Low Temperature Materials Testing

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Introduction

- Material testing is fundamental to most scientific endeavours

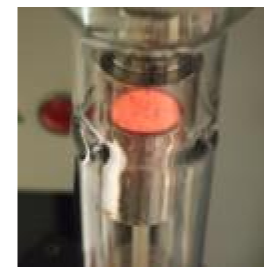
- Today – Looking at some examples:
  - Mechanical testing
  - Radiation tolerance
  - Target testing

Expertise

- Vacuum impregnation
- Surface preparation for bonding
- Radiation hardness of polymers
- Insulation for Nb_3Sn magnets
- Chemical consultancy, safety and environment, waste management
- Production of composites
- Mechanical testing to 4K
- Thermal analysis

Characterisation of polymers

- We have facilities and experience to perform:
  - Thermal analysis (DSC, TGA, DMA)*
  - Viscometry
  - Scale-up trials



* DSC: Differential Scanning Calorimetry
TGA: Thermo-Gravimetric Analysis
DMA: Dynamic Mechanical Analysis

Testing at low temperatures

- Development of materials for superconducting magnets requires testing in the cryogenic regime.
- We perform mechanical testing at 77K and 4.2K at loads up to 100kN with a range of extensometry facilities
- Servo-hydraulic ESH-Zwick load frame
- Thermal contraction to 4.2K

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50kN Testometric testing machine

- A second facility for testing at 77K, capable to 4K with an under-slung cryostat
- Custom extensometry using LVDTs and strain gauges to 4K

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Effects of irradiation on Polymers

- A collaboration between STFC Advanced Materials Group, GSi and Univ Marburg is working to characterise ion-irradiated magnet insulation materials for SiS-300
- DMA and Hyper-DSC have been used to “fingerprint” polymer insulation materials before and after irradiation
- Method development for DMA has been critical to obtain good results

➢DMA – Dynamic Mechanical Analysis
➢DSC – Dynamic Scanning Calorimeter

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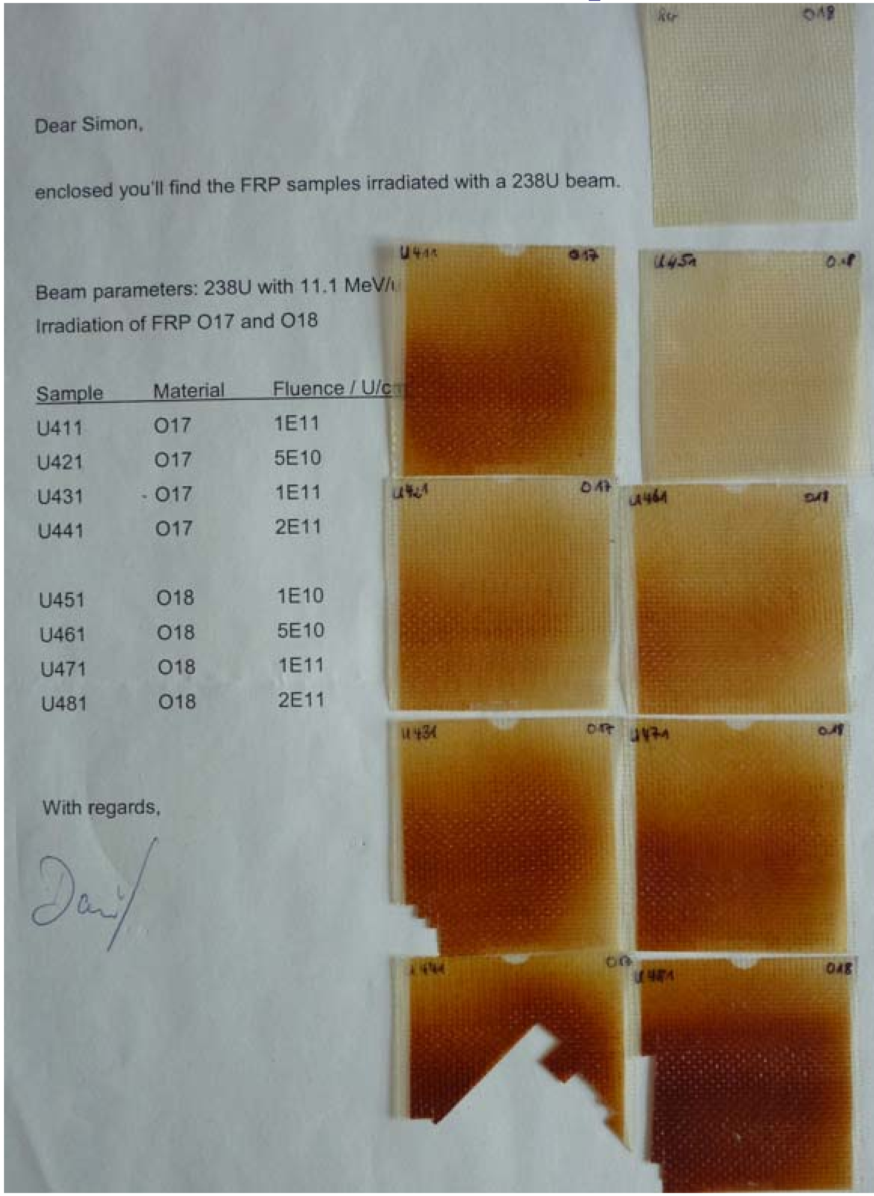


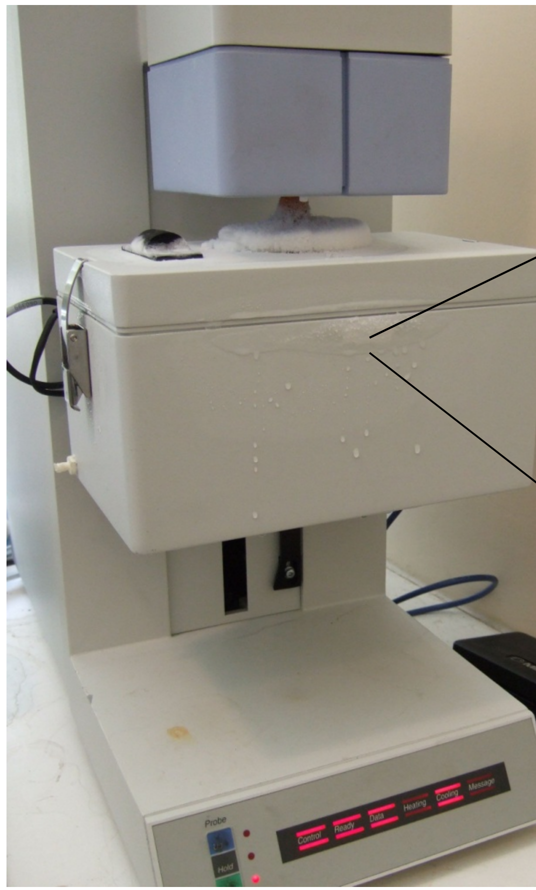
Perkin Elmer DMA7e
"a mechanical spectrometer"

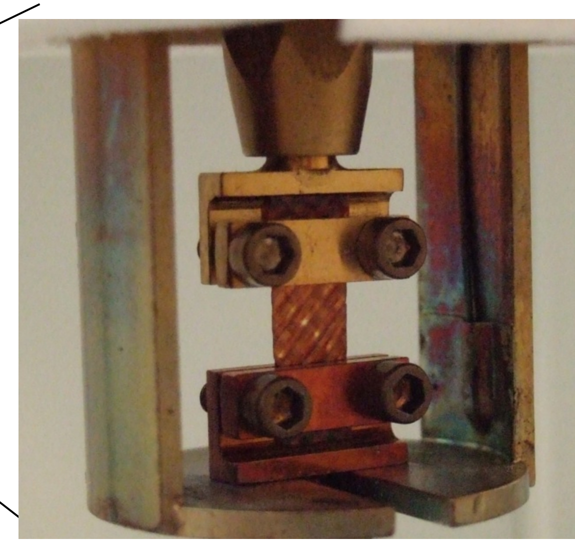
Can measure viscous liquids, solids, films with a range of measuring systems

Temperature range -160C to +200C (liquid nitrogen bath cooling)
-60C to 500C (fridge)

Force 8N (maximum)

Frequency 0.01 to 51Hz





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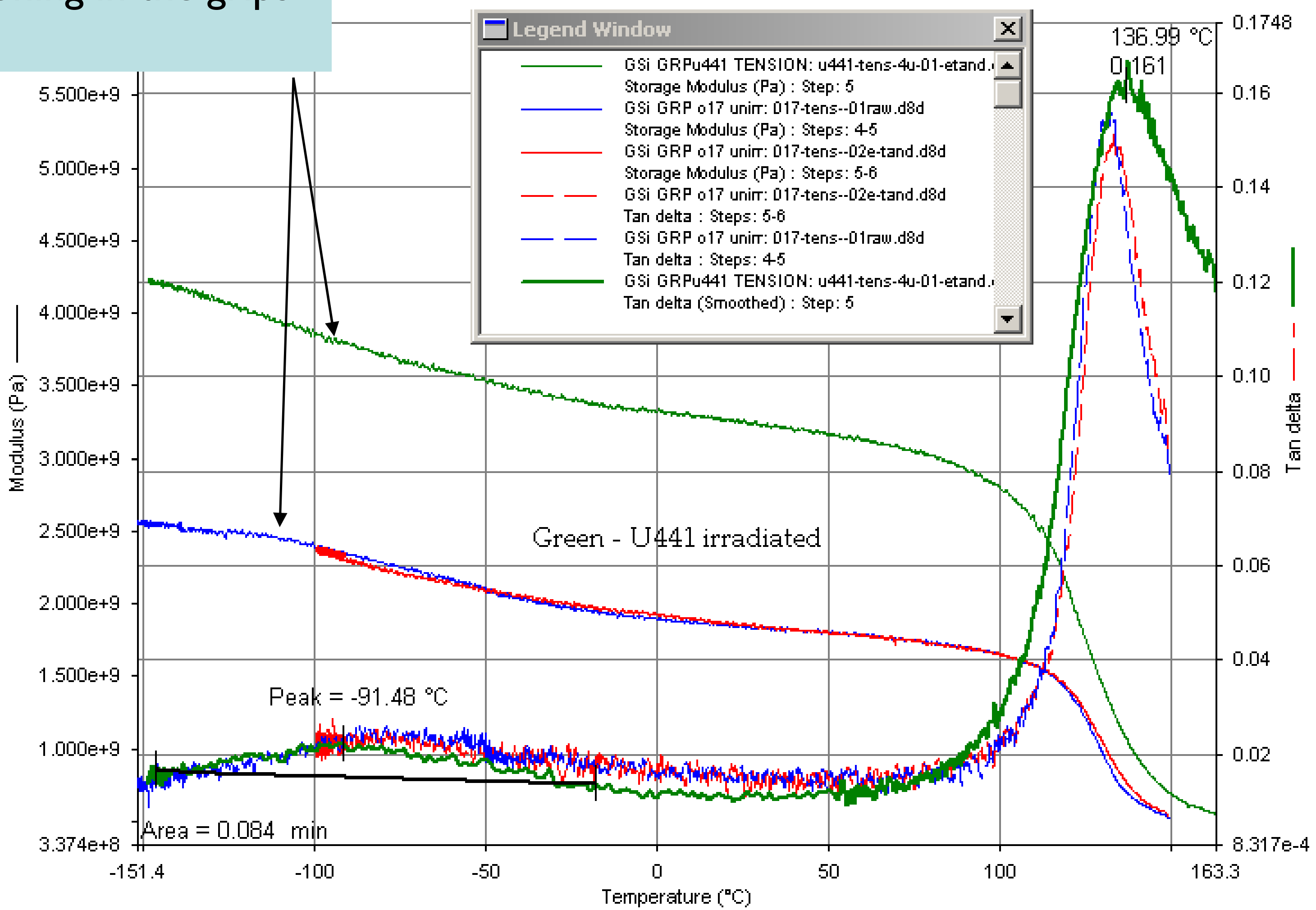
Perkin Elmer DMA7e "a mechanical spectrometer"

- An oscillating force is applied to the sample
- Always within elastic region
- Displacement is measured
- Viscoelastic properties lead to a phase lag
  - Storage modulus is in-phase component, "storage modulus"
  - Loss modulus is out-of-phase component "loss modulus"
  - Tan delta = E'/E"
- Can detect Tg with high sensitivity (10-100x low-speed DSC)
- Can detect other transitions below Tg that are not possible with other techniques
- These Secondary Transitions can relate to toughness – note that an issue with "noise" in the magnets (wires moving ?) means that toughness is an issue that needs to be understood

DMA – Irradiated and Unirradiated

NOTE Absolute modulus varies randomly, possibly related to sample length and positioning in the grips

Green – irradiated
Red – Unirradiated
Blue - Unirradiated



Hyper-DSC

Dynamic Scanning Calorimeter

- milligramme sample weights
- High speed (up to 500C/min) gives high sensitivity
- High speed avoids temperature-time effects on sample (postcure)

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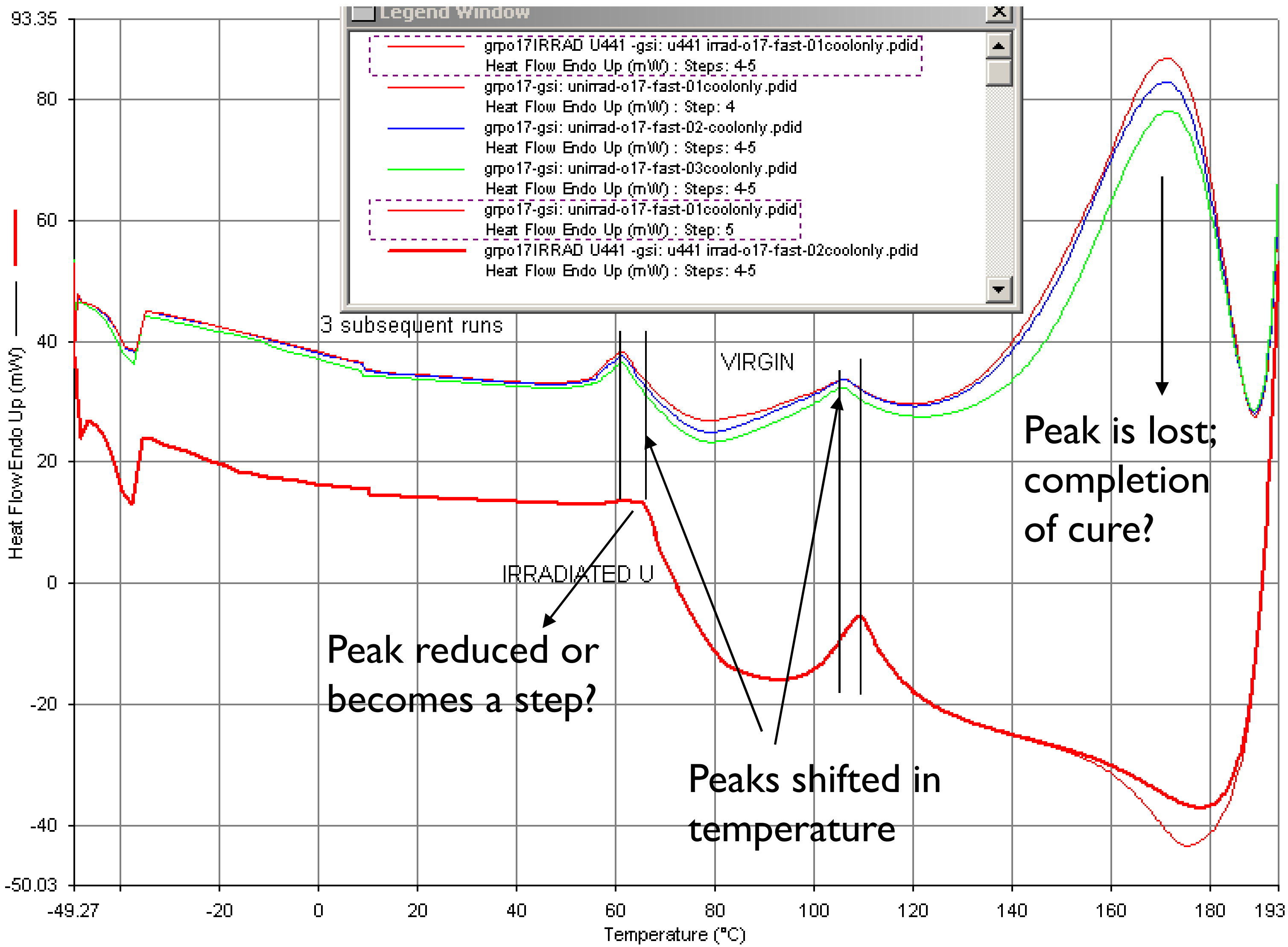
DSC: GRP virgin (top) U-238 irradiated (bottom)



25um thickness kapton, non-irradiated: 6kV

25um kapton, Ti ions

Electrical Breakdown

As an aside, electrical breakdown suffers:

25um thickness kapton, non-irradiated: 6kV

Reference	Ions/cm2	Breakdown voltage kV
Ti15	1e10	4
Ti75	1e12	3
Ti95	5e12	3.25

Summary of irradiation tests

- Both DMA and hyper-DSC show promise as tools for detecting the effects of irradiation on epoxy
- Test procedures have been developed and are being refined
- Testing on irradiated glass-epoxy, polyimide and other rad-hard polymers have been performed
- Results require interpretation

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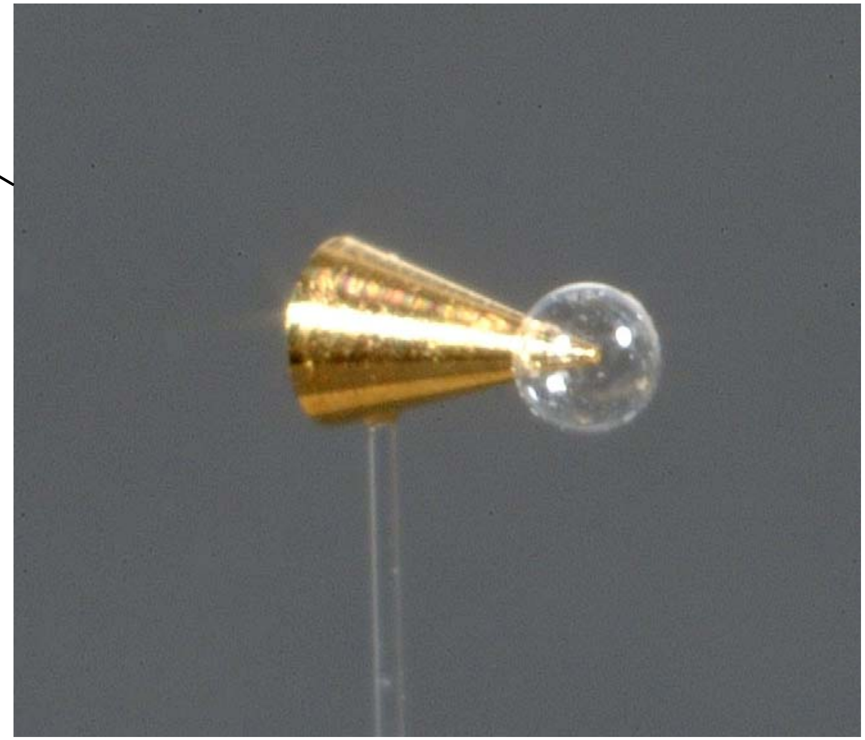
Target tests at low temperatures

- Tests on micro-balloon was done under vacuum
- Aim of the experiment was to test the glued joint between the cone and the balloon
- Balloon under vacuum
- Observed through cryostat window
- System was thermally cycled

Target tests at low temperatures

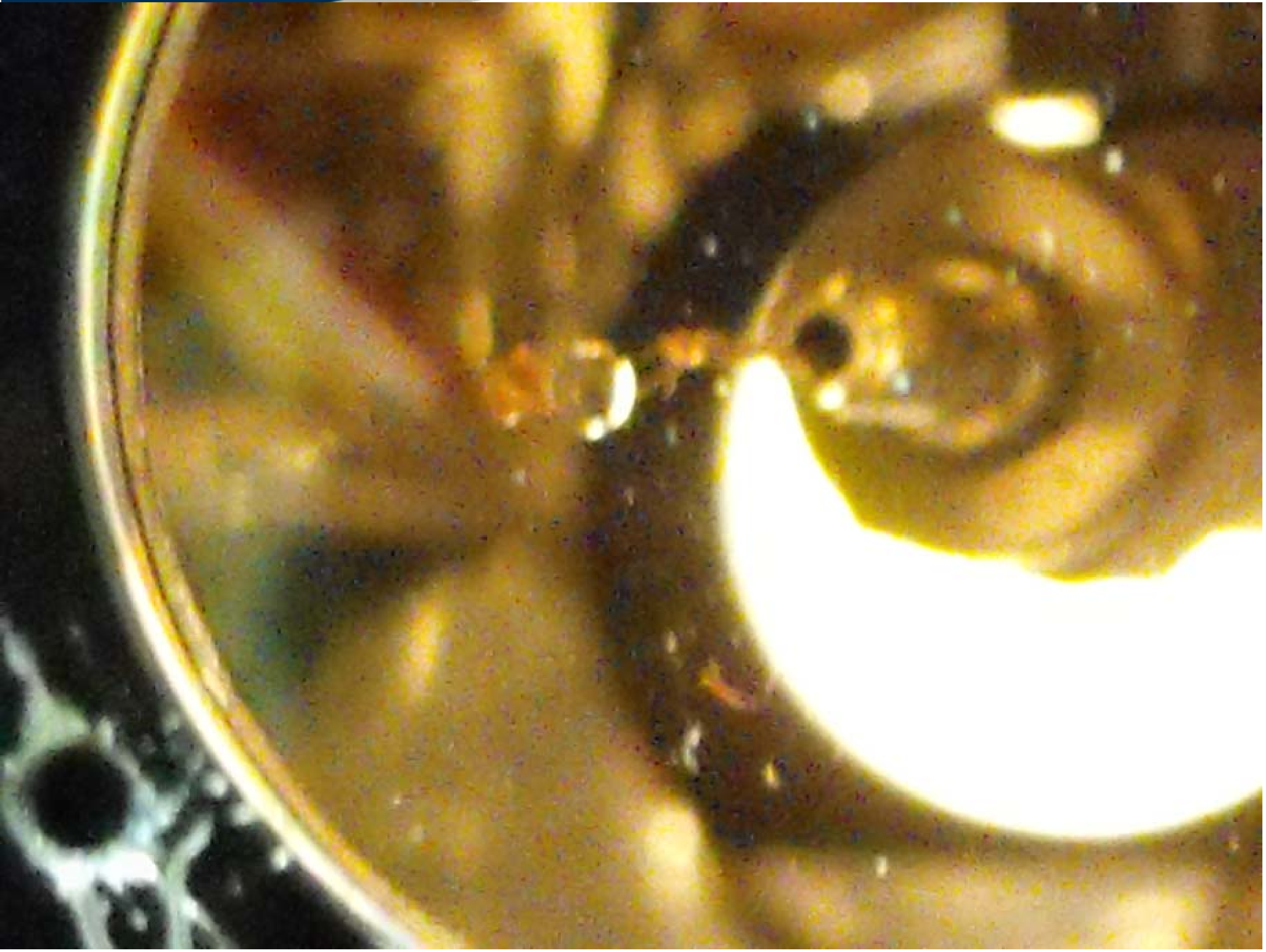
- Cryostat has windows so that the target can be monitored
- Put LN2 in the main bath and temperature cycle

Target tests at low temperatures



Test target - early tests using two part epoxy

Target tests at low temperatures



First tests were successful and tests are ongoing

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