A Novel Combination of Micromechanical Testing And Thermal Analysis

to Investigate the Temperature Dependence of Interfacial Adhesion in Fibre Reinforced Polymer Composites

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Glasgow Research Partnership in Engineering - funding

Owens Corning – glass fibres

SABIC - polypropylene

Liu Yang - measurements

Outline: TMA – Microbond Testr

- **Introduction Thermoplastic Composites**
  - Residual Thermal Stresses
  - Glass Fiber-Thermoplastic IFSS
- **IFSS Experiments**
  - Conventional Microbond Test
  - Microbond Test in the TMA
- **Results**
- **Conclusions**

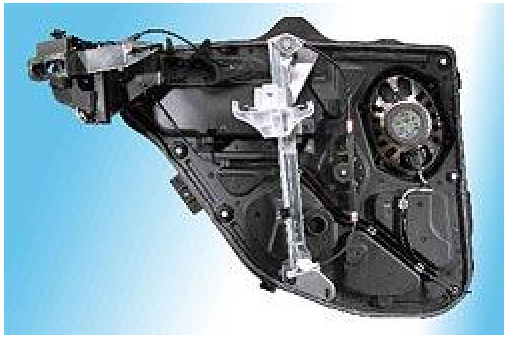
GRPE
GLASGOW RESEARCH PARTNERSHIP IN ENGINEERING

AMRL
Advanced Materials Research Laboratory

University of Strathclyde

Thermoplastic Composites Technology

- Strong continuing growth
- Attractive Performance:Price ratio
  - "Clean" processing - no chemistry
  - Intrinsically recyclable

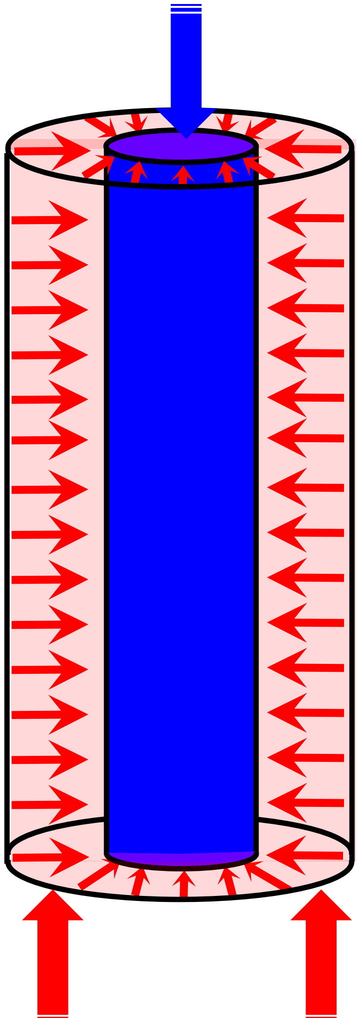


- Still room for improvement
  - particular need to better understand (and increase) GF–PP interface stress transfer capability (IFSS)

- Processed mostly by moulding

Residual Stress at the Interface

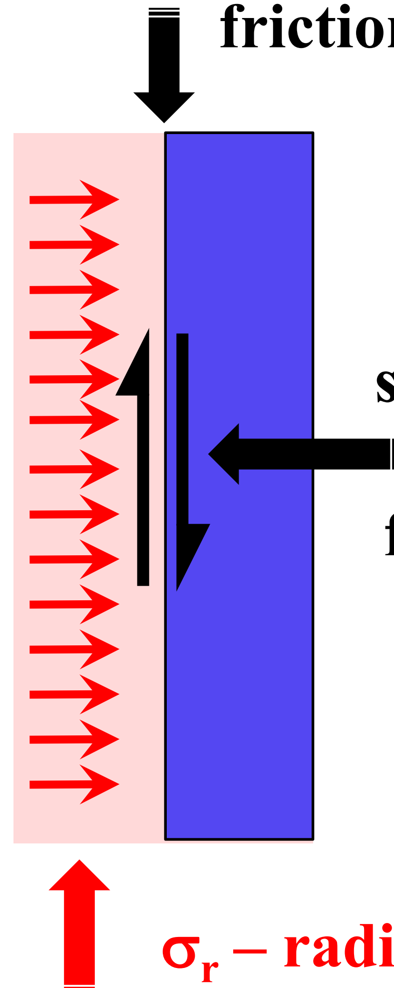
Fiber – Low LCTE (α)



Polymer – High LCTE (α)

Large Δα and ΔT from processing to room temp results in radial compressive stress at the interface

μ_s – coefficient of static friction at interface



Interfacial stress transfer capability from residual radial stress

$$\tau = \mu_s \sigma_r$$

σ_r – radial compressive stress

Modelling Interfacial Residual Stress

$$\sigma_{rm} = E_m(\alpha_m - \alpha_{fT})\Delta T$$

$$\sigma_{rm} = A_1(1 - b^2/r^2) \quad where \;\; b = F(V_f)$$

$$\text{for } A_1 \text{ solve} \begin{bmatrix} X_{11} & X_{12} \\ X_{21} & X_{22} \end{bmatrix} \begin{bmatrix} A_1 \\ A_3 \end{bmatrix} = \begin{bmatrix} (\alpha_m - \alpha_{fL})\Delta T \\ (\alpha_m - \alpha_{fT})\Delta T \end{bmatrix}$$

$$\text{where } X_{11} = 2\left(\frac{\nu_m}{E_m} + \frac{\nu_A}{E_L}\frac{V_m}{V_f}\right) \quad X_{12} = -\left(\frac{V_m}{E_L V_f} + \frac{1}{E_m}\right)$$

$$X_{21} = -\left(\frac{(1-\nu_f)V_m}{E_m V_f} + \frac{(1-\nu_m)}{E_m} + \frac{(1+\nu_m)}{E_m V_f}\right) \quad X_{22} = \frac{X_{11}}{2}$$

Nairn, J.A., Polymer Composites, 6, (1985) 123.
Wagner H.D. and Nairn J.A., Compos.Sci.Tech., 57, (1997) 1289.

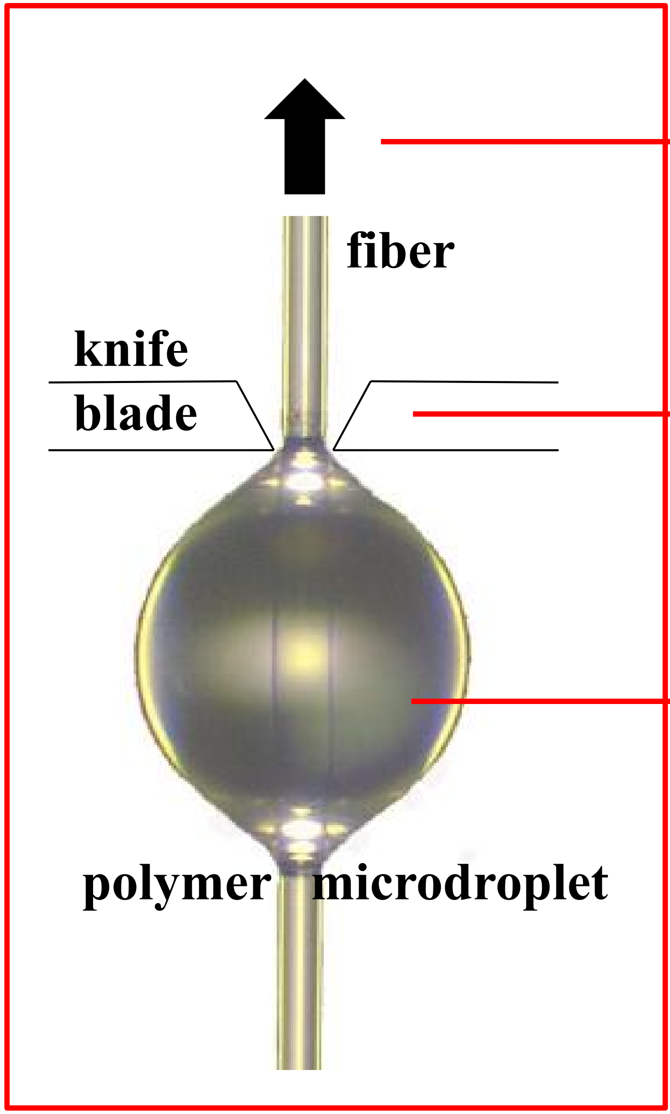
Residual Thermal Stress at Interface

In both models $\sigma_{rm} \propto \Delta T \Rightarrow$ IFSS $\propto \Delta T$

Hence the question –

is the apparent **interfacial strength** (IFSS) in thermoplastic composites **temperature dependent** ?

Microbond Test for GF-PP IFSS



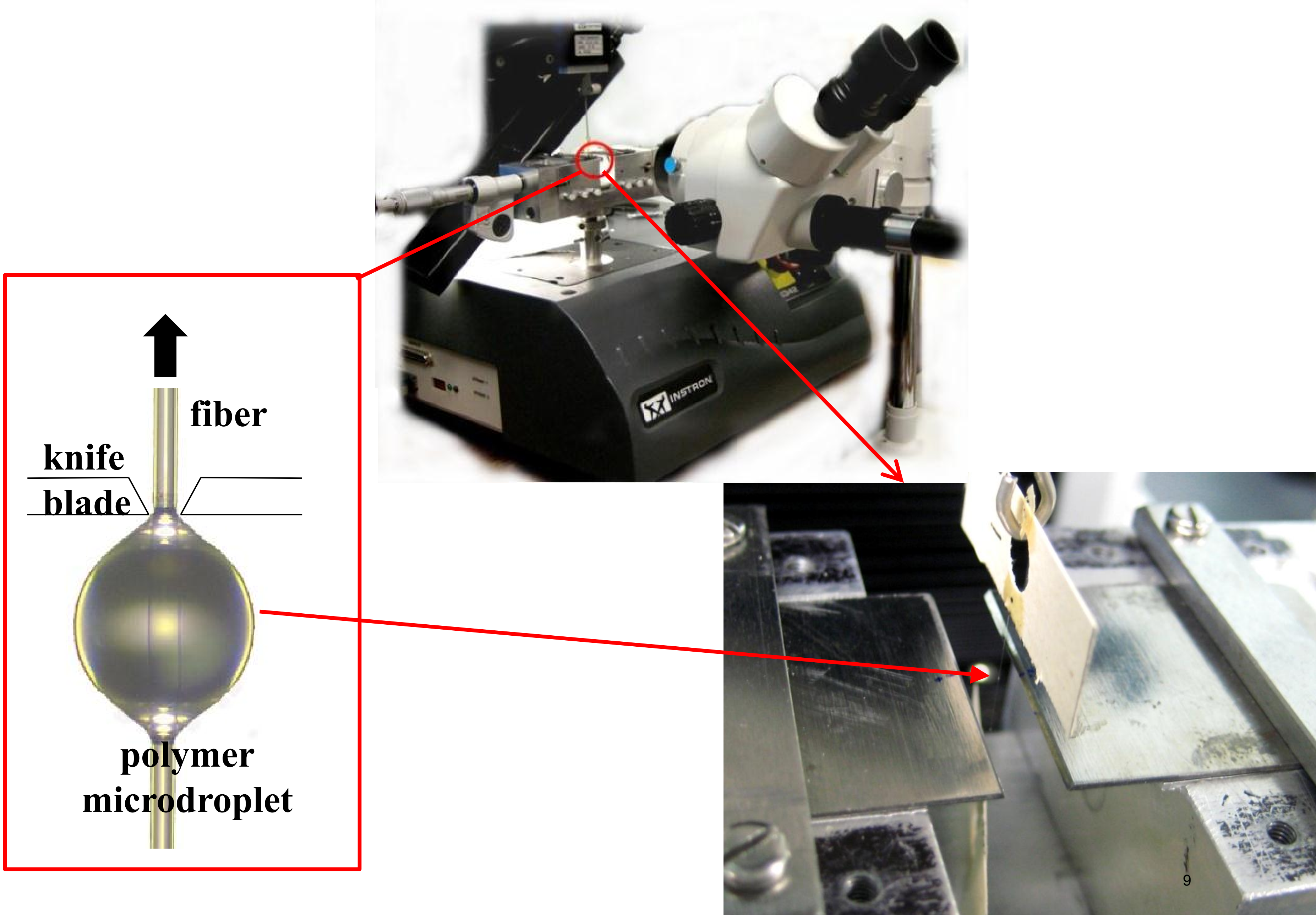
Pull and measure force to debond droplet (0.1-0.3 N)

Knife blades retain droplet

Droplet = 0.1-0.3 mm length

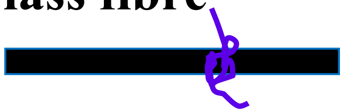
A simple concept – ***with many pitfalls in the execution***

Microbond Test for GF-PP IFSS



Microbond Samples

Glass fibre



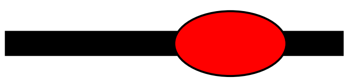
PP fibre

→

Inert Gas (N_2)
RT-220°C
Isothermal 8 min
Cool to ambient temperature

→

Glass fibre



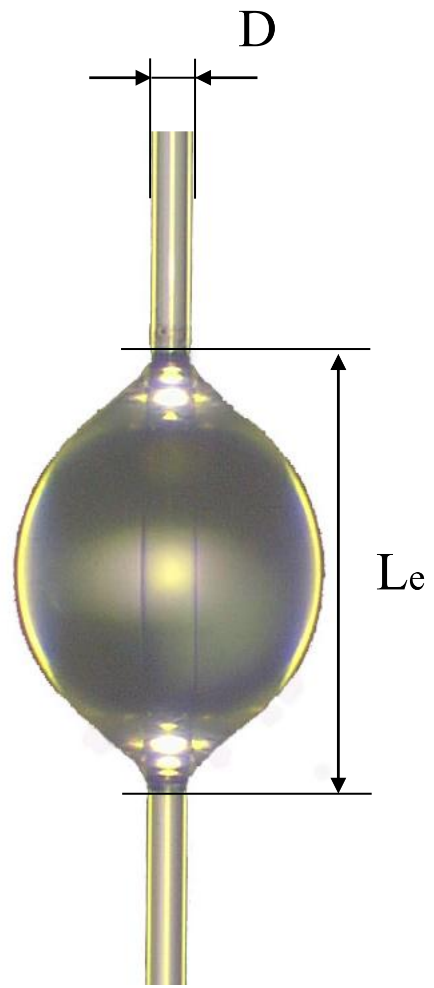
Epoxy droplet

→

In Air
1 hr at 60°C +
2 hr at 120°C
Cool to ambient inside the oven

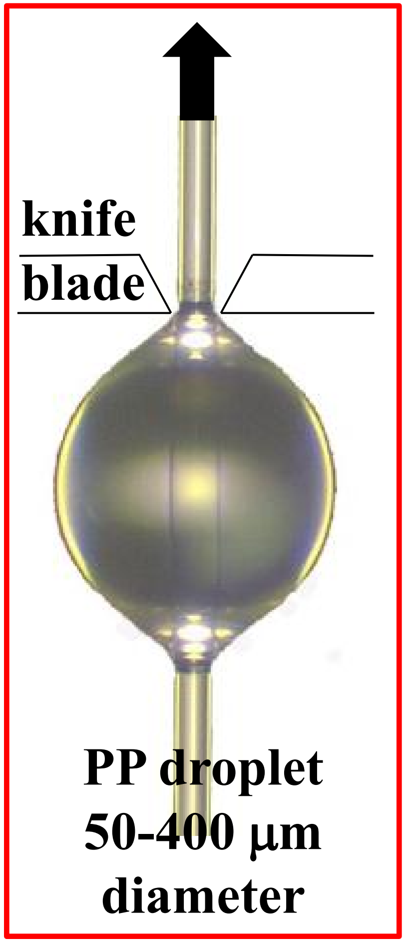
→

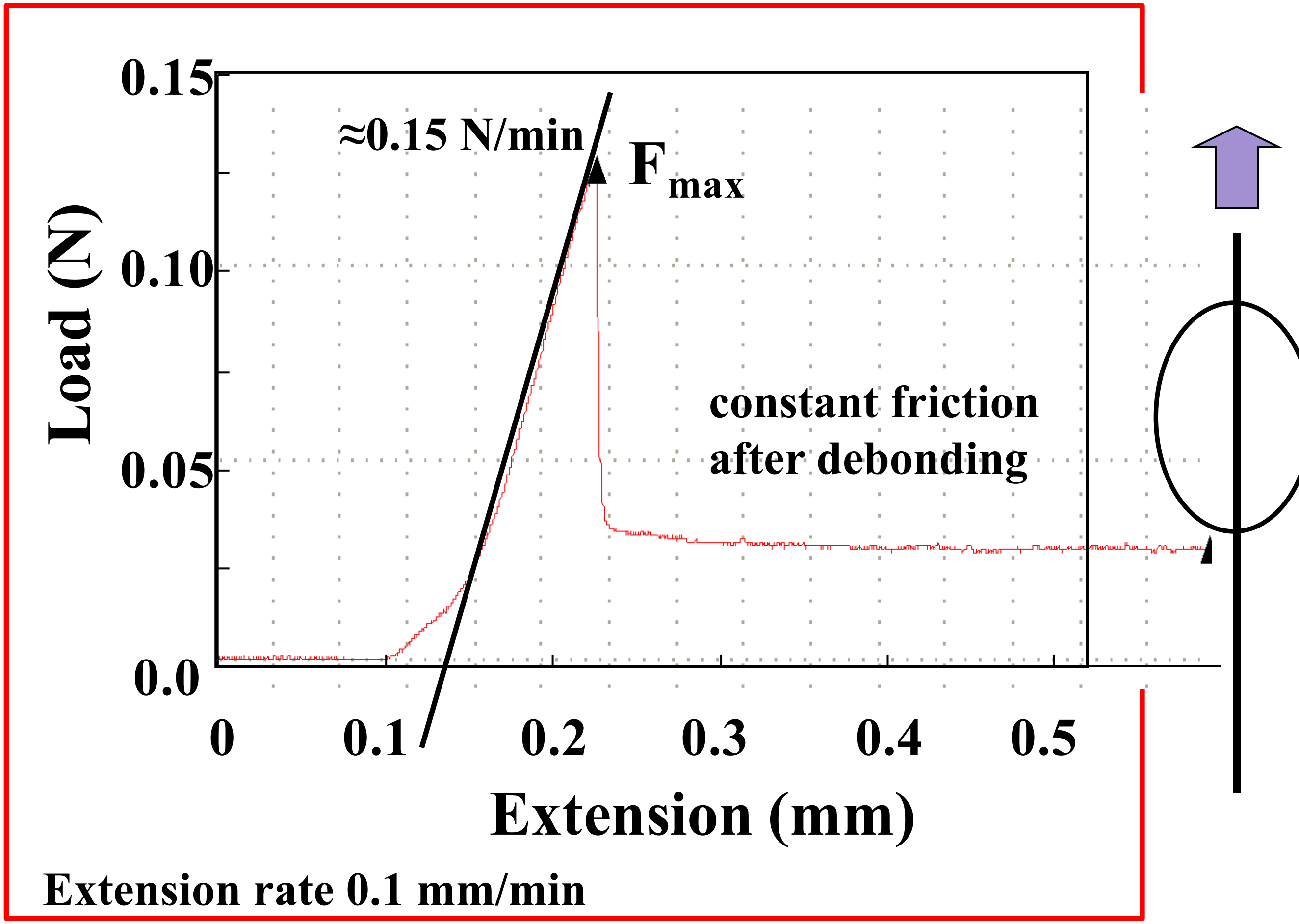
Solid polymer droplet



Embedded Interfacial Area: $A_e = \pi \times D \times L_e$

Microbond Test for GF-PP IFSS





Assuming shear stress is distributed uniformly around the interface then -

Average IFSS: $$\tau = \frac{F_{max}}{A_e}$$

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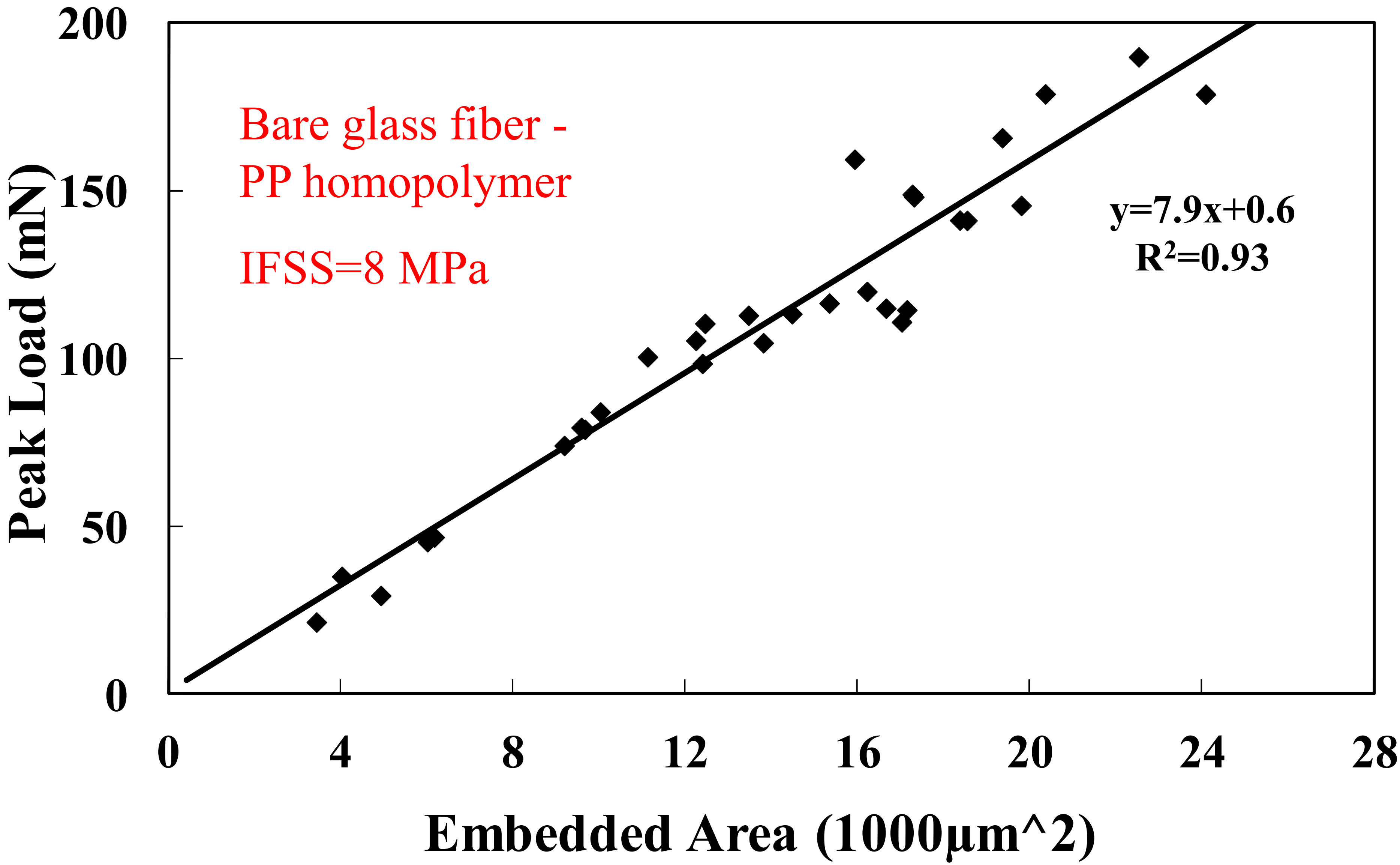
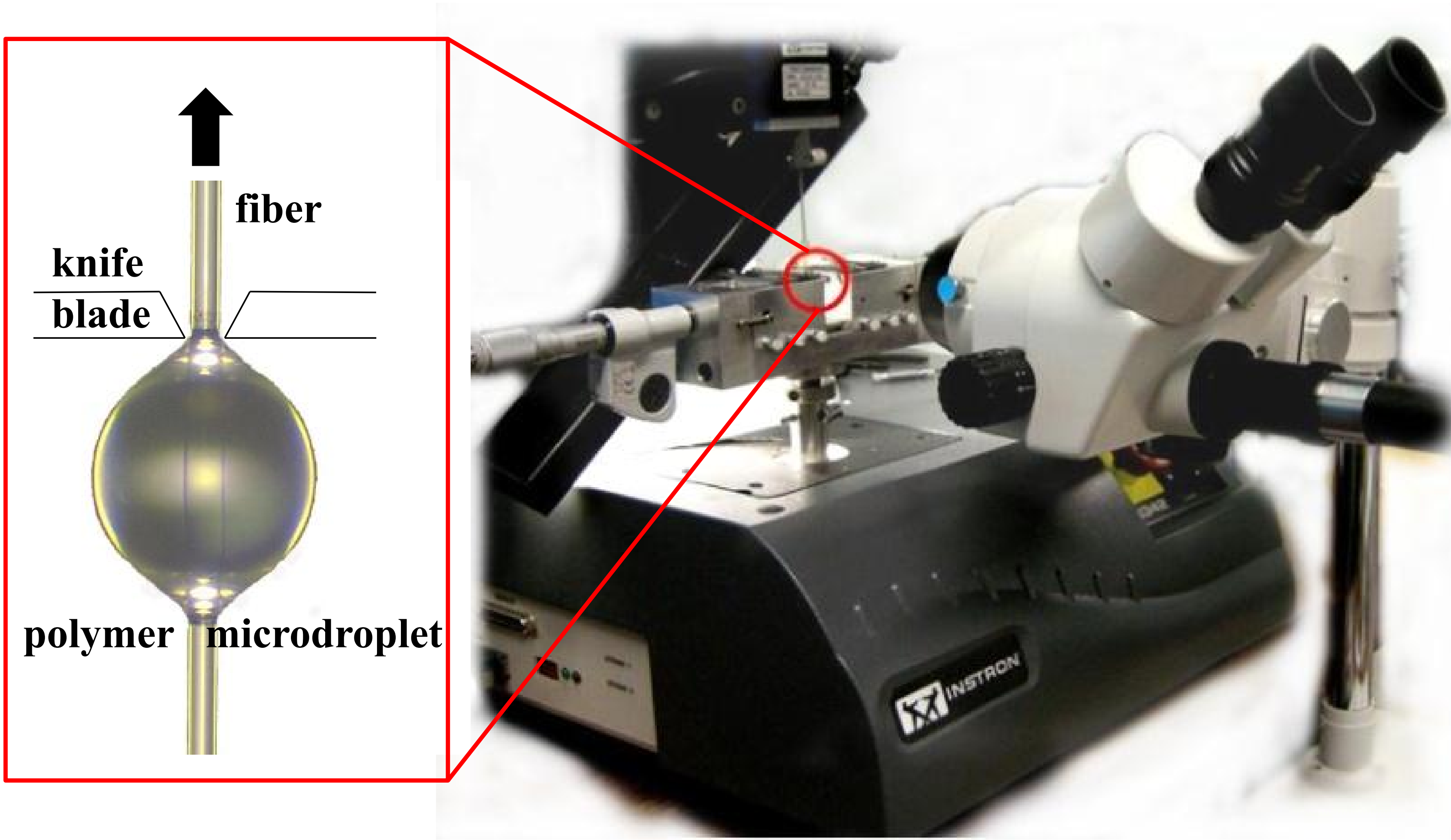
PP-Glass IFSS by Microbond Method



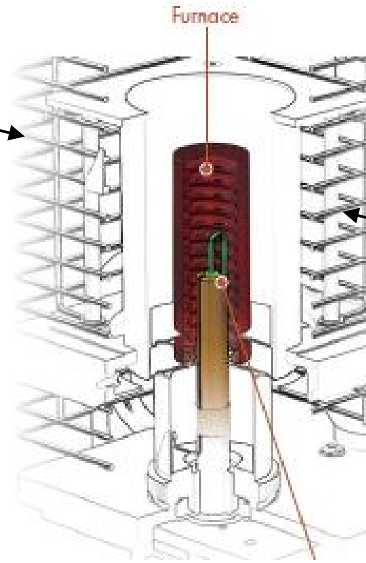
Now need to run test at different temperatures

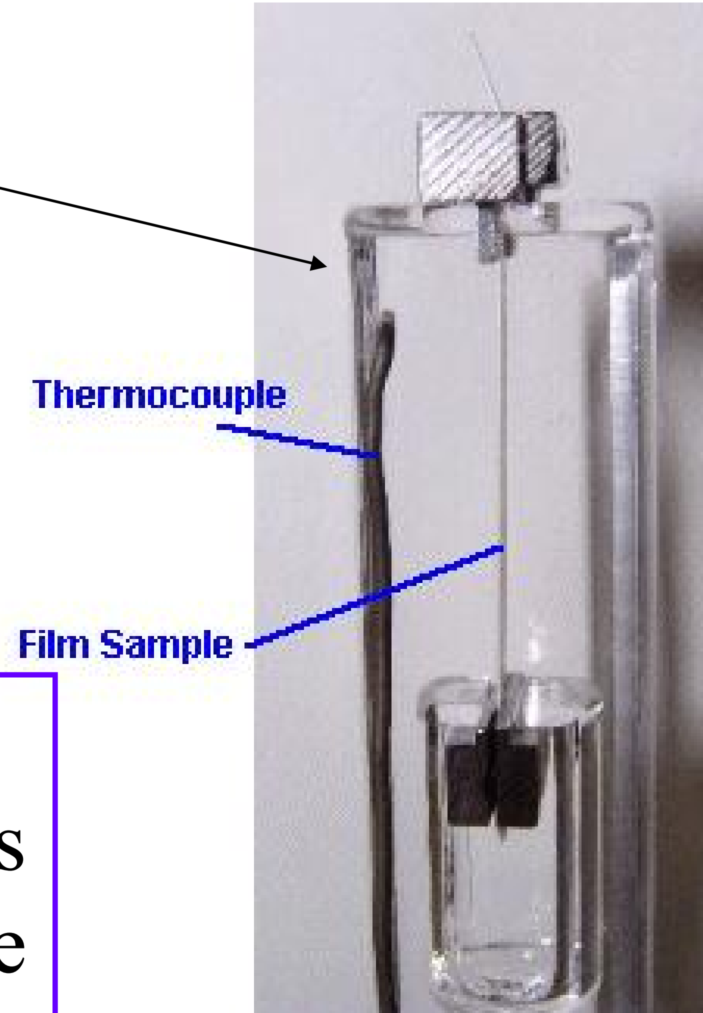
Microbond Test for IFSS



Accurate test environment temperature control is a challenge

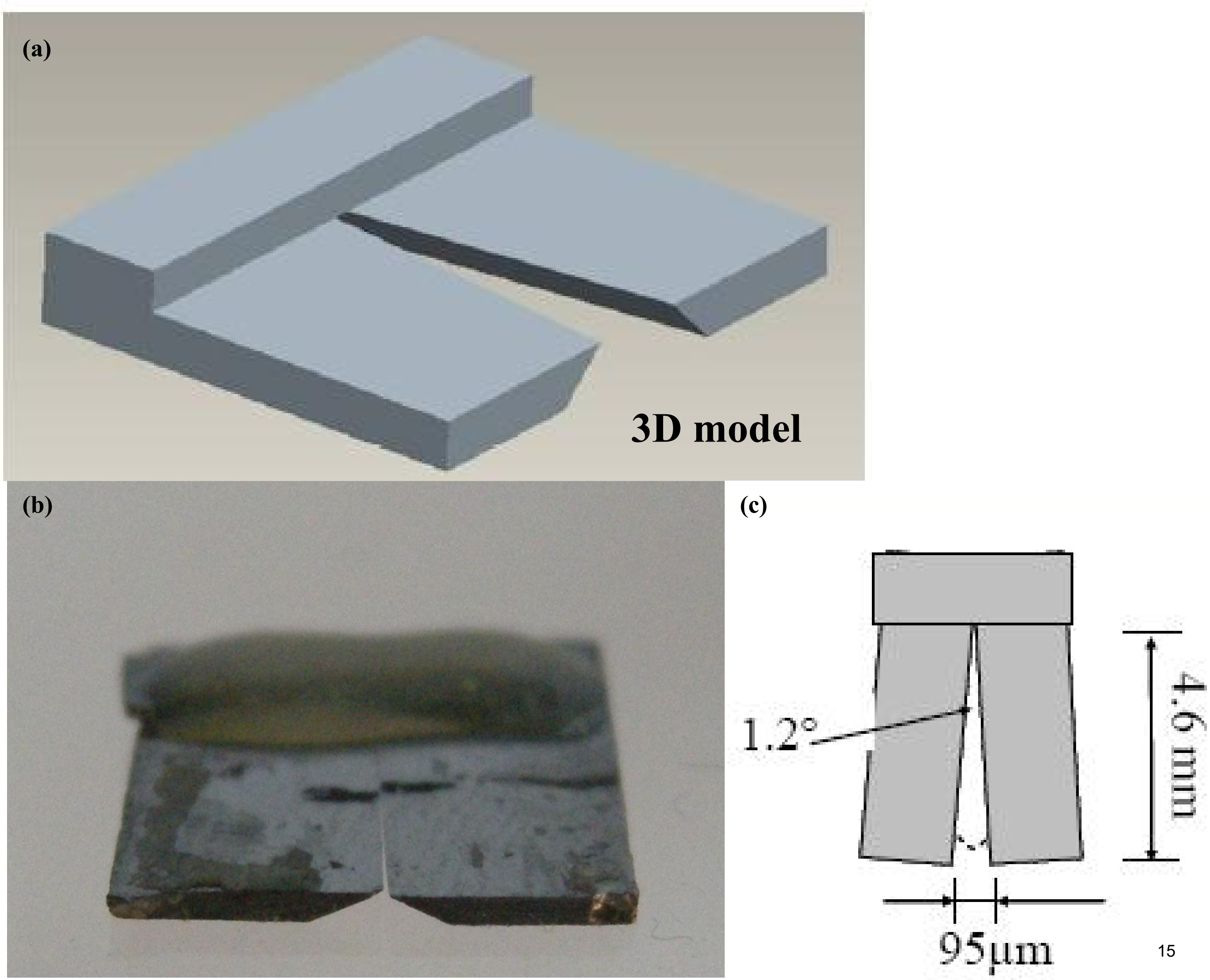
Q400 EM TMA in Fiber/Film Mode



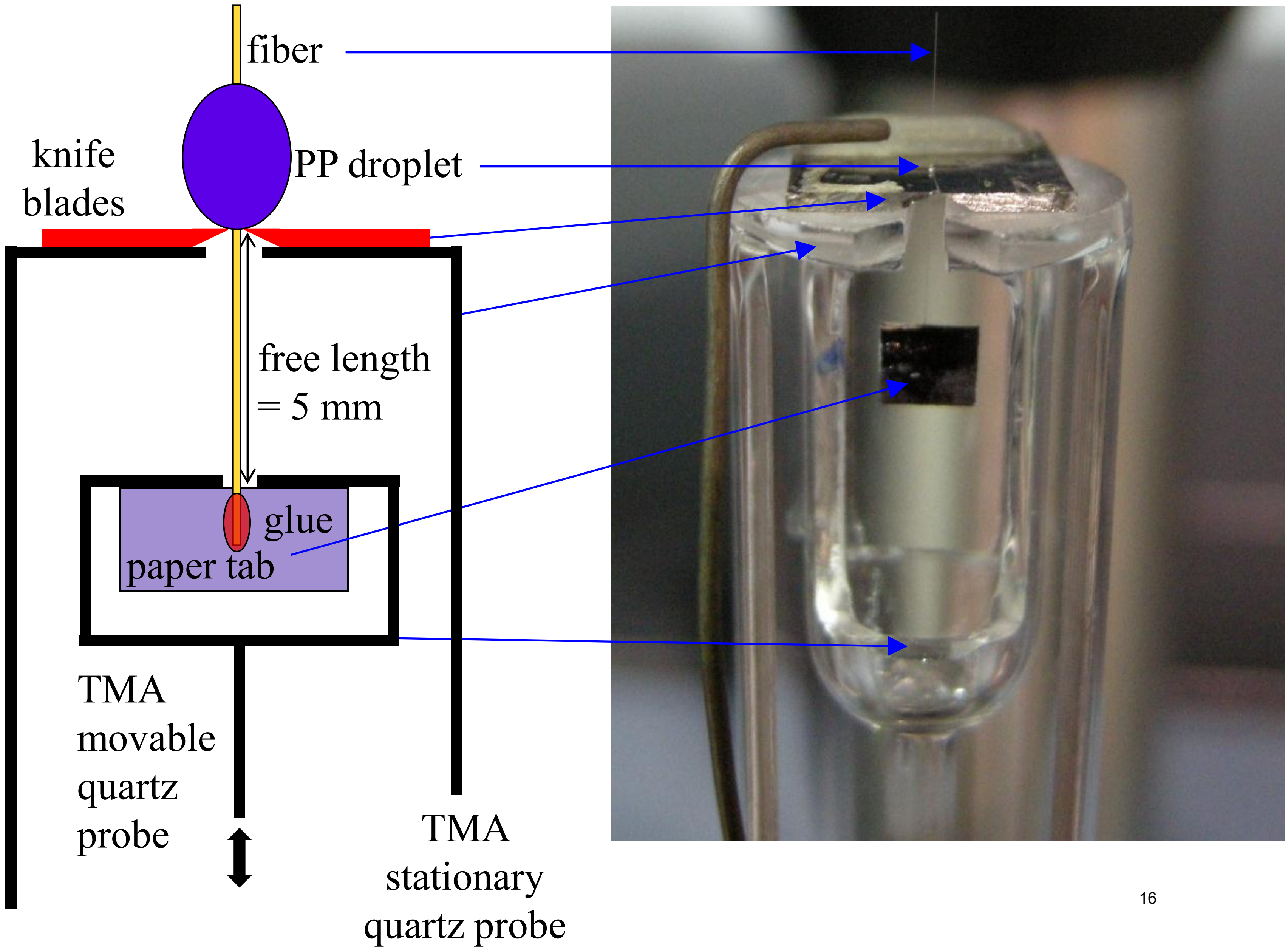


Strain-stress experiments possible on fiber/film samples in well controlled temperature environment

Knife Blades for TMA Microbond



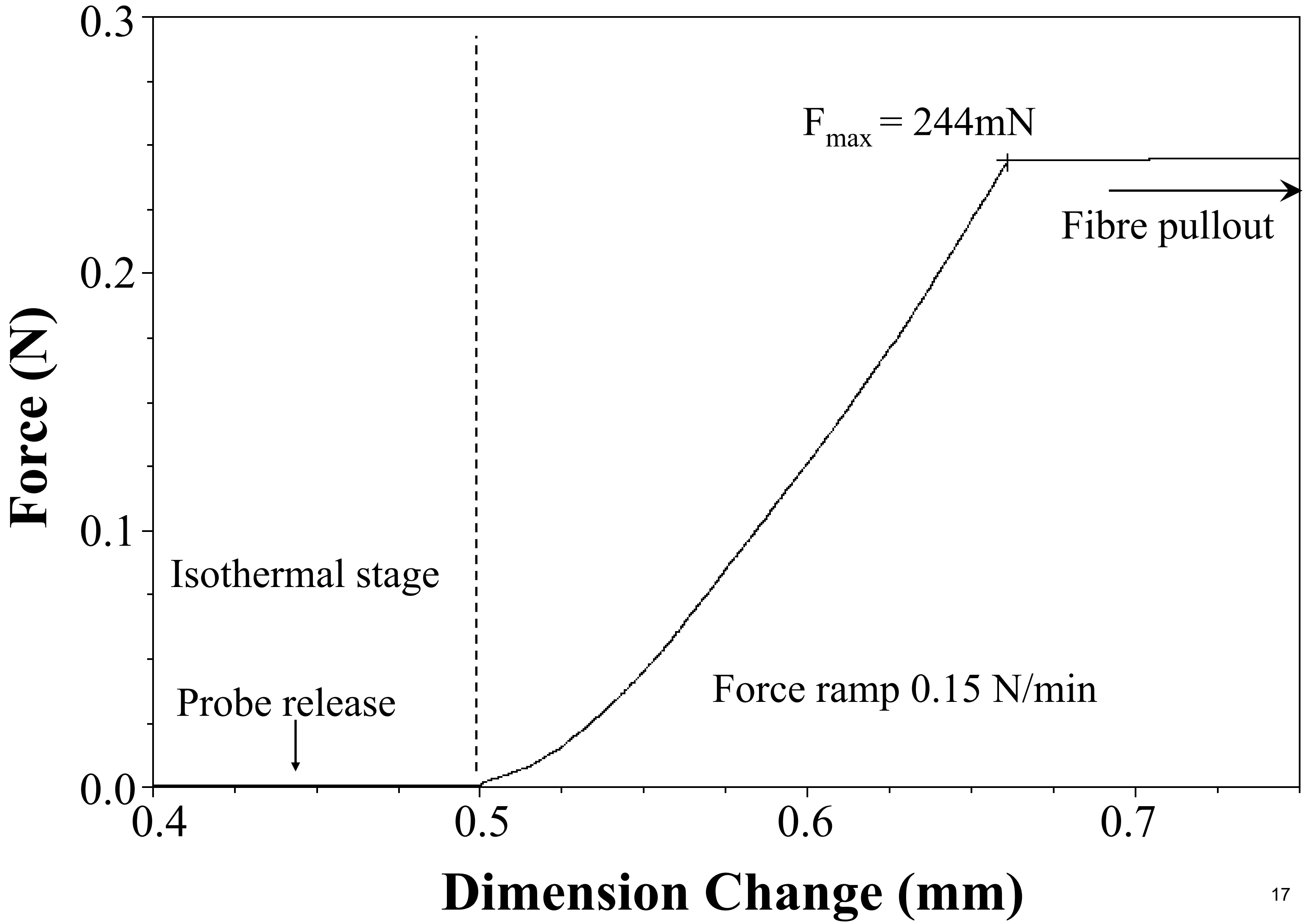
TMA Microbond Test Configuration



TMA Microbond Test

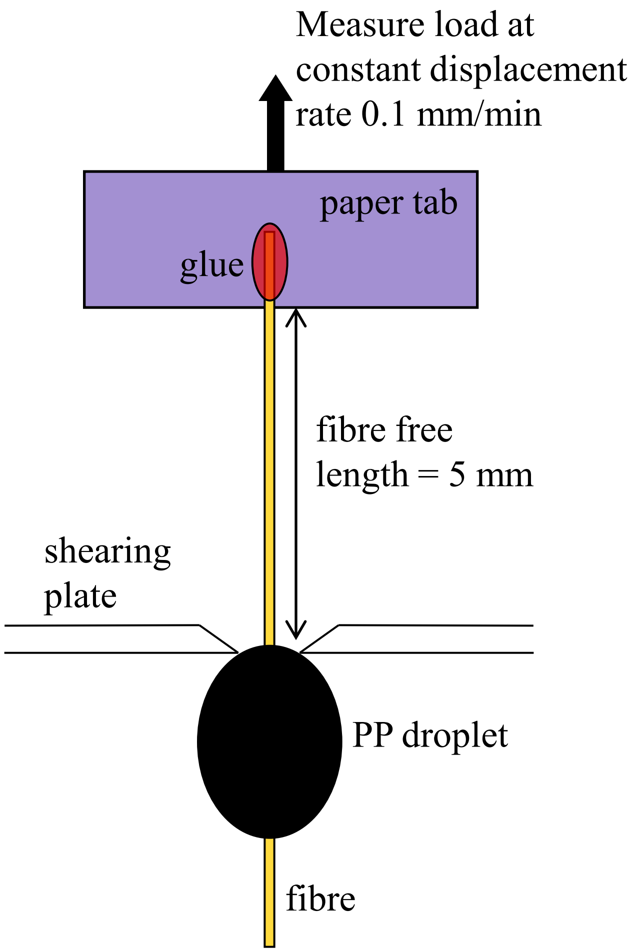
Isothermal at test temperature,

Ramp force (0.15 N/min) and measure displacement

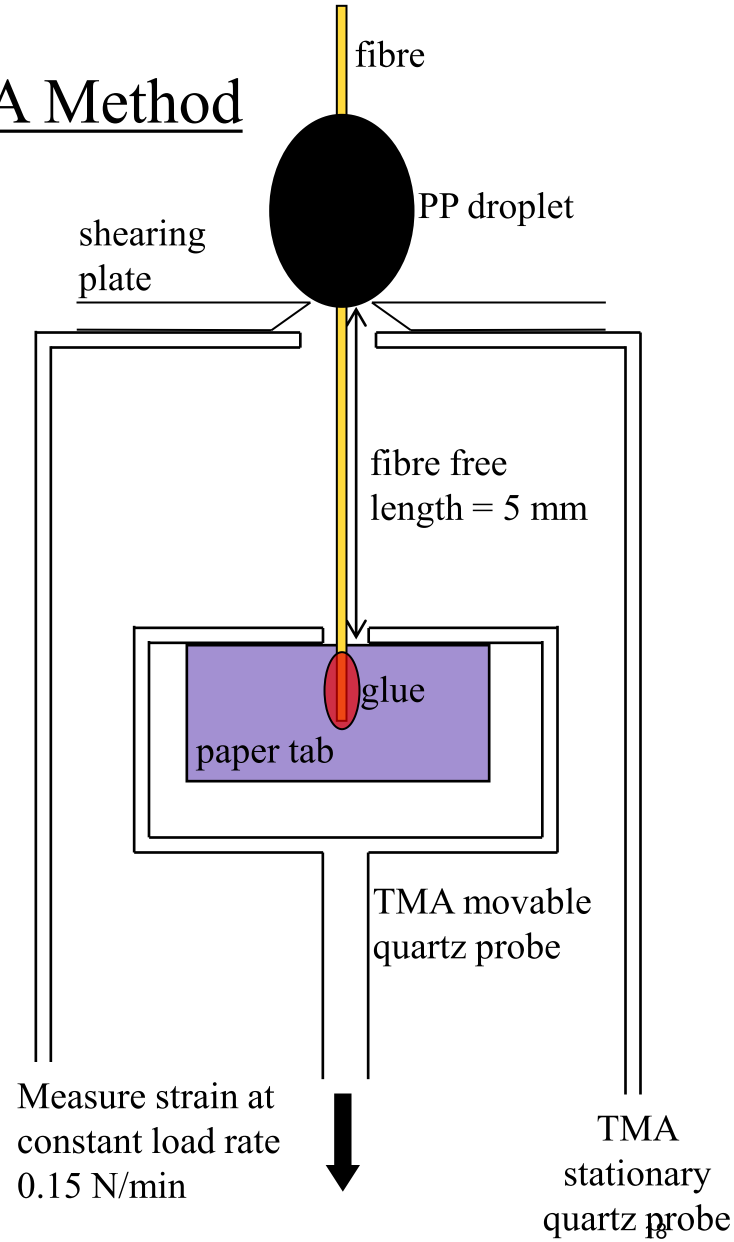


Comparison Microbond Test Configurations

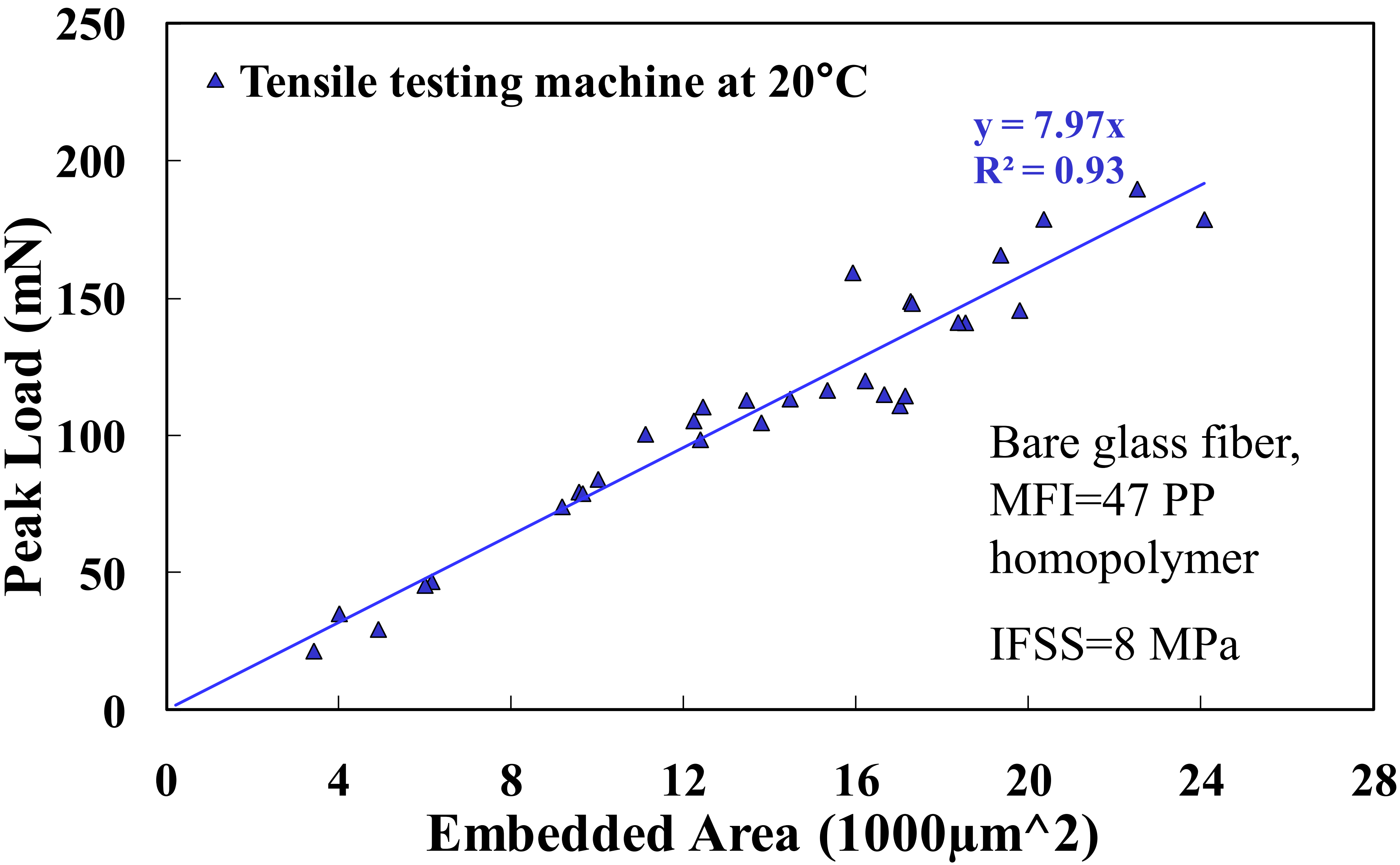
"Standard" Method



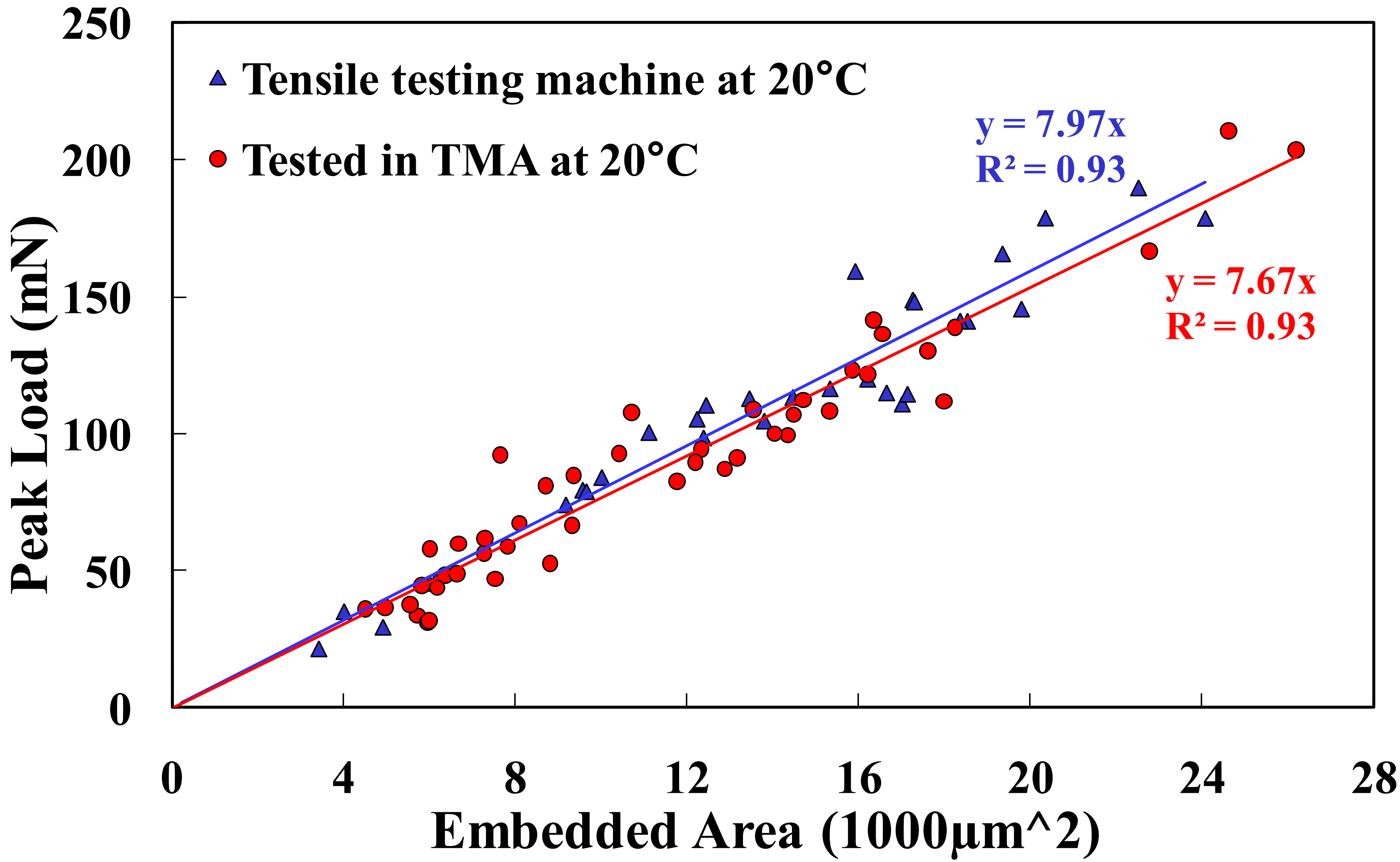
TMA Method



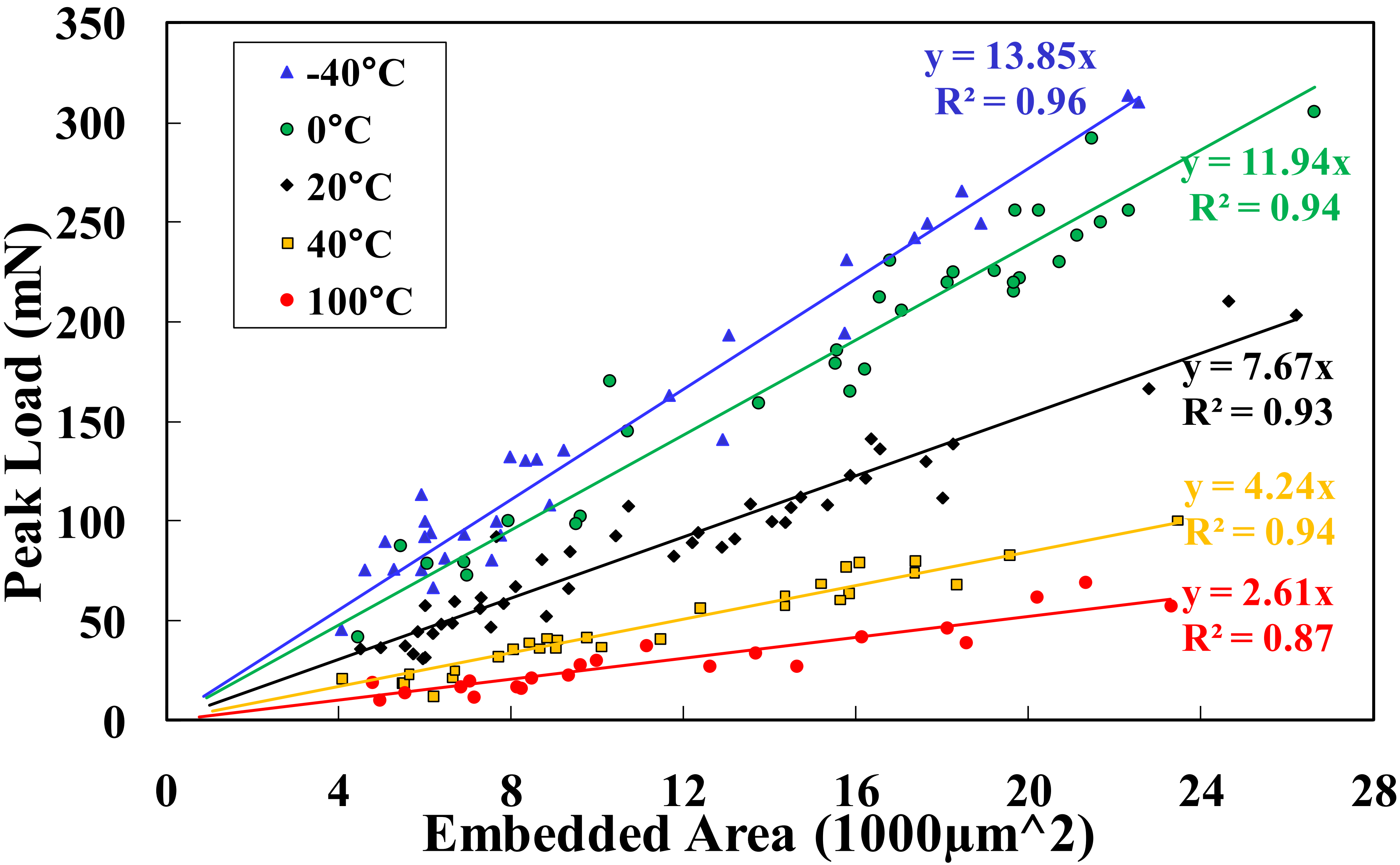
PP-Glass IFSS by Microdroplet Method



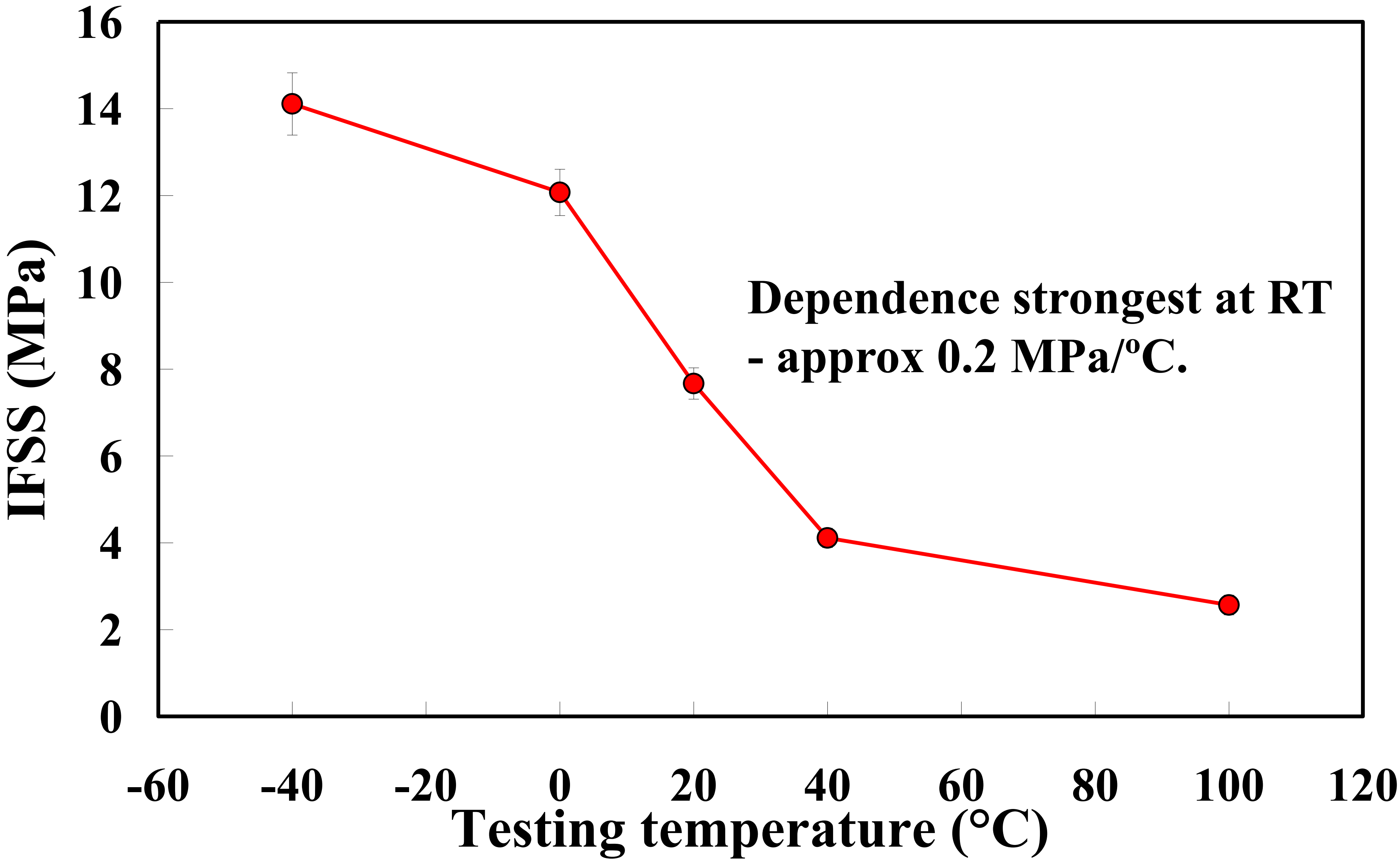
PP-Glass IFSS by TMA Microdroplet Method



PP-Glass IFSS by TMA Microbond Method



PP-Glass IFSS vs Test Temperature



Model Residual Thermal Stress at Interface

$$\sigma_{rm} = E_m(\alpha_m - \alpha_{fT})\Delta T$$

$$\sigma_{rm} = A_1(1 - b^2/r^2) \ \ where \ \ b = F(V_f)$$

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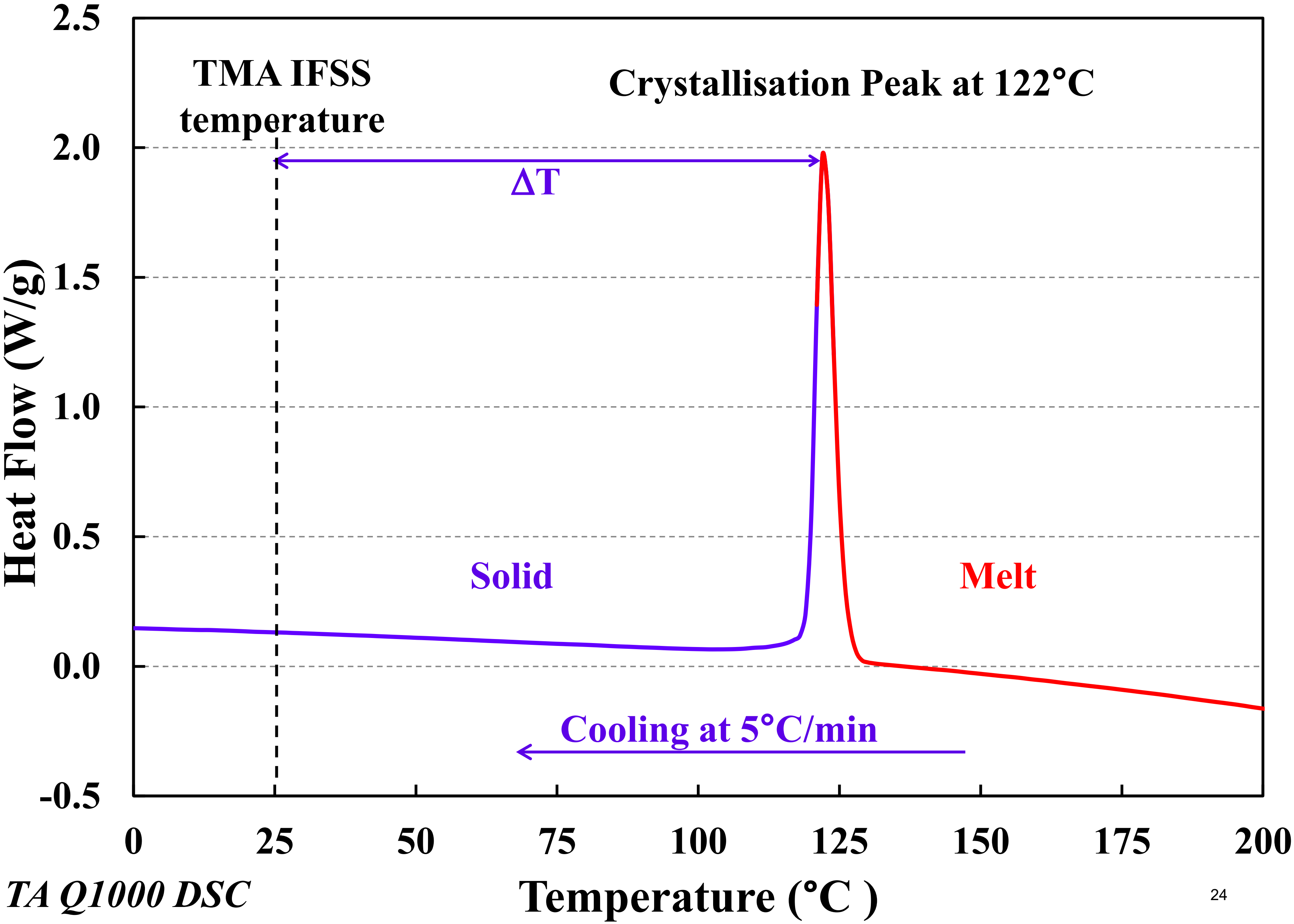
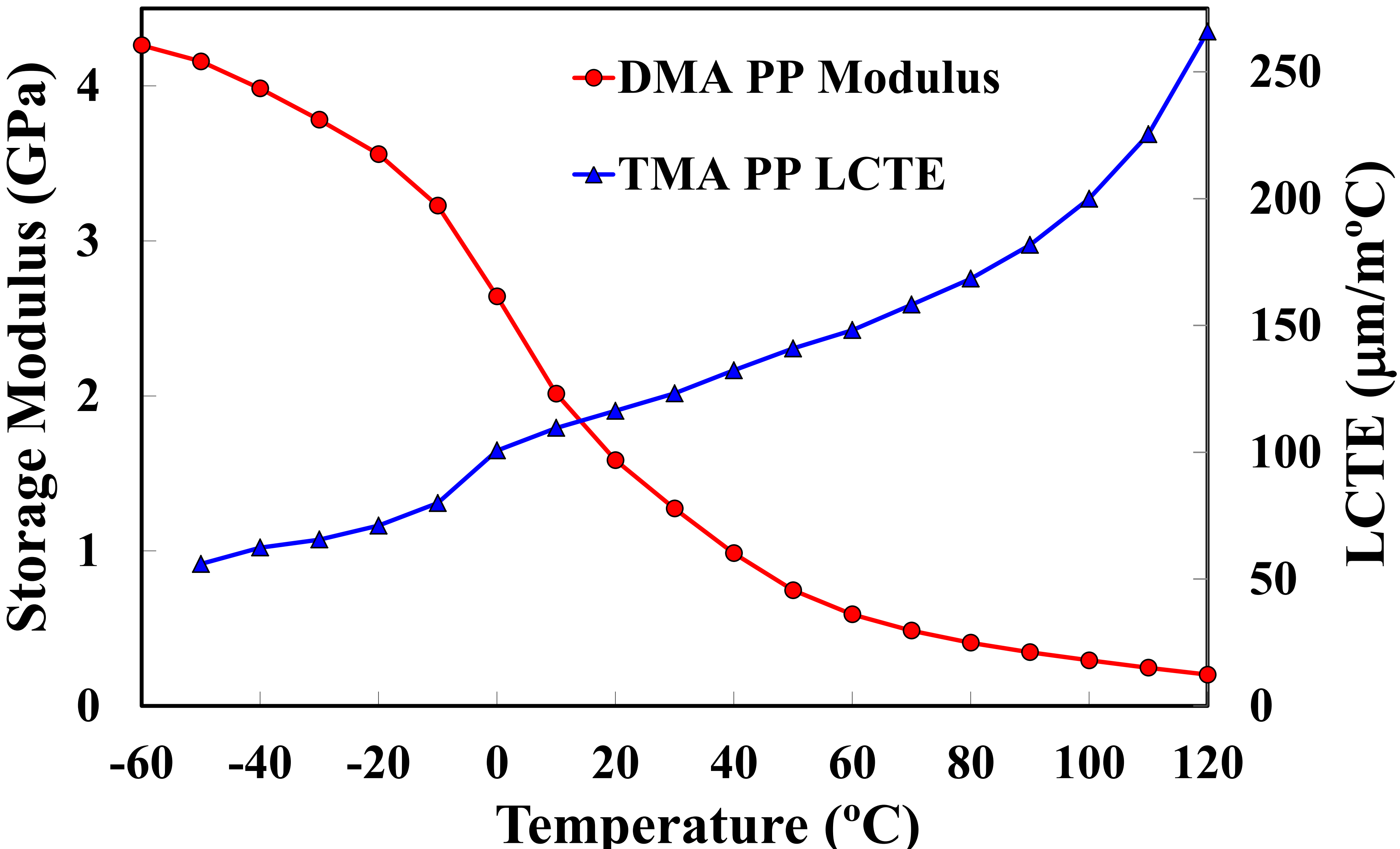
In both cases $\boldsymbol{\sigma_{rm} \propto \Delta T} \Rightarrow$ <u>IFSS $\propto \Delta T$</u>

DSC Cooling Run for PP Solidifcation



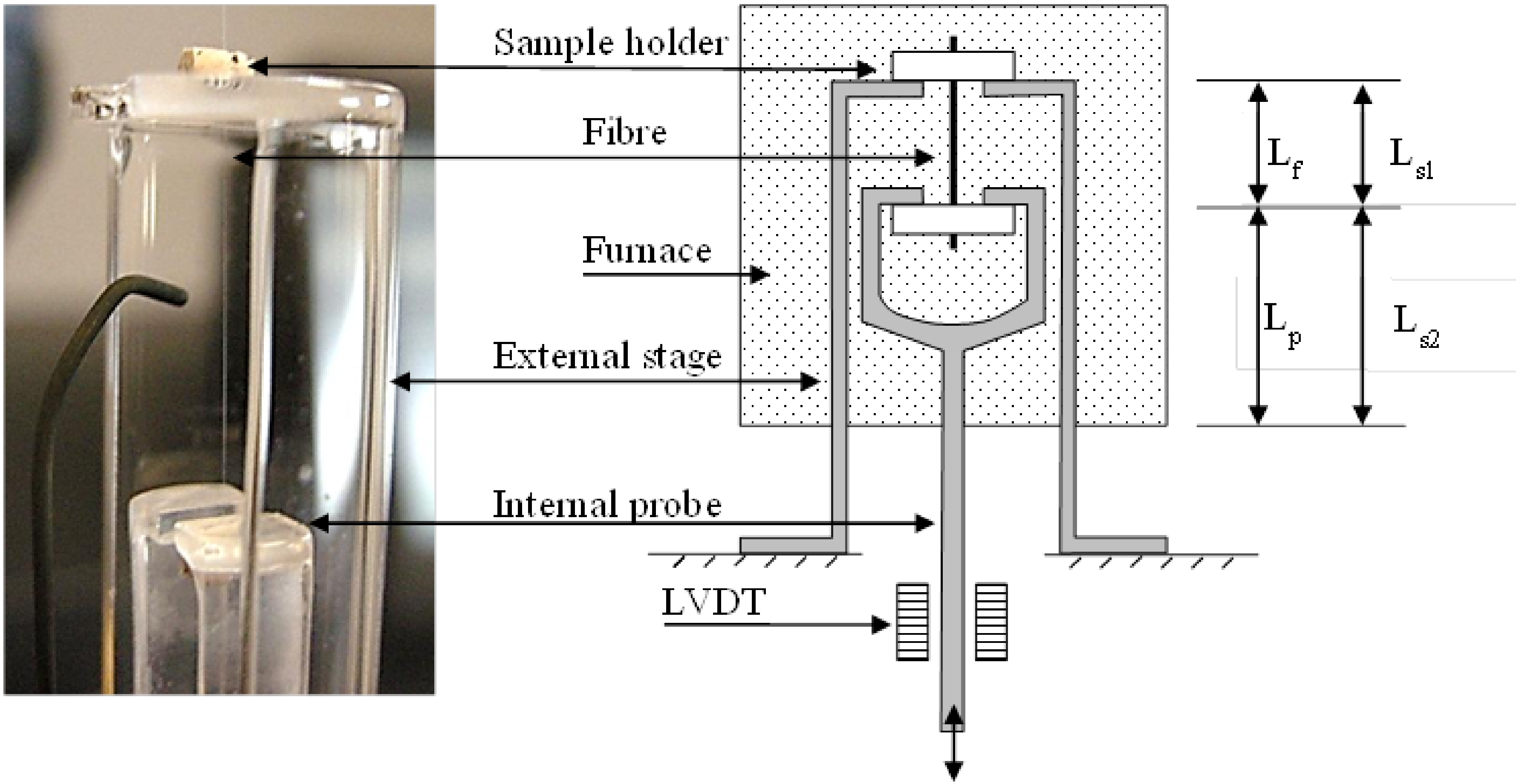
TA Q1000 DSC

Input for Residual Thermal Stress Model

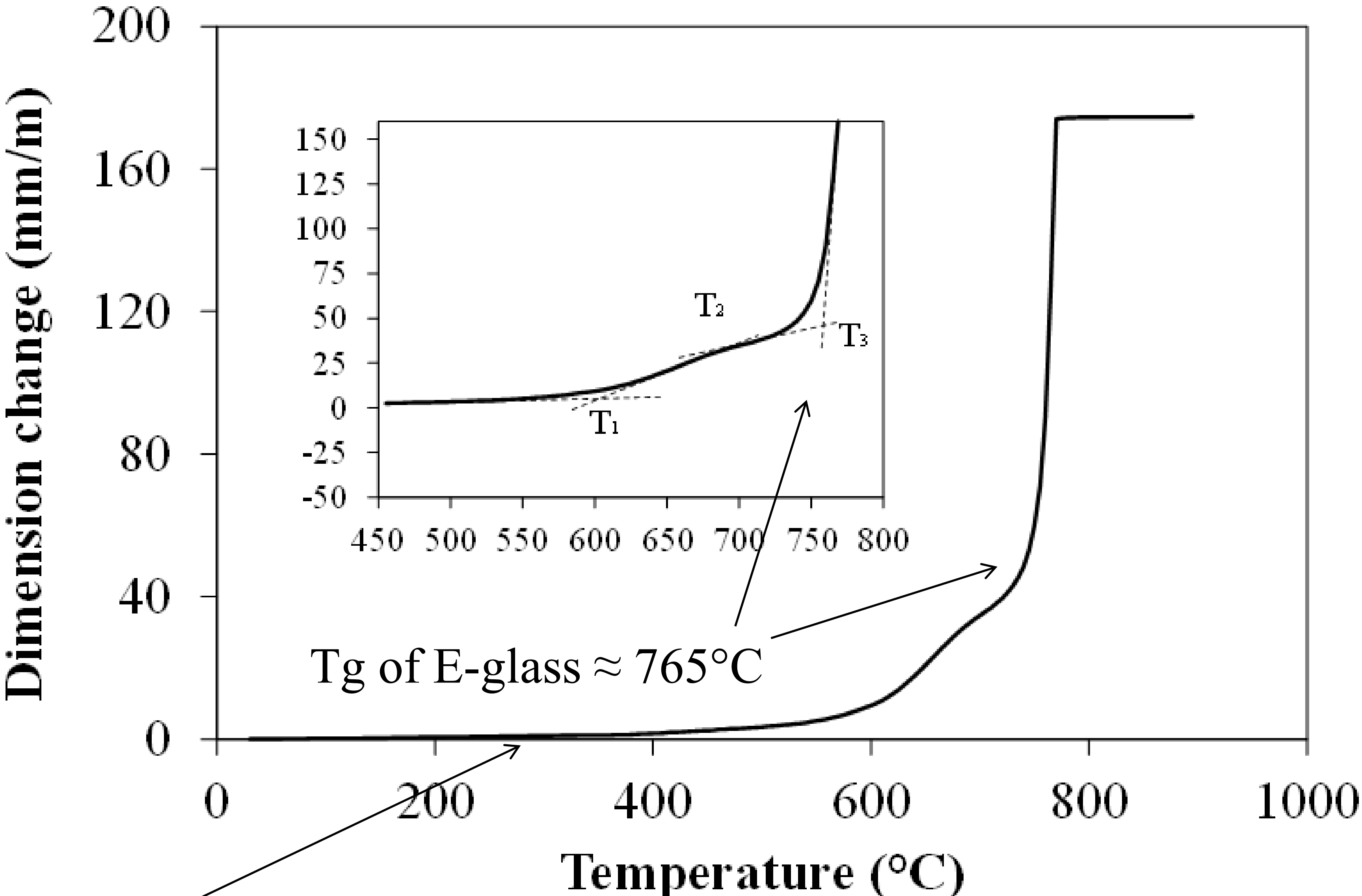


TA Q800 DMA
TA Q400 TMA

TMA of Single Glass Fibre in Q400 EM



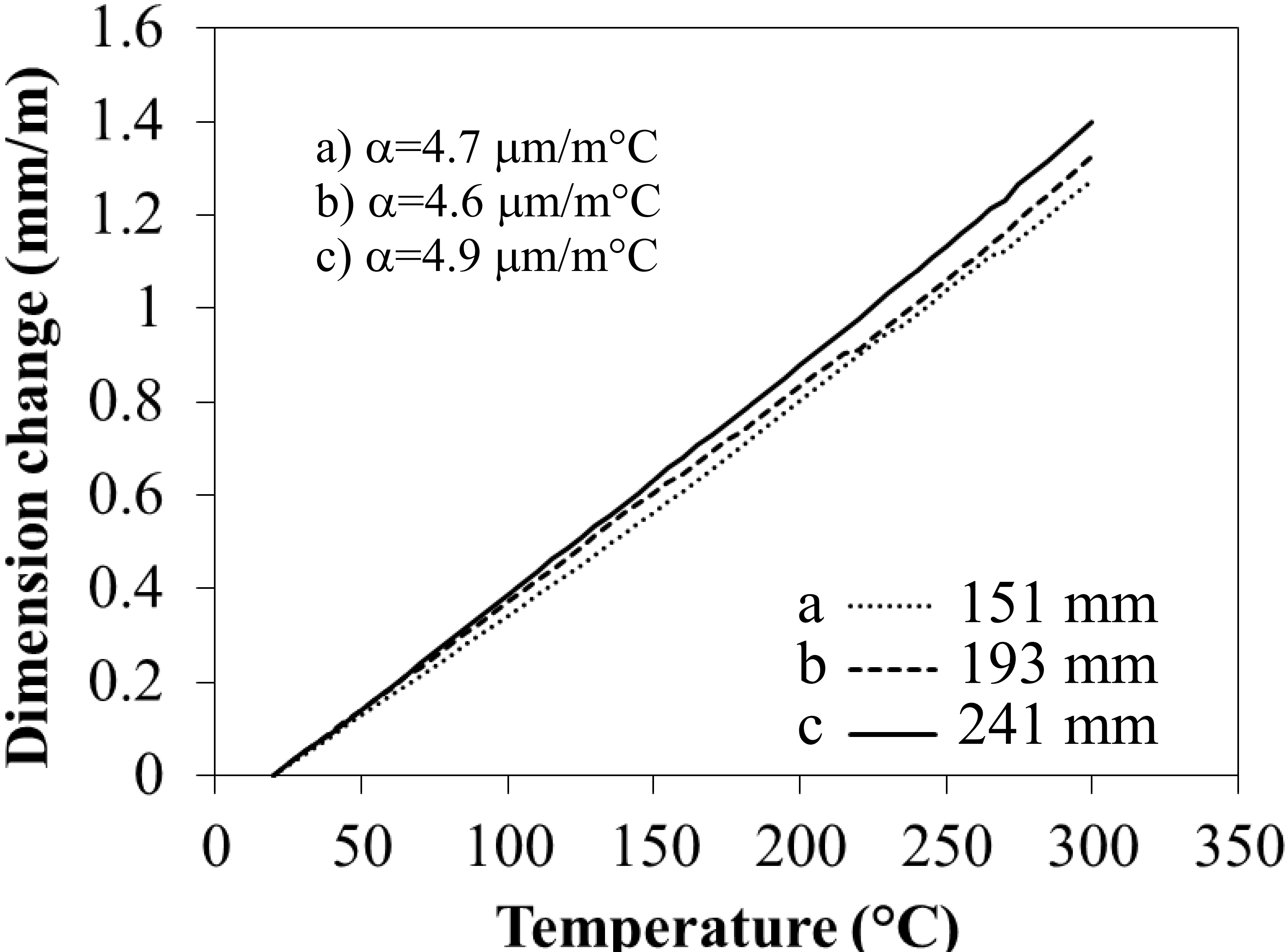
TMA of Single Glass Fibre in Q400 EM



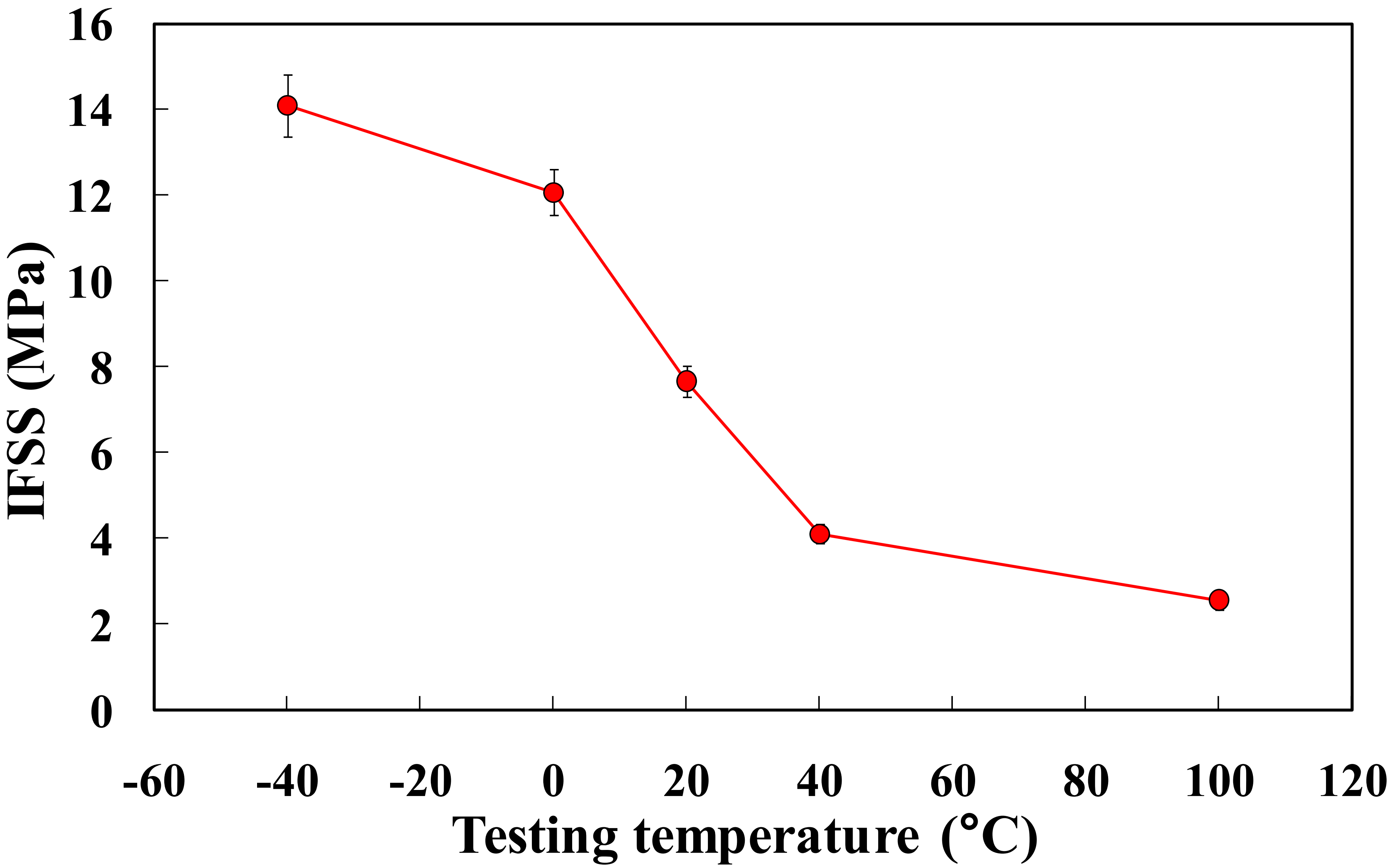
Tg of E-glass ≈ 765°C

Linear region (23-300°C) for LCTE

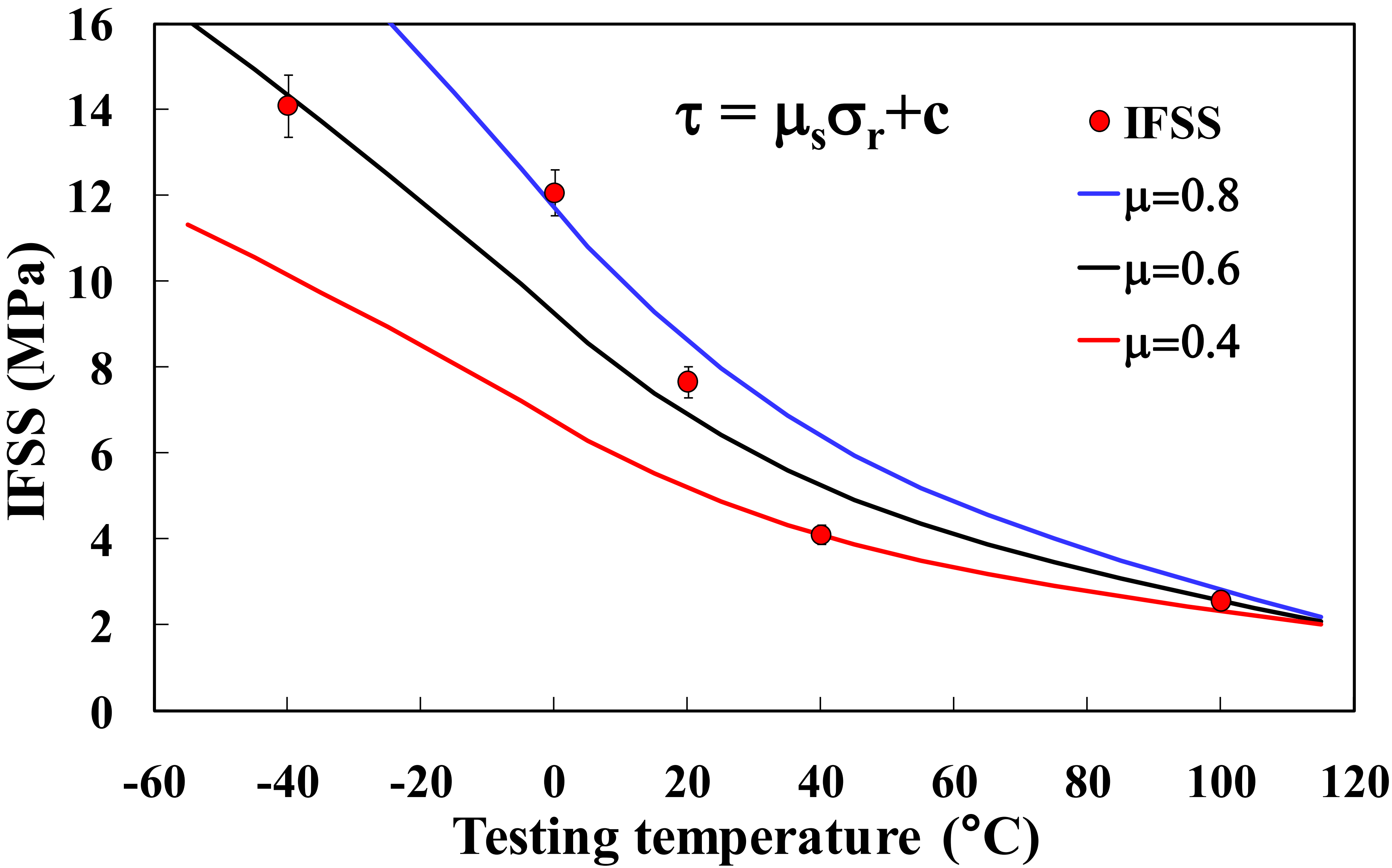
LCTE of Single Glass Fibre in Q400 EM TMA



PP-Glass IFSS vs Test Temperature



PP-Glass IFSS vs Test Temperature



Schoolenberg 1995, GF-PP static coefficient of friction = 0.65

Conclusions (GF-PP)

- **Residual compressive stress at the composite interface may contribute significantly to the apparent IFSS**
- **Magnitude of these stresses is strongly influenced by test temperature vs the solidification temperature**
- **This results in an expectation that thermoplastic composite IFSS *should be* temperature dependent**
- **The TMA microbond test has enabled measurement of the IFSS of GF–PP from -40°C up to 100 °C**
- **A strong dependence of GF-PP Interfacial Strength on Test Temperature has been observed**

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Conclusions (GF-PP)

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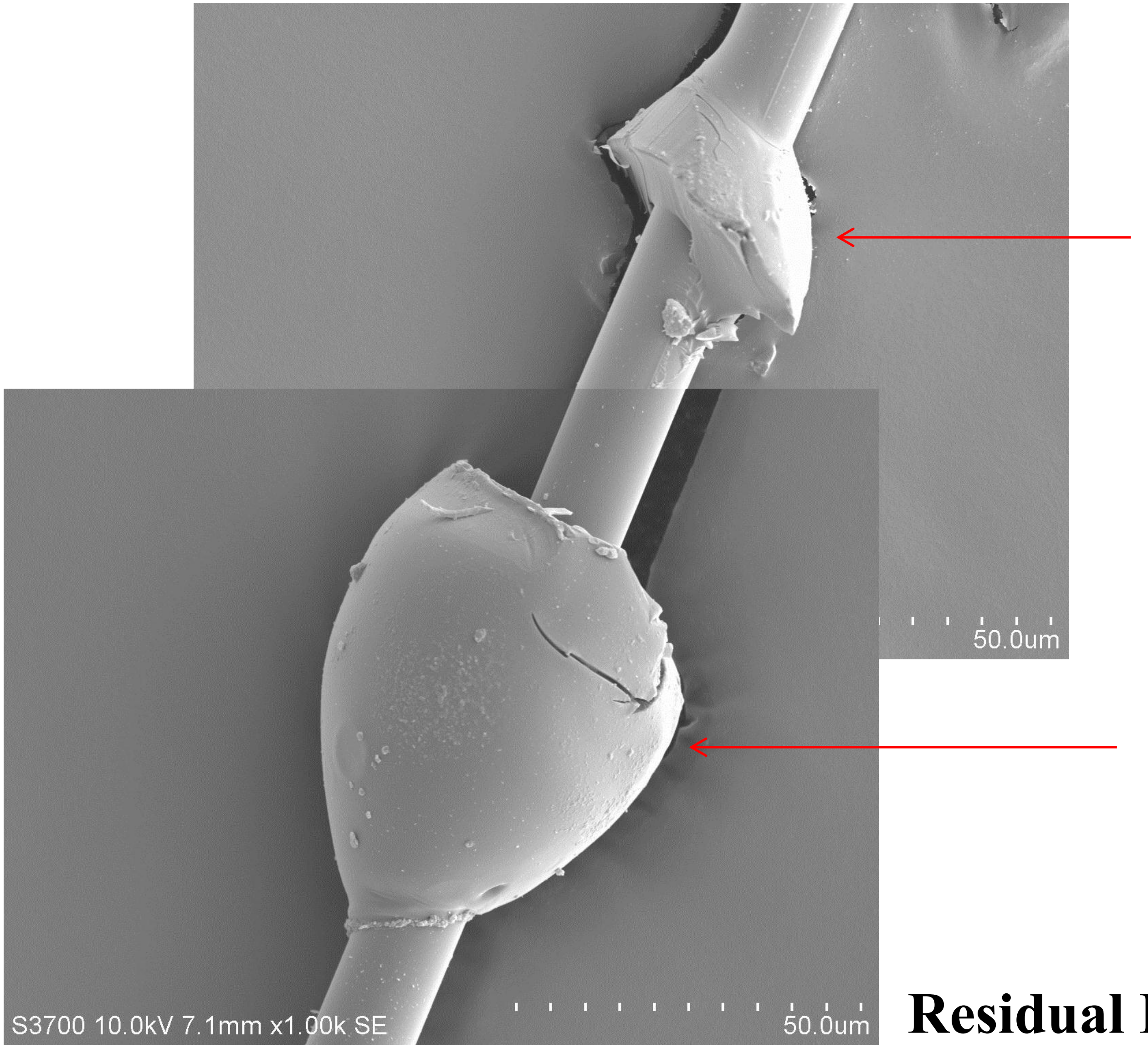
- **<u>A strong dependence of GF-PP Interfacial Strength on Test Temperature has been observed</u>**

IFSS in Other Composite Systems ?

- **IFSS correlates strongly with radial residual compressive stress in GF-PP**
- **GF-PP is a relatively low adhesion system**
- **What about other composite systems with high adhesion?**
  - **GF-Epoxy ?**
  - **GF-Polyamide ?**

GF-Epoxy IFSS by TMA Microbond

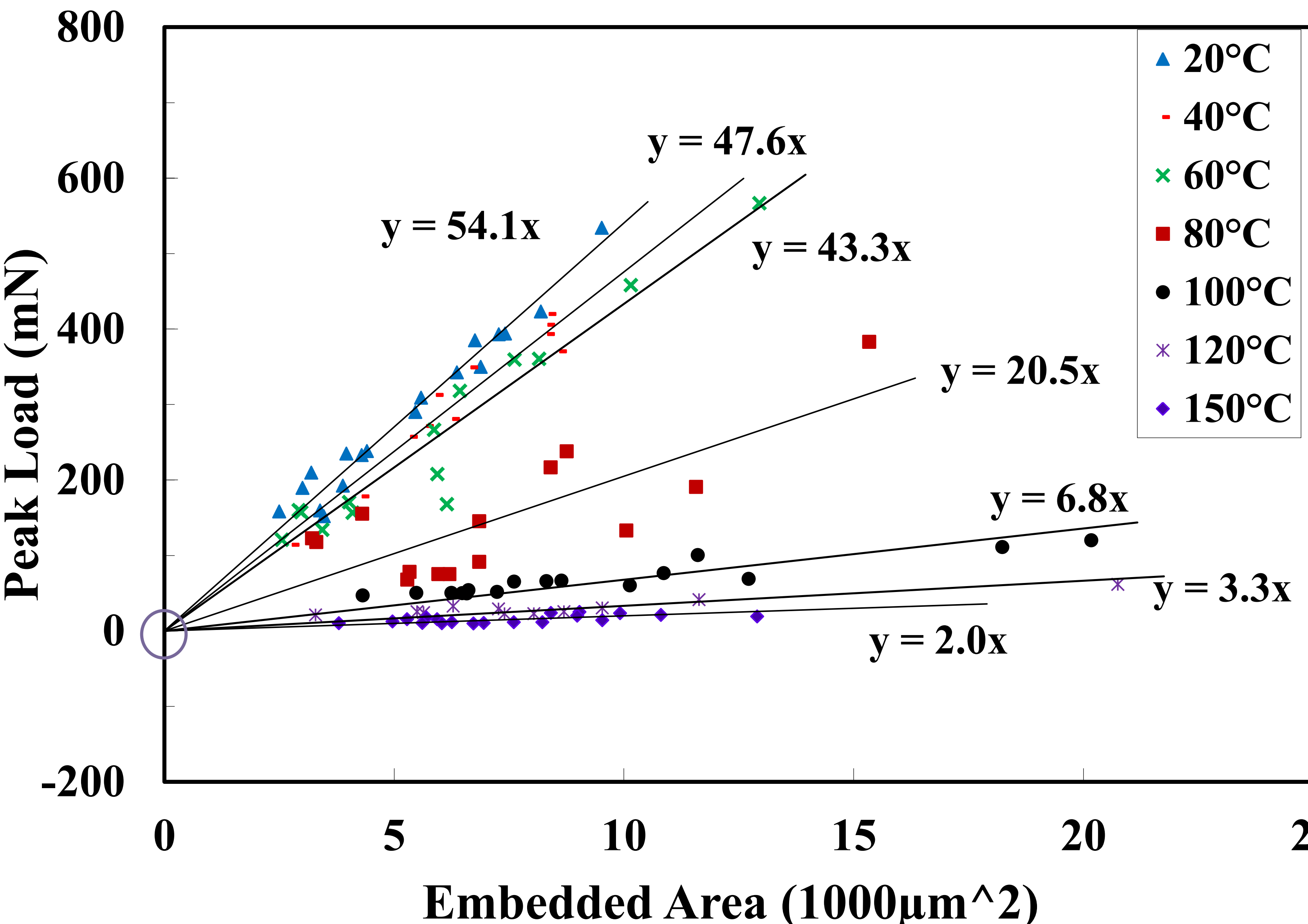


Nondebonded part

Debonded part

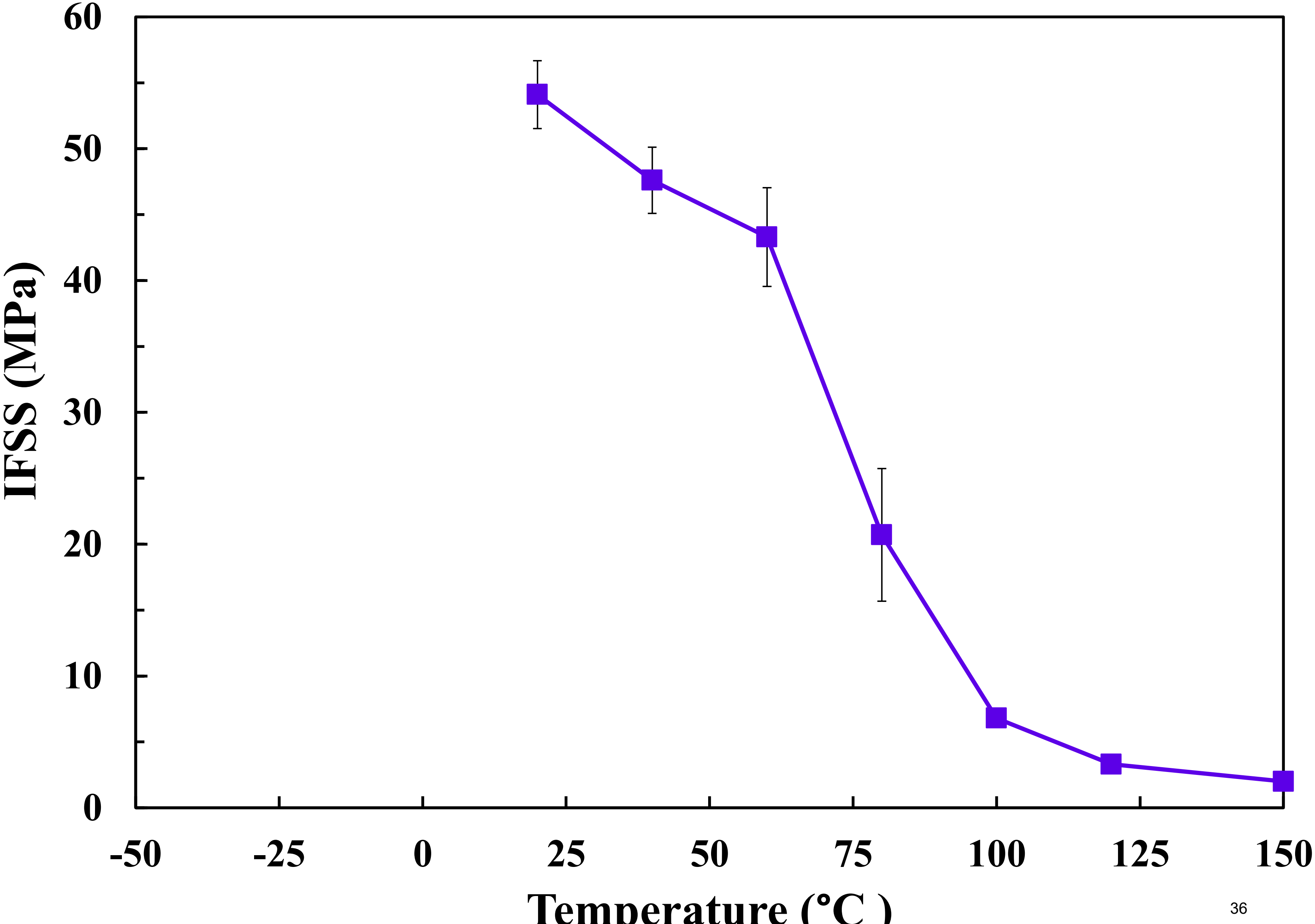
Residual Le << Original Le

GF-Epoxy IFSS by TMA Microbond

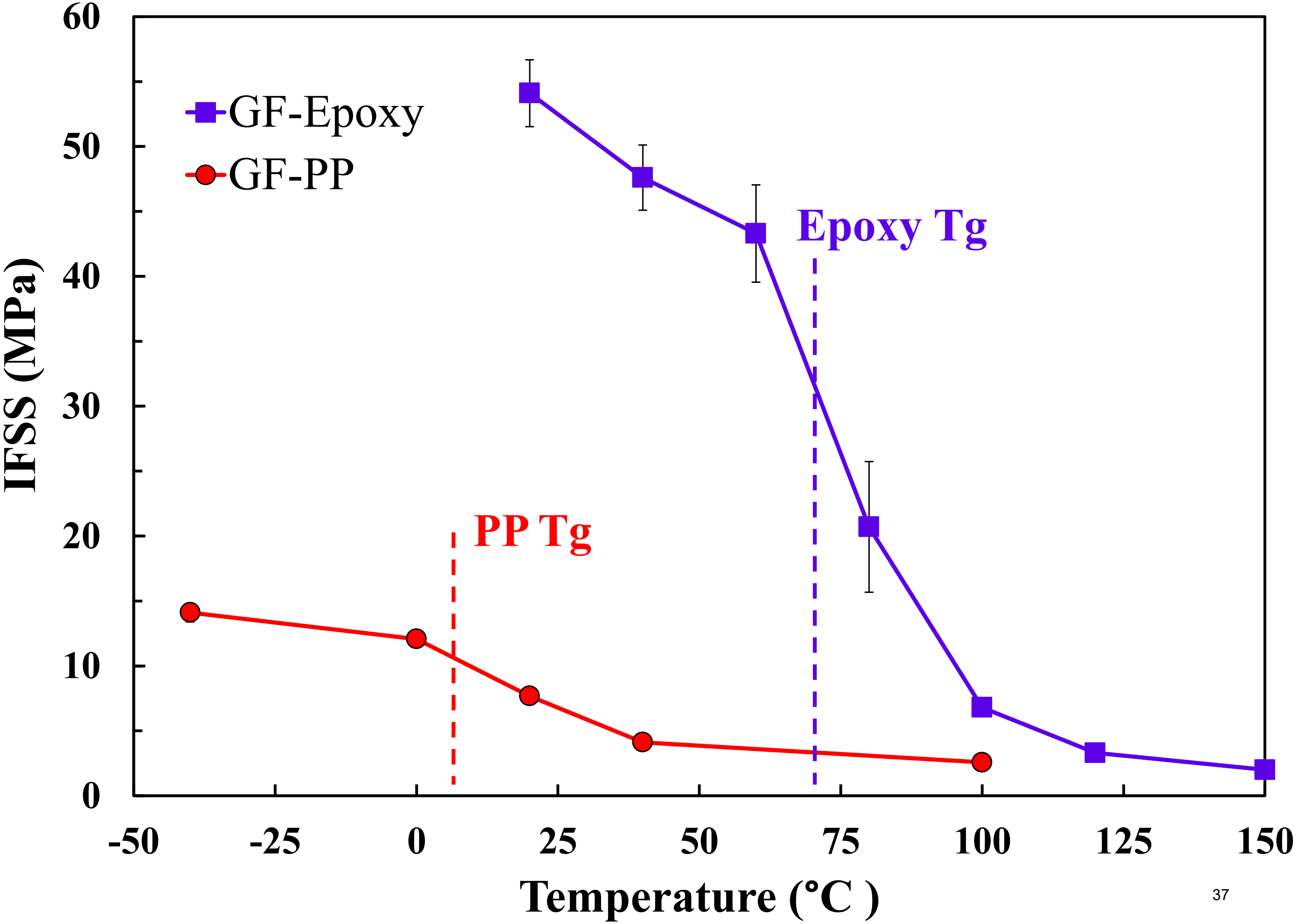


Epoxy system DGEBA&TETA

GF-Epoxy IFSS



GF-Polymer IFSS



Conclusions

- **Thermal analysis equipment can provide an excellent temperature controlled environment in which to carry out micromechanical testing**
- **A strong dependence of Interfacial Shear Strength on Test Temperature has been observed in both GF-PP and GF-Epoxy Composites**
- **Residual compressive stress at the composite interface may contribute significantly to the apparent IFSS in GF-PP**

Future Work

- **Adhesion in other Composite Systems**
  - Polyamide – Glass Fibre
  - Epoxy – Carbon Fibre
- **Test Development**
  - Microbond in the Q800 DMA ?
  - Microbond-DMA vs Temperature & Humidity