Calorimetry and scanning thermal measurements

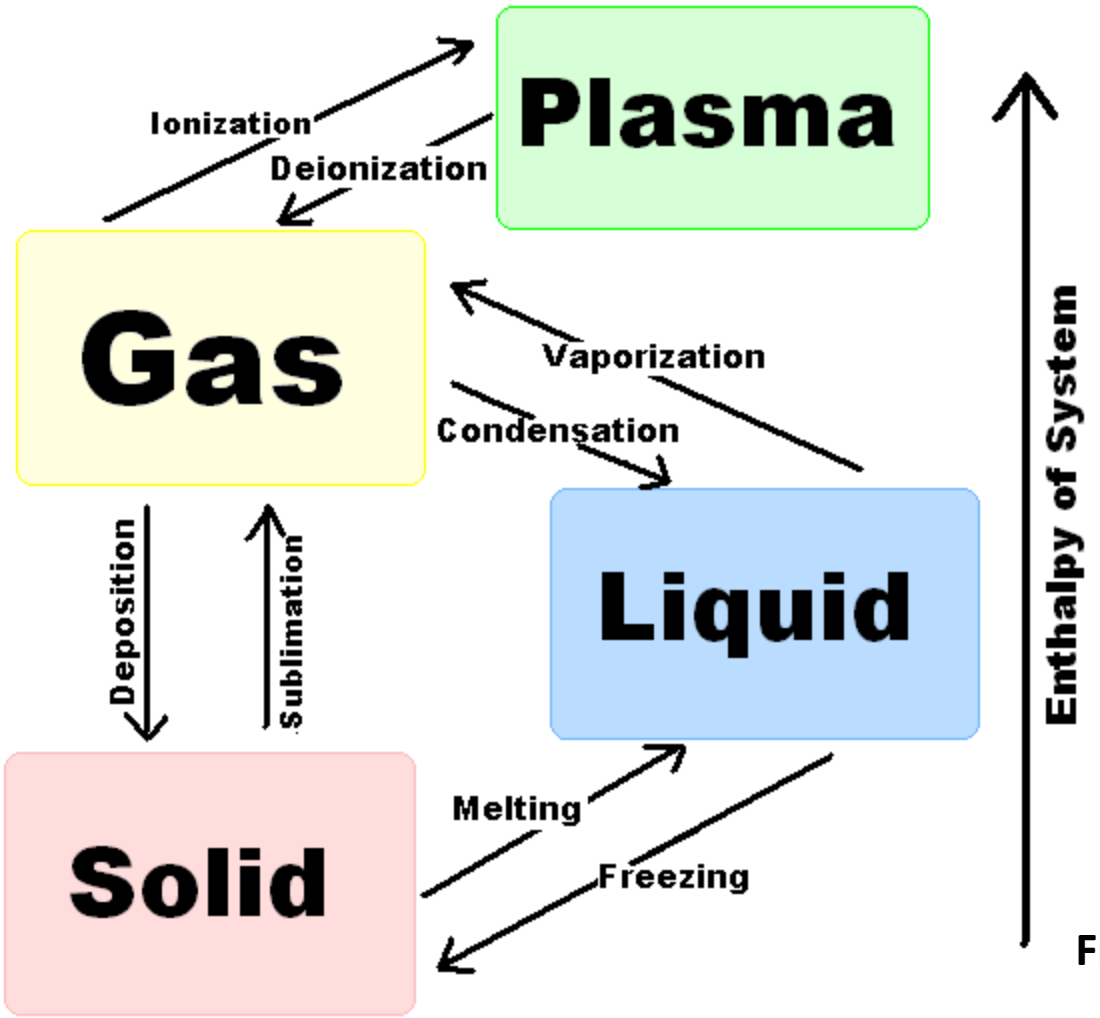
R W McCallum

Ames Laboratory

And

Materials Science and Engineering

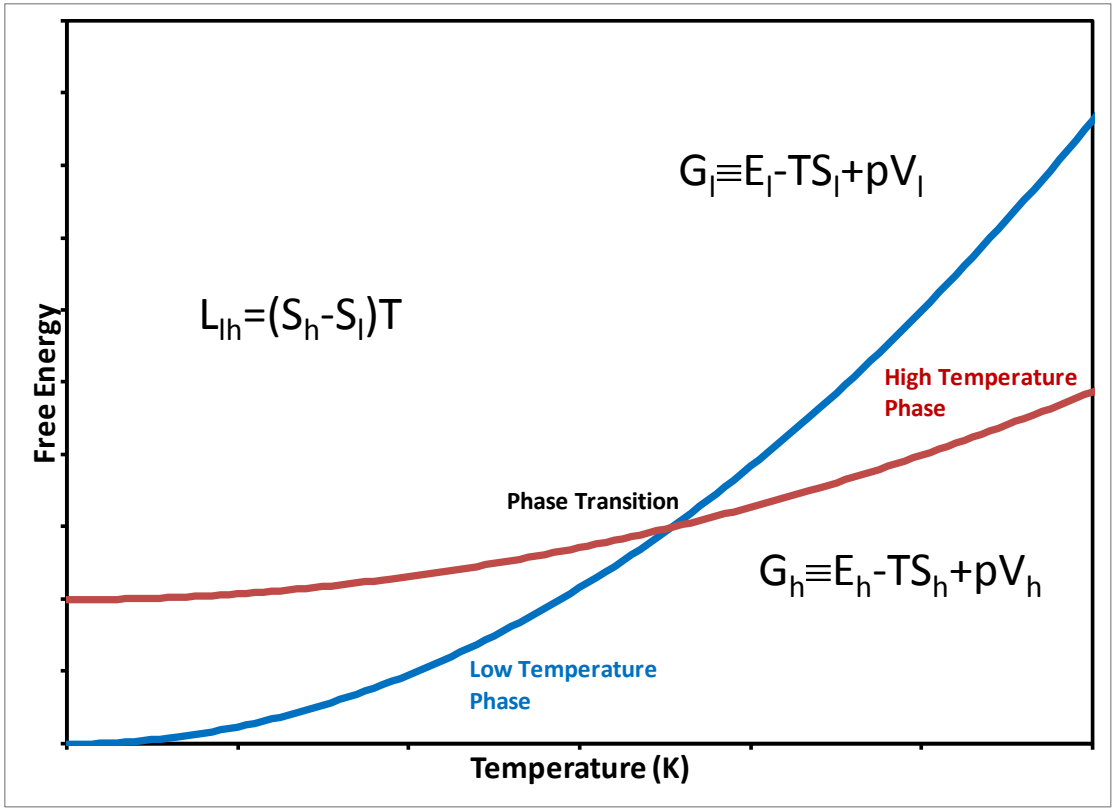
Phase transition



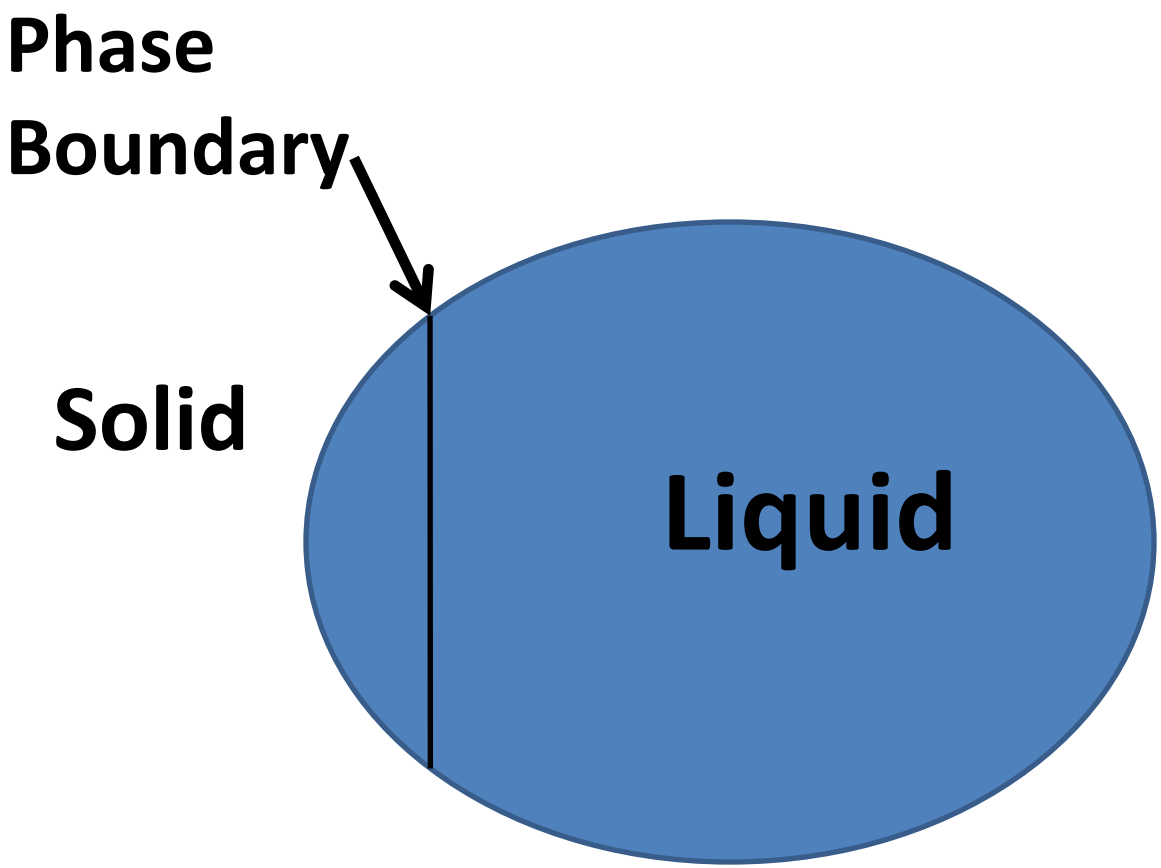
From Wikipedia,

Phase Transition

- At phase-transition point
  - the two phases have identical free energies
  - equally likely to exist.
- Below the phase-transition point
  - Low temperature phase is more stable state of the two.
- Above the phase-transition point
  - High temperature phase is more stable state of the two.



Nucleation



If the energy of the phase boundary is greater then the reduction of energy due to solid formation, the solid will not form.

Phase Transition

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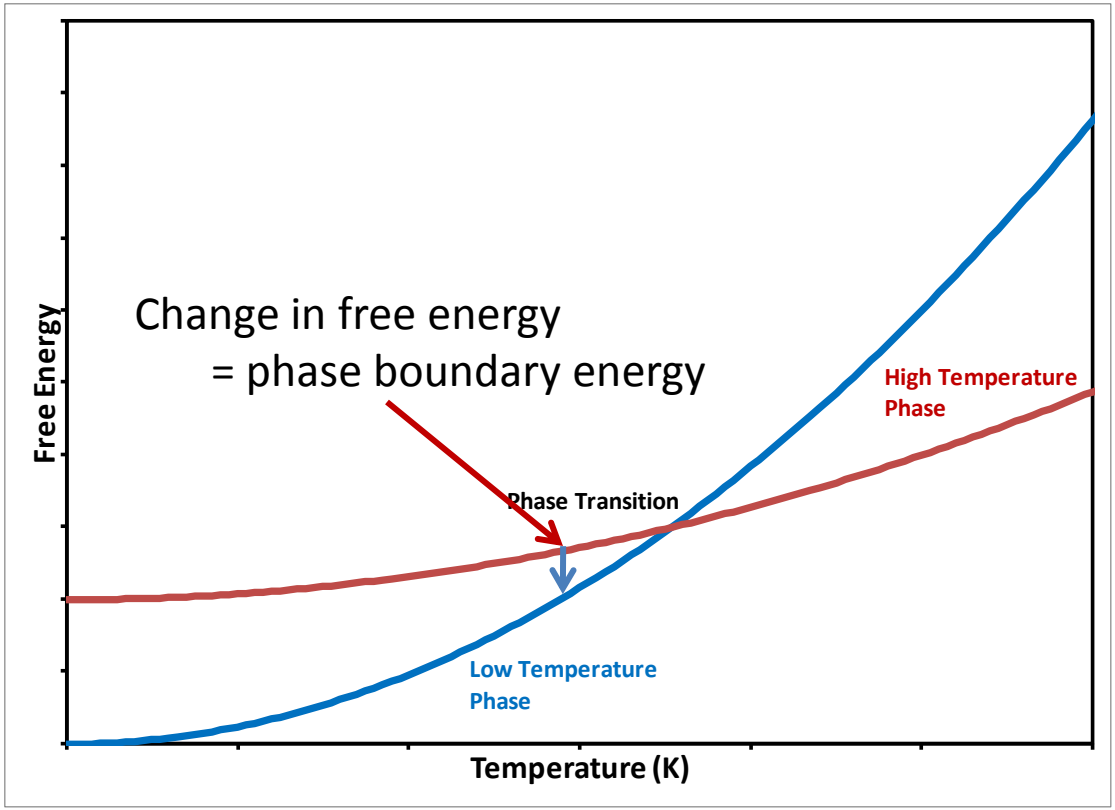
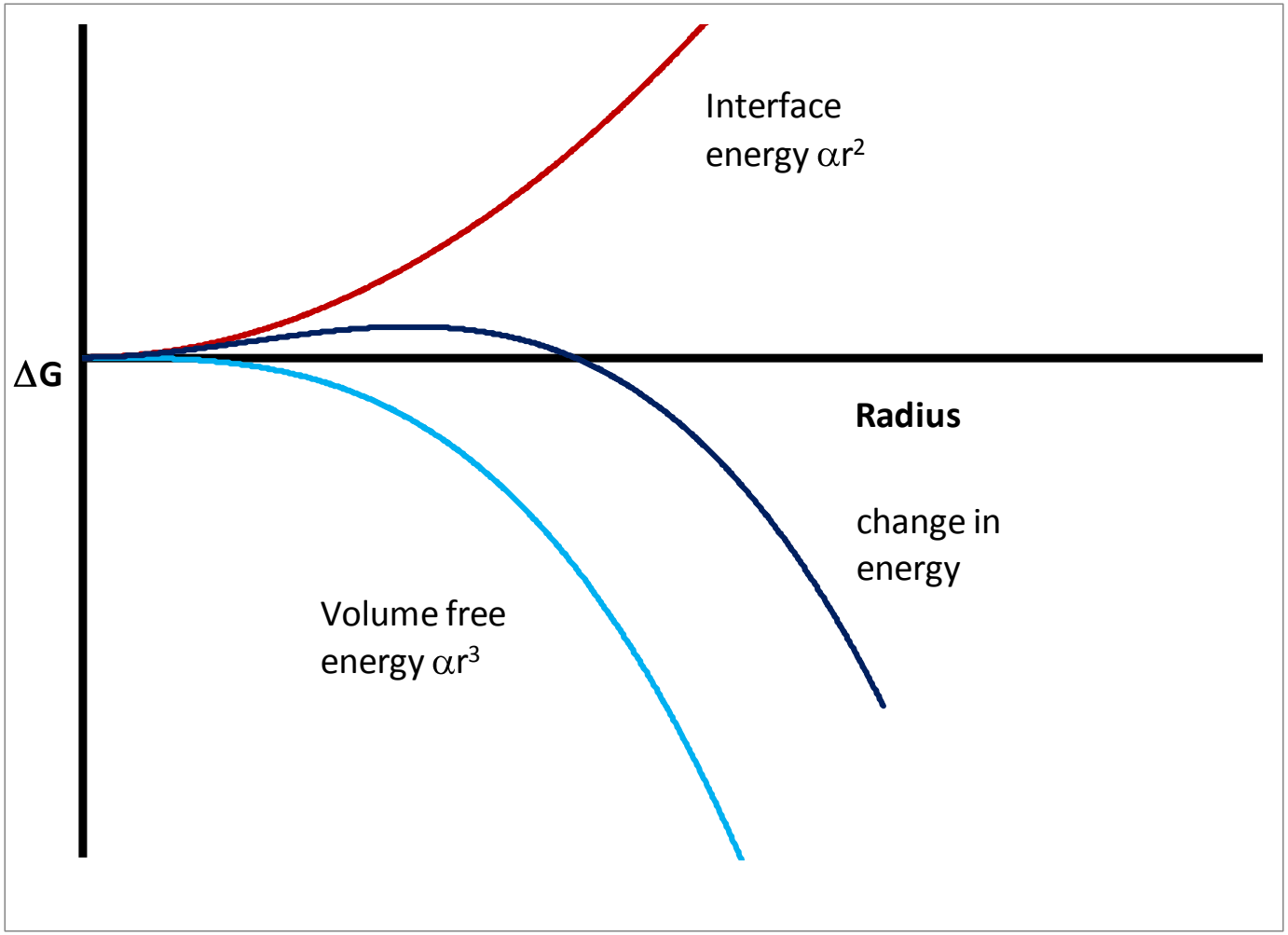


Homogeneous Nucleation

- Nucleation which occurs in the interior of a uniform substance
  - requires an interface at the boundaries of a new phase
    - Pure water freezes at −42°C rather than at its freezing temperature of 0°C if no crystal nuclei, such as dust particles, are present to form an ice nucleus.

Homogeneous nucleation barrier



Heterogeneous Nucleation

- New phase forms at preferential sites
  - Pre-existing phase boundaries
  - impurities like dust (also phase boundaries)
  - requires less energy than homogeneous nucleation.
    - the effective surface energy is lower
      - free energy barrier lower

Phase Transition

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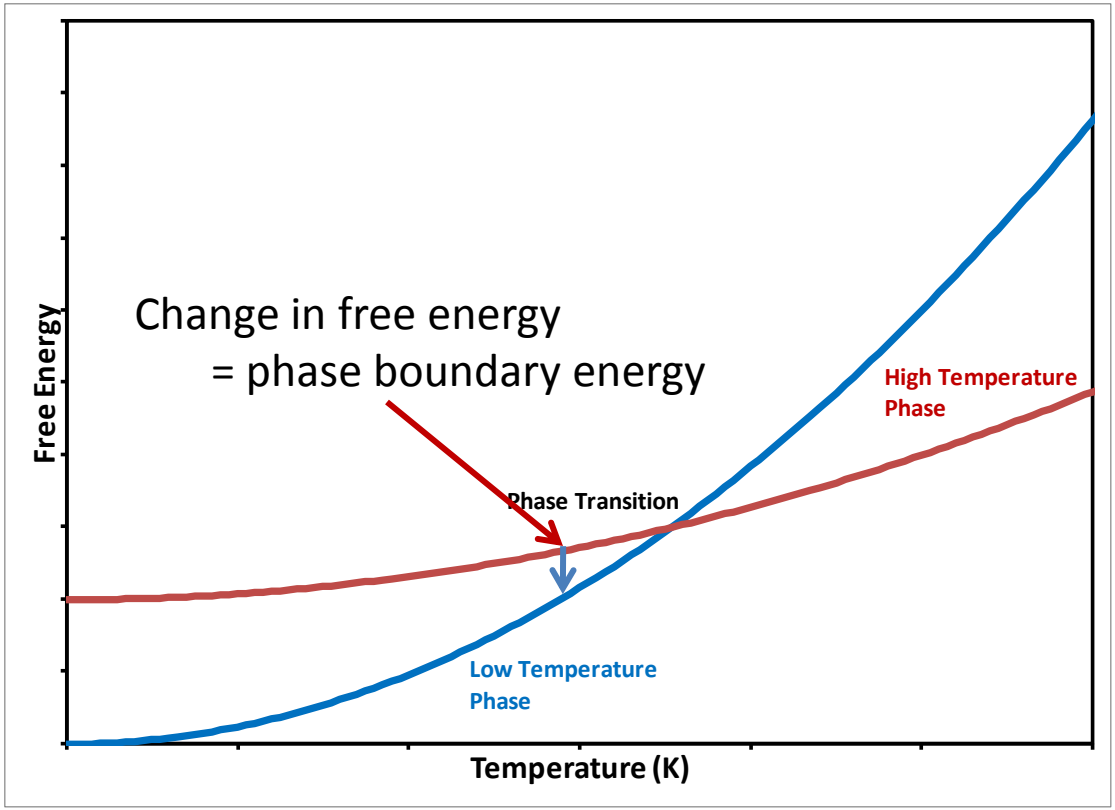
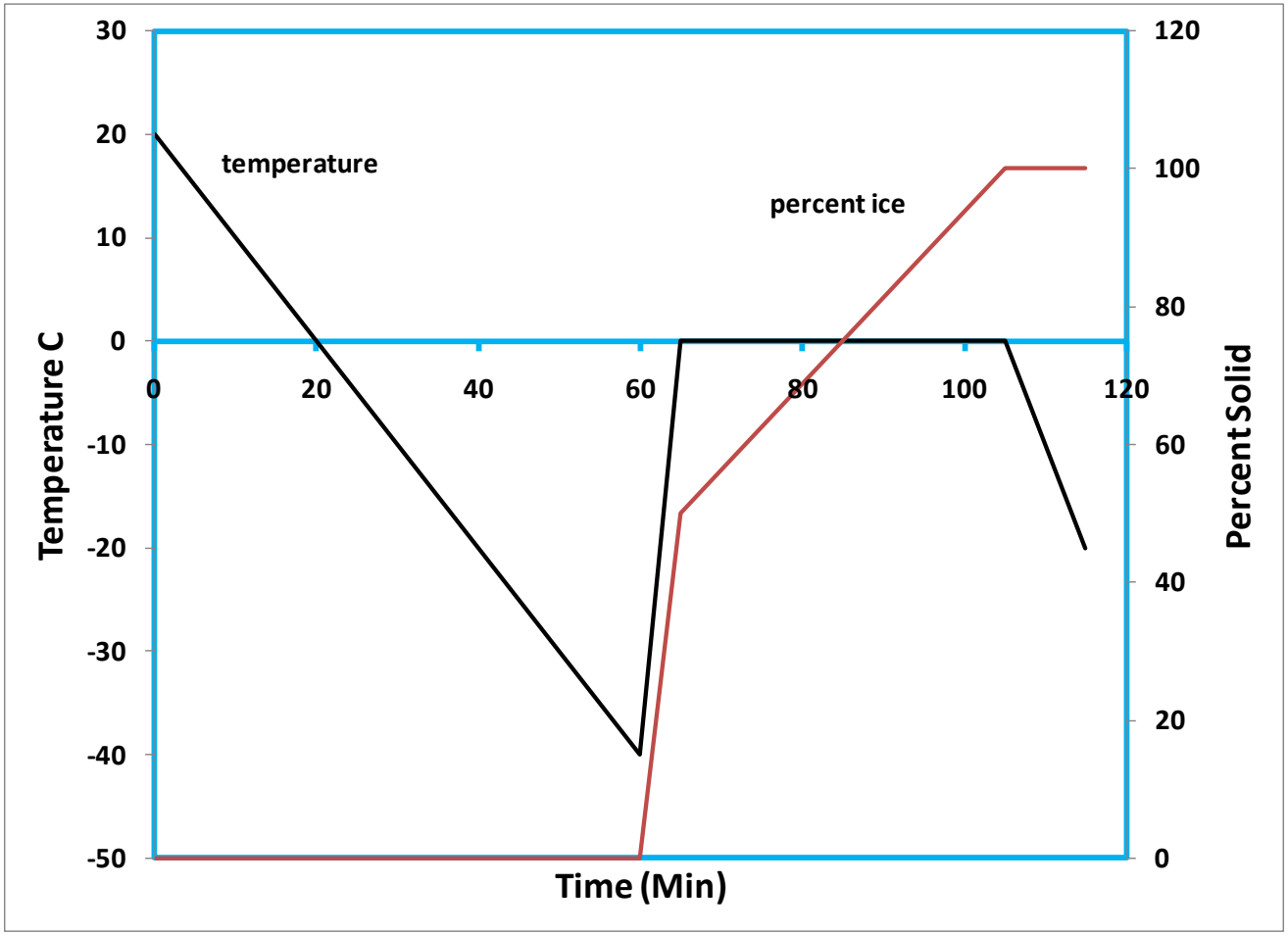
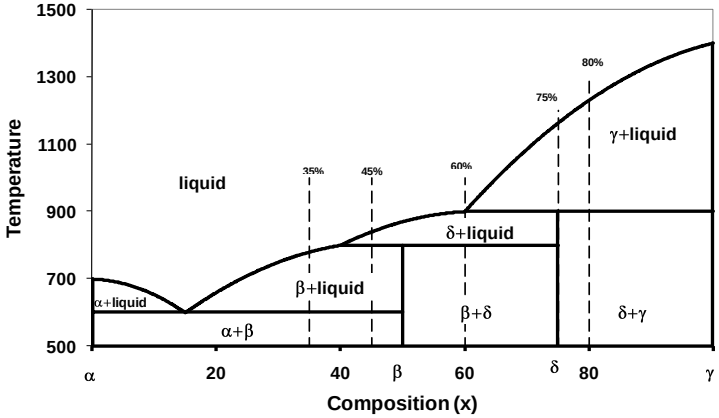
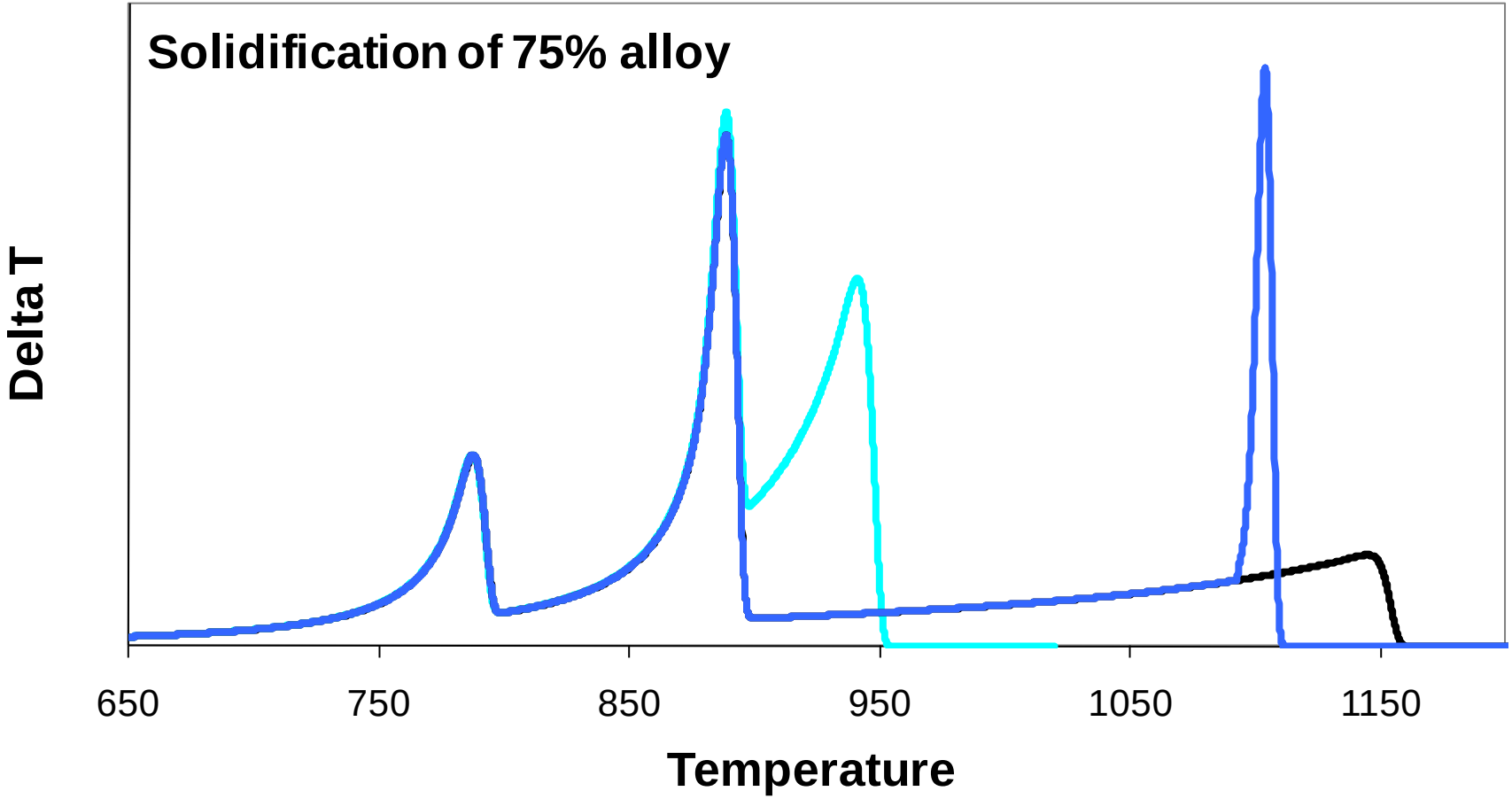


Freezing of supercooled water

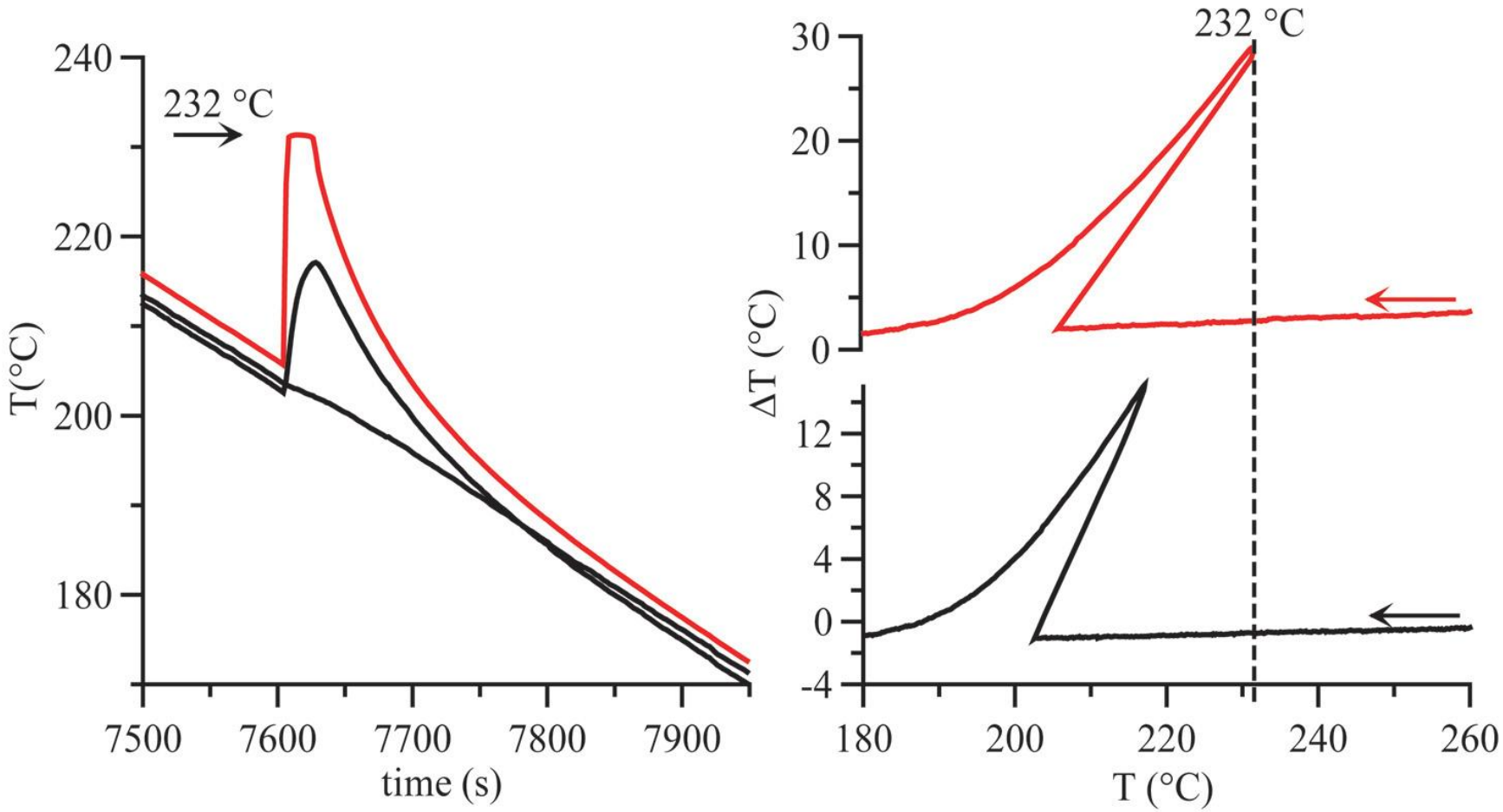




undercooling

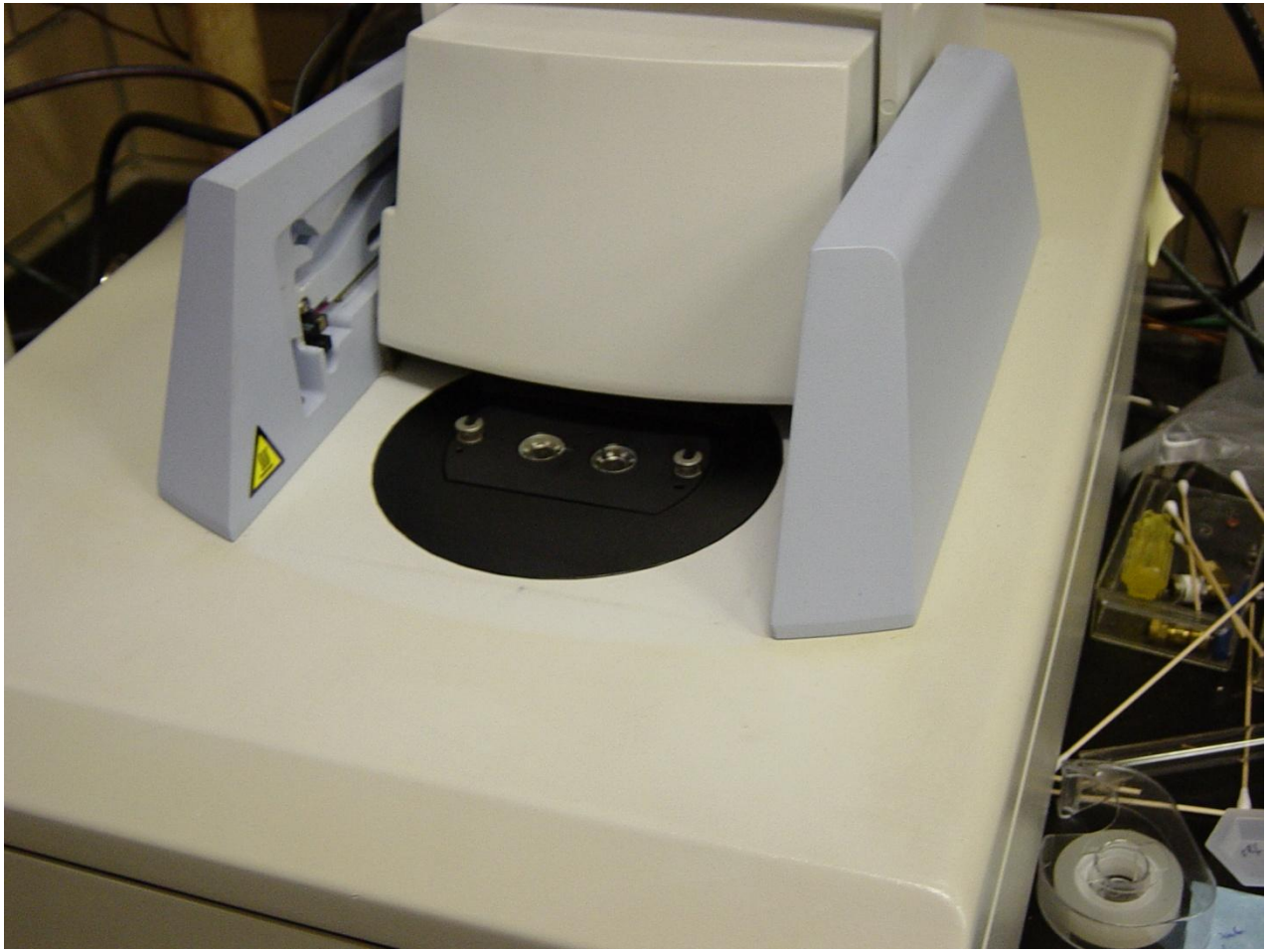


Recalescence

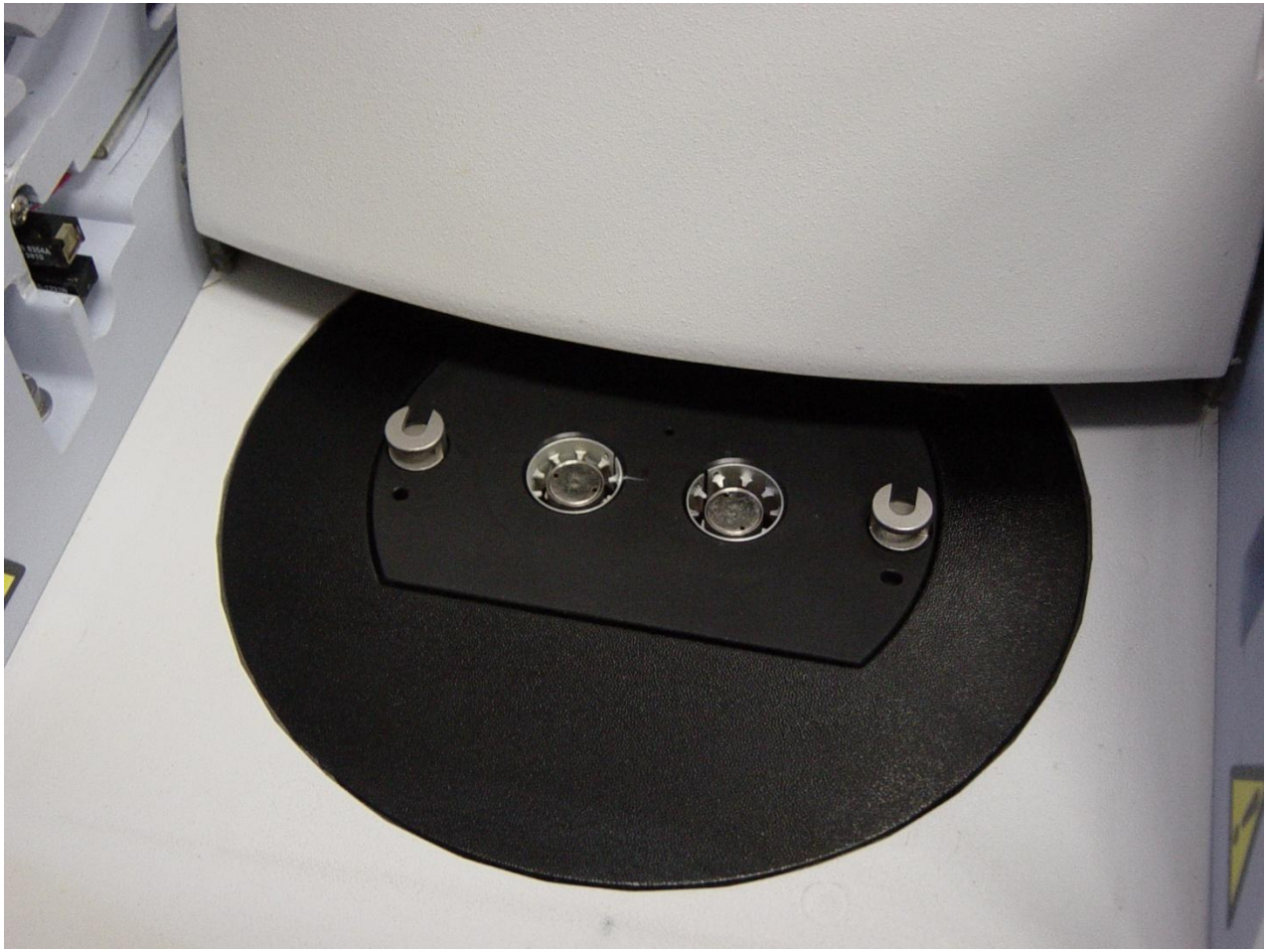


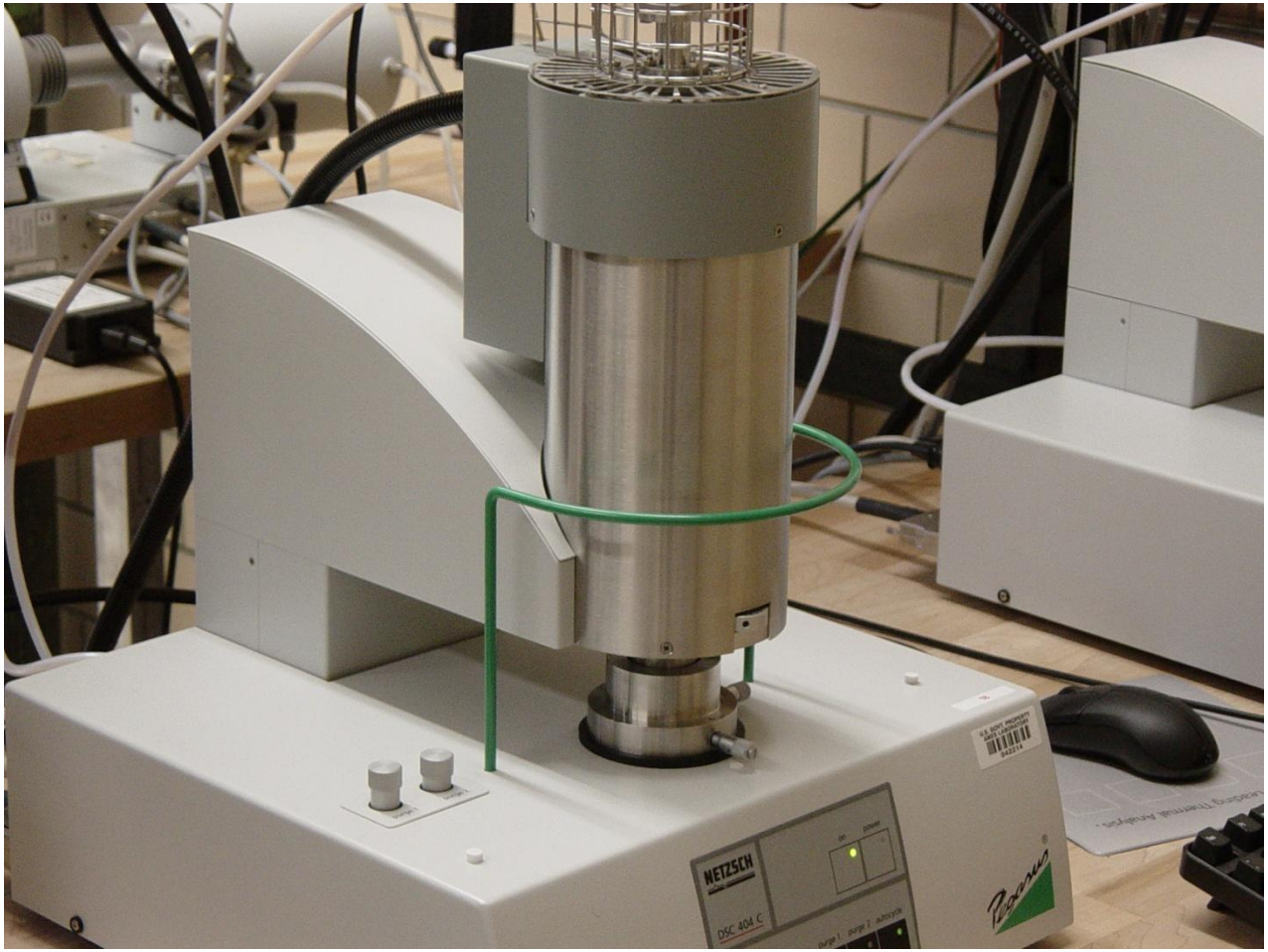
Power Compensating DSC

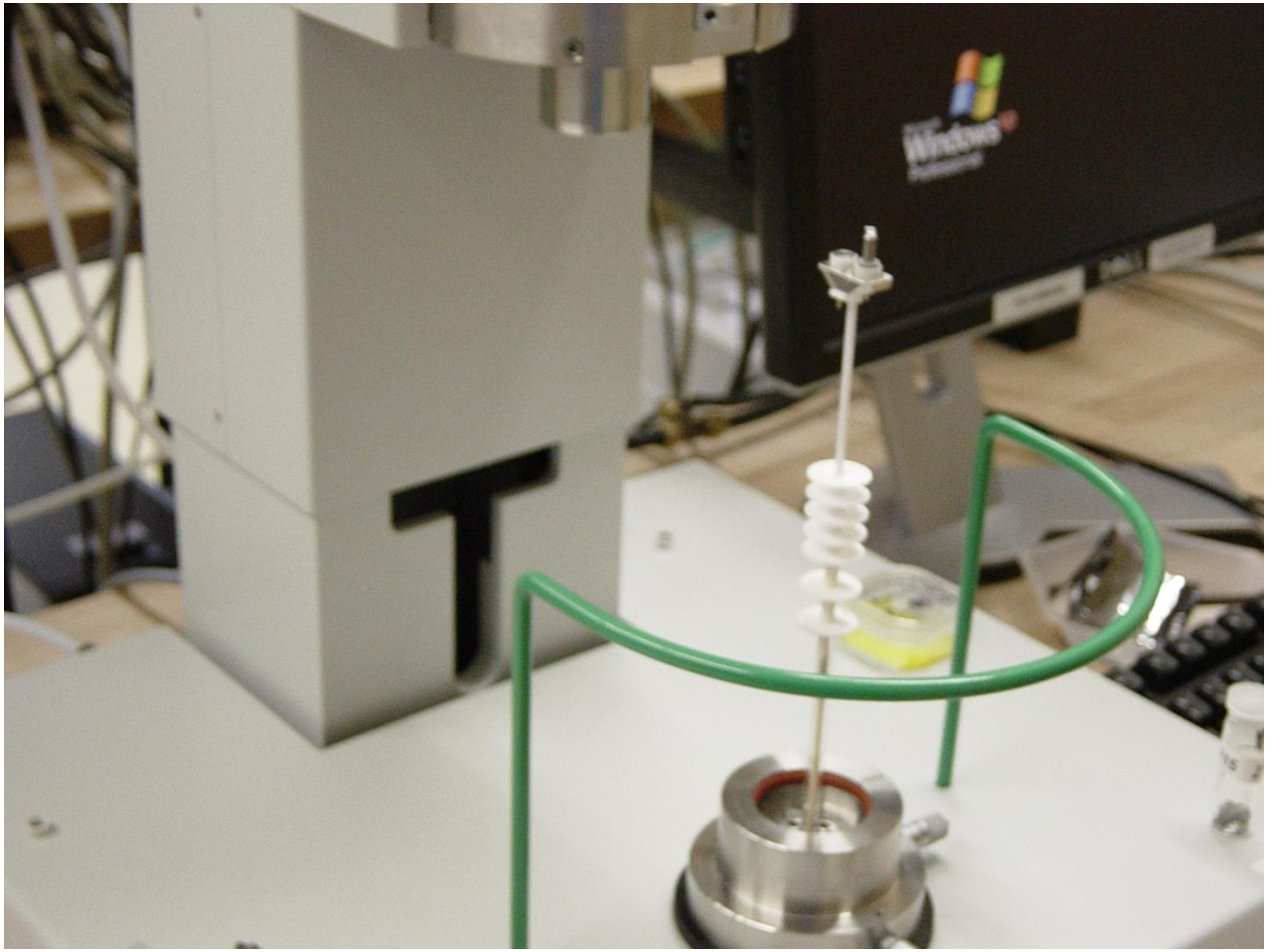
Power Compensating DSC

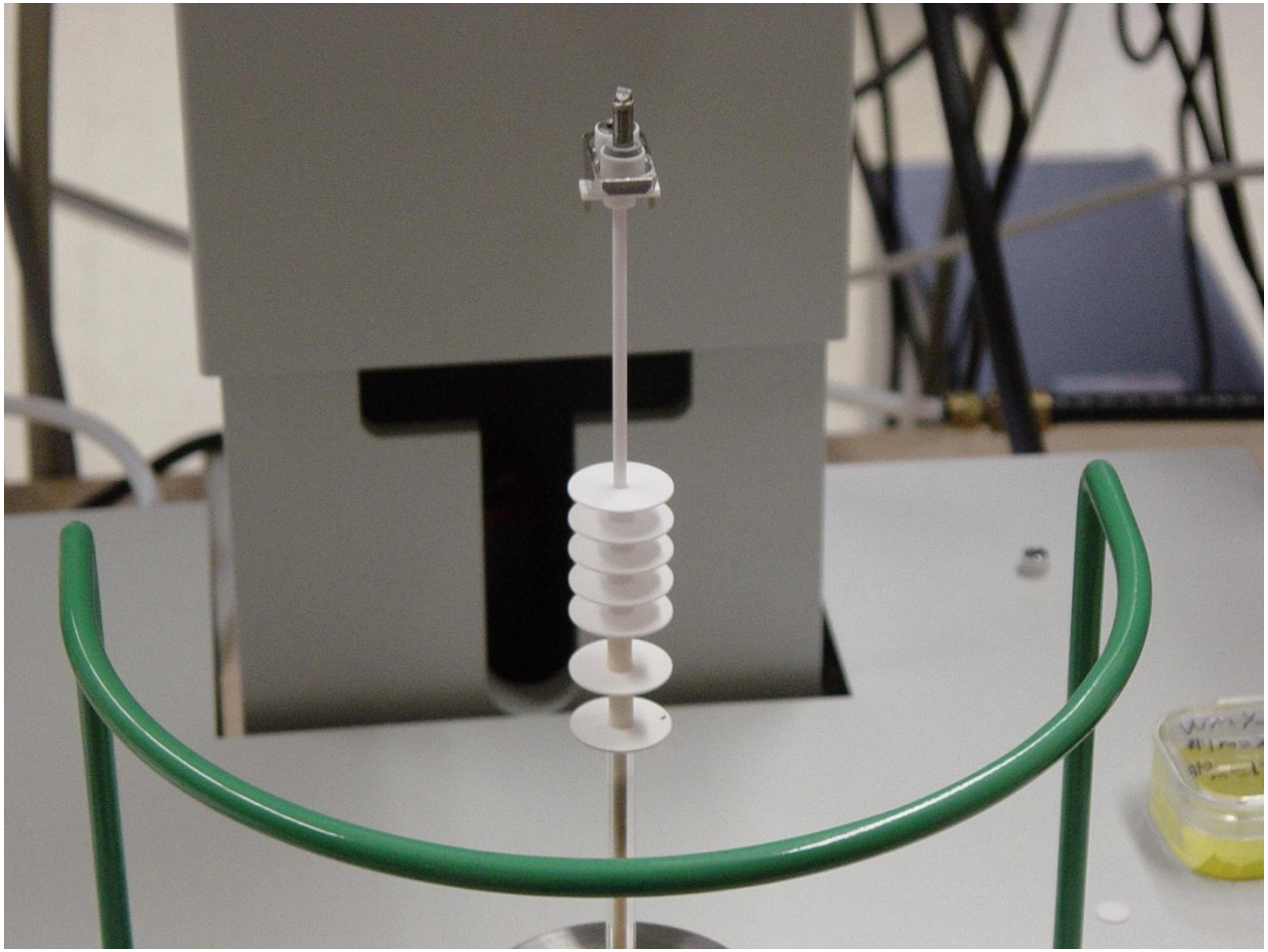


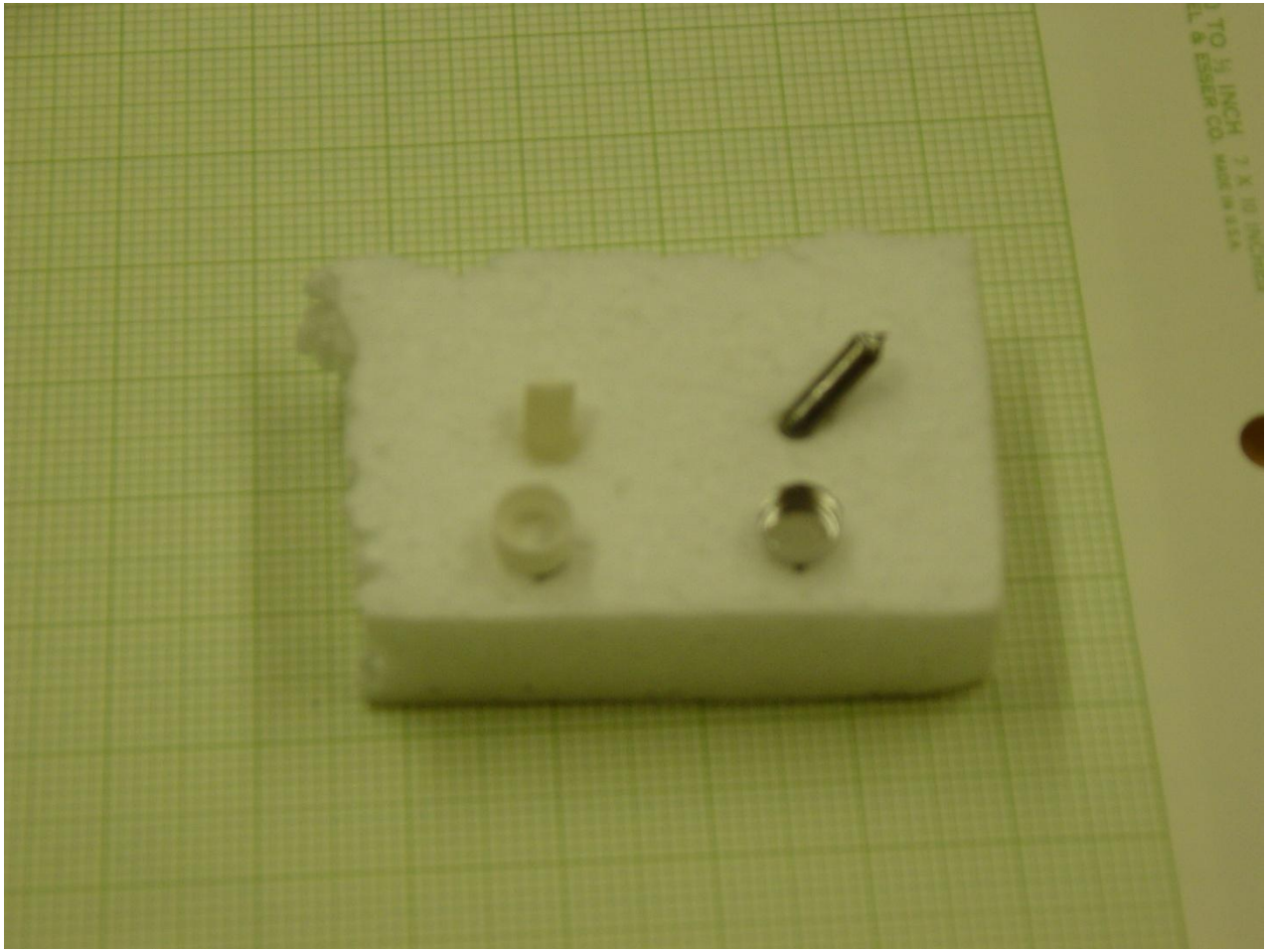
Power Compensating DSC

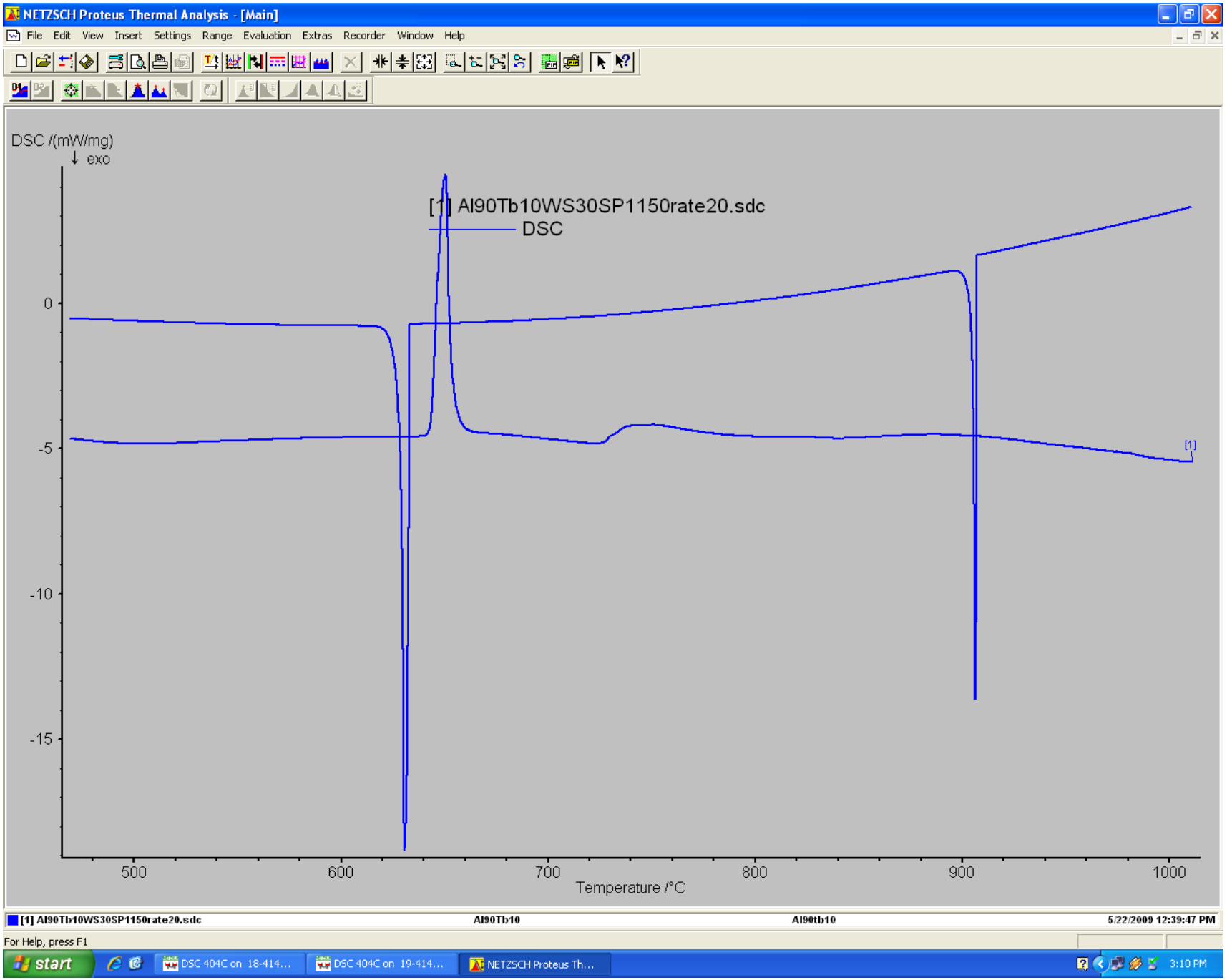


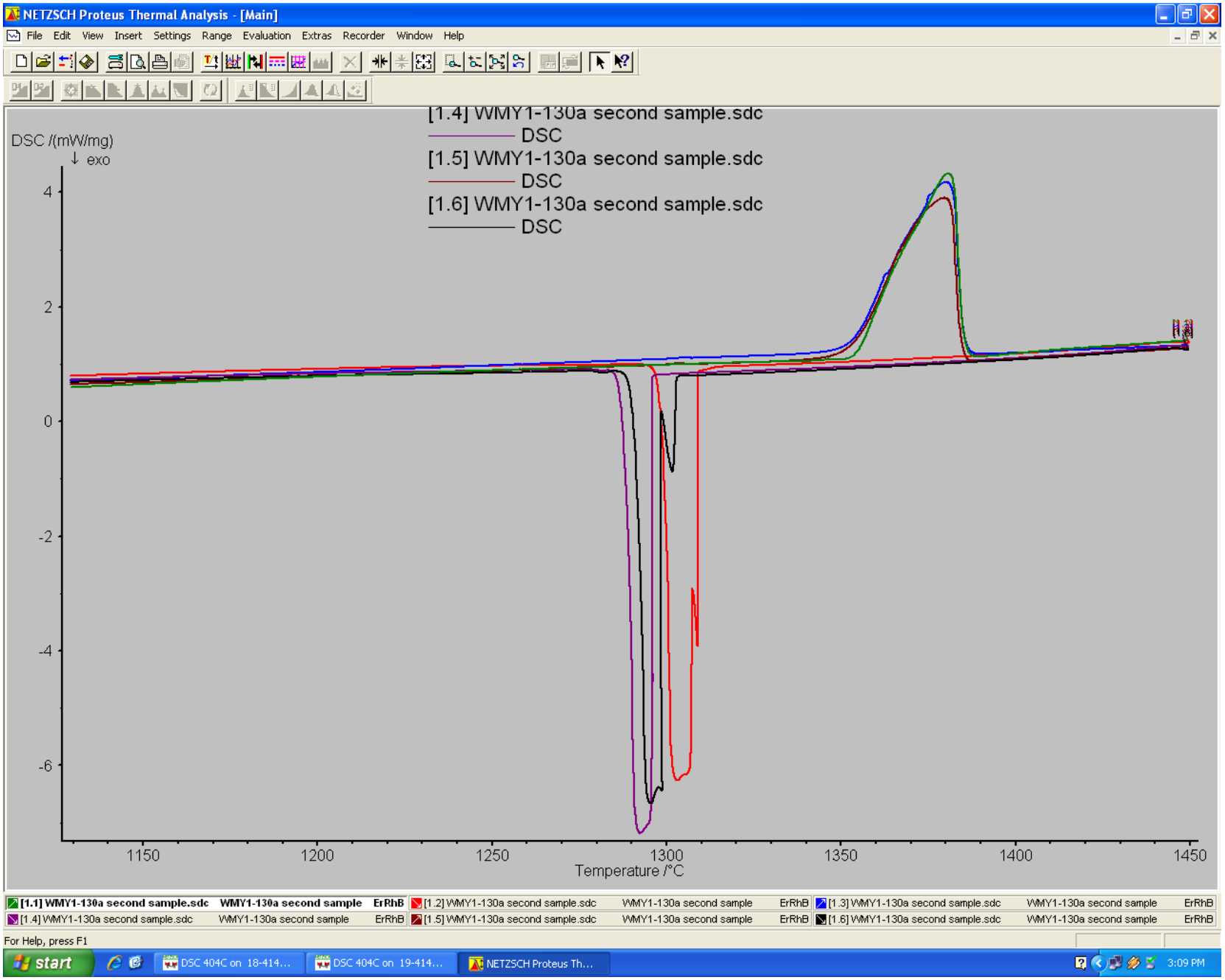

NETZSCH
DSC 404 C
ON
POWER
Porous

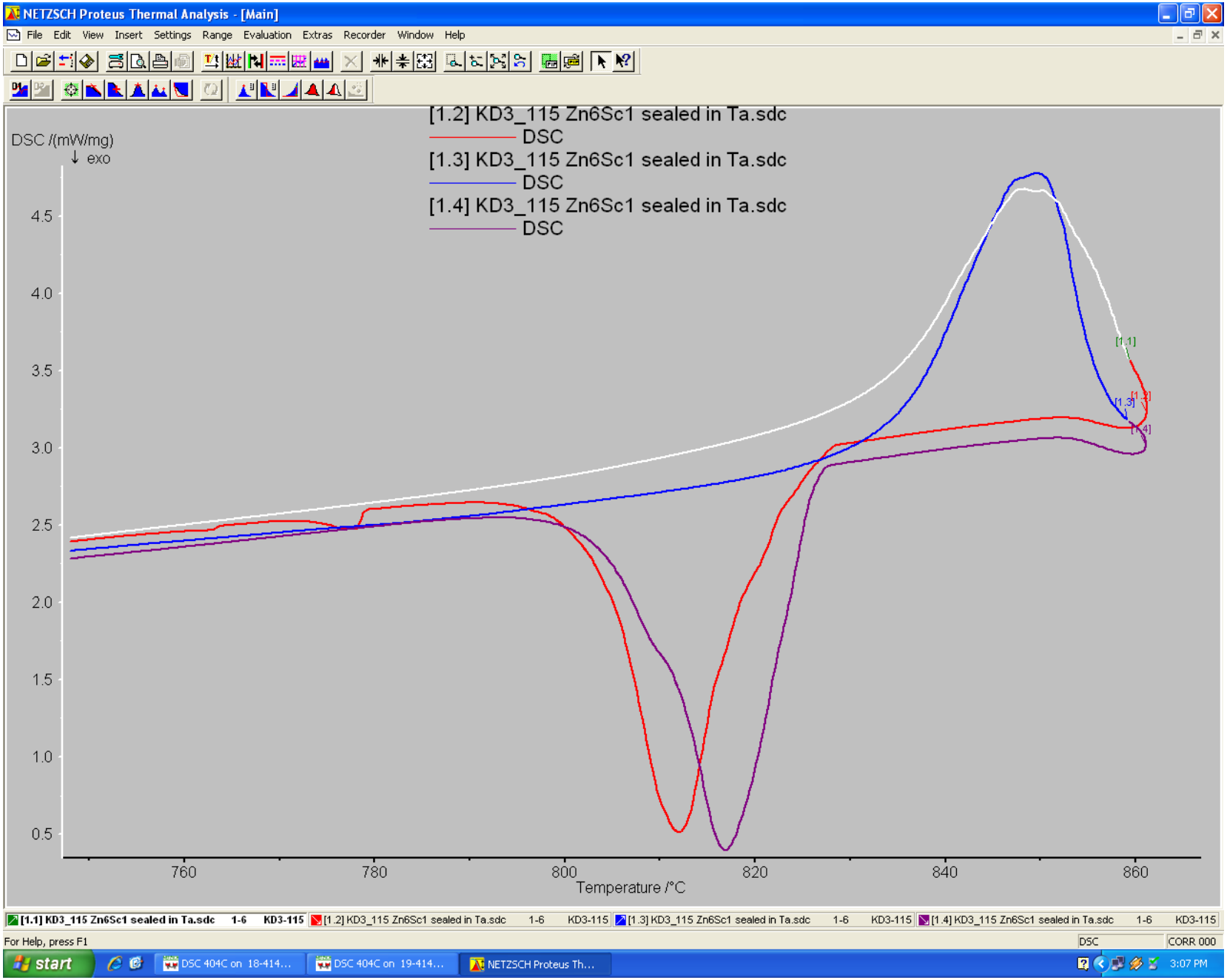

Windows



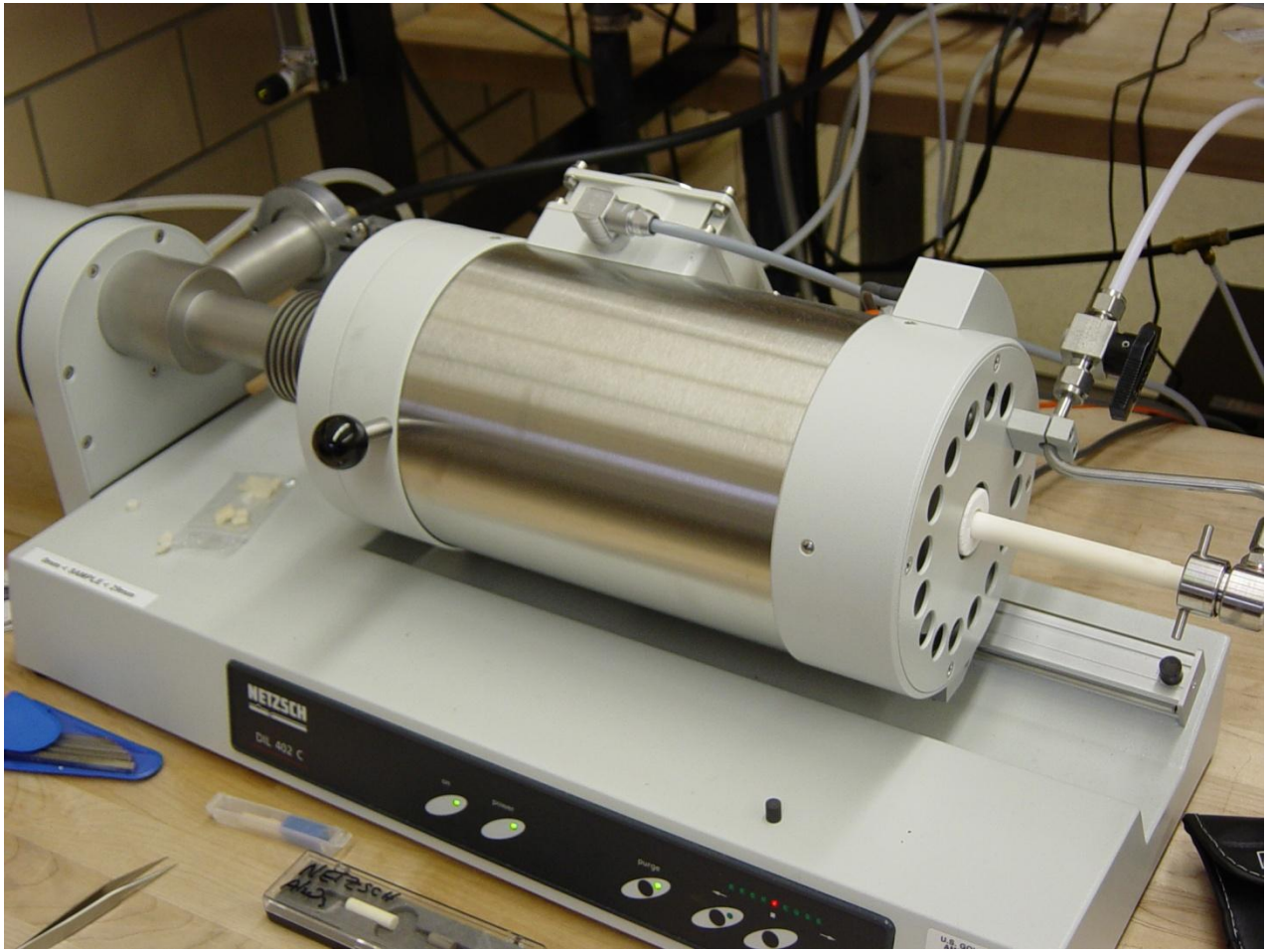



NETZSCH Proteus Thermal Analysis - [Main]
File Edit View Insert Settings Range Evaluation Extras Recorder Window Help
DSC /(mW/mg)
↓ exo
[1] Al90Tb10WS30SP1150rate20.sdc
DSC
0
-5
-10
-15
500
600
700
800
900
1000
Temperature /°C
[1]
[1] Al90Tb10WS30SP1150rate20.sdc
Al90Tb10
Al90tb10
5/22/2009 12:39:47 PM
For Help, press F1
start
DSC 404C on 18-414...
DSC 404C on 19-414...
NETZSCH Proteus Th...
3:10 PM


NETZSCH Proteus Thermal Analysis - [Main]
File Edit View Insert Settings Range Evaluation Extras Recorder Window Help
[1.4] WMY1-130a second sample.sdc
DSC
[1.5] WMY1-130a second sample.sdc
DSC
[1.6] WMY1-130a second sample.sdc
DSC
DSC /(mW/mg)
↓ exo
4
2
0
-2
-4
-6
1150
1200
1250
1300
1350
1400
1450
Temperature /°C
[1.1] WMY1-130a second sample.sdc WMY1-130a second sample ErRhB
[1.2] WMY1-130a second sample.sdc WMY1-130a second sample ErRhB
[1.3] WMY1-130a second sample.sdc WMY1-130a second sample ErRhB
[1.4] WMY1-130a second sample.sdc WMY1-130a second sample ErRhB
[1.5] WMY1-130a second sample.sdc WMY1-130a second sample ErRhB
[1.6] WMY1-130a second sample.sdc WMY1-130a second sample ErRhB
For Help, press F1
start
DSC 404C on 18-414...
DSC 404C on 19-414...
NETZSCH Proteus Th...
3:09 PM

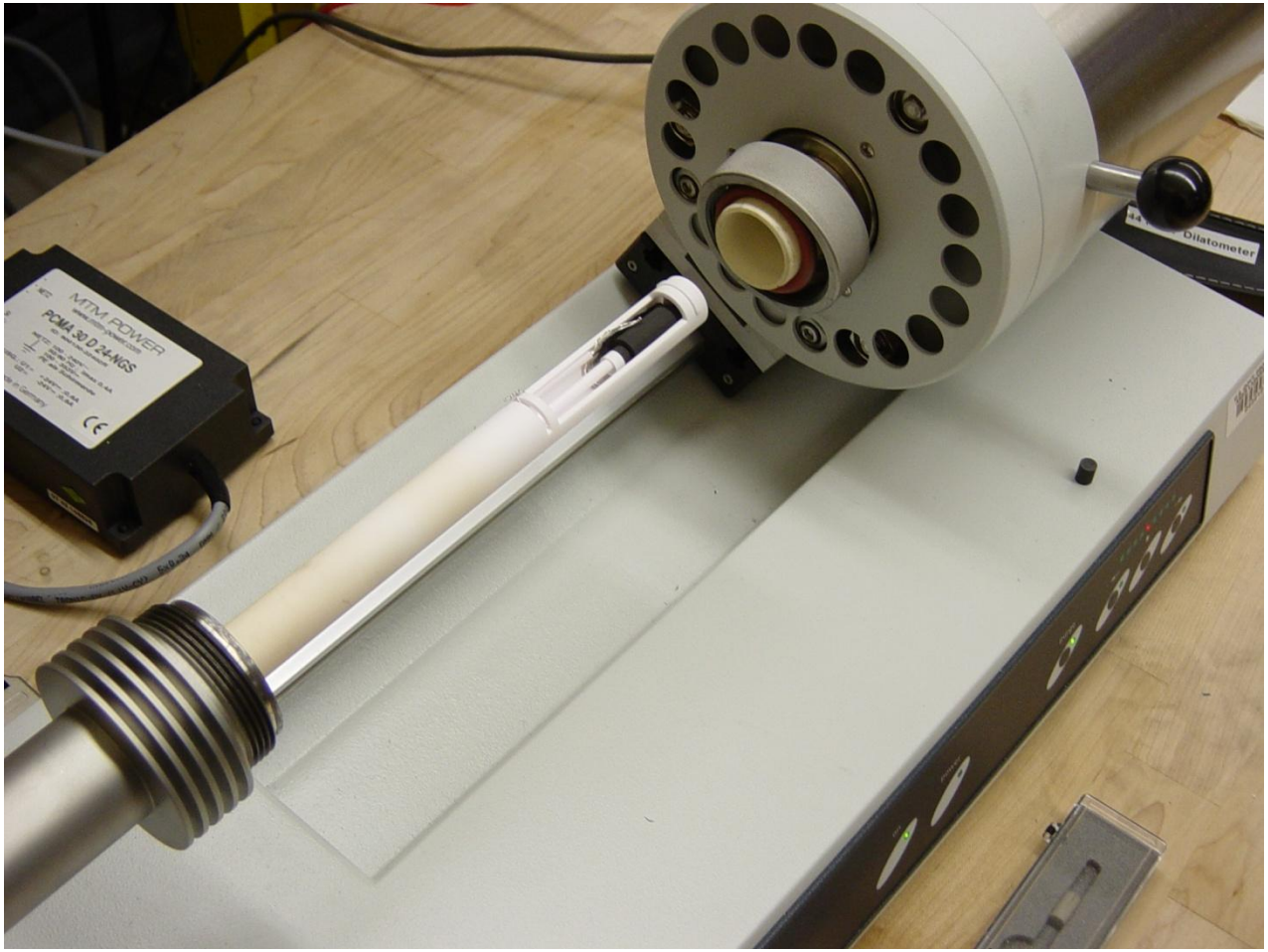

NETZSCH Proteus Thermal Analysis - [Main]
File Edit View Insert Settings Range Evaluation Extras Recorder Window Help
[1.2] KD3_115 Zn6Sc1 sealed in Ta.sdc
DSC
[1.3] KD3_115 Zn6Sc1 sealed in Ta.sdc
DSC
[1.4] KD3_115 Zn6Sc1 sealed in Ta.sdc
DSC
DSC /(mW/mg)
↓ exo
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
760
780
800
820
840
860
Temperature /°C
[1.1]
[1.2]
[1.3]
[1.4]
[1.1] KD3_115 Zn6Sc1 sealed in Ta.sdc 1-6 KD3-115
[1.2] KD3_115 Zn6Sc1 sealed in Ta.sdc 1-6 KD3-115
[1.3] KD3_115 Zn6Sc1 sealed in Ta.sdc 1-6 KD3-115
[1.4] KD3_115 Zn6Sc1 sealed in Ta.sdc 1-6 KD3-115
For Help, press F1
DSC
CORR 000
start
DSC 404C on 18-414...
DSC 404C on 19-414...
NETZSCH Proteus Th...
3:07 PM

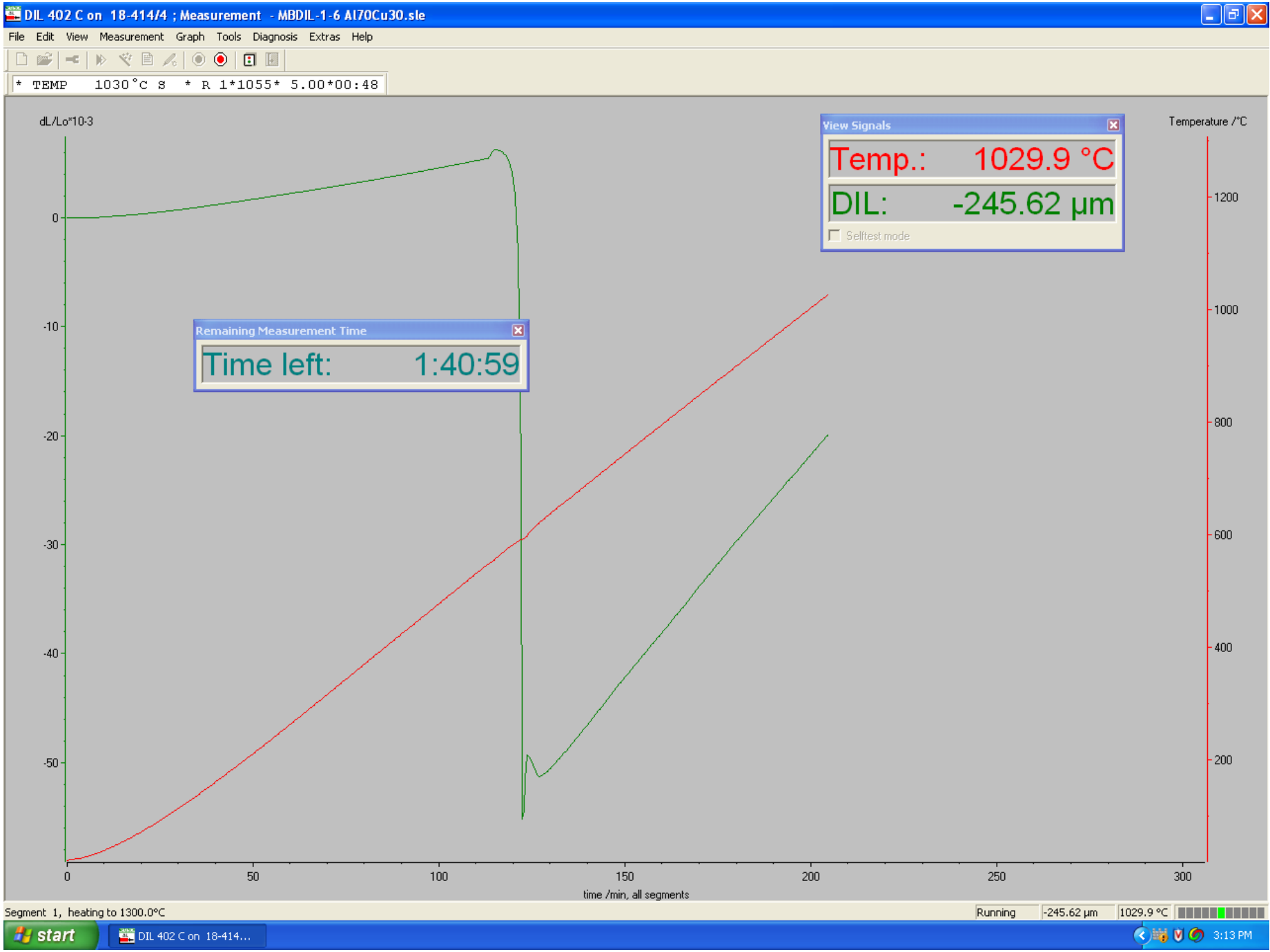
Dilatometer



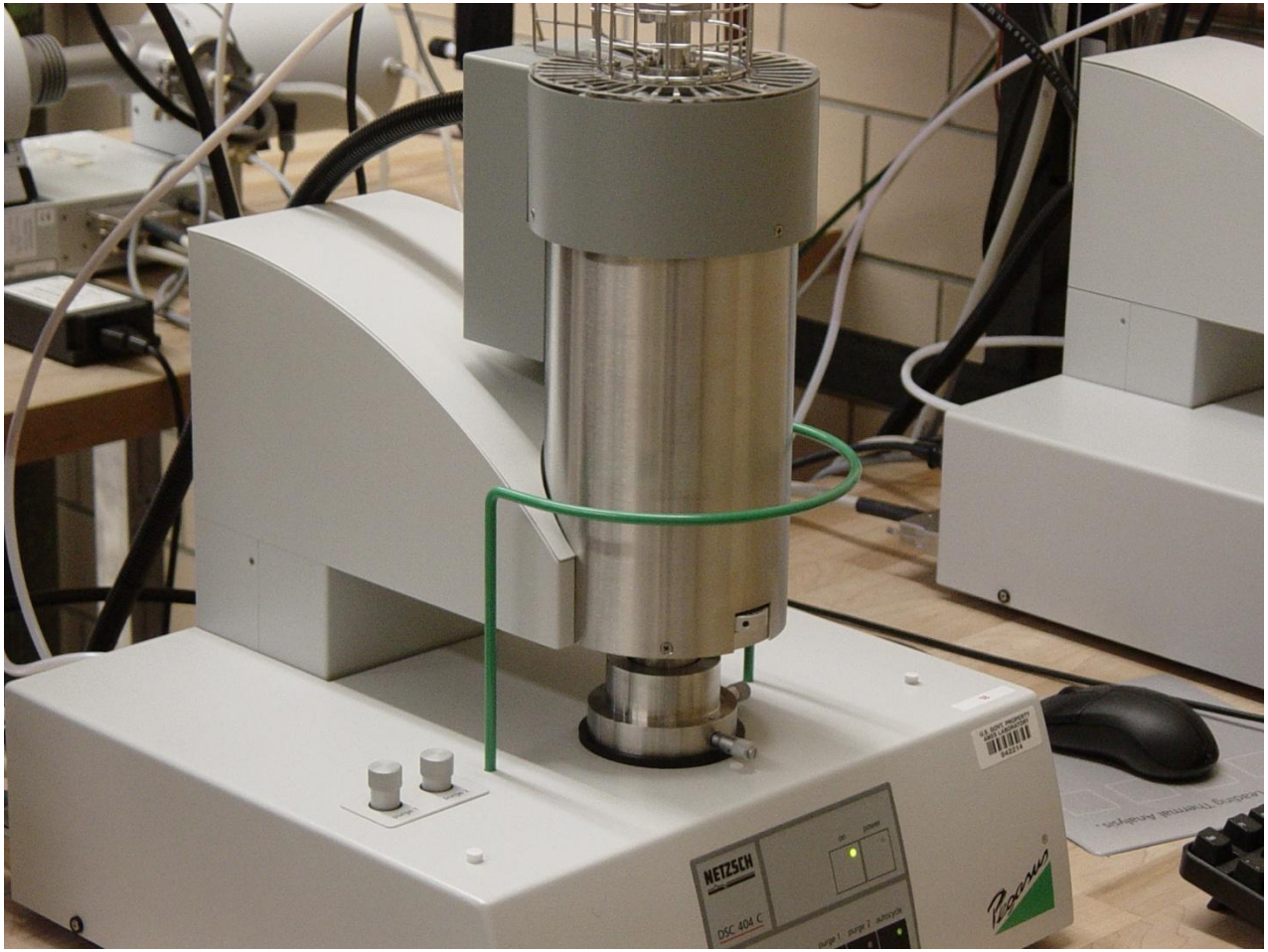
Dilatometer

Dilatometer

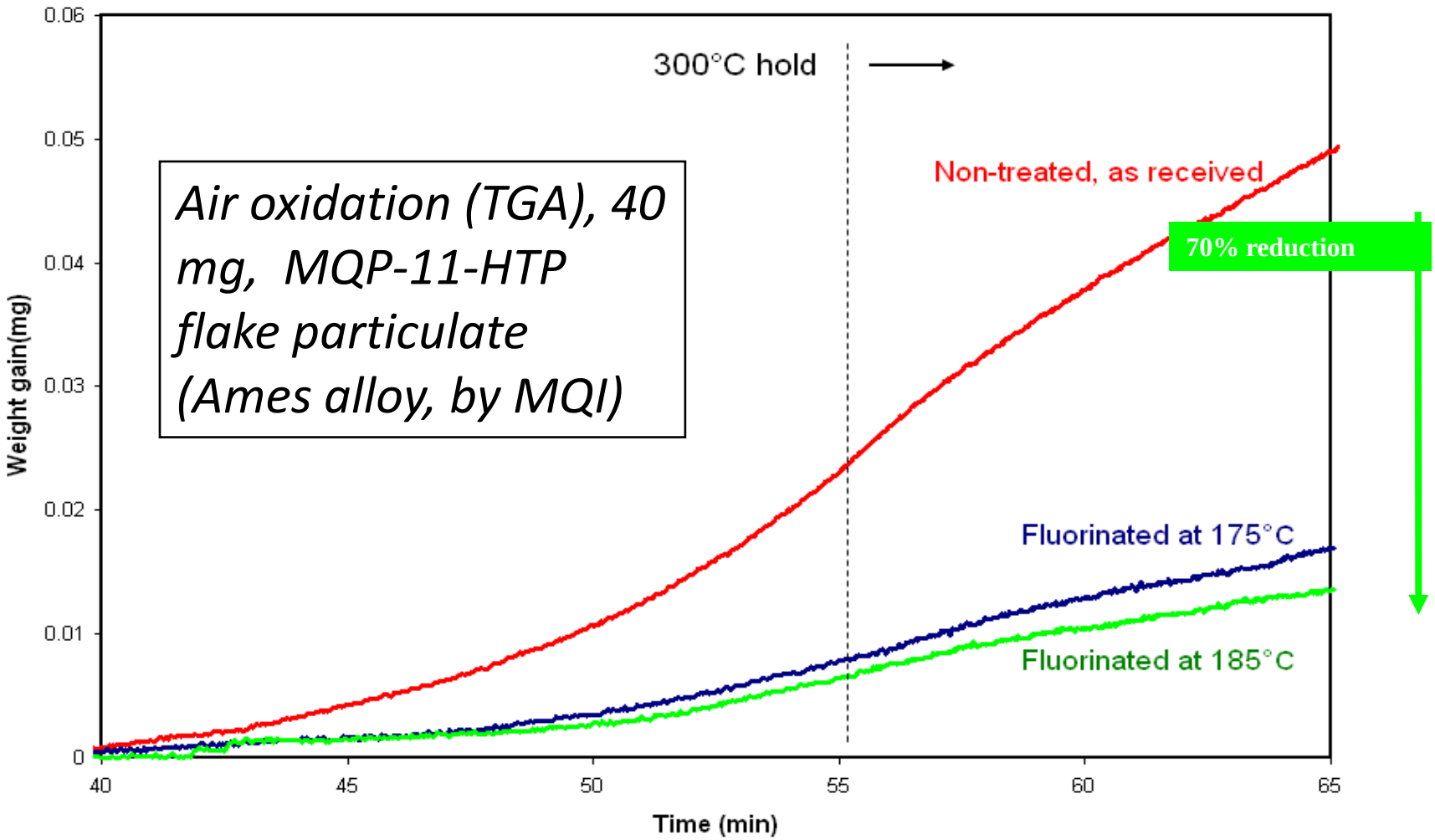



DIL 402 C on 18-414/4 ; Measurement - MBDIL-1-6 Al70Cu30.sle
File Edit View Measurement Graph Tools Diagnosis Extras Help
* TEMP 1030°C S * R 1*1055* 5.00*00:48
dL/Lo*10-3
Temperature /°C
View Signals
Temp.: 1029.9 °C
DIL: -245.62 µm
Selftest mode
Remaining Measurement Time
Time left: 1:40:59
0
-10
-20
-30
-40
-50
1200
1000
800
600
400
200
0
50
100
150
200
250
300
time /min, all segments
Segment 1, heating to 1300.0°C
Running
-245.62 µm
1029.9 °C
start
DIL 402 C on 18-414...
3:13 PM

TGA



TGA



Air oxidation (TGA), 40 mg, MQP-11-HTP flake particulate (Ames alloy, by MQI)