CELLULOSE/POLYSULFONE NANOCOMPOSITES

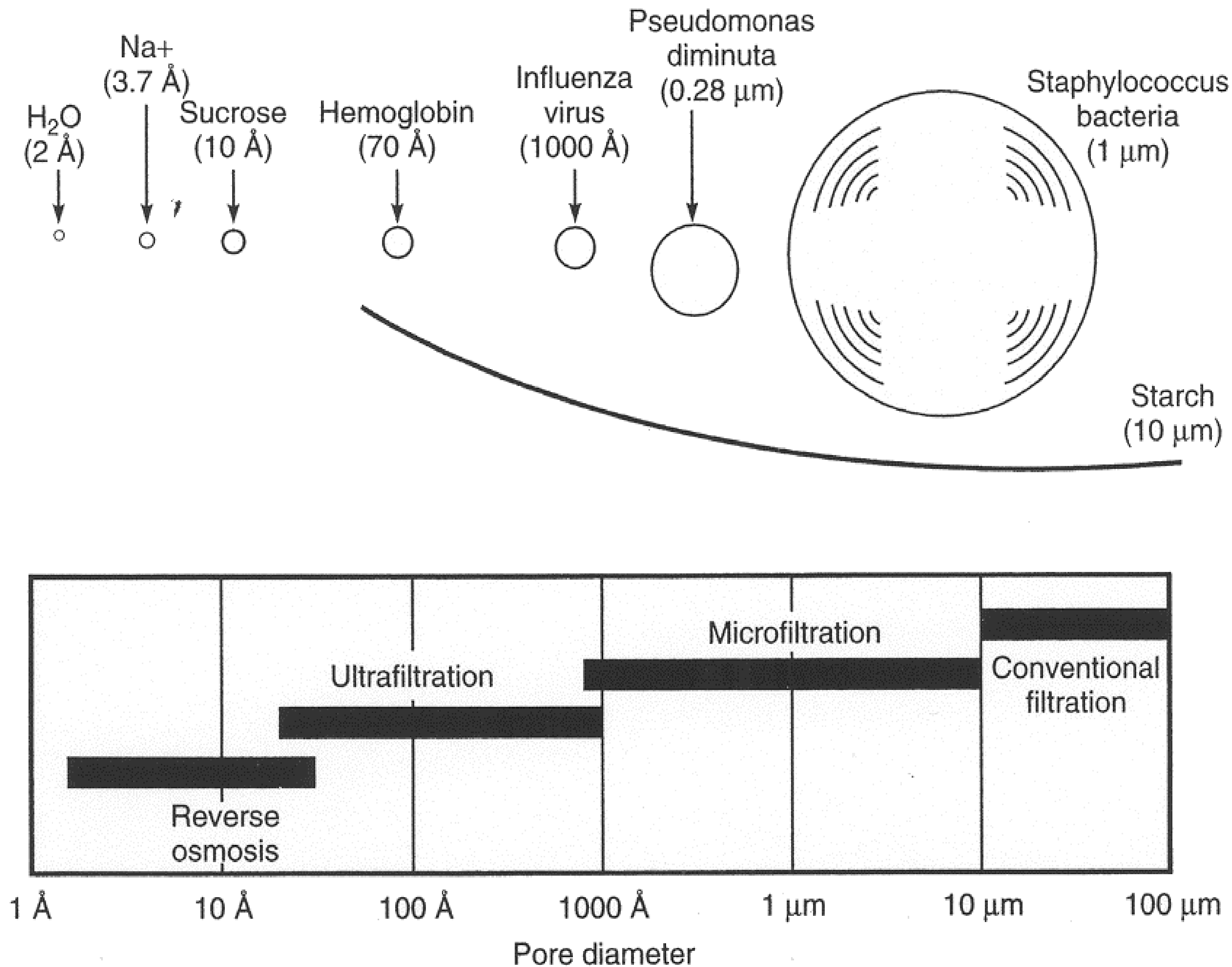
Sweda Noorani and John Simonsen

Oregon State University
Corvallis, Oregon

OREGON STATE
University

CONTENTS

Membranes



Membrane Technology and Applications, R.W. Baker, Wiley & Sons, West Sussex, England

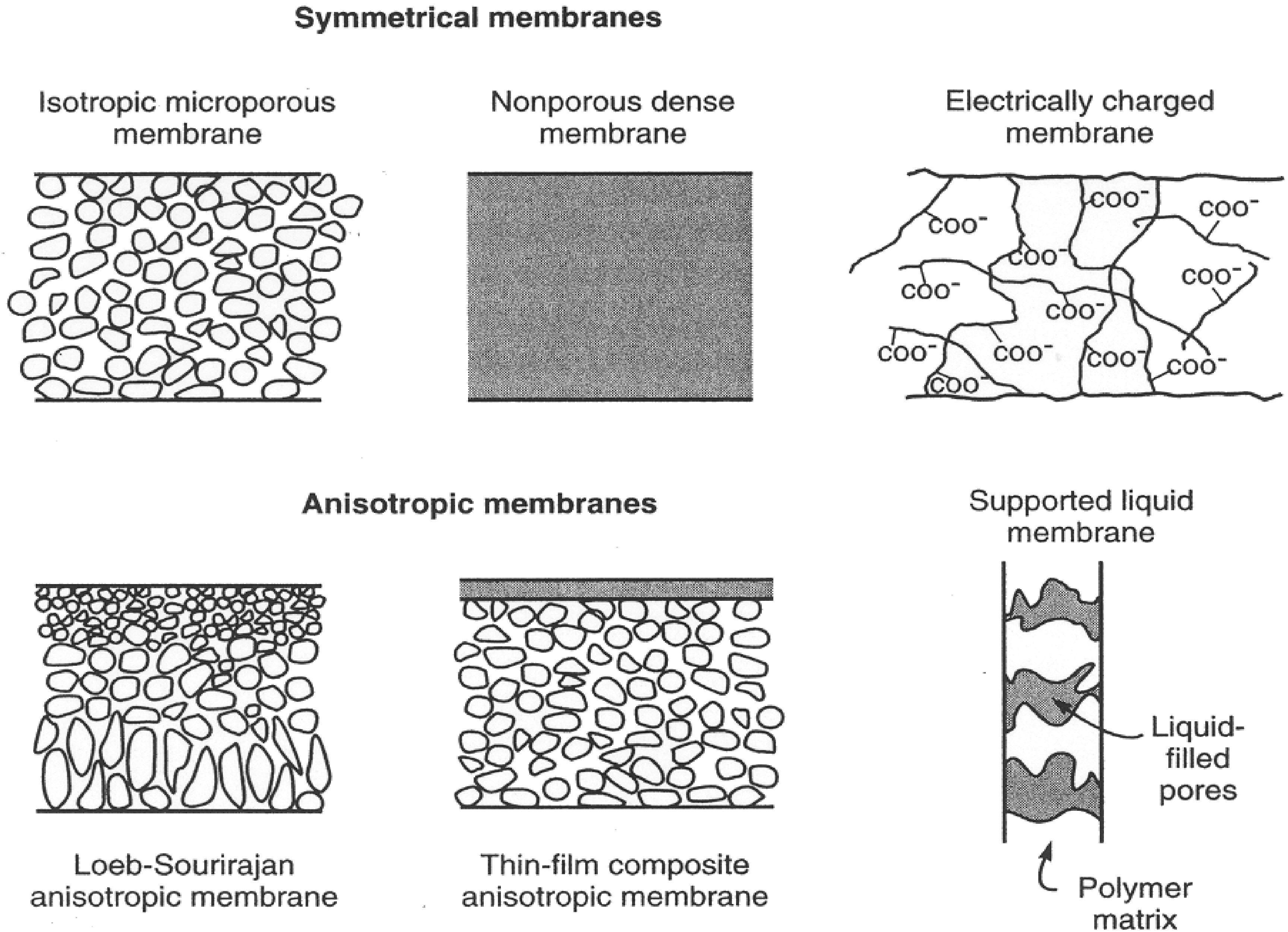
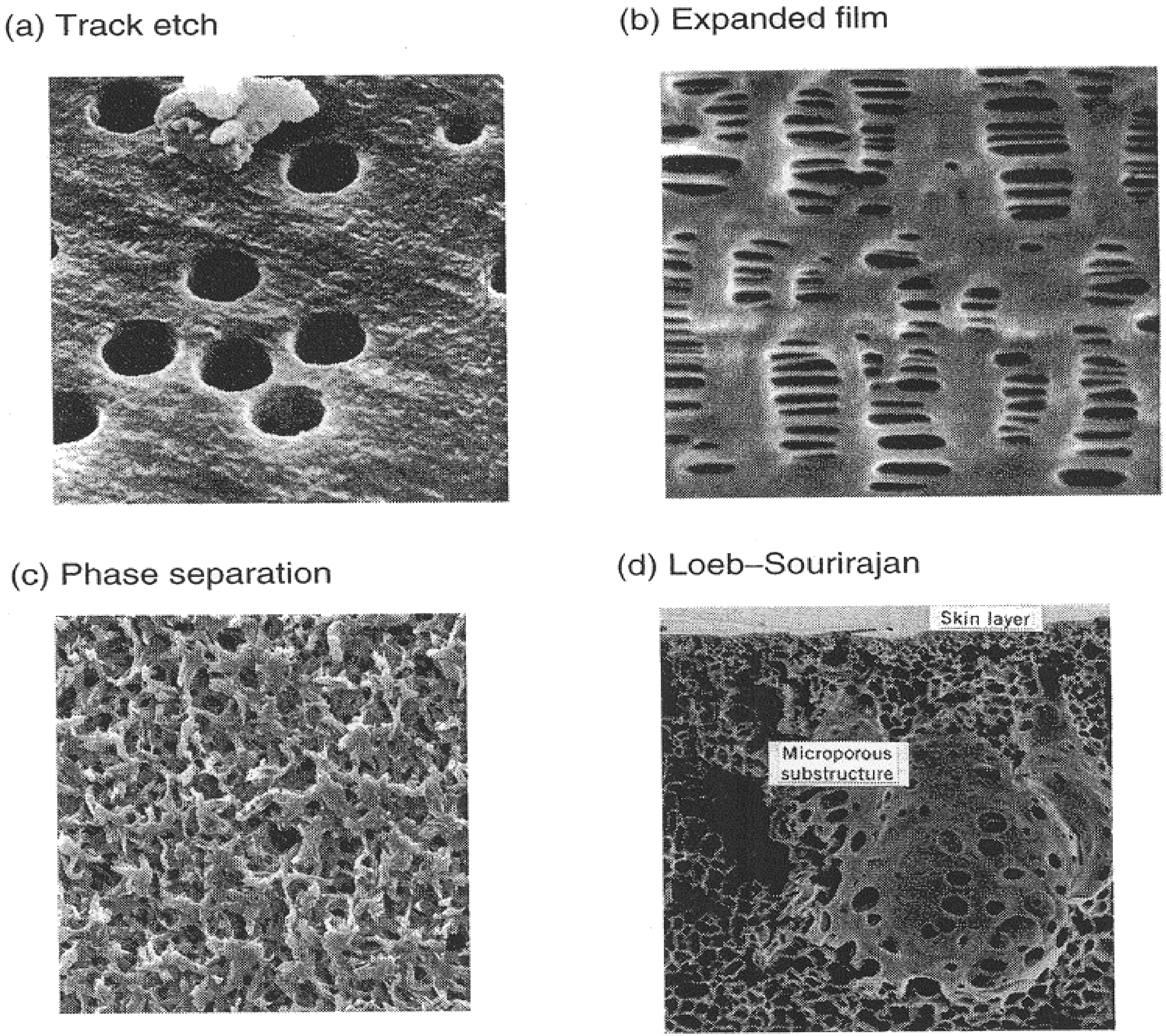


Figure 1.1 Schematic diagrams of the principal types of membranes

Membrane Technology and Applications, R.W. Baker, Wiley & Sons, West Sussex, England



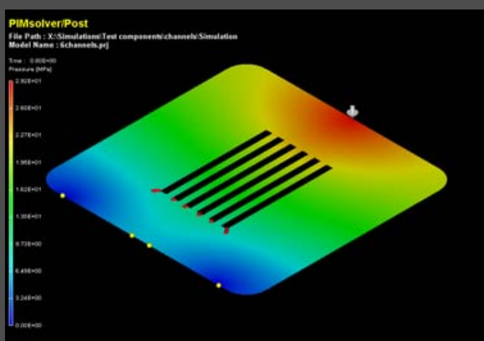
Membrane Technology and Applications, R.W. Baker, Wiley & Sons, West Sussex, England

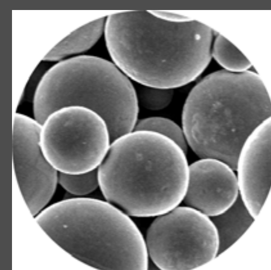
MECS

Microtechnologies in Energy and Chemical Systems

Materials & Process Design for MECS

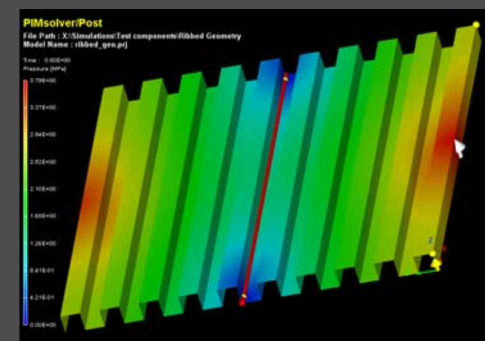
Prof. Sundar V. Atre

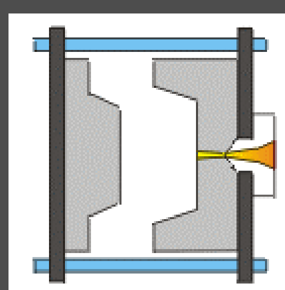




material

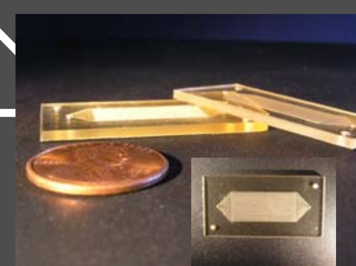
polymer
metal
ceramic
composite





process

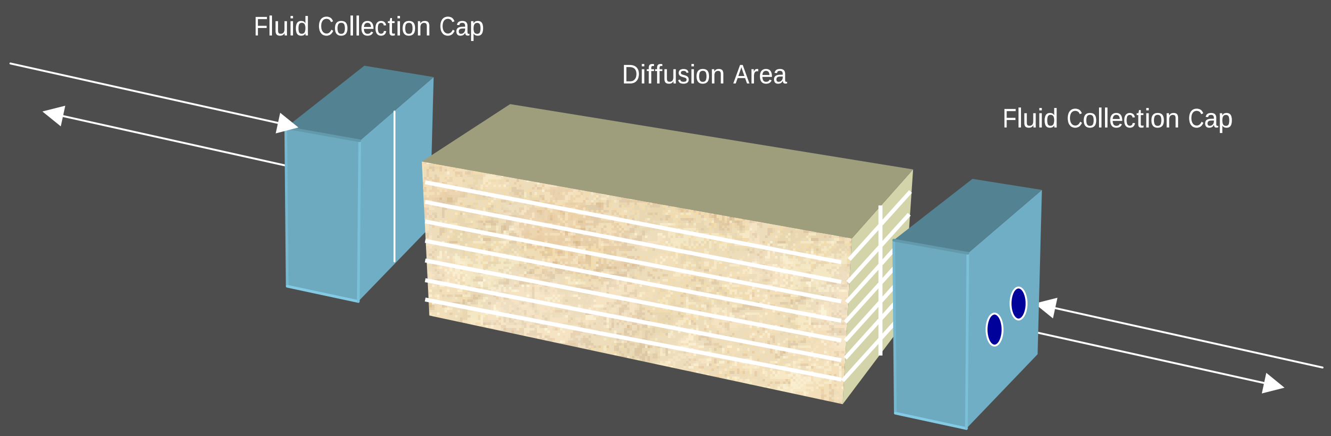
injection molding
extrusion
sintering
micromachining

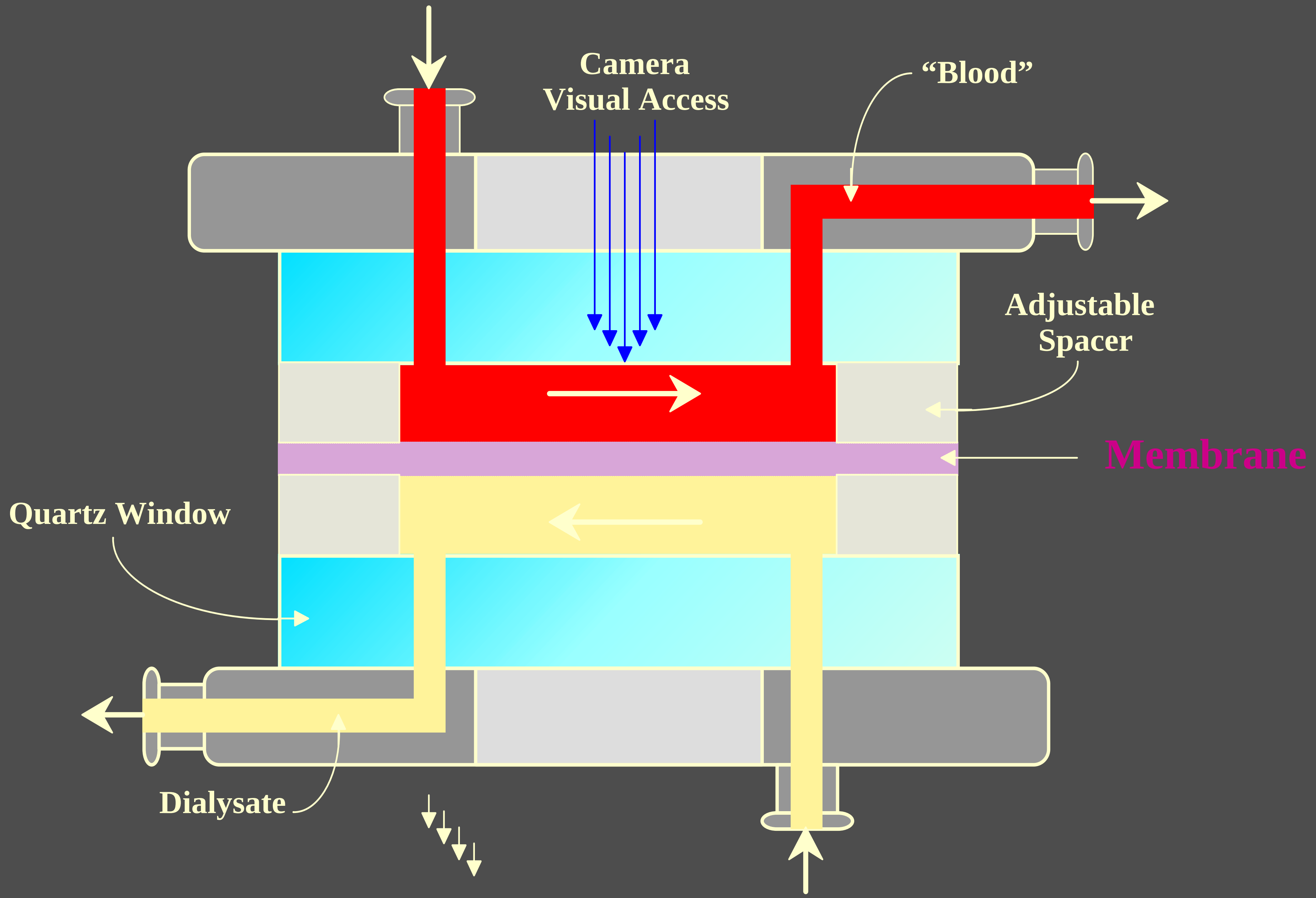


application

kidney dialysis
microreactor
thermal management
fuel cell

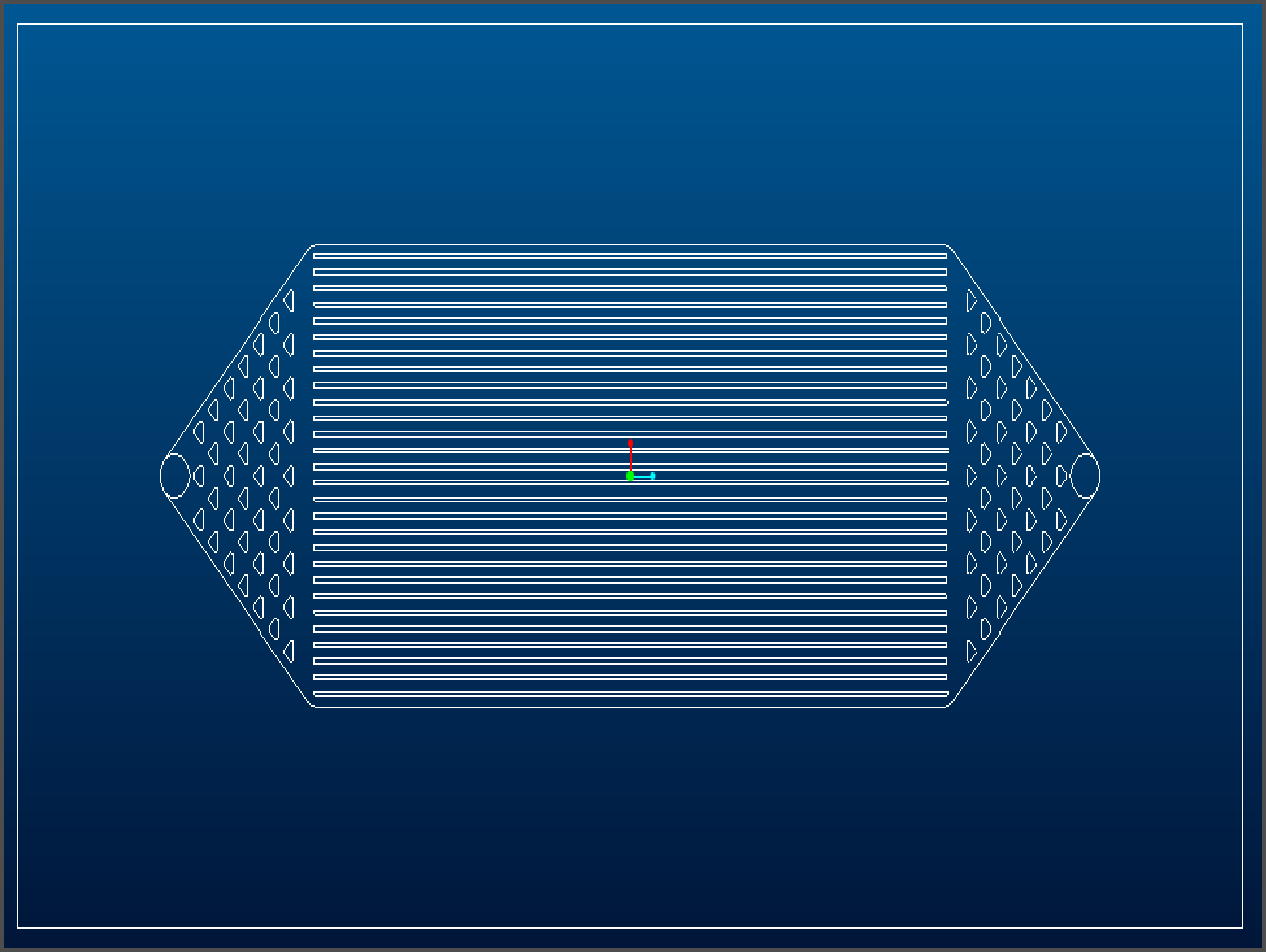
Dialyzer Unit Assembly Schematic

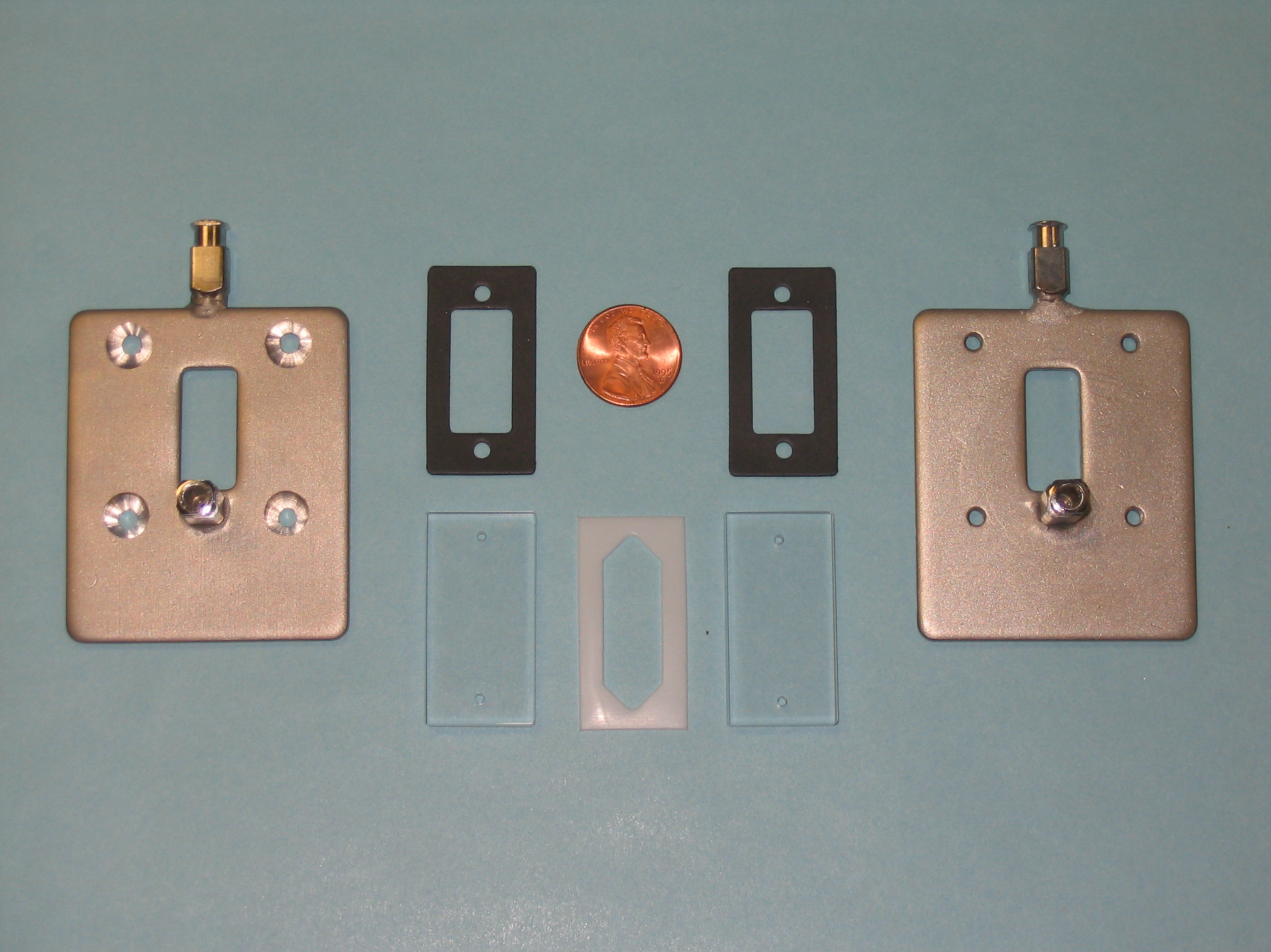


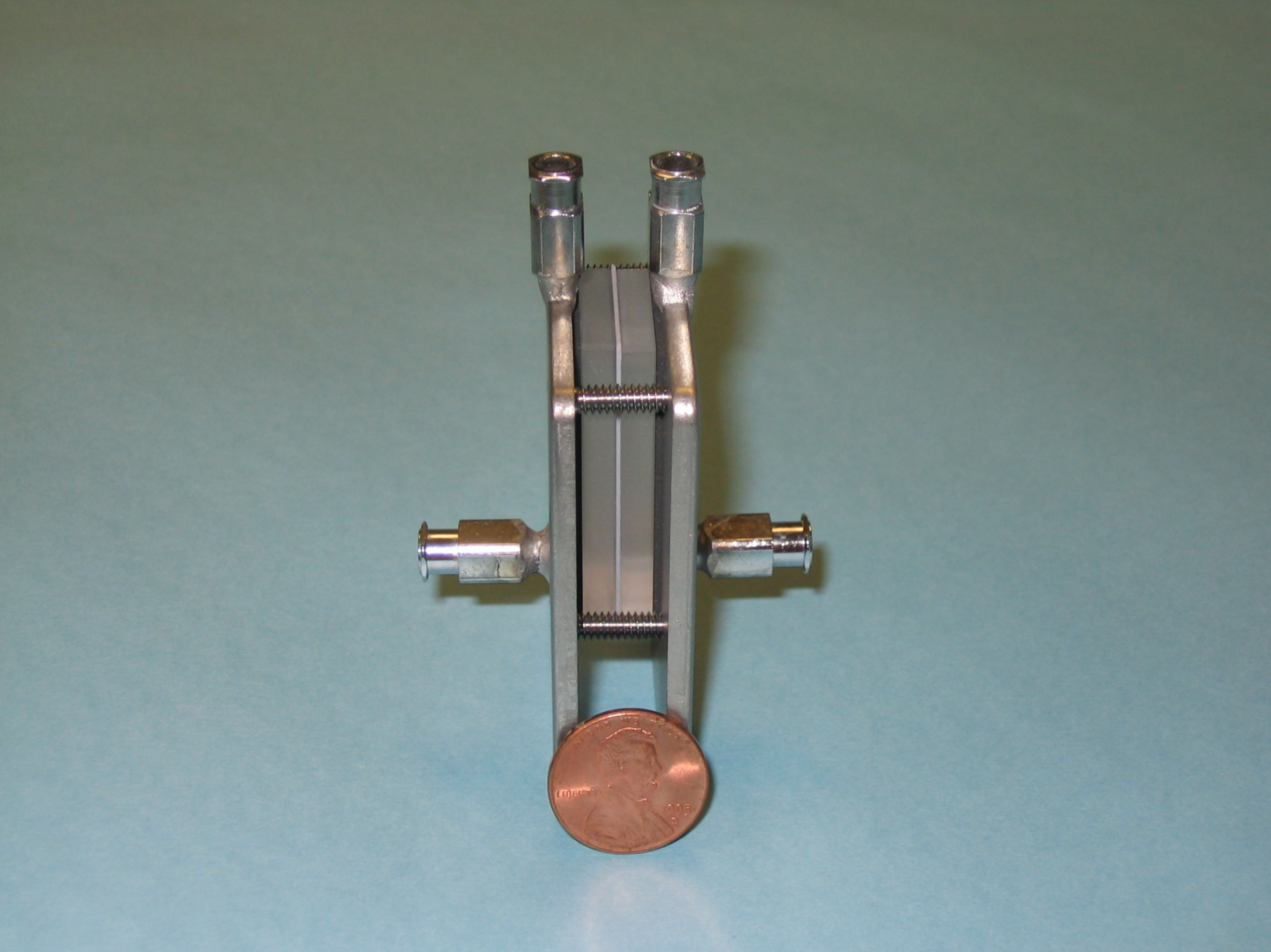


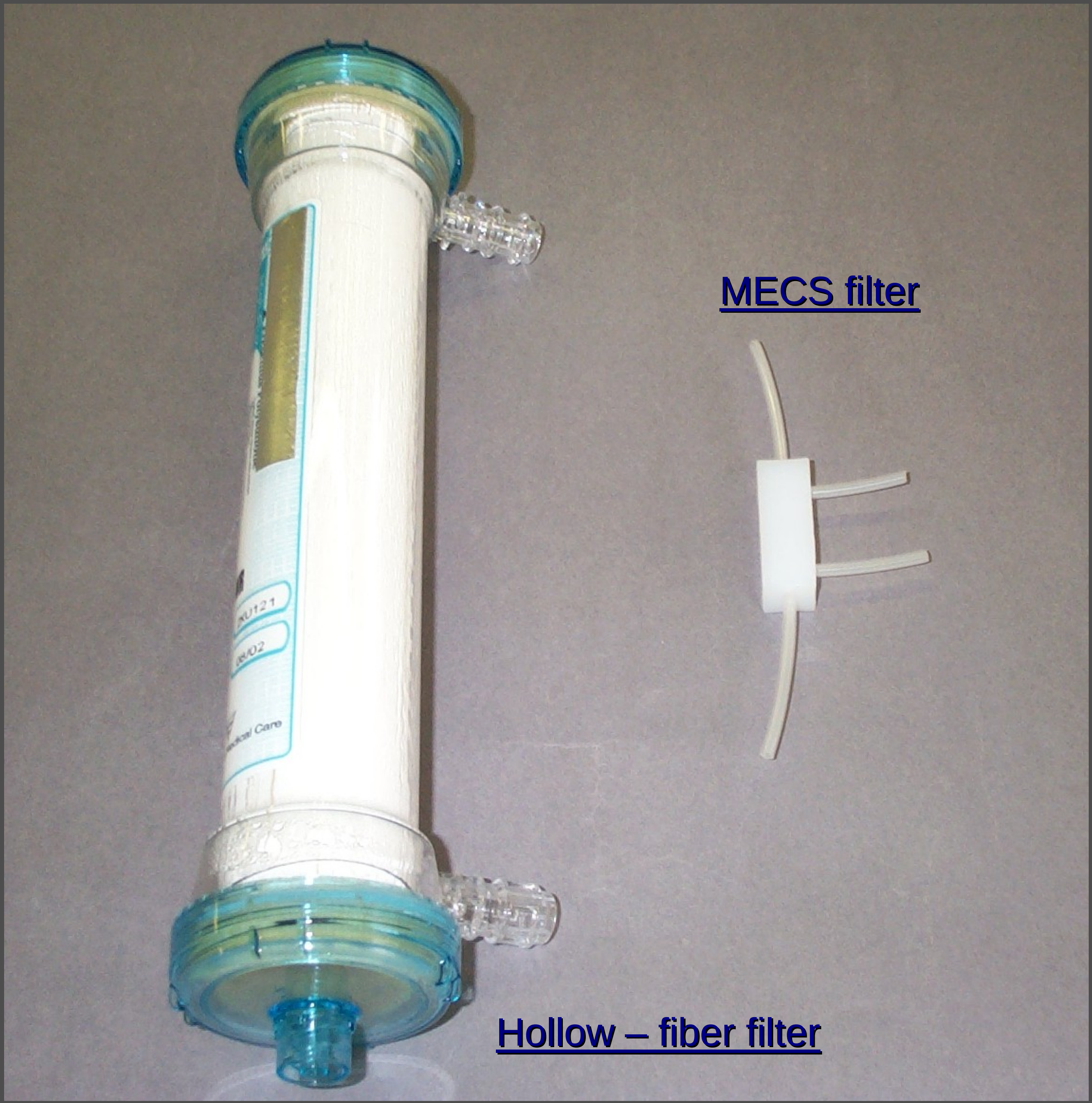
Prof. Goran Jovanovic, OSU ChE

MECS Dialyzer Design



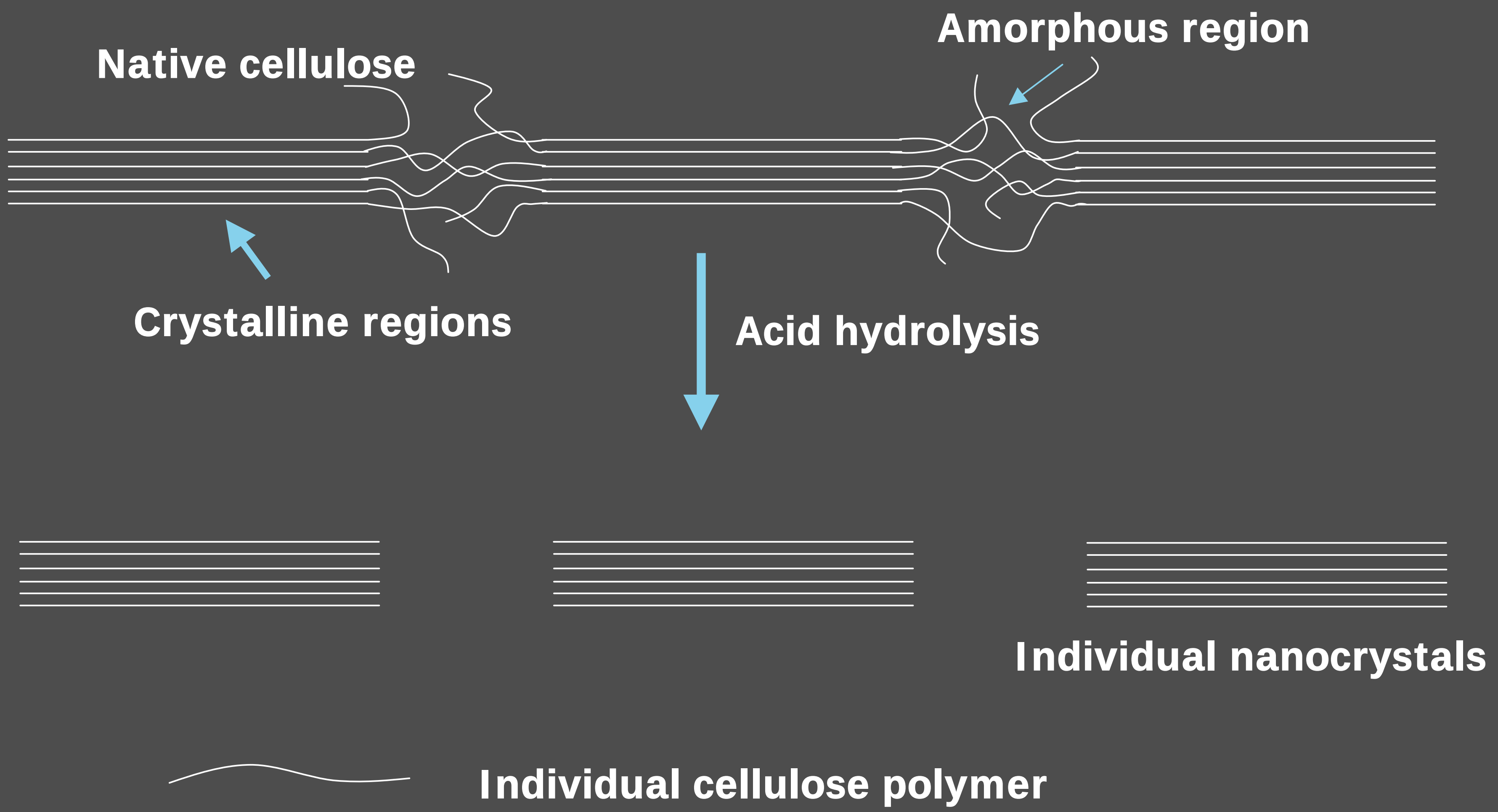





MECS filter
Hollow – fiber filter

Experimental Methods

Cellulose Nanocrystal Production



Polymer film preparation

- Solvent exchange to 1-methyl-2-pyrrolidone (NMP)
- Combine with polysulfone (PSf) in NMP
- Cast films two ways:
  - Coagulation via Loeb-Sourirajan anisotropic film
  - Solvent evaporation

Evaluation

- **Mechanical testing –Sintech 1G, Universal testing machine**
  - **tensile test mode**
  - **Data converted to stress-strain curves**
- **Thermal Analysis**
  - **Differential Scanning Calorimetry, TA Instruments DSC 2920**
    - **-Temperature range – 20-250ºC**
    - **- Heating/cooling rate – 10 ºC/min**
  - **Thermo gravimetric analysis , TA Instruments, Q500**
    - **- Temperature range – 40-600ºC**
    - **- Heating rate –10ºC/min**

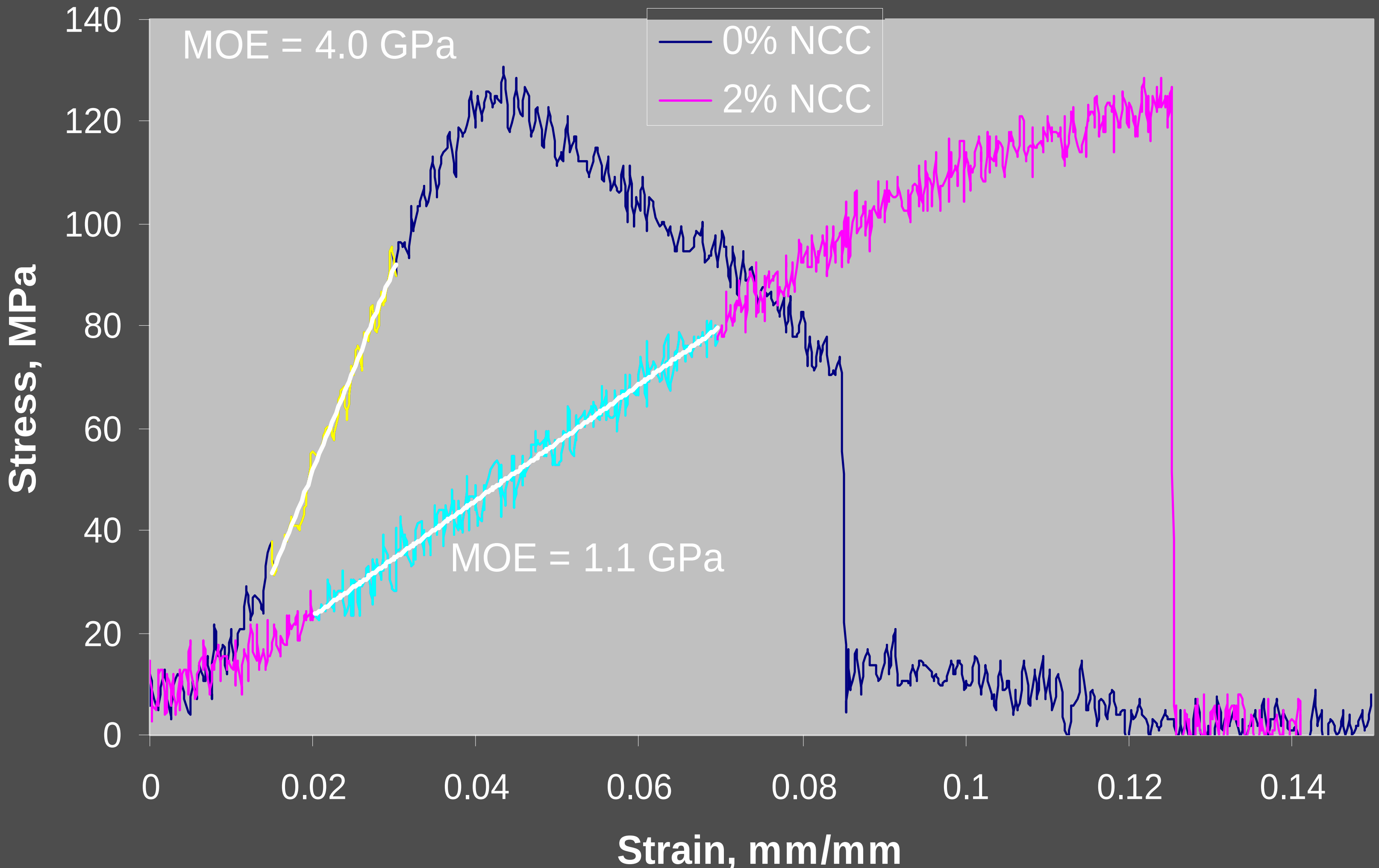
Evaluation

- Scanning electron microscopy (SEM)
  - AMRay 1000A @ 10 kV
  - Coated with Au-Pd film (8-10 nm)

- Atomic force microscopy (AFM)
  - DI Dimension 3100 (Veeco Instruments)
  - Tapping mode

Mechanical properties

Stress-strain

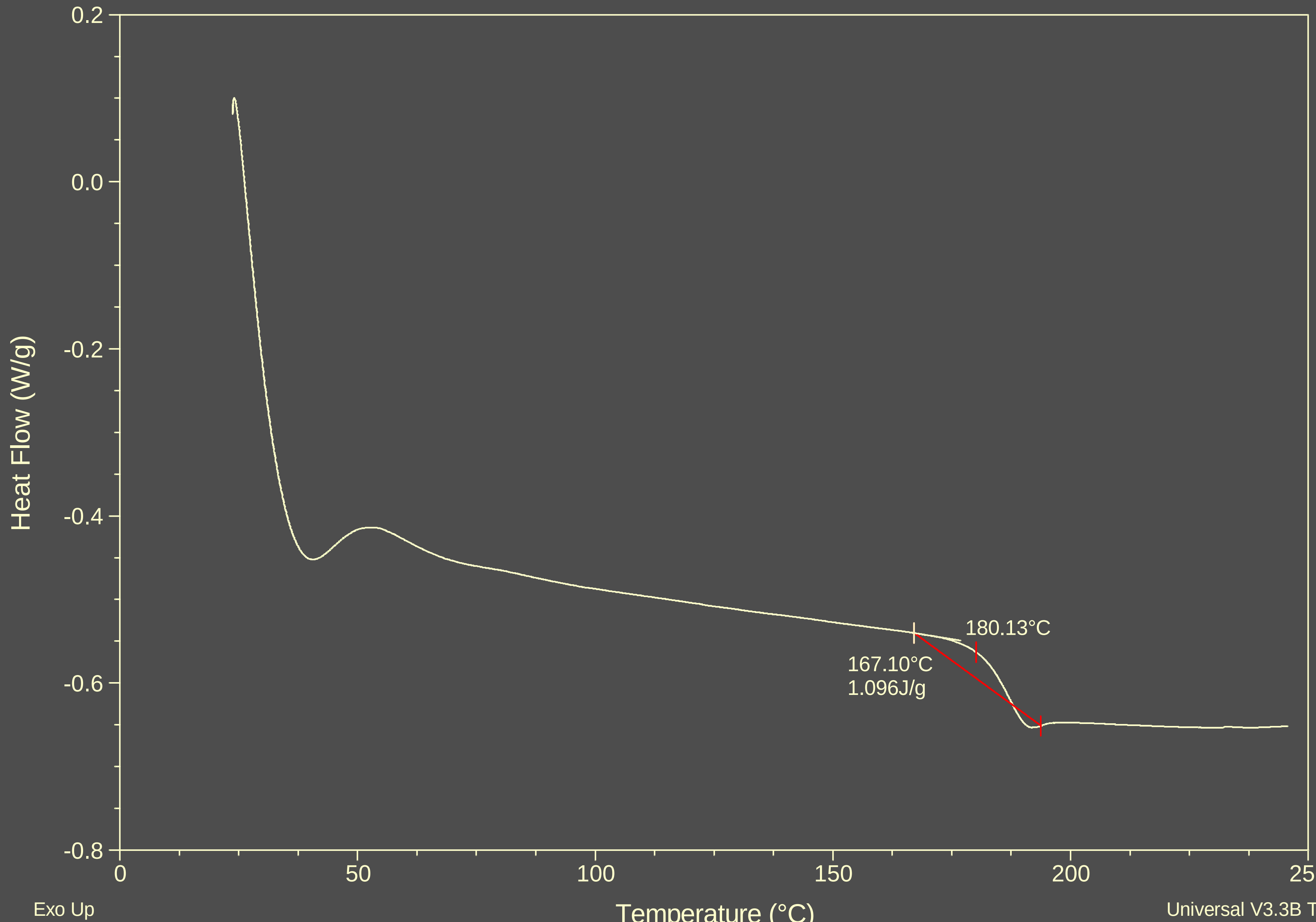


Thermal Properties

Sample: psf film nov 17,04
Size: 5.0000 mg
Method: Ramp
Comment: PSF FILM

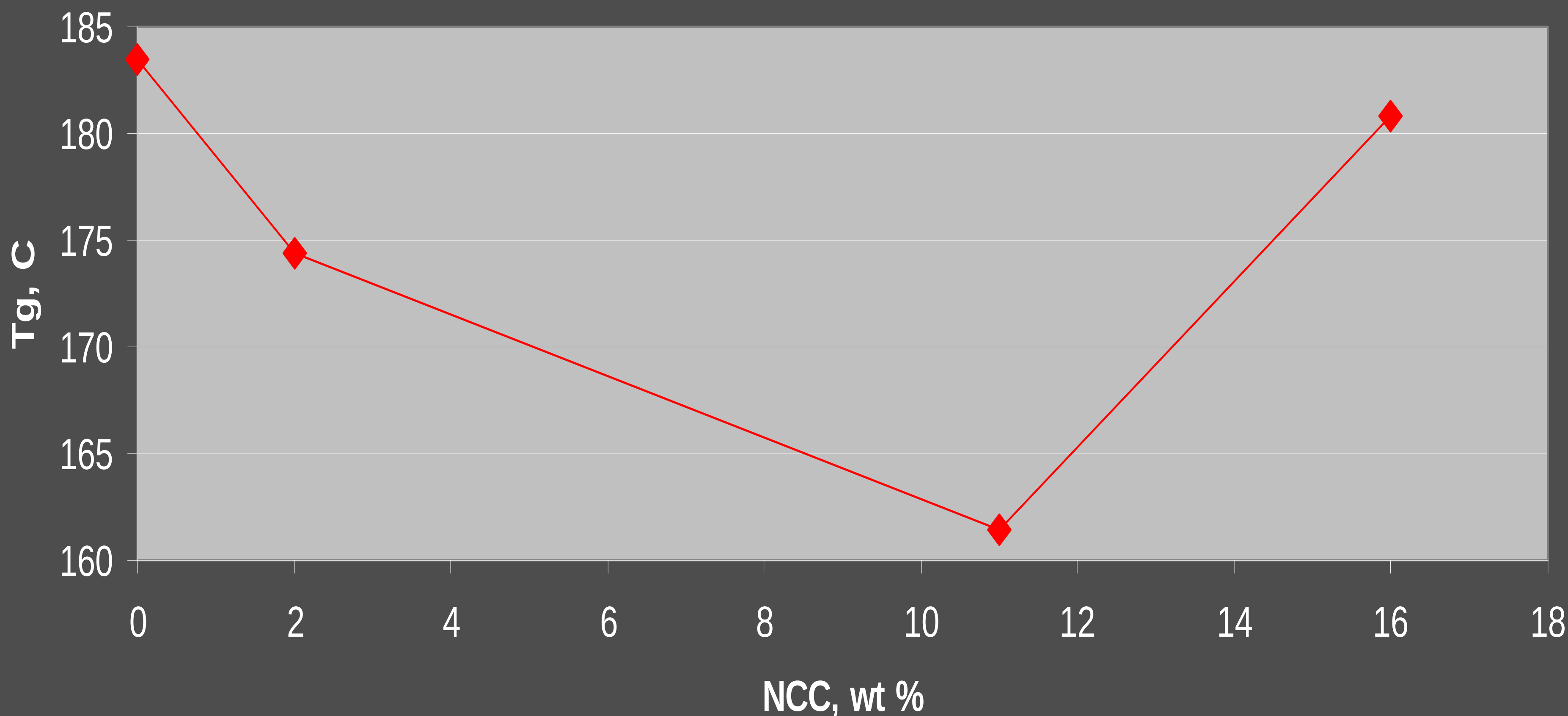
DSC

File: C:...\sweda\psf film nov 17,04(DSC).002
Operator: sweda
Run Date: 17-Nov-04 15:23
Instrument: 2920 MDSC V2.6A

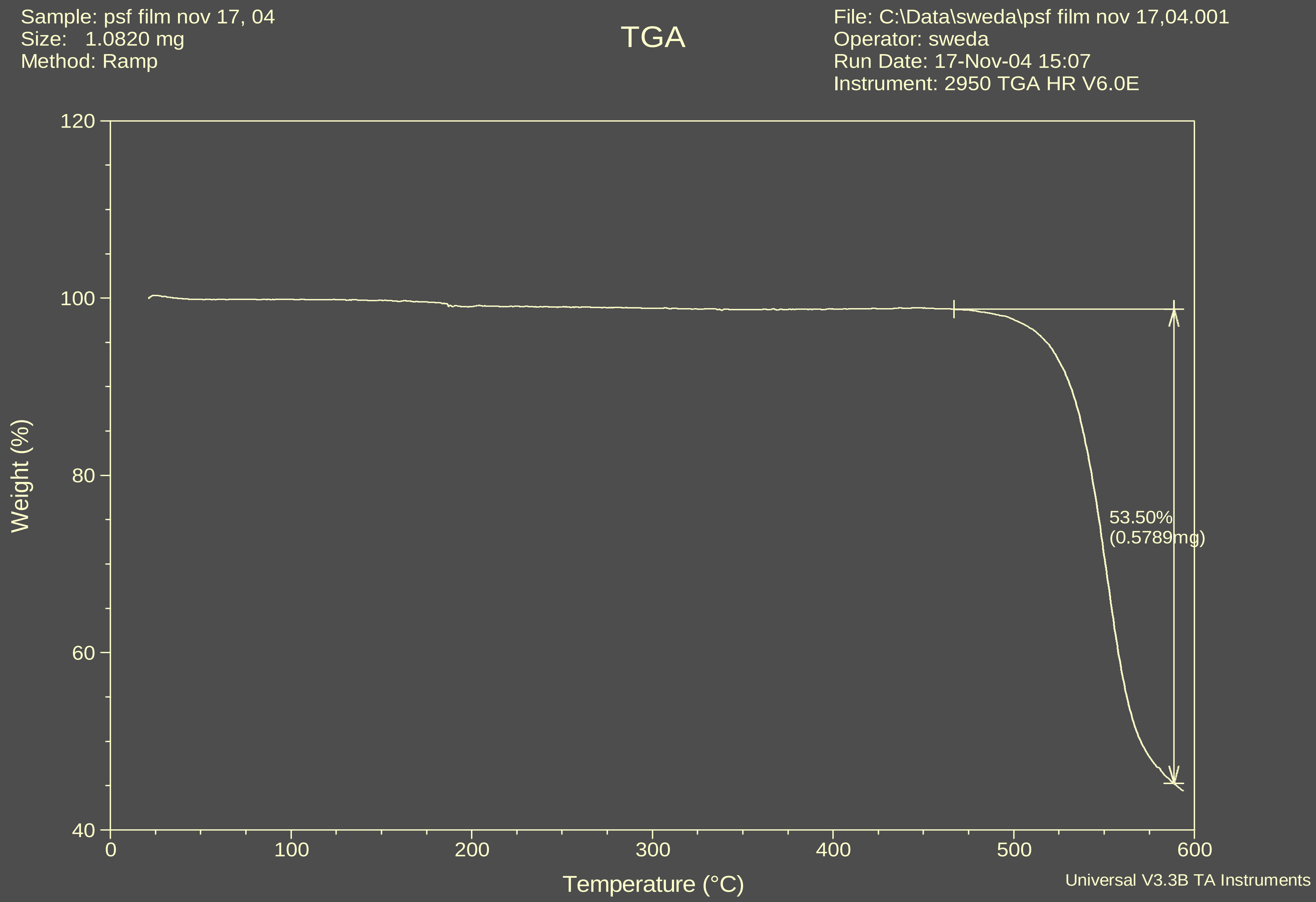


Universal V3.3B TA Instruments

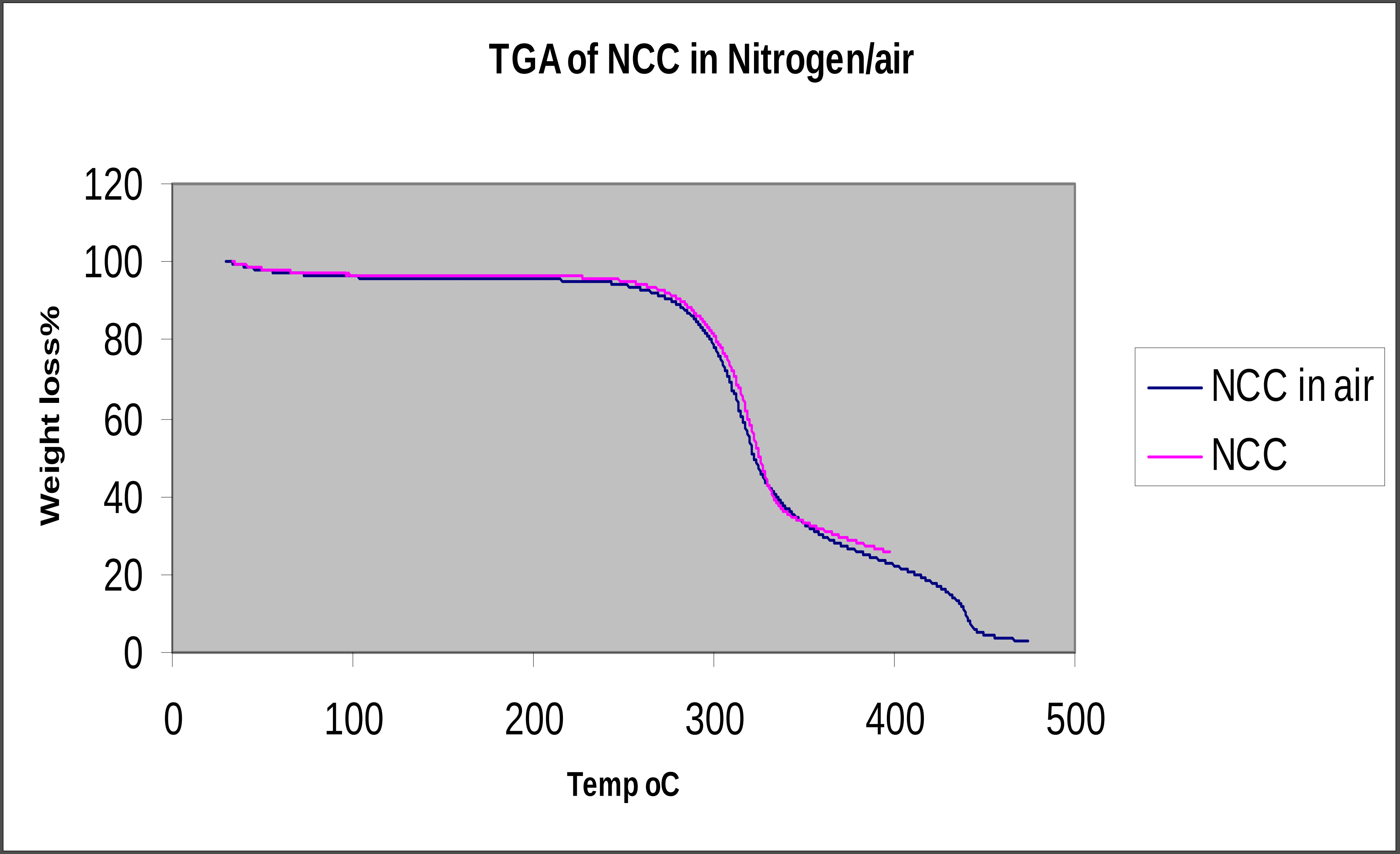
T_g of NCC-filled PSf



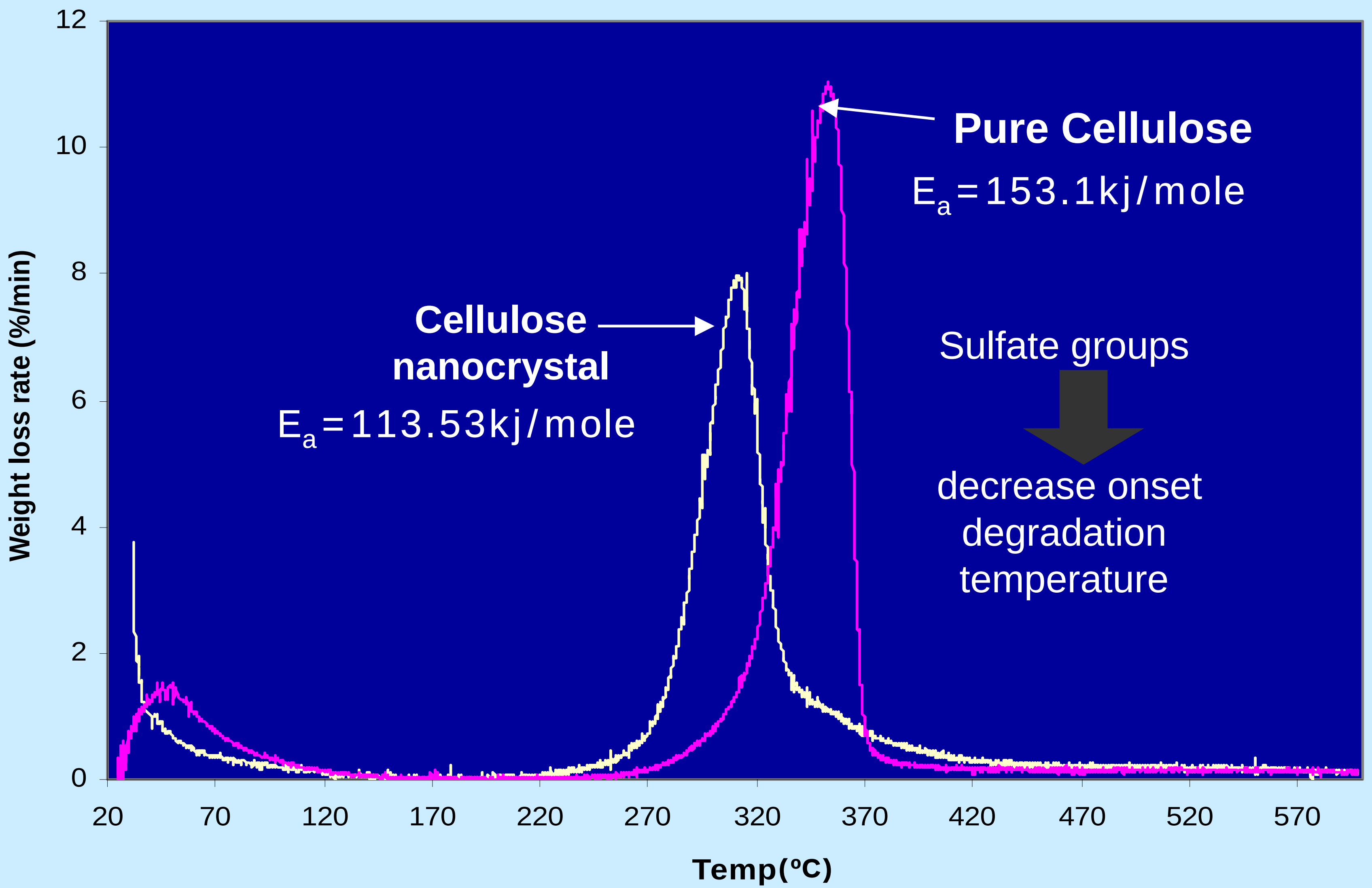
TGA Psf



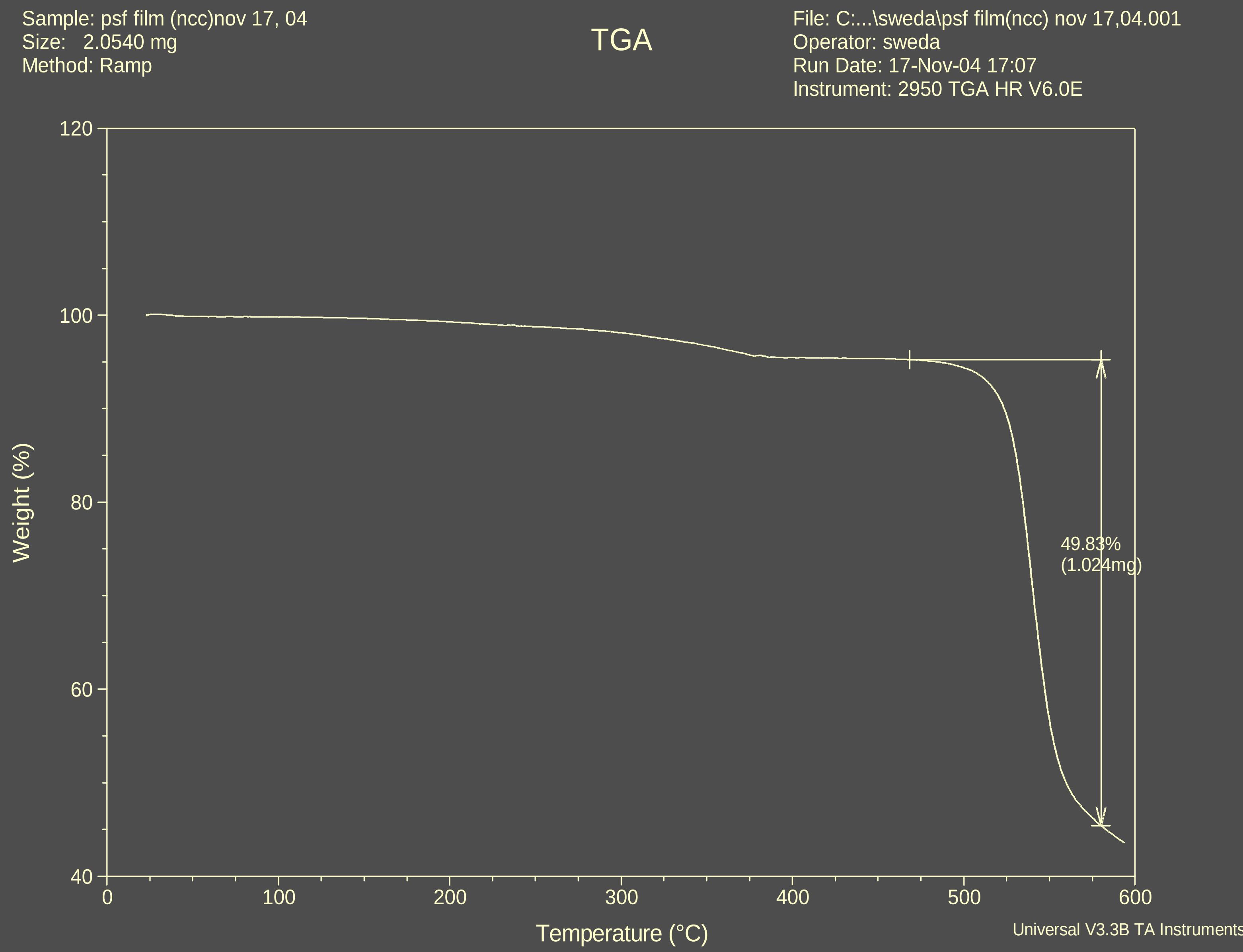
Sample: psf film nov 17, 04
Size: 1.0820 mg
Method: Ramp
TGA
File: C:\Data\sweda\psf film nov 17,04.001
Operator: sweda
Run Date: 17-Nov-04 15:07
Instrument: 2950 TGA HR V6.0E
Weight (%)
120
100
80
60
40
0
100
200
300
400
500
600
Temperature (°C)
53.50%
(0.5789mg)
Universal V3.3B TA Instruments


TGA of NCC in Nitrogen/air
Weight loss%
120
100
80
60
40
20
0
0
100
200
300
400
500
Temp oC
NCC in air
NCC

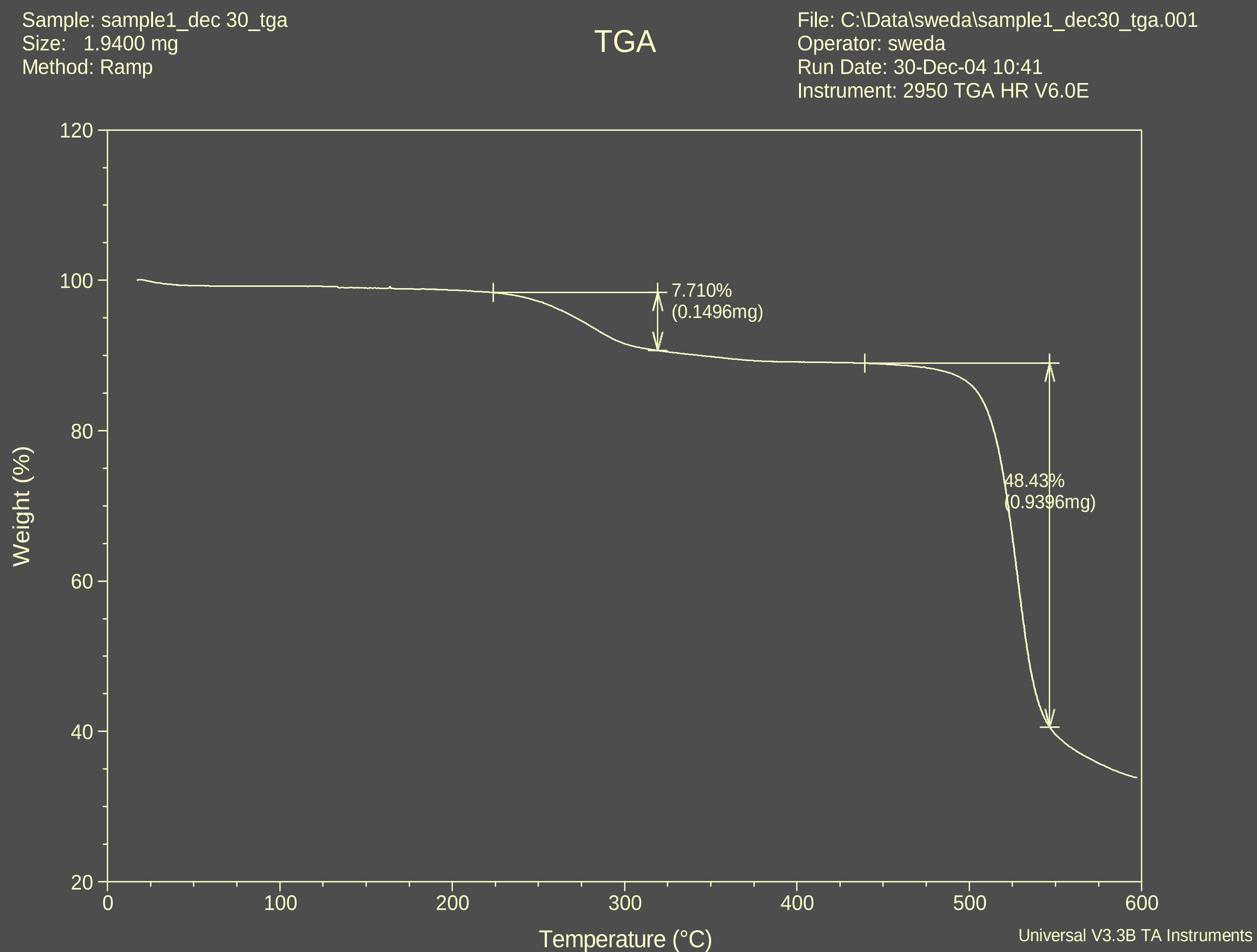
Derivative Thermogravimetric (DTG) Curves



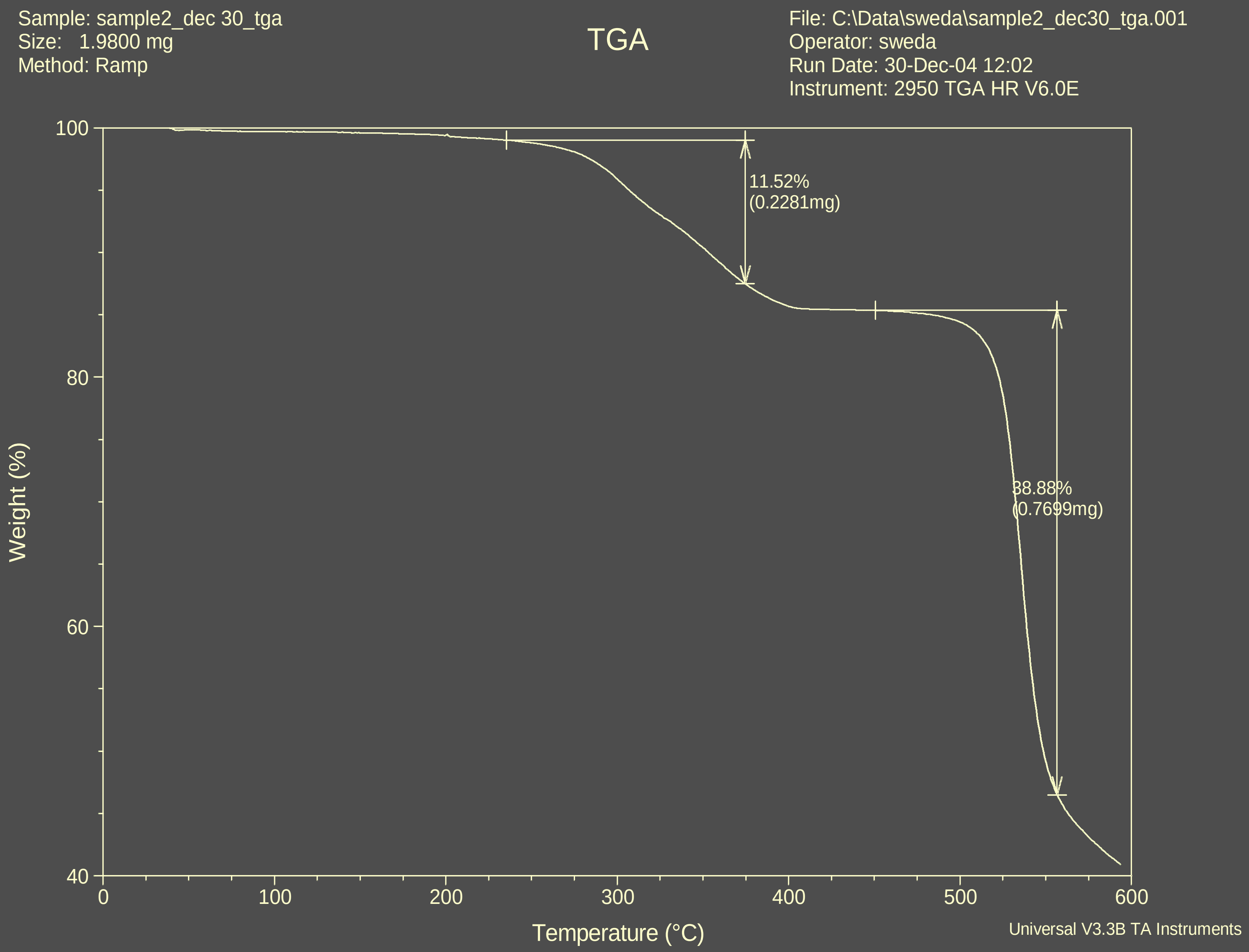
TGA (Psf film with 2% NCC)



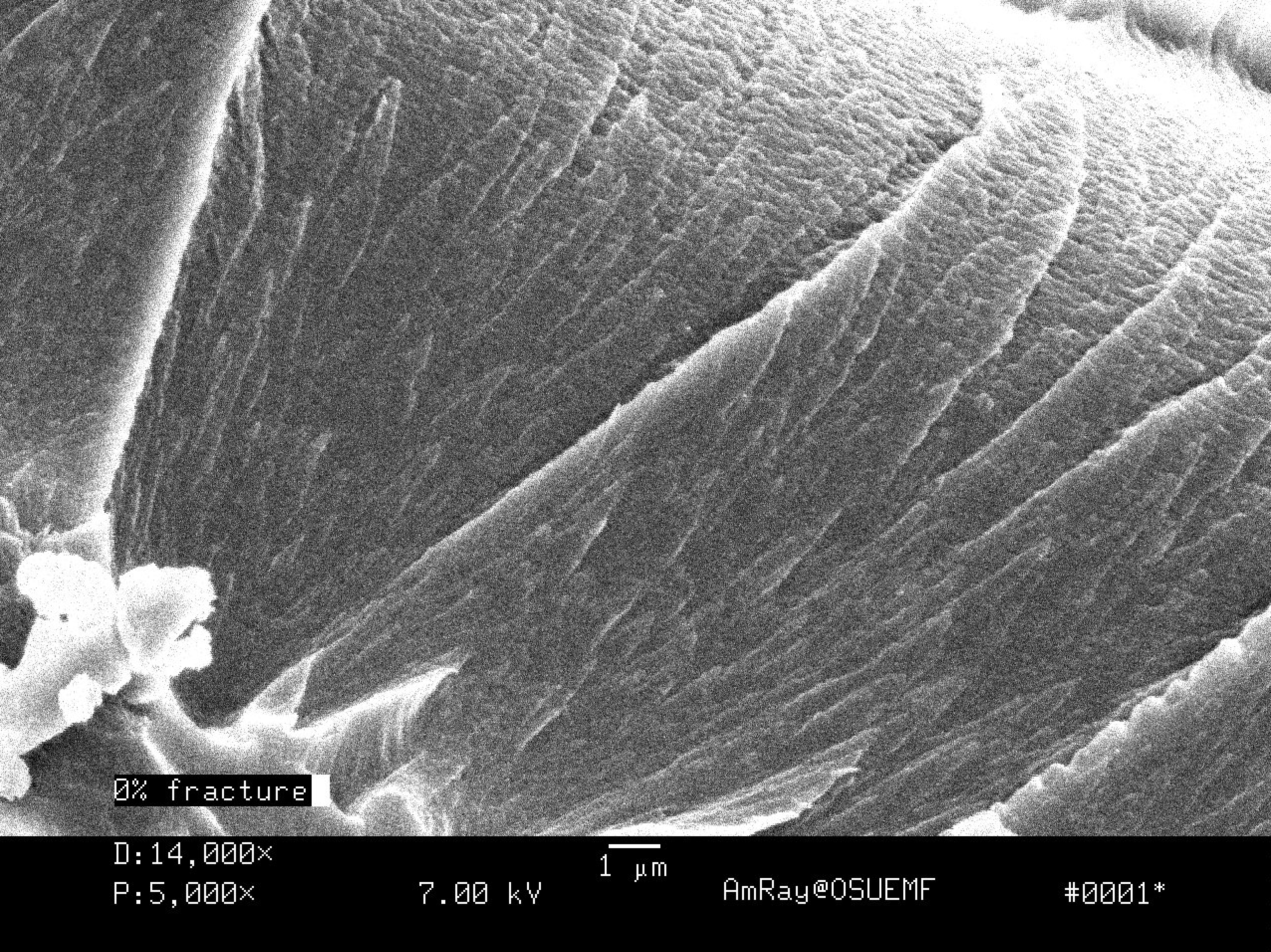
TGA-11% NCC

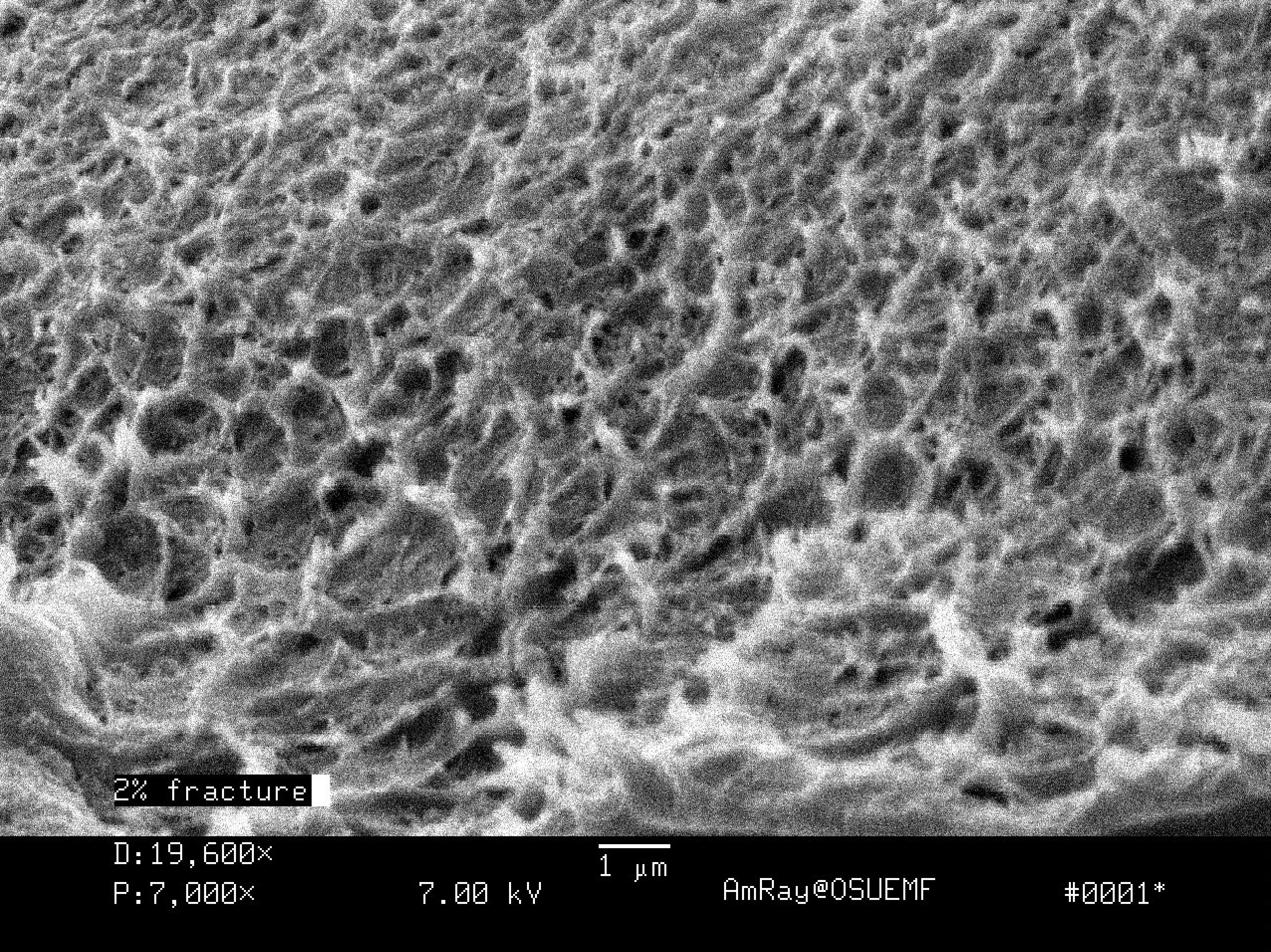

Sample: sample1_dec 30_tga
Size: 1.9400 mg
Method: Ramp
TGA
File: C:\Data\sweda\sample1_dec30_tga.001
Operator: sweda
Run Date: 30-Dec-04 10:41
Instrument: 2950 TGA HR V6.0E
7.710%
(0.1496mg)
48.43%
(0.9396mg)
Weight (%)
Temperature (°C)
120
100
80
60
40
20
0
100
200
300
400
500
600
Universal V3.3B TA Instruments

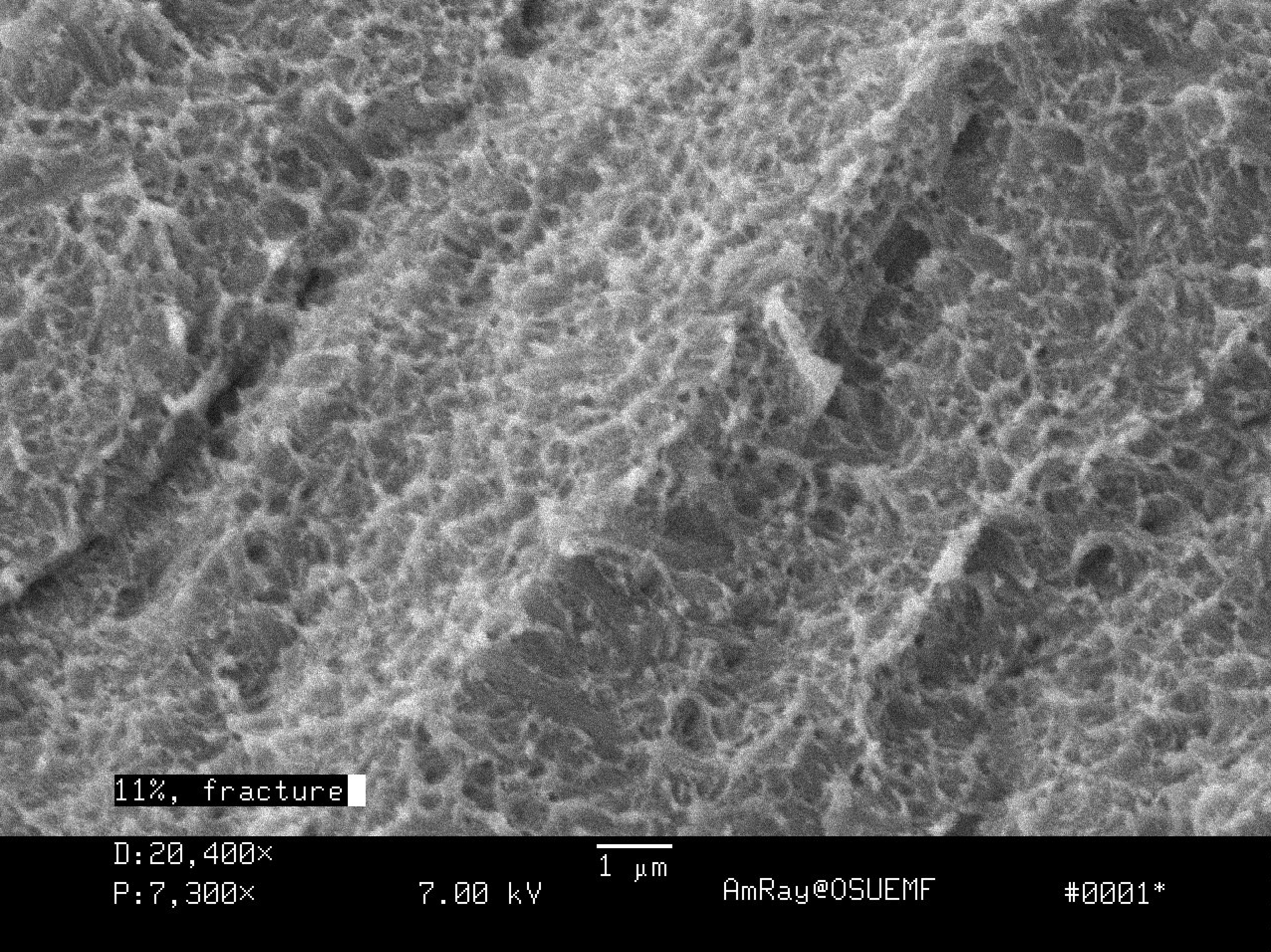
TGA-16% NCC

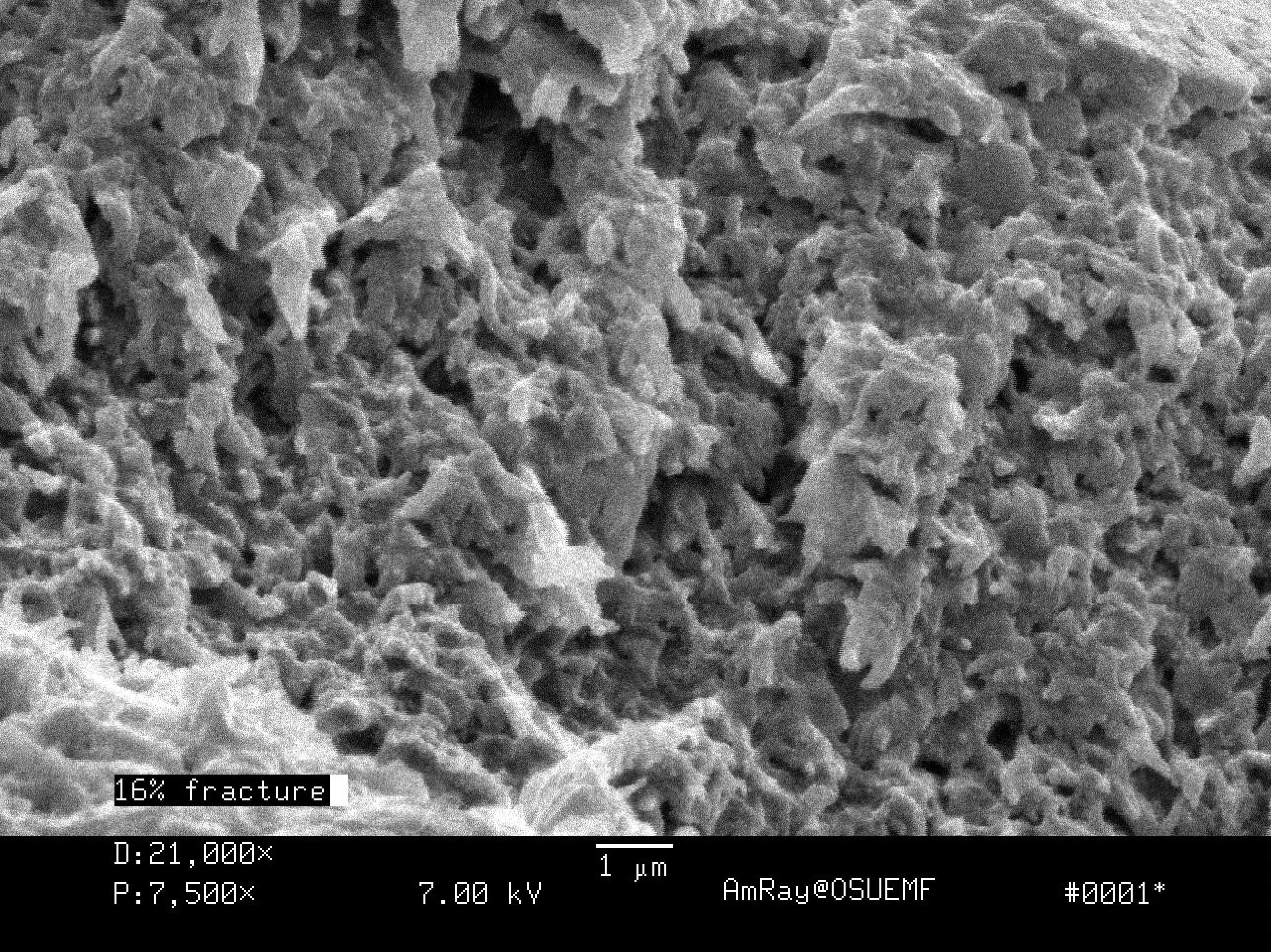

Sample: sample2_dec 30_tga
Size: 1.9800 mg
Method: Ramp
TGA
File: C:\Data\sweda\sample2_dec30_tga.001
Operator: sweda
Run Date: 30-Dec-04 12:02
Instrument: 2950 TGA HR V6.0E
11.52%
(0.2281mg)
38.88%
(0.7699mg)
100
80
60
40
Weight (%)
0
100
200
300
400
500
600
Temperature (°C)
Universal V3.3B TA Instruments

SEM

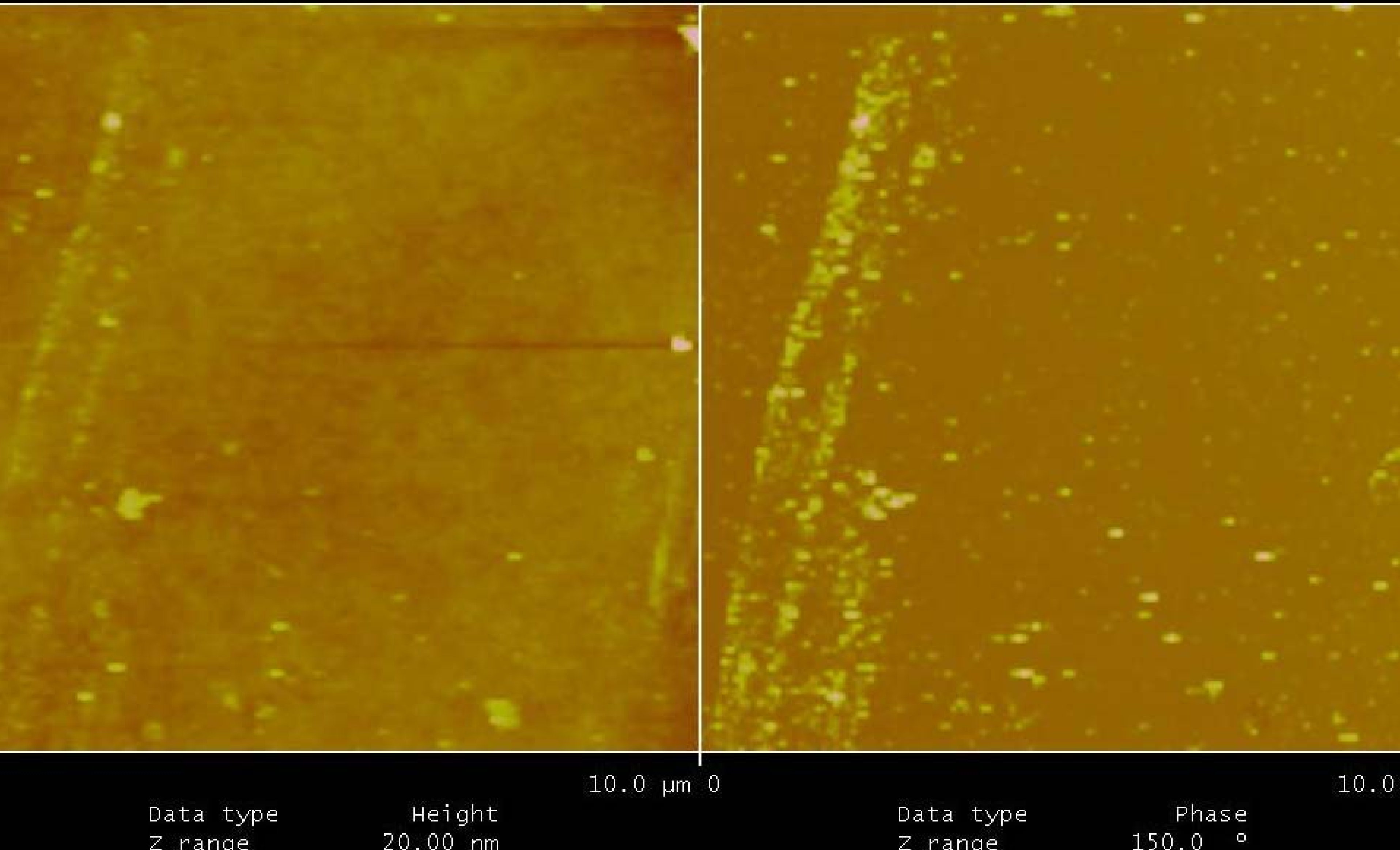

0% fracture
D:14,000×
P:5,000×
7.00 kV
1 µm
AmRay@OSUEMF
#0001*


2% fracture
D:19,600×
1 µm
P:7,000×
7.00 kV
AmRay@OSUEMF
#0001*


11%, fracture
D:20,400×
P:7,300×
1 µm
7.00 kV
AmRay@OSUEMF
#0001*

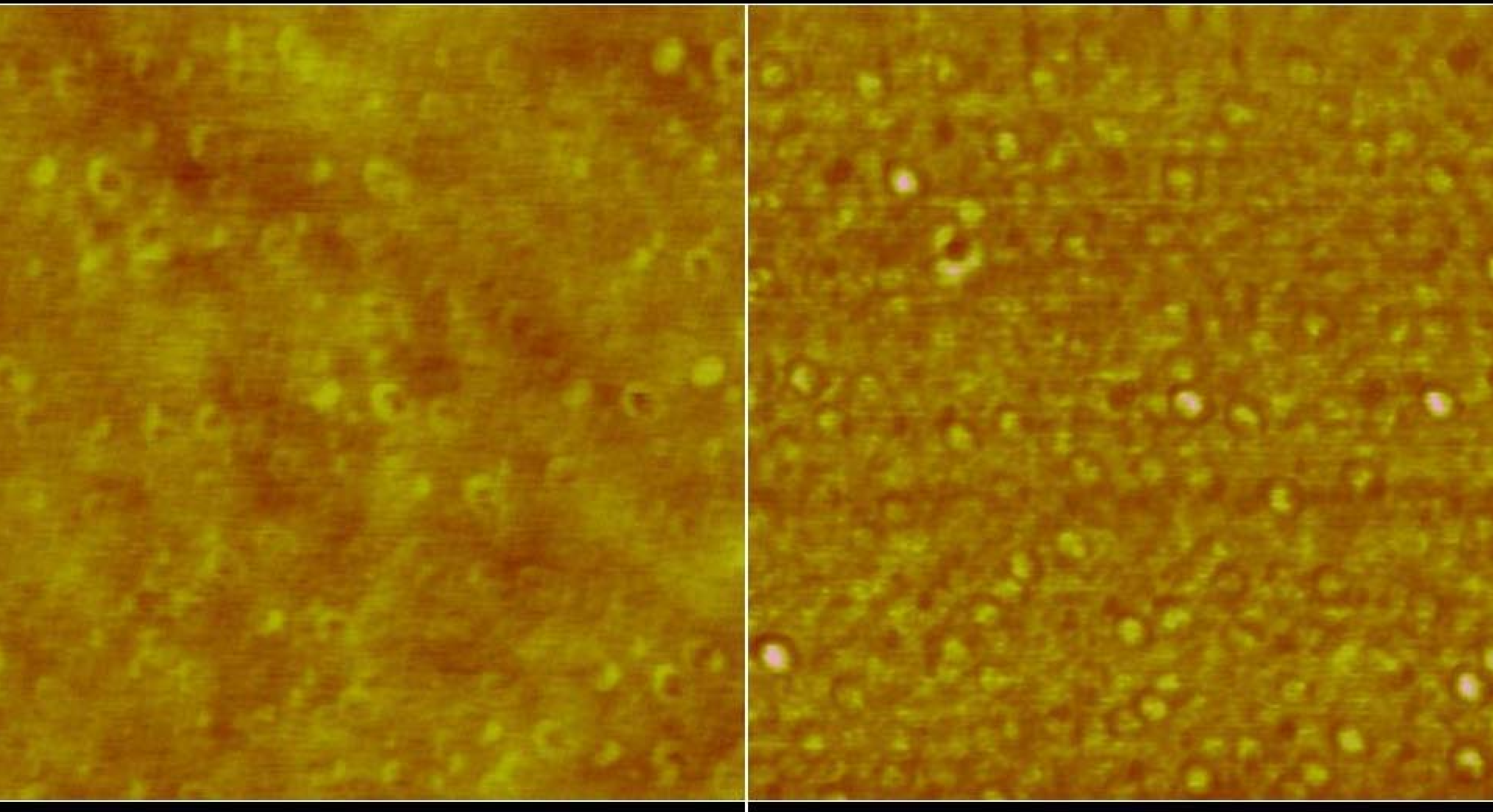

16% fracture
D:21,000×
P:7,500×
7.00 kV
1 µm
AmRay@OSUEMF
#0001*

AFM images



sf10micron2.000
/05 0% NCC in PSf second location

0% NCC



Data type Height
Z range 5.000 nm

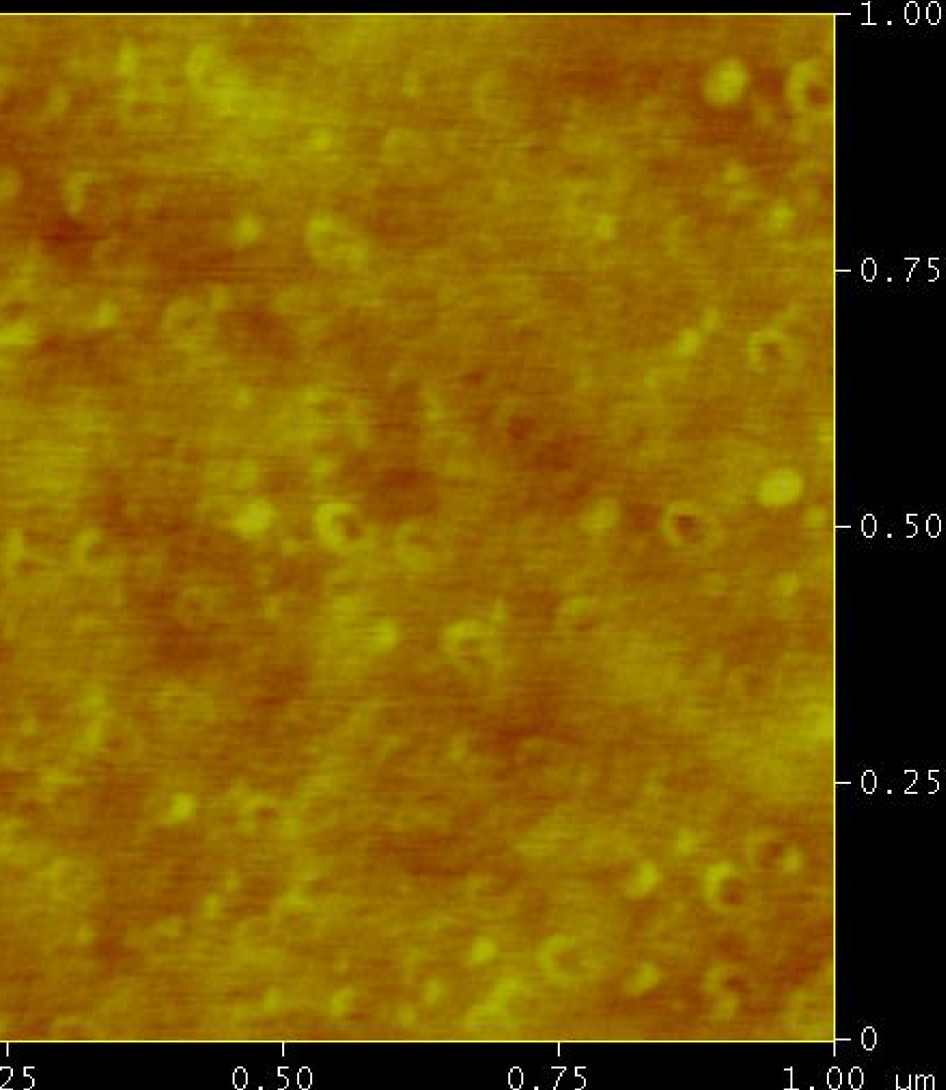
Data type Phase
Z range 70.00 °

sf1micron2.001
/05 0% NCC in PSf second location

0% NCC

Roughness Analysis

R_{ms} = 0.184 nm



NCC in PSf second location
2.001

Image Statistics

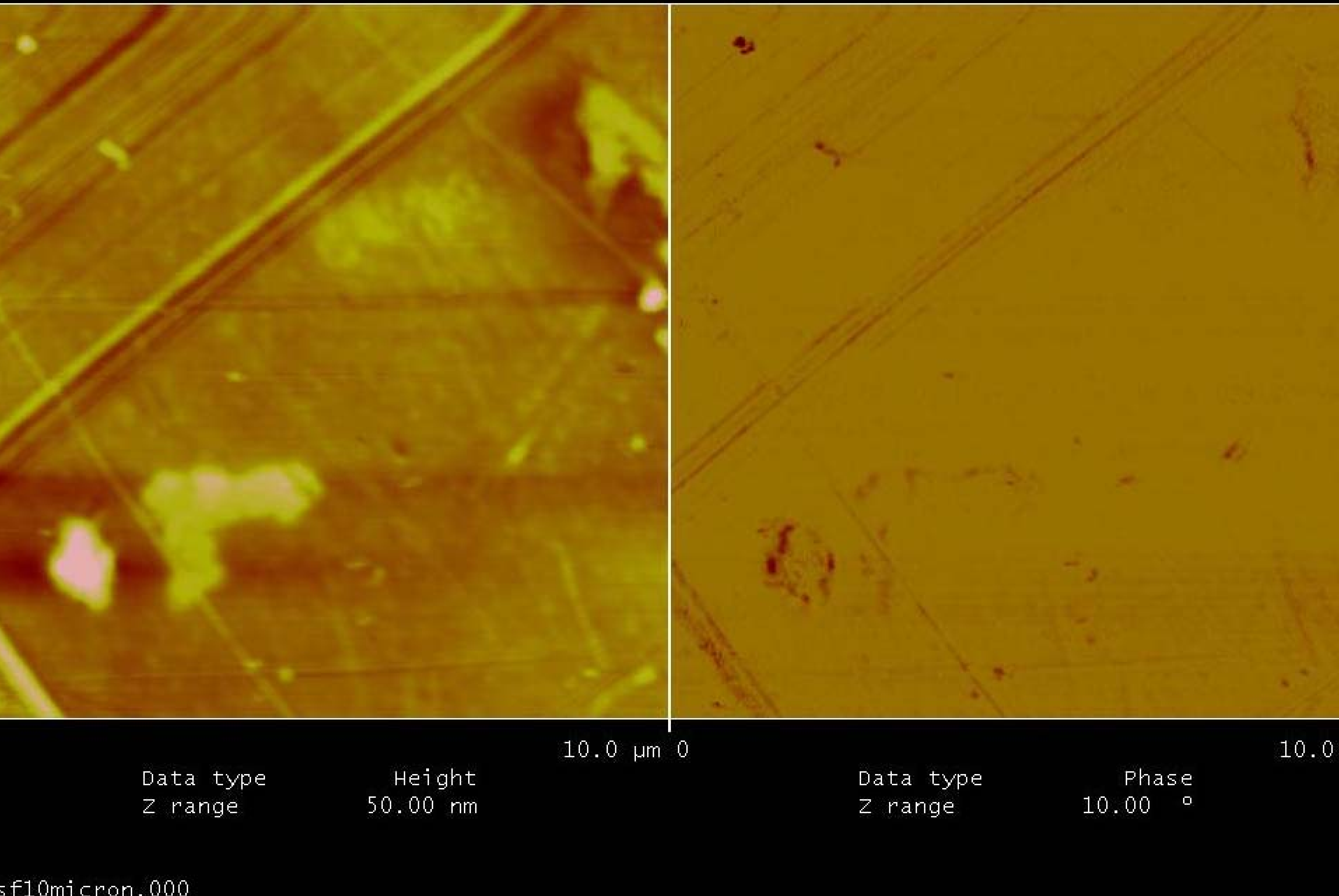
Img. Z range	1.733 nm
Img. Mean	-0.000000 nm
Img. Raw mean	-1.325 µm
Img. Rms (Rq)	0.184 nm
Img. Ra	0.143 nm
Img. Rmax	1.733 nm
Img. Srf. area	1.001 µm²
Img. Prj. Srf. area	999999 nm²
Img. Srf. area diff	0.059 %
Img. SAE	1.000

Box Statistics

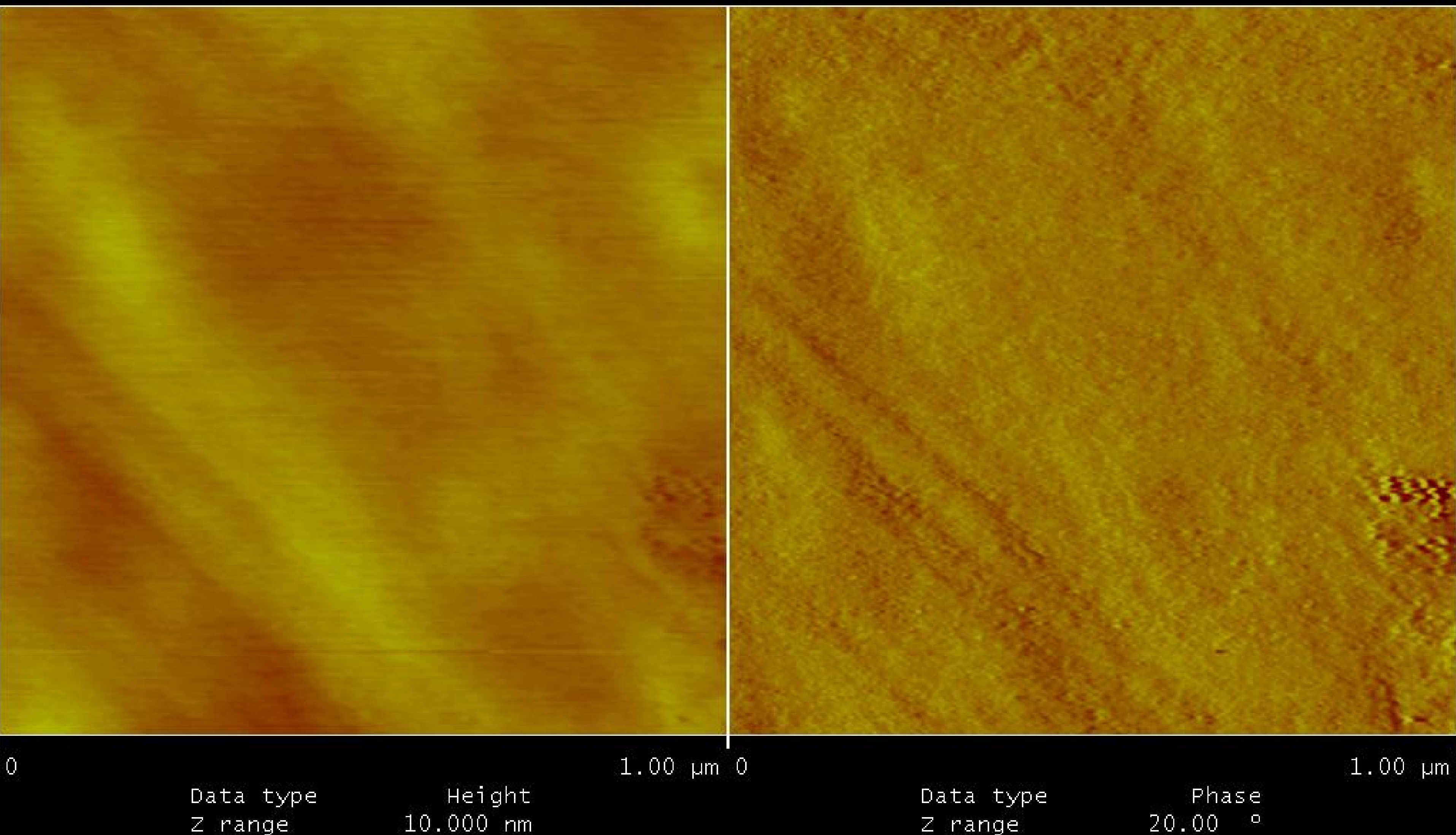
Z range	
Mean	
Raw mean	
Rms (Rq)	
Mean roughness (Ra)	
Max height (Rmax)	

0% NCC

Summit Off   Zero Cross. Off   Box Cursor

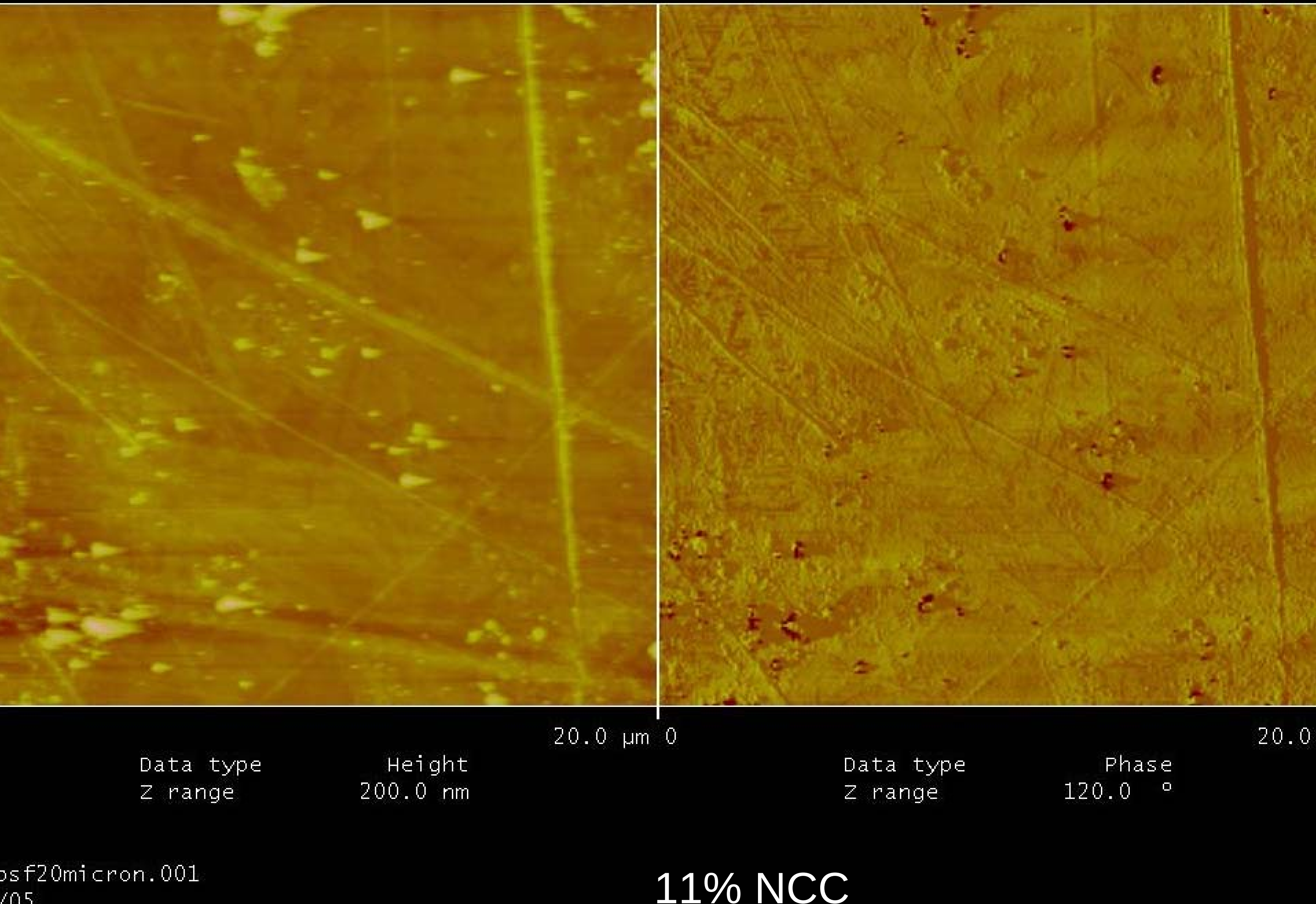

10.0 µm 0
10.0
Data type Height
Z range 50.00 nm
Data type Phase
Z range 10.00 °
sf10micron.000

2% NCC

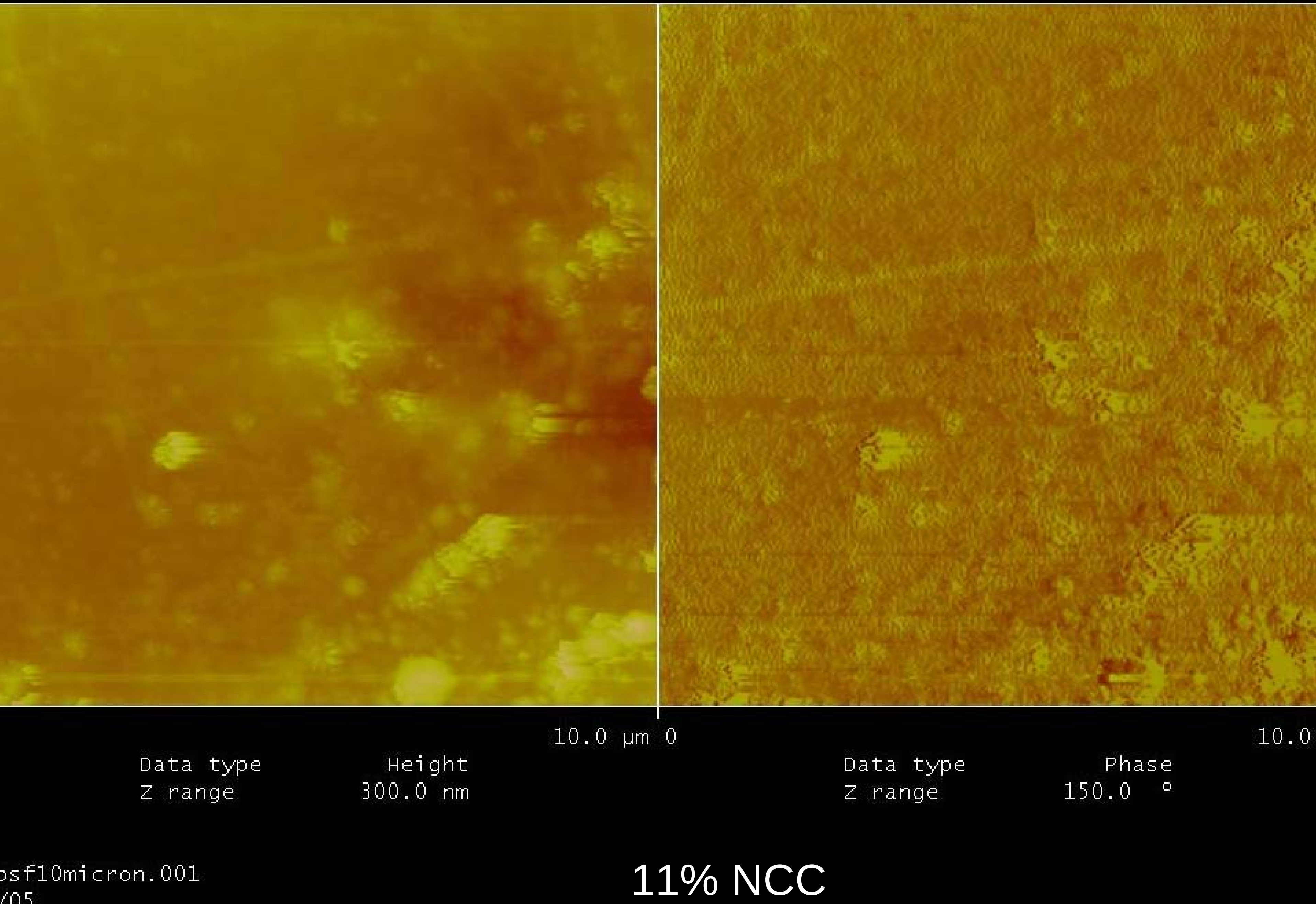


2%psf1micron.001
3/9/05 2% NCC in PSf, film

2% NCC


20.0 µm 0
Data type Height
Z range 200.0 nm
20.0
Data type Phase
Z range 120.0 °
osf20micron.001
/05

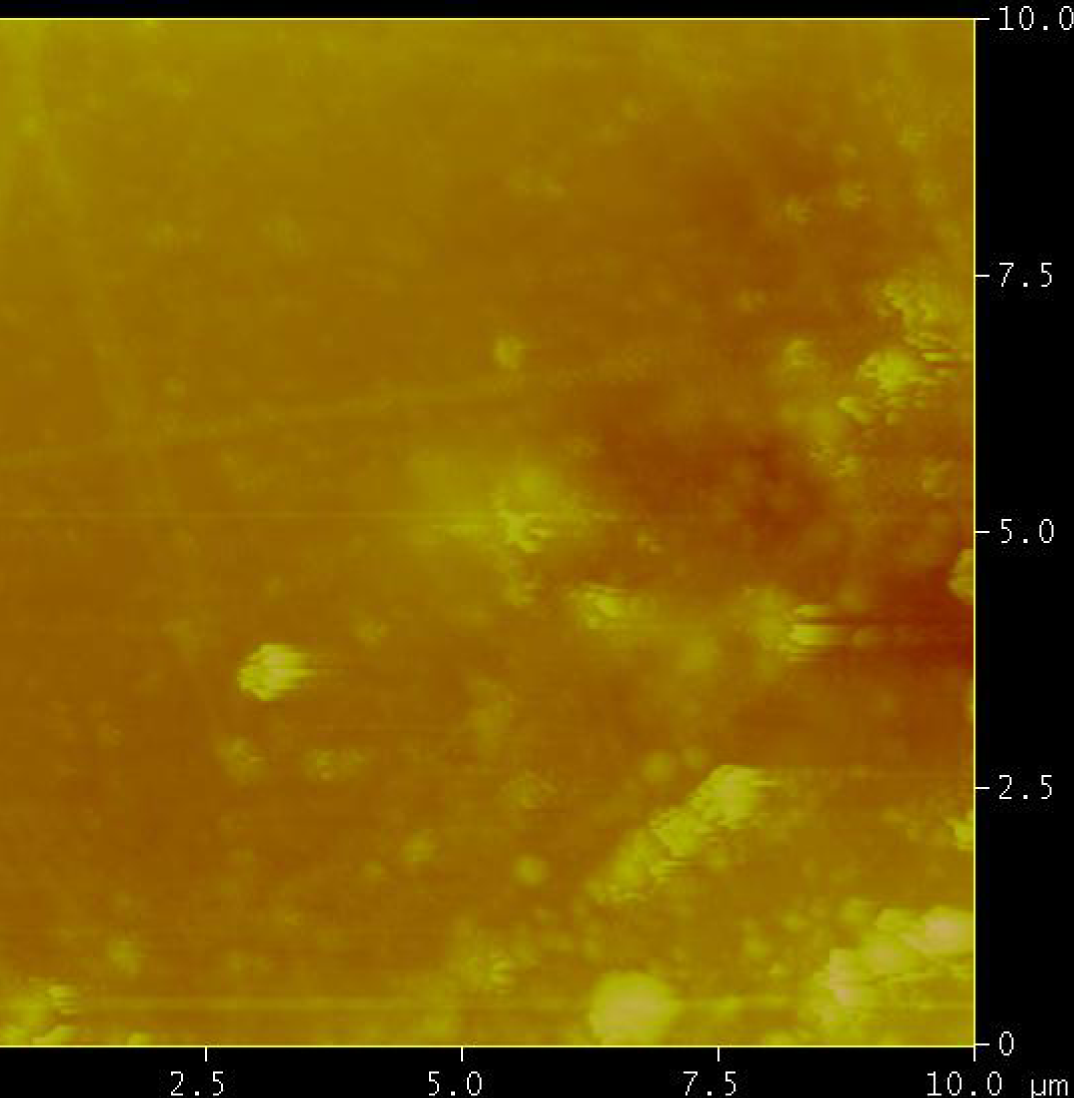
11% NCC


10.0 µm 0
10.0
Data type
Height
Z range
300.0 nm
Data type
Phase
Z range
150.0 °
sf10micron.001
/05

11% NCC

Roughness Analysis

R_{ms} = 13 nm



3/5/05
%psf10micron.001

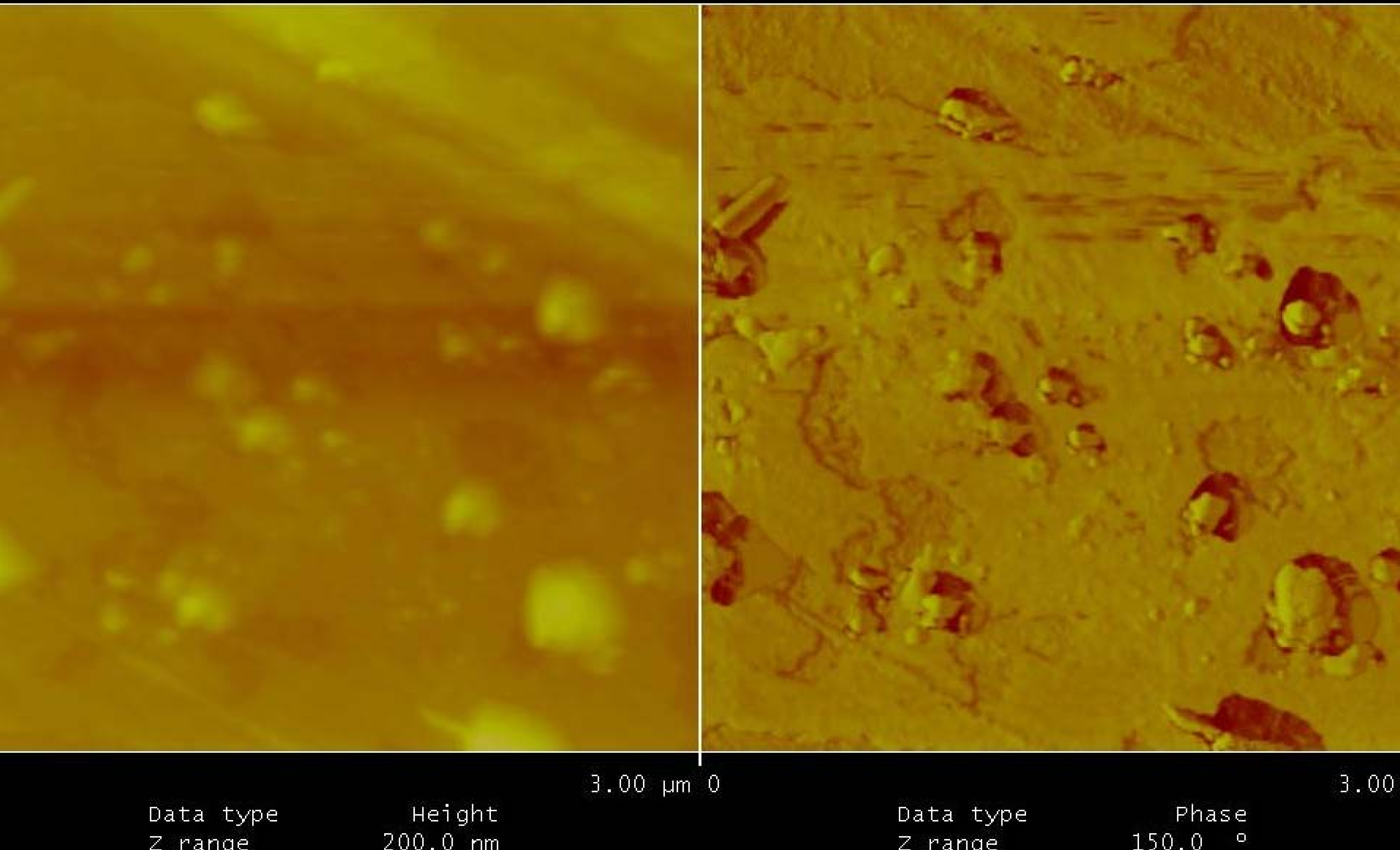
Image Statistics

Img. Z range	143.08 nm
Img. Mean	0.039 nm
Img. Raw mean	-464.60 nm
Img. Rms (Rq)	12.641 nm
Img. Ra	8.876 nm
Img. Rmax	142.41 nm
Img. Srf. area	100.91 µm²
Img. Prj. Srf. area	100.00 µm²
Img. Srf. area diff	0.915 %
Img. SAE	1.003

Box Statistics

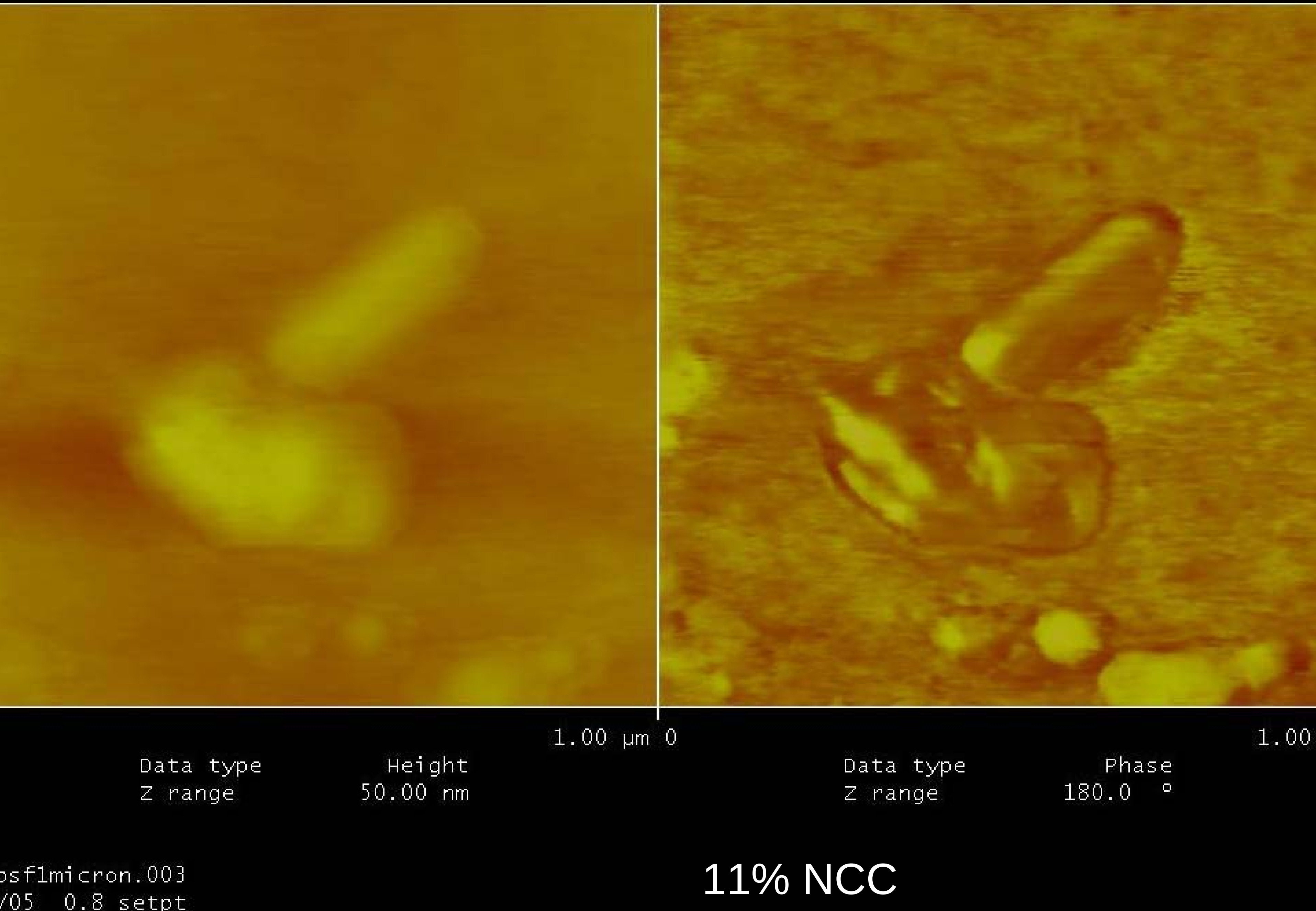
Z range	
Mean	
Raw mean	
Rms (Rq)	
Mean roughness (Ra)	
Max height (Rmax)	

11% NCC

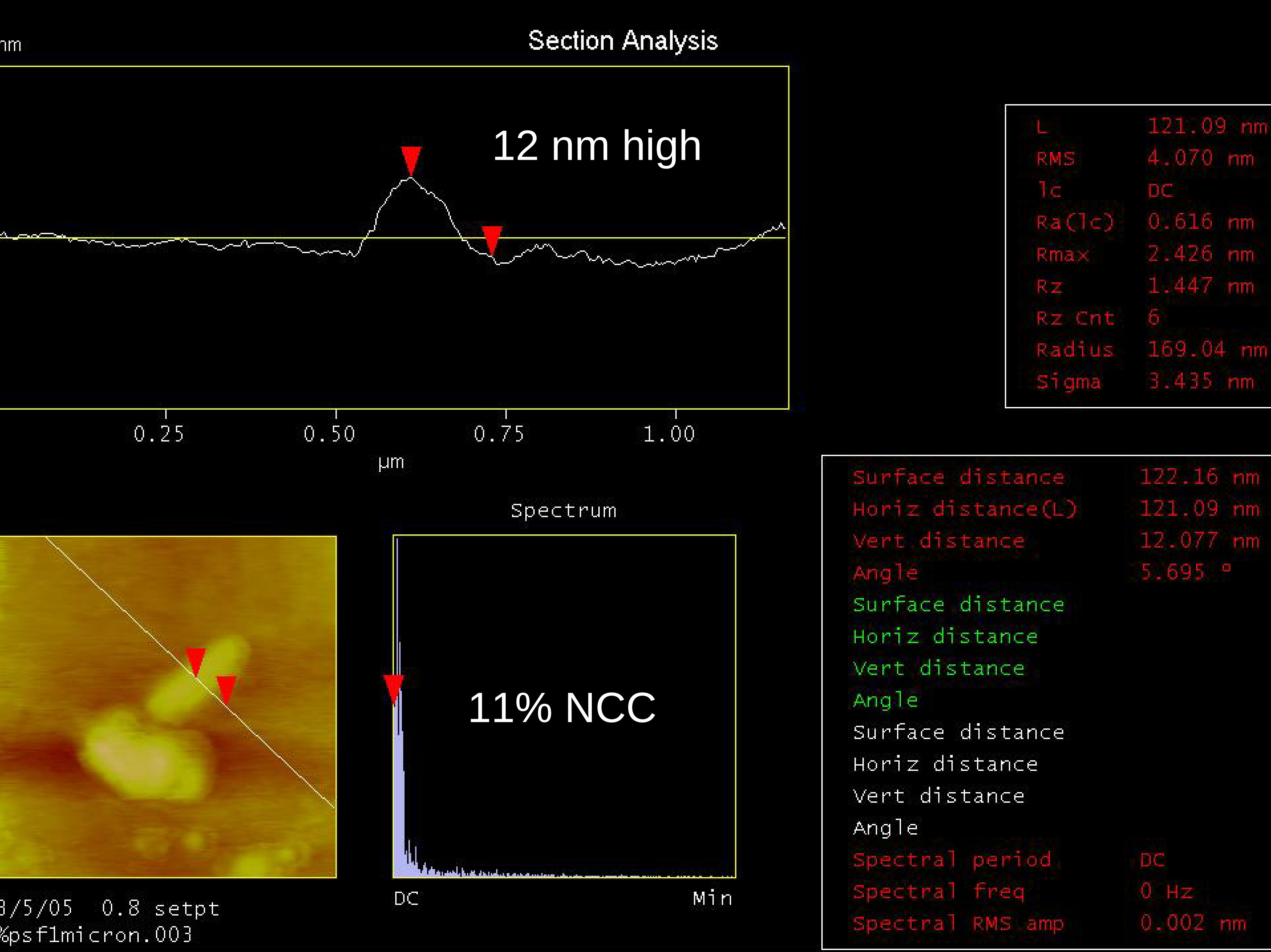


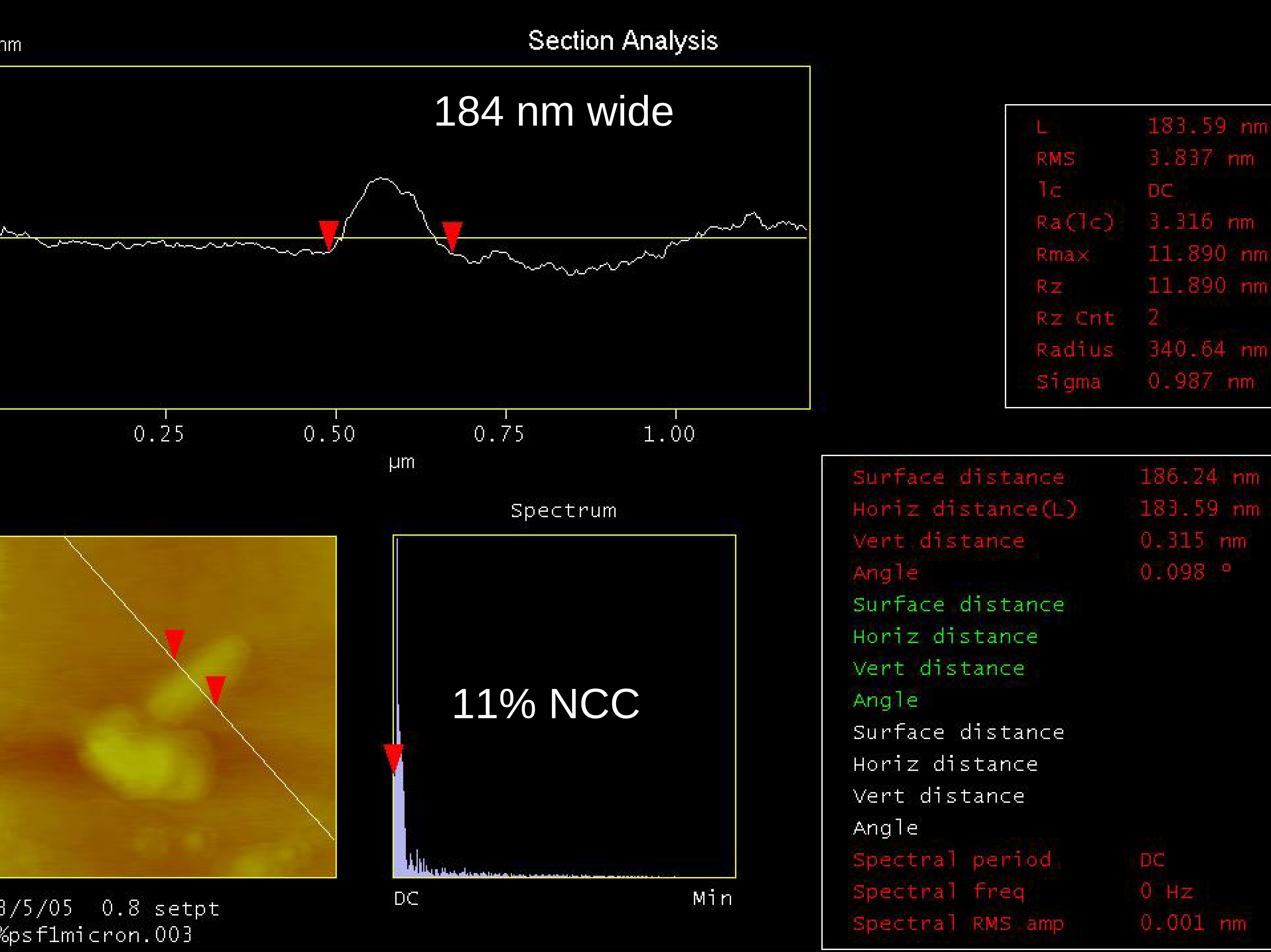
psf3micron.001
/05

11% NCC

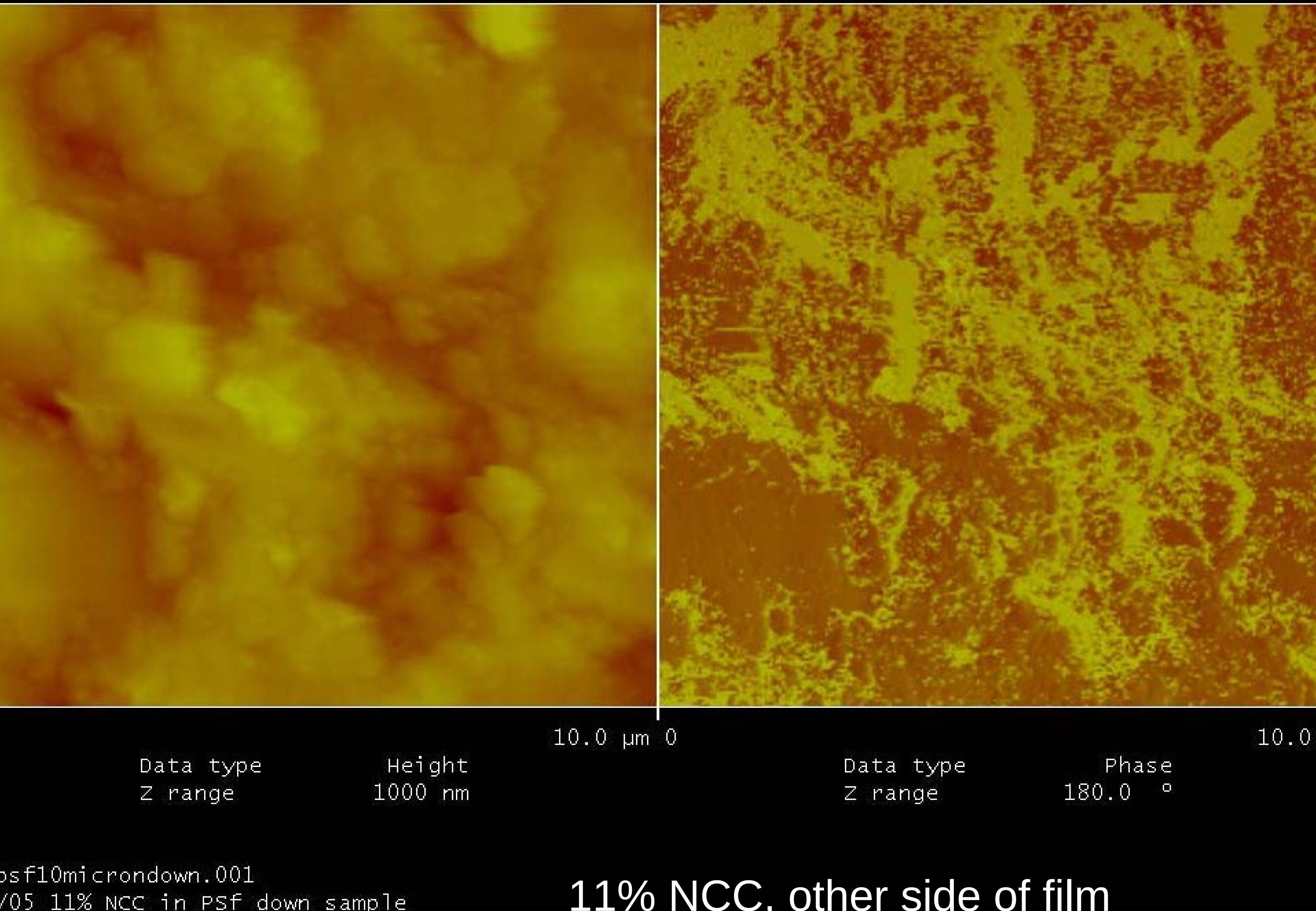

1.00 µm 0
1.00
Data type Height
Z range 50.00 nm
Data type Phase
Z range 180.0 °
osf1micron.003
/05 0.8 setpt

11% NCC


Section Analysis
nm
12 nm high
0.25
0.50
0.75
1.00
µm
L 121.09 nm
RMS 4.070 nm
lc DC
Ra(lc) 0.616 nm
Rmax 2.426 nm
Rz 1.447 nm
Rz Cnt 6
Radius 169.04 nm
Sigma 3.435 nm
Spectrum
11% NCC
DC
Min
Surface distance 122.16 nm
Horiz distance(L) 121.09 nm
Vert distance 12.077 nm
Angle 5.695 °
Surface distance
Horiz distance
Vert distance
Angle
Surface distance
Horiz distance
Vert distance
Angle
Spectral period DC
Spectral freq 0 Hz
Spectral RMS amp 0.002 nm
3/5/05 0.8 setpt
%psf1micron.003



Section Analysis
nm
184 nm wide
0.25
0.50
0.75
1.00
µm
L 183.59 nm
RMS 3.837 nm
lc DC
Ra(lc) 3.316 nm
Rmax 11.890 nm
Rz 11.890 nm
Rz Cnt 2
Radius 340.64 nm
Sigma 0.987 nm
Spectrum
11% NCC
DC
Min
3/5/05 0.8 setpt
%psf1micron.003
Surface distance 186.24 nm
Horiz distance(L) 183.59 nm
Vert distance 0.315 nm
Angle 0.098 °
Surface distance
Horiz distance
Vert distance
Angle
Surface distance
Horiz distance
Vert distance
Angle
Spectral period DC
Spectral freq 0 Hz
Spectral RMS amp 0.001 nm

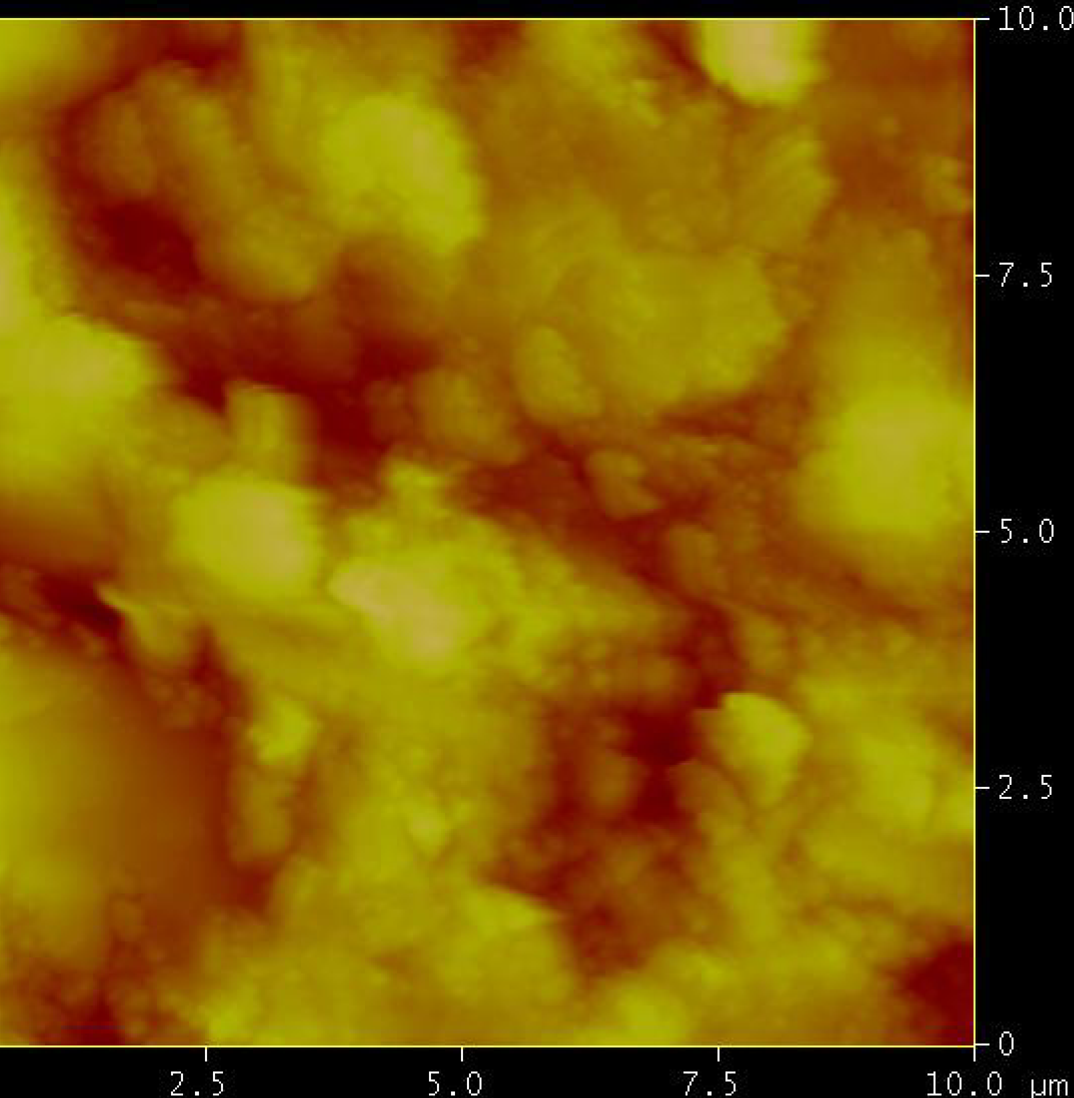


11% NCC, other side of film

Roughness Analysis

R_{ms} = 96 nm

(other side = 13 nm)



8/9/05 11% NCC in PSf down sample
%psf10microndown.001

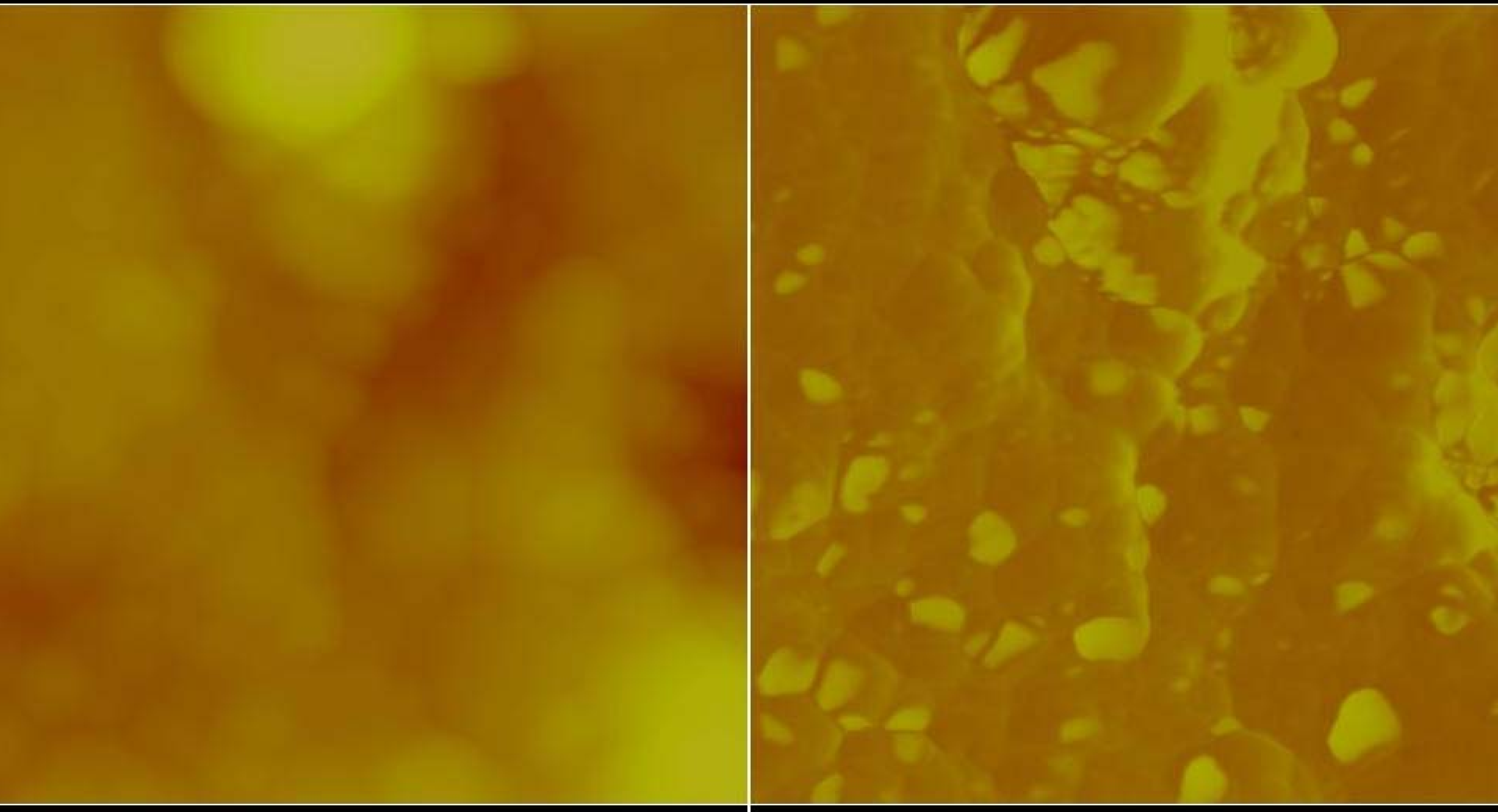
Image Statistics

Img. Z range	705.35 nm
Img. Mean	-0.000001 nm
Img. Raw mean	12.462 nm
Img. Rms (Rq)	96.118 nm
Img. Ra	77.393 nm
Img. Rmax	705.35 nm
Img. Srf. area	108.23 µm²
Img. Prj. Srf. area	100.00 µm²
Img. Srf. area diff	8.230 %
Img. SAE	1.039

Box Statistics

Z range	
Mean	
Raw mean	
Rms (Rq)	
Mean roughness (Ra)	
Max height (Rmax)	

11% NCC



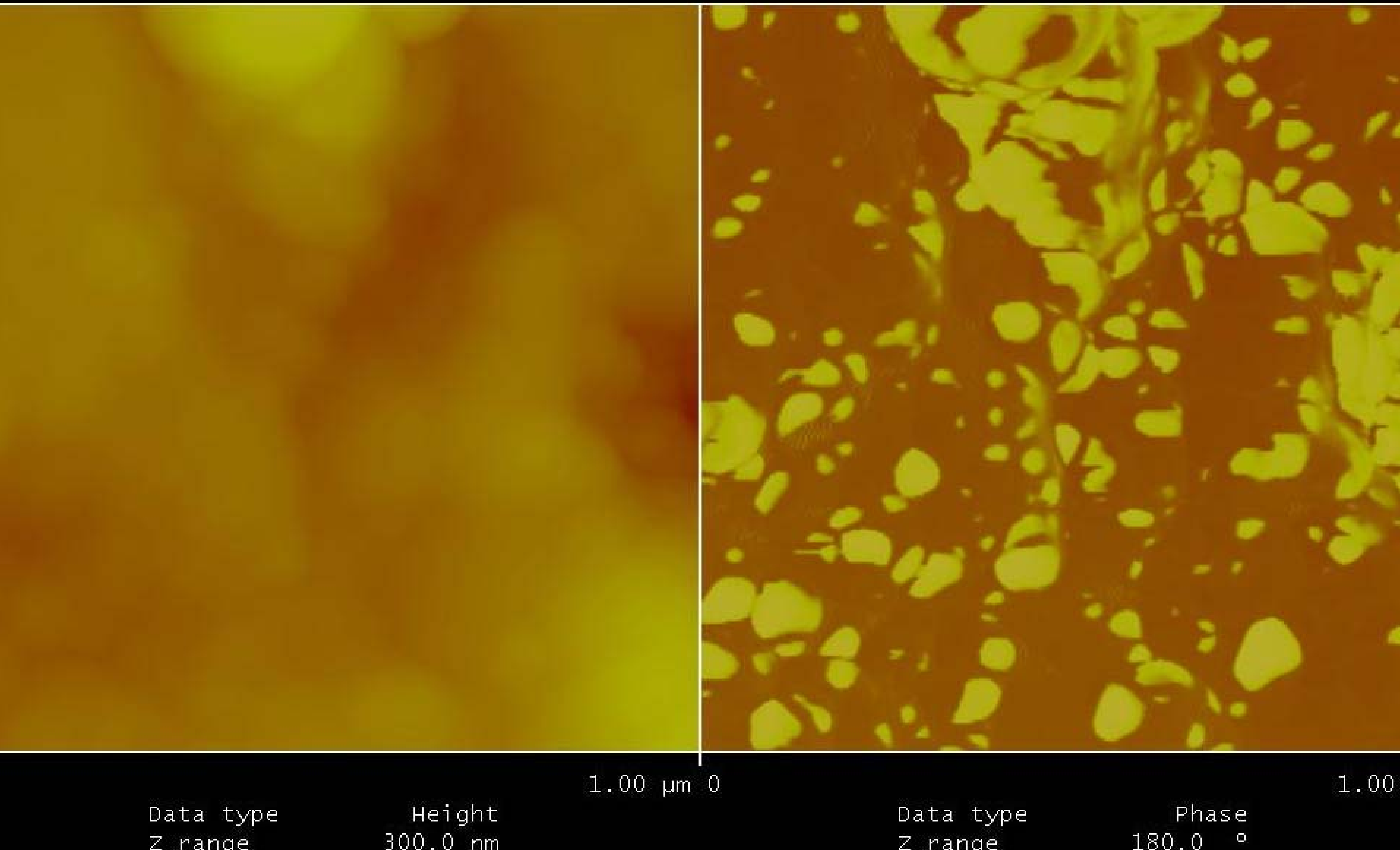
1.00 µm 0 1.00

Data type Height
Z range 300.0 nm

Data type Phase
Z range 180.0 °

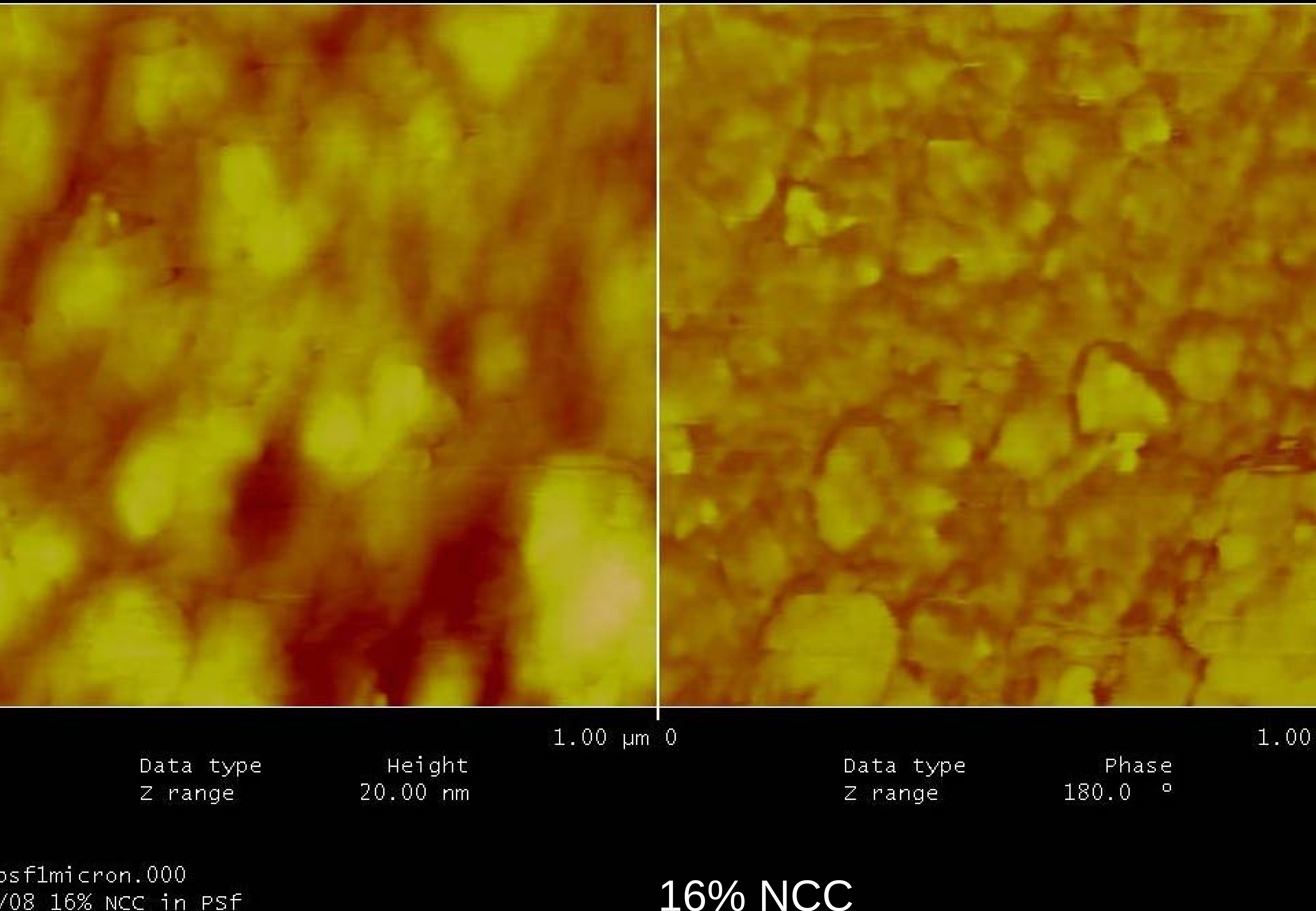
psf1microndownlt.002
/05 11% NCC in PSf down sample light tapping

11% NCC, light tapping



osf1microndownht.003
/05 11% NCC in PSf down sample hard tapping

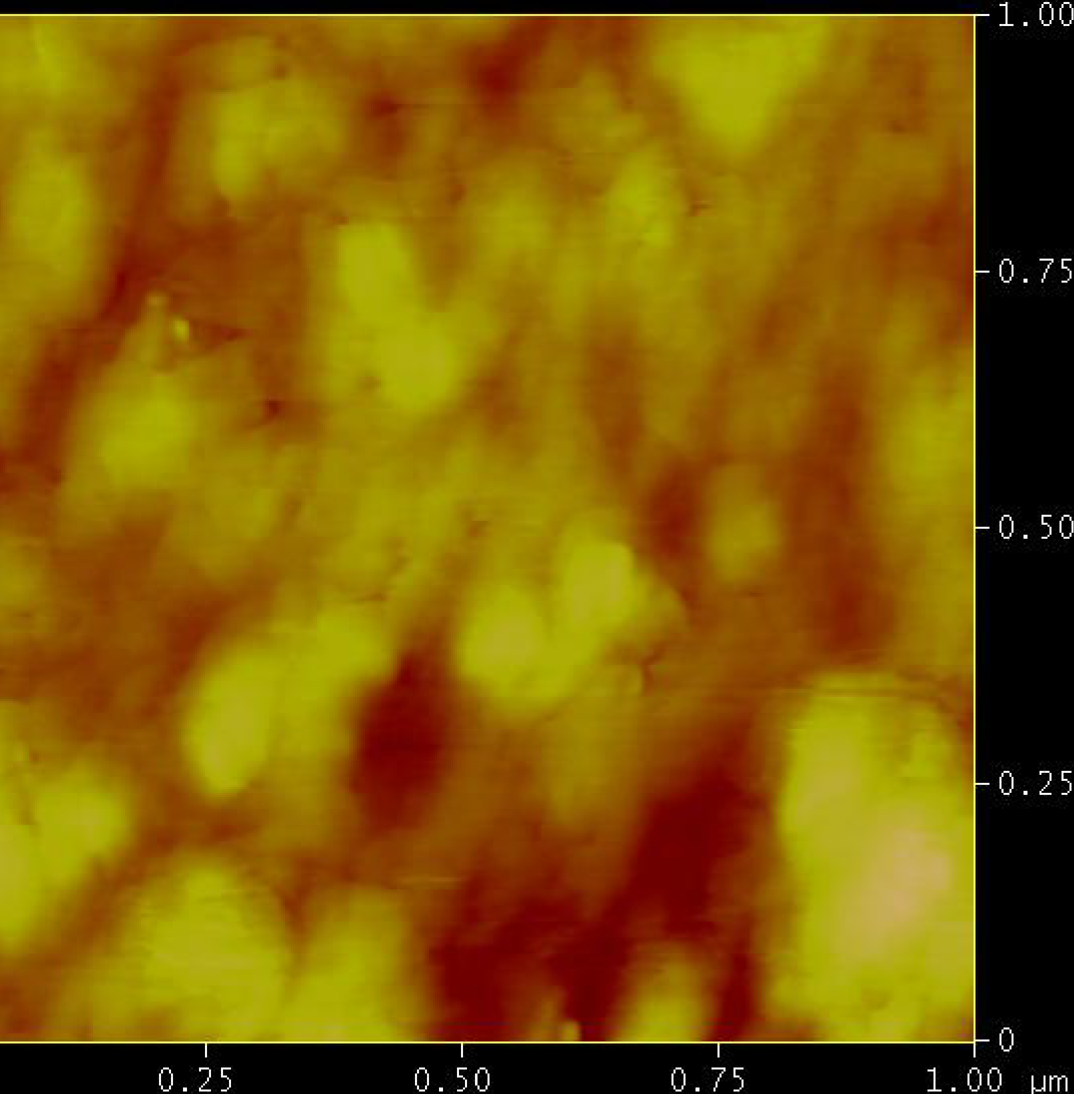
11% NCC, hard tapping


1.00 µm 0
Data type Height
Z range 20.00 nm
1.00
Data type Phase
Z range 180.0 °
psf1micron.000
/08 16% NCC in PSf

16% NCC

Roughness Analysis

R_{ms} = 1.9 nm



3/8/08 16% NCC in PSf
%psf1micron.000

Image Statistics

Img. Z range	12.912 nm
Img. Mean	0.000000 nm
Img. Raw mean	633.59 nm
Img. Rms (Rq)	1.853 nm
Img. Ra	1.460 nm
Img. Rmax	12.912 nm
Img. Srf. area	1.003 µm²
Img. Prj. Srf. area	999999 nm²
Img. Srf. area diff	0.305 %
Img. SAE	1.001

Box Statistics

Z range	
Mean	
Raw mean	
Rms (Rq)	
Mean roughness (Ra)	
Max height (Rmax)	

16% NCC

CONCLUSIONS

- NCC can be dispersed in PSf
  - Quality of the dispersion is less than perfect
- NCC effects the T_g of the PSf
- TGA indicates 2% NCC in PSf behaves differently, close association?
- NCC stiffens, but does not strengthen composite
  - Interphase needs improvement
- Hard tapping on AFM has promise as technique to visualize
- Loadings of 16% cause dry out, poor properties

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