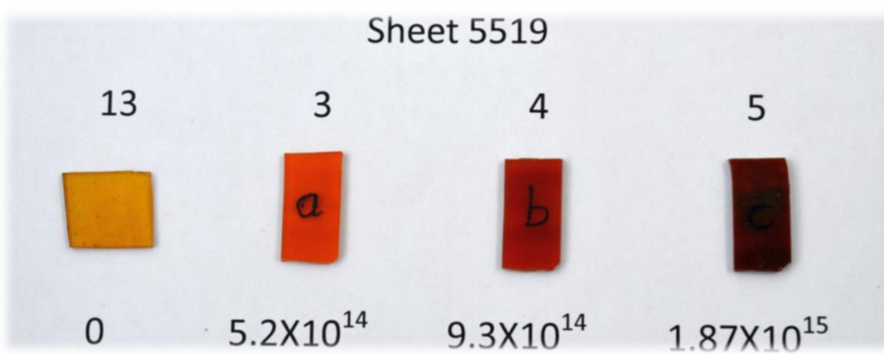
Radiation Damage in Epoxy Resins

Steve Robertson, Simon Canfer



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- Composite materials are widely used as detector structures in HEP, eg ATLAS detectors
  - Main structure
  - Other adhesives for assembly
- Low Z materials
- Properties of the organic matrix can change as a result of accumulated radiation dose in LHC detectors

Radiation hardness of polymers

To some extent the chemical structure can be used to predict radiation hardness:

Good: Aromatic rings, high crosslink density

Bad: long aliphatic chains, low crosslink density

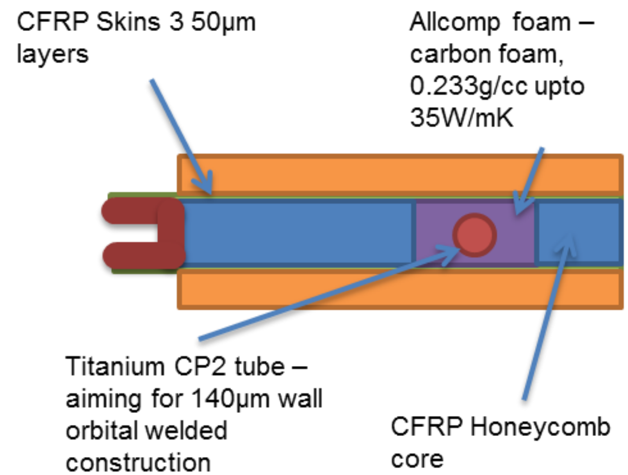
This tends to imply rigid, brittle polymers!

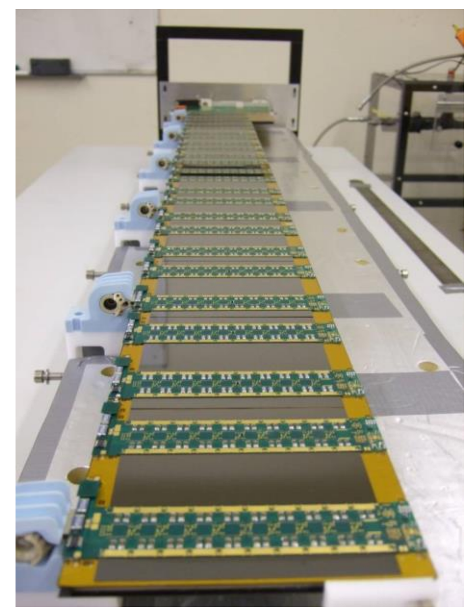
Radiation type is a large factor and often overlooked, e.g. Kapton has been shown to be more damaged by neutrons than gamma (Ref. Abe 1987).

We set out to determine if a flexible, low Tg epoxy could offer sufficient radiation hardness for a HEP detector.

ATLAS Stave

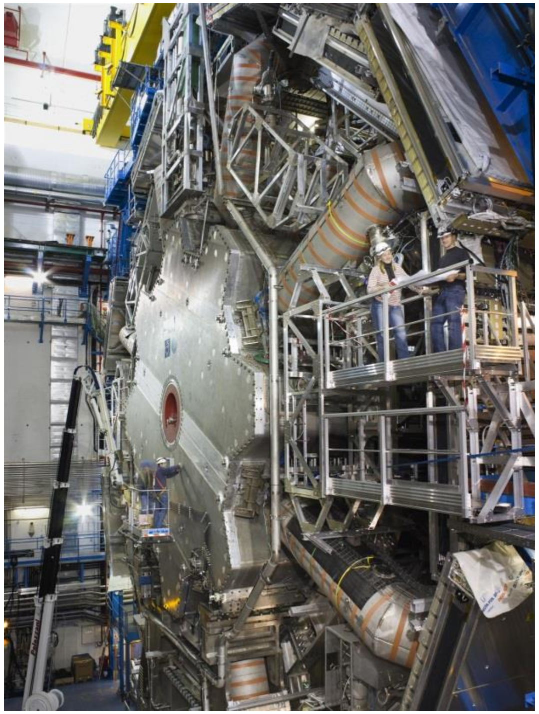
- Stave components rely on adhesive bonding
- Differences in CTE mean that flexible materials are desirable
- How do material properties change with radiation dose?
- If stiffness increases (as expected) then stress states within stave also change





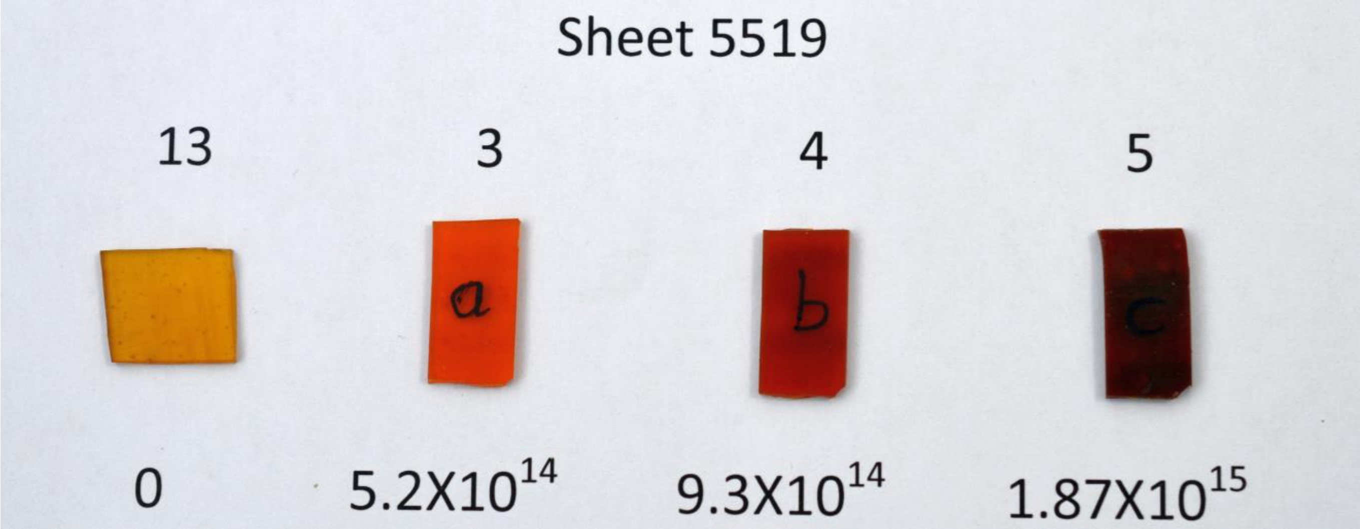
Our flexible Epoxy Resin System

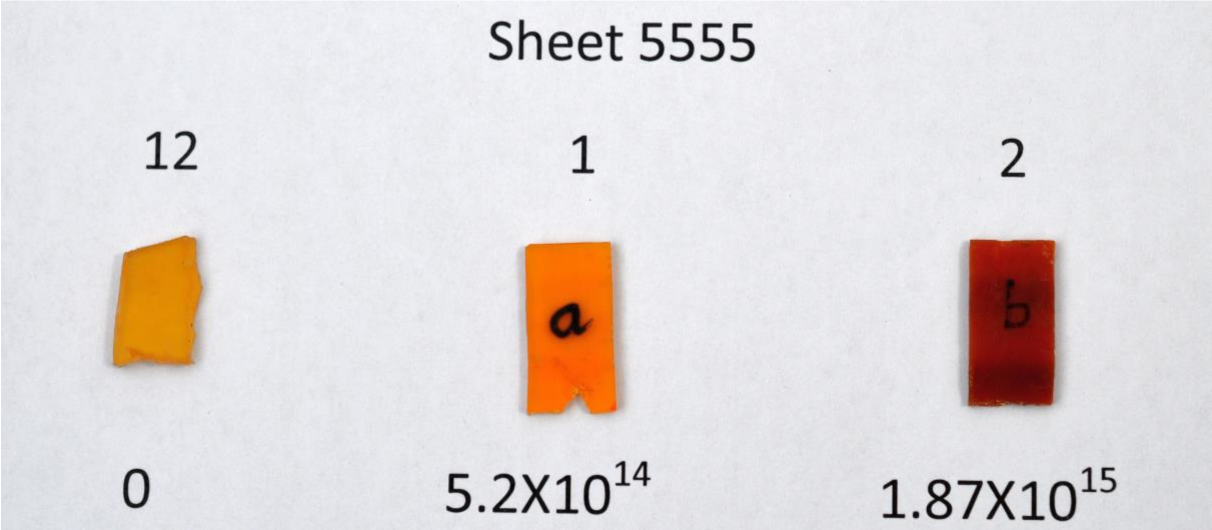
- Based on AECT Resin:
  - DGEBF / PPGDGE / DETD
- Additional flexibliser POPDA
- Two Test Resins:
  - 5519: 16% flexibliser
  - 5555: 50% flexibliser



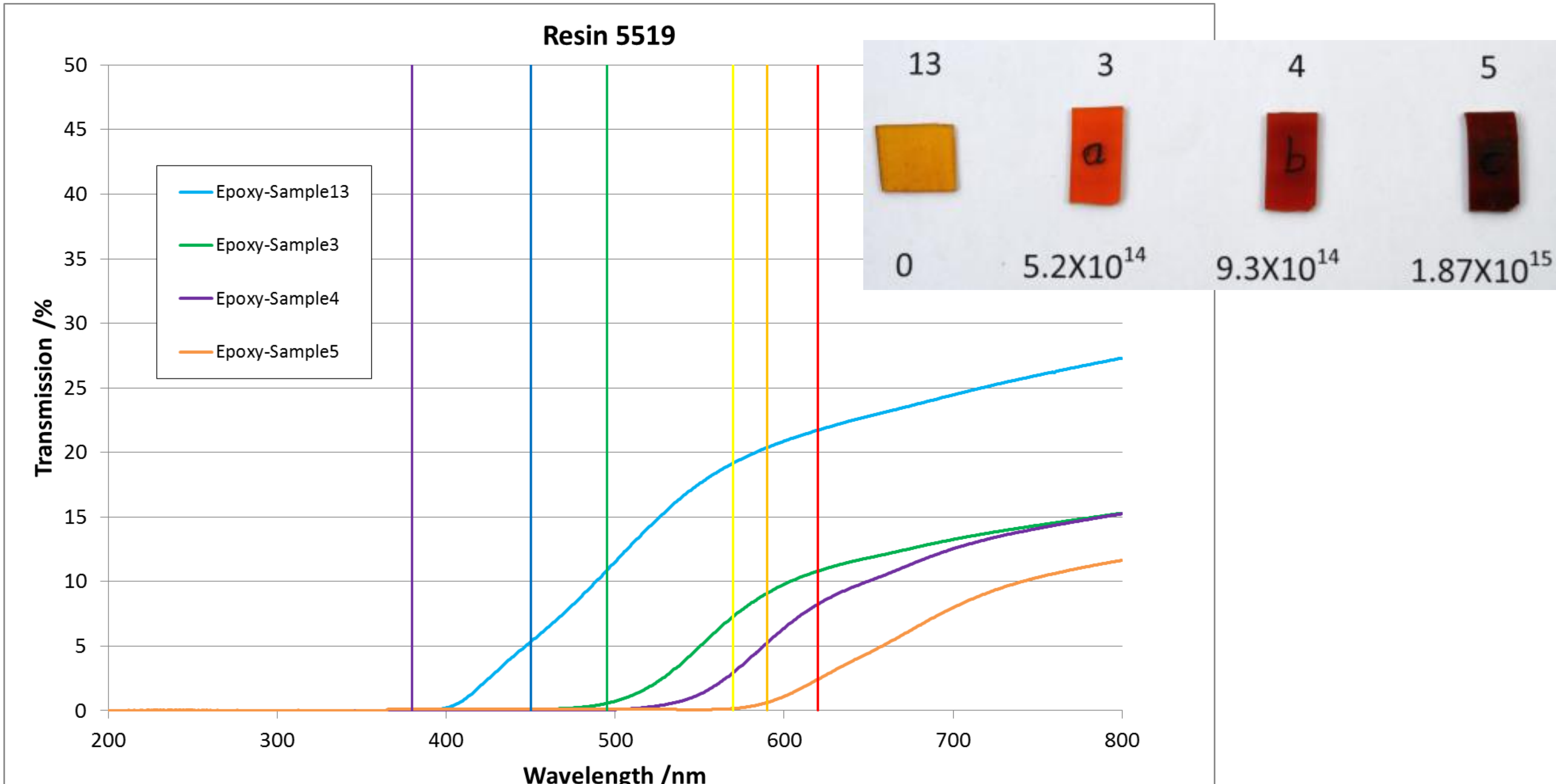
Samples

26MeV Protons, Birmingham cyclotron
Courtesy:
Dr. John Wilson





UV-Vis Spectroscopy



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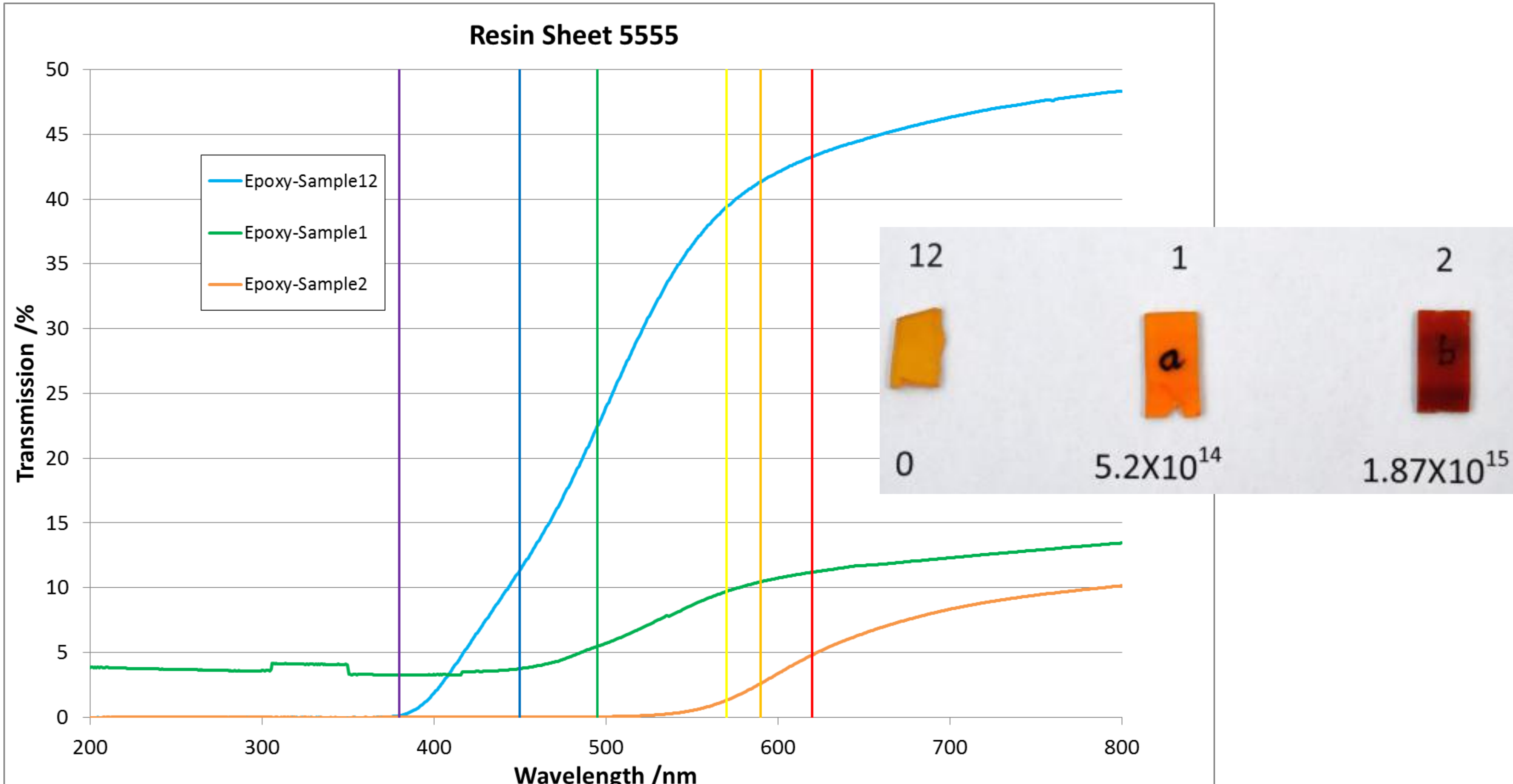
UV-Vis Spectroscopy



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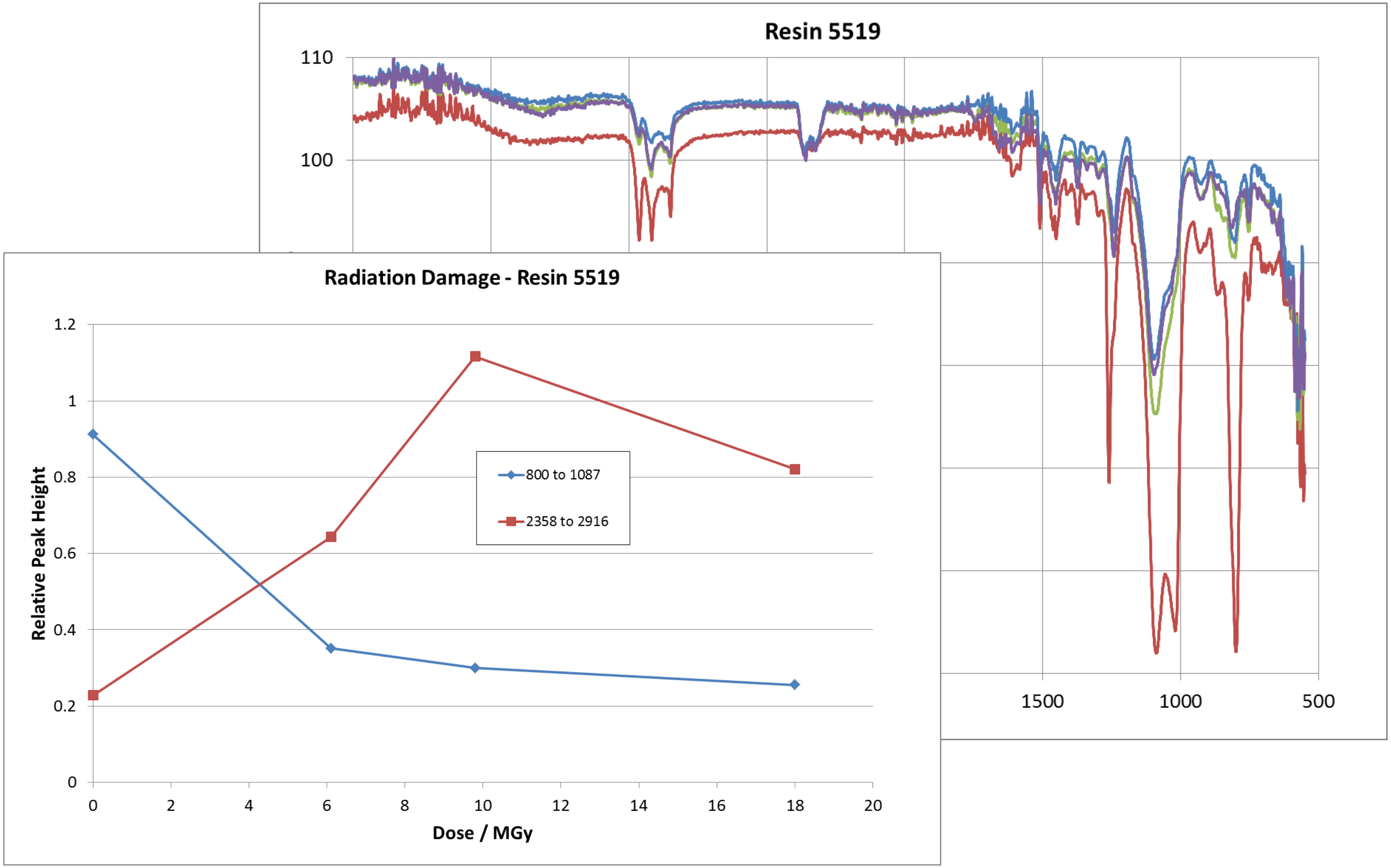
FTIR Spectroscopy



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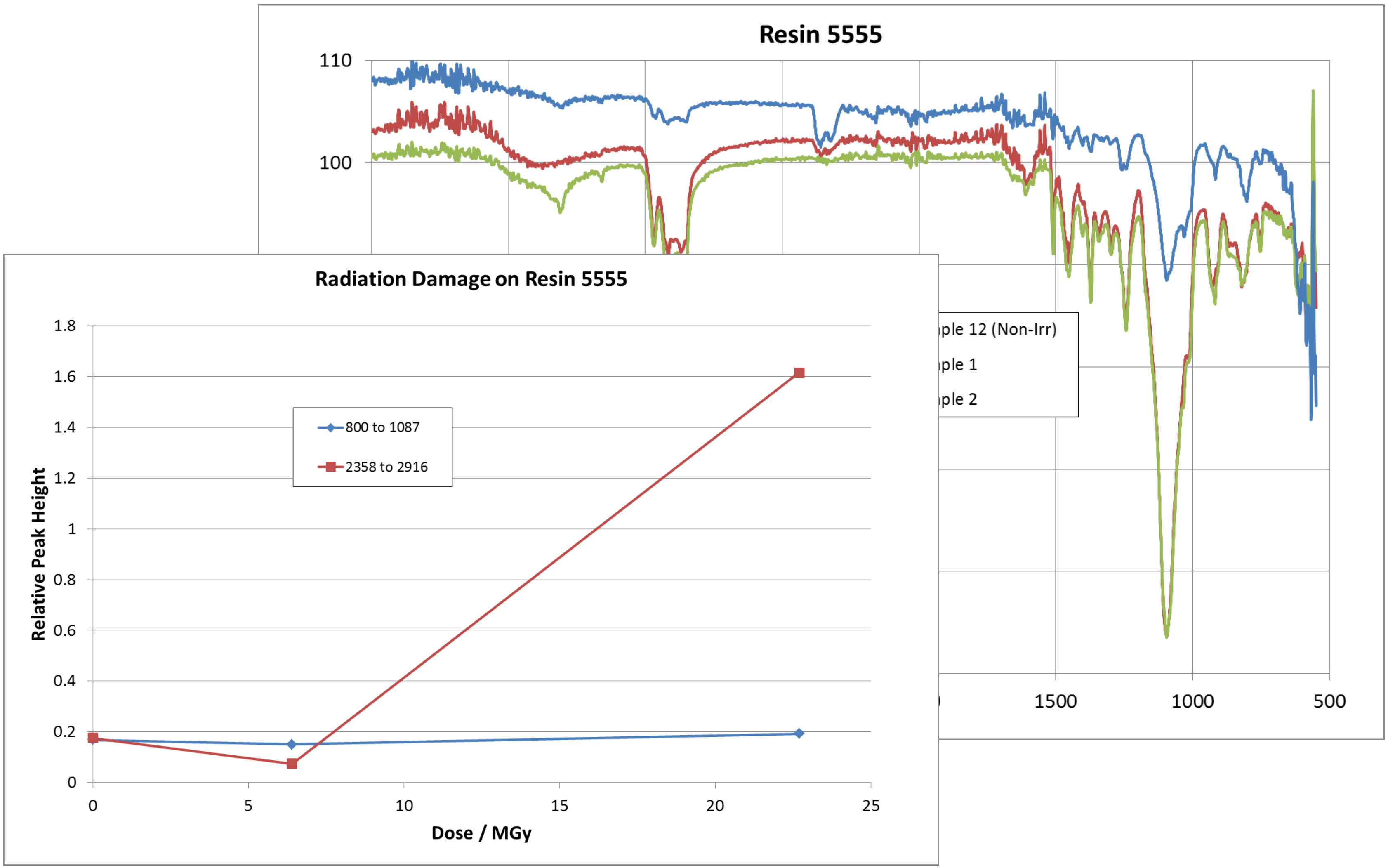
FTIR Spectroscopy



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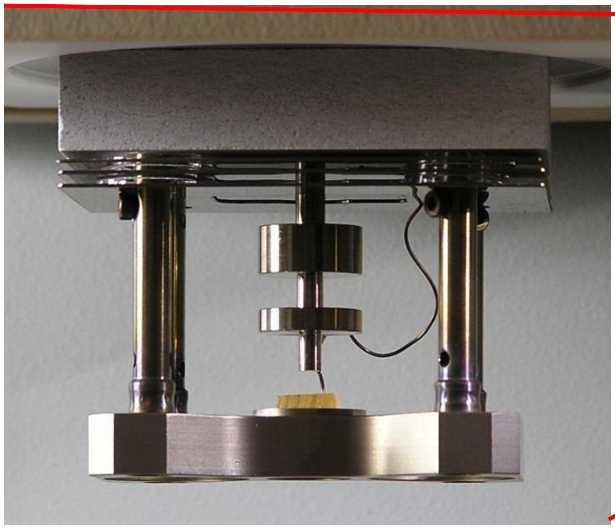
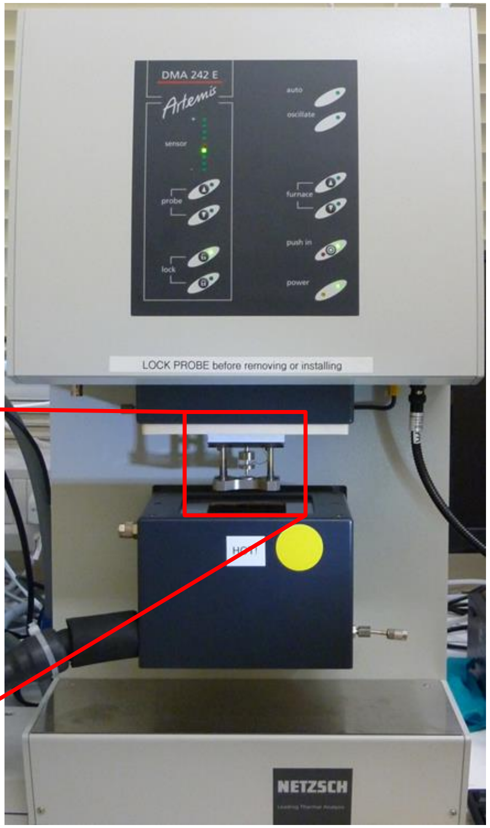
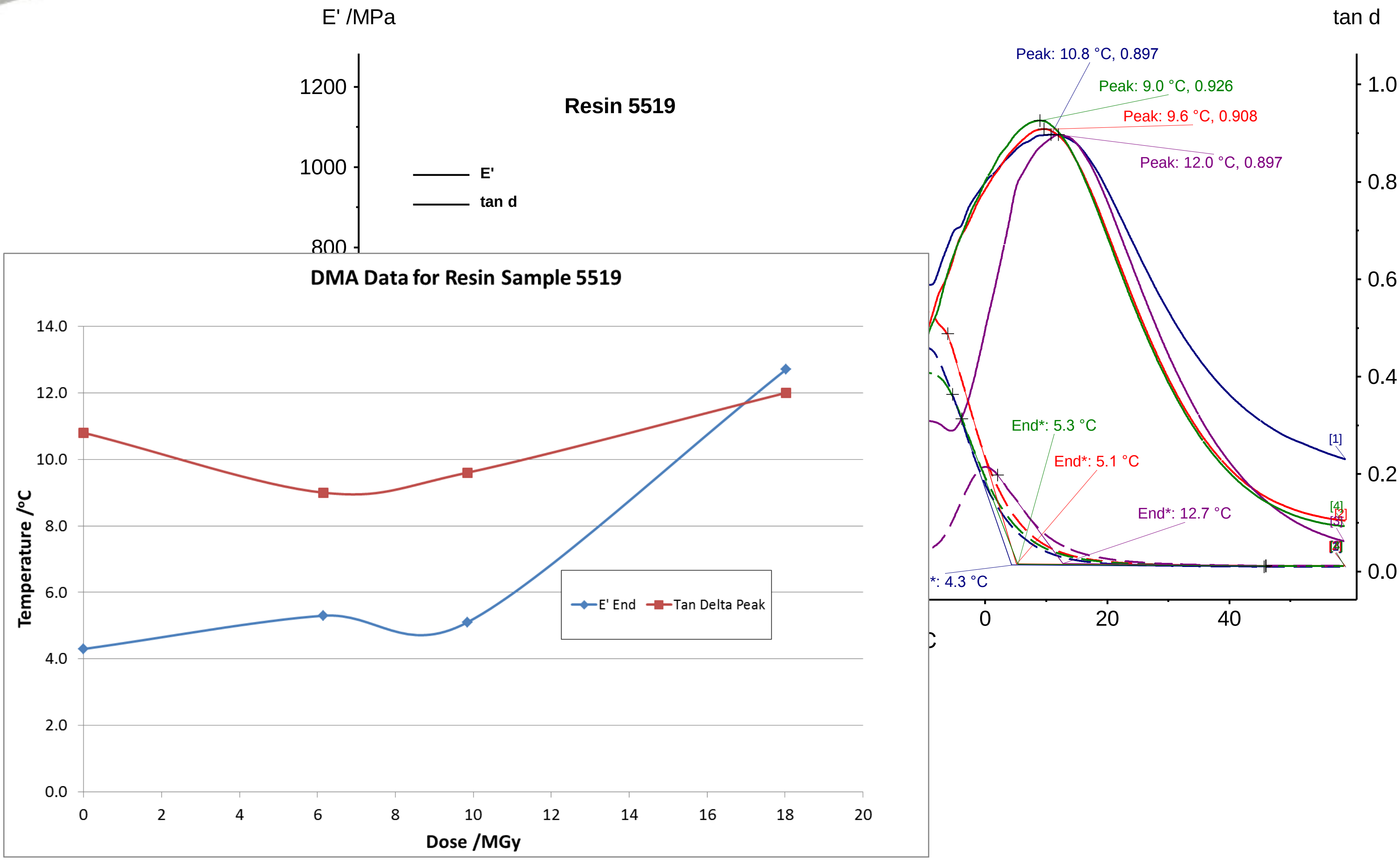
Dynamic Mechanical Analysis

- Applies oscillating forces to sample.
- Many geometries (tensile, penetration, flexure)
- Measures mechanical changes in materials as they are heated (e.g. glass transition)



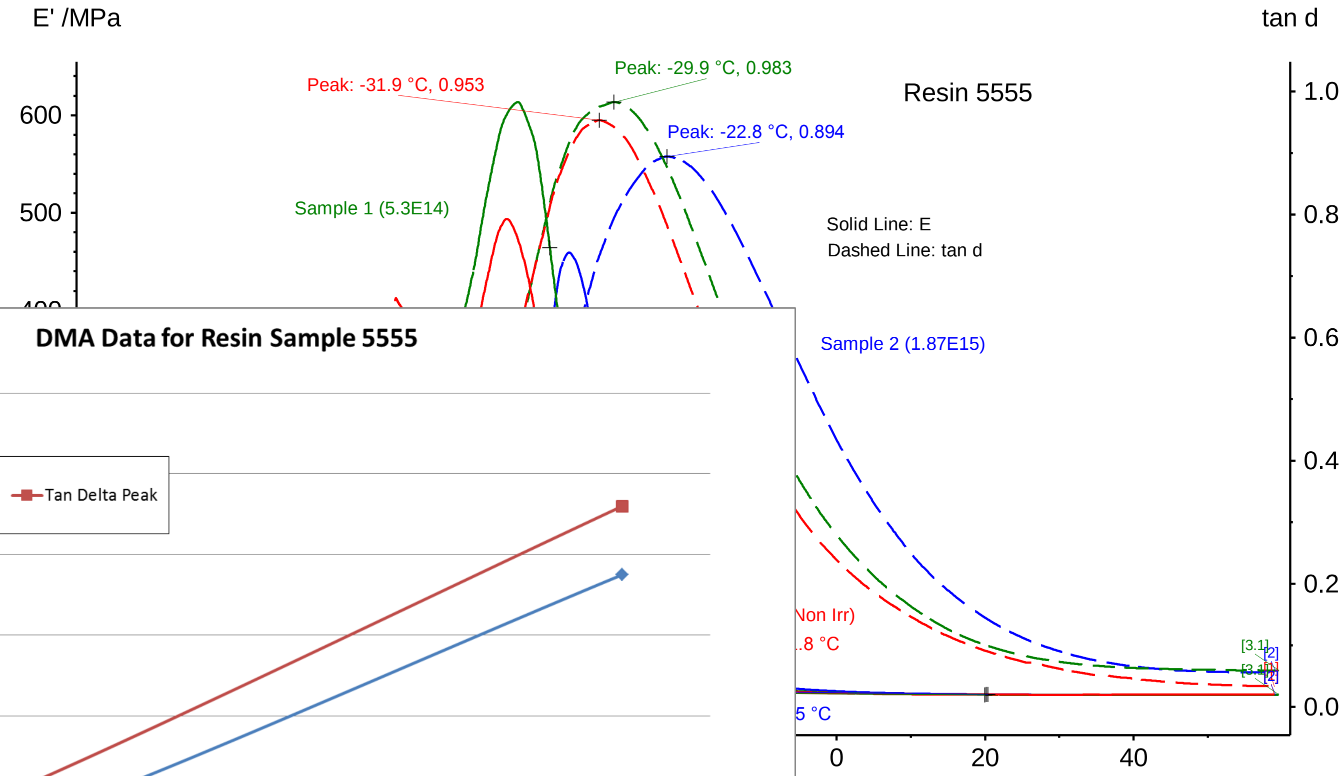


DMA Results

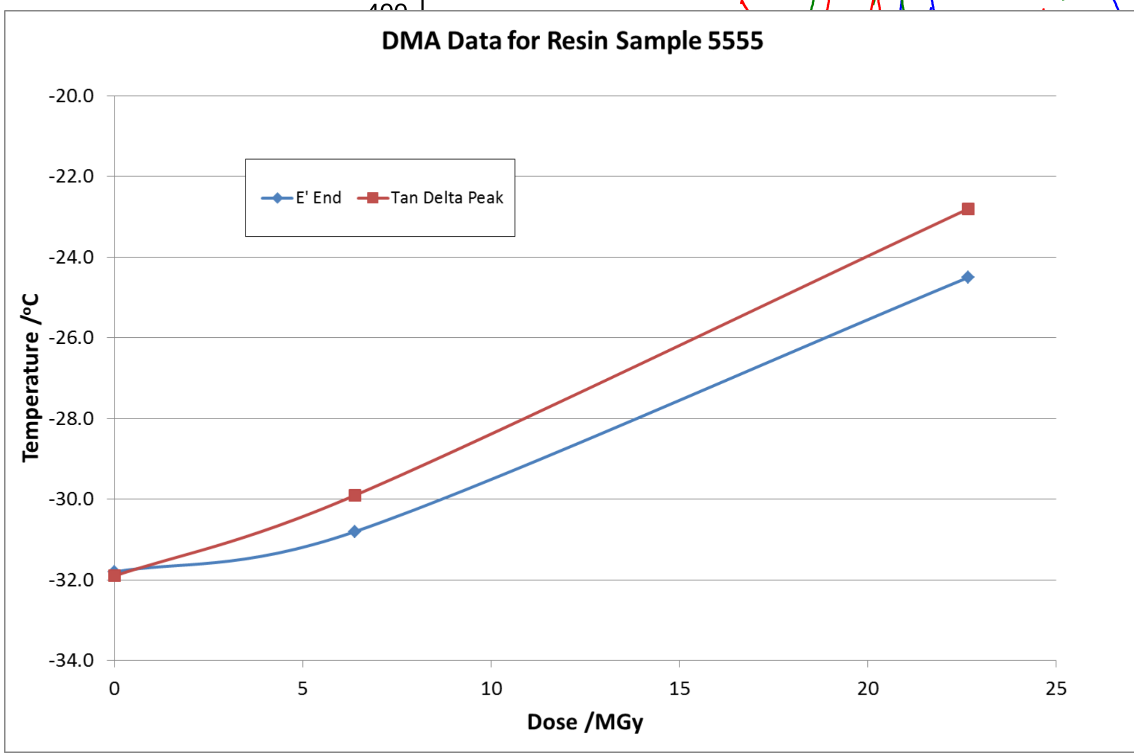


E' /MPa
tan d
Resin 5519
E'
tan d
1200
1000
800
1.0
0.8
0.6
0.4
0.2
0.0
0
20
40
Peak: 10.8 °C, 0.897
Peak: 9.0 °C, 0.926
Peak: 9.6 °C, 0.908
Peak: 12.0 °C, 0.897
End*: 5.3 °C
End*: 5.1 °C
End*: 12.7 °C
*: 4.3 °C
[1]
[4]
DMA Data for Resin Sample 5519
Temperature /°C
Dose /MGy
E' End
Tan Delta Peak
14.0
12.0
10.0
8.0
6.0
4.0
2.0
0.0
0
2
4
6
8
10
12
14
16
18
20

DMA Results



E' /MPa
tan d
Resin 5555
Peak: -31.9 °C, 0.953
Peak: -29.9 °C, 0.983
Peak: -22.8 °C, 0.894
Sample 1 (5.3E14)
Sample 2 (1.87E15)
Solid Line: E
Dashed Line: tan d
600
500
1.0
0.8
0.6
0.4
0.2
0.0
0
20
40



DMA Data for Resin Sample 5555
E' End
Tan Delta Peak
Temperature /°C
Dose /MGy
-20.0
-22.0
-24.0
-26.0
-28.0
-30.0
-32.0
-34.0
0
5
10
15
20
25

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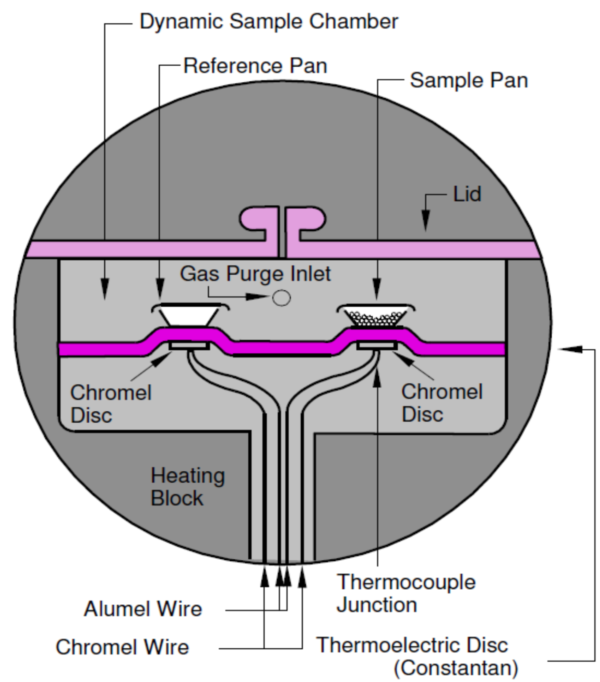
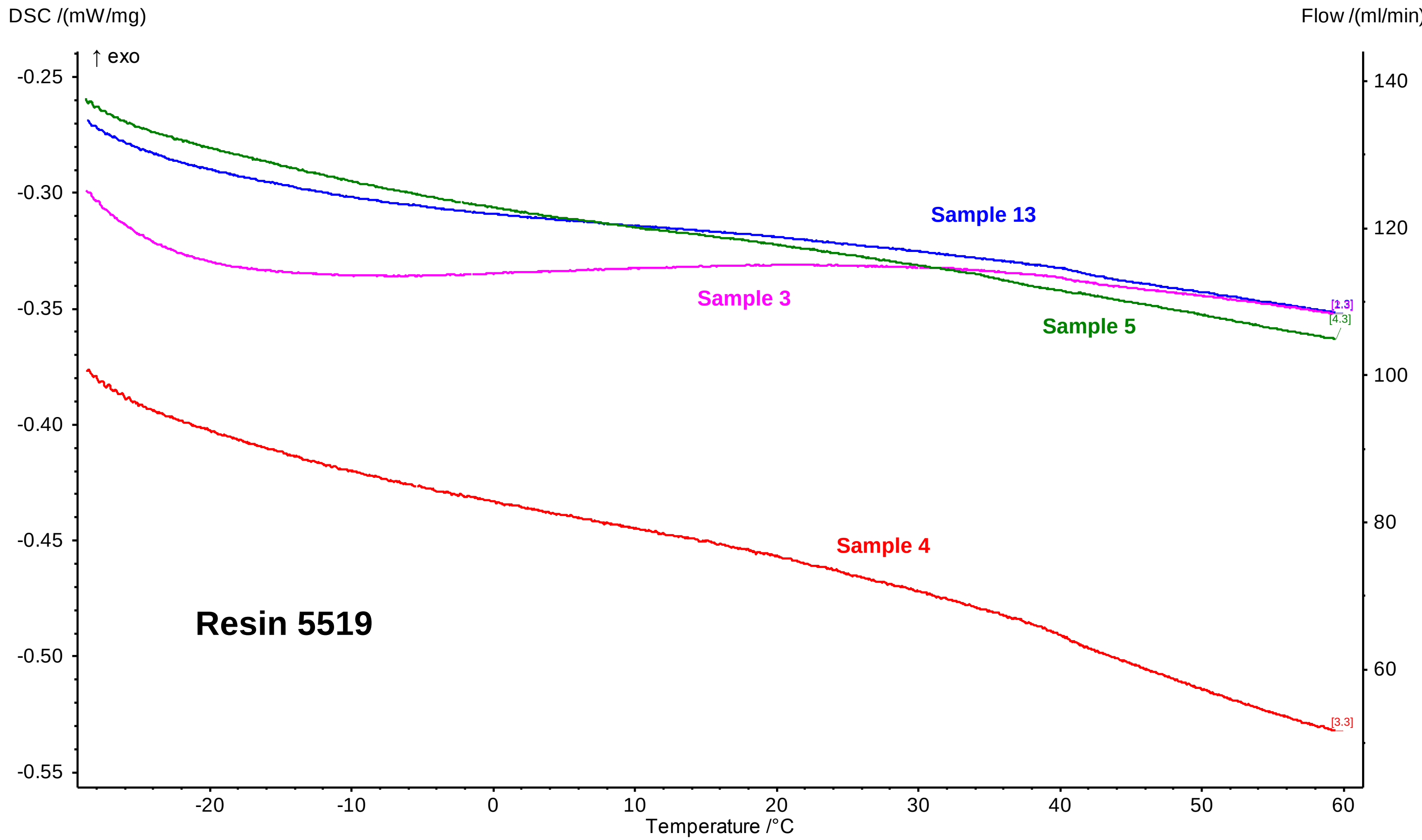
Differential Scanning Calorimetry

- Measures heat flow to/from sample as it is heated
- Measures exo- & endothermic changes in a material
- Glass transitions, relaxations, (melting points).



Differential Scanning Calorimetry



Summary

- UV-Vis Spectroscopy can be used to follow the radiation dose.
- FTIR can show some of the damage processes occurring in the resin.
- DMA shows increasing Tg with radiation dose.
- DSC is not useful, as materials are above Tg at room temperature.

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Future Studies

- Irradiated further samples for intermediate data points
- Effect of other radiation types
- Involvement of community
  - e.g. M. Koziel, Frankfurt
- Develop a flexible resin with lower cure temperature (use of accelerators).

AIDA Common database

AIDA: an FP7 EC project

The AIDA database aims to hold information on materials, detectors and electronic components tested after irradiation.

www.tinyurl.com/aidaimhotep

Written by Lesley Wright at (STFC-RAL) in Linux, Apache, MySQL, Perl

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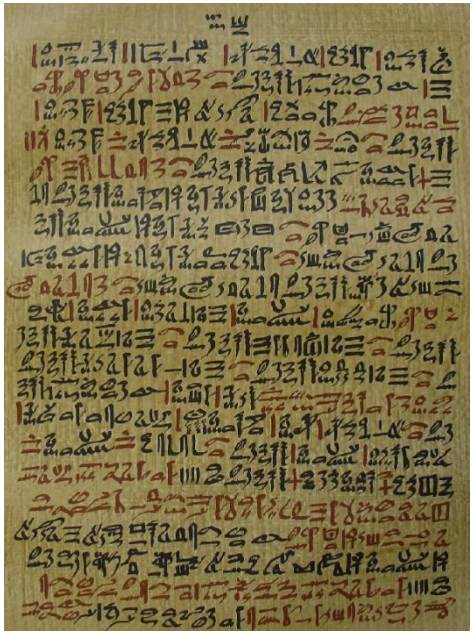
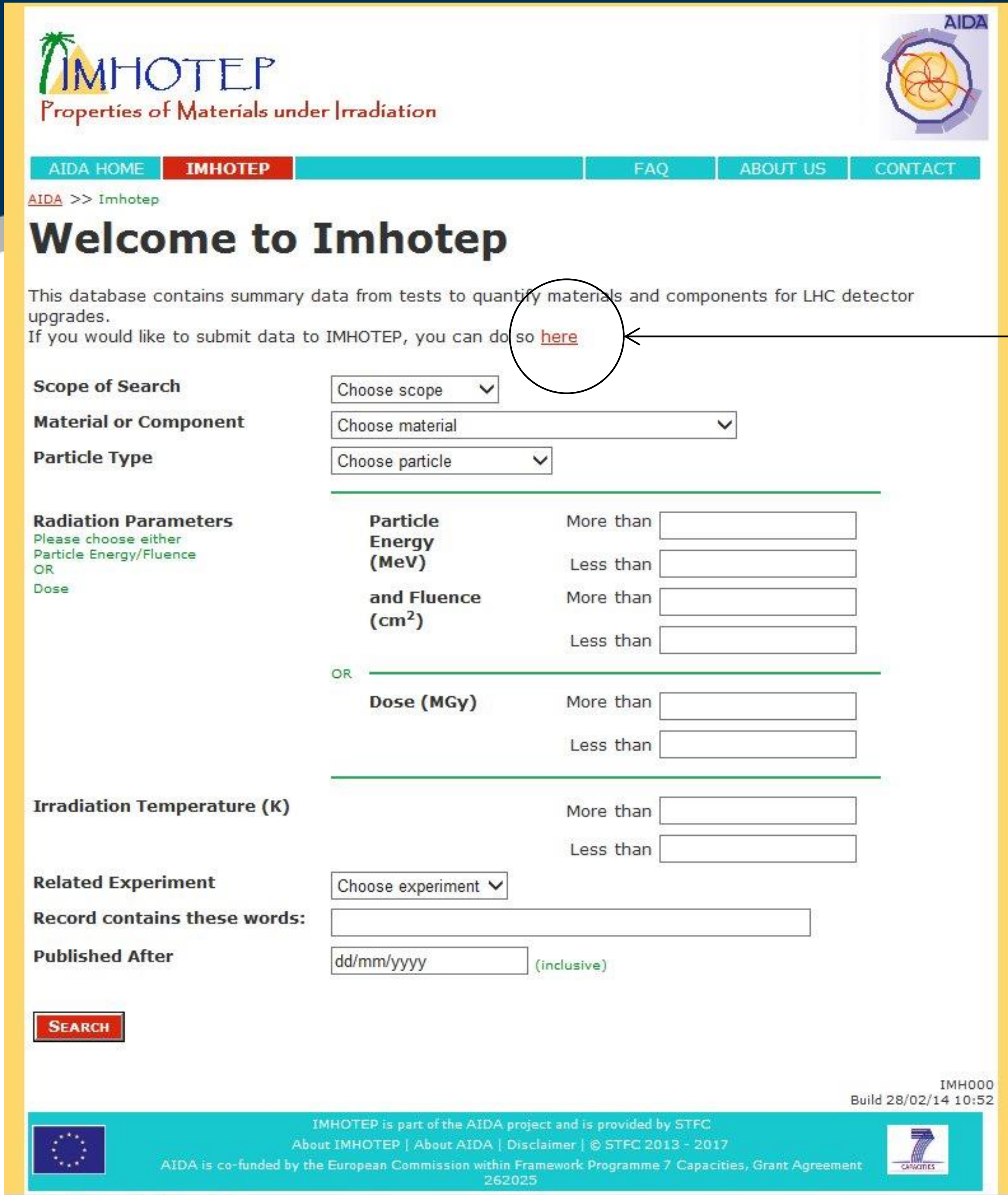
Imhotep

"Chancellor of the King of Egypt, Doctor, First in line after the King of Upper Egypt, Administrator of the Great Palace, Hereditary nobleman, High Priest of Heliopolis, Builder, Chief Carpenter, Chief Sculptor, and Maker of Vases in Chief."

- Third Dynasty (~2650-2600BC) polymath.
- Architect of the Pyramid of Djoser (Step Pyramid)
- Possible author of the "Edwin Smith Papyrus": a medical treatise.





IMHOTEP

Properties of Materials under Irradiation

AIDA

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AIDA >> Imhotep

Welcome to Imhotep

This database contains summary data from tests to quantify materials and components for LHC detector upgrades.
If you would like to submit data to IMHOTEP, you can do so here

Scope of Search Choose scope

Material or Component Choose material

Particle Type Choose particle

Radiation Parameters
Please choose either Particle Energy/Fluence
OR
Dose

Particle Energy (MeV)	More than	
	Less than	
and Fluence (cm^2)	More than	
	Less than	

OR

Dose (MGy)	More than	
	Less than	

Irradiation Temperature (K) More than / Less than

Related Experiment Choose experiment

Record contains these words:

Published After dd/mm/yyyy (inclusive)

SEARCH

IMH000
Build 28/02/14 10:52

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Link to data entry page

IMHOTEP
Properties of Materials under Irradiation

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AIDA >> Imhotep >> Submit Dataset

Imhotep: Suggest Datasheet - Suggest Dataset

If you would like to submit a datasheet or link to Imhotep, please use the form below

The Data Manager will review all submissions and decide whether to include them

Mandatory Field

Your Name

Your Affiliation
Organisation or similar affiliation

Your Email Address

Please confirm that you have the right to submit this datasheet or link
You own this datasheet, or have permission from the datasheet owner, or it is in the public domain

Scope of Search — Choose type; or enter new value

Sample Material or Identifier — Choose material; or enter new value

Grade

Sample Geometry or Test Type — Choose geometry; or enter new value

Sample Dimensions

Particle Type — Choose particle type; or enter new value

Particle Energy (MeV) — e.g. 20000

Particle Flux ($/cm^2/h$)
Use 5.23E12 notation
Enter values on separate rows, smallest to largest
- Discrete Values
- Range

Dose (MGy) — e.g. 0.038

OR — Either Dose or Fluence must be given

Particle Fluence (cm^2)
Use 5.23E12 notation
Enter values on separate rows, smallest to largest
- Discrete Values
- Range

Non-Ionizing Dose (MGy) e.g. 0.038

Irradiation Temperature (K)

Additional Irradiation Conditions

Time of measurement after irradiation (hours) Whole hours eg 2

Observables

Observable	Value
breakdown voltage kV/mm	
compressive strength MPa	
induced absorption vs wavelength and recovery	
light output changes	
radioactivation	
tensile strength MPa	
tensile yield strain %	
or enter new Observable	

Measurement Conditions

Related Experiment(s)

- ☐ ATLAS
- ☐ CMS
- ☐ ITER TF
- ☐ LHC

You can also enter a new experiment

Sample Properties Reference or Data Sheets

You can upload up to three datasheets below, give links to datasheets, or both

This system only accepts PDF files

Choose file to upload

Browse...

Location of datasheet on web

Publication Date

DD/MM/YYYY Enter date as dd/mm/yyyy eg 24/09/2013

Title of Publication

Reference DOI

Choose file to upload

Browse...

Location of datasheet on web

Publication Date

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Main Citations

Source of Information Choose Information Source

or enter new value

Comments

SUBMIT Submit Datasheets for Inclusion into Imhotep

RETURN TO SEARCH

IMH003
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IMHOTEP - Results

13 records have been found from your search. Click on a record to see its details.
You can also download this record set as a .csv file which can be viewed using MS Excel.

	Sample material / identifier	Geometry/Test Type	Observable	Dose (Mgy)	Datesheet Publication Date
Details	Cyanate ester 40% epoxy 60% S-glass composite	tensile ISO37:2005	tensile yield strain %	50.0000	20/03/2014
Details	Cyanate ester 40% epoxy 60% S-glass composite	ASTM D149-breakdown voltage	breakdown voltage kV/mm	50.0000	21/03/2014
Details	Cyanate ester 40% epoxy 60% S-glass composite	tensile ISO37:2005	tensile strength MPa	50.0000	20/03/2014
Details	Epoxy anhydride S-glass composite	ASTM D149-breakdown voltage	breakdown voltage kV/mm	50.0000	21/03/2014
Details	Epoxy anhydride S-glass composite	tensile ISO37:2005	tensile strength MPa	50.0000	20/03/2013
Details	Epoxy anhydride S-glass composite	tensile ISO37:2005	tensile yield strain %	50.0000	21/03/2014
Details	Epoxy RAL237	tensile ISO37:2005	tensile strength MPa	50.0000	21/03/2014
Details	Epoxy RAL237	tensile ISO37:2005	tensile yield strain %	50.0000	21/03/2014
Details	Epoxy RAL237	ASTM D149-breakdown voltage	breakdown voltage kV/mm	50.0000	21/03/2014
Details	Epoxy RAL237+41% dolomite filler	Compression	compressive strength MPa	107.0000	18/09/2012
Details	Epoxy RAL71 S-glass composite	ASTM D149-breakdown voltage	breakdown voltage kV/mm	50.0000	20/03/2013
Details	Epoxy RAL71 S-glass composite	tensile ISO37:2005	tensile yield strain %	50.0000	21/03/2014
Details	Epoxy RAL71 S-glass composite	tensile ISO37:2005	tensile strength MPa	50.0000	20/03/2013

DOWNLOAD this produces a .csv file that can be opened in Excel

RETURN TO SEARCH

Thank you

www.tinyurl.com/aidaimhotep

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Database hardware

Very robust system using 4 servers:

All data entry will be done on Lutra (backed up by Aonyx), and then exported nightly across the firewall to Tarka (backed up by Mijbil) on which the query system will run. Aonyx will be in a different building to Lutra (probably in the ATLAS centre at RAL).

The urls can be swapped between the pairs of machines to allow for power outages, patching and other similar occurrences; this process will be invisible to users.