



Low temperature DMA with liquid N₂: installation and initial results

Polymer laboratory meeting

David Mate Parragh, Christian Scheuerlein

11/04/2024

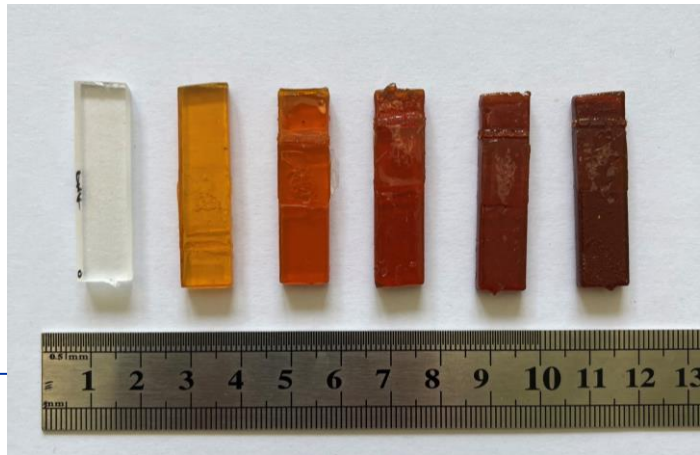
Installation for liquid N₂ DMA

With the help of Christian all the required equipment was installed for the liquid N₂ DMA (dewar, connectors, etc.)

Capabilities:

- DMA temperature sweep starting from -160 °C (or -150 °C) switching to using only compressed air at 50 °C (adjustable)
- Cool down from room temperature takes around 30-50 min
- 50 l liquid N₂ dewar sufficient for at least 2 DMA tests


First tests on PU SIKA before and after 10 MGy gamma irradiation

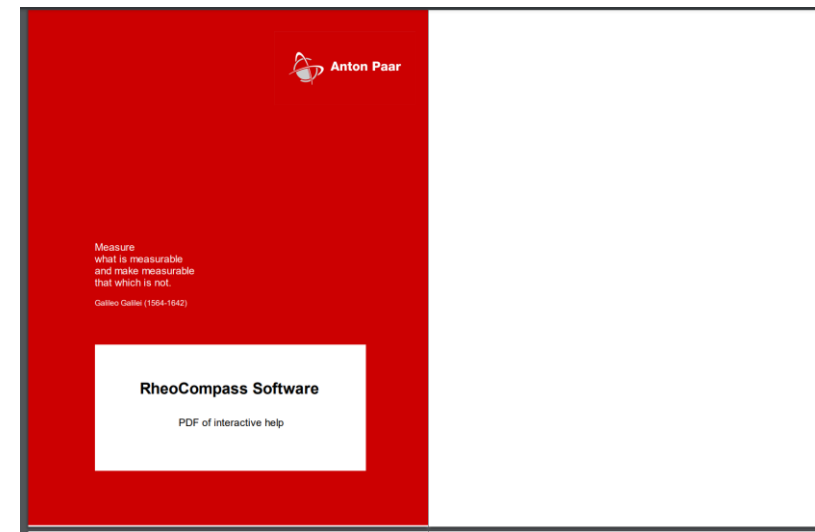


SOP and manual updated on EDMS

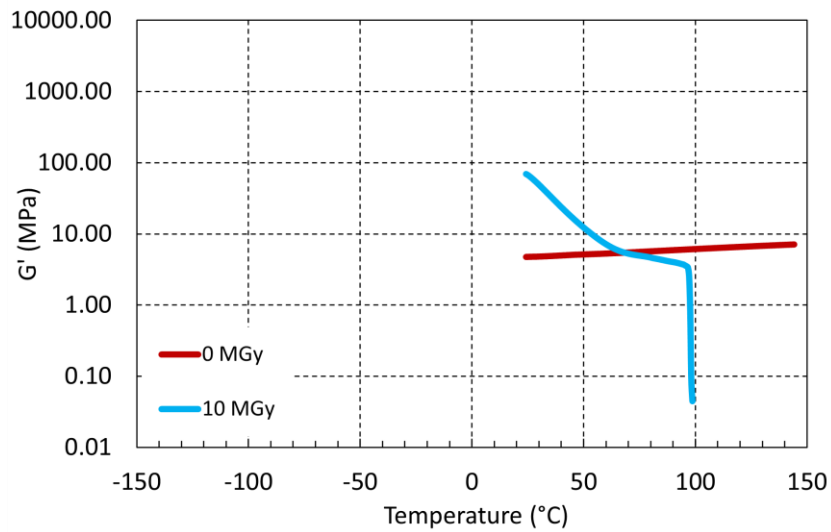
Standard operating procedure of DMA is updated to include the liquid N₂ operation mode as well, and the manual of the new updated software is uploaded to EDMS as well (RheoCompass 3.12)

- DMA temperature sweep in torsion mode with the Anton Paar MCR 702e <https://edms.cern.ch/document/3017673/1>
- RheoCompass 3.12 manual <https://edms.cern.ch/document/2887791/2>

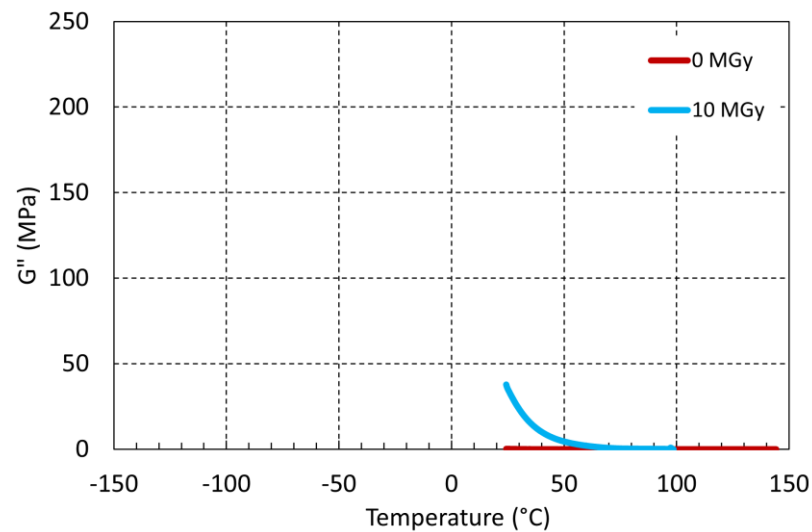
 Polymer Laboratory Laboratoire des polymères	N° EDMS 3017673
	Version : 01 Date : 12/01/2024 Page 1 / 7
Standard Operating Procedure	
DMA temperature sweep in torsion mode with the Anton Paar MCR 702e	



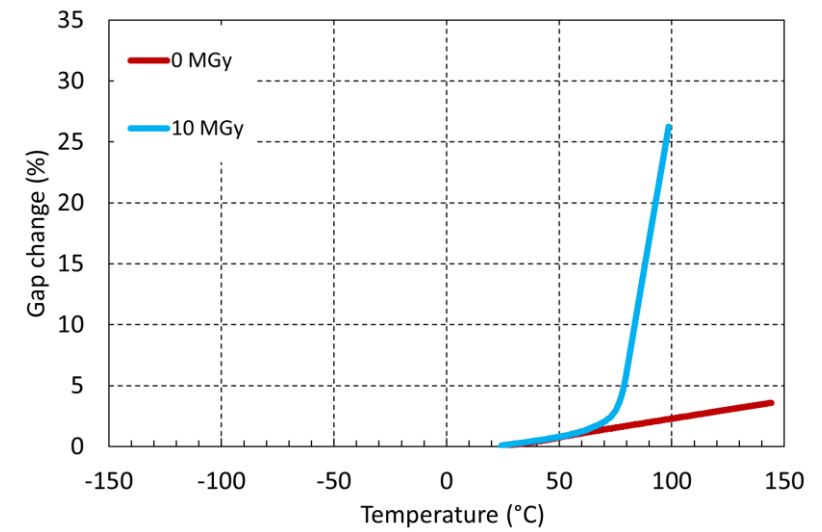
DMA temperature sweep of PU SIKA after gamma irradiation (Steris 2022)



Storage modulus $G'(T)$

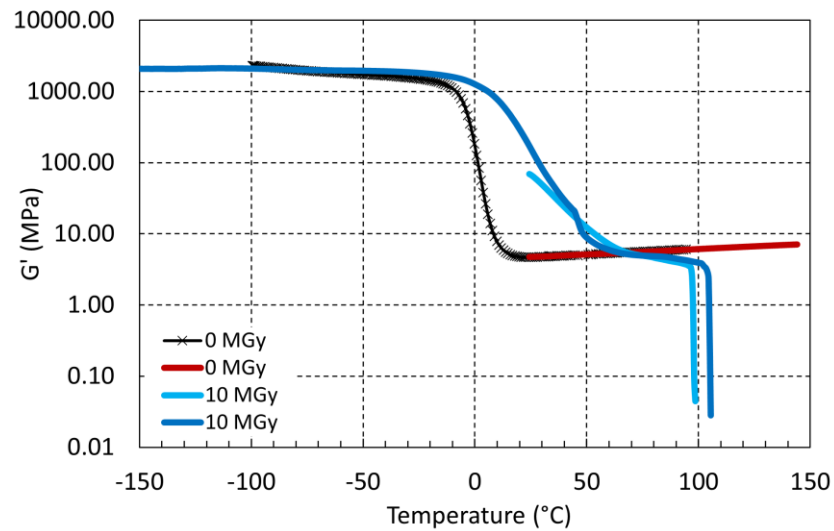


Loss modulus $G''(T)$

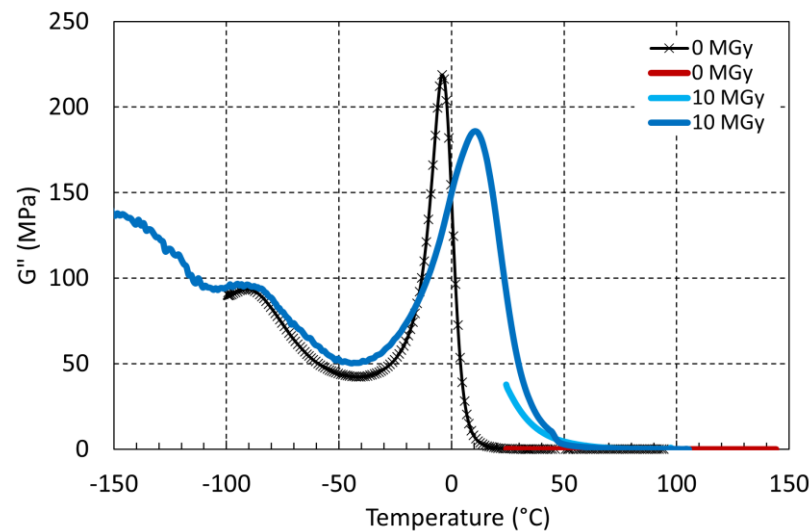


Thermal expansion $\Delta L_r(T)$

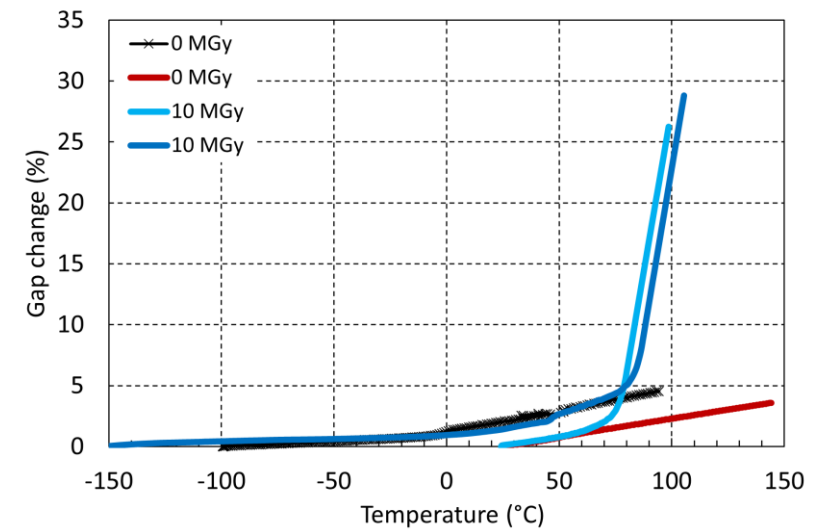
DMA temperature sweep of PU SIKA after gamma irradiation (Steris 2022)



Storage modulus $G'(T)$

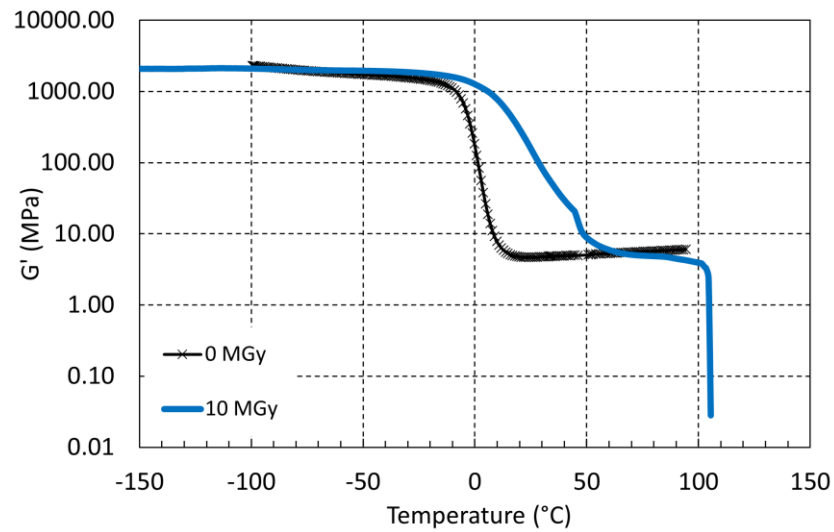


Loss modulus $G''(T)$

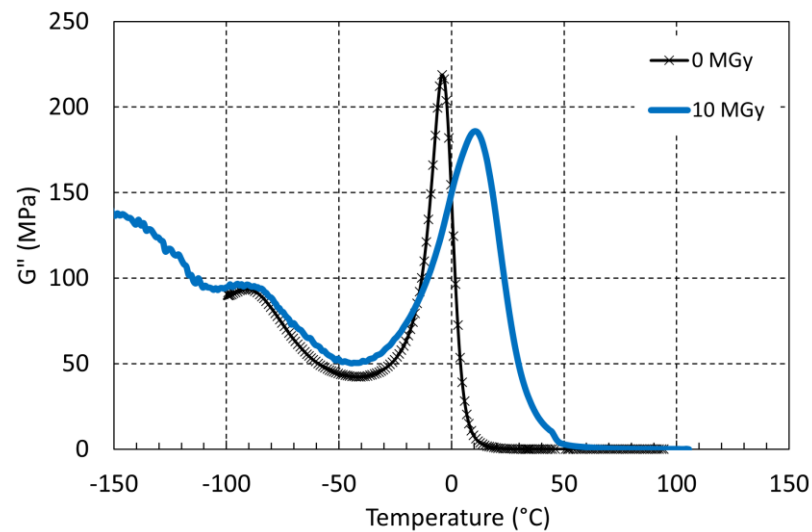


Thermal expansion $\Delta L_r(T)$

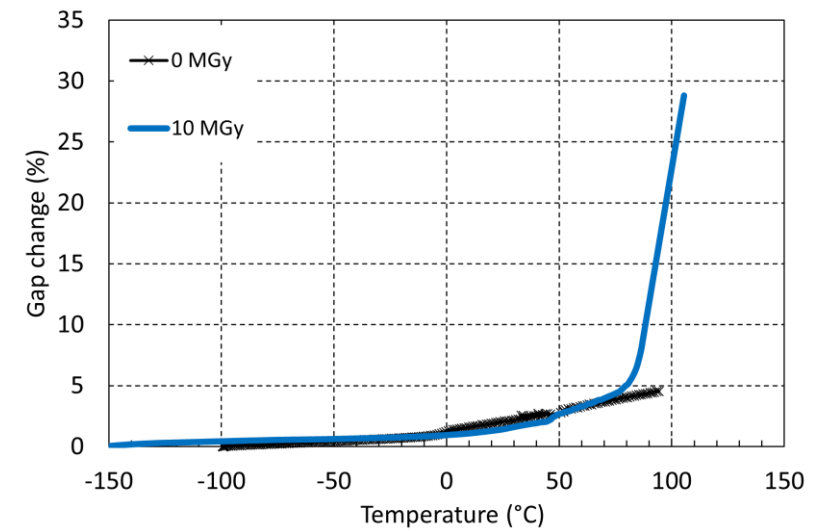
DMA temperature sweep of PU SIKA after gamma irradiation (Steris 2022)



Storage modulus $G'(T)$

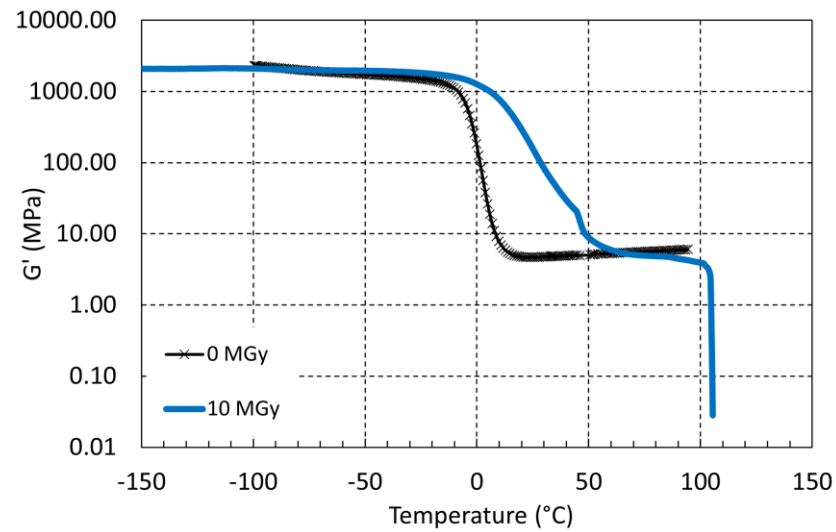


Loss modulus $G''(T)$

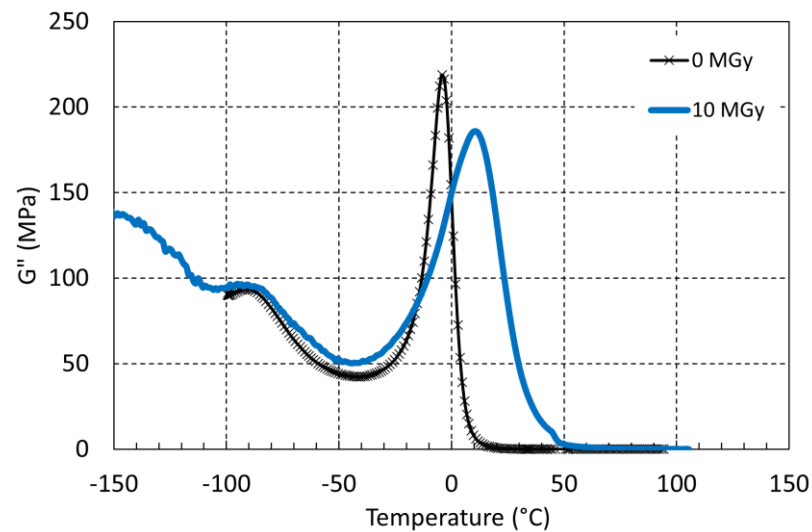


Thermal expansion $\Delta L_r(T)$

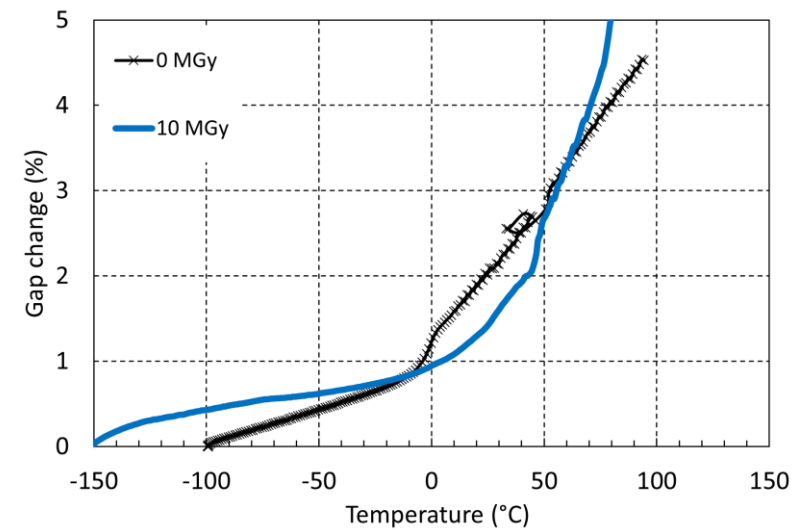
DMA temperature sweep of PU SIKA after gamma irradiation (Steris 2022)



Storage modulus $G'(T)$



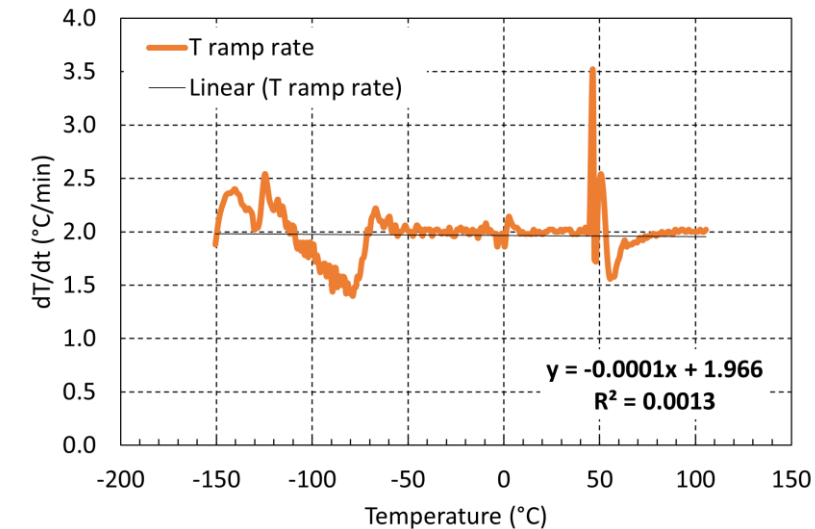
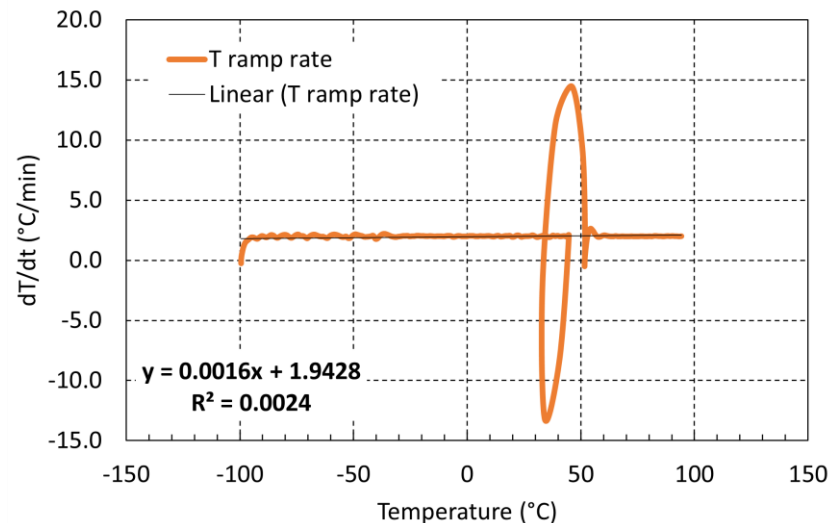
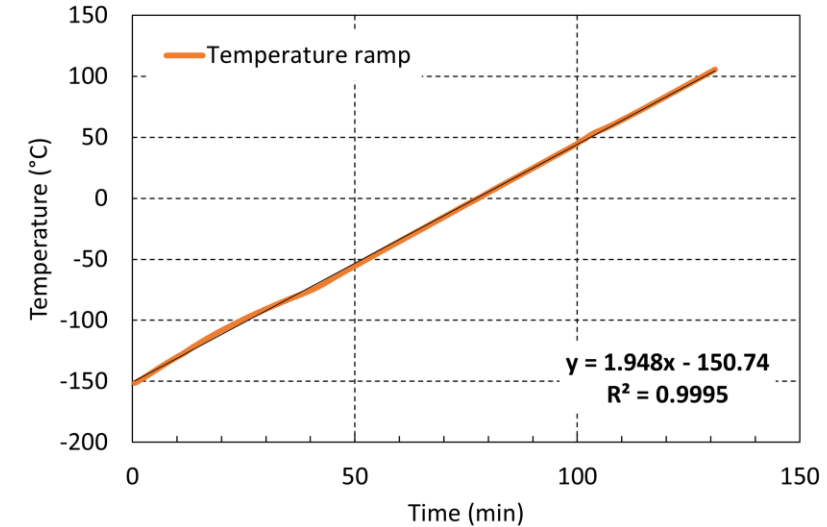
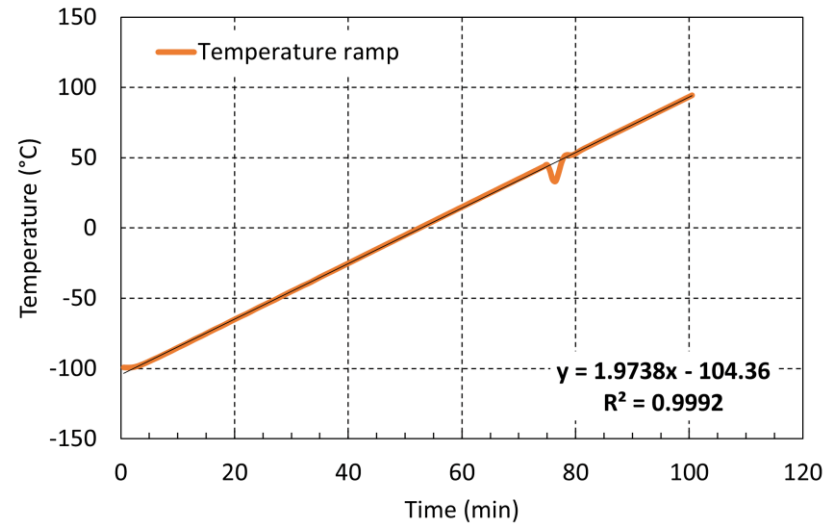
Loss modulus $G''(T)$



Thermal expansion $\Delta L_r(T)$

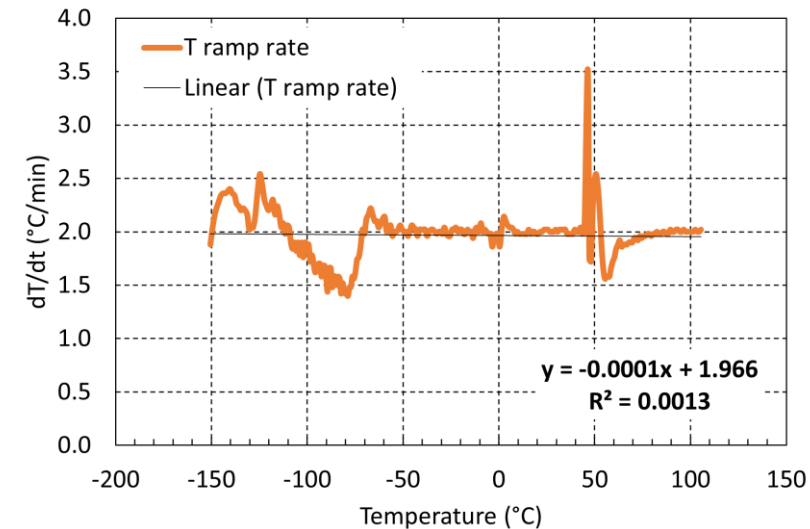
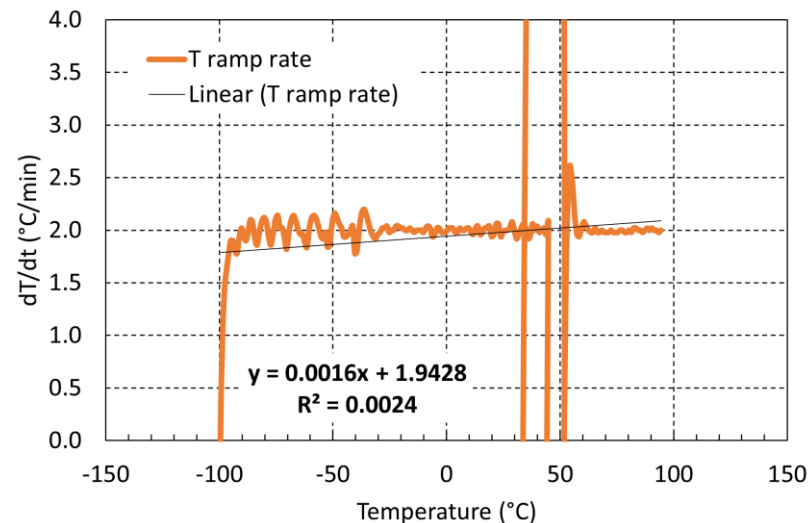
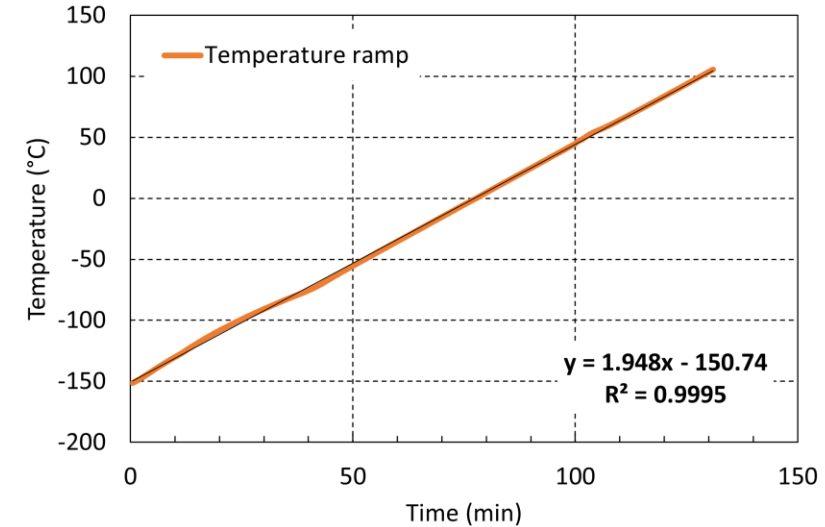
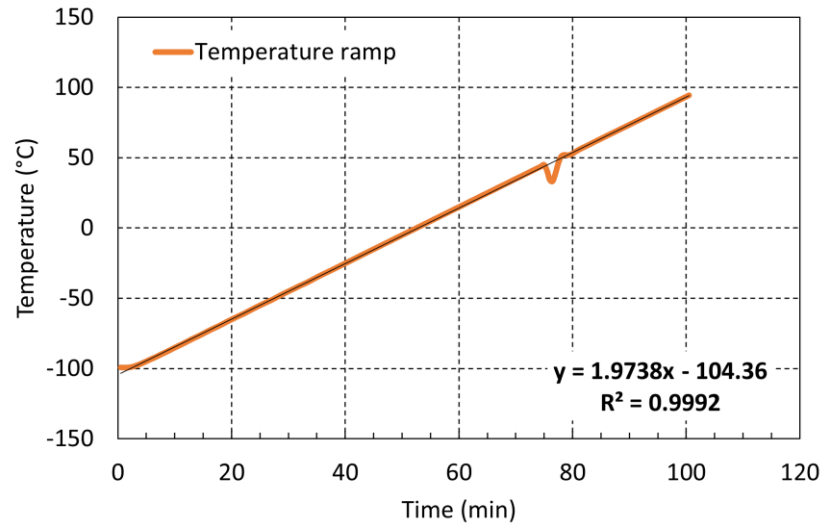
Temperature control

- Excellent temperature control during 1st test using a refilled (~50 l) dewar, with small instability between 45 and 50 °C as N₂ + air heating is switched to using only air
- Maintained temperature control during 2nd DMA test as well starting with a ~20 l dewar from -150 °C



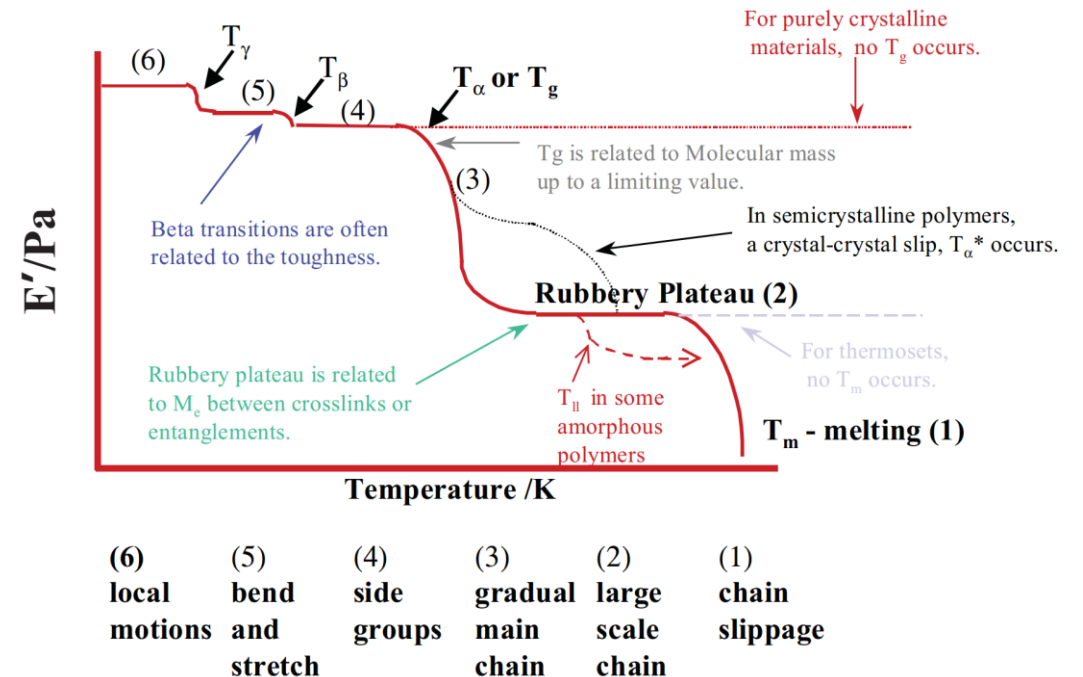
Temperature control

- Excellent temperature control during 1st test using a refilled (~50 l) dewar, with small instability between 45 and 50 °C as N₂ + air heating is switched to using only air
- Maintained temperature control during 2nd DMA test as well starting with a ~20 l dewar from -150 °C



Conclusion / outlook

- Liquid N₂ cooling installed for DMA and operates well for low temperature T sweeps
- T_g can now be determined of materials with below room temperature T_g
- Sub-T_g transitions can also be studied (e.g. beta-transition)
- ~2 measurements per week feasible with 50 l dewar



https://resources.perkinelmer.com/lab-solutions/resources/docs/app_thermaldynmechanalybasicspart2.pdf



home.cern