Practice in testing of materials

A short notes for beginners to survive

Version (20210408A)

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Thermal test

- Thermal gravimetric analysis (TGA)
- Differential Scanning Calorimetry (DSC)
- Dynamic Mechanical Analyser (DMA)

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TGA

https://en.wikipedia.org/wiki/Thermogravimetric_analysis

Thermogravimetric analysis or **thermal gravimetric analysis** (**TGA**) is a method of thermal analysis in which the mass of a sample is measured over time as the temperature changes. This measurement provides information about physical phenomena, such as phase transitions, absorption, adsorption and desorption; as well as chemical phenomena including chemisorptions, thermal decomposition, and solid-gas reactions (e.g., oxidation or reduction).

We can use it

- to determine the water content in a polymer (right figure: we estimate that at 260°C the polyurethane is fully dried).
- to find the maximum heating temperature to avoid decomposition.

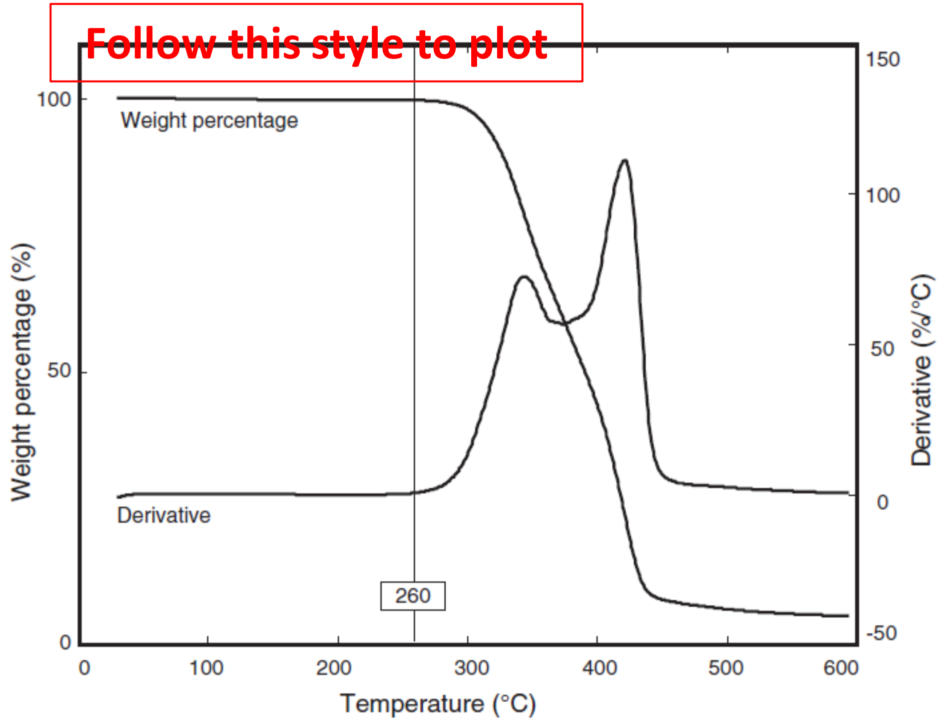

Figure 10: Typical thermogravimetric analysis (TGA) result of polyurethane (PU). (Reproduced from [29] with permission from The Royal Society of Chemistry).

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DSC

https://en.wikipedia.org/wiki/Differential_scanning_calorimetry

Normally, two cycles at a heating/cooling speed of 10°C/min. The result of the 1st cycle may be affected by the "history".

We can find

- Tg (glass transition)
- Tm/Tc (melting/crystallization)

of a material.

The heat flow (vertical axis) should be in w/g to avoid the size effect.

Follow this style to plot

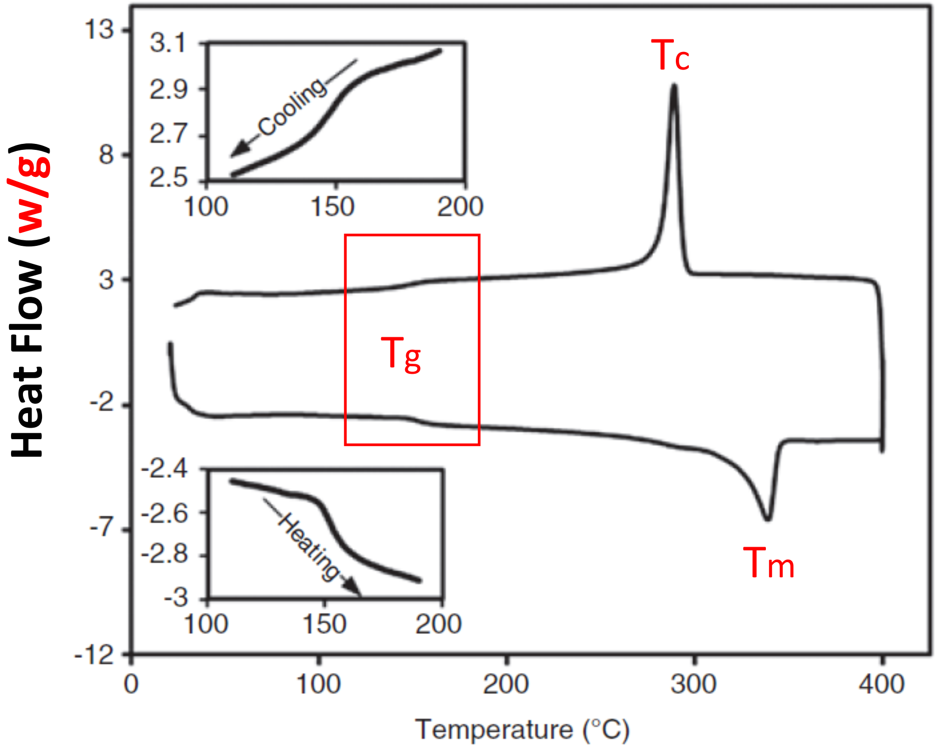

Figure 11: Differential scanning calorimetry (DSC) result of poly(ether ether ketone) (PEEK). Insets: zoom-in view of the glass transition range in heating (bottom) and cooling (top). (Reproduced from [15] with permission from Wiley Periodicals, Inc.).

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Cyclic DSC test is able to find the influence of “thermal” treatment.

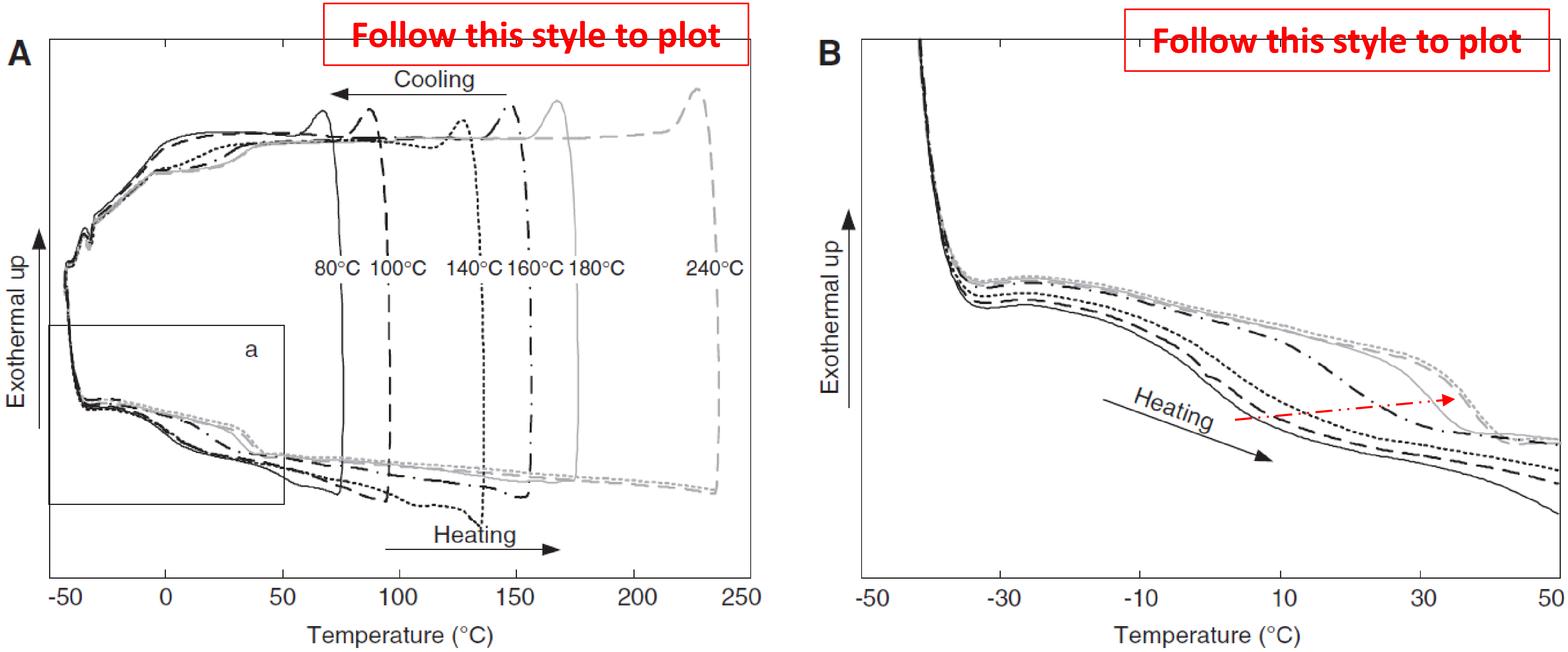

Figure 13: Cyclic differential scanning calorimetry (DSC) curves of water saturated polyurethane (PU). (A) Overall view (reproduced from [77] with permission from Elsevier Ltd.); (B) zoom-in of a.

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DMA

https://en.wikipedia.org/wiki/Dynamic_mechanical_analysis

The applied heating rate is normally around 15°C/min or the same as that used in DSC test.

This is a way to find softening of a material if you cannot find any apparent transition by DSC test within a particular temperature range.

This is very much useful in studying the shape memory effect of polymers to find the actual mechanism behind.

The modulus could be used as an estimation of the Young's modulus.

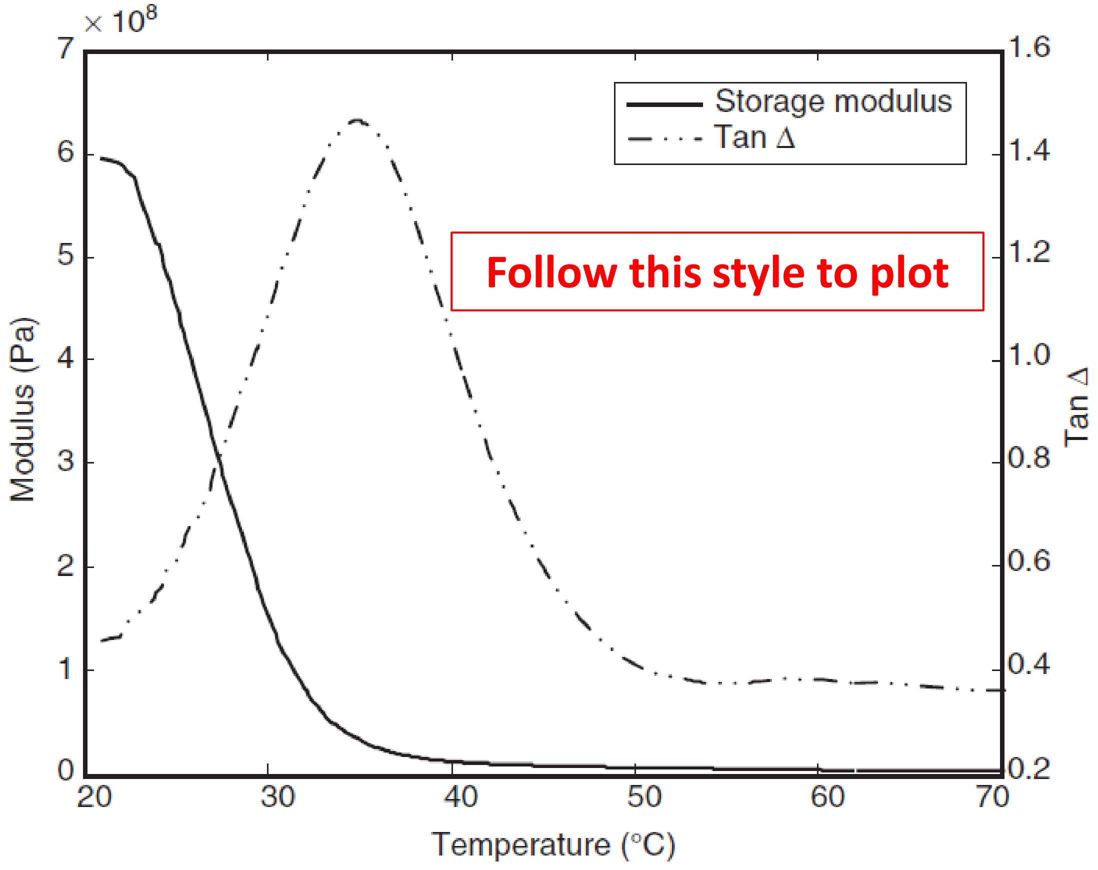


Figure 12: Typical dynamic mechanical analysis (DMA) result of polyurethane (PU). (Reproduced from [29] with permission from The Royal Society of Chemistry).

Mechanical test

Typical steps

- **Sample preparation**

  Strip, dog-bone, fibre/wire, cylindrical …

- **Testing parameters (load cell, loading speed, control)**
  - Estimate the maximum load, and find right load cell (less than 50x of max load)
  - Work out loading speed (based on the required **strain rate**)
  - Type of test and control

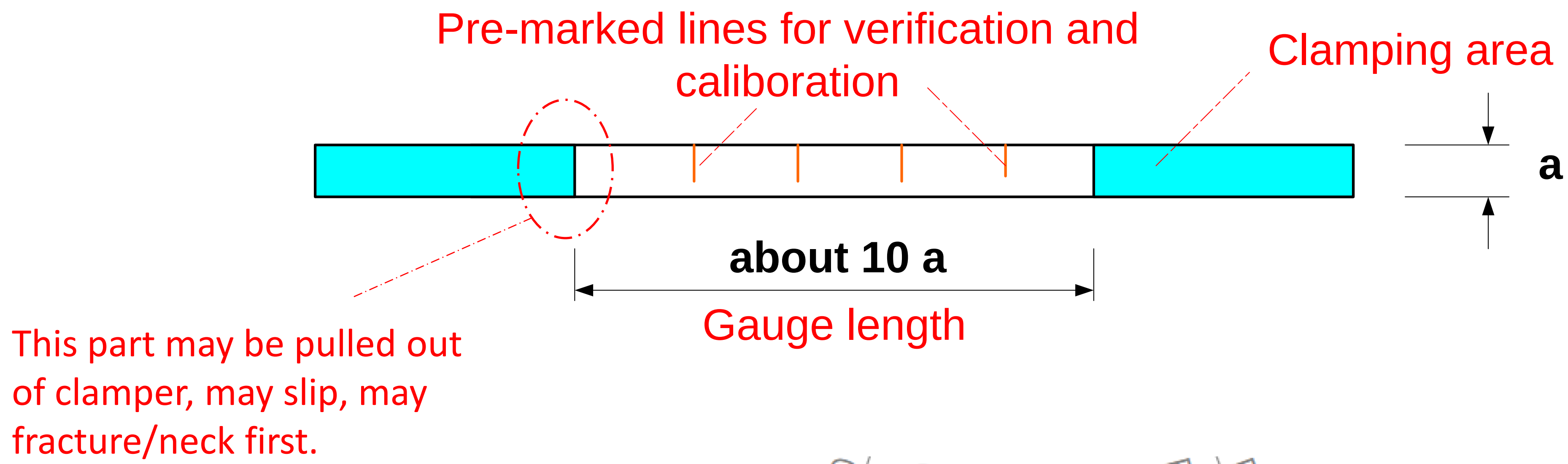
    Single stretching? fracture? cyclic? A cyclic fracture test is able to provide a good understanding of a material.

    Strain rate control (strain change/second) to avoid the size effect

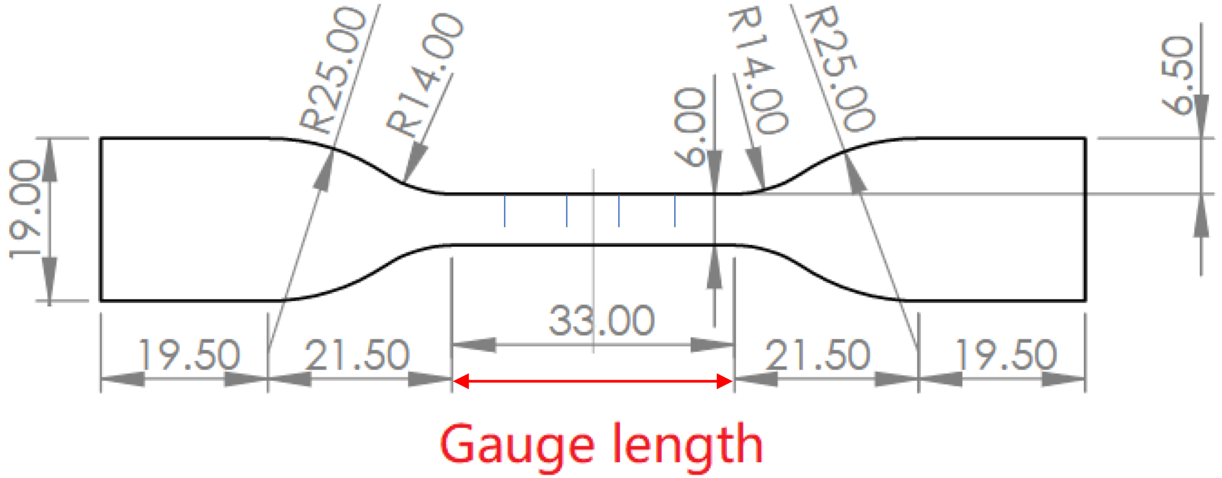
- **Results analysis**
  - Is the result reliable?

Samples

Strip



Dog-bone (typical dimension)



Engineering strain/stress or true/real stress/strain

$$\sigma_{true} = \sigma_{engineering} * (1 + \varepsilon_{engineering})$$

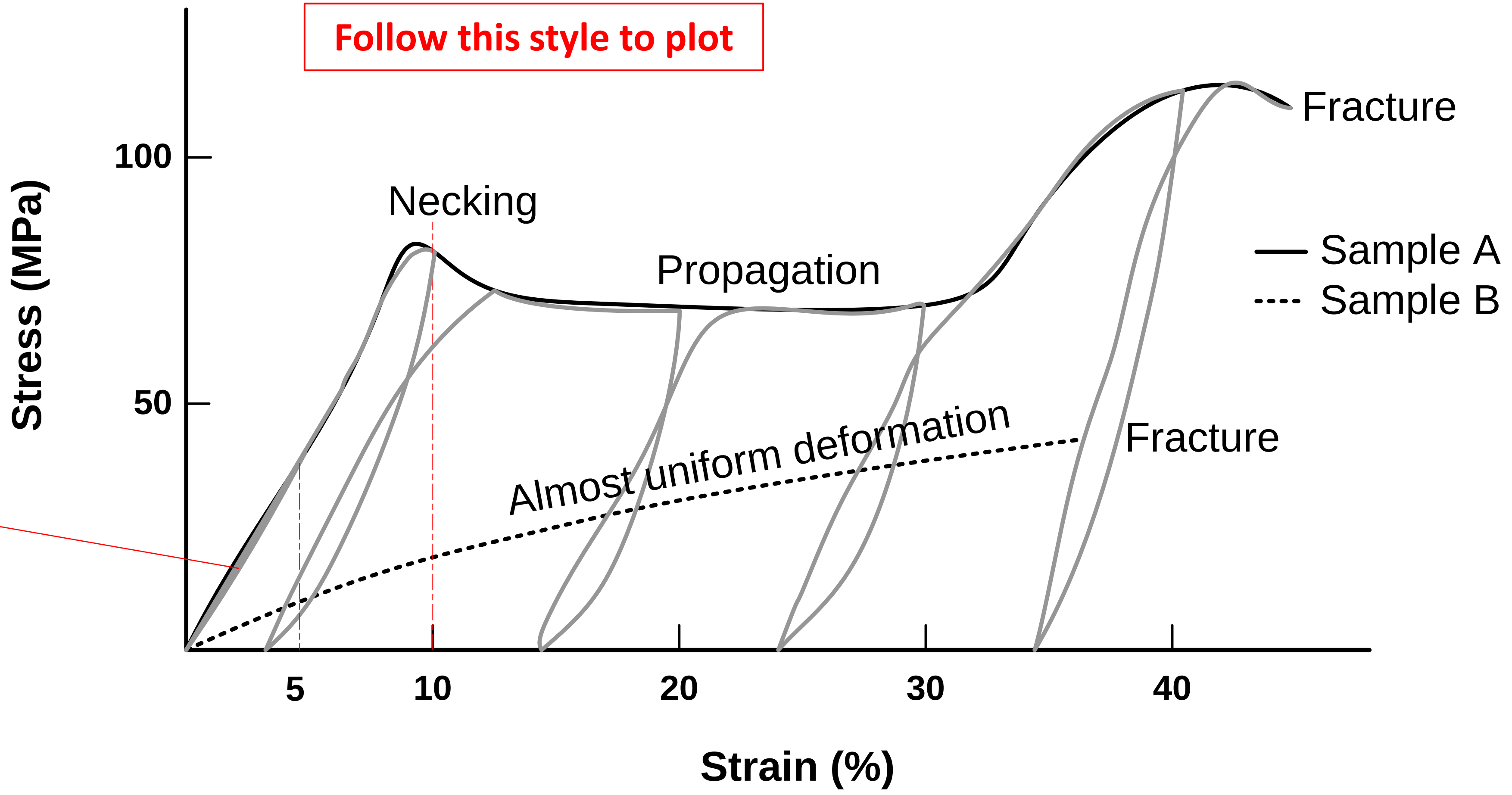
$$\varepsilon_{true} = \ln(1 + \varepsilon_{engineering})$$

Typical types of test

- Fracture

- Cyclic

Follow this style to plot



The 1st cycle to 5% is about 100% elastic.

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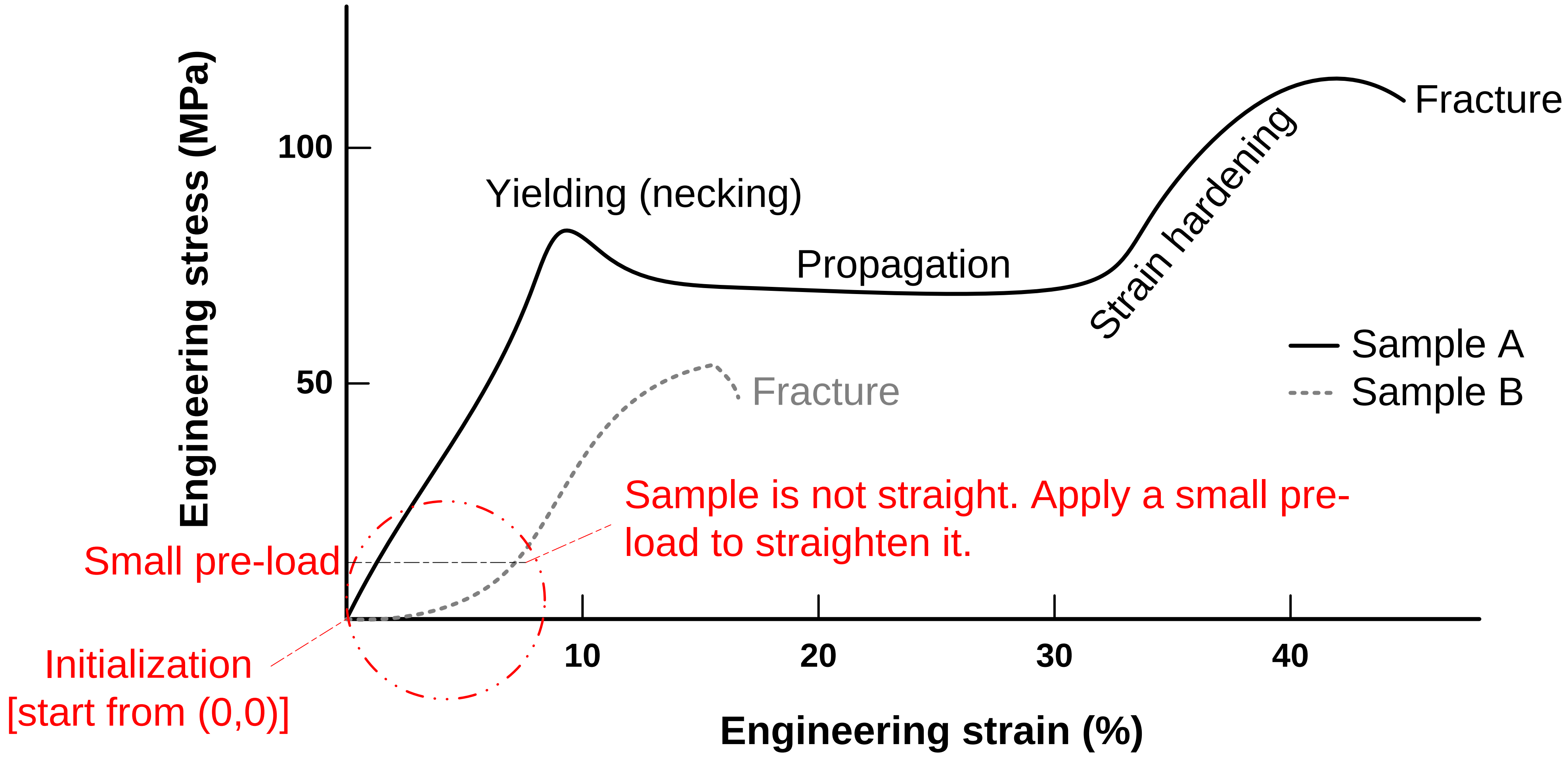
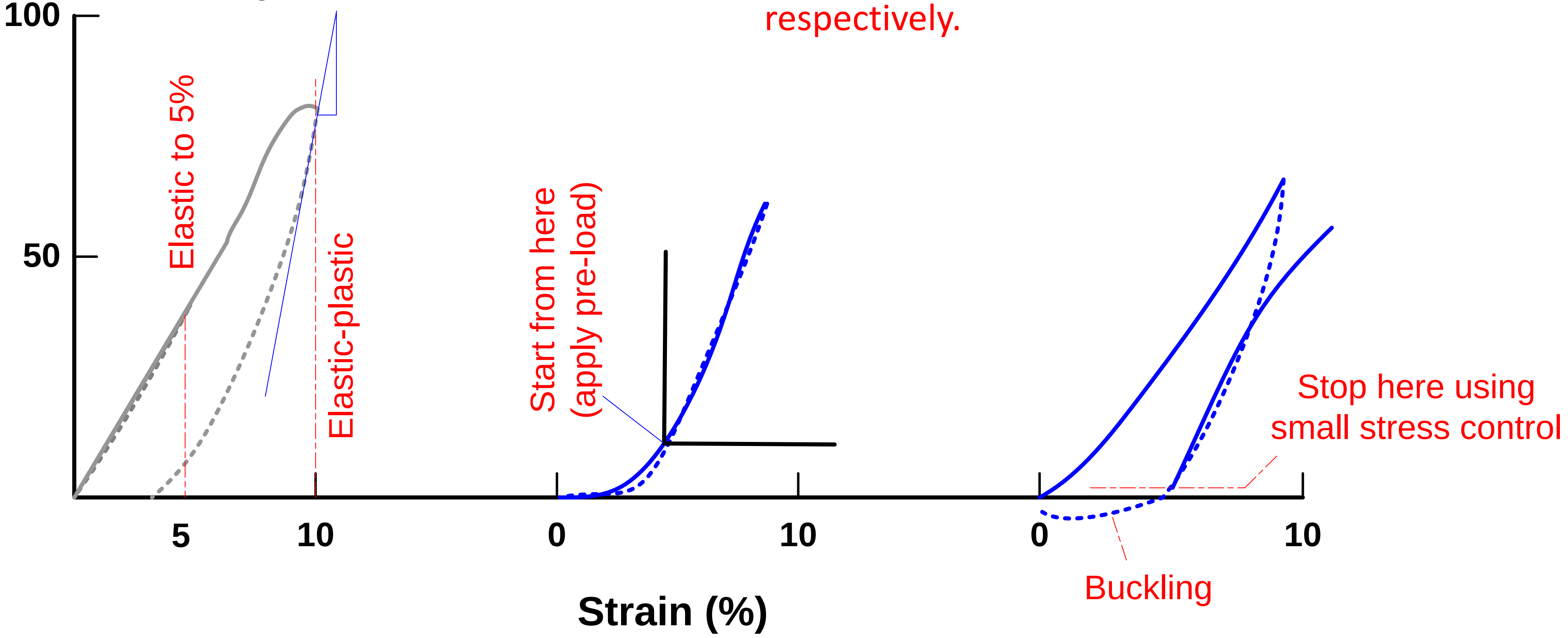
Stress vs. strain curve



In cyclic loading test, we normally use same speed in loading/unloading, but using strain and stress control in loading and unloading, respectively.



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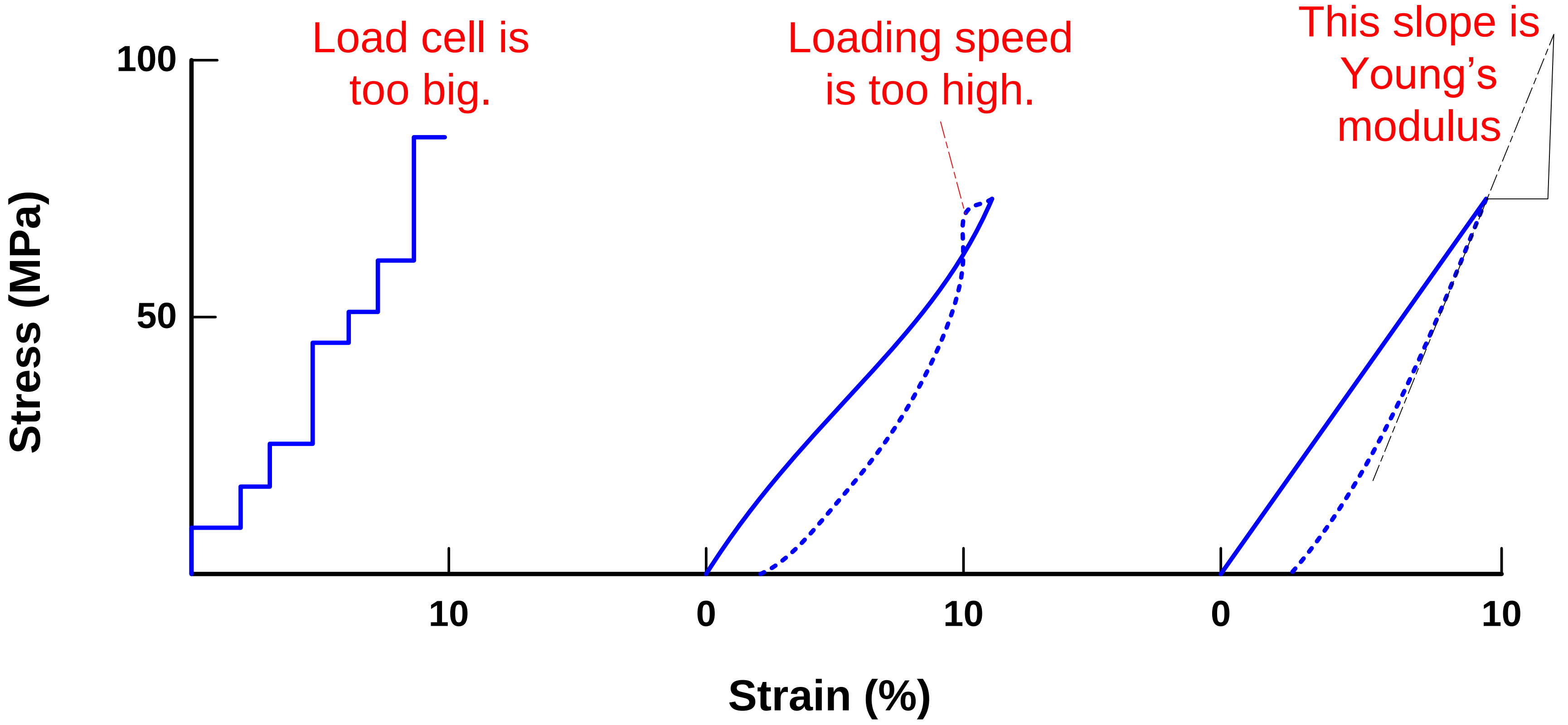
Loading speed in STRAIN RATE and load cell

Strain rate (in /s) = (speed of clamp, in mm/s)/(gauge length, in mm)

Typically, 10^{-3}/s (high speed) or 10^{-4}/s (low speed)

Load cell



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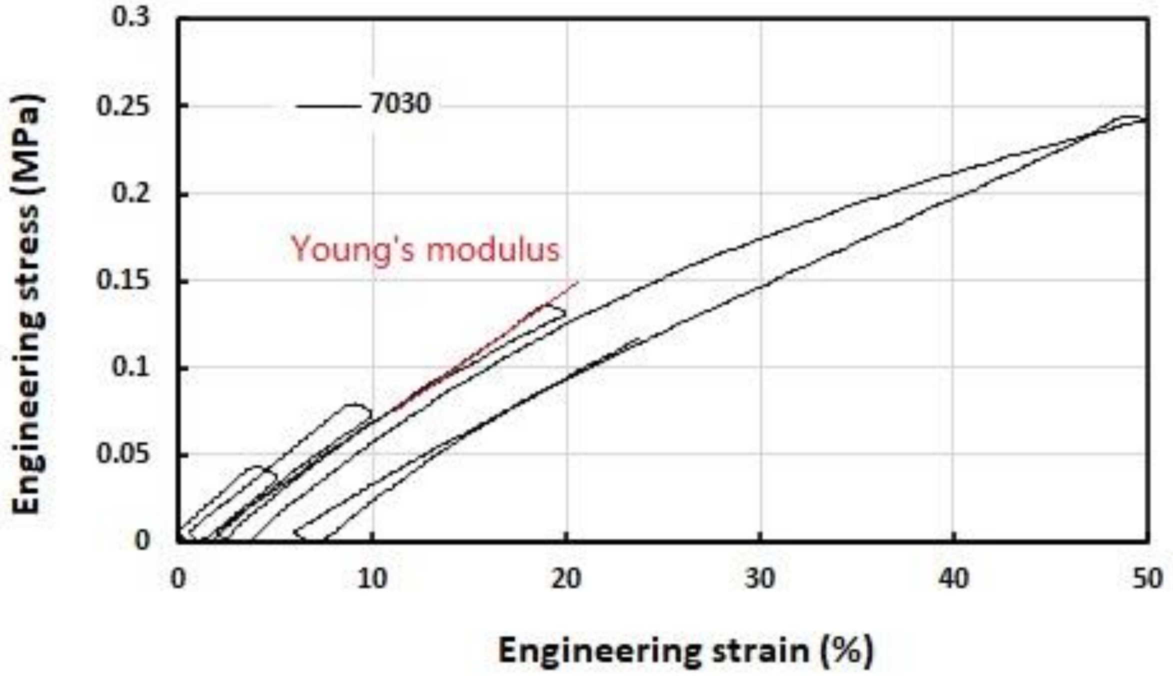
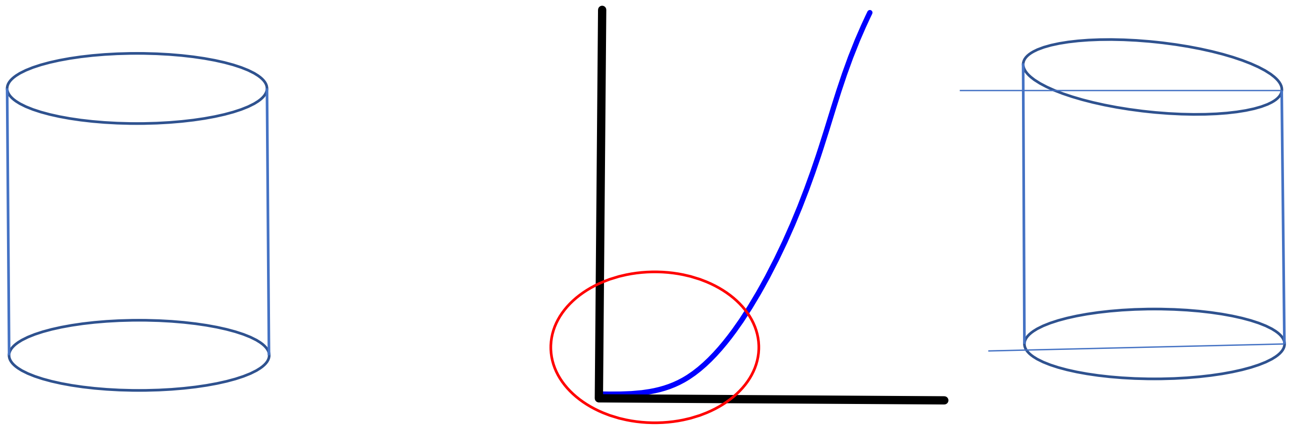
Example



0.3
0.25
0.2
0.15
0.1
0.05
0
Engineering stress (MPa)
7030
Young's modulus
0
10
20
30
40
50
Engineering strain (%)

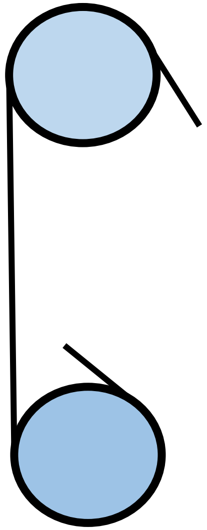
Additional issues

Compression test: cylindrical sample, short height (about 2 : 1 or less) to avoid buckling.



Check sample surfaces

Wire/fibre: using cylindrical clamp, wrapping around for a few turns.



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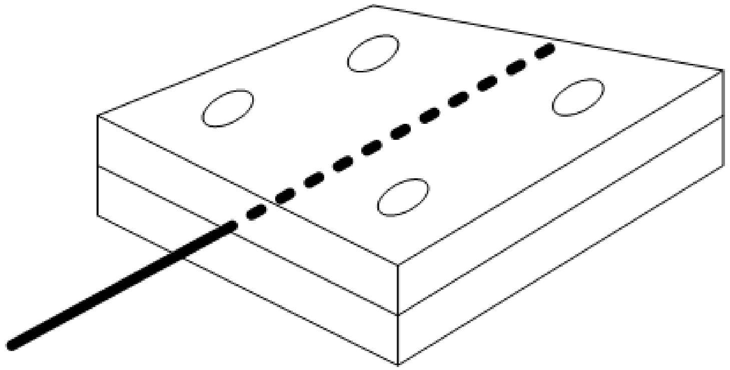
Short/thin sample: using "adapter" to increase area for good clamping.

Either using screw/nut or using clamper



For thin fibre/film, glue (e.g., superglue) may be used

3D surface scanning

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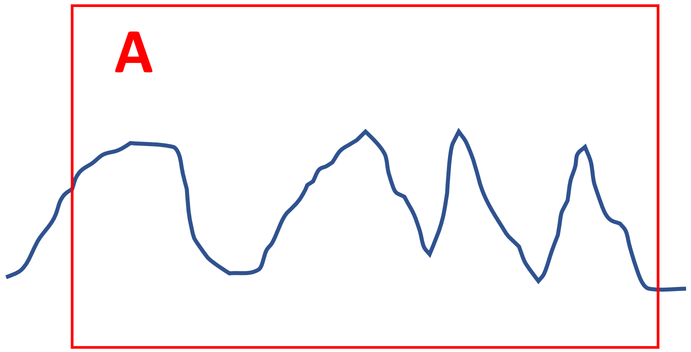
Testing mode

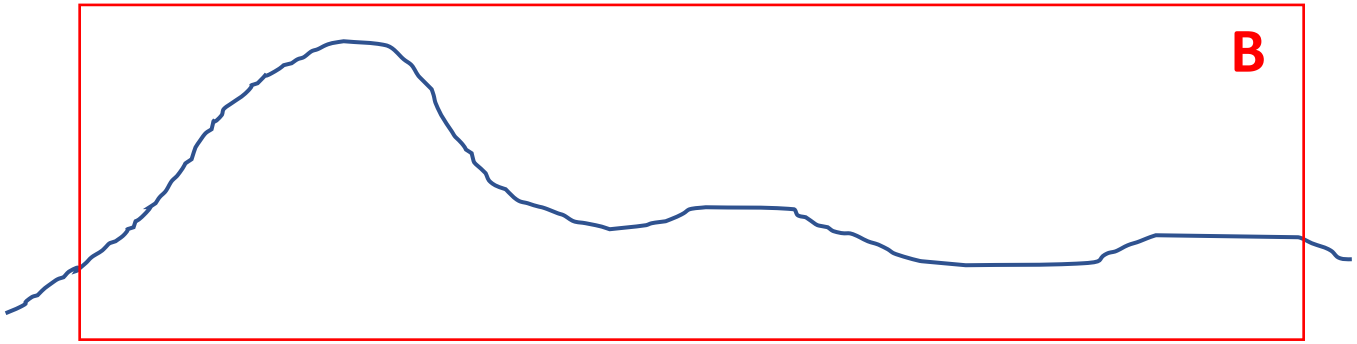
- **Contact mode**
  (maybe not applicable for soft/easily scratchable materials)
- **Non-contact mode**
  (maybe not applicable for transparent materials)

Testing speed/step/area

Depending on the surface features to be observed



Smaller step to catch the smaller sized features (reduce the scanning area to reduce testing time)

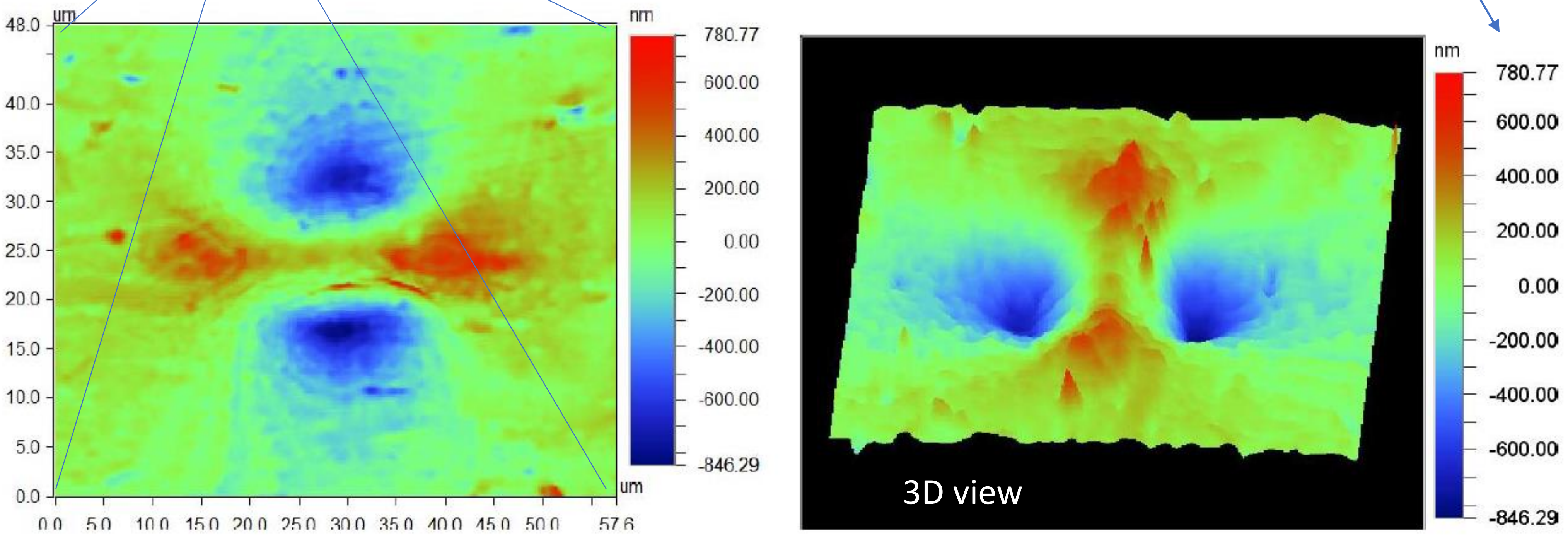


Larger step to catch the larger sized features (scanning over a larger area)

Typical ways to present the results

Make sure these four corners are at about the same height (using the software to adjust)

Scale bar must be included if vertical axis is not shown



(a) (b)

Fig. 1. A typical micro butterfly. (a) 2D view; (b) 3D view.

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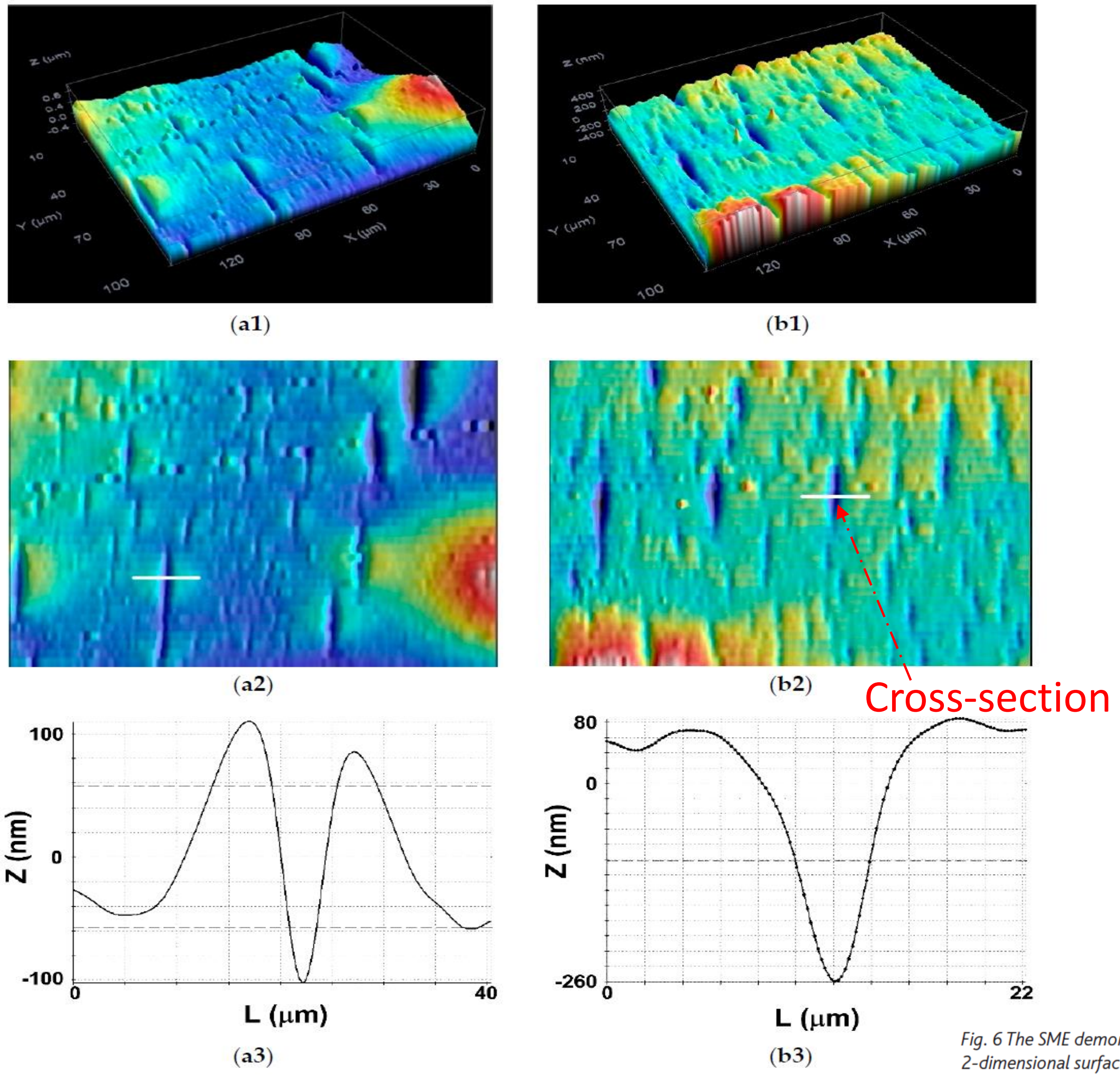


Figure 18. Typical surface morphology within necked area for samples programmed at 128 °C. Top row (1): 3D view; middle row (2): top view (area: 140 × 100 μm); bottom row (3) cross-section of the crack marked in middle row (2). Left: 30% strained; right: 100% strained.

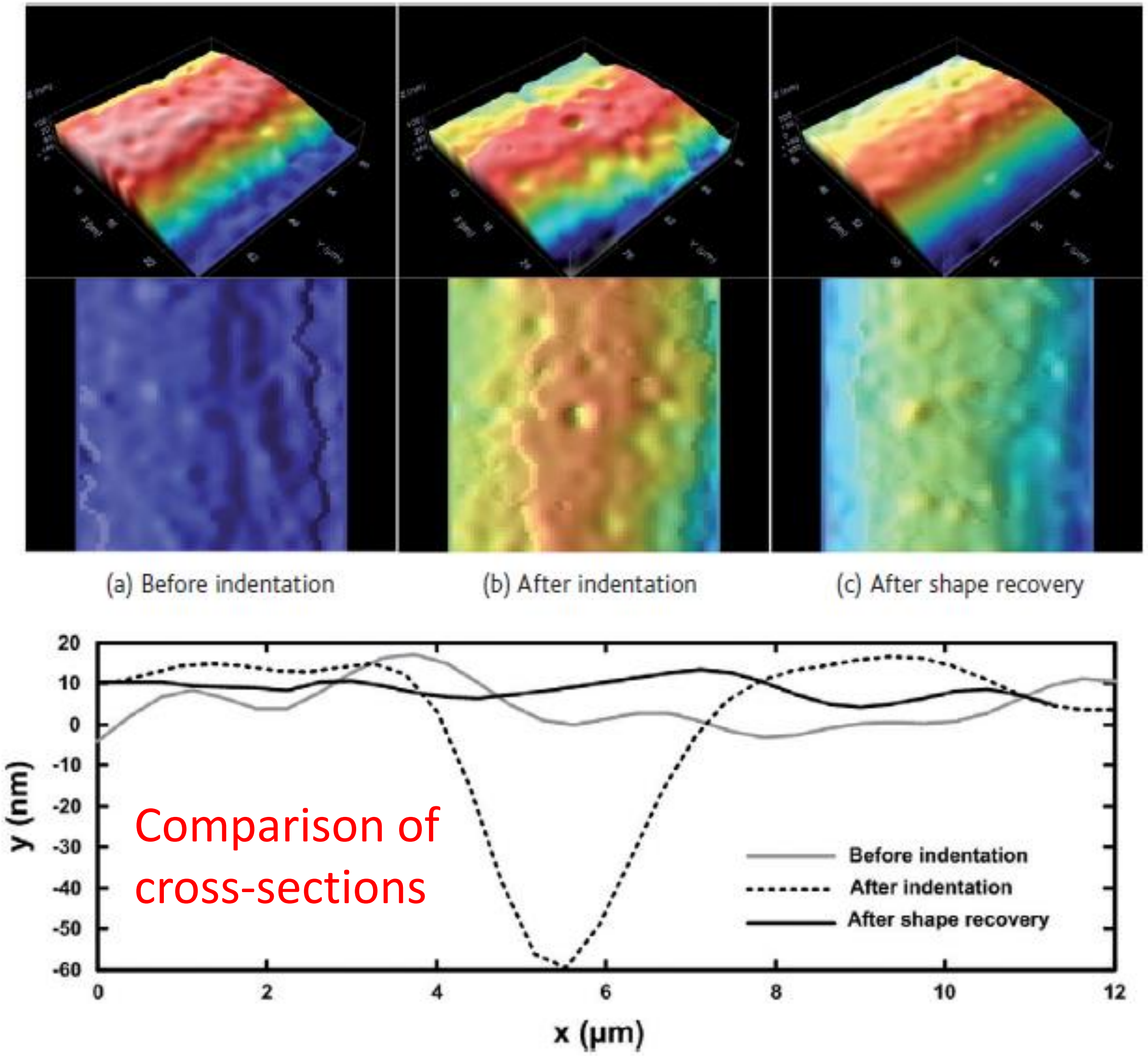
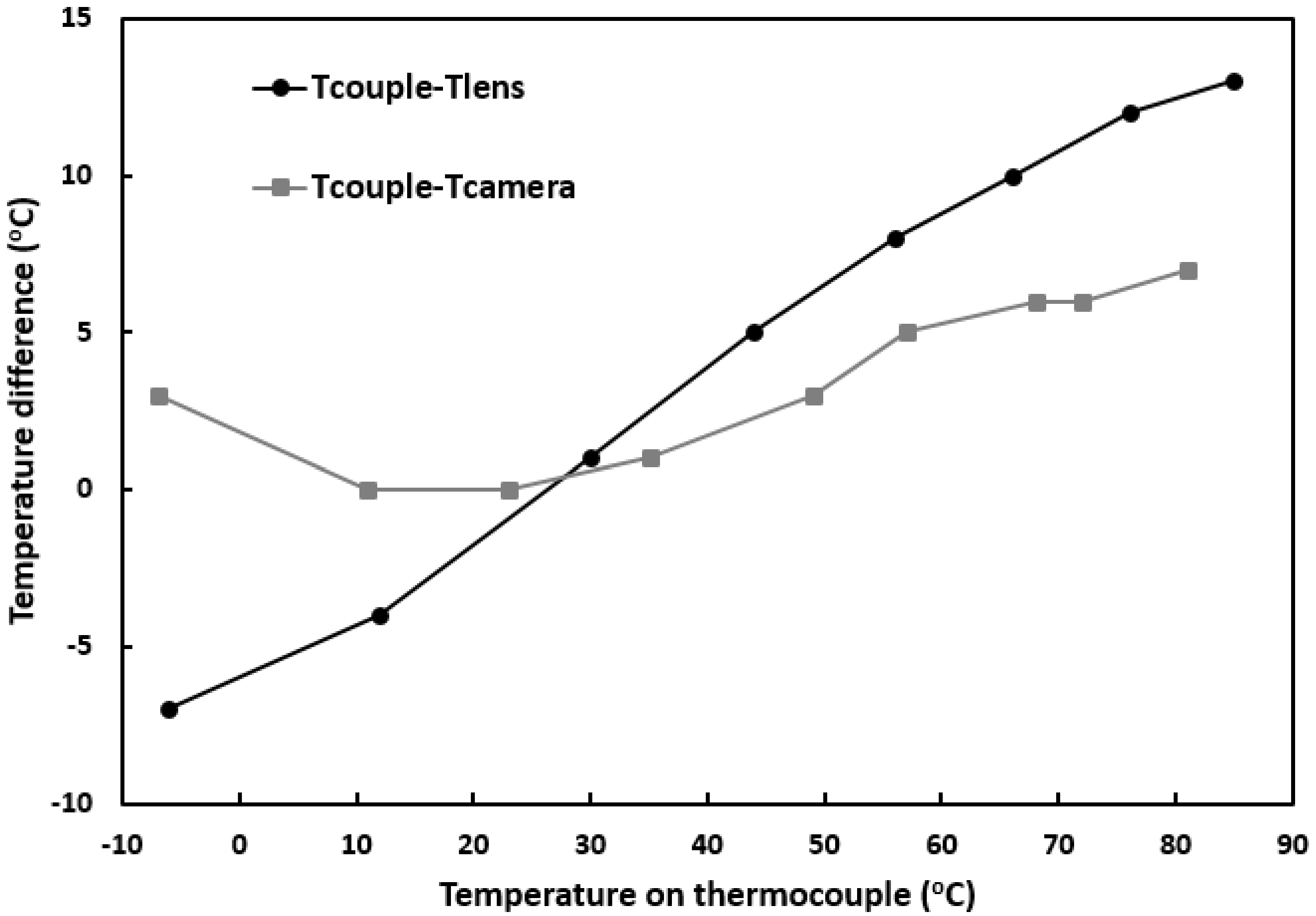


Fig. 6 The SME demonstrated by indentation test (using a Berkovich diamond indenter) in a 170 nm thick thin film polyurethane SMP. Top: 3-dimensional and 2-dimensional surface scanning images. Bottom: cross-sectional view.

Temperature

It may take ½ hour until the required temperature inside an oven/heat chamber is reached and stabilized (e.g., influence of clamps).

Always using a thermal couple to monitor the surface temperature of a tested sample.



- **Thermal <u>couple</u>**
- **Infrared <u>camera</u>**
- **USB <u>lens</u> (SEEK Thermal)**

Temperature calibration

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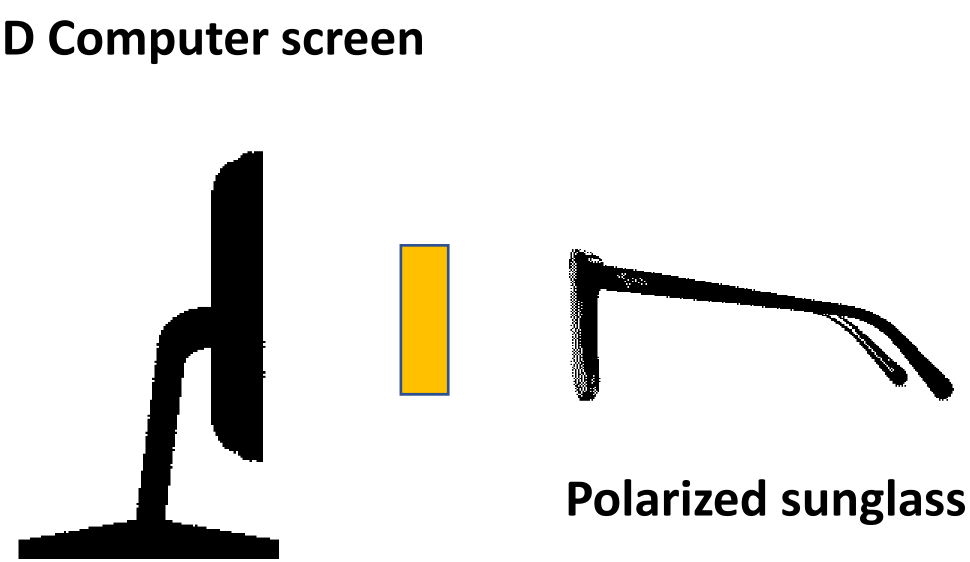
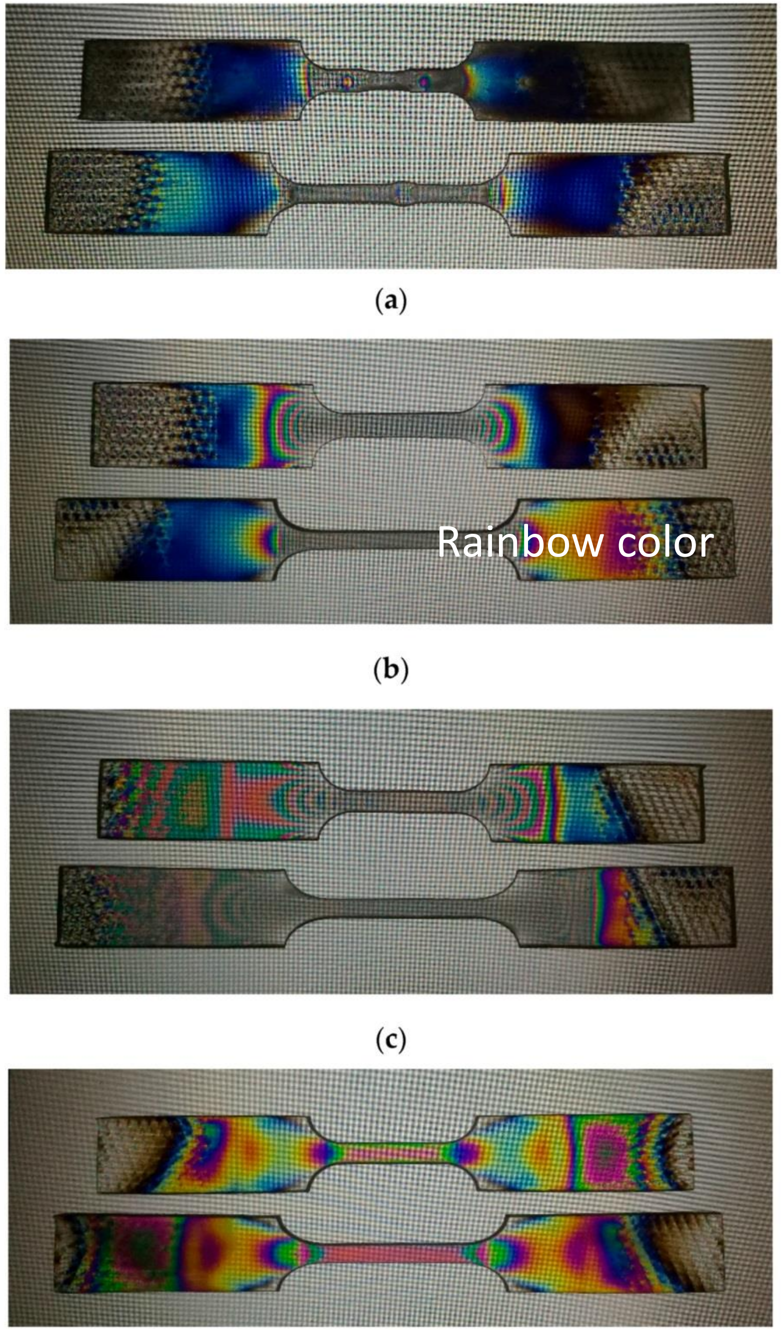
Photo-elasticity

A quick way to reveal stress distribution



Experimental setup



(a)

(b)

(c)

(d)

Figure 12. Images of the photo-elastic effect in samples programmed to two maximum programming strains of 30% (**top**) and 100% (**bottom**) at 128 °C (**a**); 135 °C (**b**); 145 °C (**c**) and 160 °C (**d**).

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Reference: *Appl. Sci.* **2017**, *7*(8), 848; https://doi.org/10.3390/app7080848

Camera/video

Always use a tripod, check focus, use a proper background, use a reference/ruler, save into smaller sized video if quality is not compromised…

Reference: European Polymer Journal 72 (2015) 282–295

Step by step shape recovery upon gradual heating of programmed sample

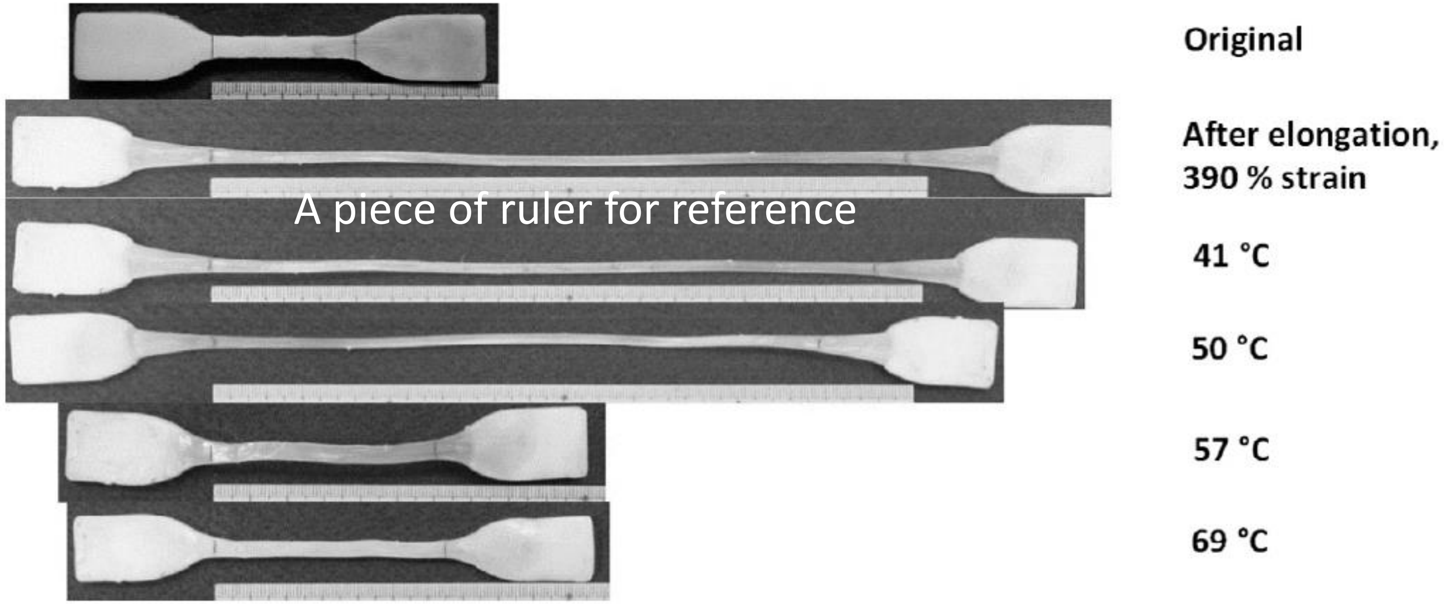

Fig. 7. Step by step heating to 41 °C, 50 °C, 57 °C and finally 69 °C.

Step by step shape recovery upon gradual heating of fractured sample

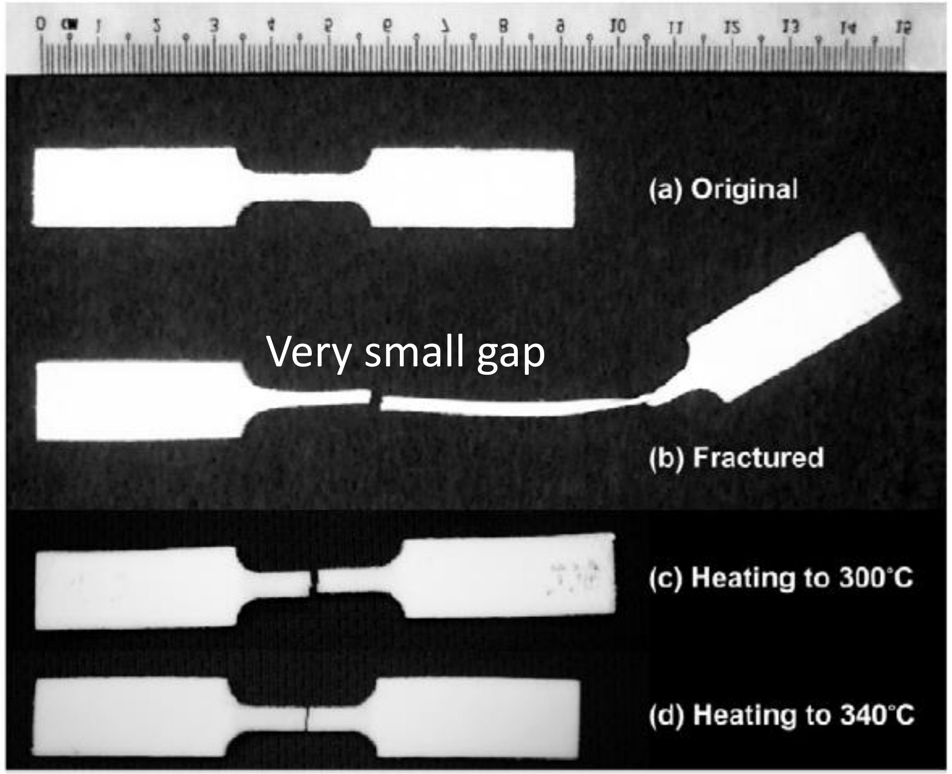

Figure 5. Shape recovery of a fractured sample after heating to 300 and 340 °C.

Reference: Smart Mater. Struct. 22 (2013) 125023

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Shape memory behaviour in polymeric materials

References

Characterization of polymeric shape memory materials, Journal of Polymer Engineering, Vol. 37, 2017, 1-20

- Characterization of shape recovery via creeping and shape memory effect in ether-vinyl acetate copolymer (EVA), Journal of Polymer Research, Vol. 20, 2013, 150
- Two-step shape recovery in heating-responsive shape memory polytetrafluoroethylene (PTFE) and its thermally assisted self-healing, Smart Materials and Structures, Vol. 22, 2013, 125023
- Characterization of the thermo-responsive shape memory effect in polyether ether ketone (PEEK), Journal of Applied Polymer Science, Vol. 131, 2014, 39844
- Thermo-responsive shape-memory effect and surface features in polycarbonate (PC), Applied Science, Vol. 7, 2017, 848
- Shape/temperature memory phenomena in un-crosslinked poly-e-caprolactone (PCL). European Polymer Journal, 72, 2015, 282-295
- Self-surface wrinkling atop acrylonitrile butadiene styrene (ABS) via heating-responsive shape memory effect, Surface Review and Letters, 26(8), 2019, 1950044
- Influence of long-term storage on shape memory performance and mechanical behavior of pre-stretched commercial poly(methyl methacrylate) (PMMA), Polymers, 11(12), 2019, 1978

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Some key issues

1. For shape memory hybrids, the important composition parameter is volume ratio, but not weight ratio.
2. Shape fixity ratio includes both short term shape fixity ratio and long term shape fixity ratio.

Herein, we may define the short term shape fixity ratio (R_f^s) as:

$$R_f^s = \frac{\varepsilon_r^s}{\varepsilon_{max}} \quad (1)$$

the long term shape fixity ratio (R_f^l) as:

$$R_f^l = \frac{\varepsilon_r^l}{\varepsilon_{max}} \quad (2)$$

and the shape recovery ratio (R_r) as:

$$R_r = \frac{\varepsilon_r^l - \varepsilon_R}{\varepsilon_r^l} \quad (3)$$

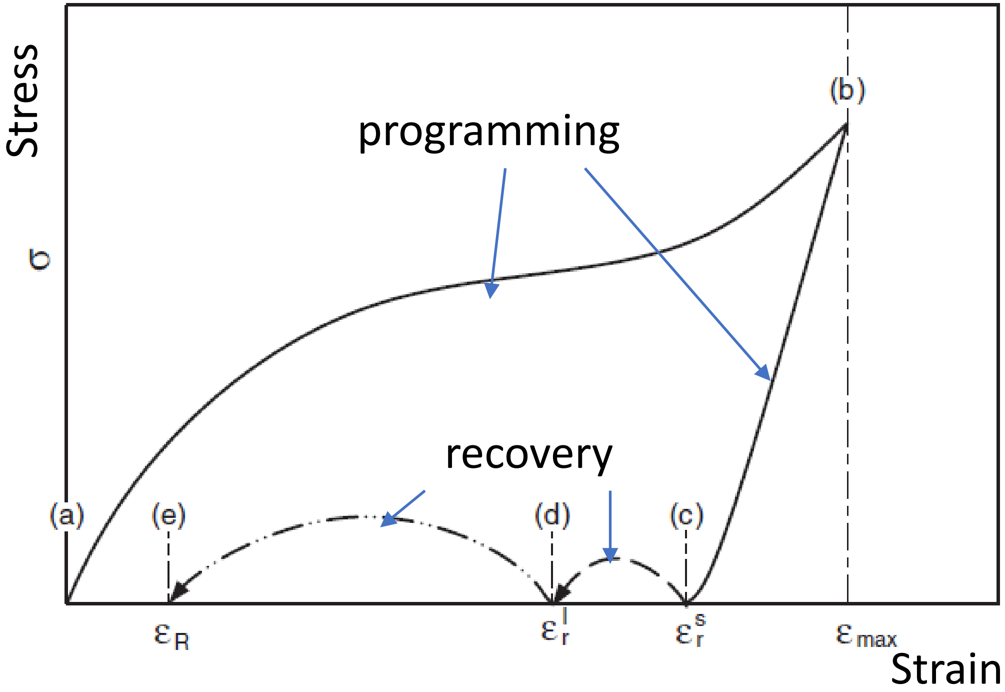

Figure 20: Illustration of a typical shape memory effect (SME) cycle in uni-axial tension. (Reproduced from [7]).

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3. Monitoring of creeping after programming

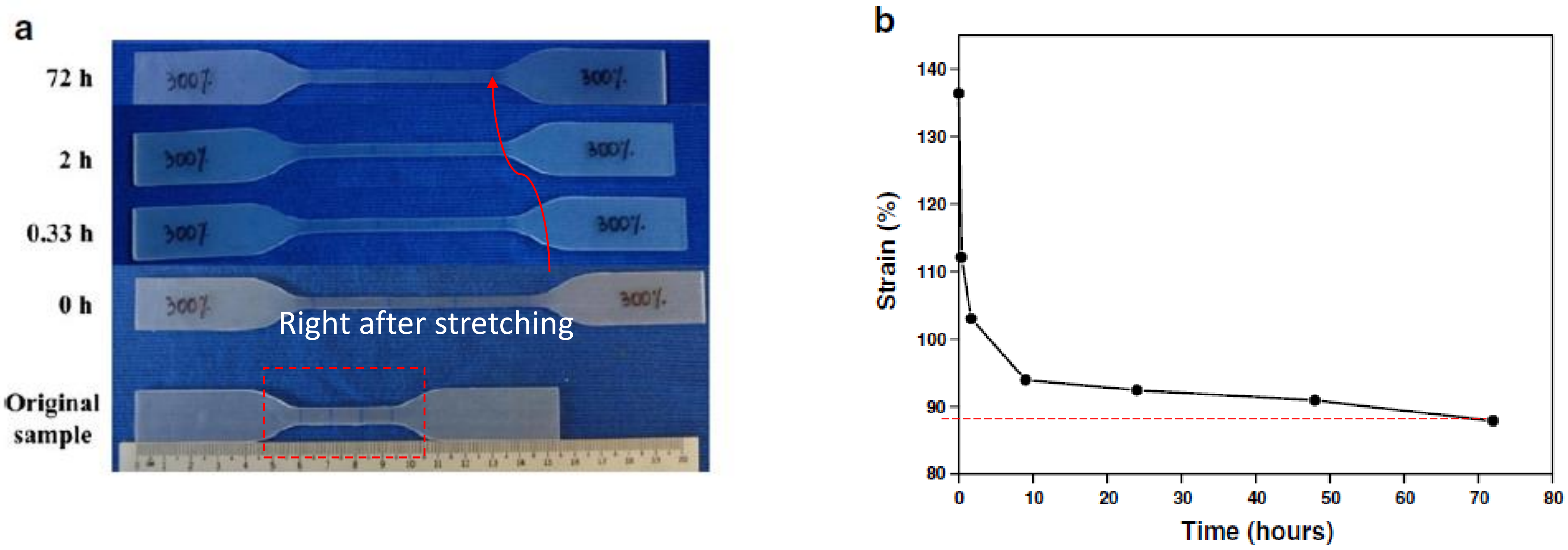


Fig. 8 a Recovery sequence (*bottom to top*) of EVA after 300 % pre-stretching at room temperature. The original shape is shown for comparison. **b** Evolution of the residual strain induced by creep at room temperature as a function of time

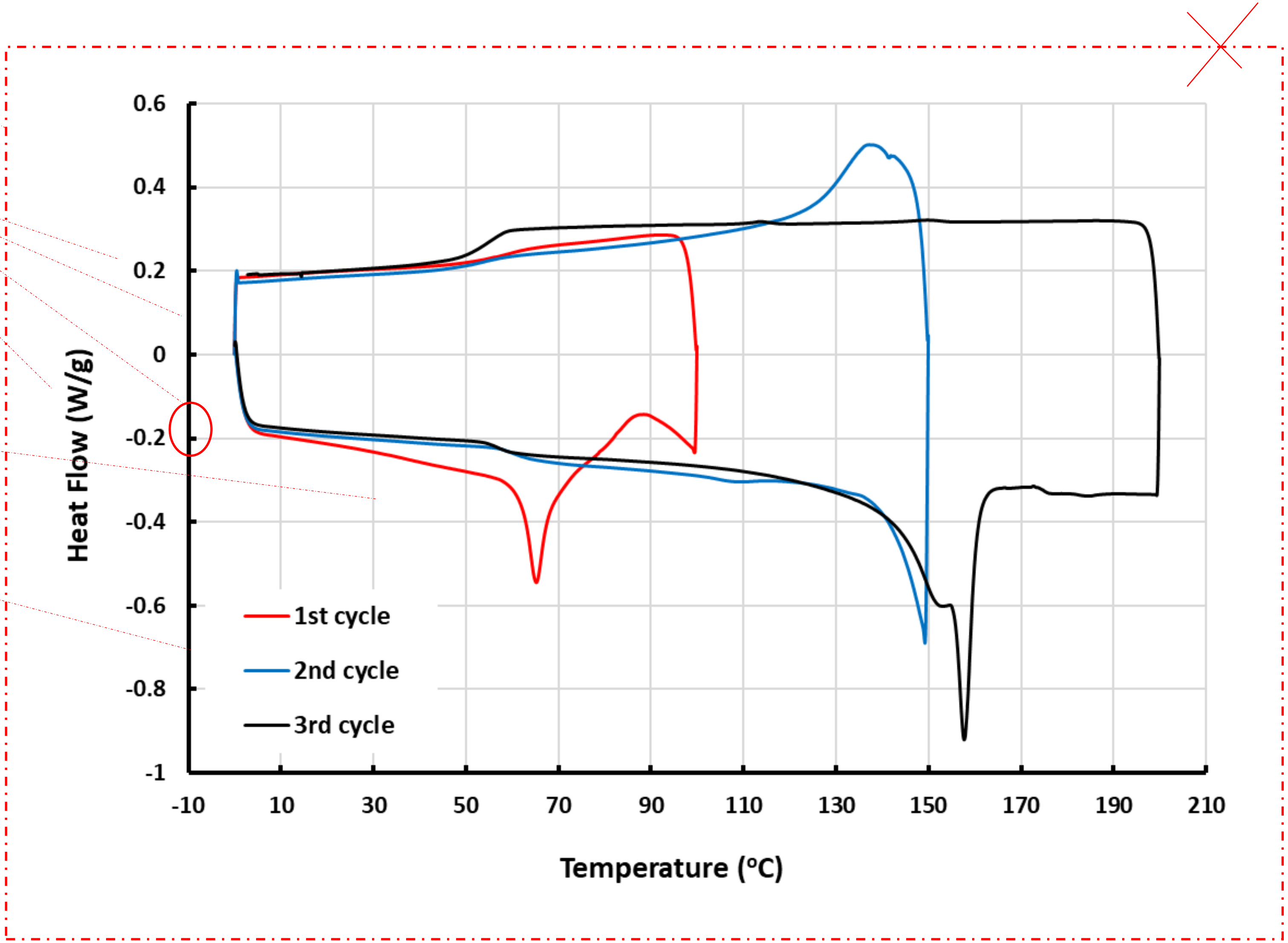
Characterization of shape recovery via creeping and shape memory effect in ether-vinyl acetate copolymer (EVA), Journal of Polymer Research, Vol. 20, 2013, 150

Typical format for “engineering” figure

SMT

- No border for figure.
- All axis title, axis, tick and number **<u>in black</u>** whenever possible.
- Major gridline, if really necessary, in thin line.
- Axis in black thicker line.
- The fond size of axis title is +2 than the axis number and legend.
- All fond in bold.
- Using different colours or line styles for lines.

Most of the default options in Excel are terrible bad.



Data collection, processing and analysis

If the resulted data file is too big, e.g., 60 M, the post-data process (in particular, using excel to plot) is going to take very long.

There are two ways to solve this problem.

- Reduce the data collection frequency. This is particularly useful, if you carry out a mechanical test at very low strain rate, or cyclic loading.
- Using, e.g., MATLAB, to pre-process the data to reduce the size. Take average over, e.g., five data points.

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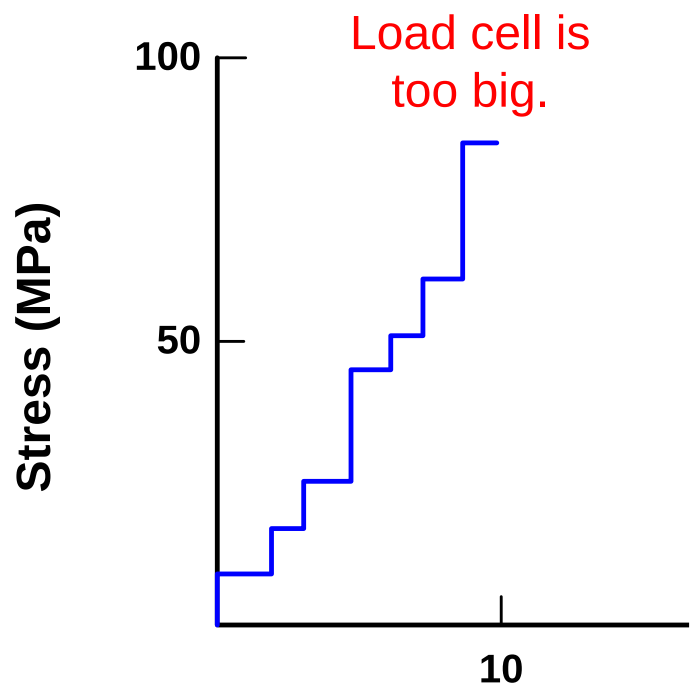

If the curve is in steps-style (as shown in left figure), or occasionally with sparks,

Smooth the curve by taking average of every, e.g., five data points, i.e.,

(1,2,3,4,5)/5….New data point (1)

(2,3,4,5,6)/5….New data point (2)

(3,4,5,6,7)/5….New data point (3)

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- Always save a few copies of the original data. Record the testing parameters.
- Always have a trial first to get familiar with the equipment and check if the result makes sense.
- A cyclic till fracture test is a quick way to get basic information of a <u>new</u> material.
- Process the result and compare with previous ones as soon as the experiment is finished. This is to check if the result obtained is reliable. Do not wait over, e.g., a few weeks, until a set of tests are finished.
- Experiment should repeat a few times, whenever possible.

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Average and deviation