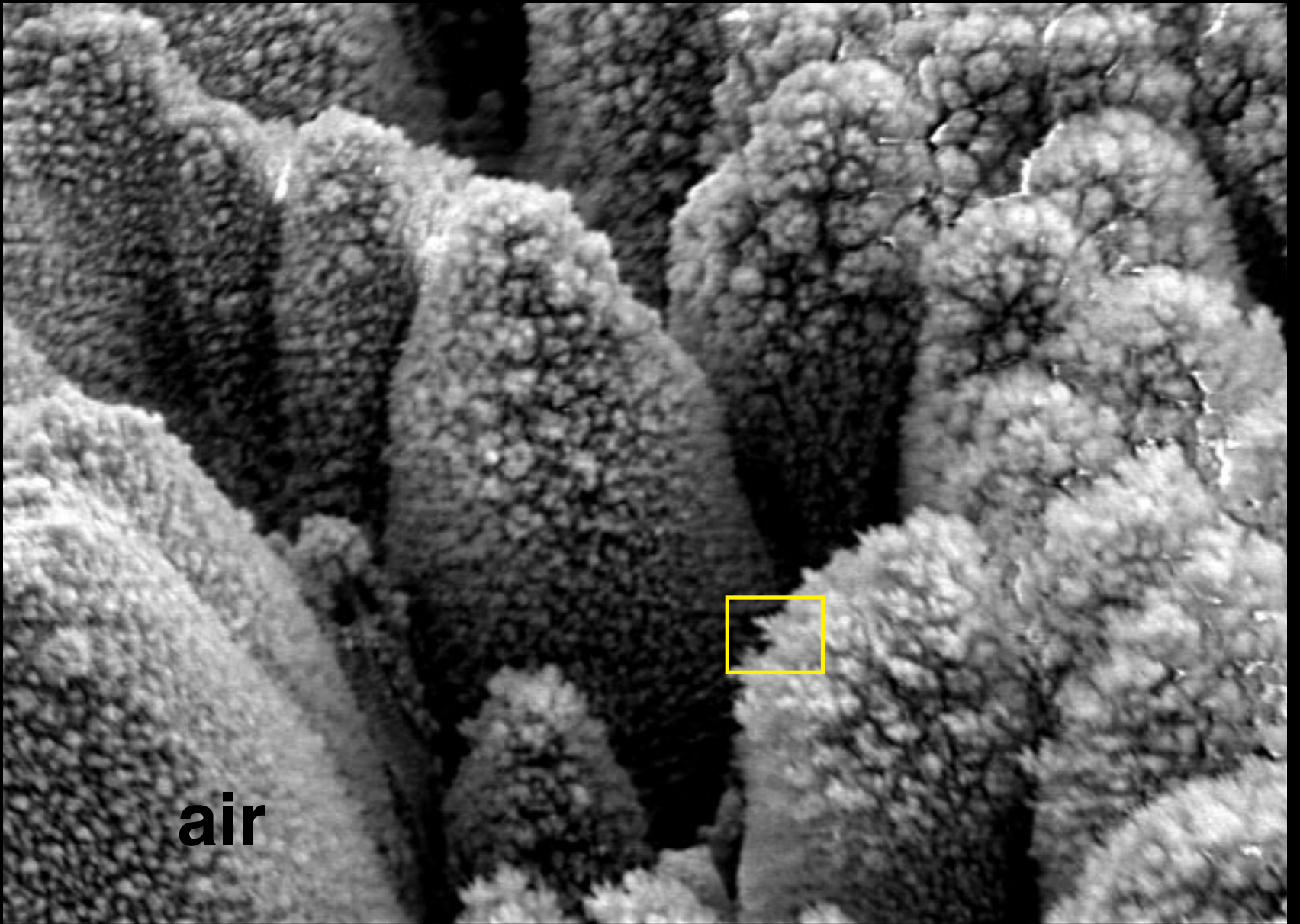
x3000  
#240

10  $\mu$ m  
SF6

5kV

14mm





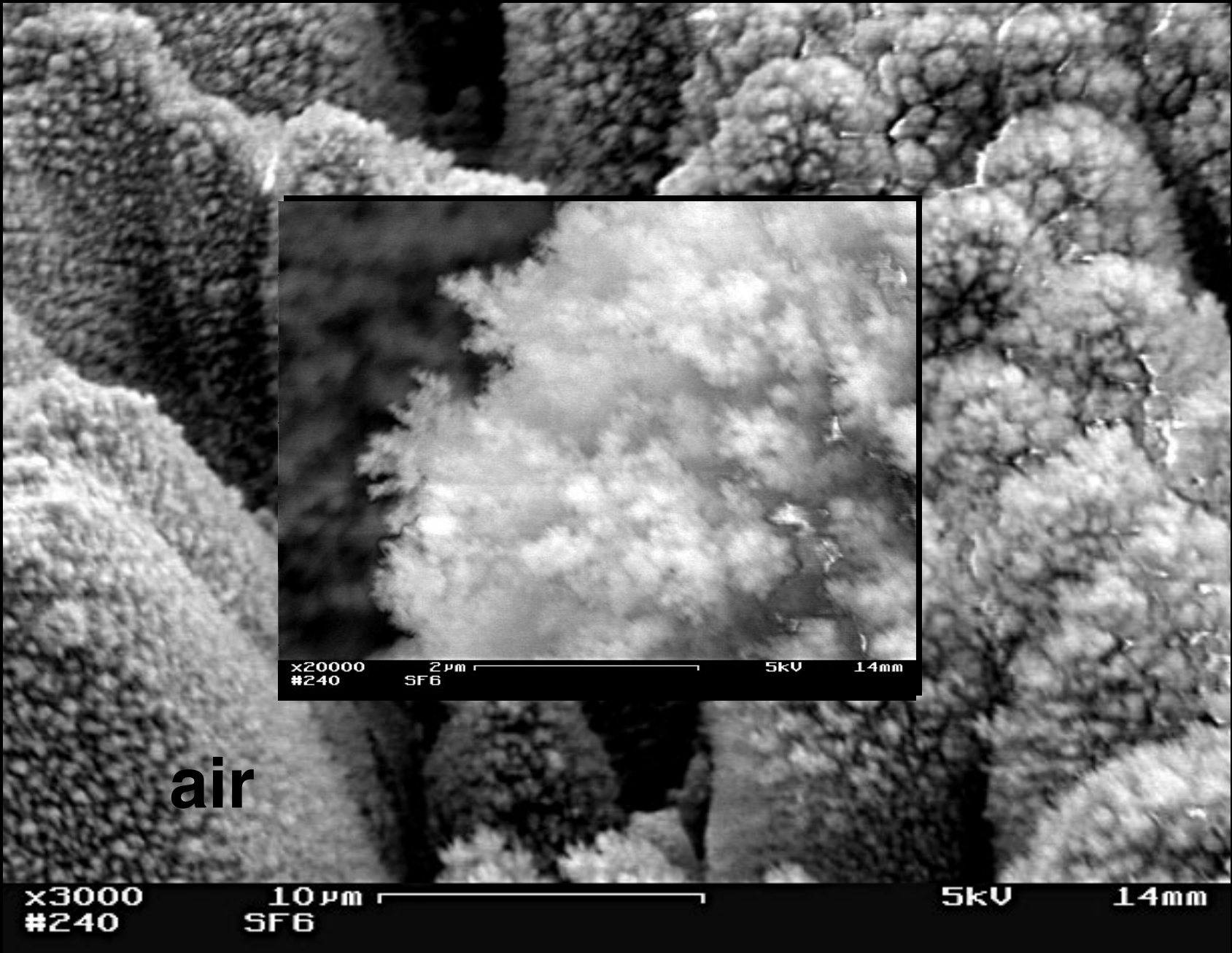
air

x3000  
#240

10  $\mu$ m  
SF6

5kV

14mm



air

x3000  
#240

10  $\mu$ m  
SF6

5kV

14mm

x20000  
#240

2  $\mu$ m  
SF6

5kV

14mm

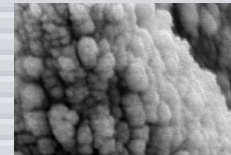
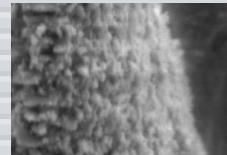
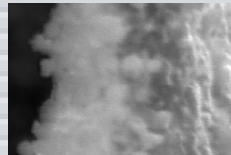
# Structural and chemical analysis

---

	$\text{SF}_6$	$\text{Cl}_2$	$\text{N}_2$	air
IR absorption	high	medium	low	low
field emission	high	low	medium	low
SIMS	high S	?	?	high O

---

nanostructure





## *Structural and chemical analysis*

- ▶ **significant incorporation of ambient species**
- ▶ **nanostructured surface layer**
- ▶ **sulfur content correlates with IR absorption**

# Outline

- ▶ **Properties**
- ▶ **Structural and chemical analysis**
- ▶ **Outlook**



# Outlook

*New Scientist* 13, 34 (2001)

A forest of silicon spikes could revolutionise solar cells and give you painless injections. **Bruce Schechter** peers into the mysterious world of black silicon

## TALL, DARK AND STRANGER

WE ALL love stories of serendipity. They seem to hark back to a time when a fogged plate or a filthy Petri dish today, when

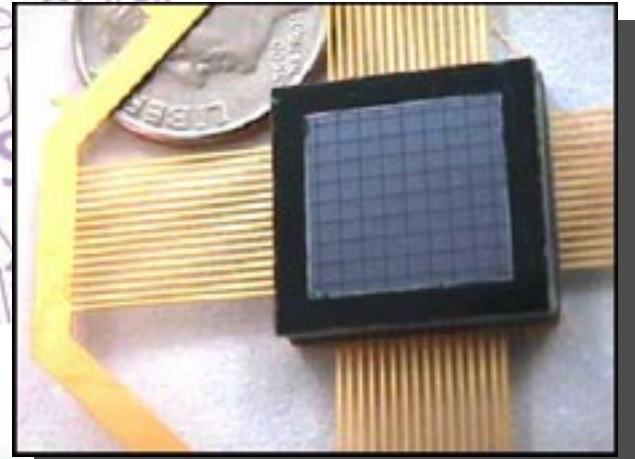
semiconductors with a powerful laser. In the early 1990s, Mazur's was the first academic lab in the world to get its hands on a femtosecond laser. This device produces pulses of light that are hundreds of times brighter than the Sun. and extremely

around the laboratory," he claims. Well, it was almost the only reason a short laser pulse will break down into sulphur and fluorine radicals, which will attack a silicon substrate. "Hydrogen fluoride is used to etch silicon. I thought maybe the SF<sub>6</sub> would do it and then the fluorine would so with the silicon," Mazur explains. "I was more than

# Outlook

## ► detector technology

A forest of silicon spikes could revolutionise solar cells and give you injections. Bruce peers into the mysterious world of black silicon



# TALL, DARK AND STRANGER

We'll all have stories of weird days. They seem to lack back to the time when a dog bit a child or a fire broke out in a room when

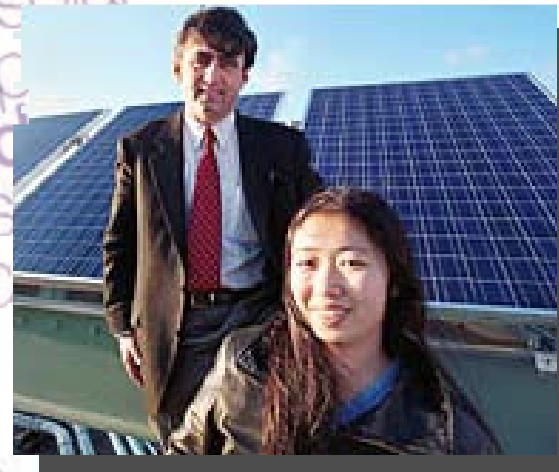
semiconductors with a powerful laser. In the early 1960s, Major's was the first academic lab in the world to do so. It was a hard-core attempt to see if there were any advantages to light that included wavelengths shorter than the Sun's and extremely

around the "laser" line. He claims that it was a great idea to make a laser that would break down into sulfur and fluorine radicals. That's the way to make a laser. But there's a problem: the silicon would have to be made of silicon. Most ex-

# Outlook

- ▶ detector technology
- ▶ solar cells

A forest of silicon spikes could revolutionise solar cells and give you power injections. Bruce S... peers into the mysterious world of black silicon



## TALL, DARK AND STRANGER

...around the laboratory. "He claims...  
...the early 1960s. Marz's was the first...  
...hard over a ten-year period. This...  
...sunlight is much brighter than the Sun...  
...and extremely



# Outlook

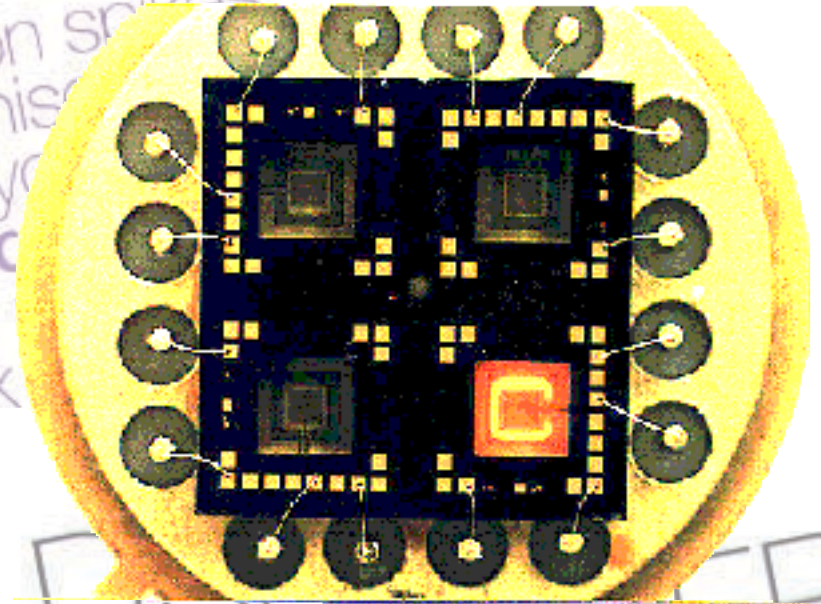
- ▶ detector technology
- ▶ solar cells
- ▶ display technology



# Outlook

- ▶ detector technology
- ▶ solar cells
- ▶ display technology
- ▶ sensors

A forest of silicon spikes  
could revolutionise  
cells and give you  
injections. Bruce  
peers into the  
world of black



TALL, DARK  
AND STRANGER

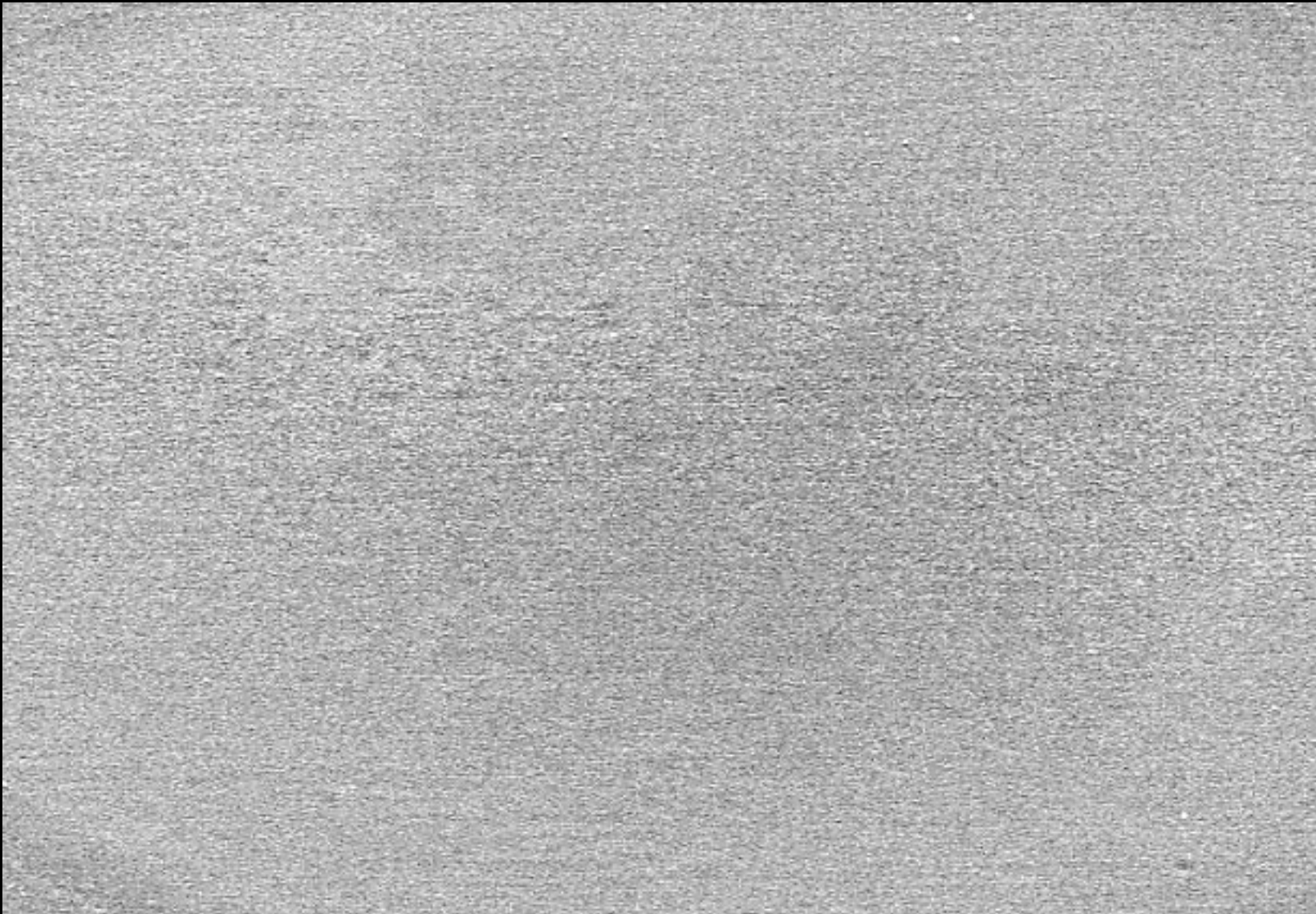
We'll all have stories of weird jobs. This  
one to ask for a long time when a legend  
of a life's time. You don't  
know when

semiconductors with a potential use for  
the early 1960s. Major's was the first  
academic lab in the world to do so.  
hard over a ten-year period. This was  
an idea as far as light that included  
lighter than the Sun  
and extremely

around the laboratory," he claims.  
With it was a great the only place  
that I've ever seen. It's hard to see  
into a pipe and through the pipes  
with a flashlight. It's a very dark place. But  
there is a light to each silicon  
through the holes. It would be  
one that the silicon would be  
the silicon. Most ex

# *Outlook*

- ▶ **development of spikes**
- ▶ **spike formation through grids**
- ▶ **cell adhesion**
- ▶ **functionalization**



x2000  
#3548  
512 x 480

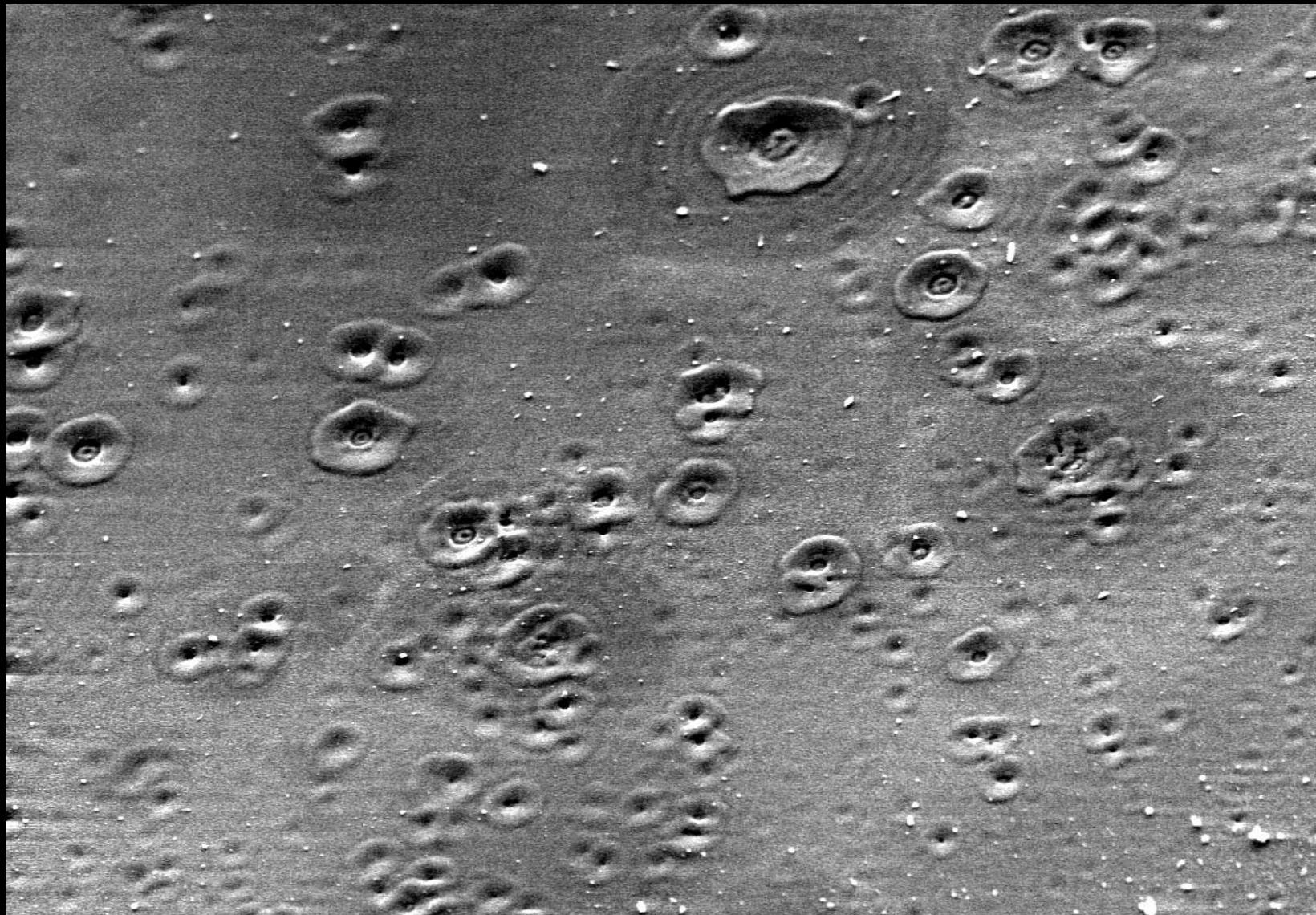
20  $\mu$ m

10kV

15mm

0000





x2000  
#3548  
512 x 480

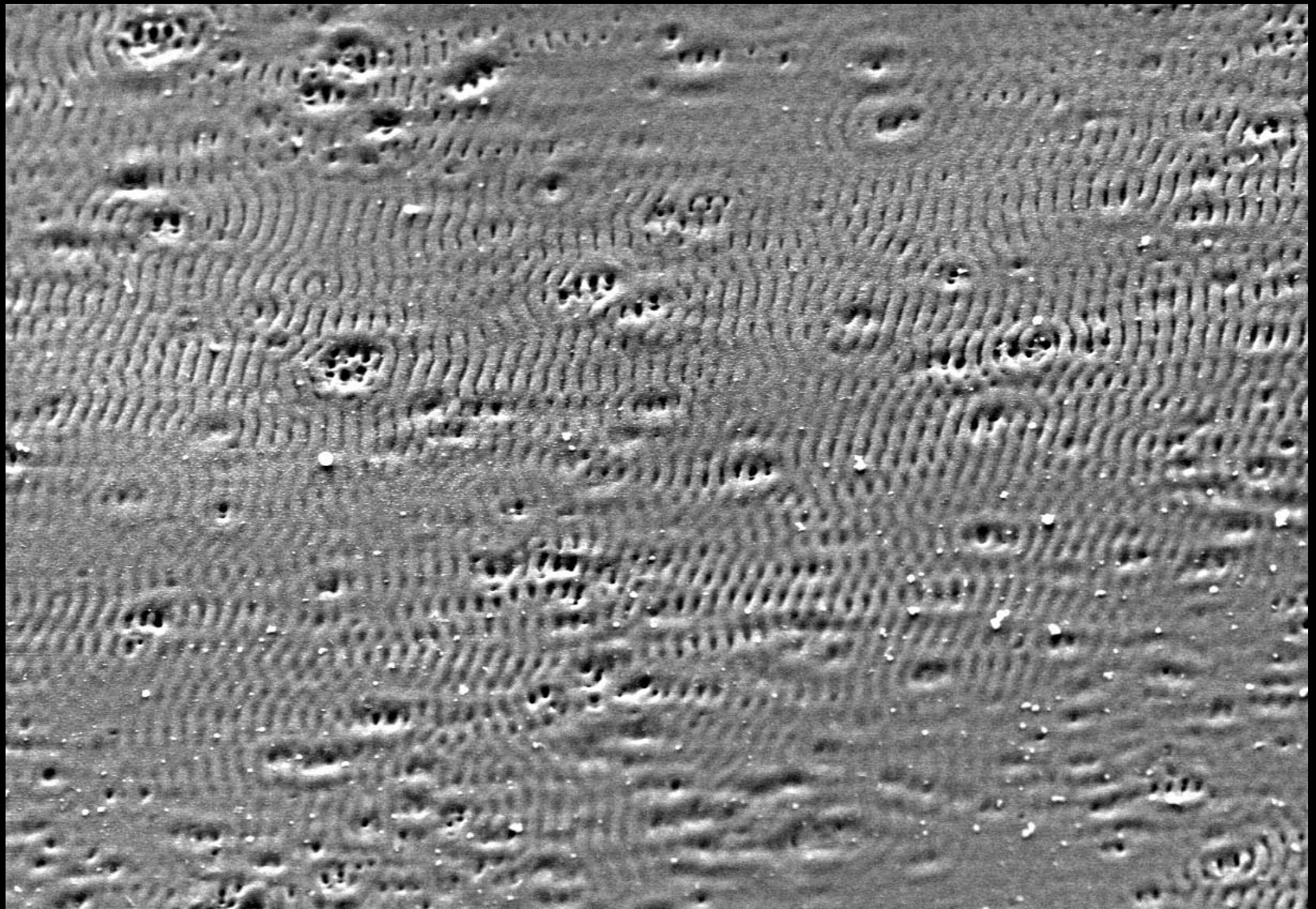
20  $\mu\text{m}$

10kV

15mm

0001





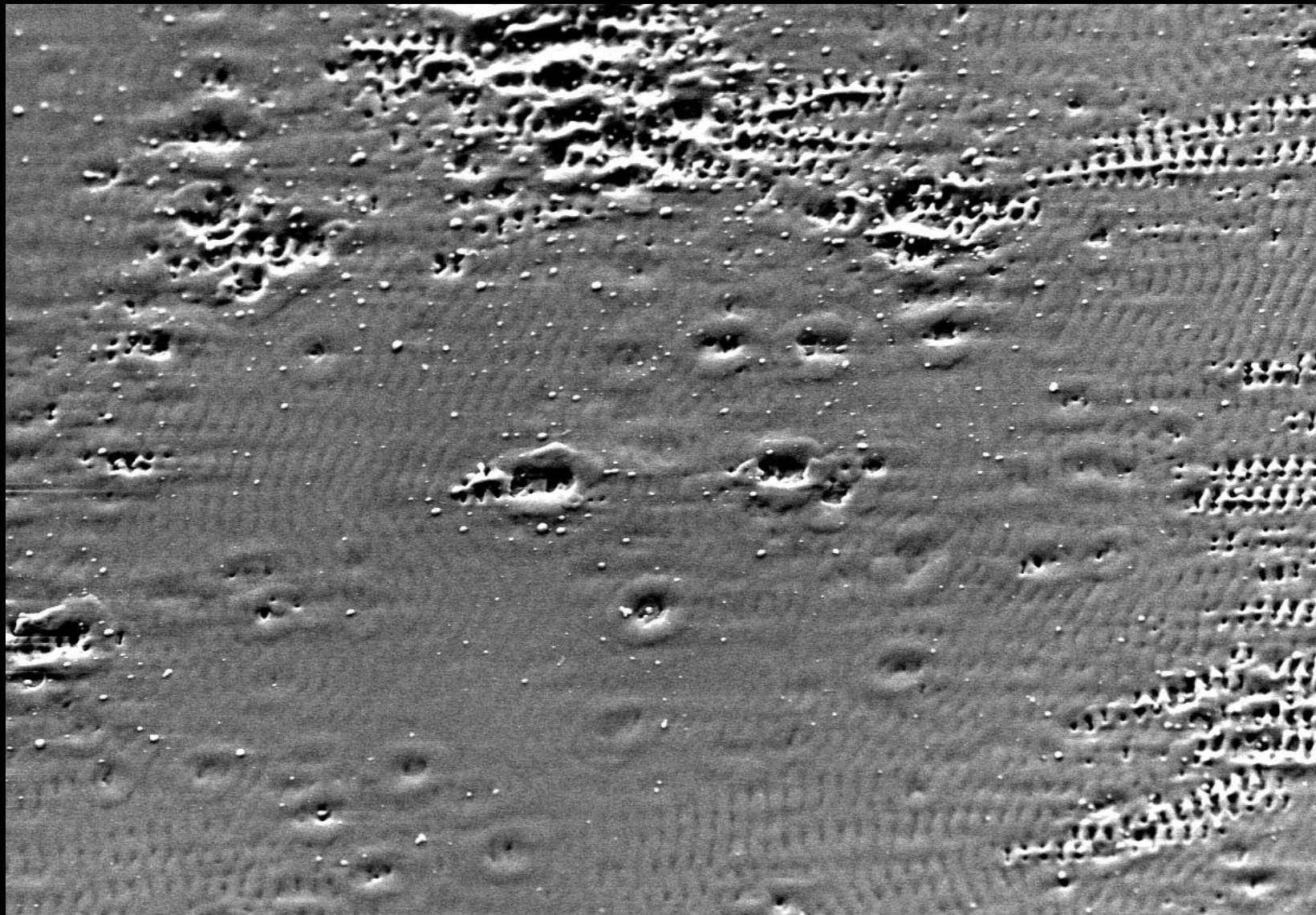
x2000  
#3548  
512 x 480

20  $\mu$ m

10kV

15mm

0002



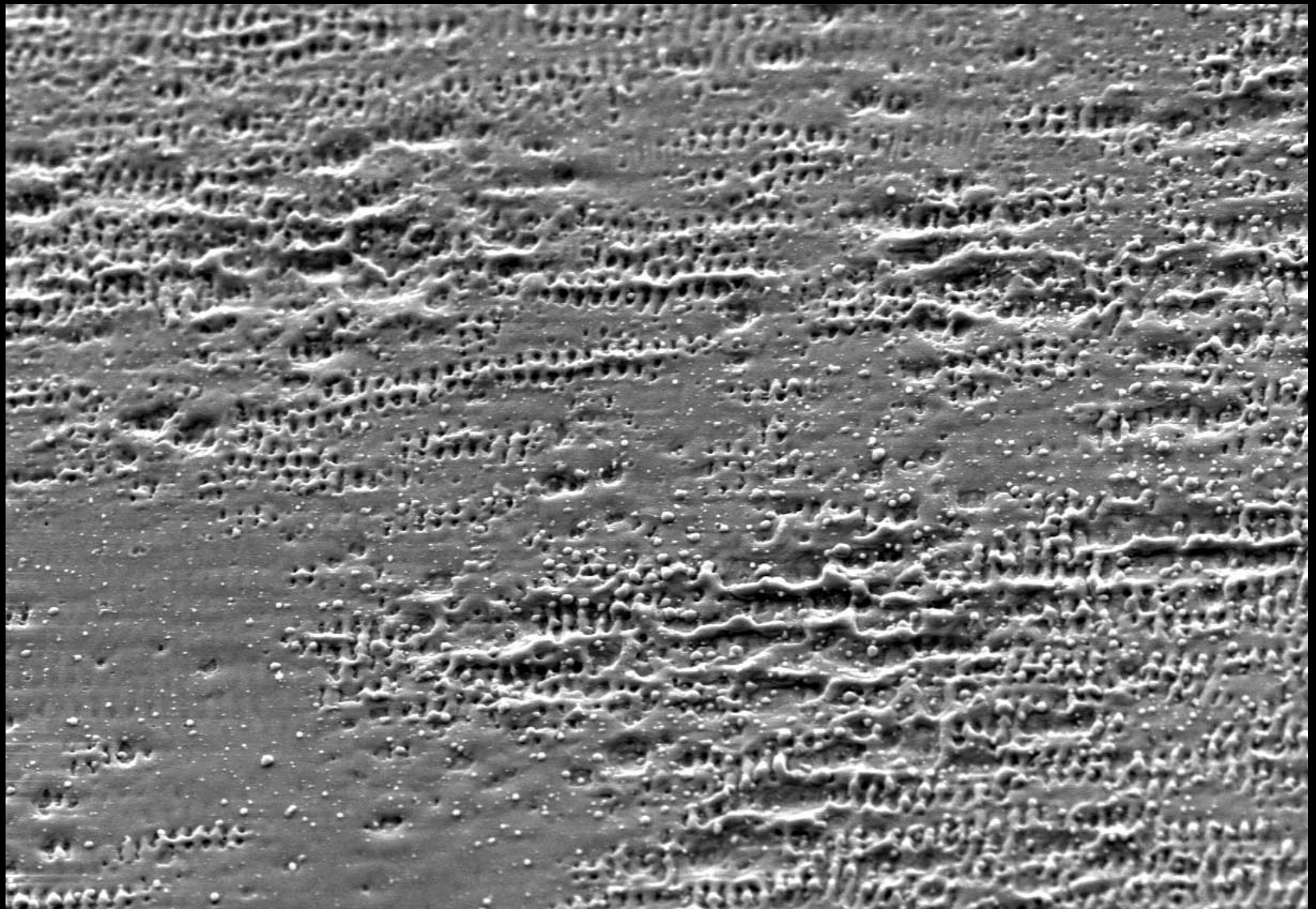
x2000  
#3548  
512 x 480

20  $\mu\text{m}$

10kV

15mm

0003



x2000  
#3548  
512 x 480

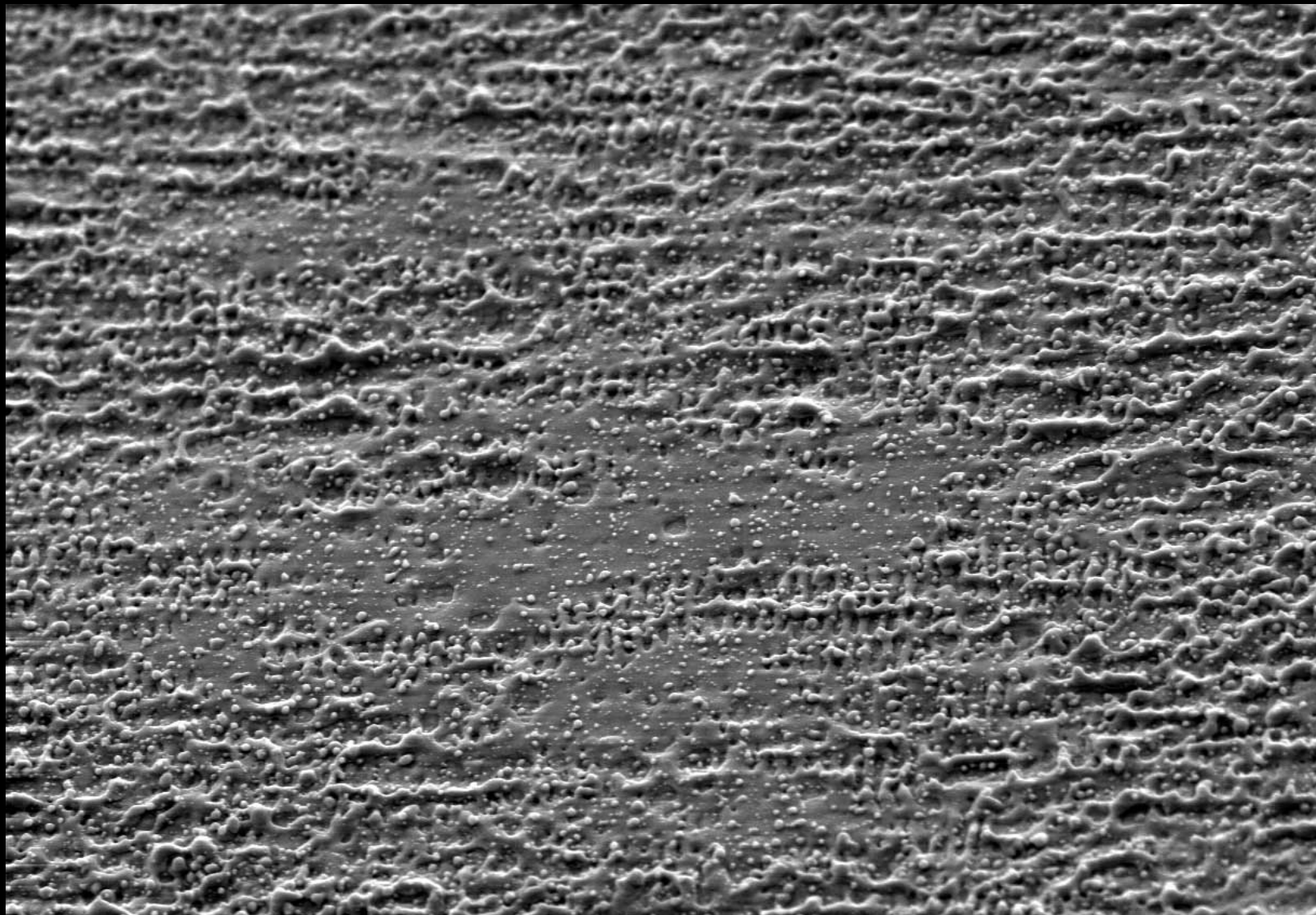
20 μm

10kV

15mm

0004





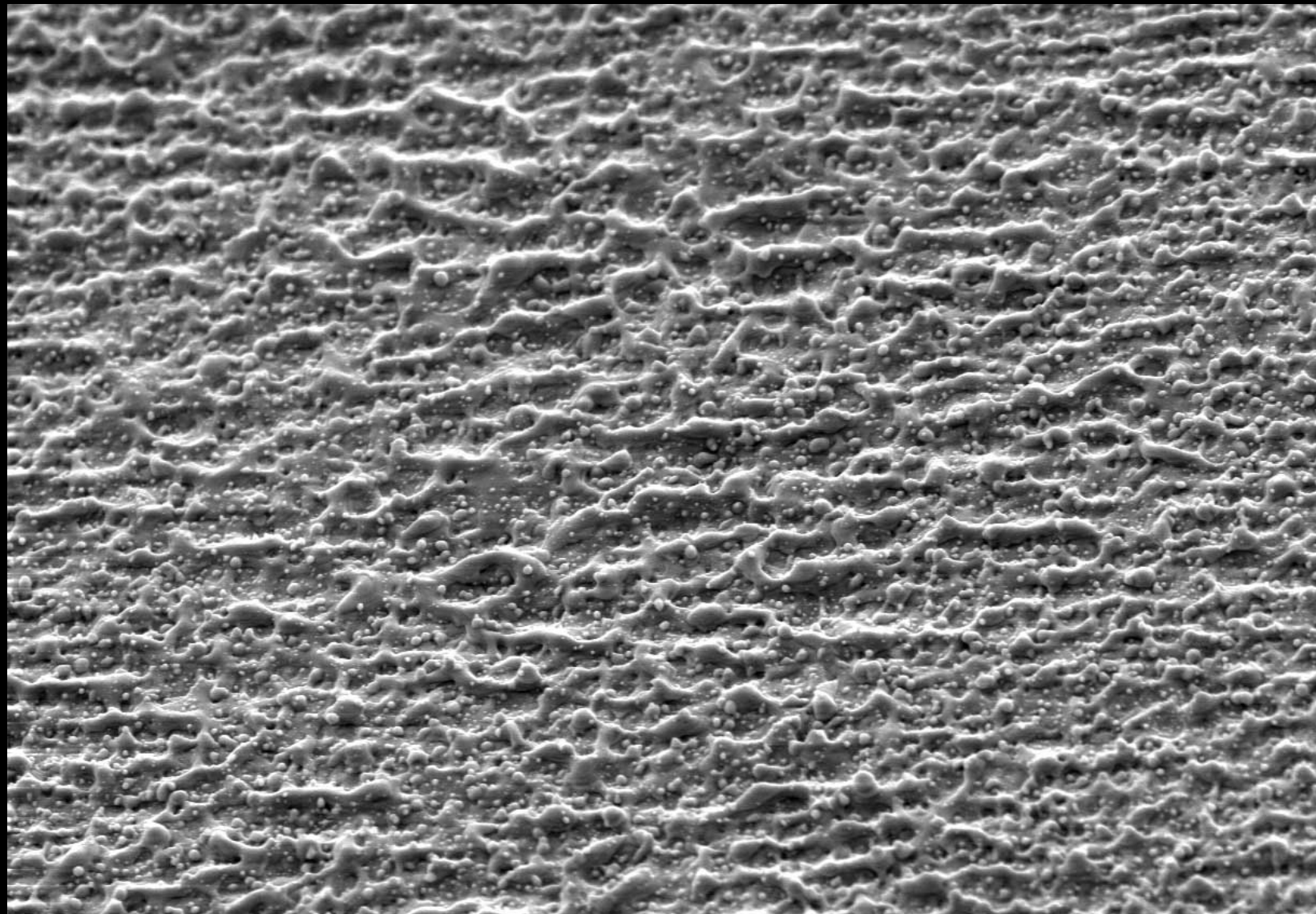
x2000  
#3548  
512 x 480

20 μm

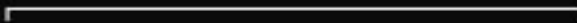
10kV

15mm

0005



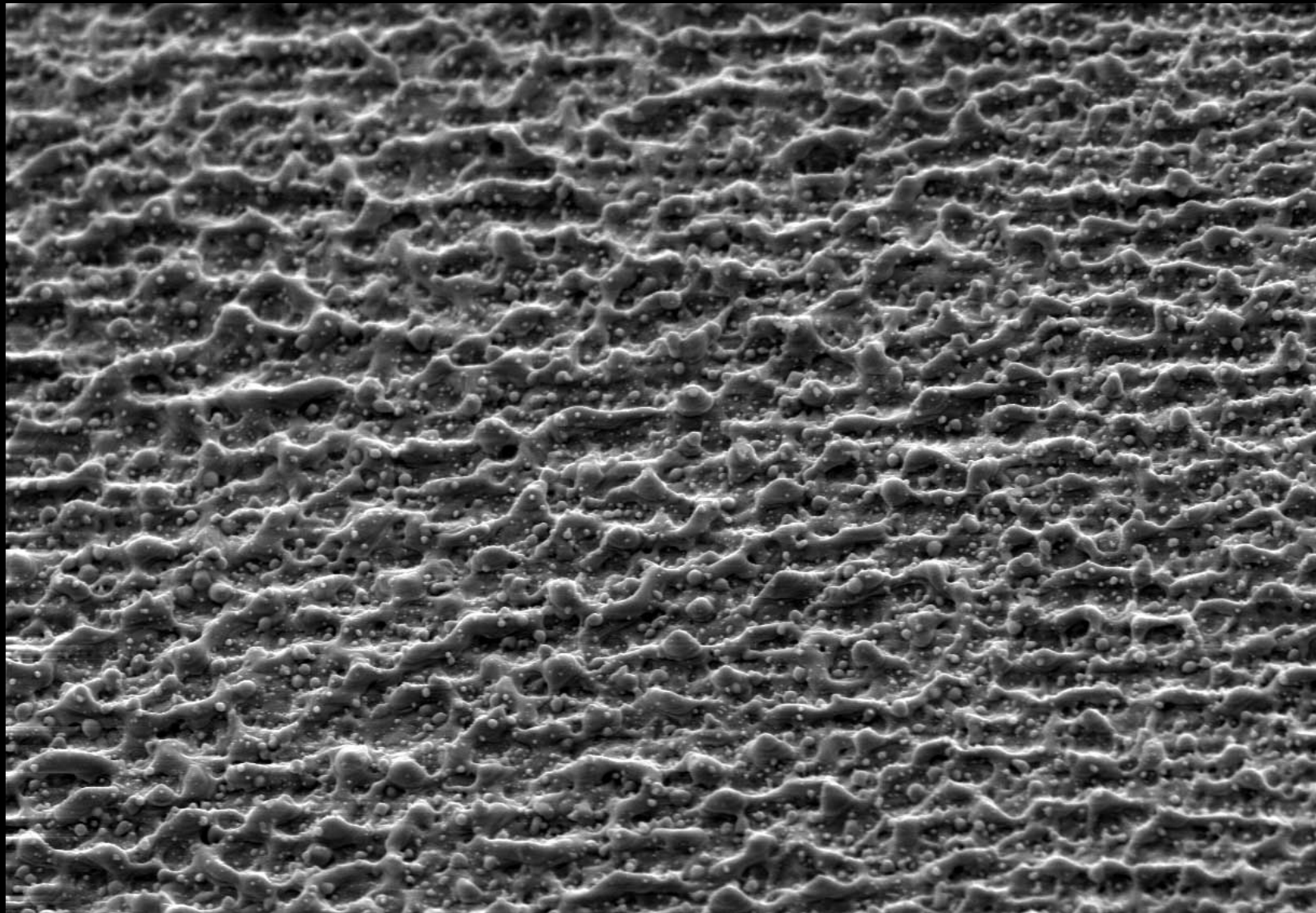
x2000  
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512 x 480

20  $\mu\text{m}$  

10kV

15mm

0006



x2000  
#3548  
512 x 480

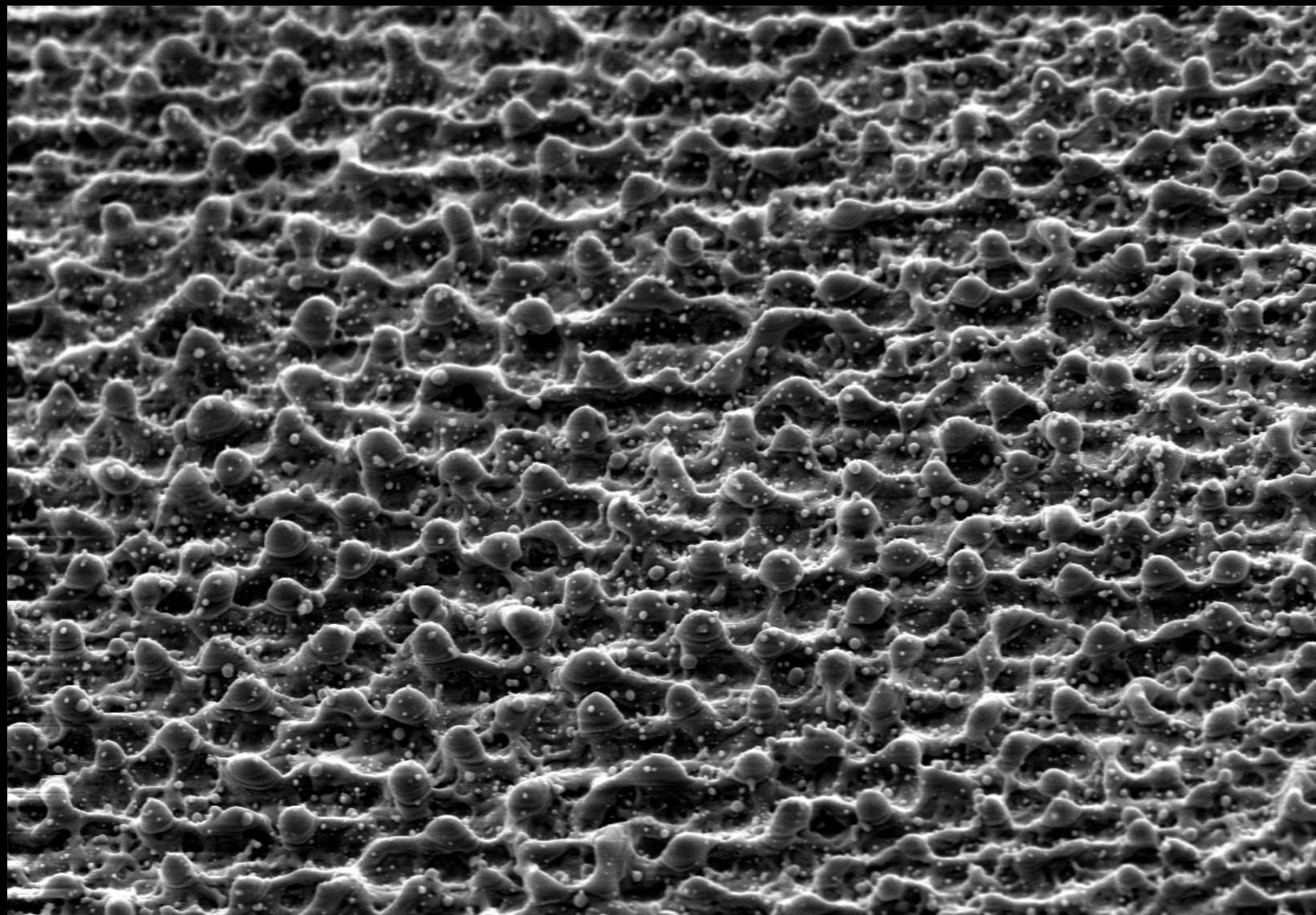
20  $\mu$ m

10kV

15mm

0008





x2000  
#3548  
512 x 480

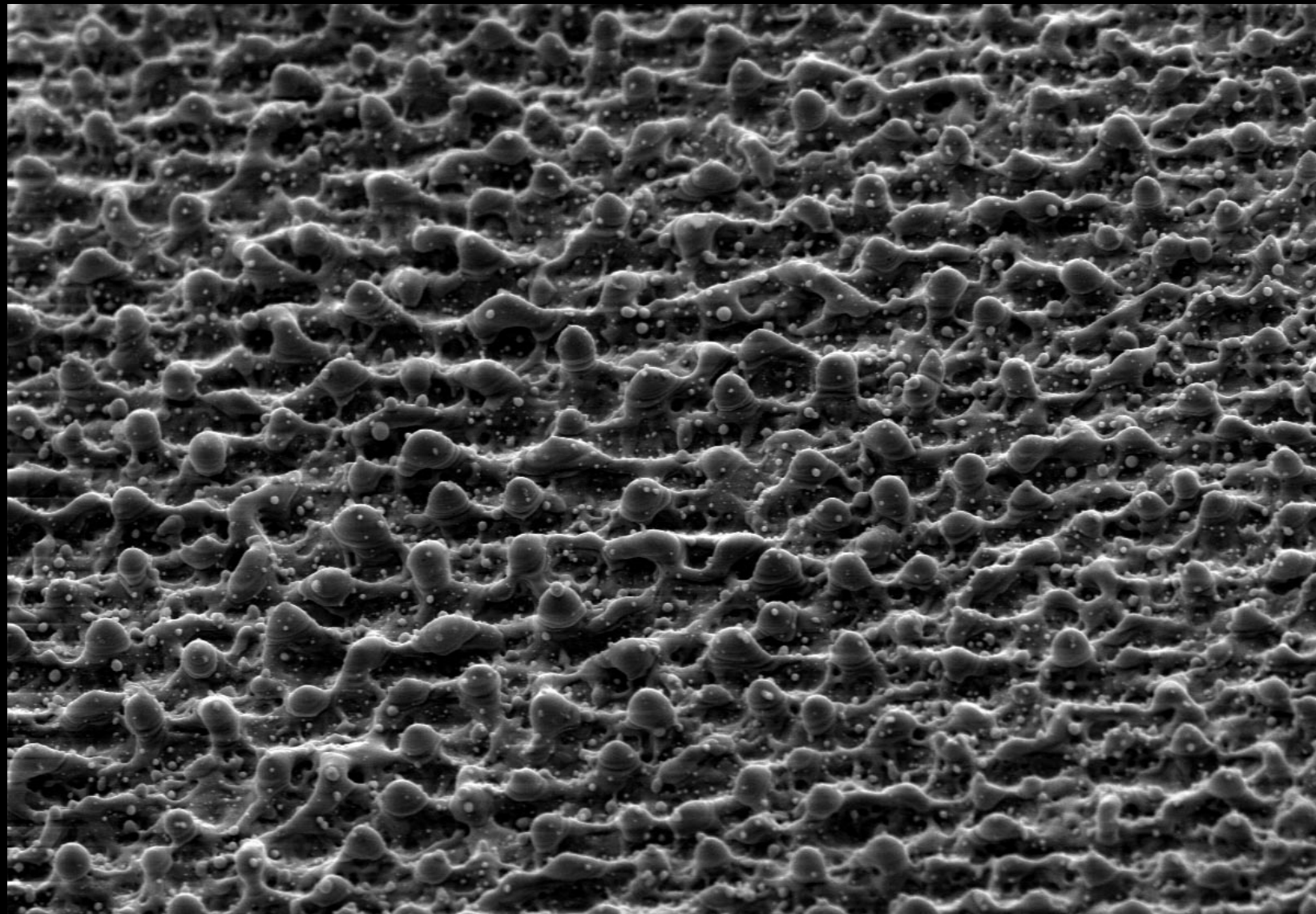
20  $\mu$ m

10kV

15mm

0010





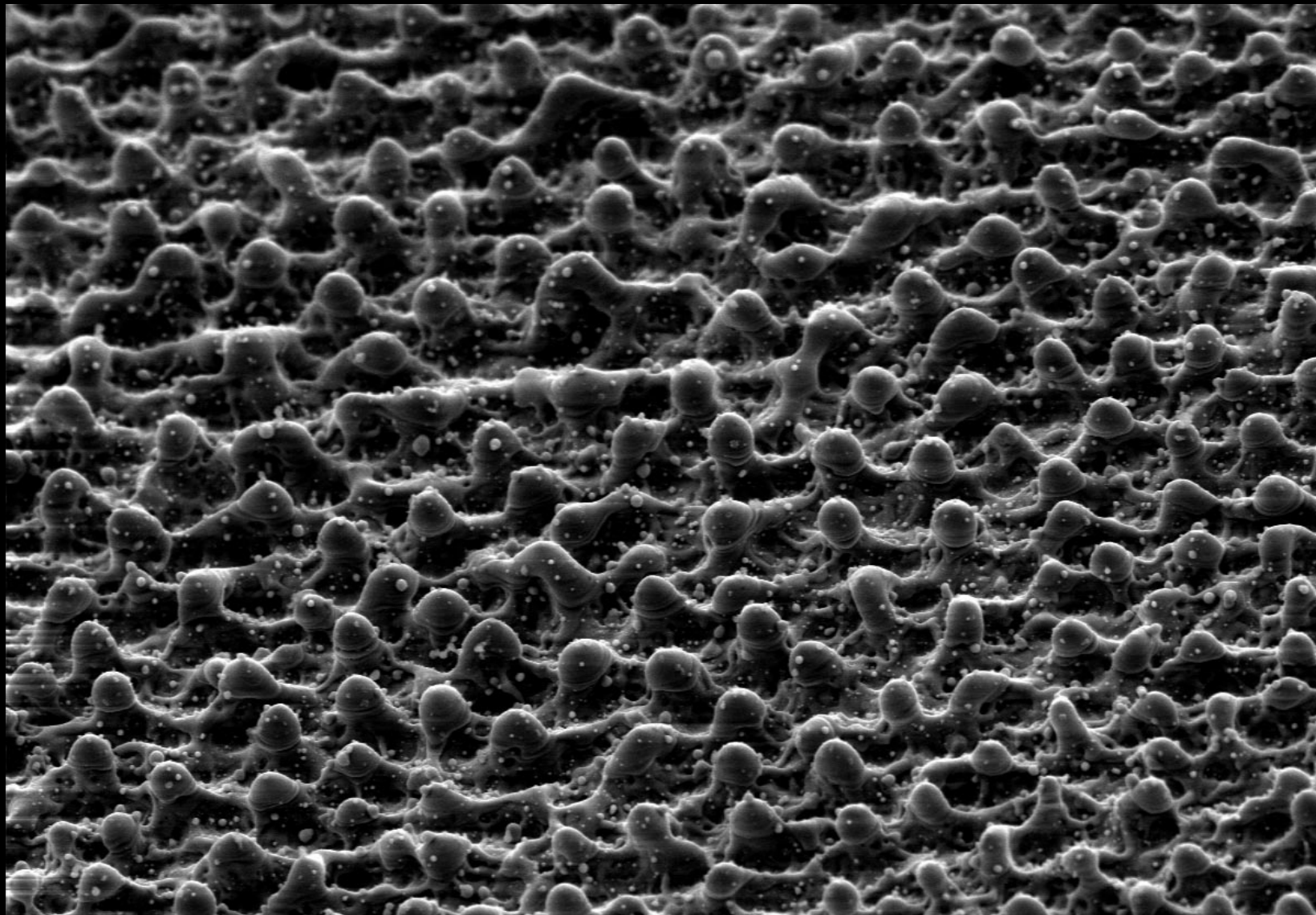
x2000  
#3548  
512 x 480

20  $\mu$ m

10kV

15mm

0012



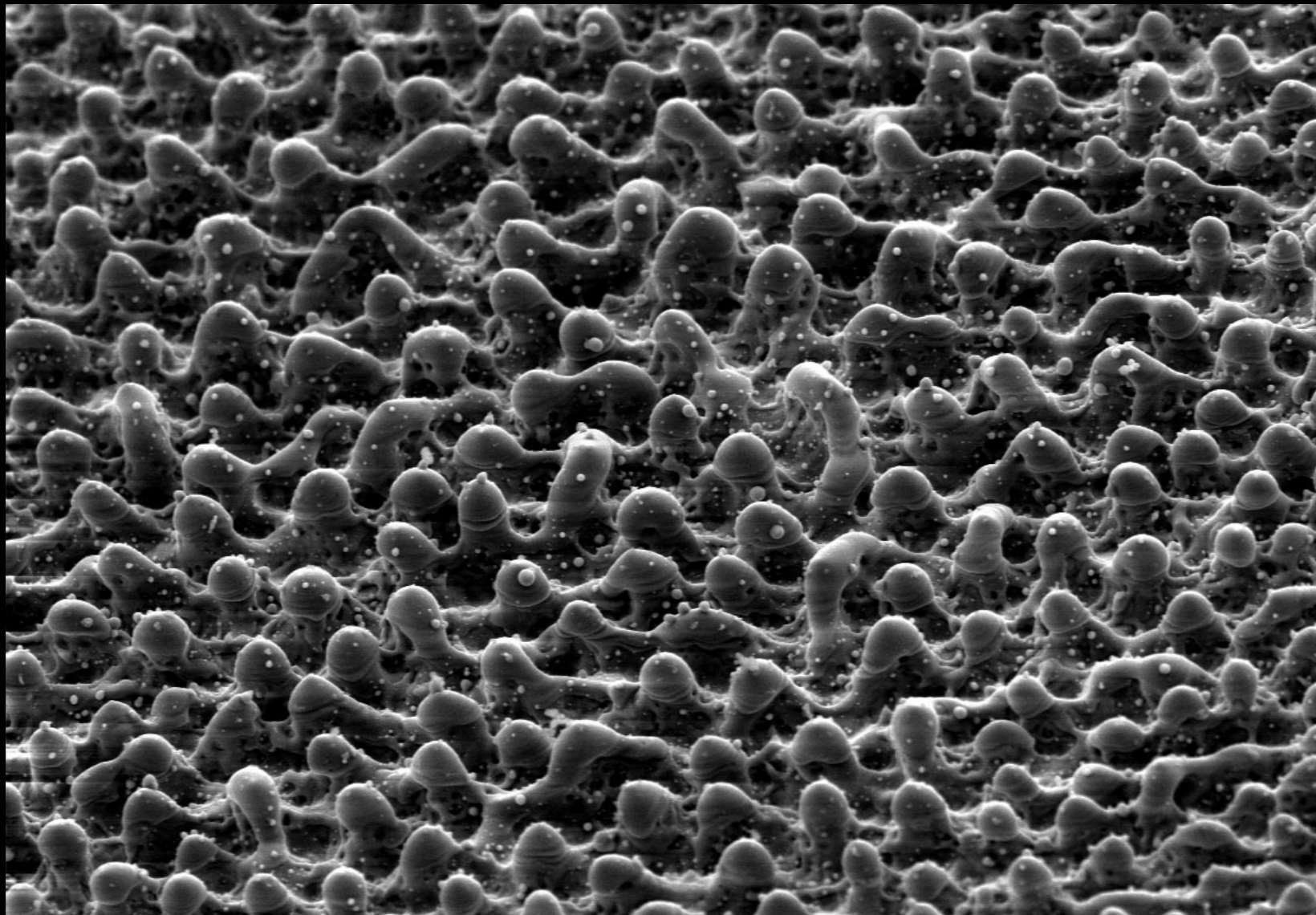
x2000  
#3548  
512 x 480

20  $\mu$ m

10kV

15mm

0015



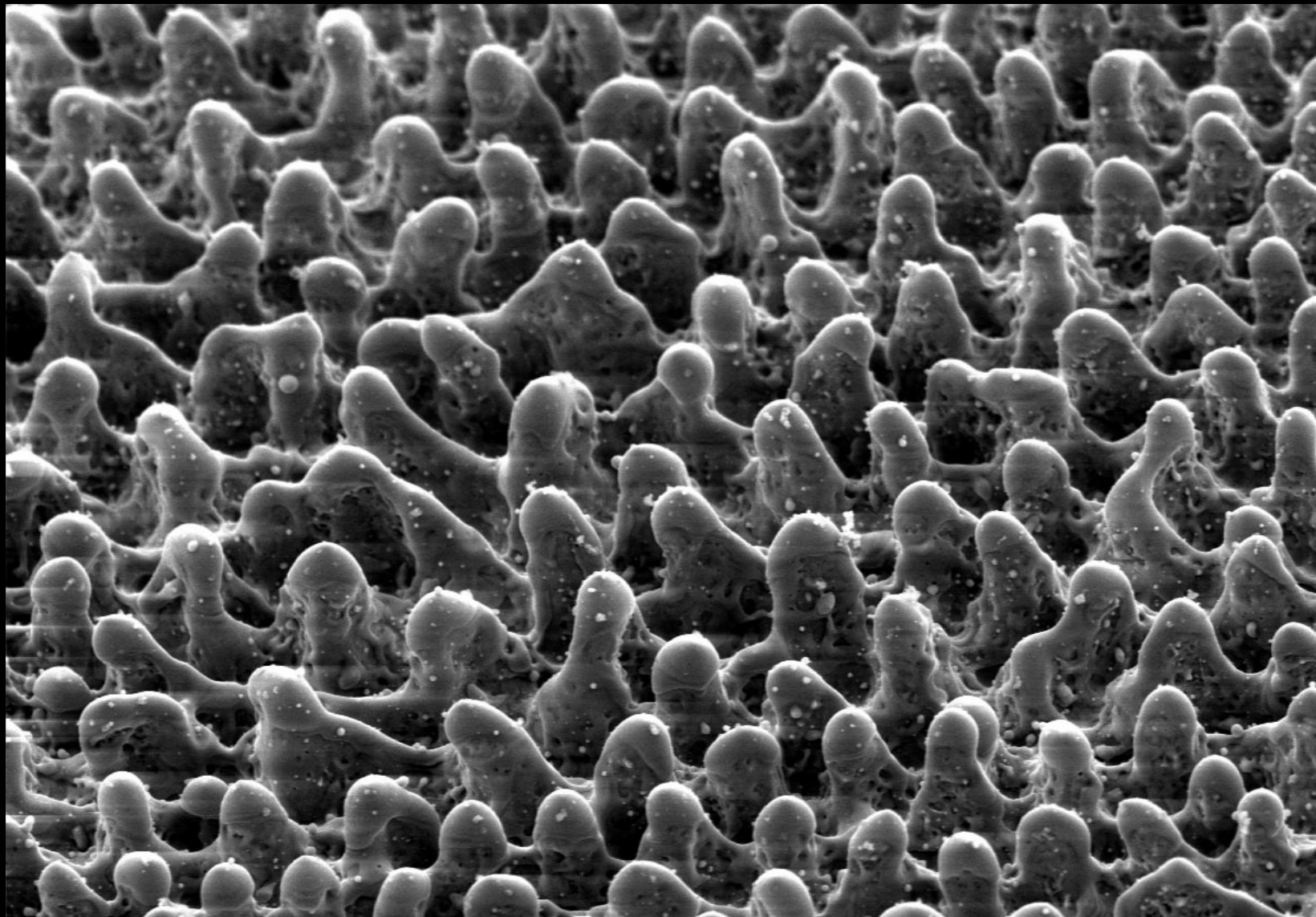
x2000  
#3548  
512 x 480

20  $\mu$ m

10kV

15mm

0020



x2000  
#3548  
512 x 480

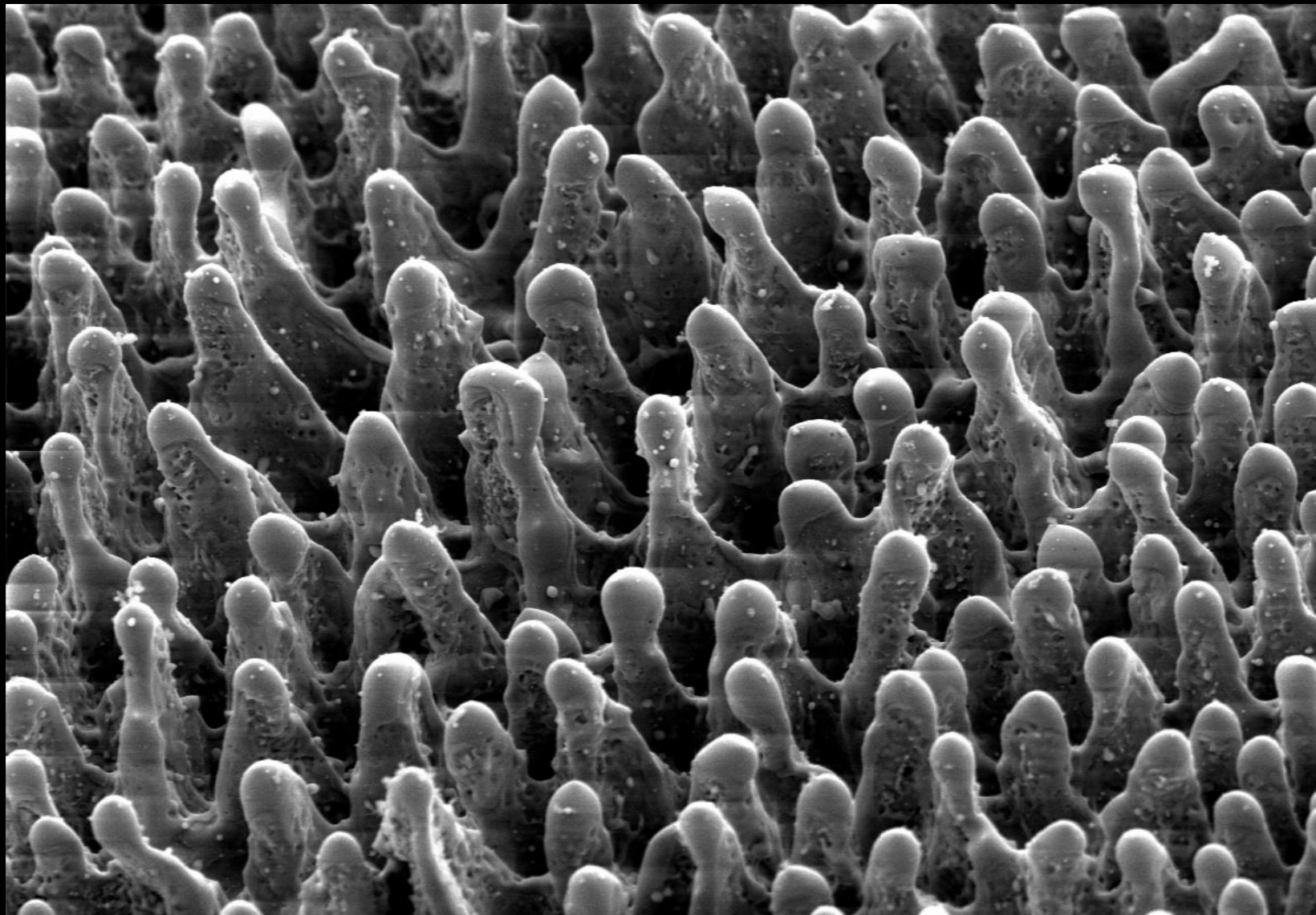
20  $\mu$ m

10kV

15mm

0030





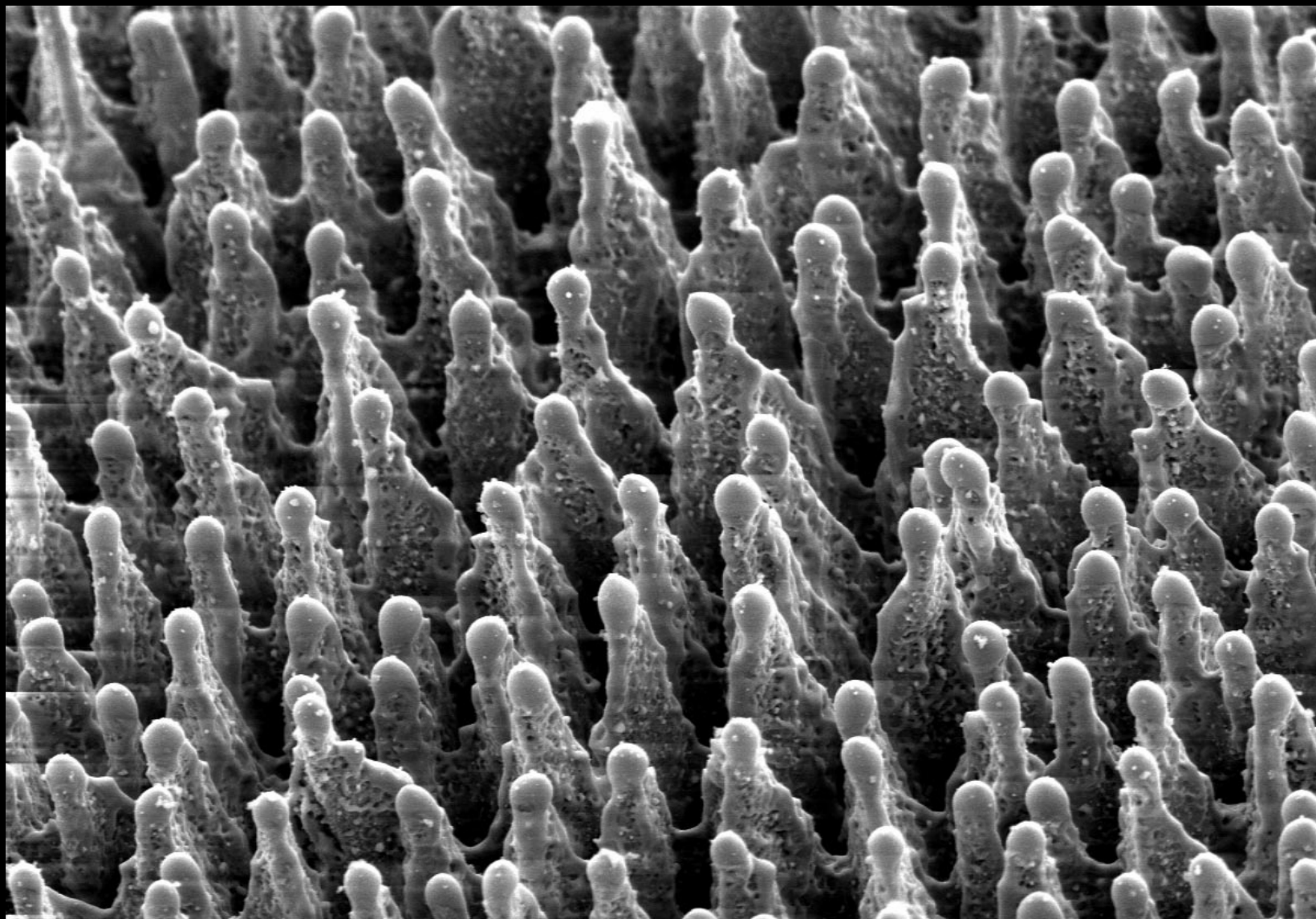
x2000  
#3548  
512 x 480

20  $\mu$ m

10kV

15mm

0050



x2000  
#3548  
512 x 480

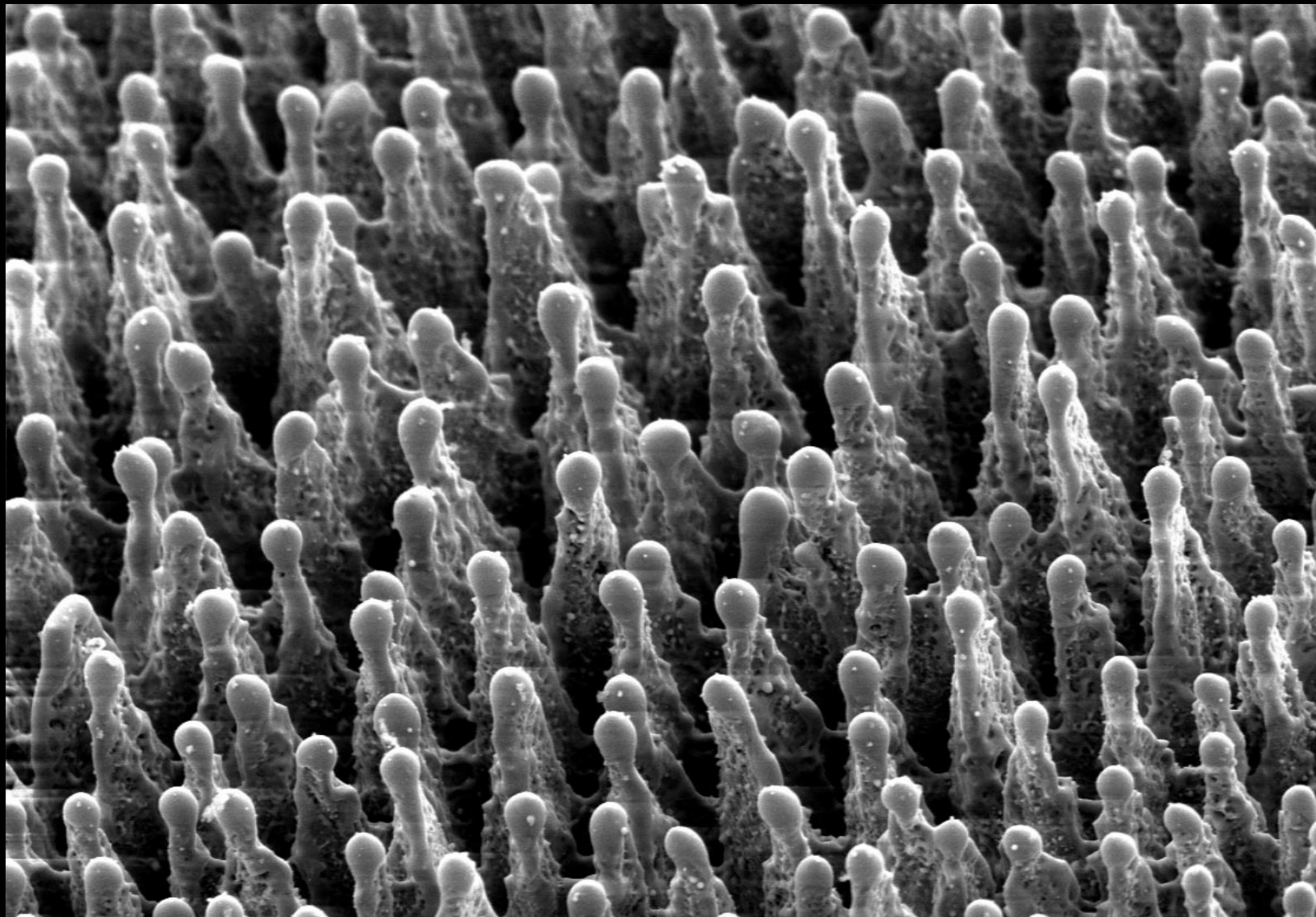
20  $\mu$ m

10kV

15mm

0070





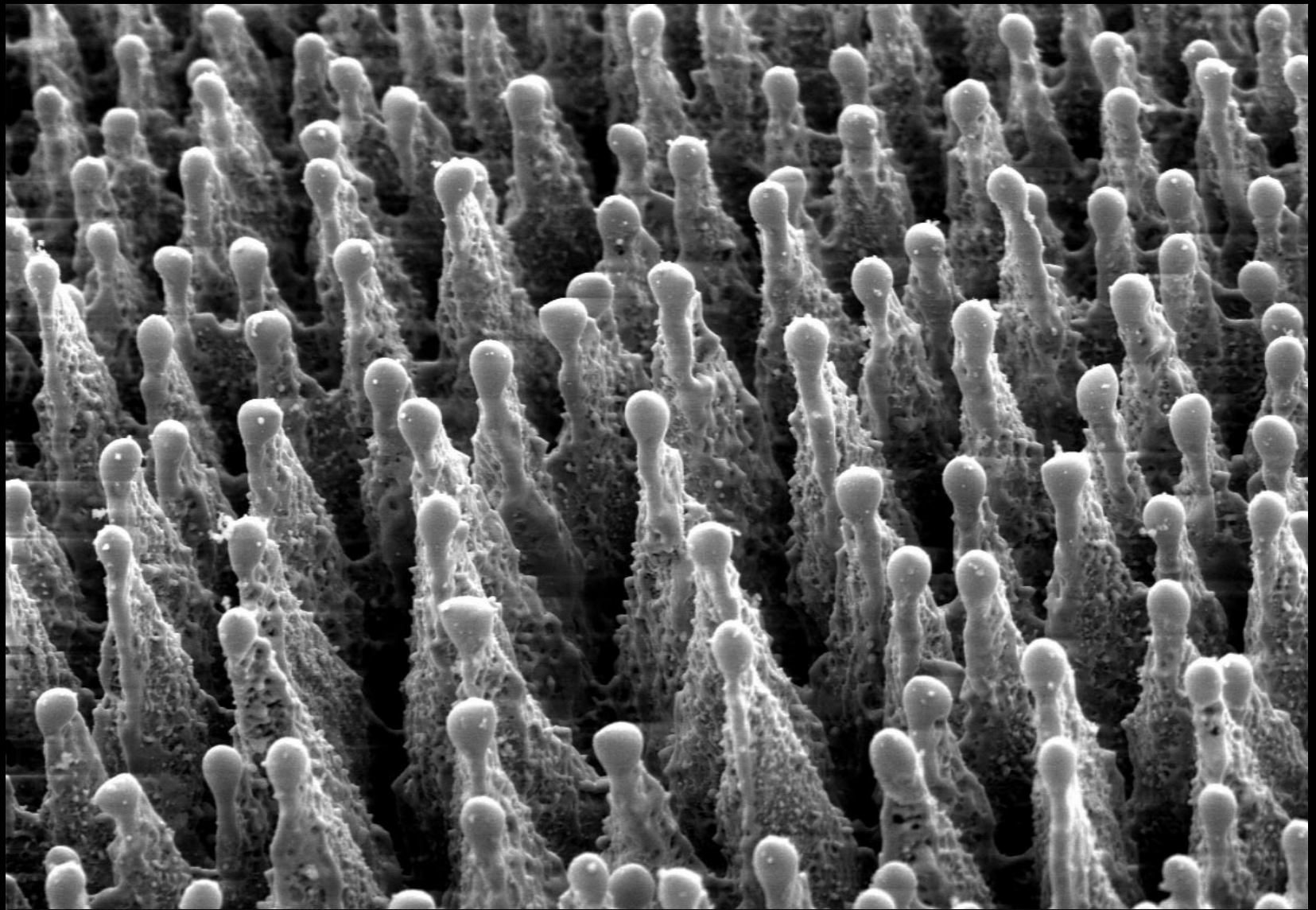
x2000  
#3548  
512 x 480

20  $\mu$ m

10kV

15mm

0100



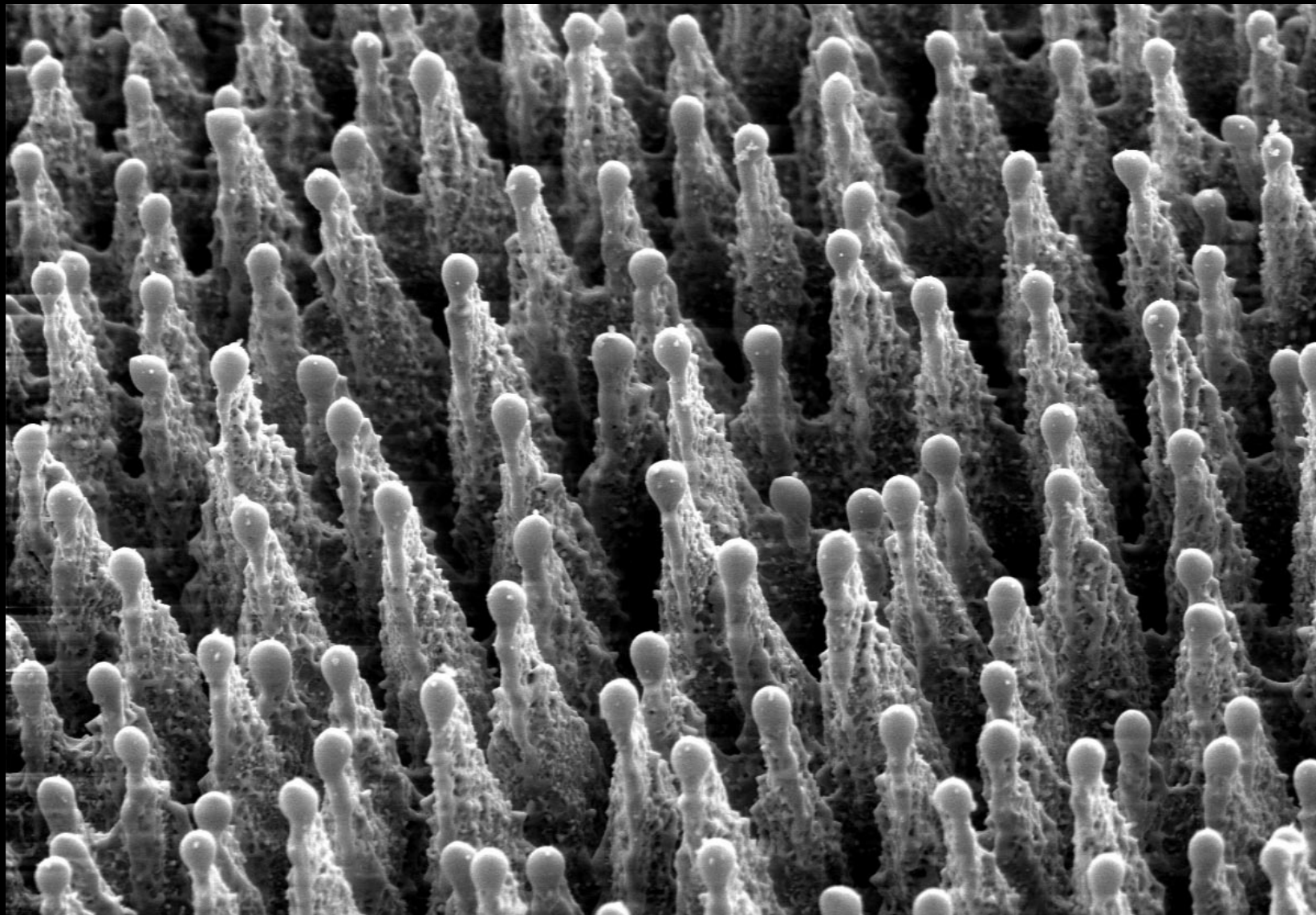
x2000  
#3548  
512 x 480

20  $\mu$ m

10kV

15mm

0200



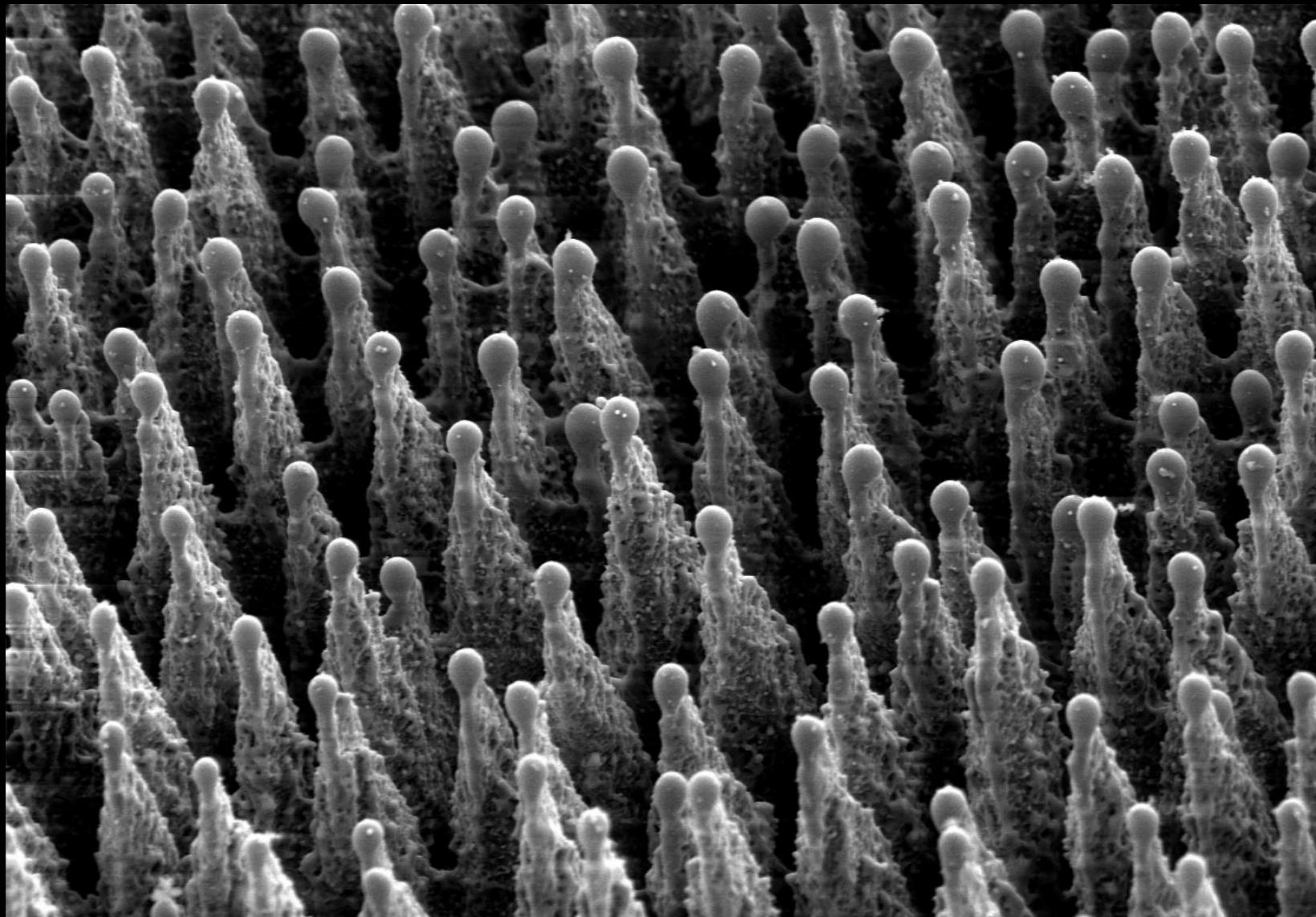
x2000  
#3548  
512 x 480

20  $\mu$ m

10kV

15mm

0400



x2000  
#3548  
512 x 480

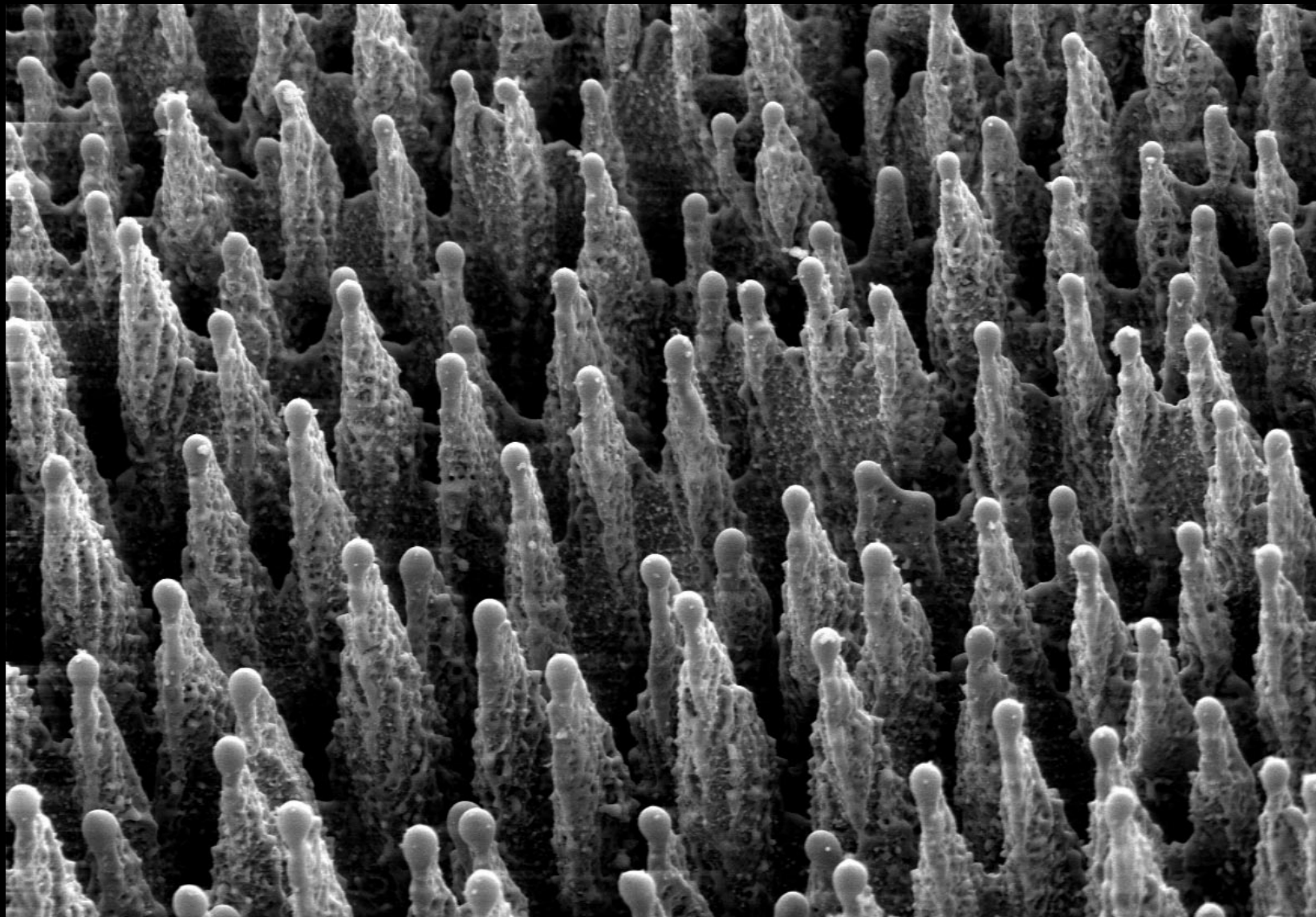
20  $\mu$ m

10kV

15mm

0600





x2000  
#3548  
512 x 480

20  $\mu$ m

10kV

15mm

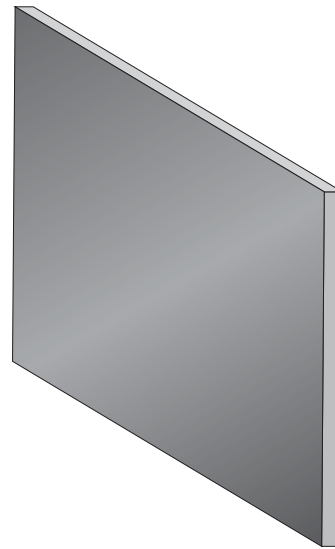
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# *Outlook*

**can ordering of spikes be improved by using a grid?**



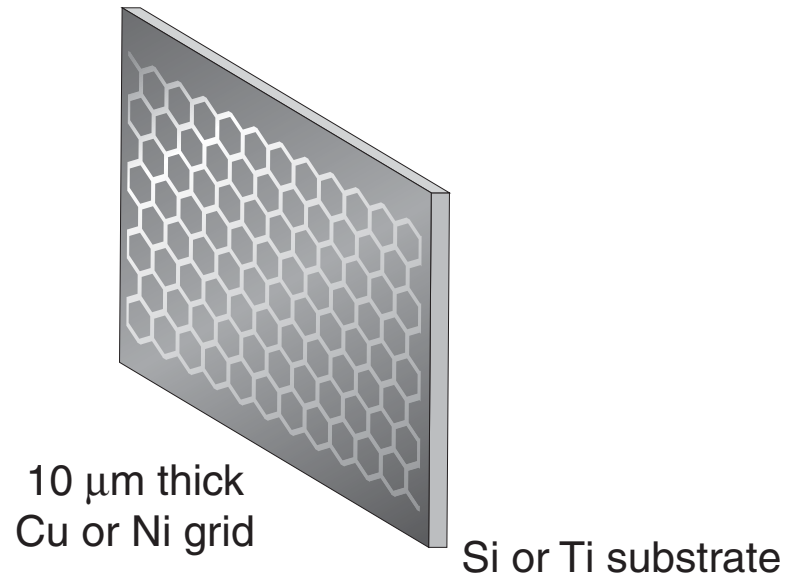
# Outlook



Si or Ti substrate

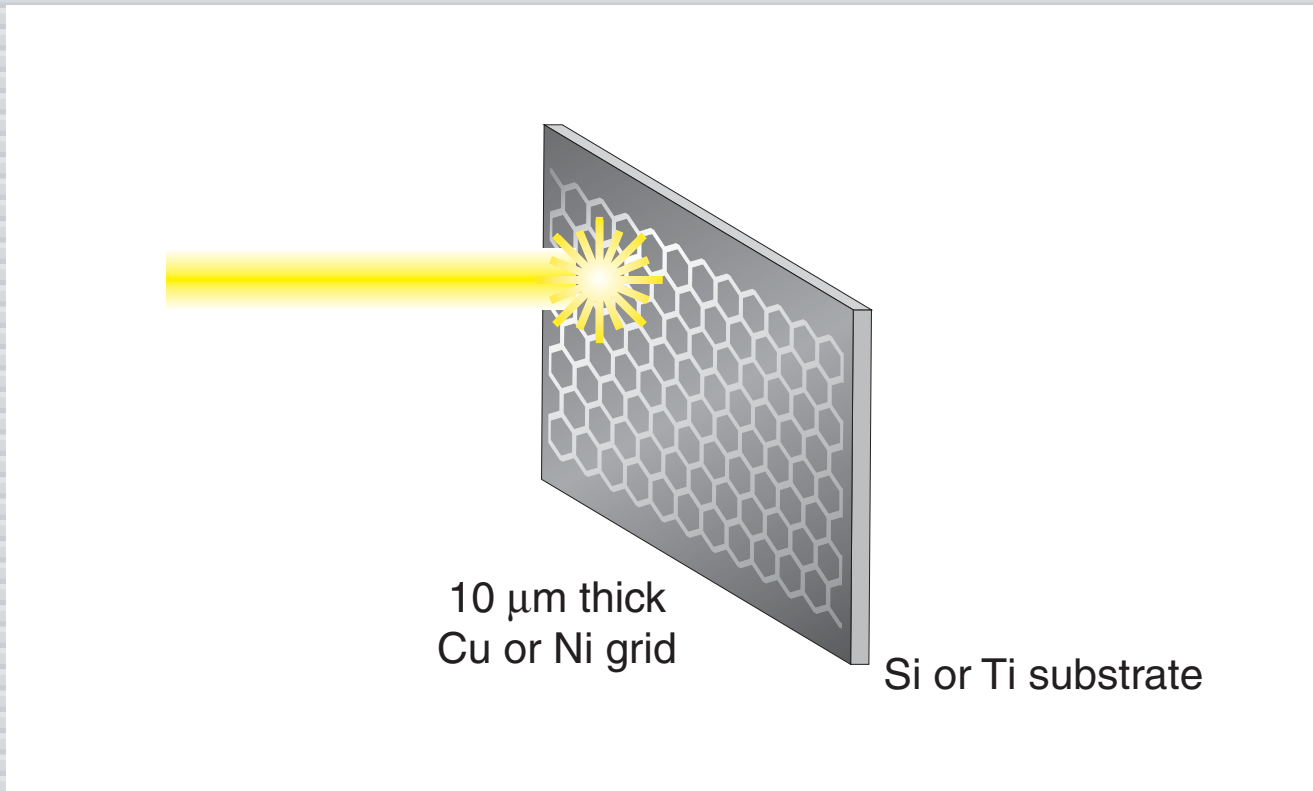
# Outlook

**place grid in front of substrate**



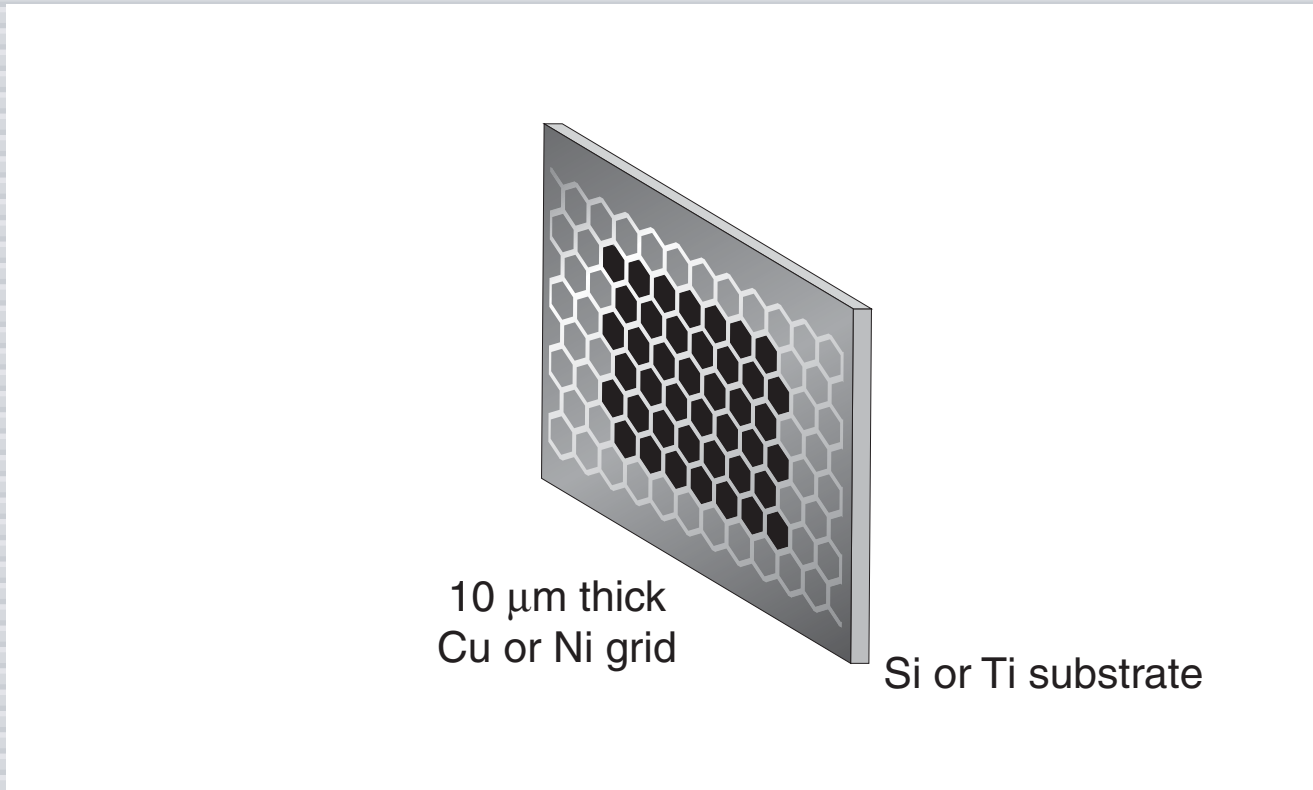
# Outlook

## scan laser beam



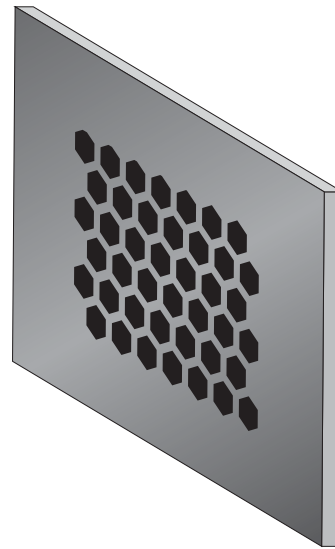
# Outlook

## scan laser beam

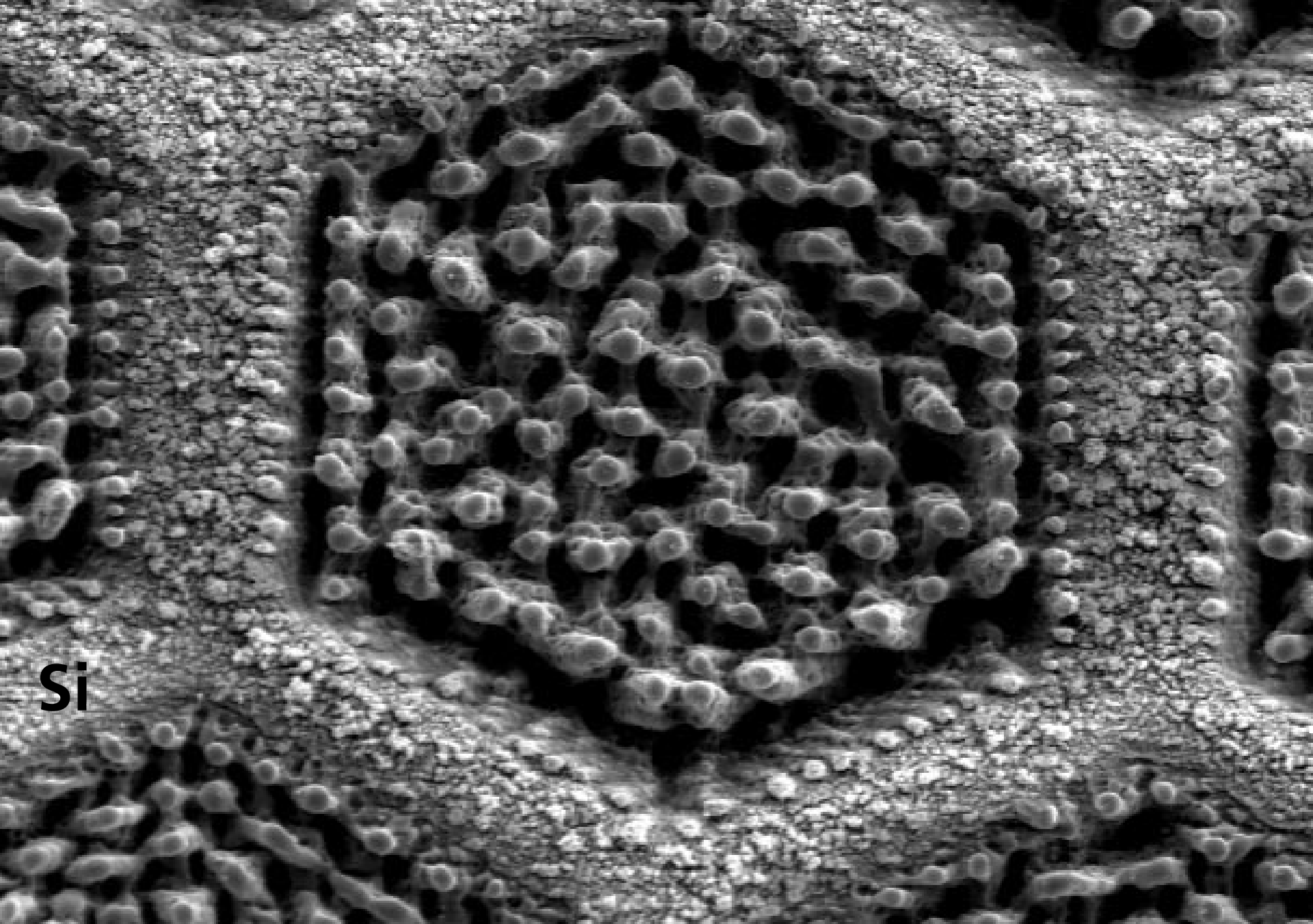


# *Outlook*

**remove grid**



Si or Ti substrate



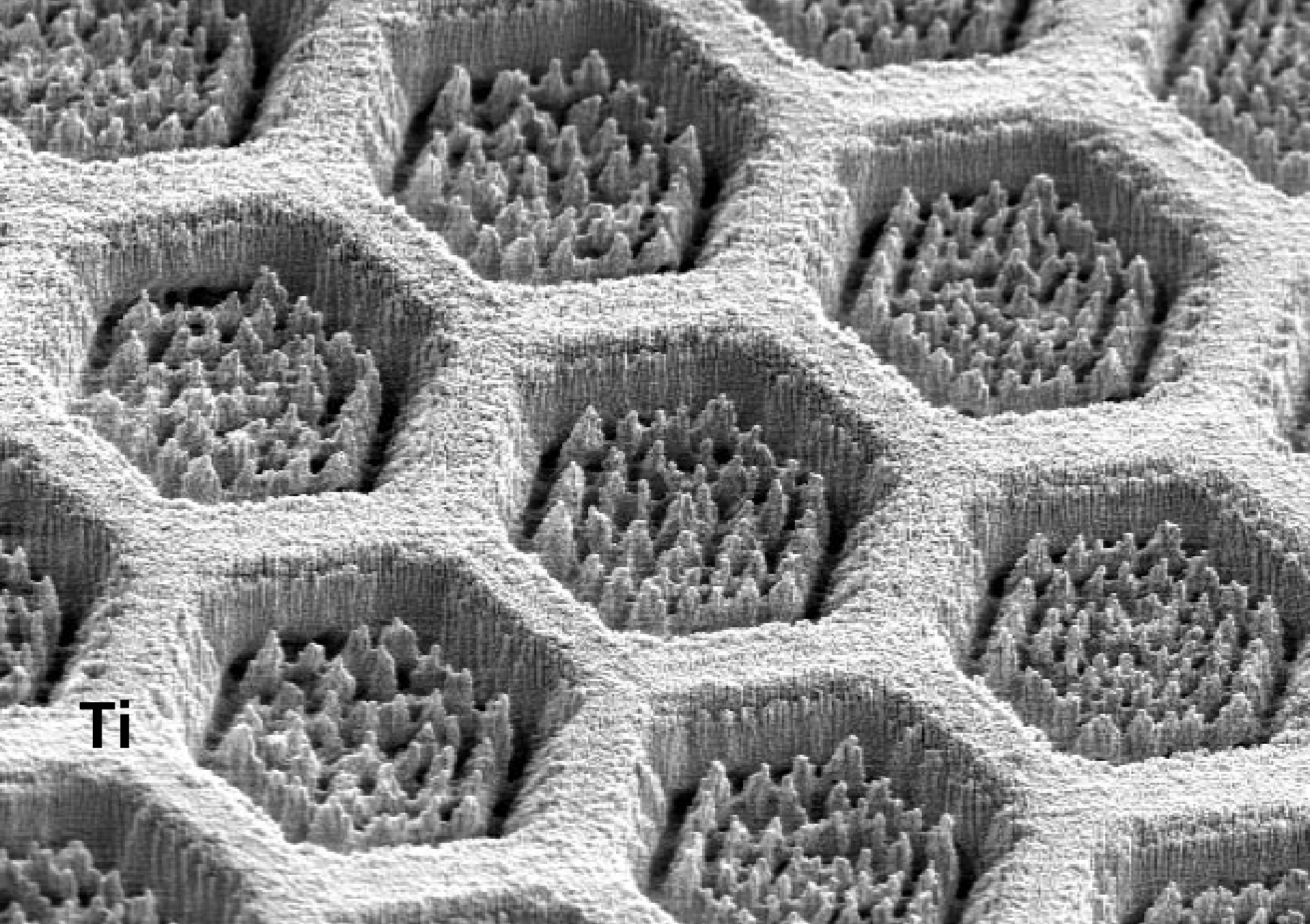
Si

x2000  
512 x 480

20  $\mu$ m

5kV 24mm  
H300.TIF



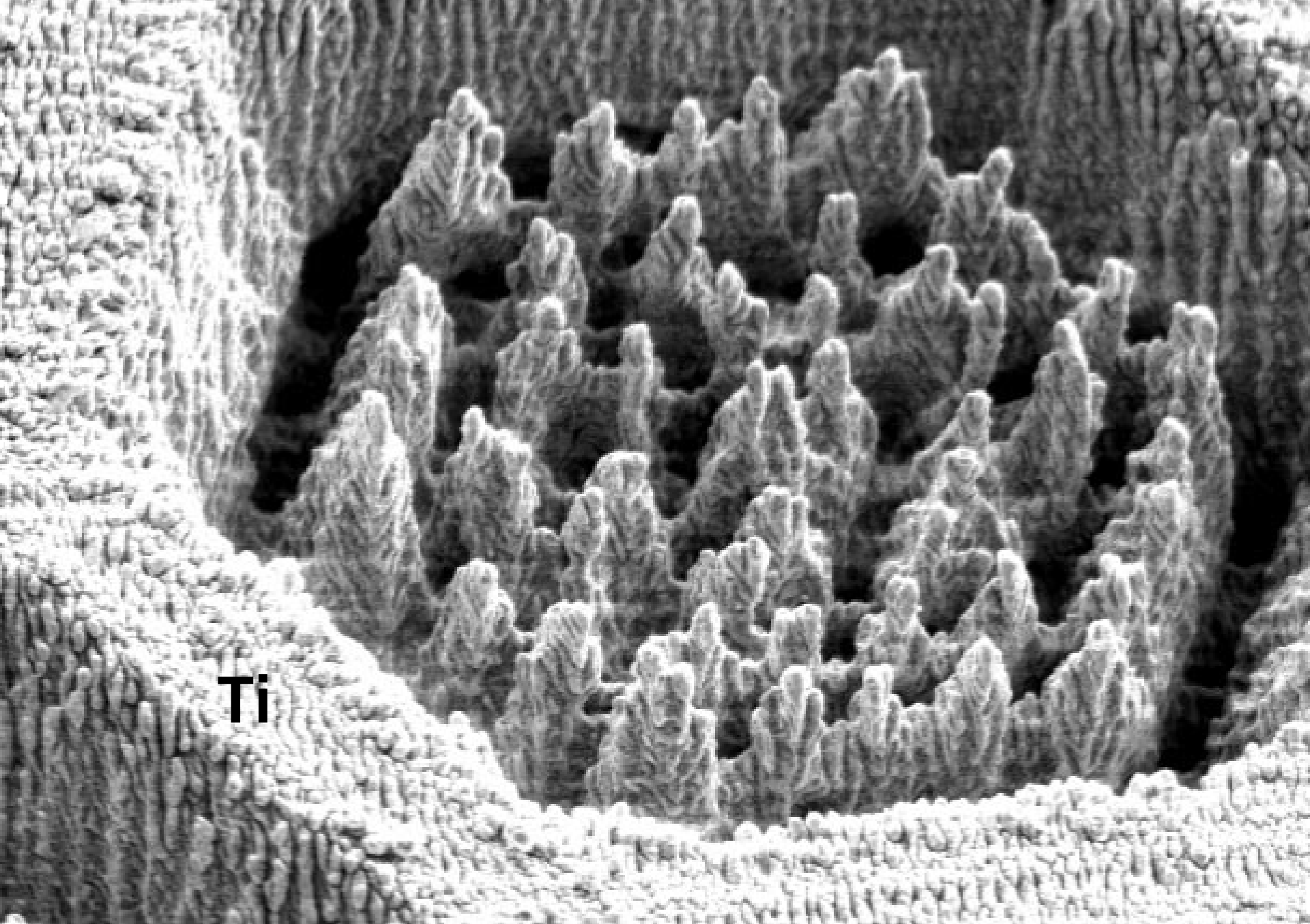


Ti

20  $\mu$ m

5kV

17mm



Ti

10  $\mu$ m

5kV

17mm

# *Outlook*

# *Summary*

## **Microstructured silicon**

- ▶ **fabricated by simple, maskless process**

# *Summary*

## **Microstructured silicon**

- ▶ **fabricated by simple, maskless process**
- ▶ **can be integrated with microelectronics**

## **Microstructured silicon**

- ▶ **fabricated by simple, maskless process**
- ▶ **can be integrated with microelectronics**
- ▶ **generates IR photocurrent**



# *Summary*

## **Microstructured silicon**

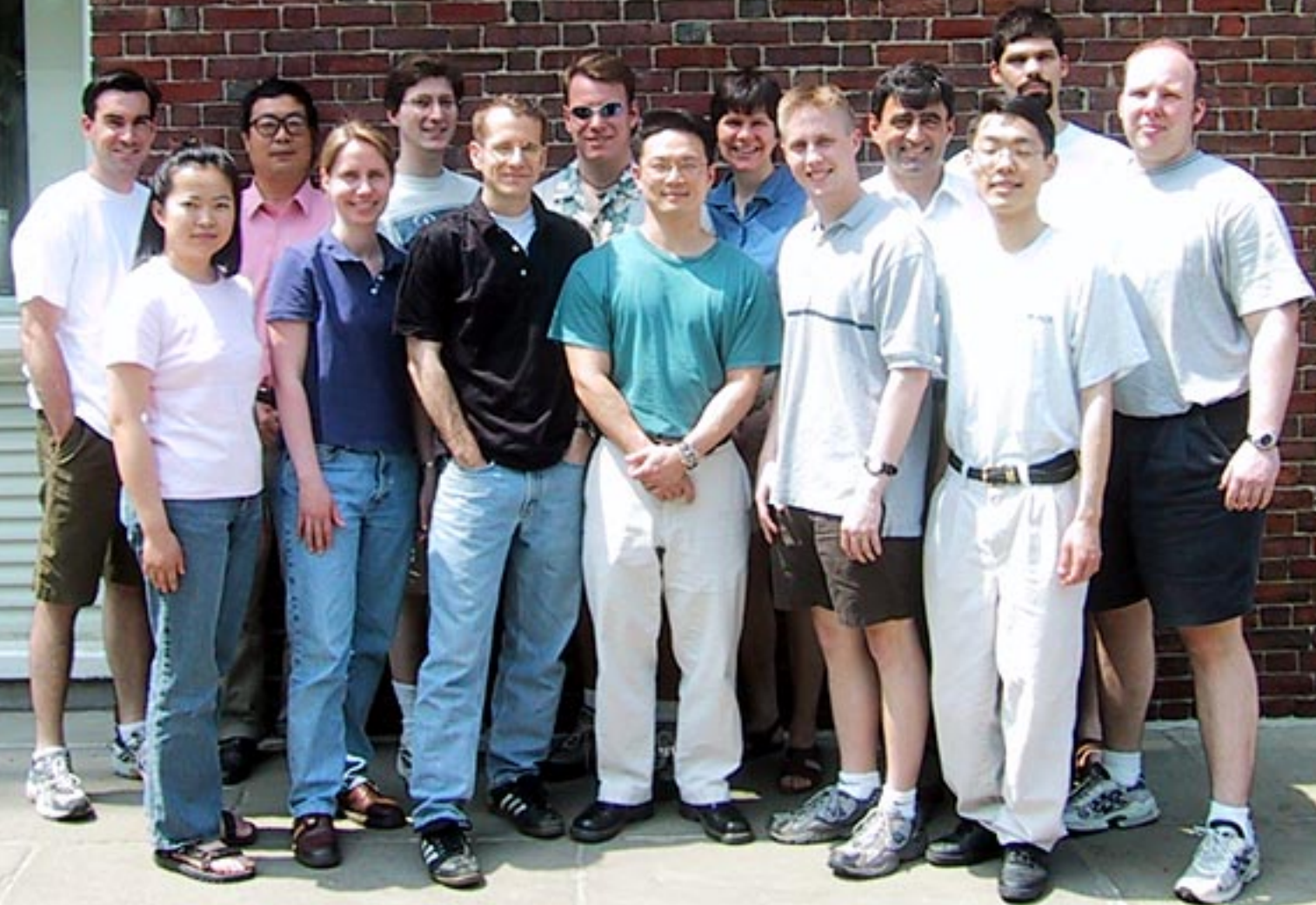
- ▶ **fabricated by simple, maskless process**
- ▶ **can be integrated with microelectronics**
- ▶ **generates IR photocurrent**
- ▶ **provides stable, high field emission current**

# *Summary*

## **Microstructured silicon**

- ▶ **fabricated by simple, maskless process**
- ▶ **can be integrated with microelectronics**
- ▶ **generates IR photocurrent**
- ▶ **provides stable, high field emission current**
- ▶ **is durable**

CORDON MCKAY  
LABORATORY OF  
APPLIED SCIENCE





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**Dr. Alf Bjørseth (Scanwafer)**

**Dr. Tom Mates (UCSB)**

**Dr. John Chervinsky (Harvard University)**

**Prof. Cynthia Friend (Harvard University)**

**Prof. Mike Aziz (Harvard University)**

**For a copy of this talk and  
additional information, see:**

**<http://mazur-www.harvard.edu>**

# Materials

SF<sub>6</sub>

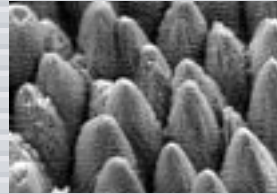
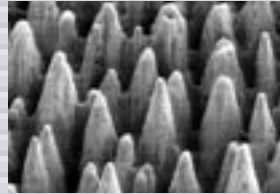
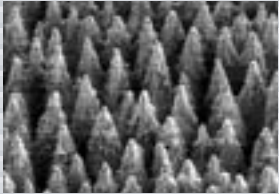
Cl<sub>2</sub>

N<sub>2</sub>

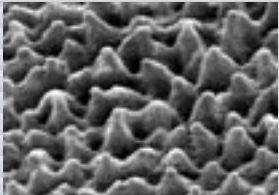
air

vacuum

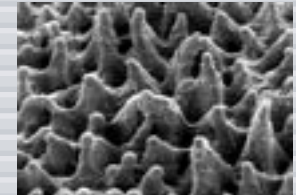
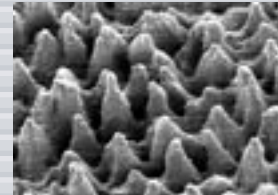
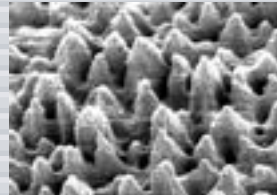
Si



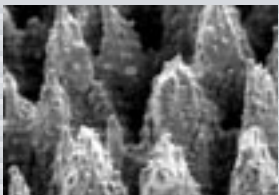
Ti



reacts



Only in SF<sub>6</sub>:



Ge

InP

No spikes in SF<sub>6</sub>: Ag, Al, Cu, Pd, Pt, Rh, Ta and GaAs

# Materials

SF<sub>6</sub>

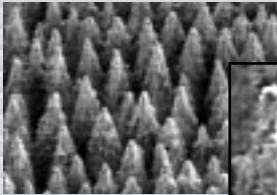
Cl<sub>2</sub>

N<sub>2</sub>

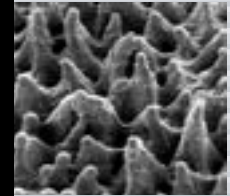
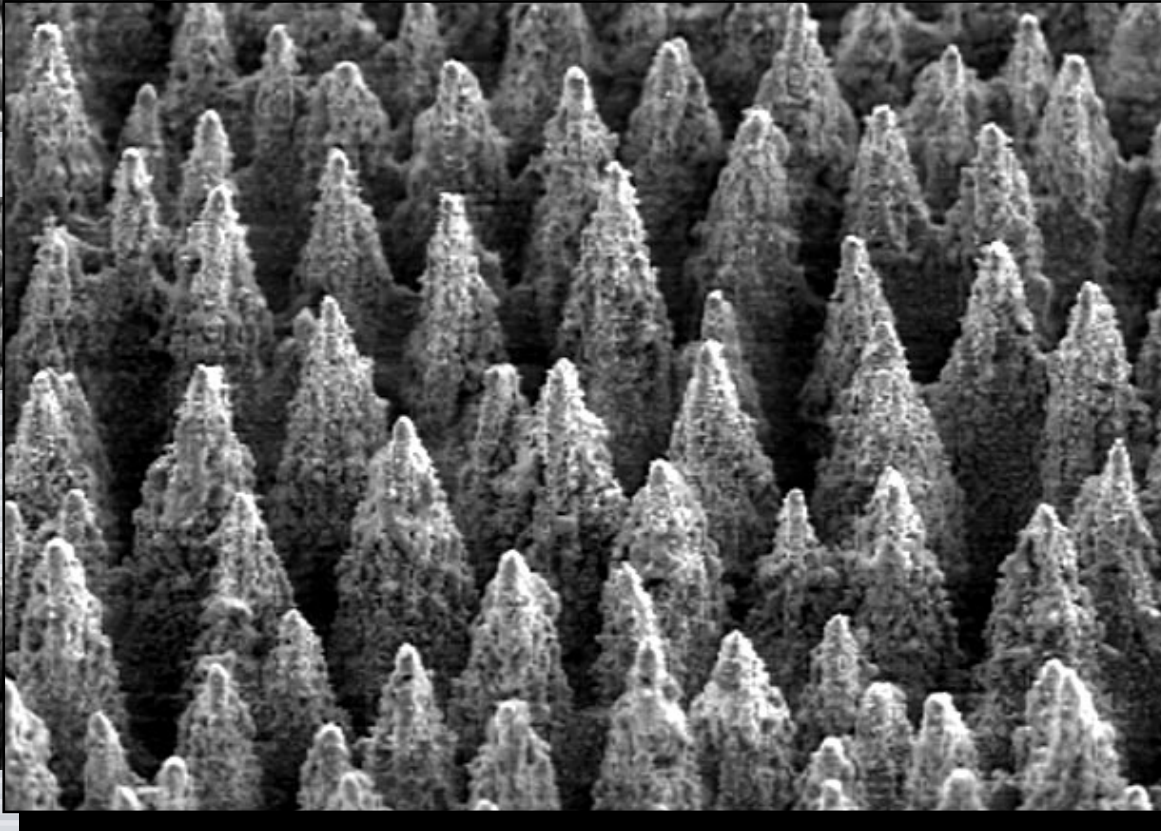
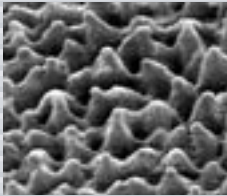
air

vacuum

Si

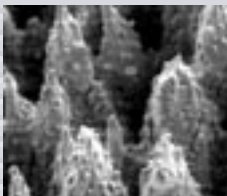


Ti



Only in SF<sub>6</sub>

Ge



No spikes in SF<sub>6</sub>: Ag, Al, Cu, Pd, Pt, Rh, Ta and GaAs



# Materials

SF<sub>6</sub>

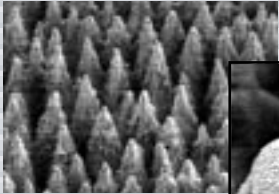
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N<sub>2</sub>

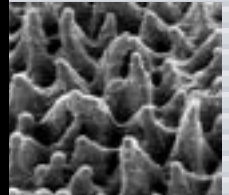
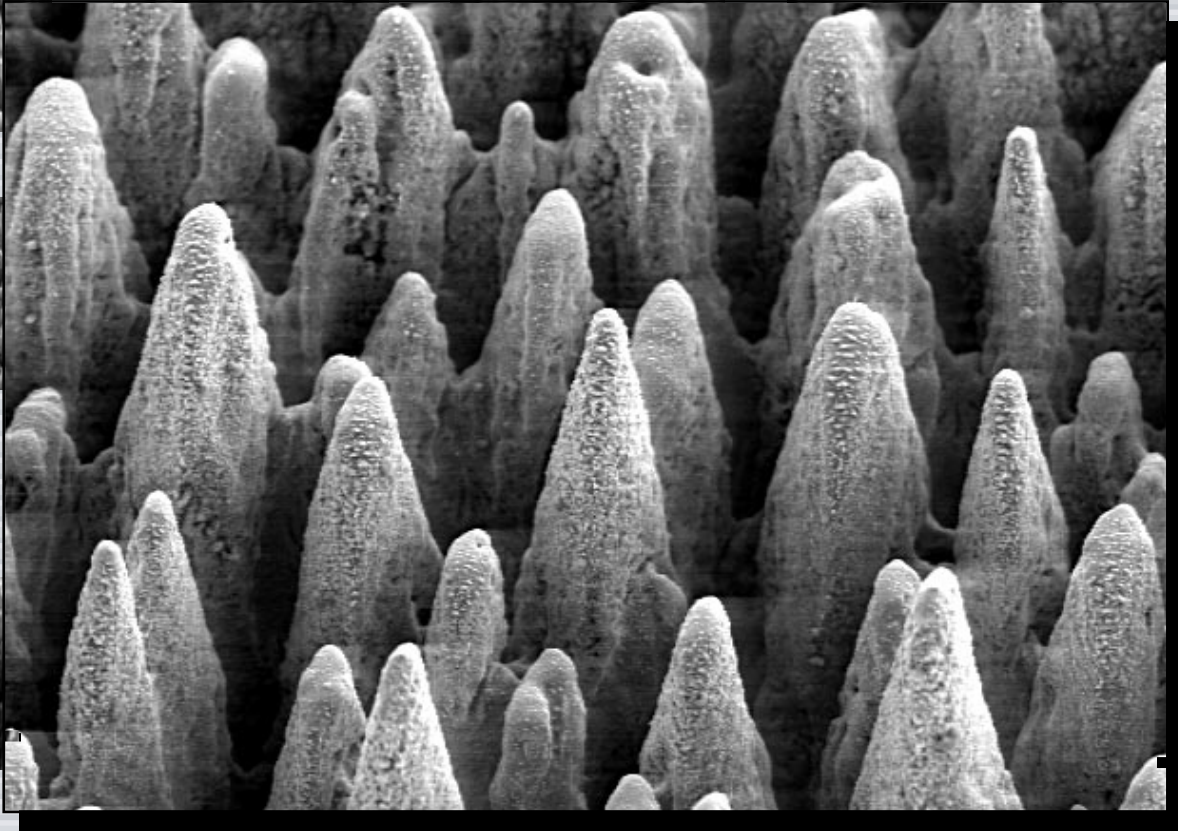
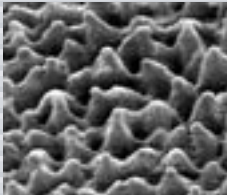
air

vacuum

Si

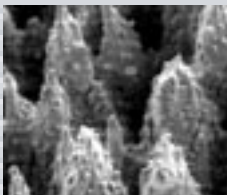


Ti



Only in SF<sub>6</sub>

Ge



No spikes in SF<sub>6</sub>: Ag, Al, Cu, Pd, Pt, Rh, Ta and GaAs

# Materials

SF<sub>6</sub>

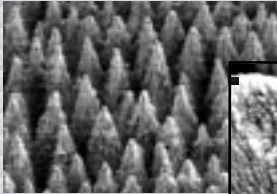
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N<sub>2</sub>

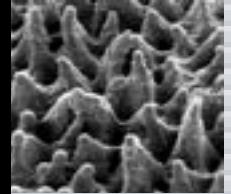
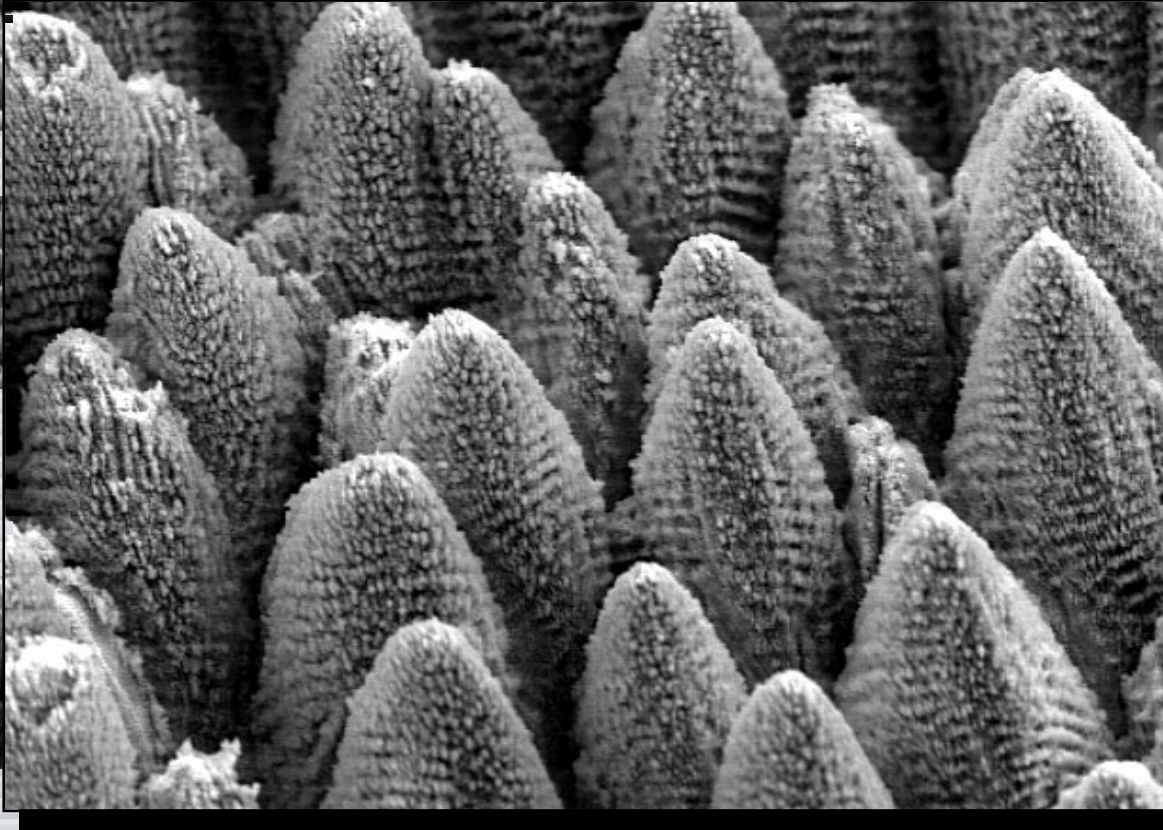
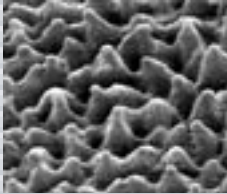
air

vacuum

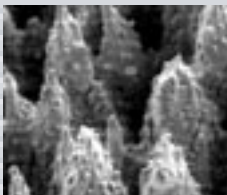
Si



Ti



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# Materials

SF<sub>6</sub>

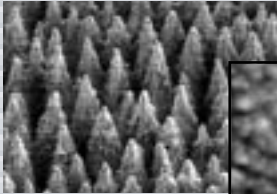
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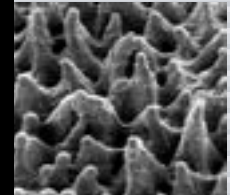
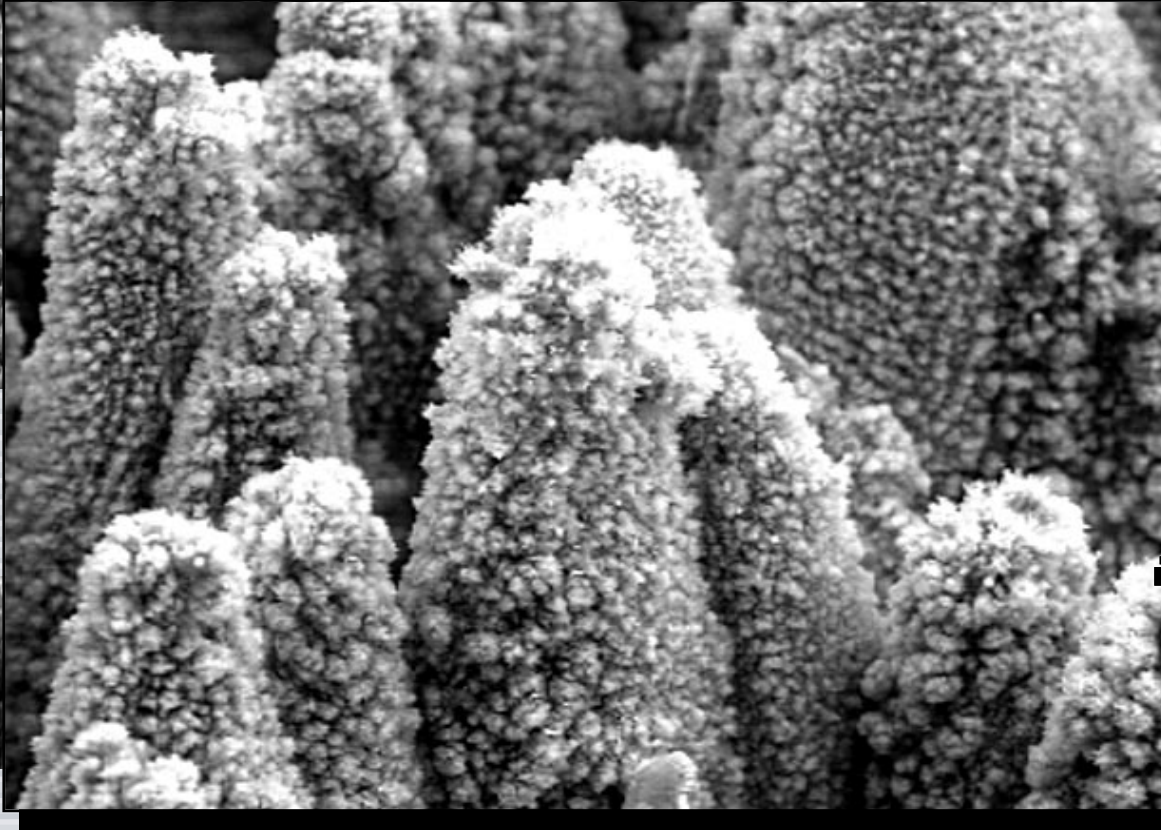
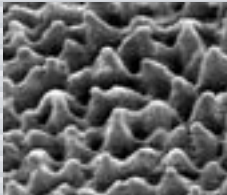
air

vacuum

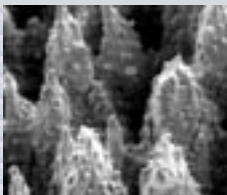
Si



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SF<sub>6</sub>

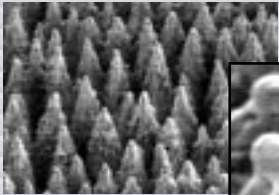
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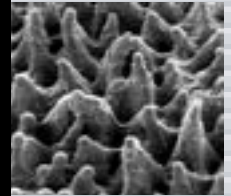
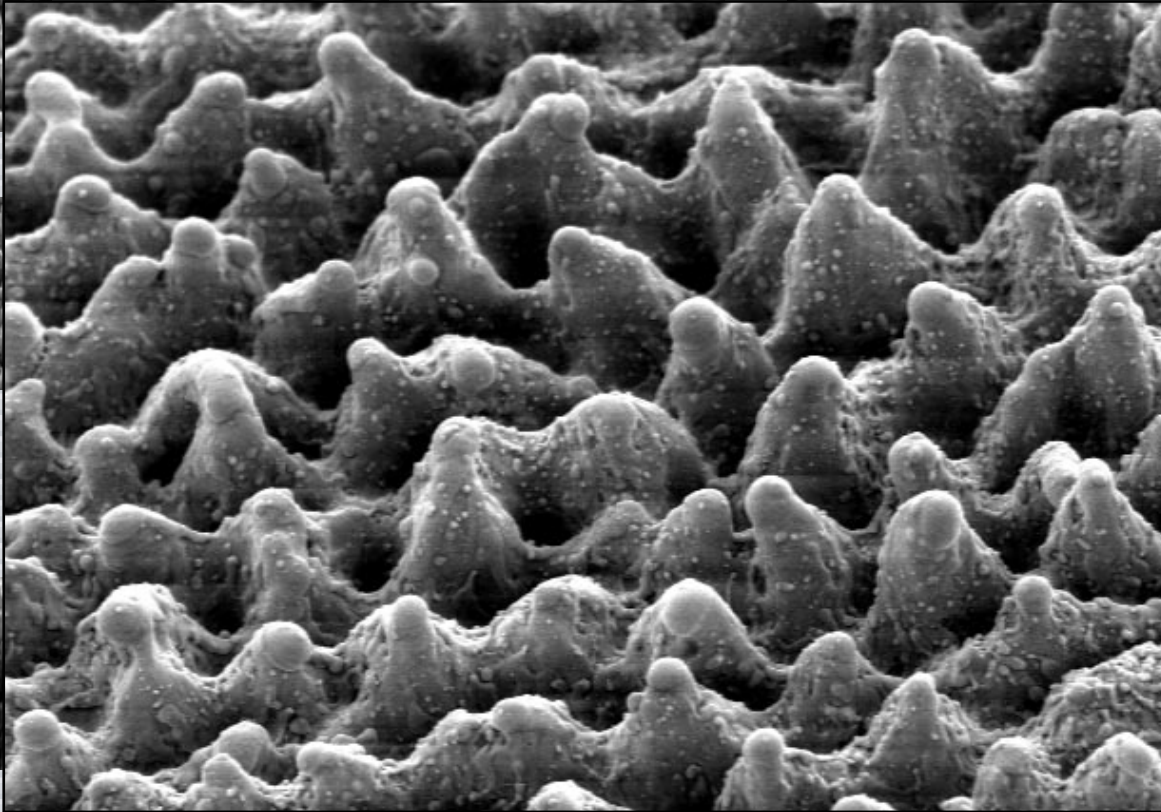
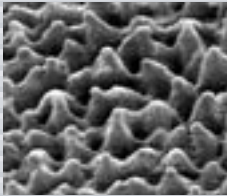
air

vacuum

Si

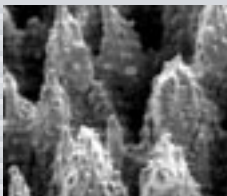


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SF<sub>6</sub>

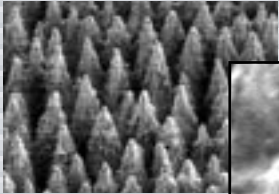
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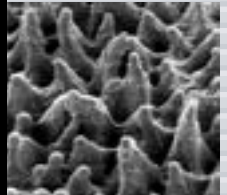
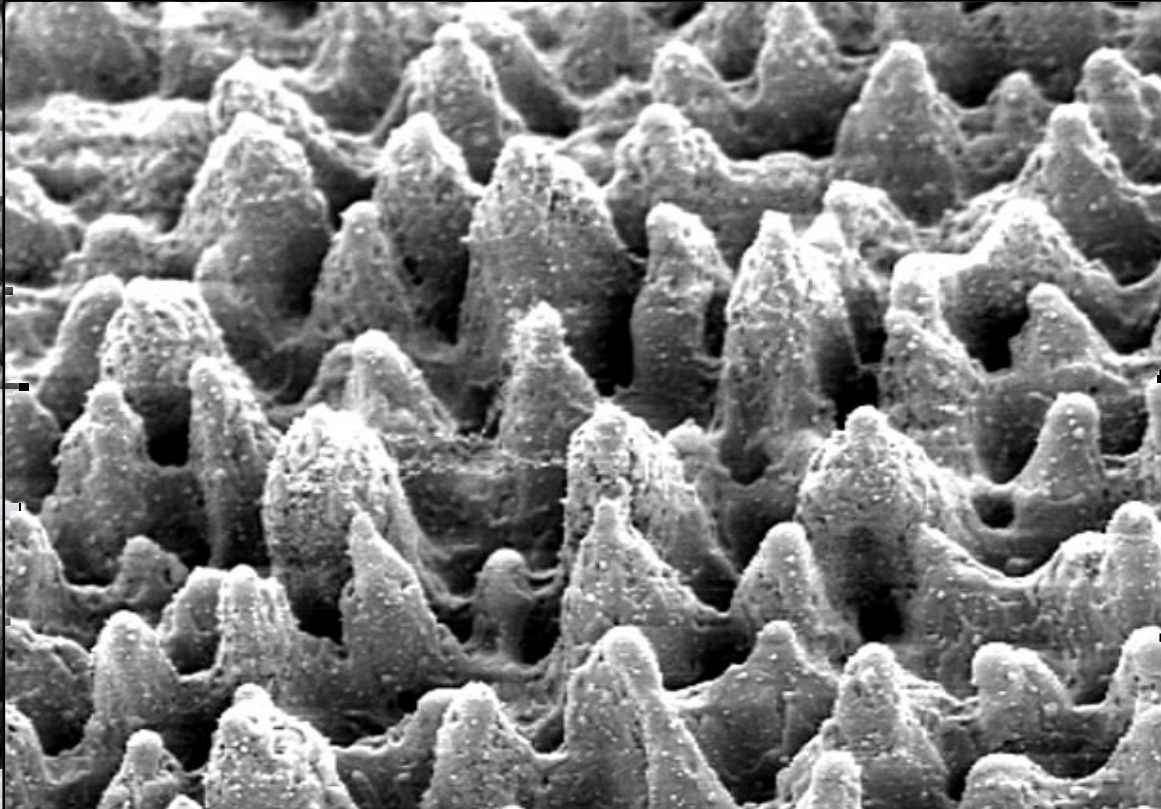
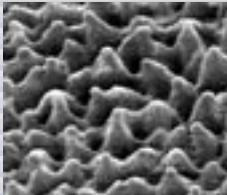
air

vacuum

Si

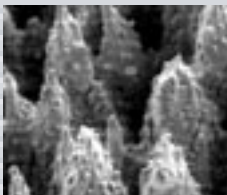


Ti



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# Materials

$SF_6$

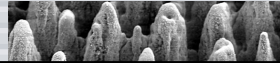
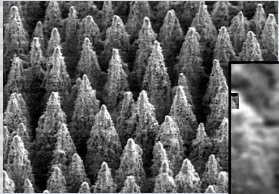
$Cl_2$

$N_2$

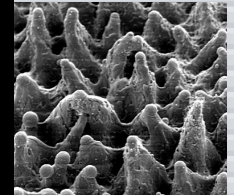
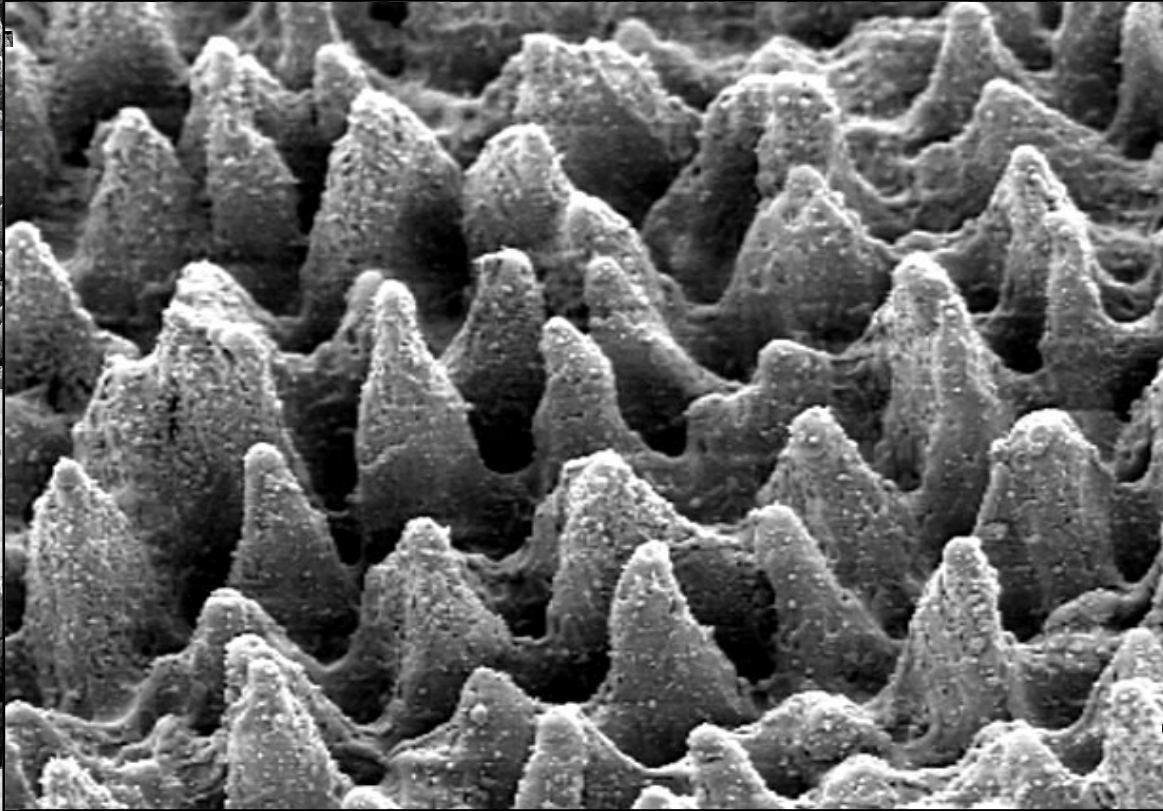
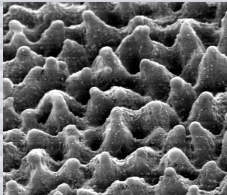
air

vacuum

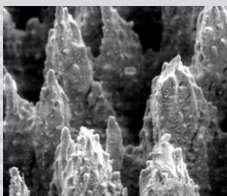
Si



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SF<sub>6</sub>

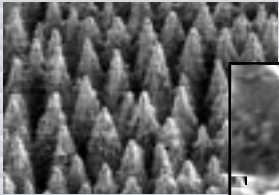
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N<sub>2</sub>

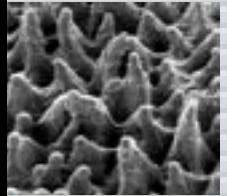
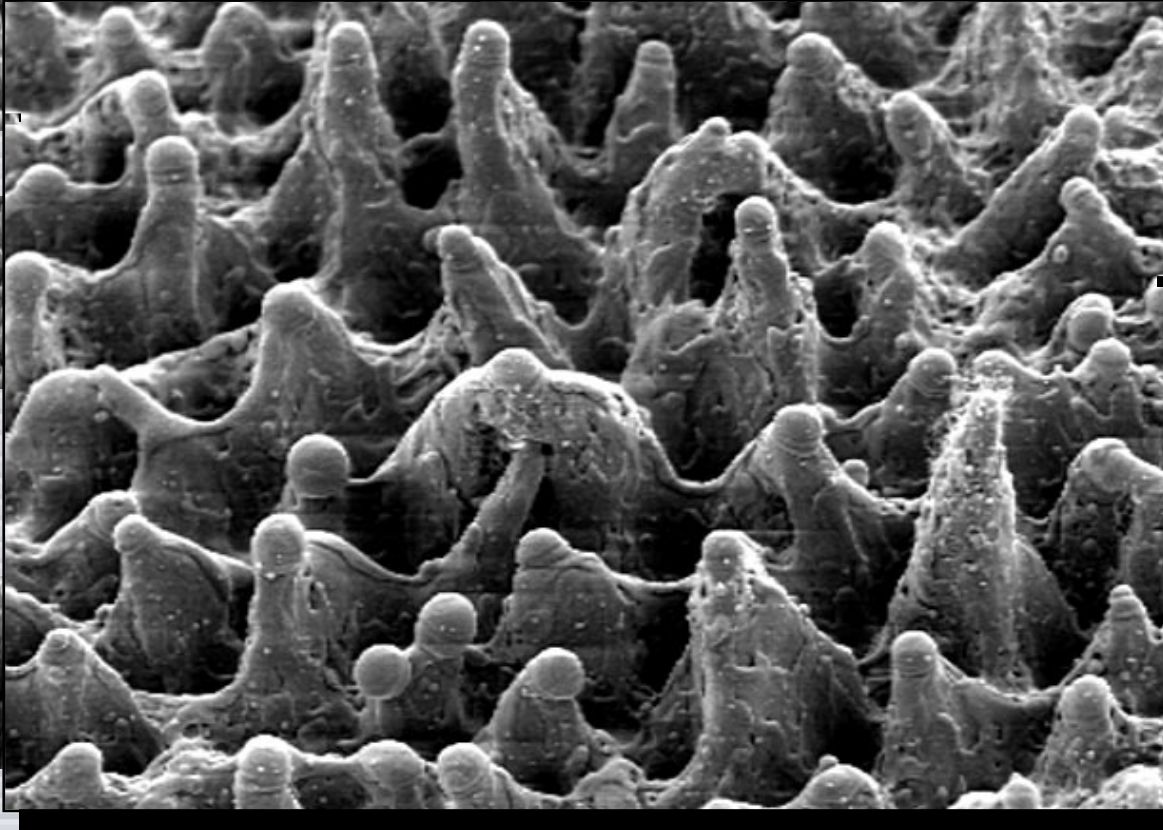
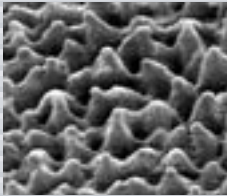
air

vacuum

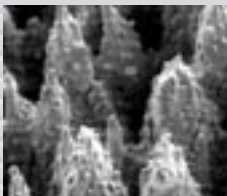
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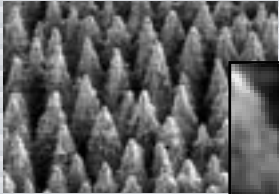
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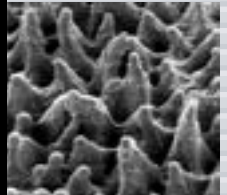
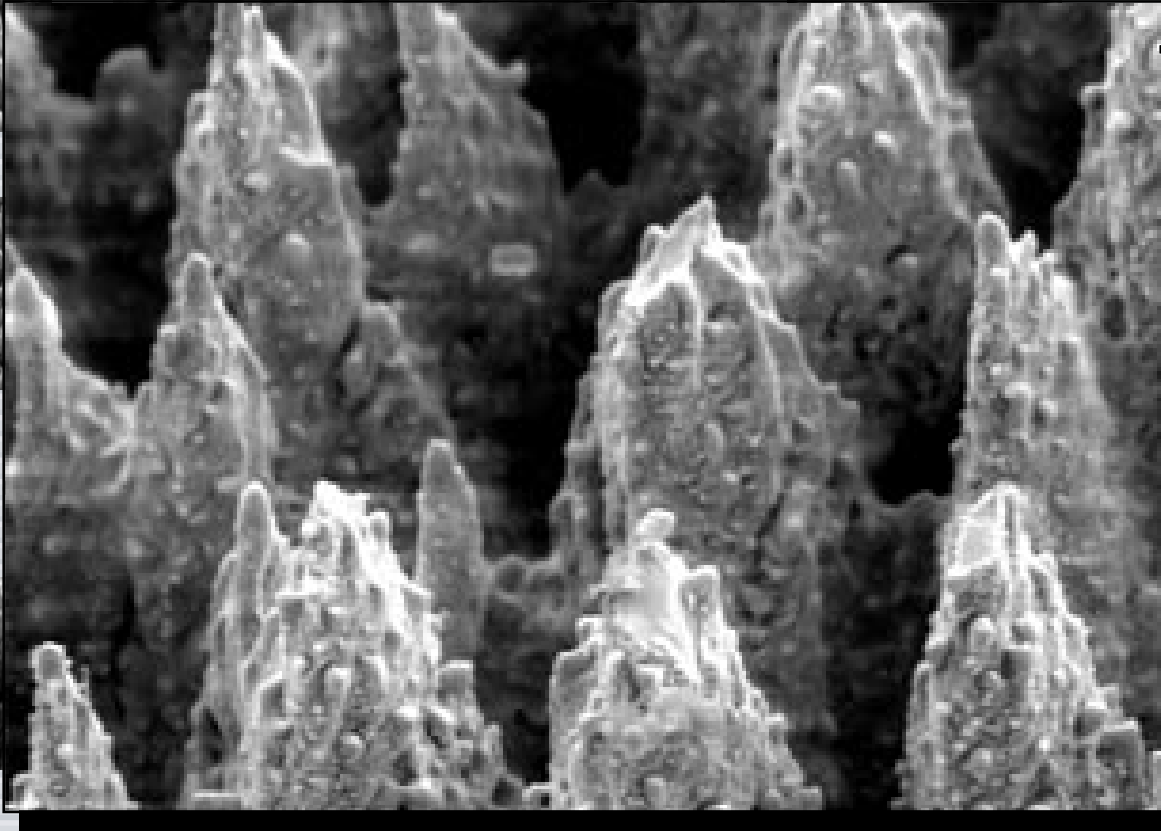
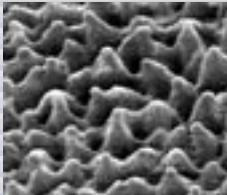
air

vacuum

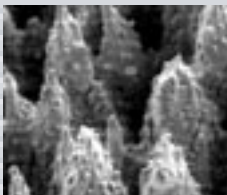
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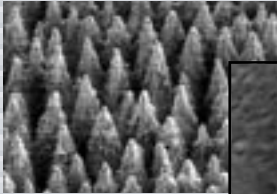
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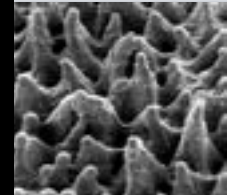
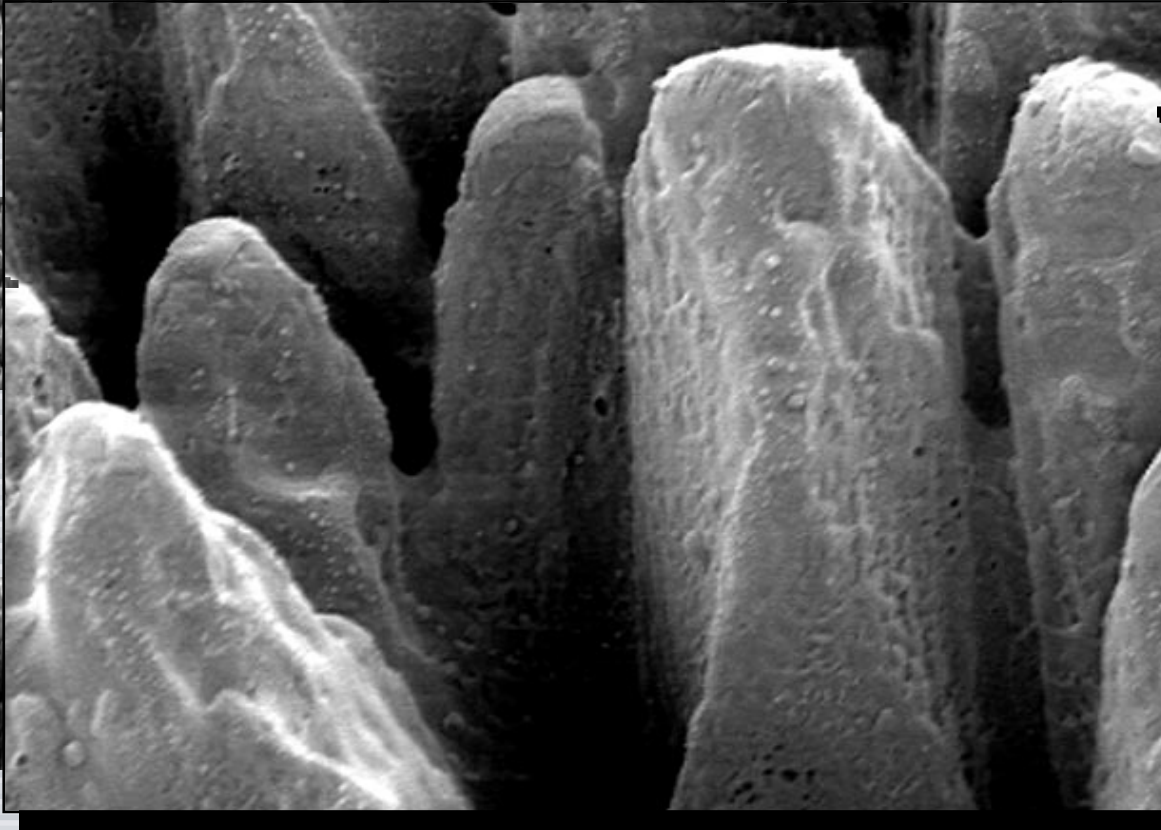
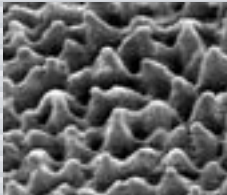
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vacuum

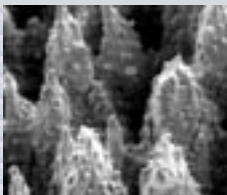
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