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Magnet Insulation materials for FAIR: Dynamic Mechanical Analysis and High-Speed Differential Scanning Calorimetry of glass-epoxy before and after U-238 ion irradiation

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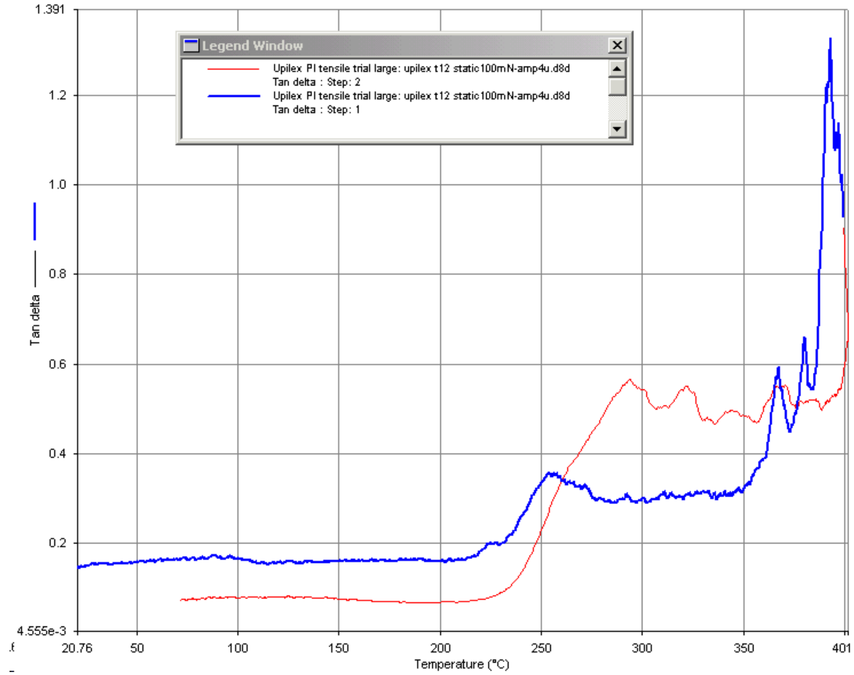
Summary

- A collaboration between CCLRC, 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 on polyimide, tension

- Polyimide is challenging for the DMA because:
- Very high modulus drop at high temperatures
- No glass fibre support
- Leads to very low forces
- Low signal-to-noise
- Experience with GRP should help with method development on



Polyimide (kapton 25micron), 20 to 400°C.

4 micron amplitude control on DMA. Note noisy results.

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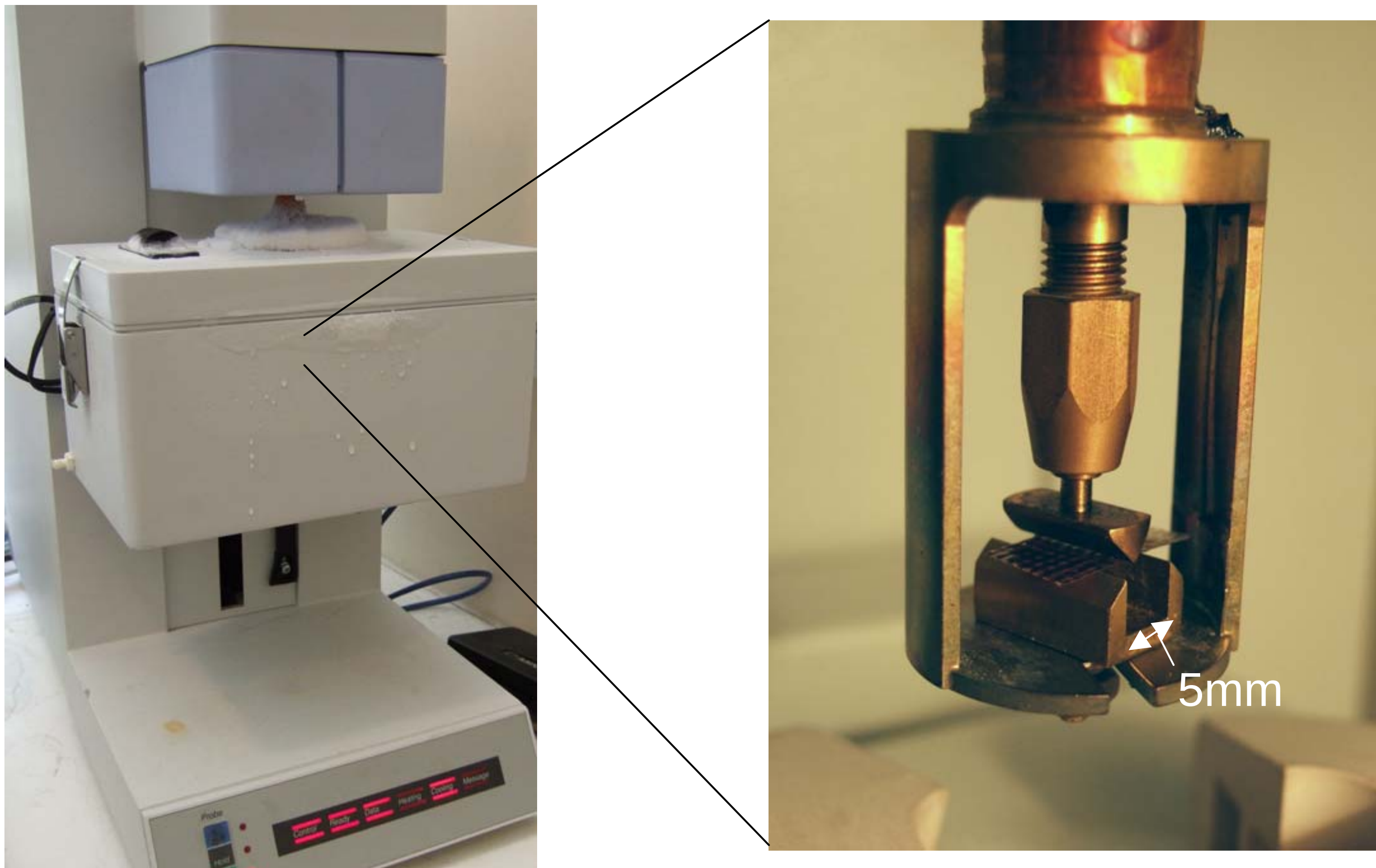
Perkin Elmer DMA7e
“a Mechanical Spectrometer”



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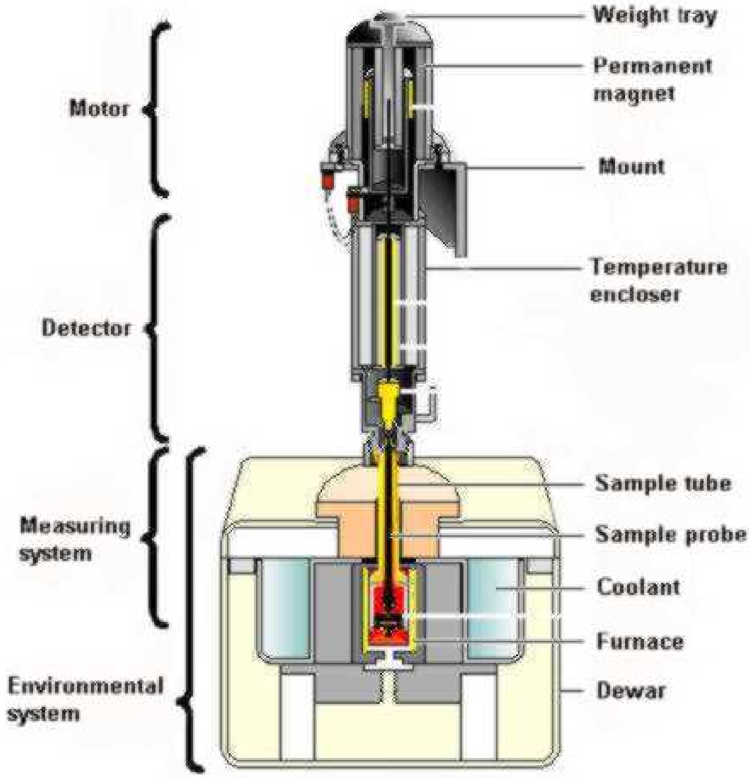


(Perkin Elmer image)

- 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

Frequency 0.01 to 51Hz

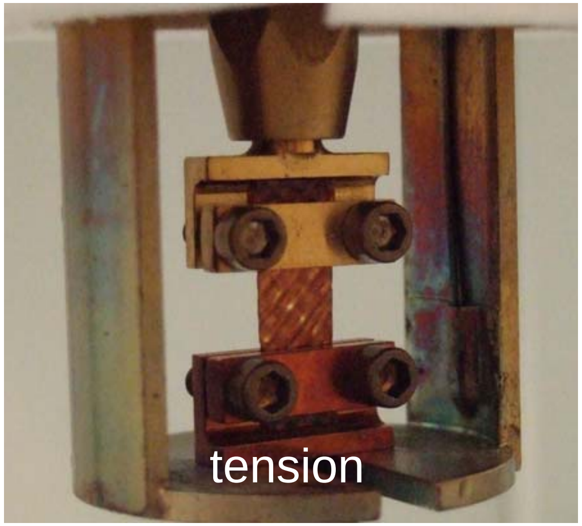
DMA

- 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.

Loading modes

- 3-point bend at 0/90deg to fibres:
  +simple set-up
  – Low force on thin laminates, poor signal-to-noise
- 3-point bend at 45deg to fibres:
  +simple set-up, +More strain on resin so potentially more information
  - Even lower force
- Tension at 45 deg to fibres:
  +High force so **excellent** signal-to-noise
  -time consuming set-up

TENSION was chosen



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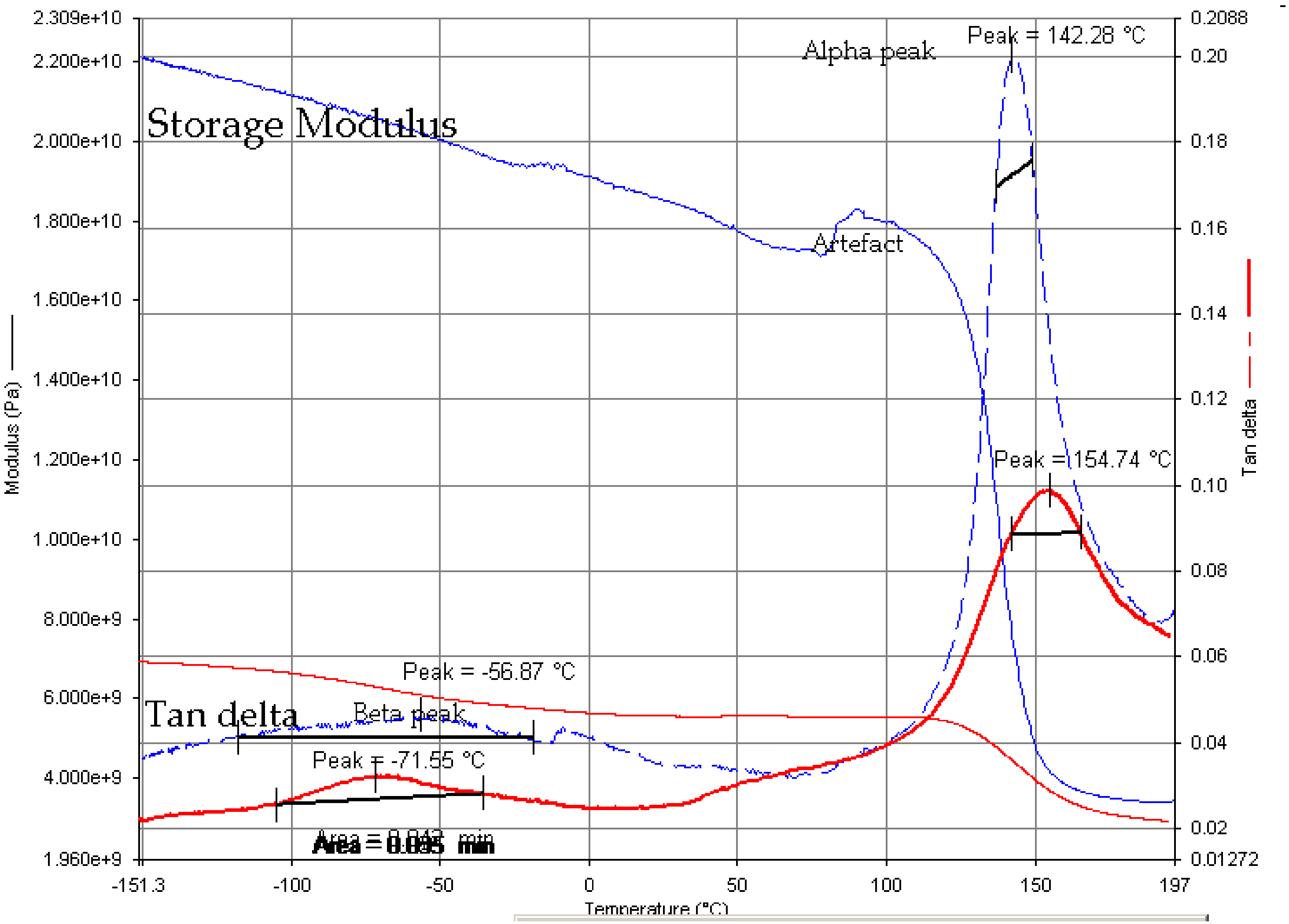
Early DMA results showed many “artefacts”
First results – 45deg 3point bend
Irradiated red, unirradiated blue



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Hyper-DSC

- milligram sample weights
- High speed (up to 500C/min) gives high sensitivity
- High speed avoids temperature-time effects on sample (postcure)
- Technique uses two cells and measures the difference in energy going into a reference and sample chamber

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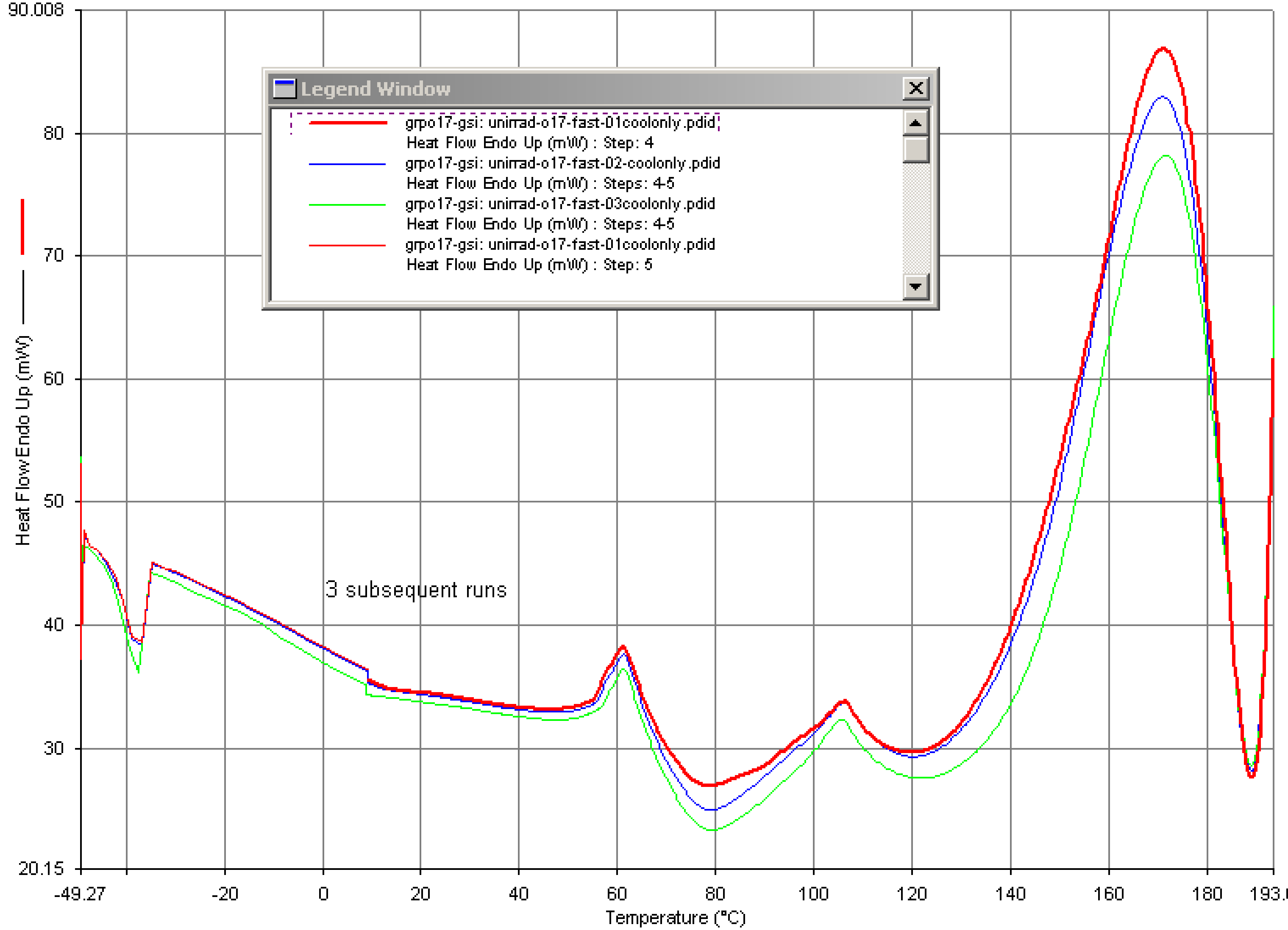
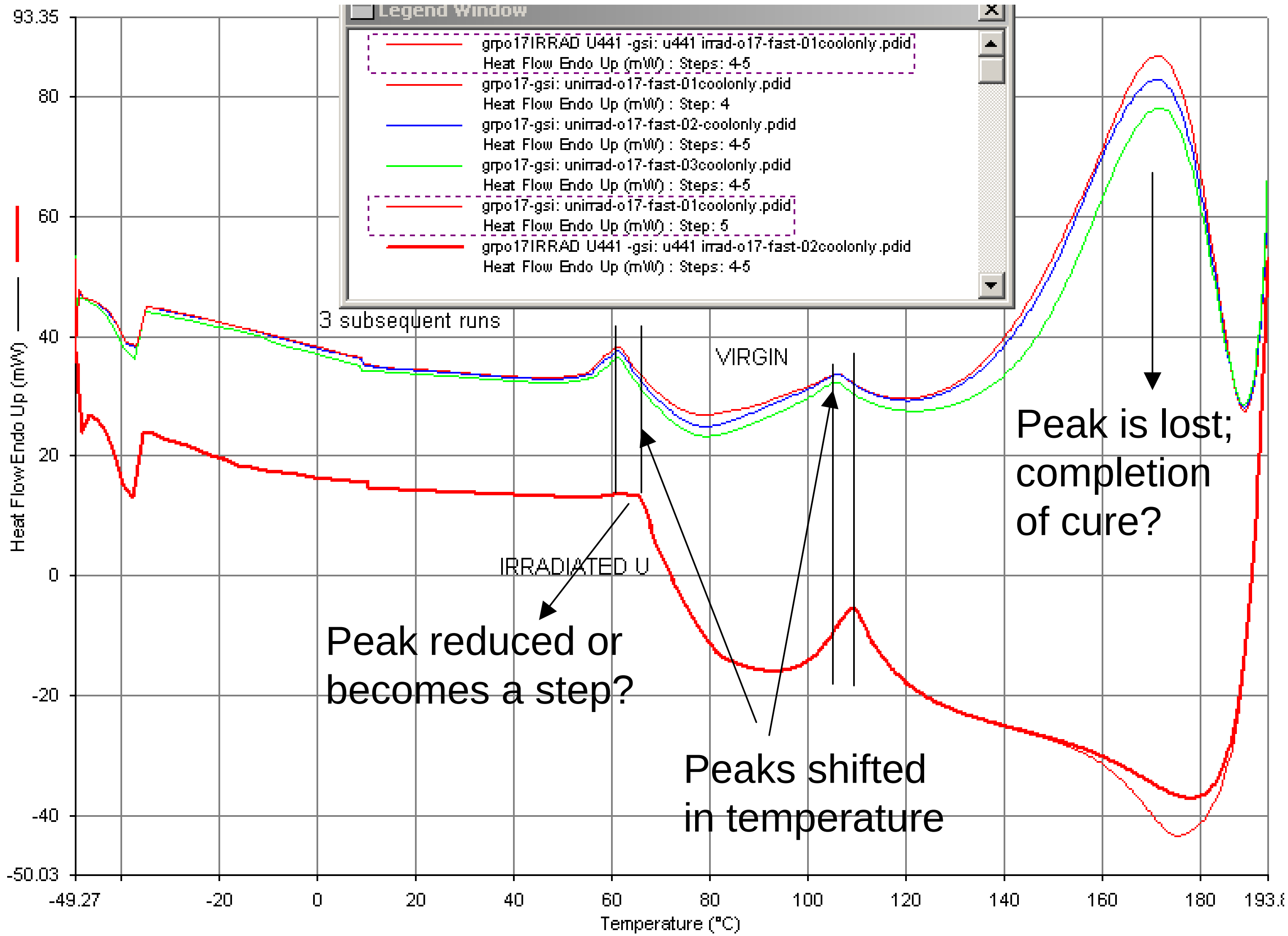
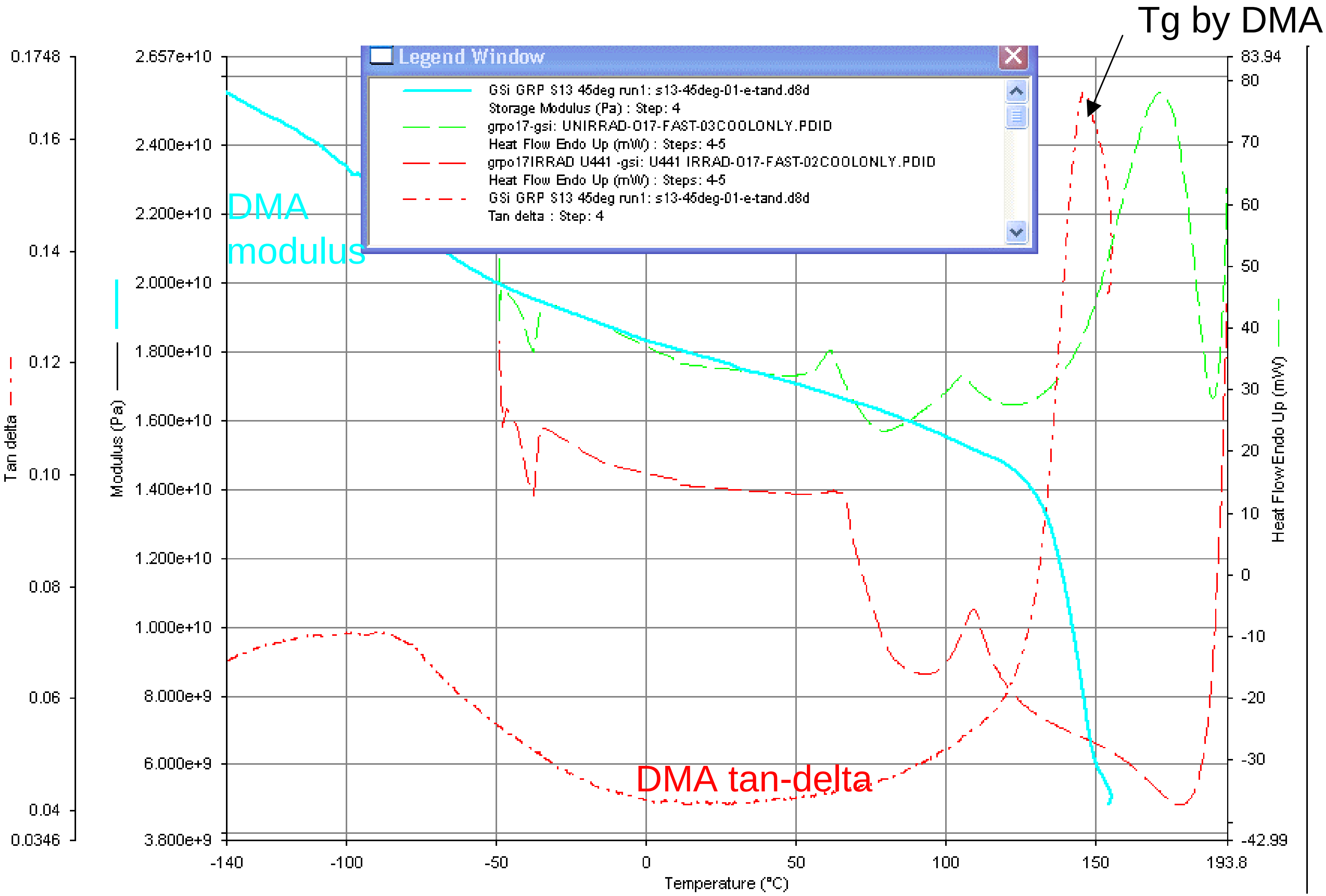
Reproducibility – cooling curves 400C/min GRP O17 (S-glass, Epoxy Bis-F/DETD)



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



DSC compared to DMA – work in progress



Conclusions

- Both DMA and hyper-DSC show promise as tools for detecting the effects of irradiation on epoxy
- Test procedures have been developed that use small samples
- Testing on irradiated glass-epoxy and polyimide will be performed
- Results require interpretation

Future Work

- Very little work done in the area of irradiation with ions.
- Need to compile a database of rad hard materials – irradiation with ions is a very useful complement to “conventional” work.
- Want to go back and understand polyimide and other materials.
- Need to correlate the various test methods and understand the results. (Including Daniel’s FTIR work)
- Fatigue work is important and this is being looked at.
- There is a lot to do and this work is quite unique.

END

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Appendices

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Test method

- DMA can be used in many loading modes
  – tension, 3-point bend...
  – controlled strain or controlled stress
- Control can be difficult due to orders of magnitude changes in sample modulus, hence force, during test
- PID parameters for position need to be tuned to keep stable amplitude and “clean” curves
- Parameters:
  – 3 point bend, 5mm span
  – 5C/min ramp rate (low to avoid temperature gradients)
  – Amplitude control to use full force range (eg 55 micron)
- ***Test method development is critical***

Summary of test procedure DMA

- Measure specimen and load into machine
- Start machine in “dynamic control”
- Fill liquid nitrogen bath
- Allow to cool to -170C
- Check load limit has not been reached, check amplitude is stable
- Start test
- Refill nitrogen at end of heating run
- Save raw data
- Produce these graphs:
  - Position vs time
  - Amplitude vs time
  - E and tan delta vs temperature
- Check position and amplitude are within limits
- 3 reproducible results are required

(NB this is a summary only and is NOT the actual procedure to be followed)

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