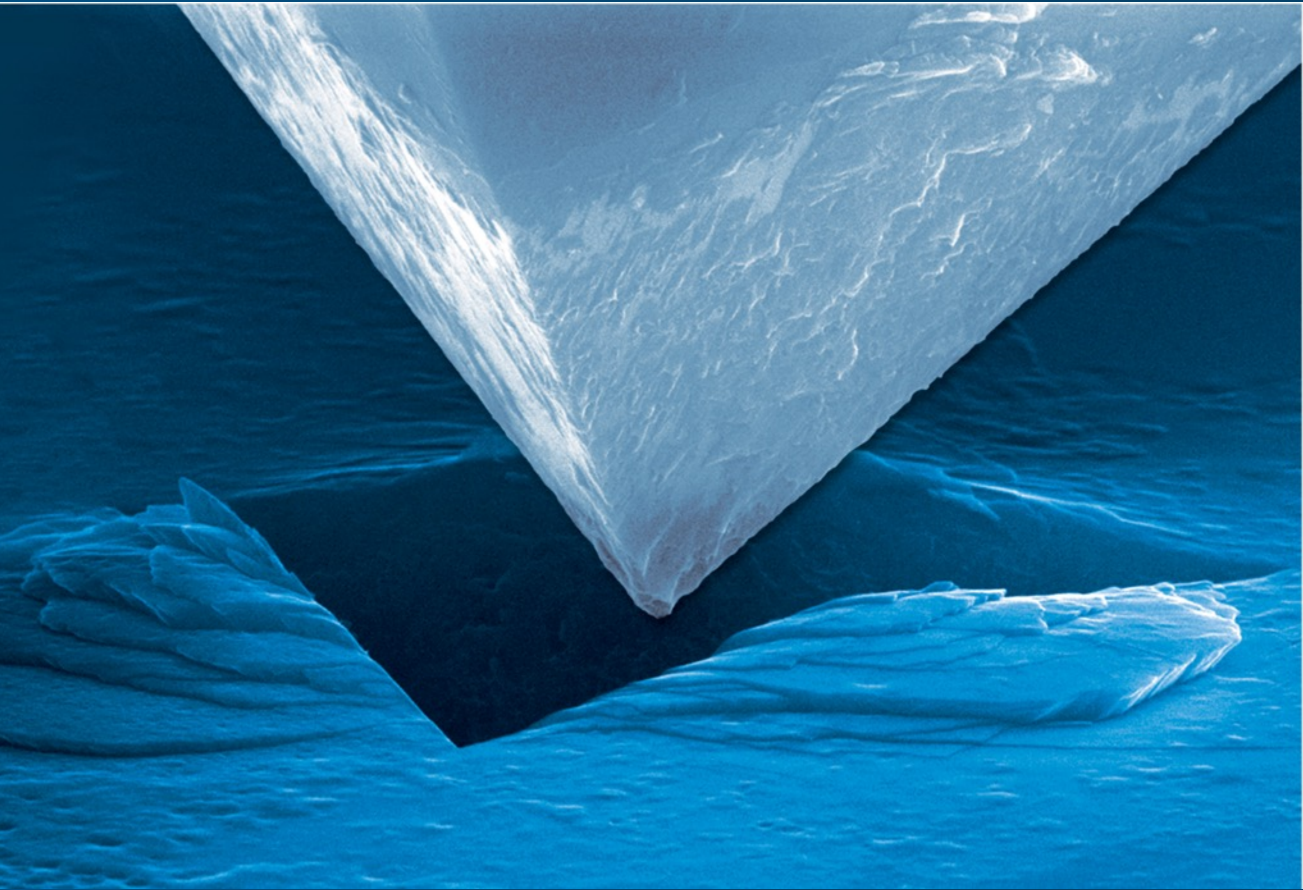
Nan-Scale Mechanical Measurements at Environmental Controls

16 June, 2023

Outlines:

- Applications at High Temperature
- Applications at Low Temperature
- Applications at Humidity Levels



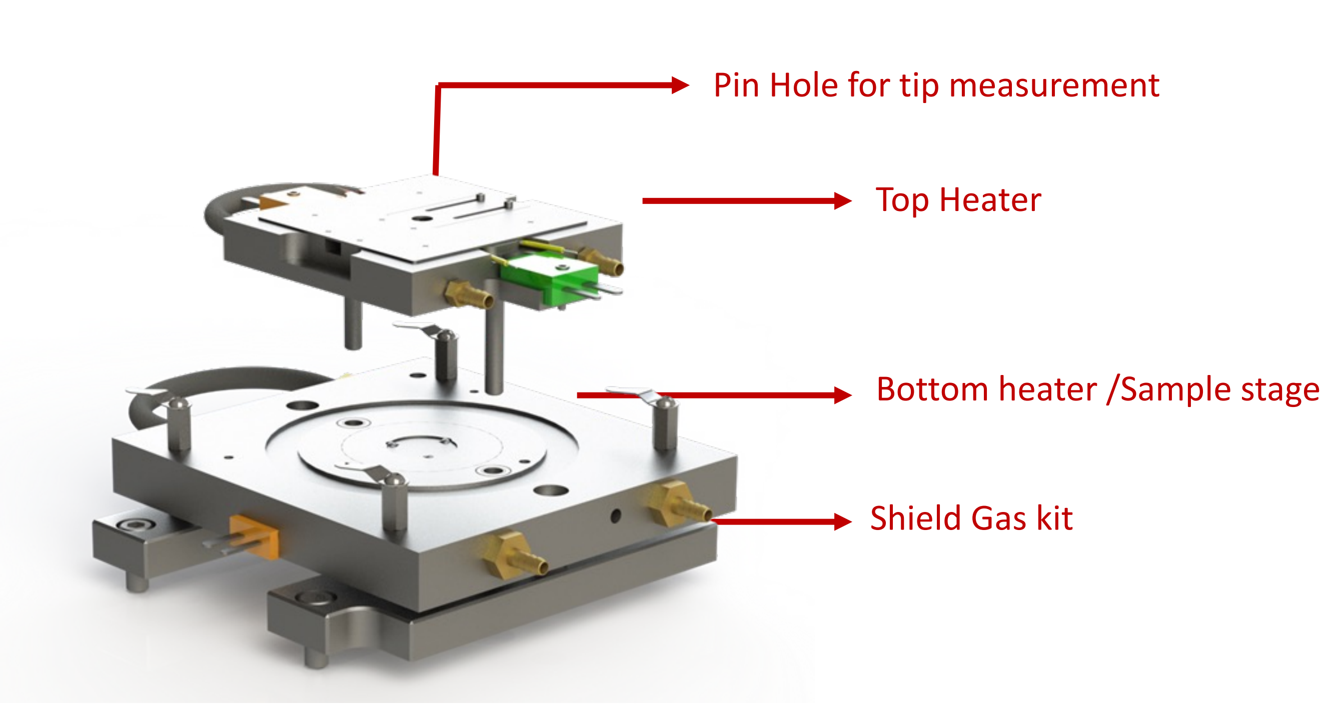
In-Operanto Environmental Chamber

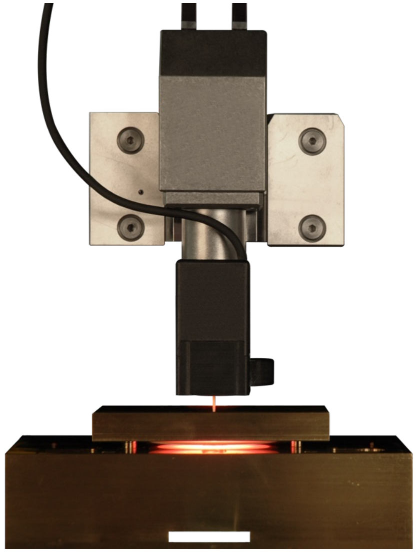
xSol® - Heating, Cooling, Humidity

Heating: 800°C • Cooling: -120°C • Humidity: 10-75% RH

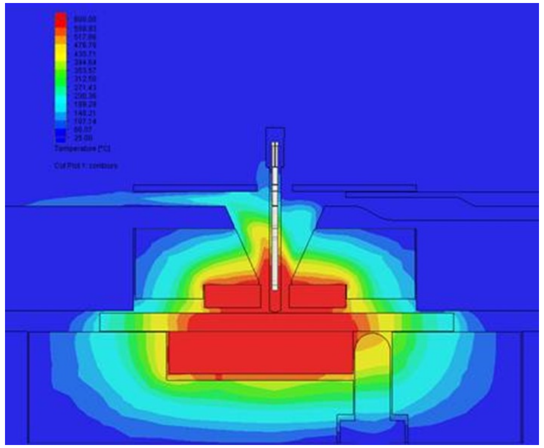
Applications at High Temperature

Controlled Atmosphere Systems

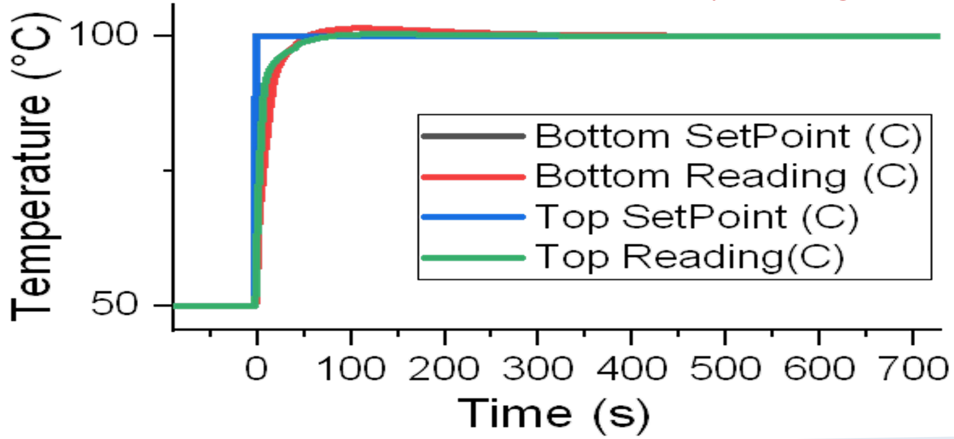




Heating Chamber



Passive Tip heating



xSol® - Heating, Cooling, Humidity

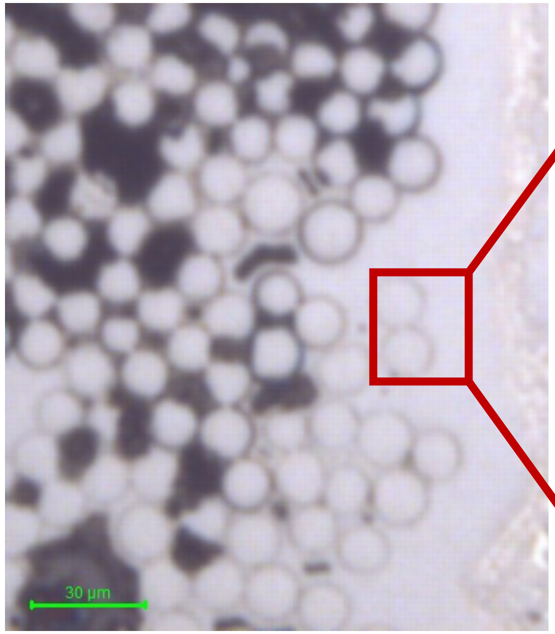
Heating: 800°C • Cooling: -120°C • Humidity: 90% RH

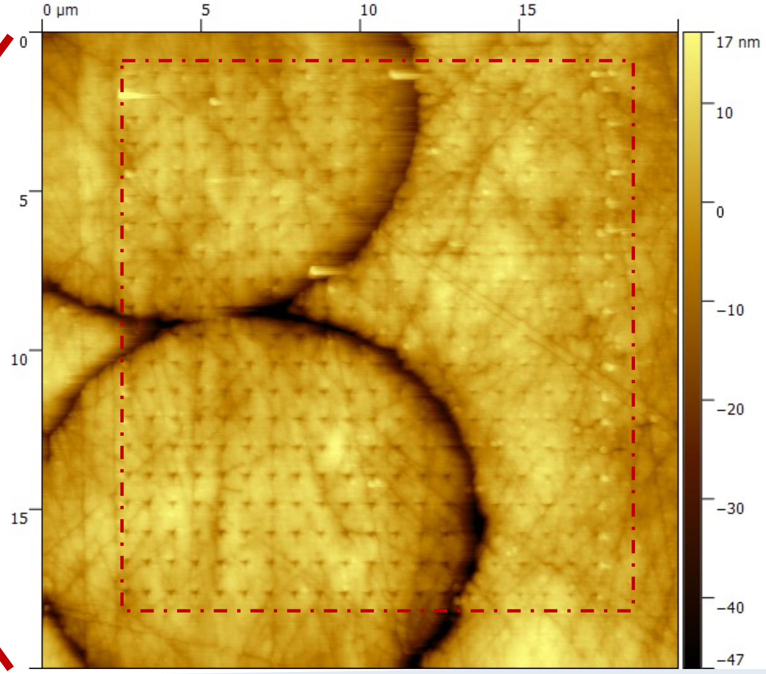
Application Case of XPM: Fiber-Matrix Composite

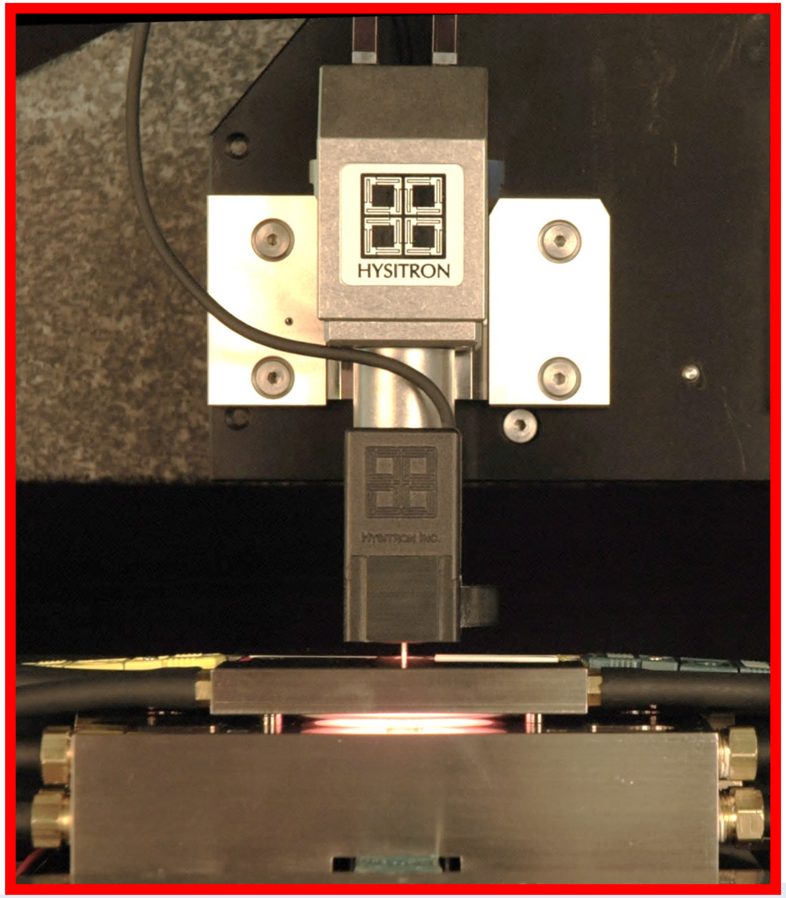
XPM for small size components at temperature levels

SILICON CARBIDE FIBER-MATRIX SAMPLE TESTED AT 400°C AND 800°C USING XPM HIGH THROUGHPUT INDENTATION

20X20 GRID PERFORMED IN 100 SECONDS

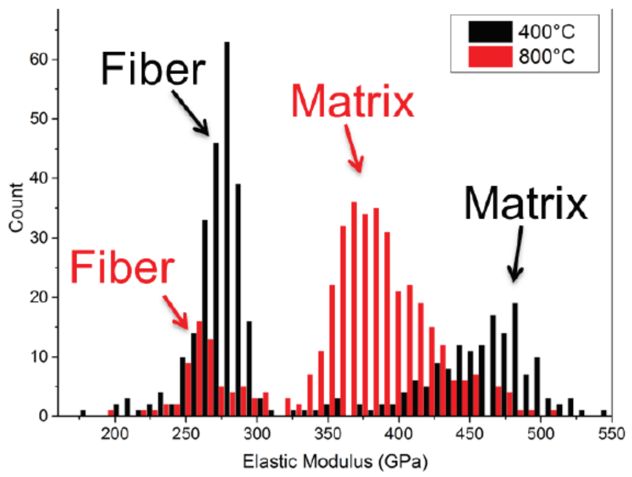




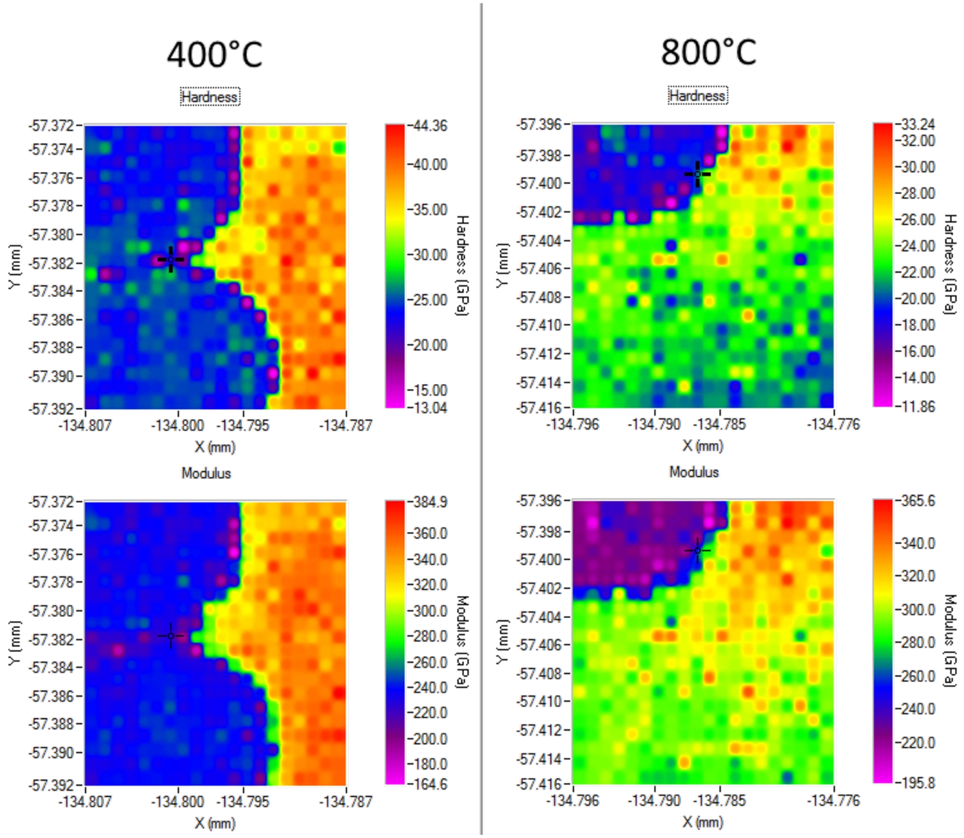


Application Case of XPM: Fiber-Matrix Composite

XPM for small size components at temperature levels



Histograms of silicon carbide fibre-matrix composite hardness and elastic modulus results obtained from XPM indentation testing at 400°C and 800°C

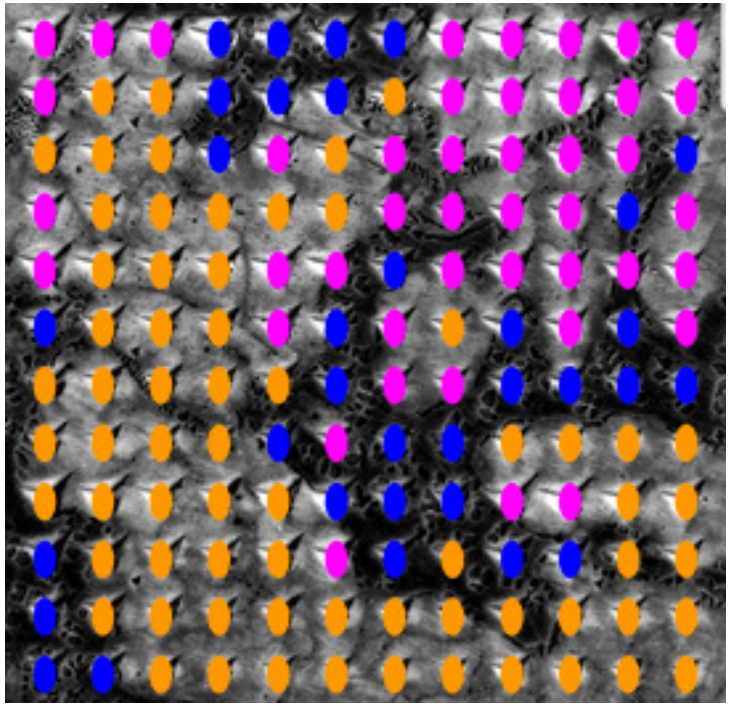


Fiber hardness and modulus, while lower, are also retained better at high temperature

Three Cluster Data at Temperature Levels

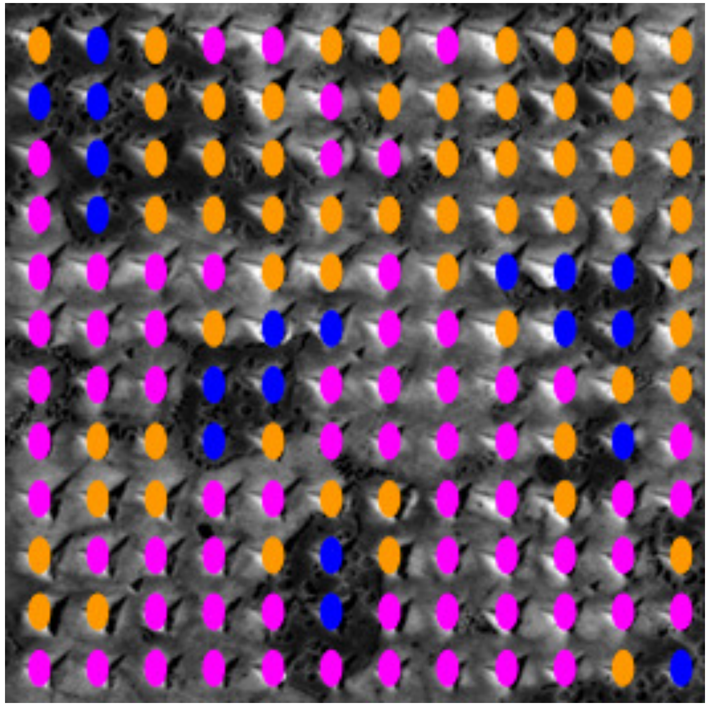
72% data usage: cluster 1 = Interface

RT



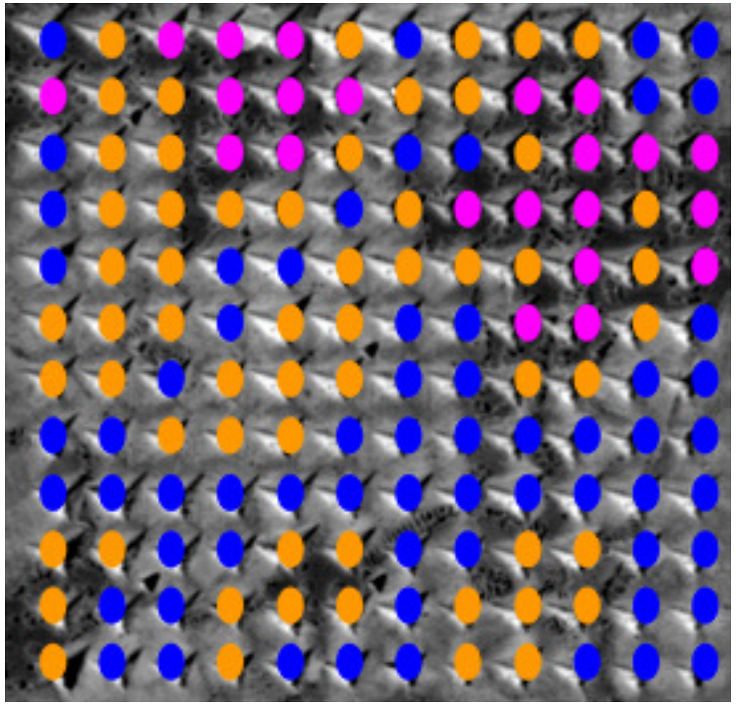
FCC: 4.0± 0.4 GPa
BCC: 6.1± 0.5 GPa

300°C



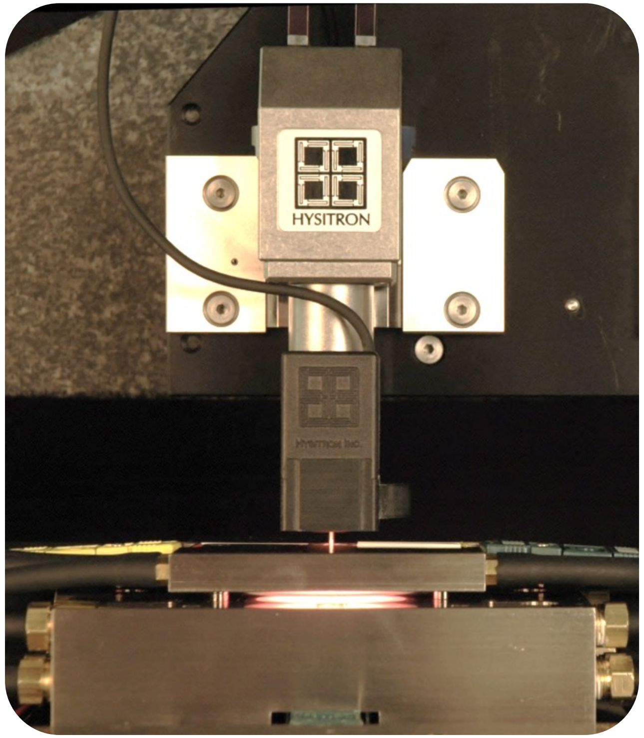
FCC: 3.6± 0.3 GPa
BCC: 5.6± 0.5 GPa

400°C

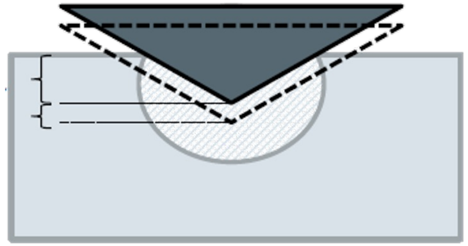


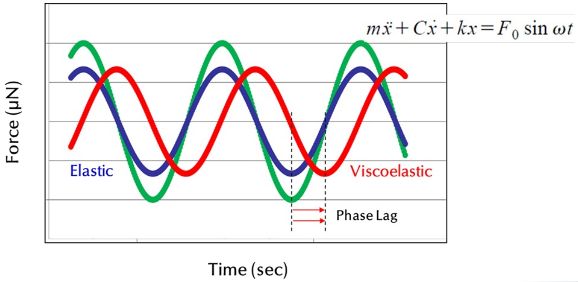
FCC: 3.6± 0.2 GPa
BCC: 5.5± 0.4 GPa

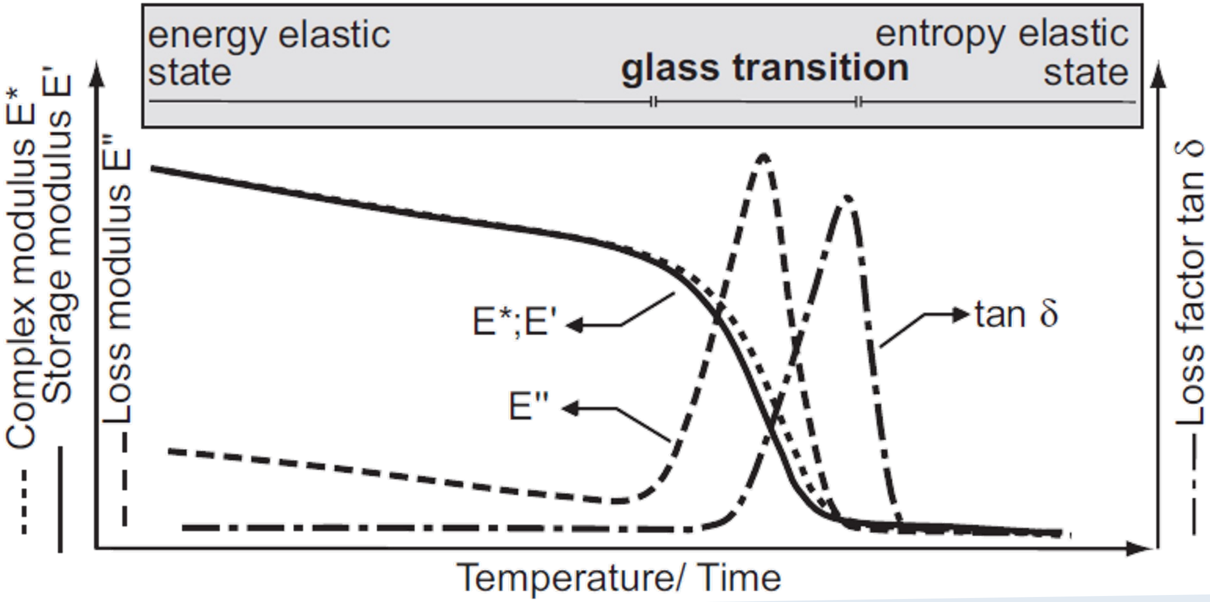
Technique for Glass Transition Temperature Measurements



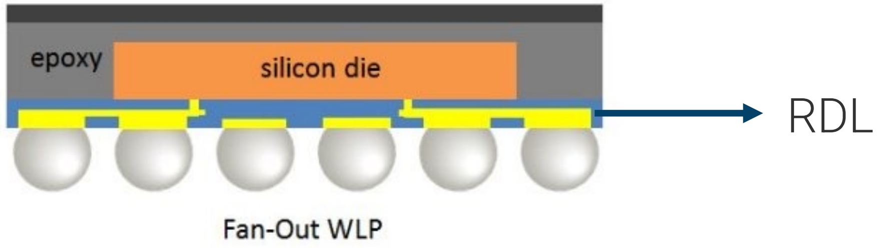
- Using automatically continuous DMA tests for Tg measurements is convenient for users.
- Compared to the rules using storage modulus, it is easy to apply the rule of tan-delta peak for determination of Tg.
- Practically, Tg determined by tan-delta peak is the end of glass transition.

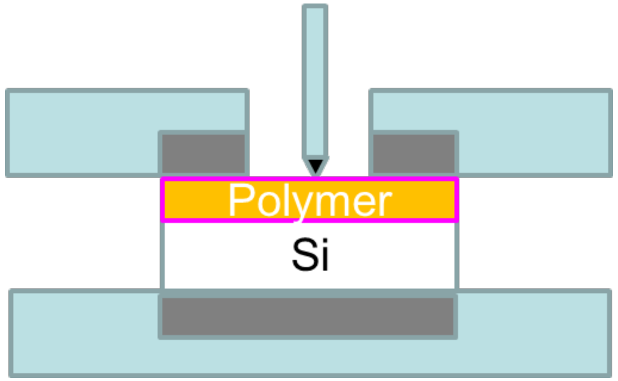




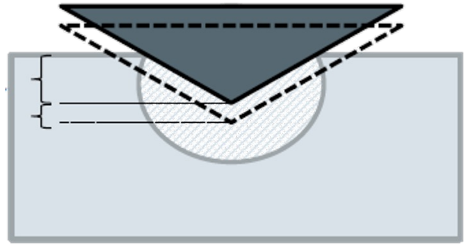


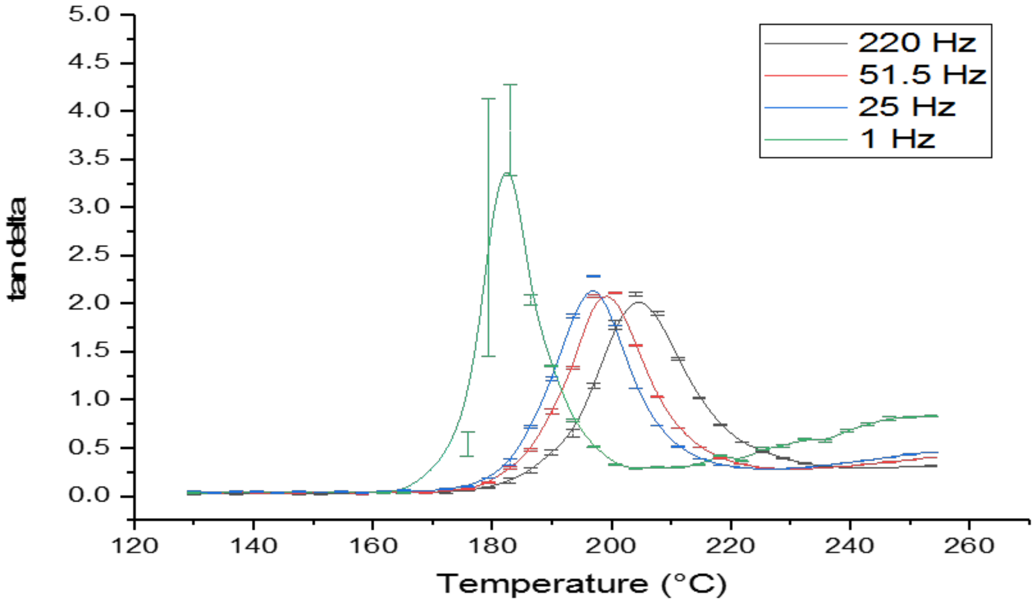
Oscillation Frequency Effect on Tg Measurements

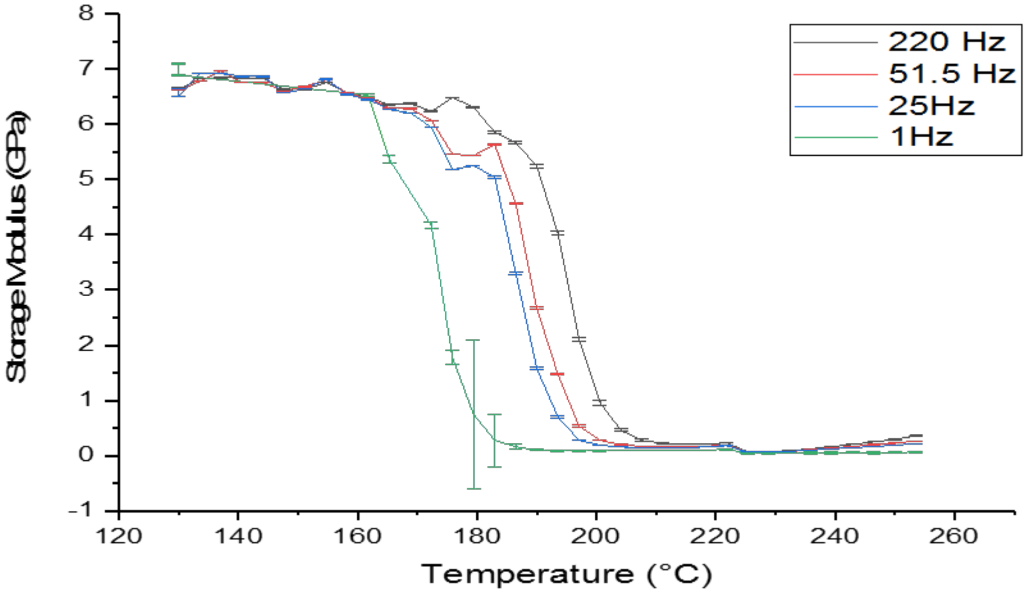




Nano-DMA



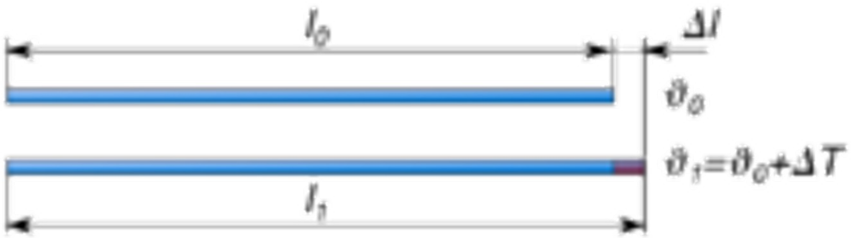




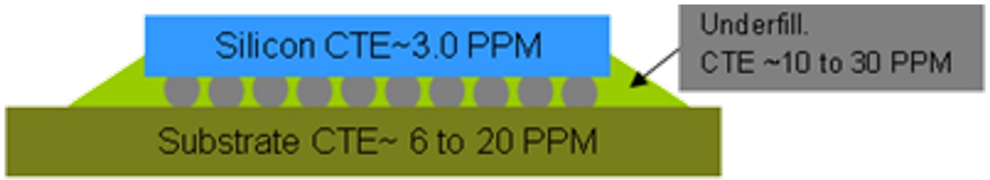
Overview of Linear Coefficient of Thermal Expansion

Material	CTE (ppm/°C)
AlAs	4.9
AlP	4.5
Alumina	6-7
AsSb	4
Copper	16.7
Cu/I/Cu	8.4
Cu/Mo/Cu	6
Cu/Mo-Cu/Cu	6-10
E-glass	54
Epoxy	55
Fused Silica	0.55
Gallium Arsenide (GaAs)	6.86
GaP	4.5
GaSb	7.75
Germanium (Ge)	5.8

Material	CTE (ppm/°C)
InAs	4.52
InP	4.75
InSb	5.37
Invar	1.3
Kovar	5.9
Molybdenum	7.0-7.1
Polymers	50-200
S-glass	16
Silicon	2.6
Silicon Nitride (Si_3N_4)	3.2
Silicone resins	30-300
Tin-Lead Solder	27
Titanium	9.5
Tungsten	5.7-8.3



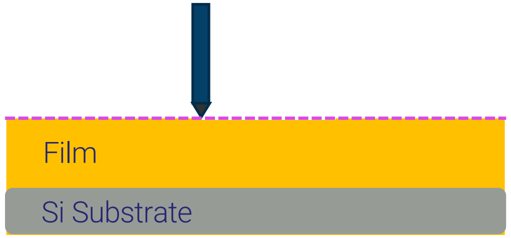
$$\frac{\Delta L}{L} = \alpha_L \Delta T$$



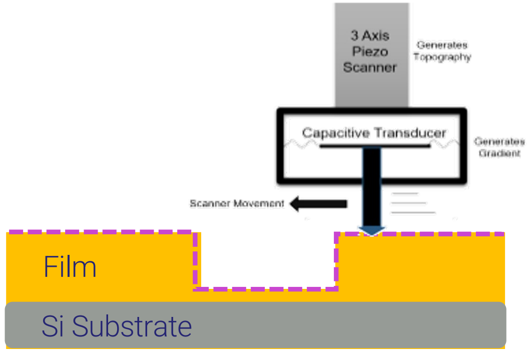
Solution for Thin Film's CTE Measurements

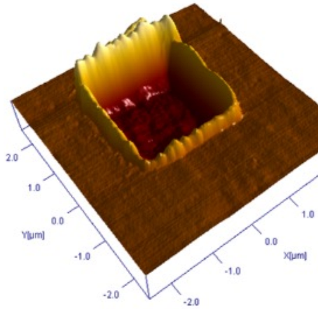
SPM Method

1. Nano Wear on the surface

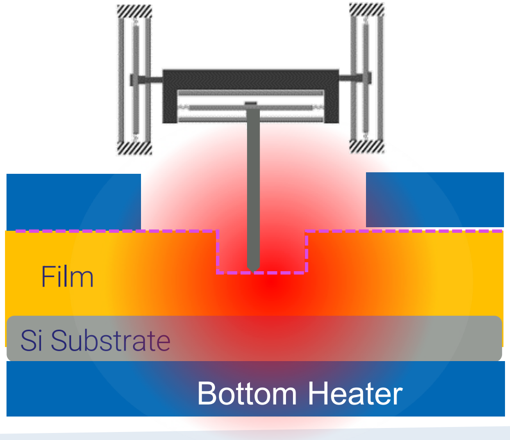


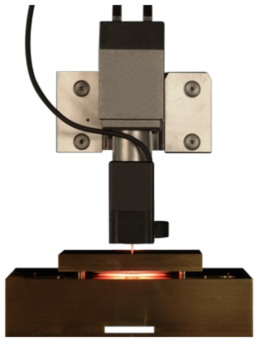
2. SPM to measure the hole depth





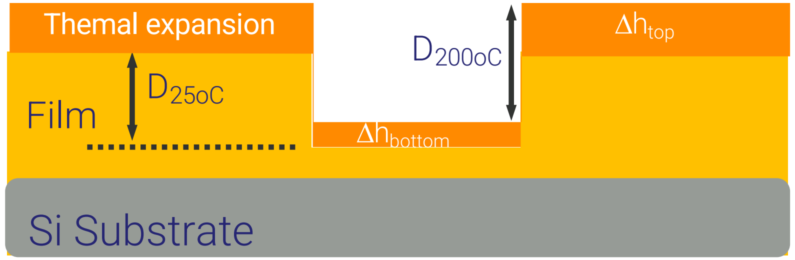
3. Heating the sample & SPM





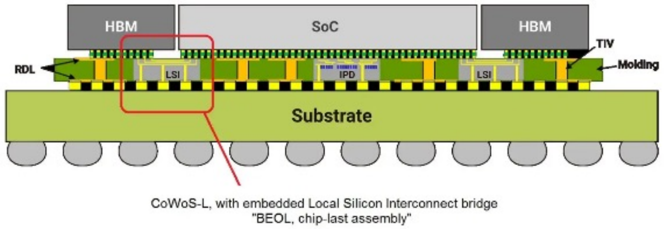
Hysitron xSol stage

4. SPM to measure the hole depth Changing

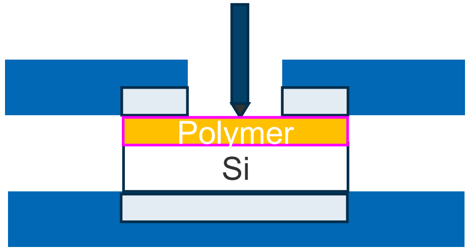


Thin PBO Film’s CTE Measurements

SPM Method

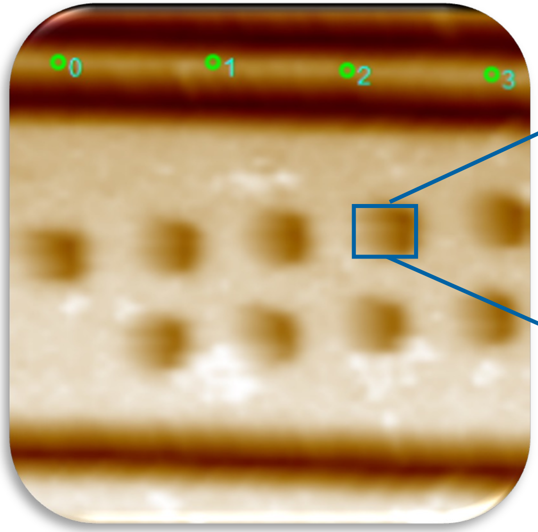


https://semiwiki.com/

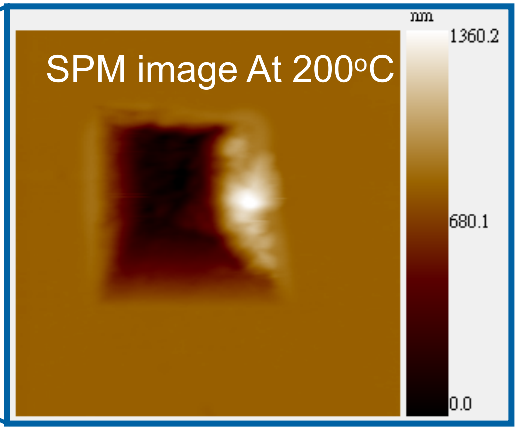


Advantages:

- Direct measurements
- High spatial resolution



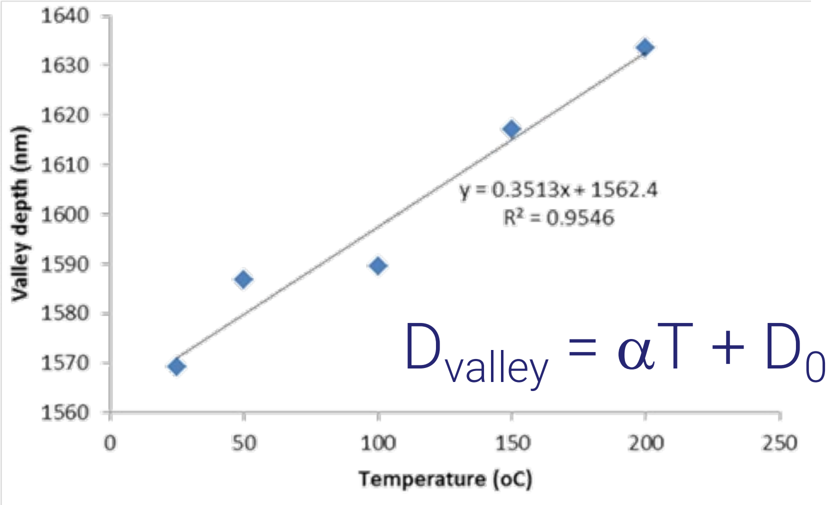
Post-wear images of polymer film



Whole Image Statistics

Projected Area = 400 瘞^2
RMS Roughness (Rq) = 179.149 nm
Average Roughness (Ra) = 107.798 nm
Mean Height = -52.4029 nm
Max Height = 580.534 nm
Min Height = -847.151 nm
Peak-to-Valley = 1427.68 nm

Valley depth increasing from 838.3nm to 847.2nm form 25oC – 200 oC

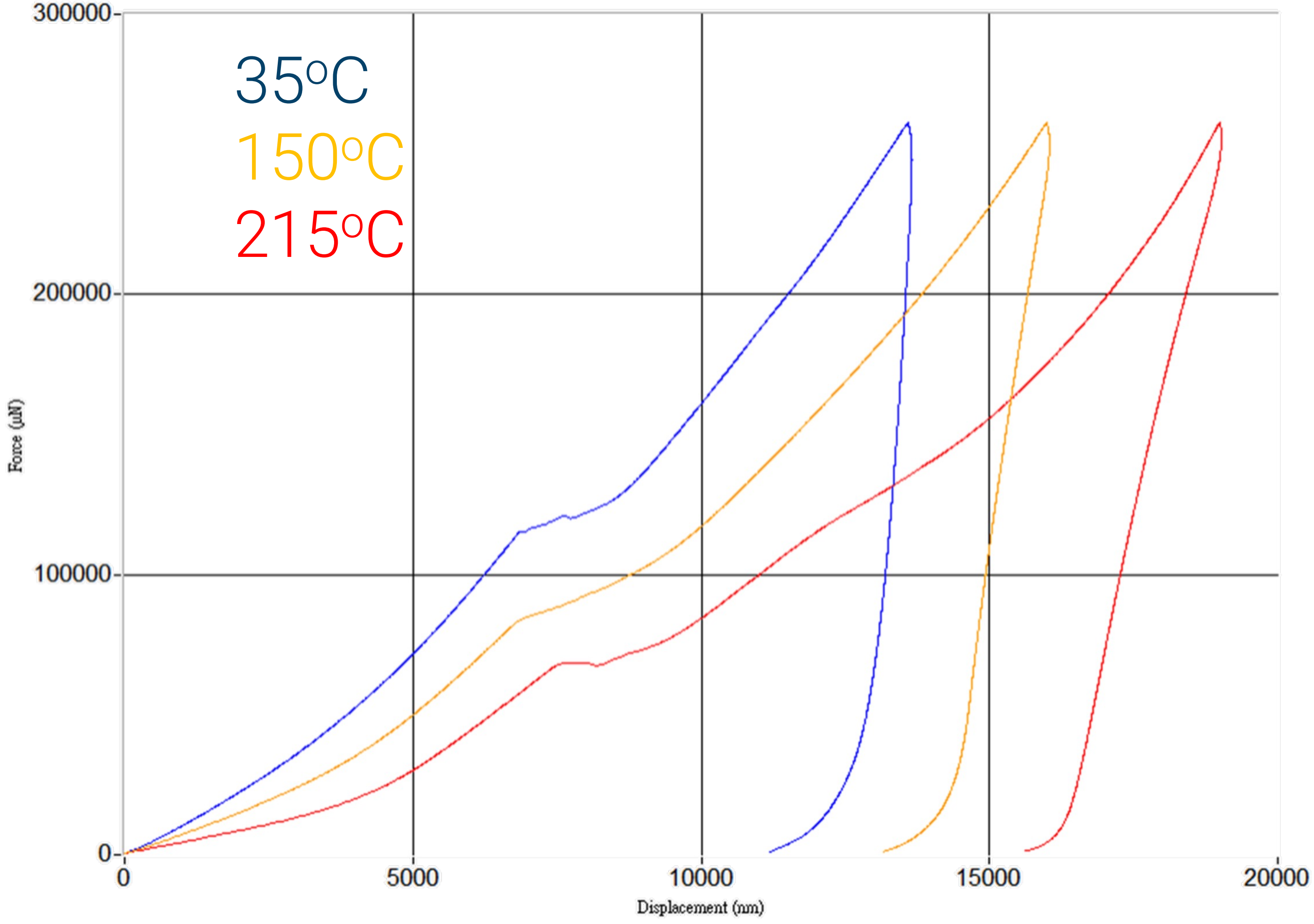


$$D_{valley} = \alpha T + D_0$$

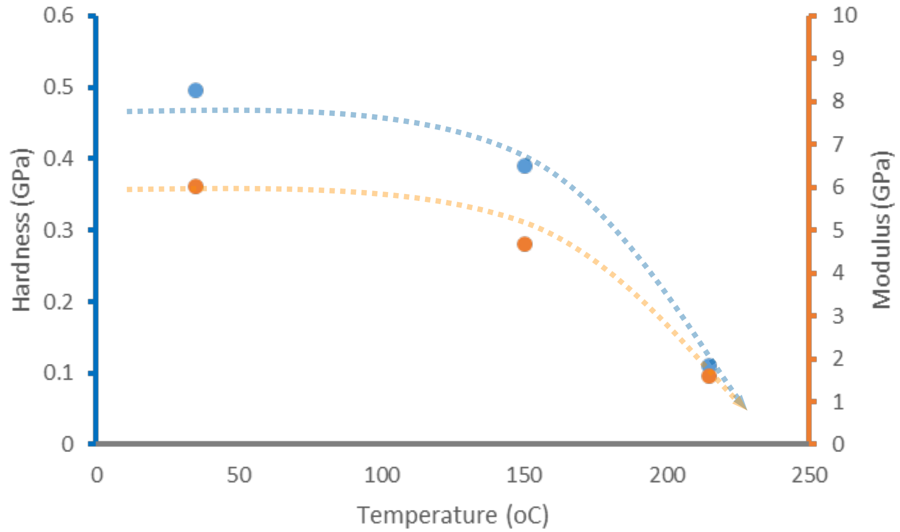
$$CTE = \alpha / D_0 = 0.3513(nm/^{o}C)/1562.4(nm) = 224.8ppm/^{o}C$$

High Load Indentations at Elevated Temperature

Polymer Film on Copper

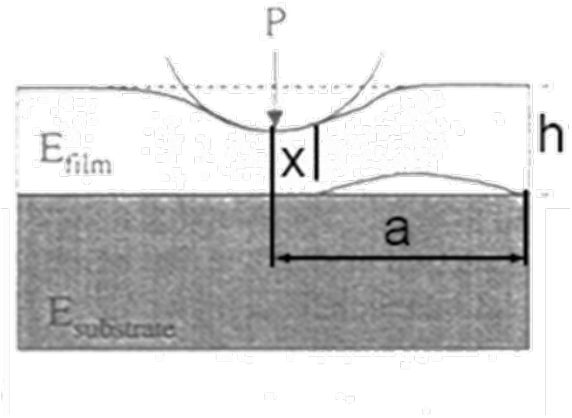


T (°C)	L_{crit} (uN)	H (GPa)	E (GPa)
35	115218.2	0.4963	6.02
150	84091.46	0.3906	4.69
215	68276.05	0.1101	1.59



Adhesion Work Calculation at Temperature Levels

Polymer Film on Copper



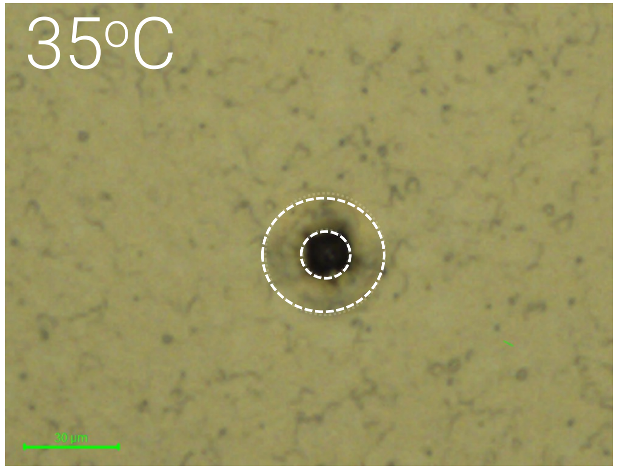
Developed by Rosenfeld et al. (1990)

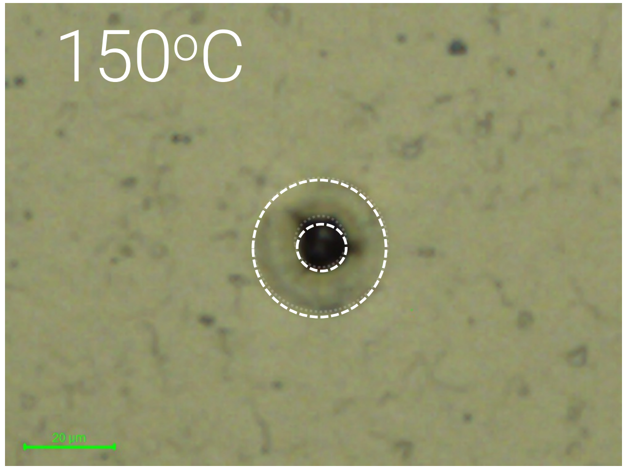
Film hardness — Film thickness

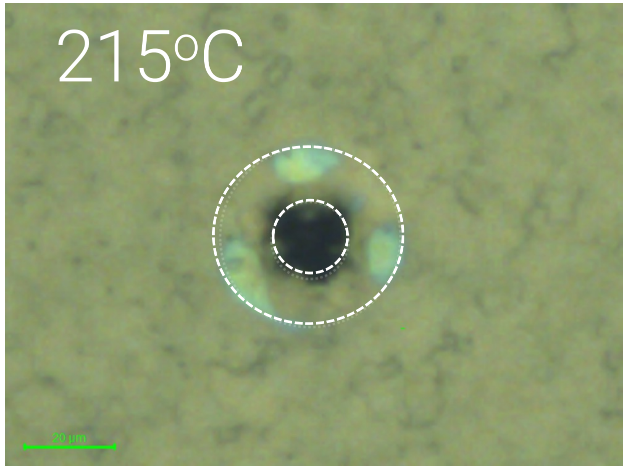
$$G = \frac{0.627 H^2 h (1 - \nu_f^2)}{E_f} \left(\frac{1}{1 + \nu_f + (a/x)^2 (1 - \nu_f)} \right)^2$$

Delaminated radius — Contact radius

3D Omniprobe
2N ,10 N with 80 um



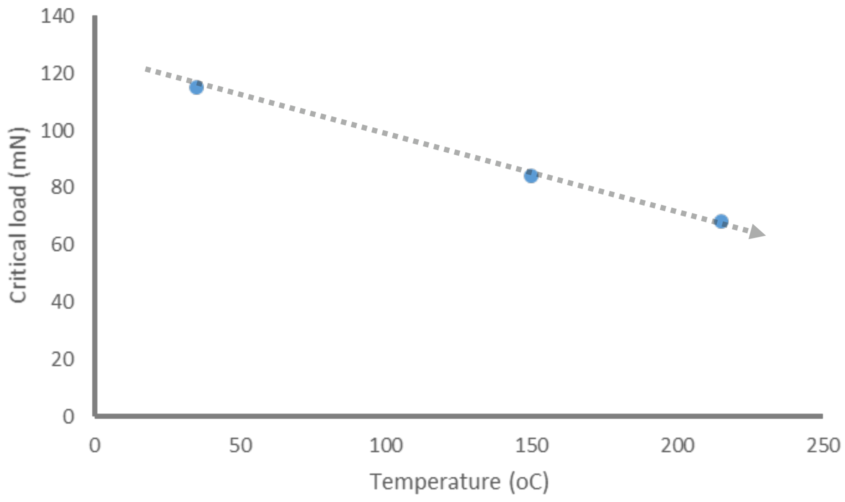


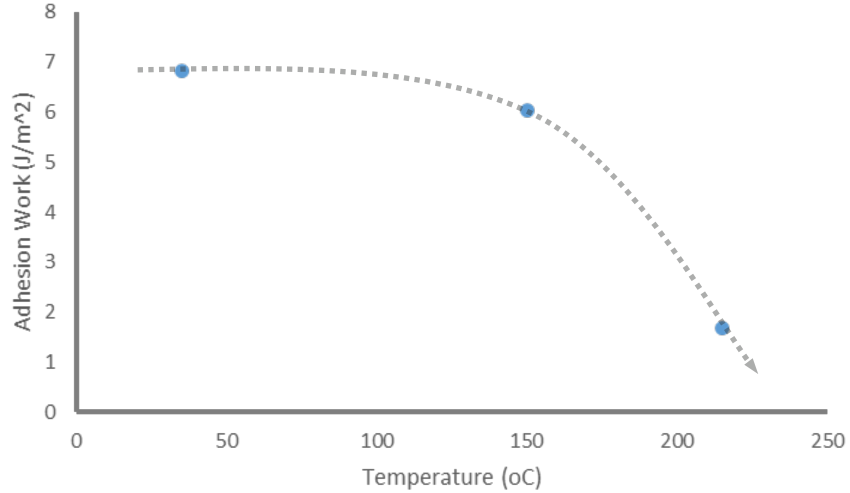


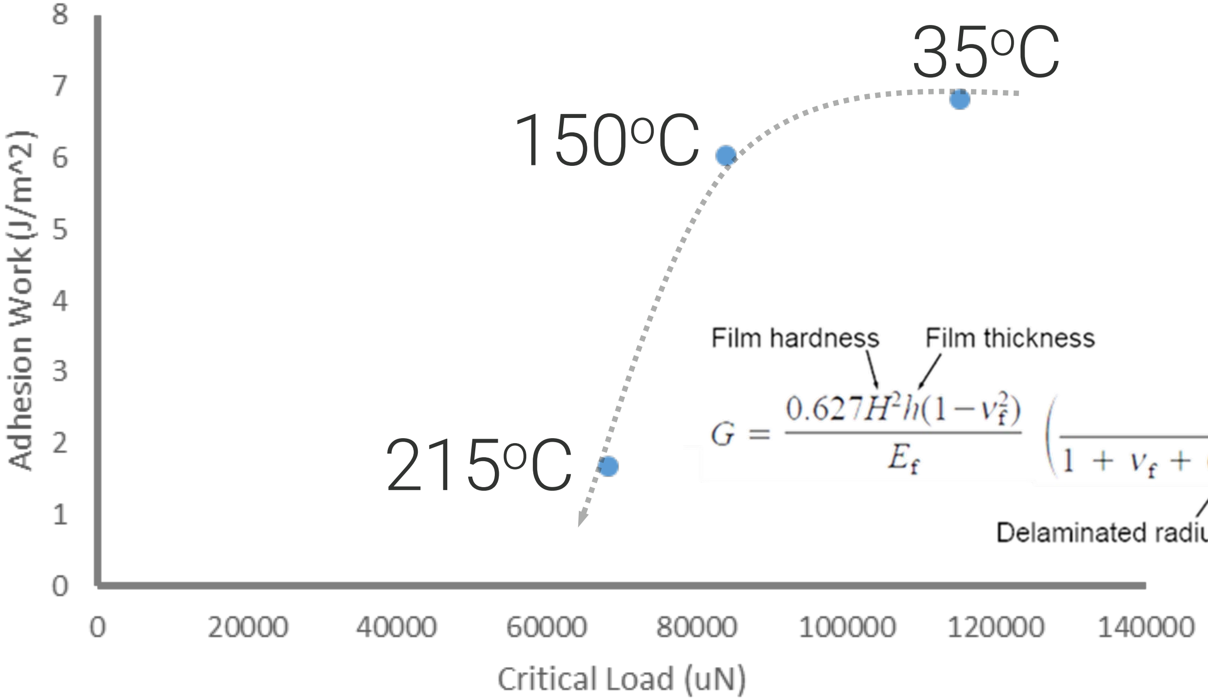
Adhesion Work Calculation at Temperature Levels

Polymer Film on Copper

H (GPa)	T (°C)	E (GPa)	Lcrit (uN)	G (J/m^2)
0.49	35	6.02	115218.2	6.81
0.39	150	4.69	84091.5	6.04
0.11	215	1.59	68276.1	1.68





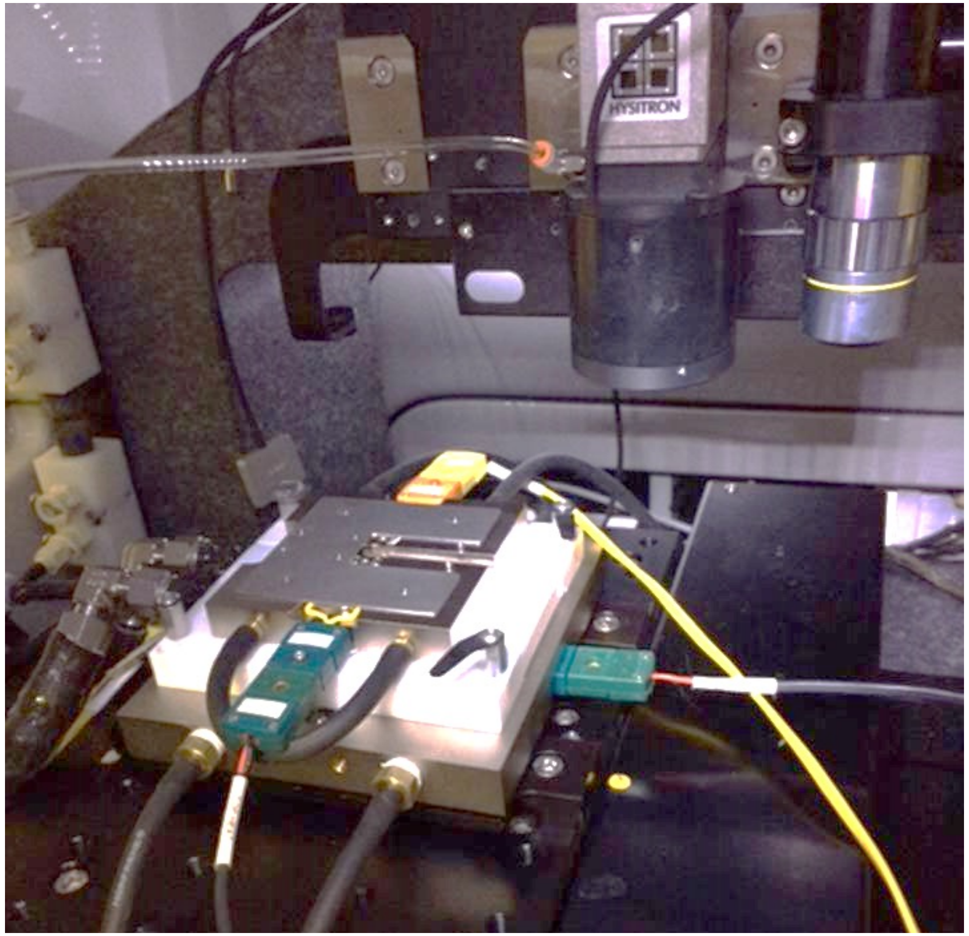


$$G = \frac{0.627 H^2 h (1-\nu_f^2)}{E_f} \left(\frac{1}{1 + \nu_f + (a/x)^2 (1-\nu_f)} \right)^2$$

Applications at Low Temperature

xSol Cooling Stage System Overview

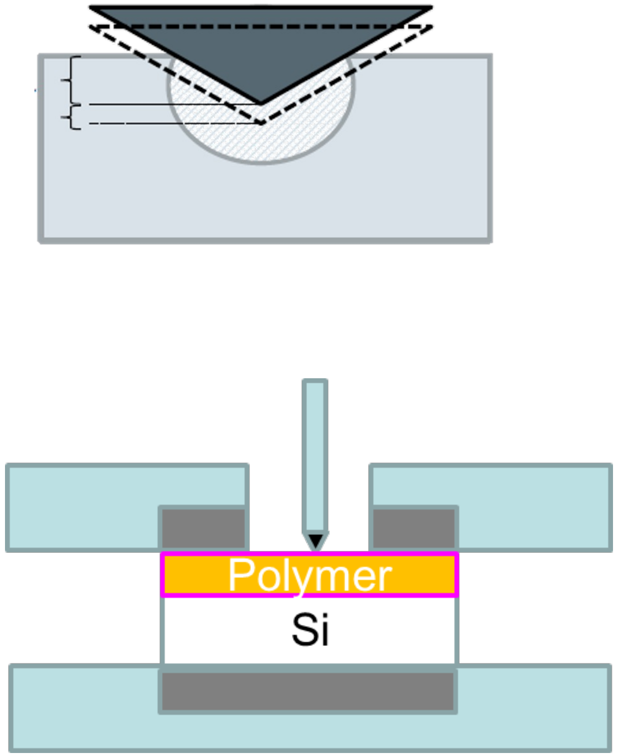
- Nitrogen gas passes through liquid N2 to cool
- Cold gas enters the chamber through a port on the side of the enclosure to passively cool the chamber
- A heater is used to actively hold the temperature steady at the set temperature
- Achieves cooling capabilities down to -160°C

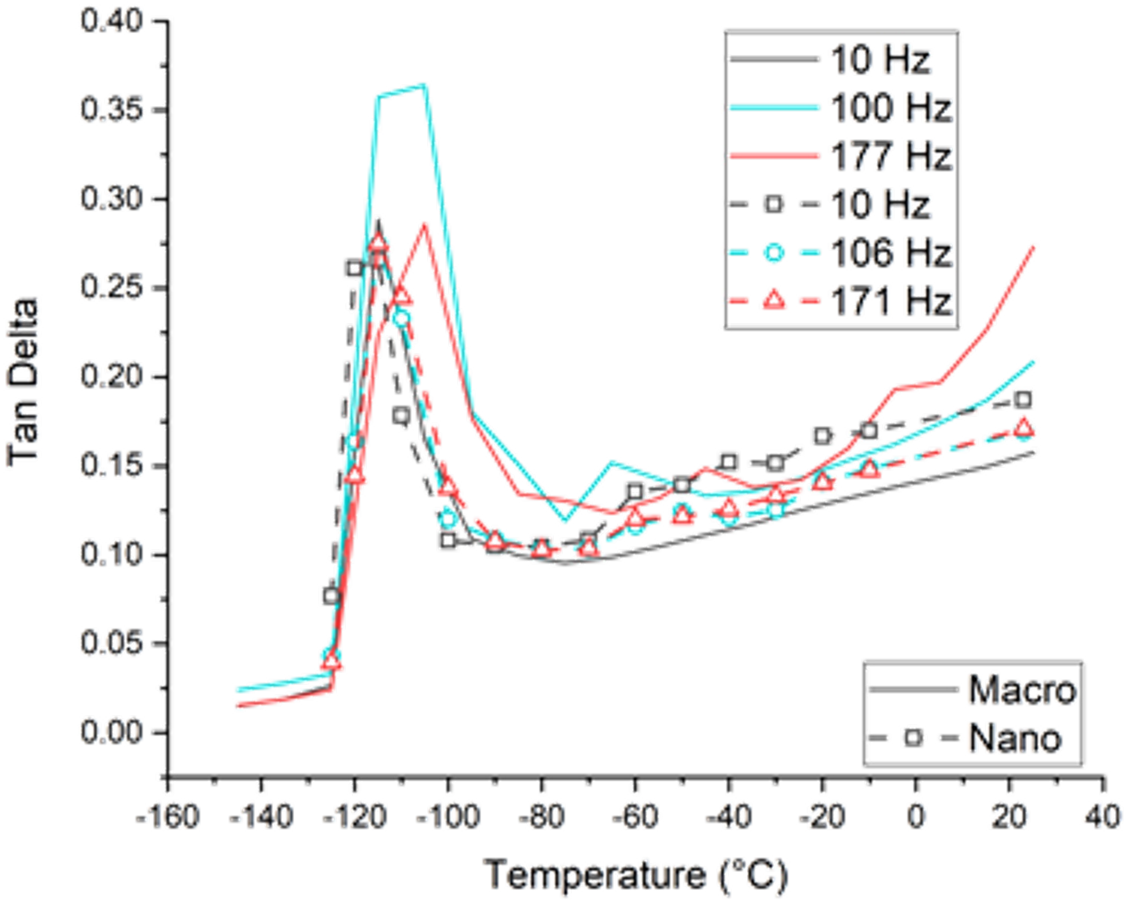


Internal heat exchanger/sample chamber

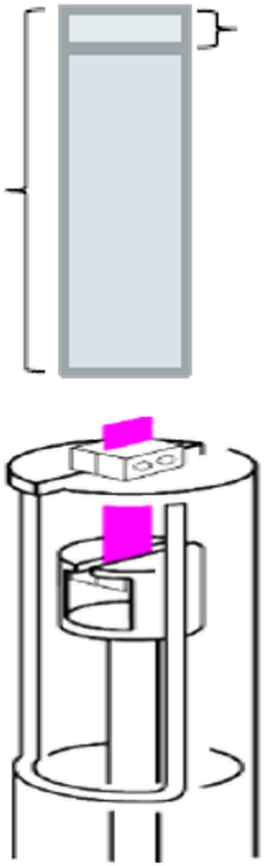
Comparisons between Nano and Macro:
Frequency Effects on 500um PDMS

Nano-DMA



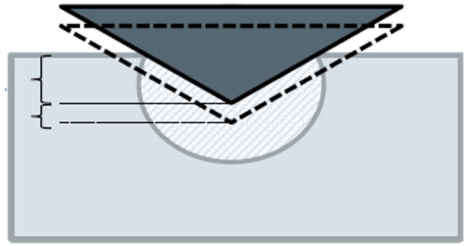


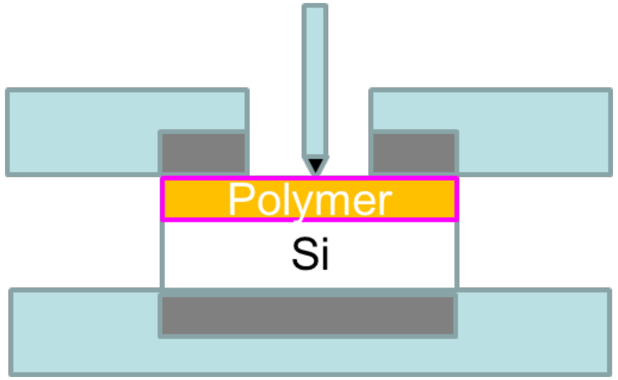
Macro-DMA

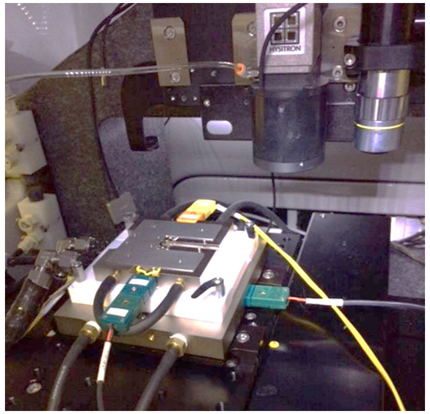


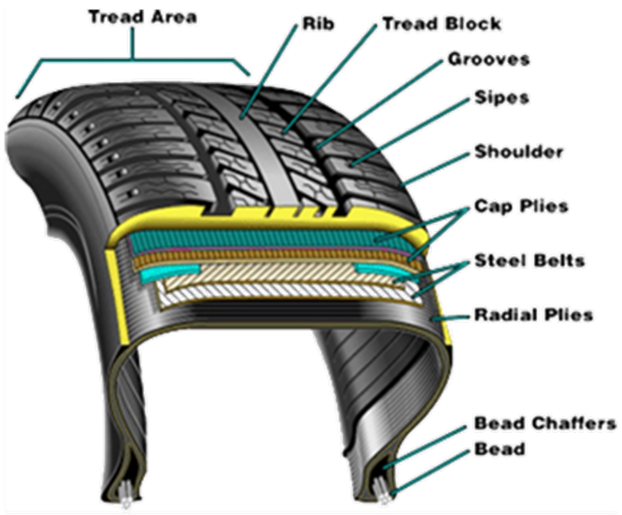
Source: TA instrument

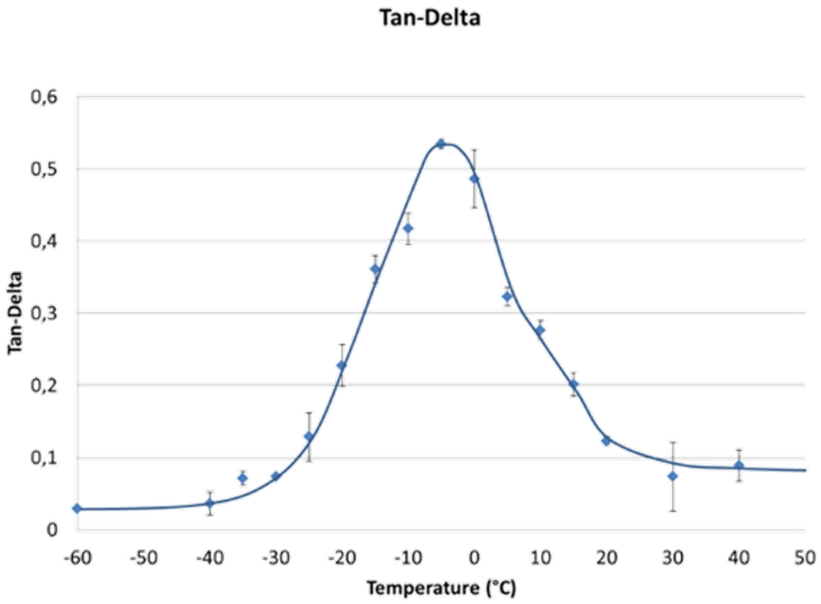
xSol Heating & Cooling on Tire Rubber

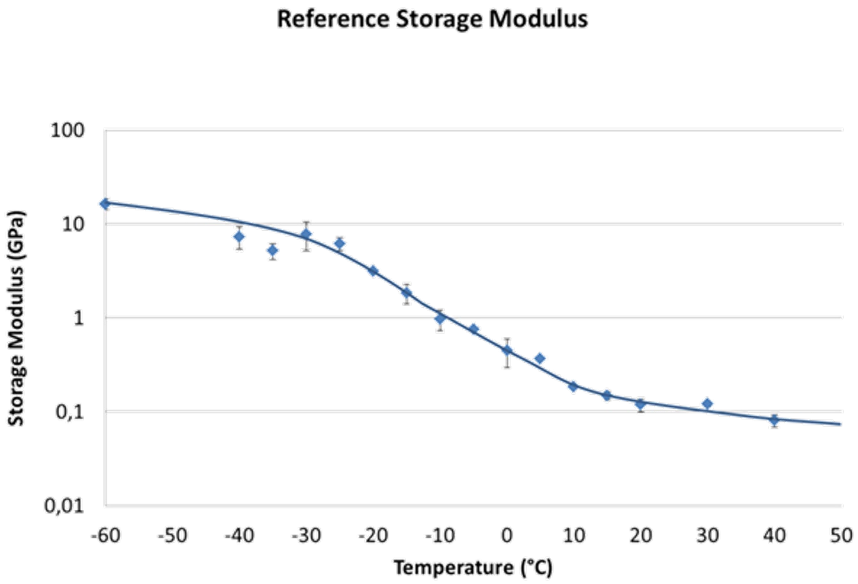




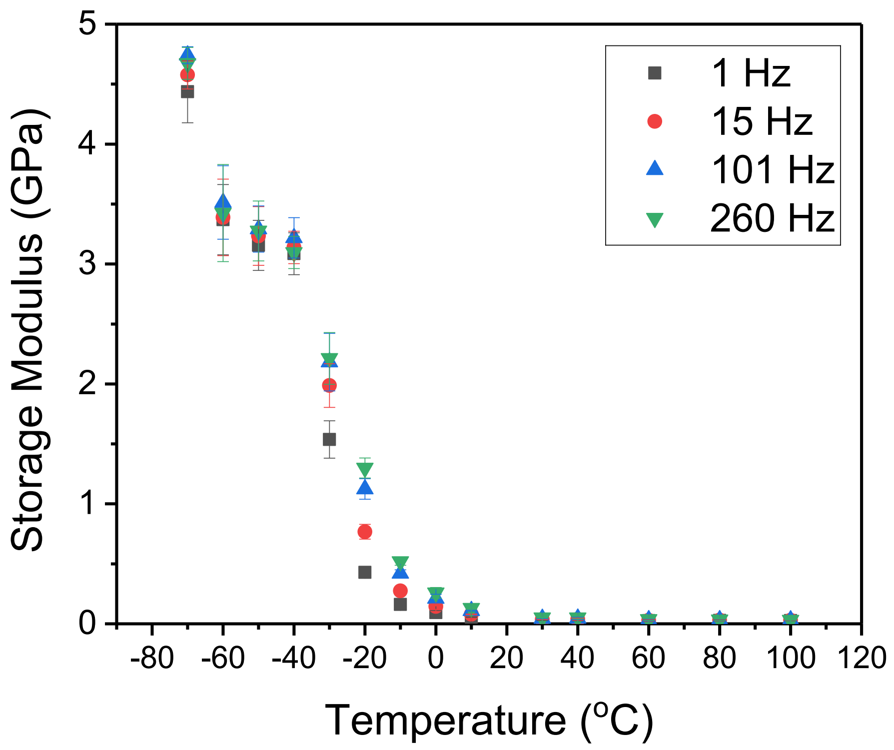




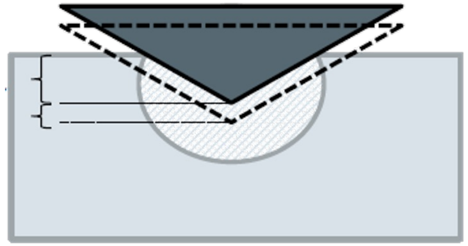


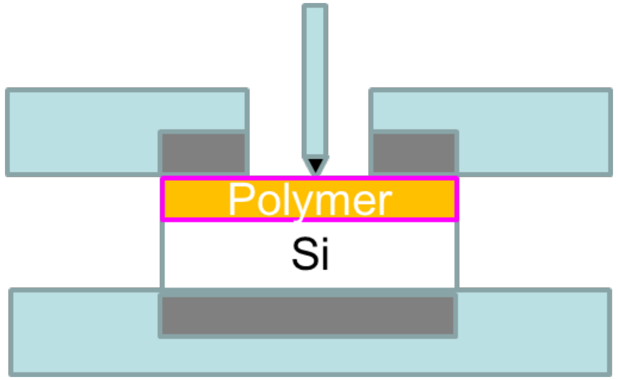


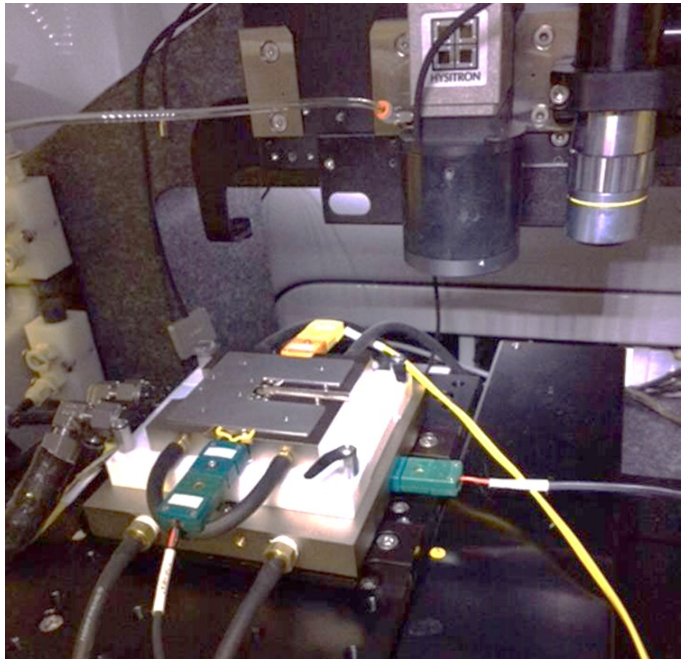
Low Temperature Testing of Polymer Films



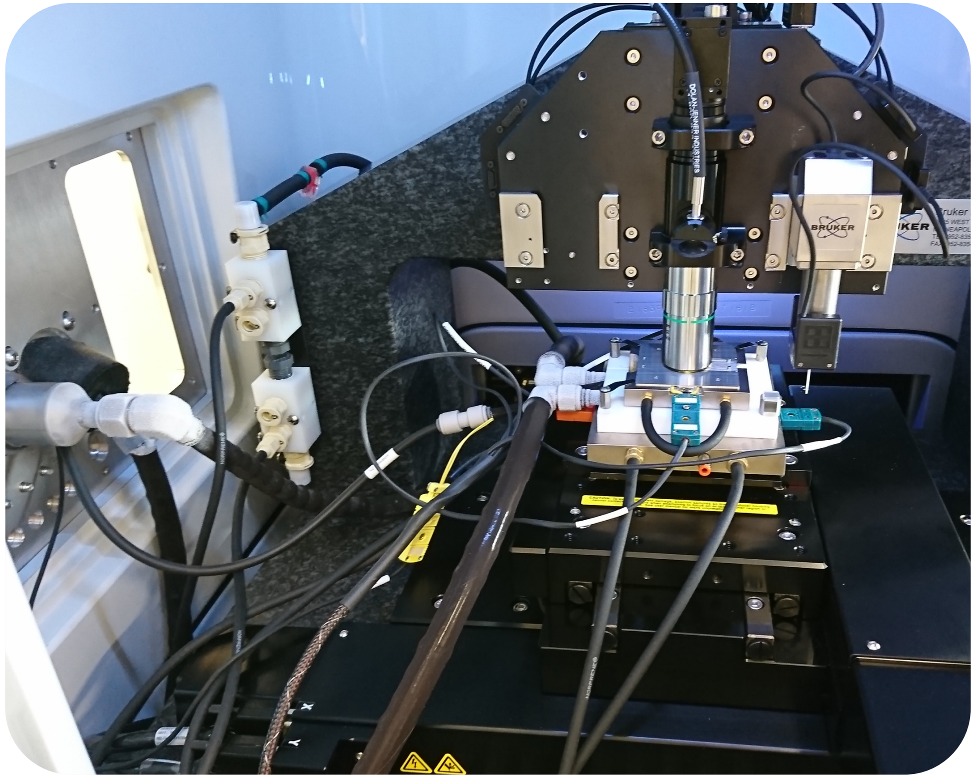
10μm thick polymer film

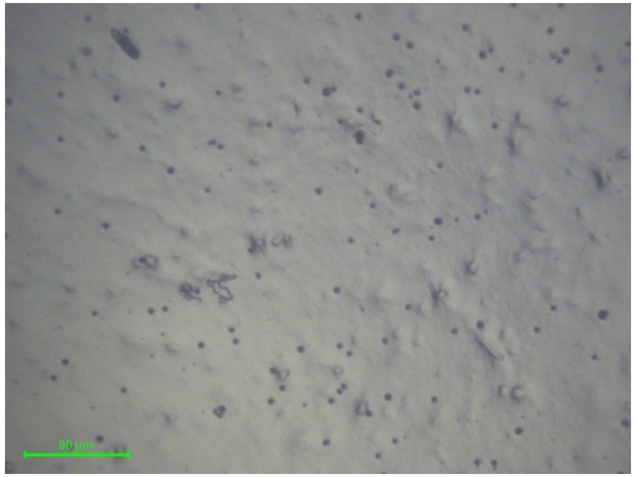


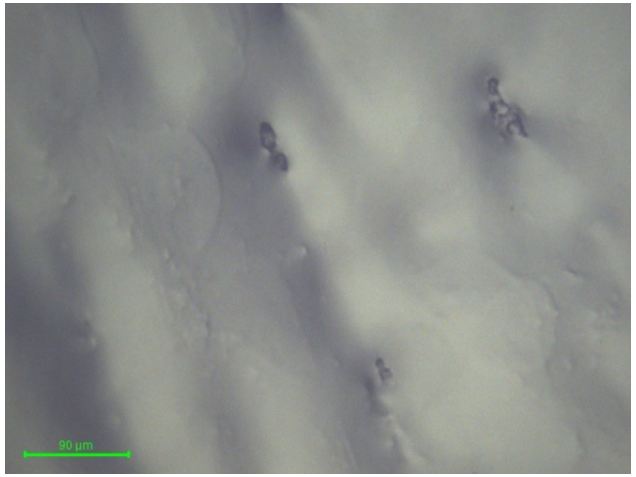




Two 'Over The Counter' Adhesives







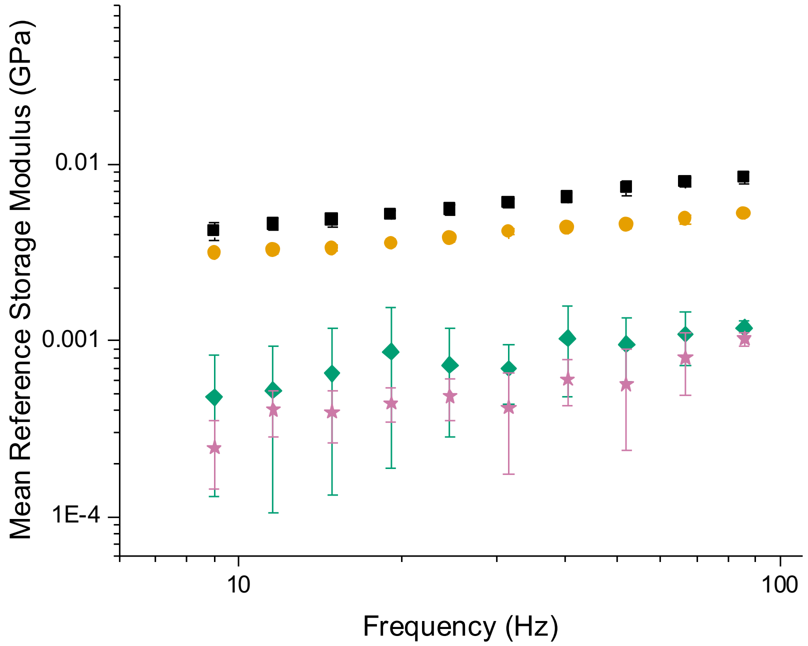
Adhesive 1

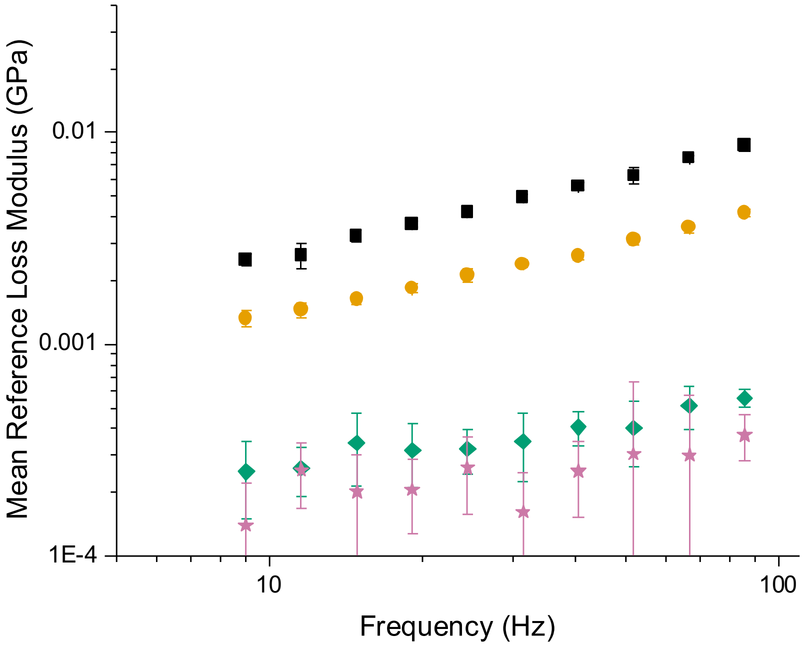
'wall sticky'

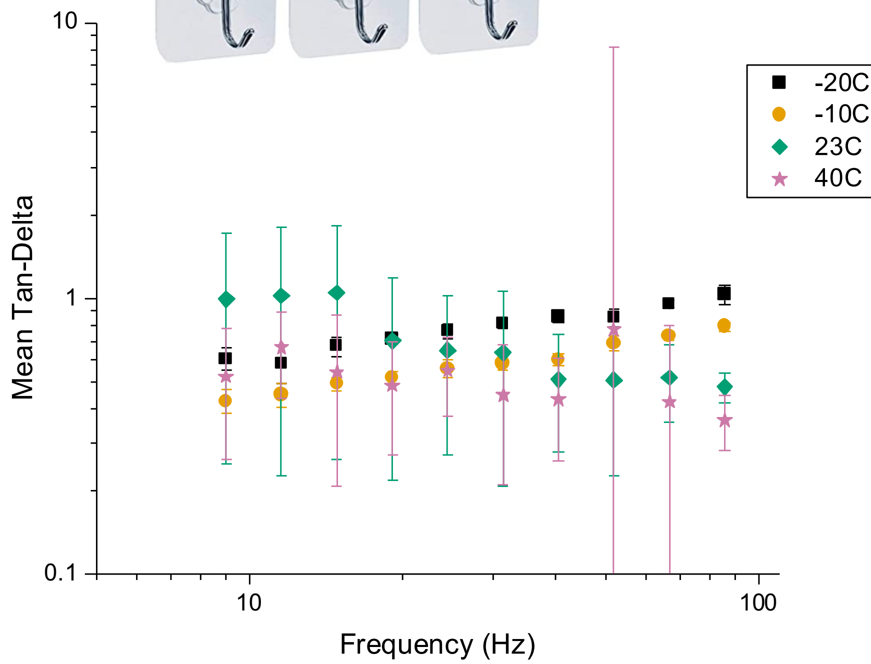
Adhesive 2

'construction'

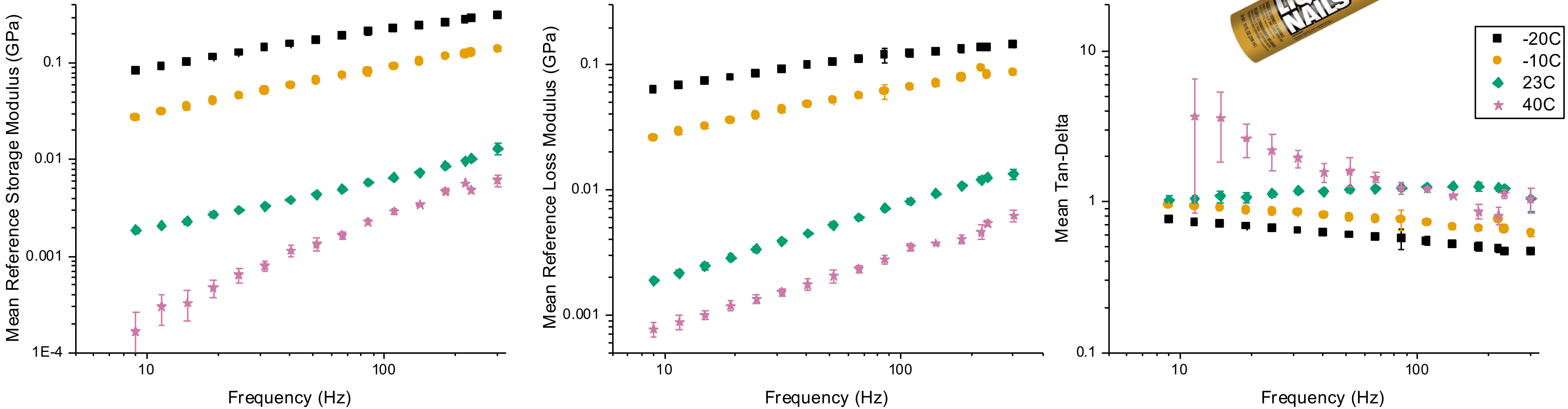
Adhesive 1 - Wall Sticky



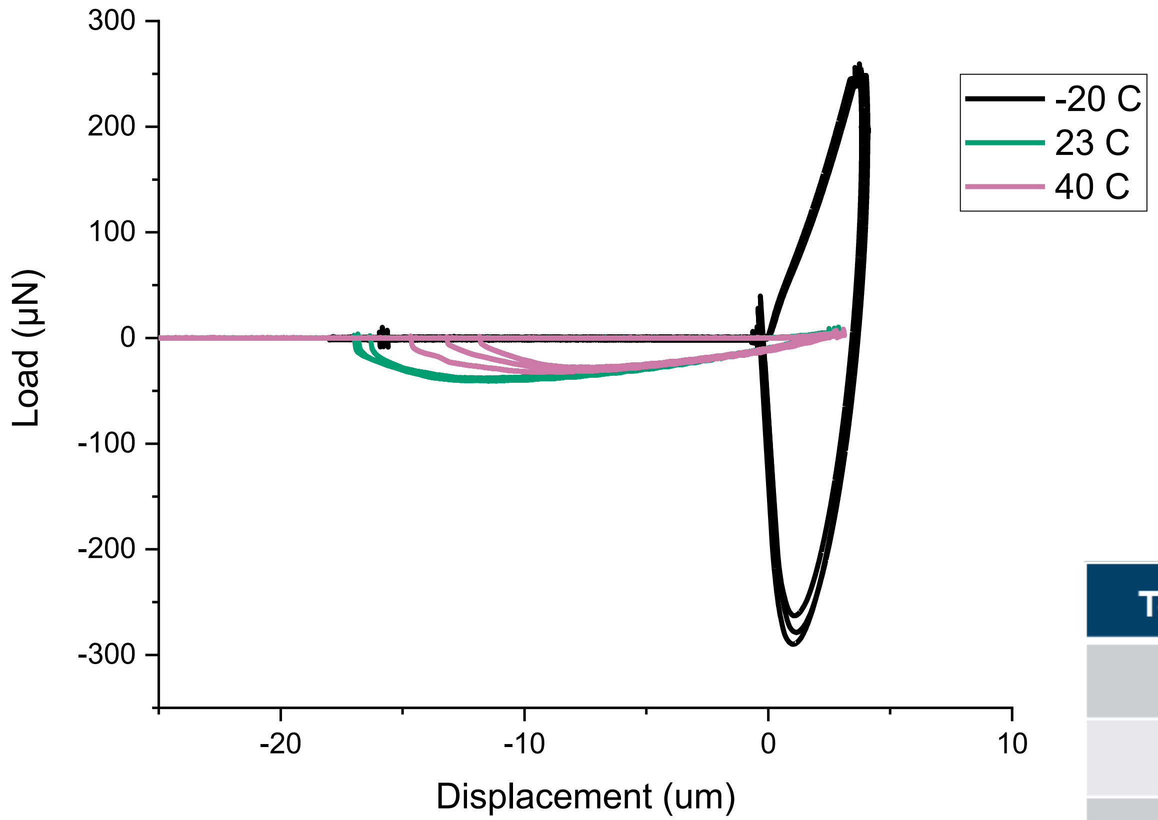


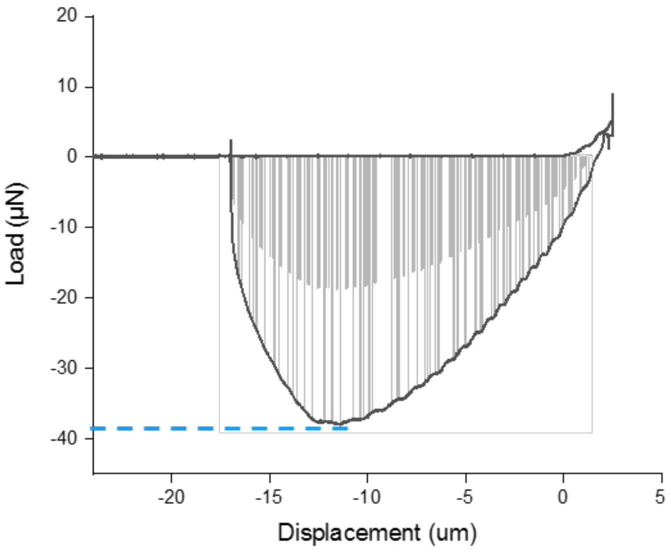


Adhesive 2 – 'Construction'



Adhesive 2 – ‘construction’
Measuring adhesion



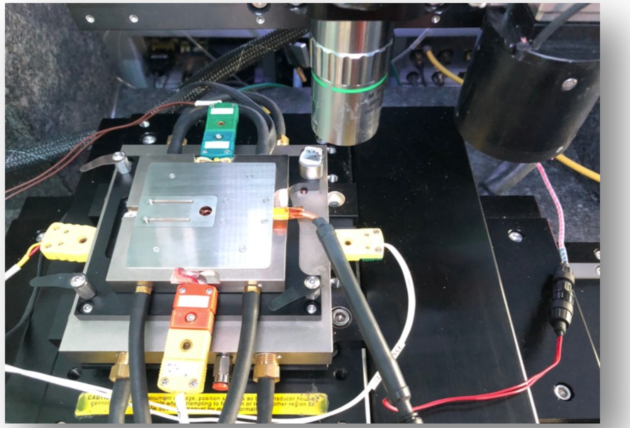


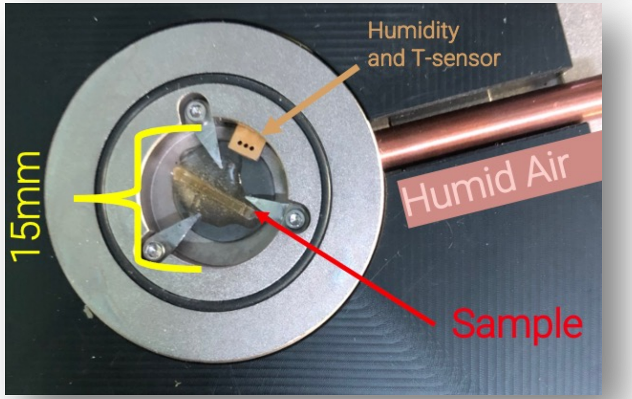
Temperature (°C)	Pull –off Force (uN)	Work (uJ)
-20	-276.97±13.41	378.59±16.49
23	-39.55±1.30	232.82±4.71
40	-29.73±2.17	151.98±28.46

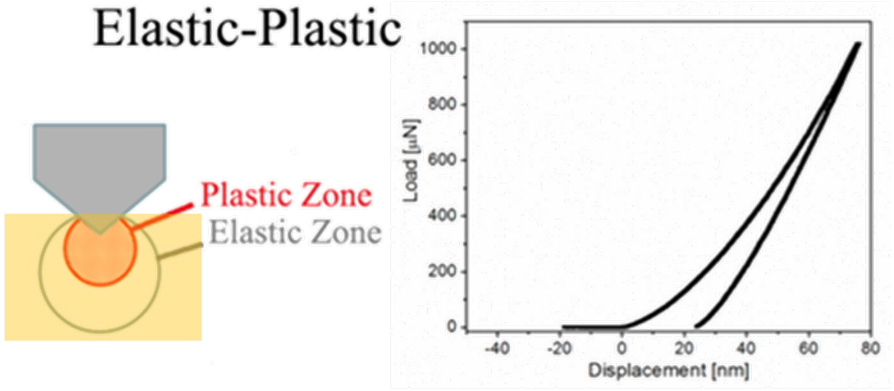
Applications at Humidity Levels

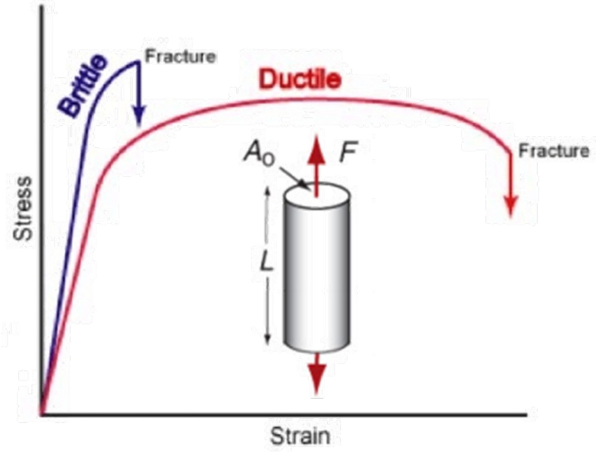
xSol® Humidity Control

Humidity, Temperature, Mechanical Property Results of a Polymer Film





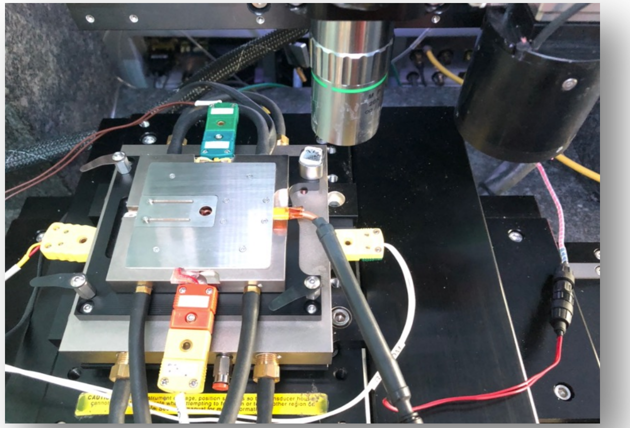


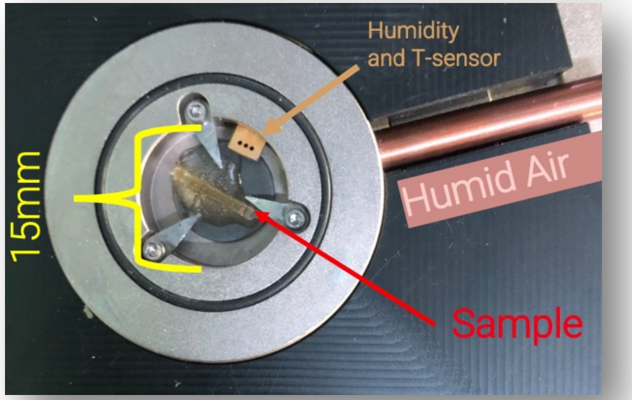


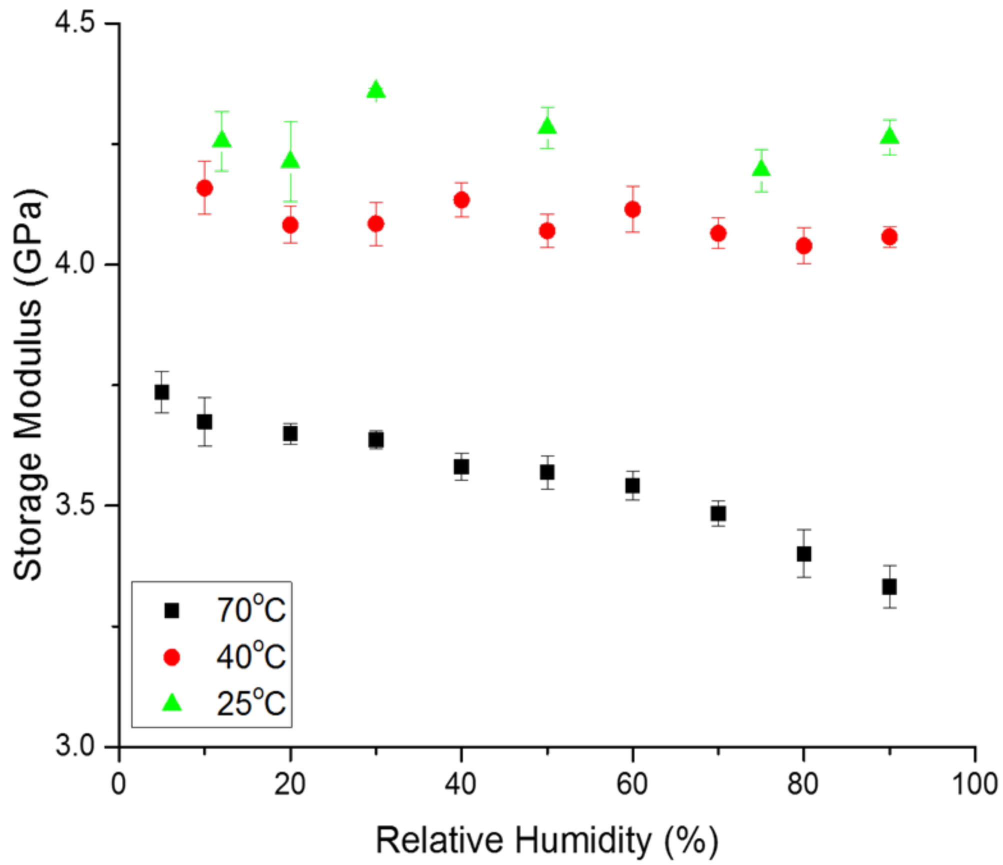
https://sites.google.com/site/polymorphismmyhomepage/investigating-material-failures

xSol® Humidity Control

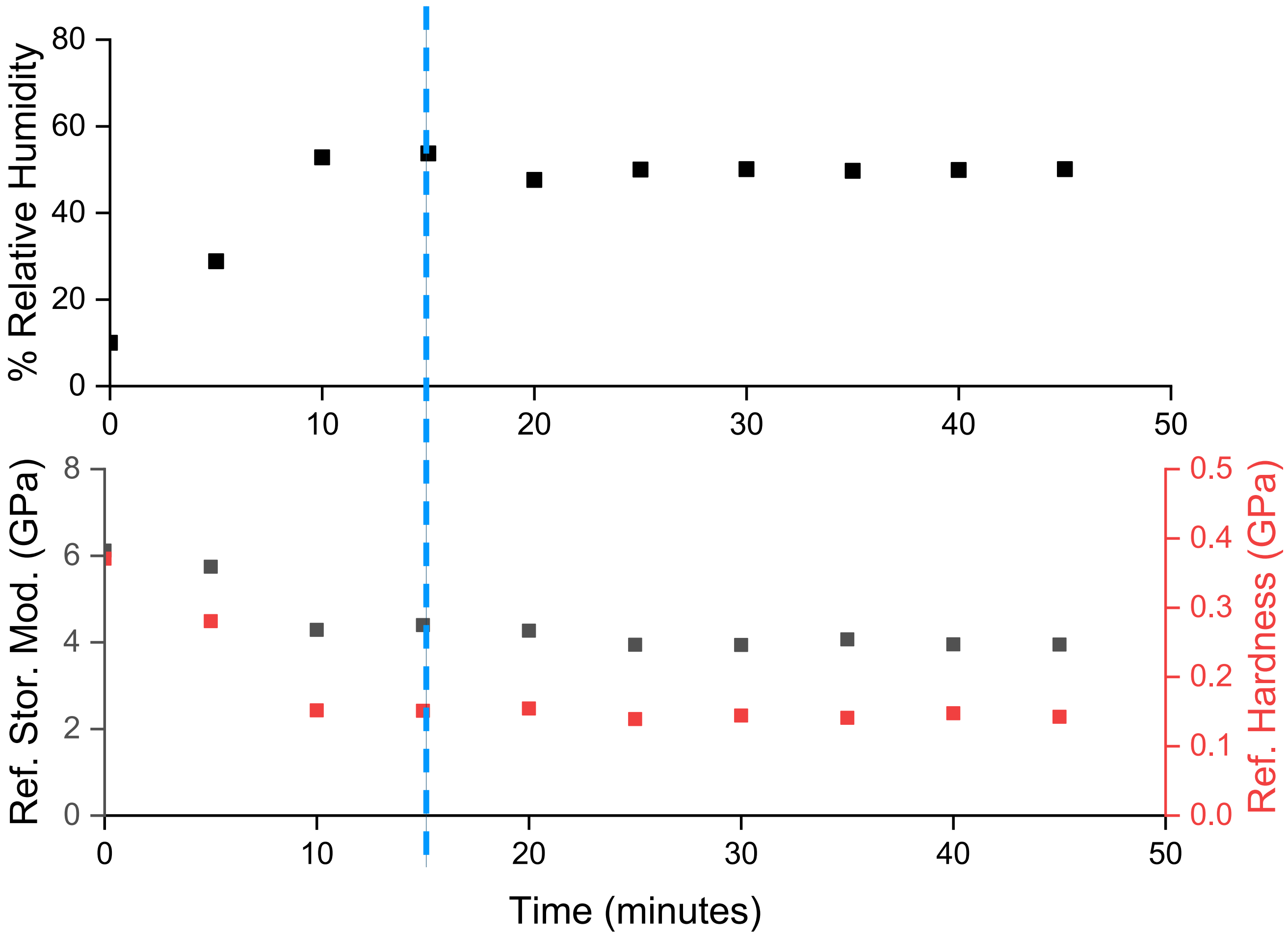
Humidity, Temperature, Mechanical Property Results of a Polymer Film

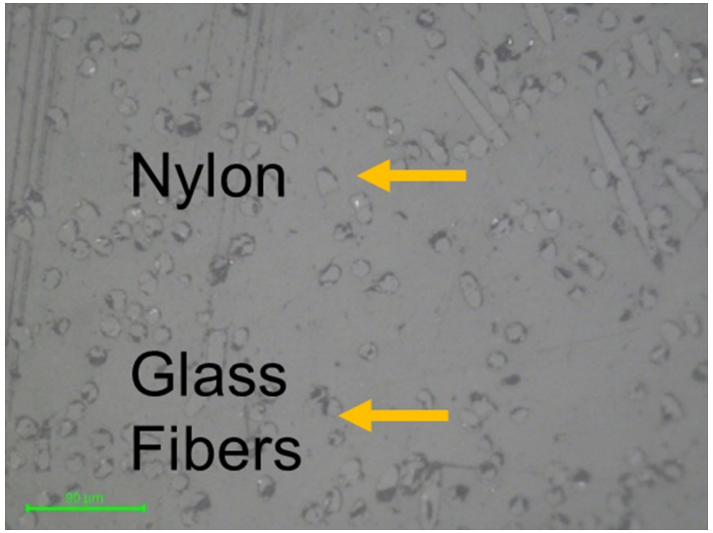






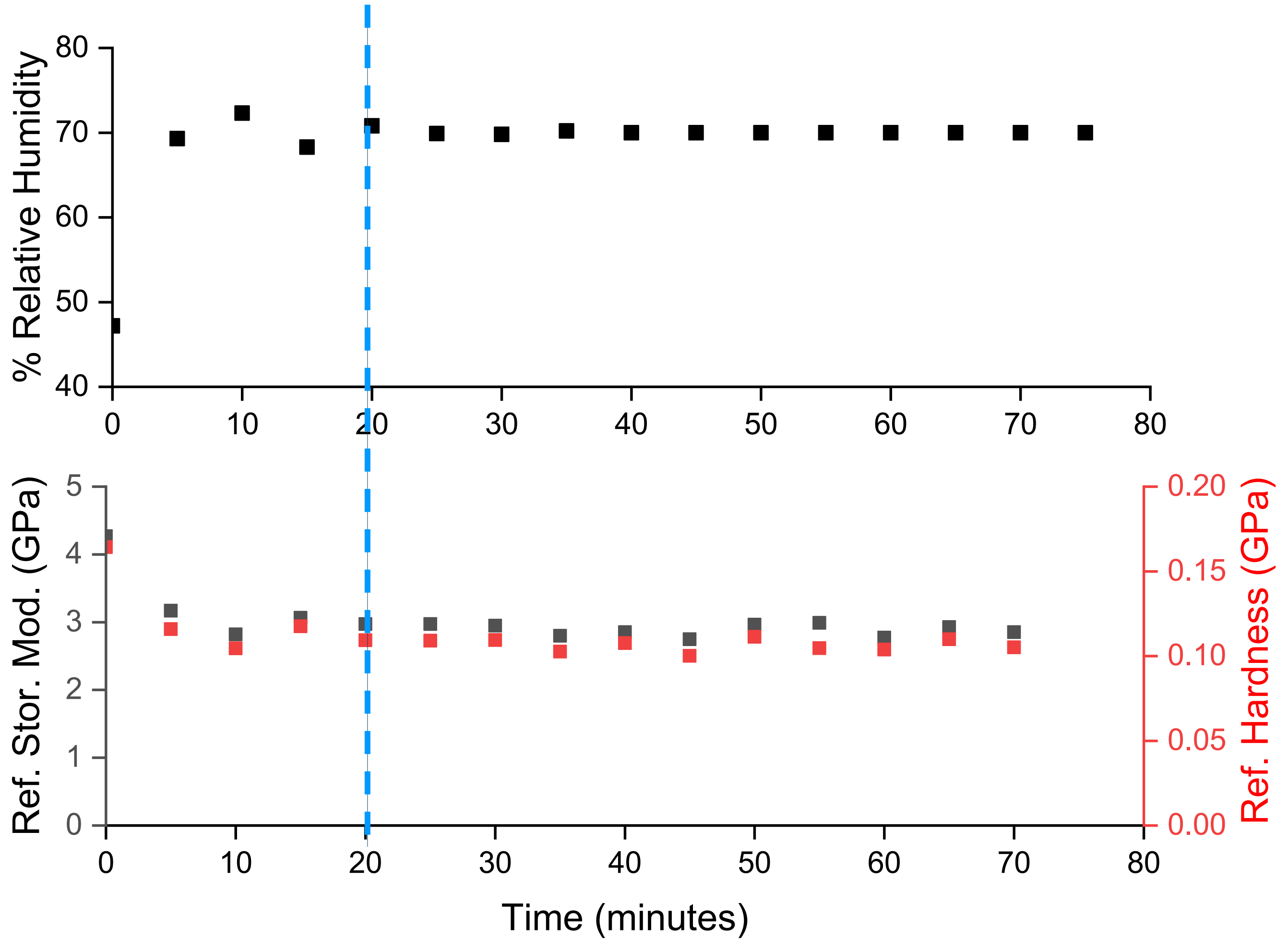
Humidity Testing of Glass Filled Nylon 66

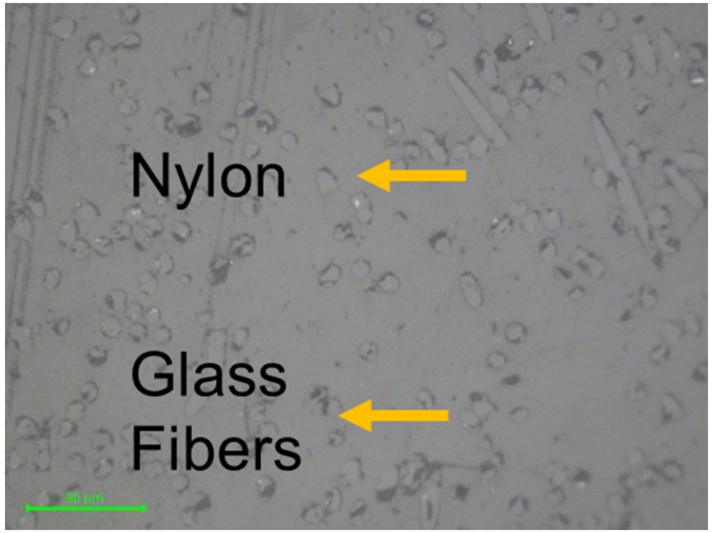




- 10% --> 50 % RH < 20 mins
- Measured humidity inside xSol
- Frequency = 20 Hz
- Room temperature

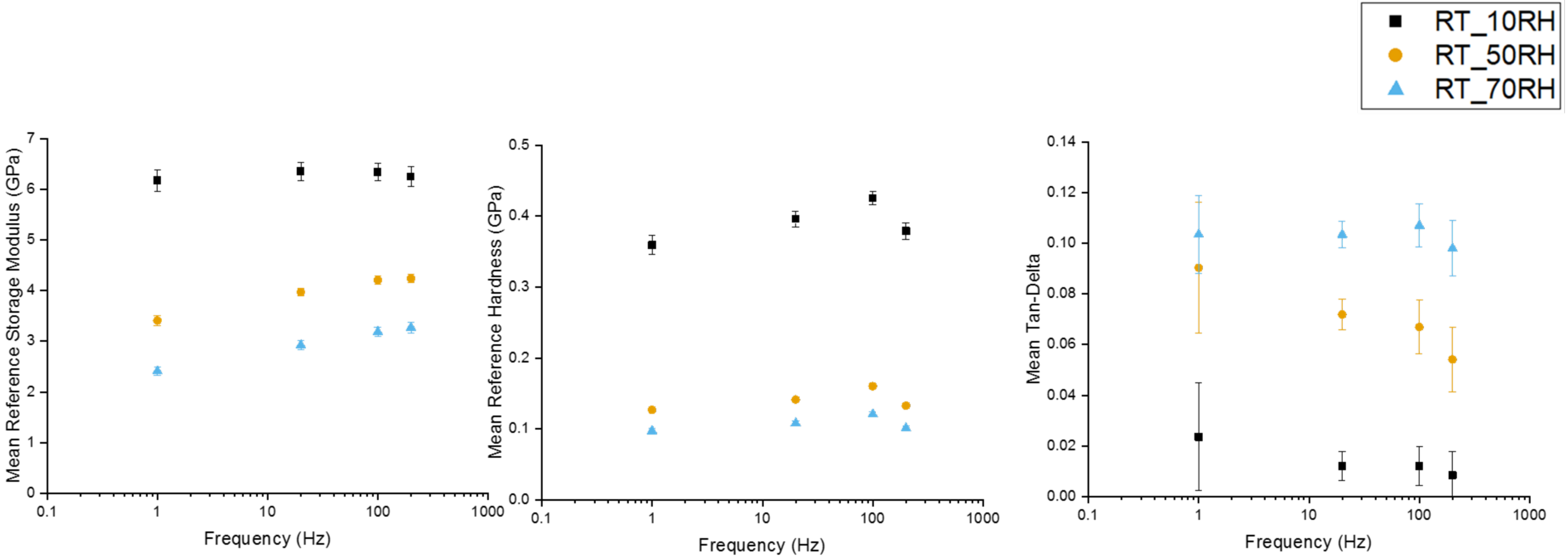
Humidity Testing of Glass Filled Nylon 66





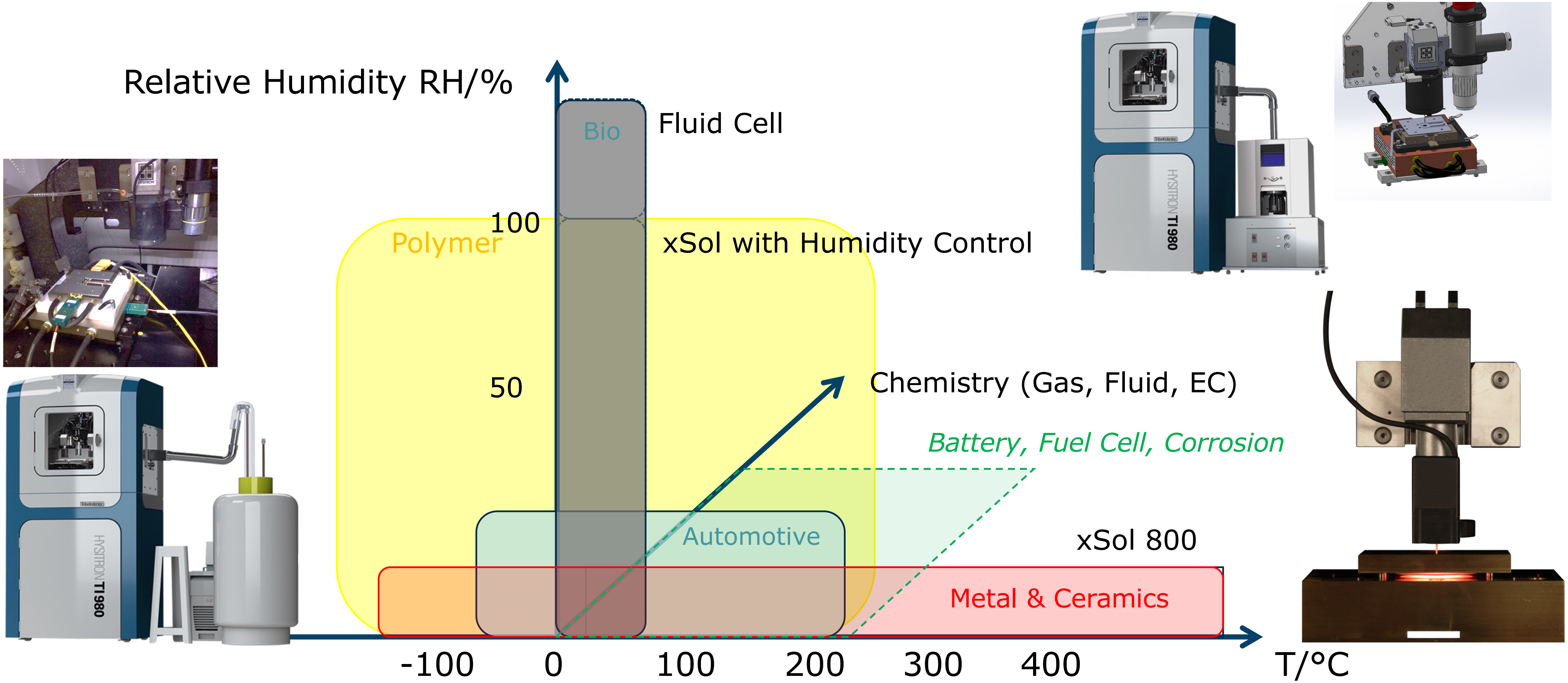
- 50 % --> 70% RH < 20 mins
- Measured humidity inside xSol
- Frequency = 20 Hz
- Room temperature

Humidity Testing of Glass Filled Nylon 66



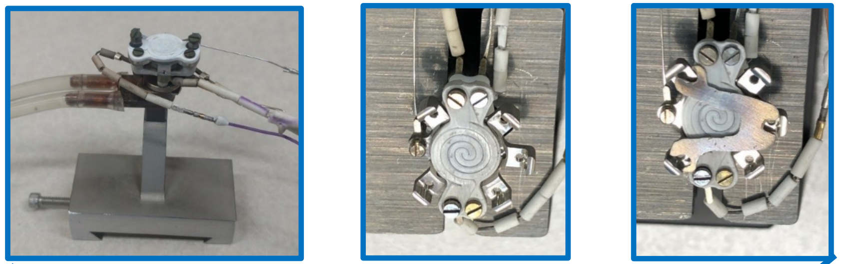
Take-Away

Expanding & Exploring a Larger Parameter Space

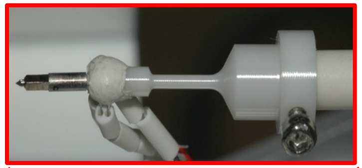


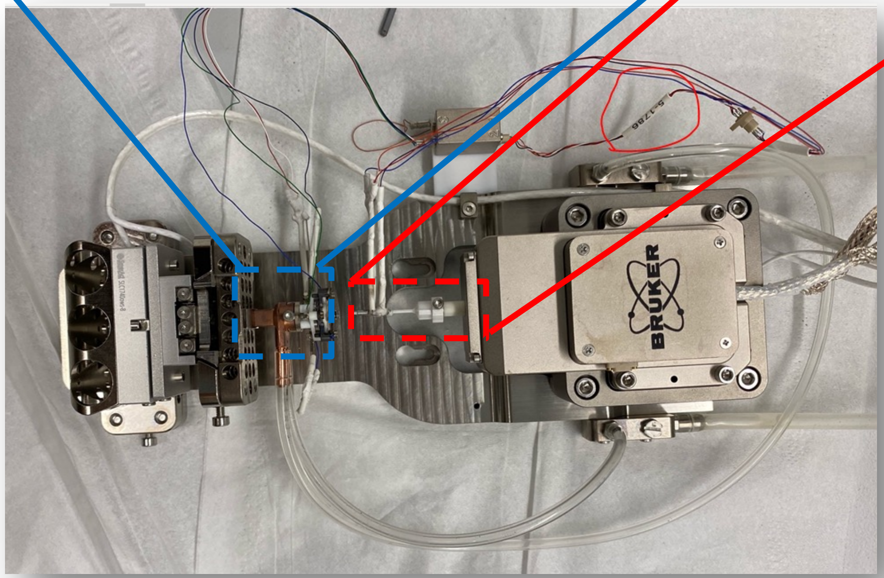
800°C Heating Option for *in-situ* SEM Tool of PI-89

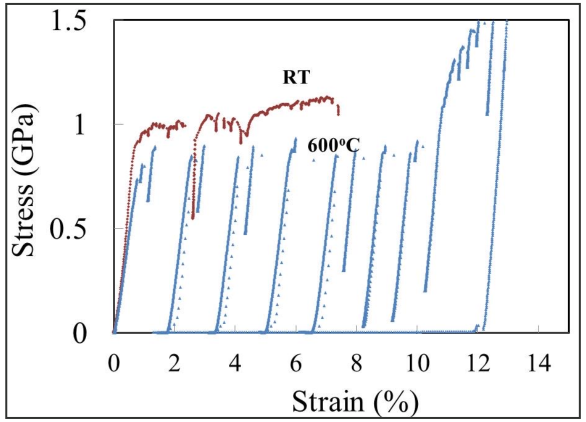
Sample Heater

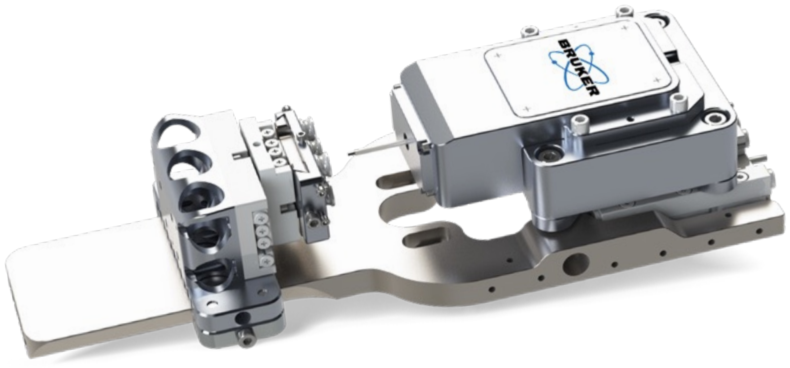


Probe Heater









- Independent tip and sample heaters, with separate feedback control
- Water cooling and thermal breaks to reduce heat flow into the system
- 800°C maximum temperature
- Up to 30°C/min ramp rates

www.bruker.com/Nanomechanical-testing

Innovation with Integrity