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Accelerated Creep Characterization of High Density Polyethylene by the Stepped Isothermal Method

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Creep behaviour and testing

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Morphology

Processing determines:

- Degree of crystallinity
- Molecular orientation
- Geometry dependent morphological variations

Loading

- Mode (tension, compression, shear)
- Magnitude (linear viscoelasticity, non-linear viscoelasticity)

Temperature

- Thermal treatment/ preconditioning
- Application temperature relative to glass transition

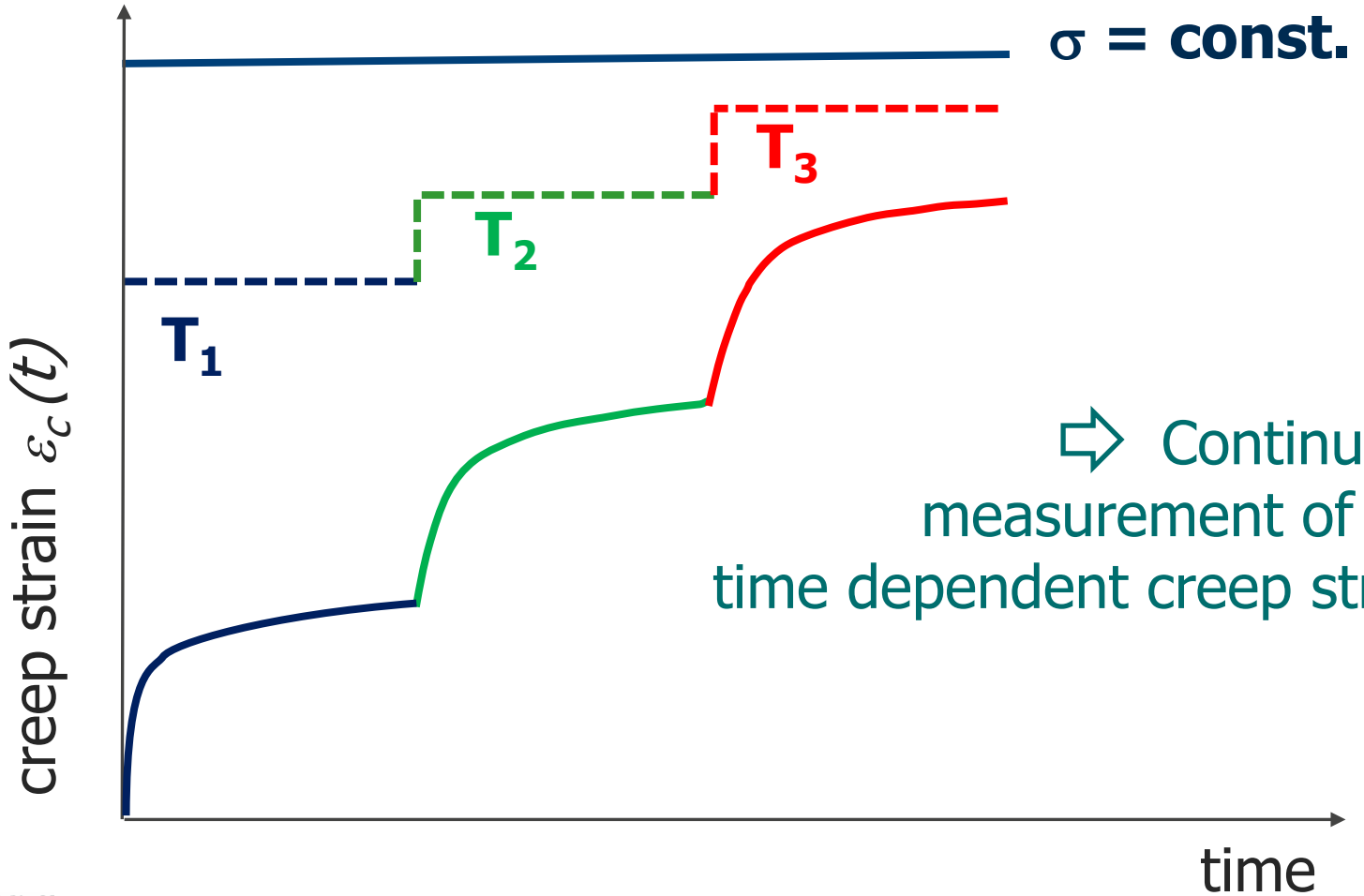
C r e e p b e h a v i o u r

Accelerated testing by Time-Temperature Superposition requires:

- Stable morphology within the temperature range of testing
- Loading within the linear-viscoelastic range
- Limited temperature range according to the underlaying molecular relaxation process

Stepped Isothermal Method SIM

Basic test procedure



⇨ Continuous measurement of the time dependent creep strain

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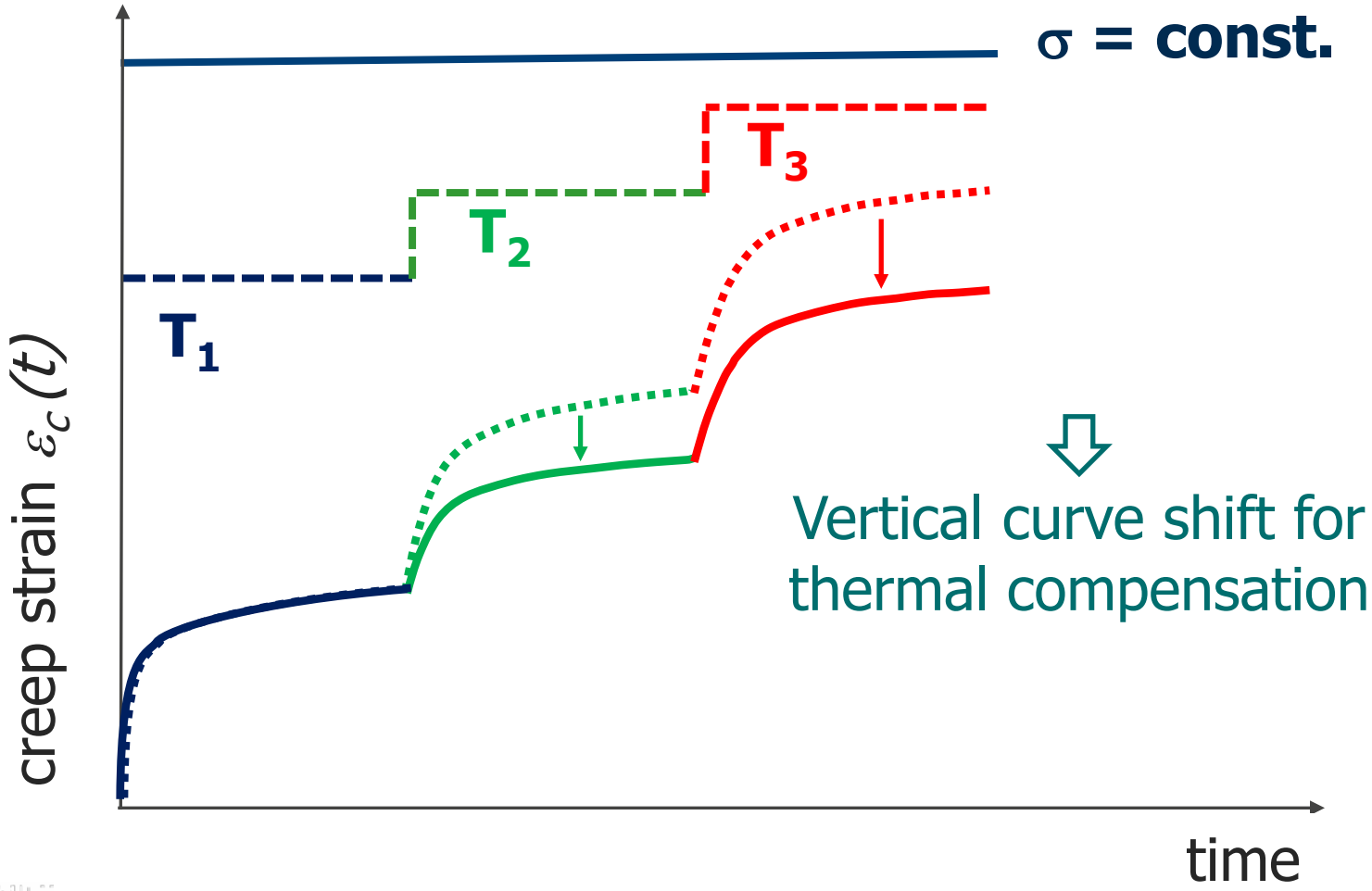
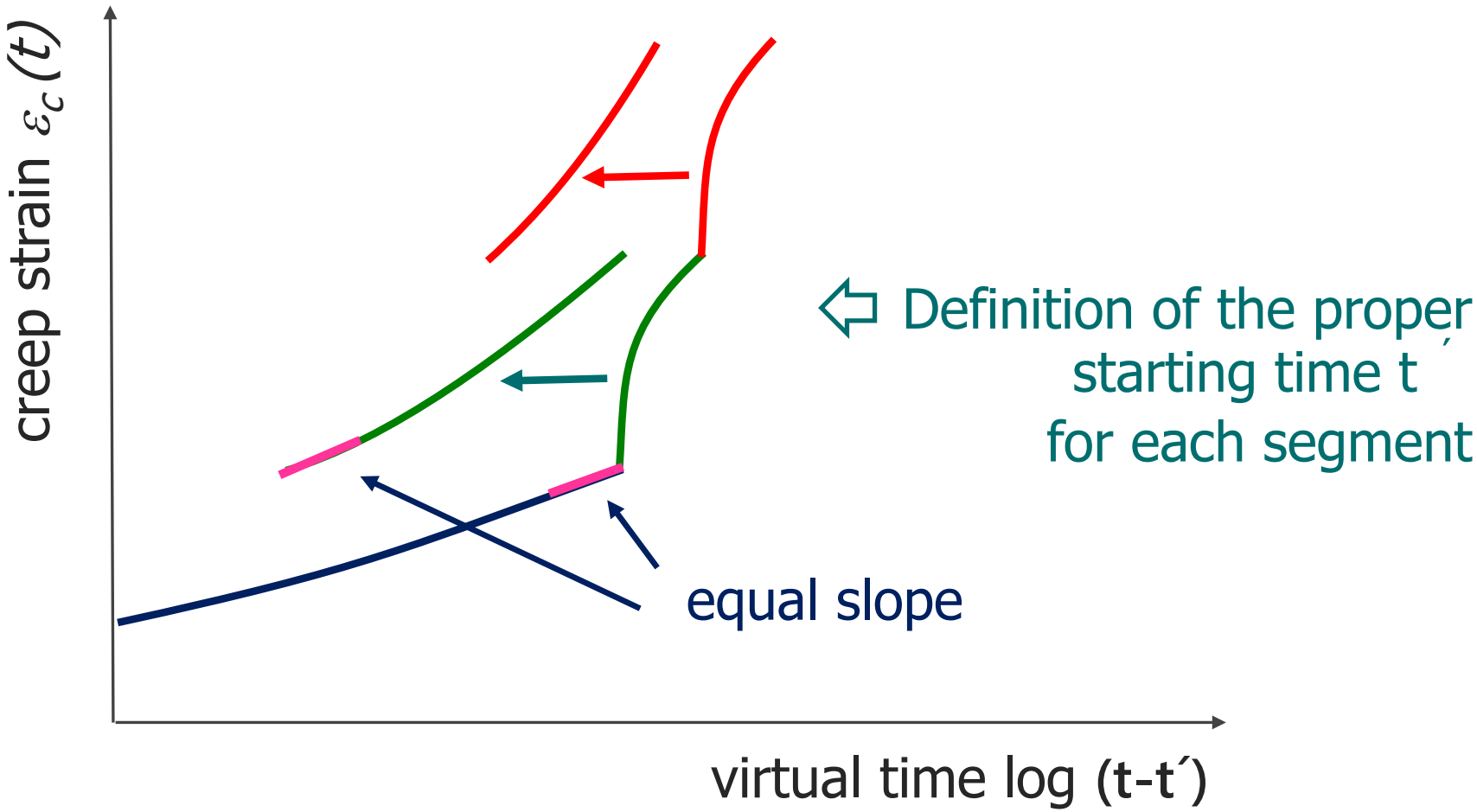
Curve correction referring to thermal expansion



Stepped Isothermal Method SIM

Virtual starting times => separated creep curves



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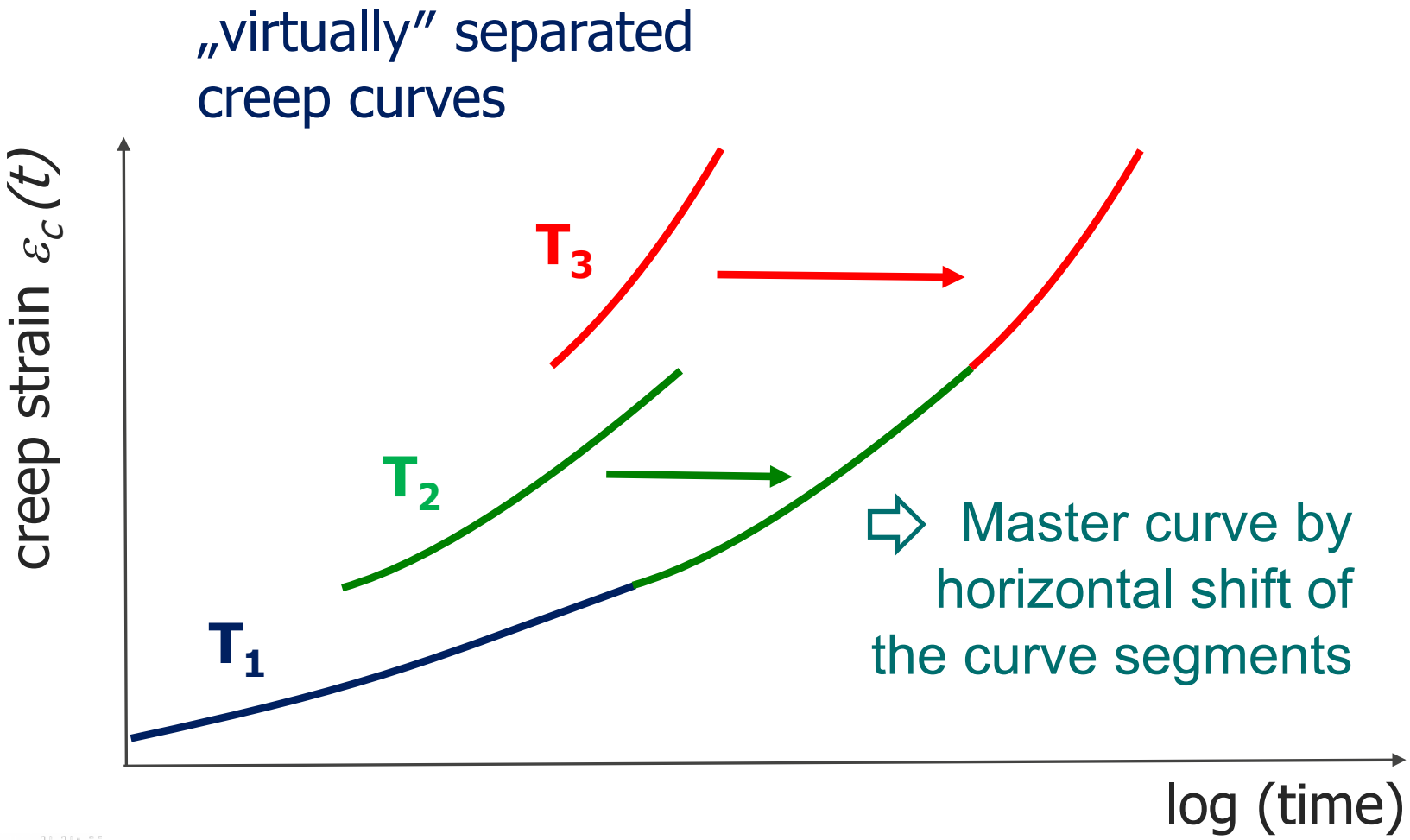
Conventional time temperature superposition TTSP



Experimental

Material

High density polyethylene (HDPE)

Density: 0.953 g/cm³

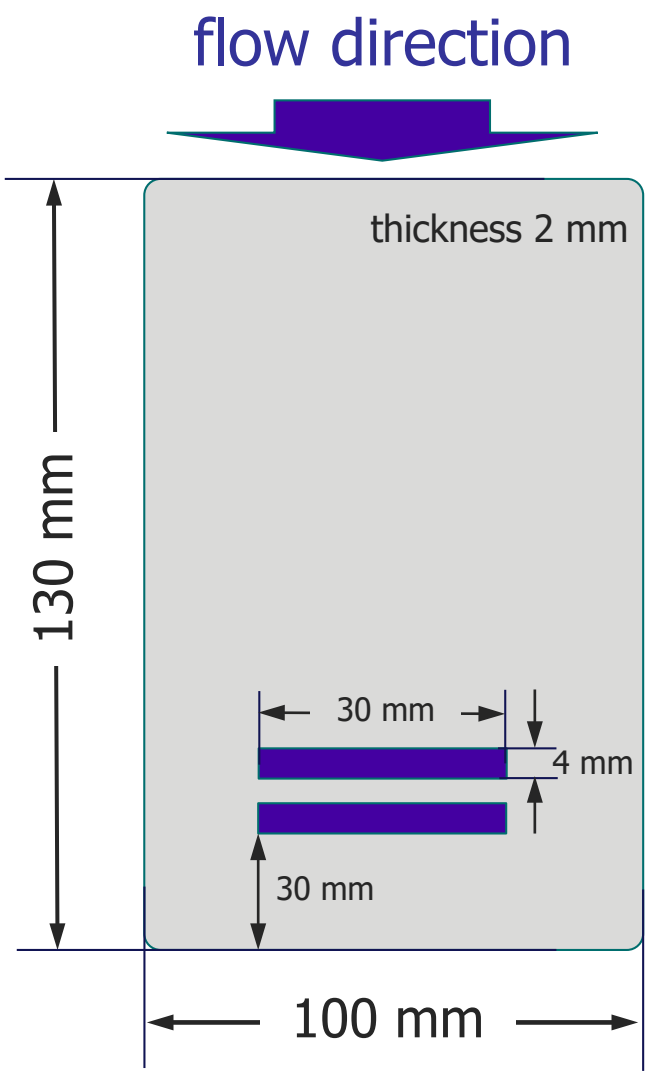
Young´s modulus: 1.2 GPa

Specimen preparation

bar specimens (30 x 4 x 2 mm)

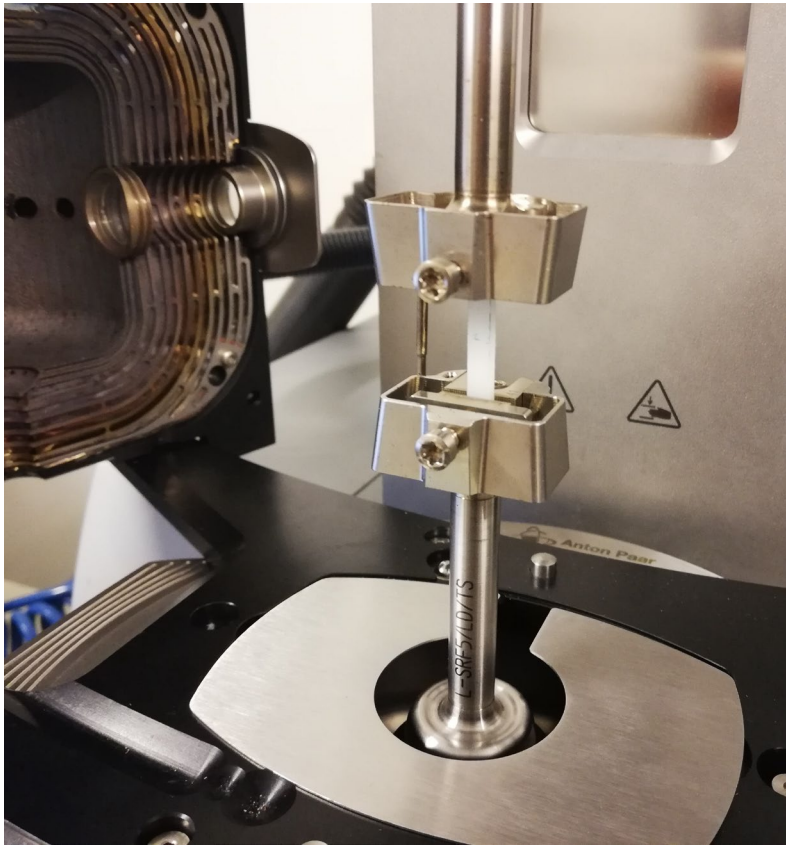
cut out from injection moulded plates (130 x 100 x 2 mm)

perpendicular to the flow direction in the lower section of the plates



Experimental

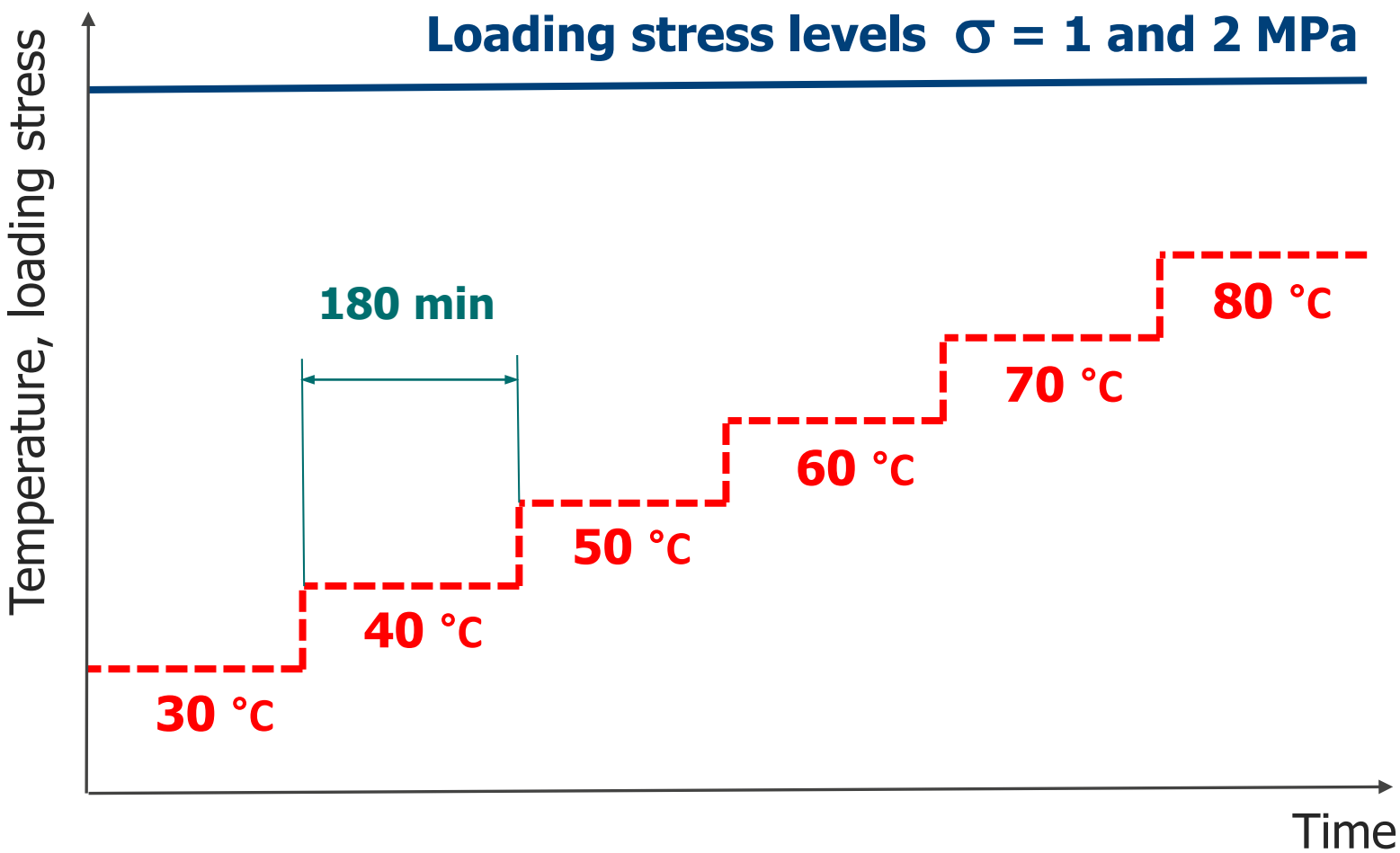
Dynamic Mechanical Analyzer MCR 702 MultiDrive (Anton Paar, GmbH, Austria)



Test parameters for SIM/Creep-Testing

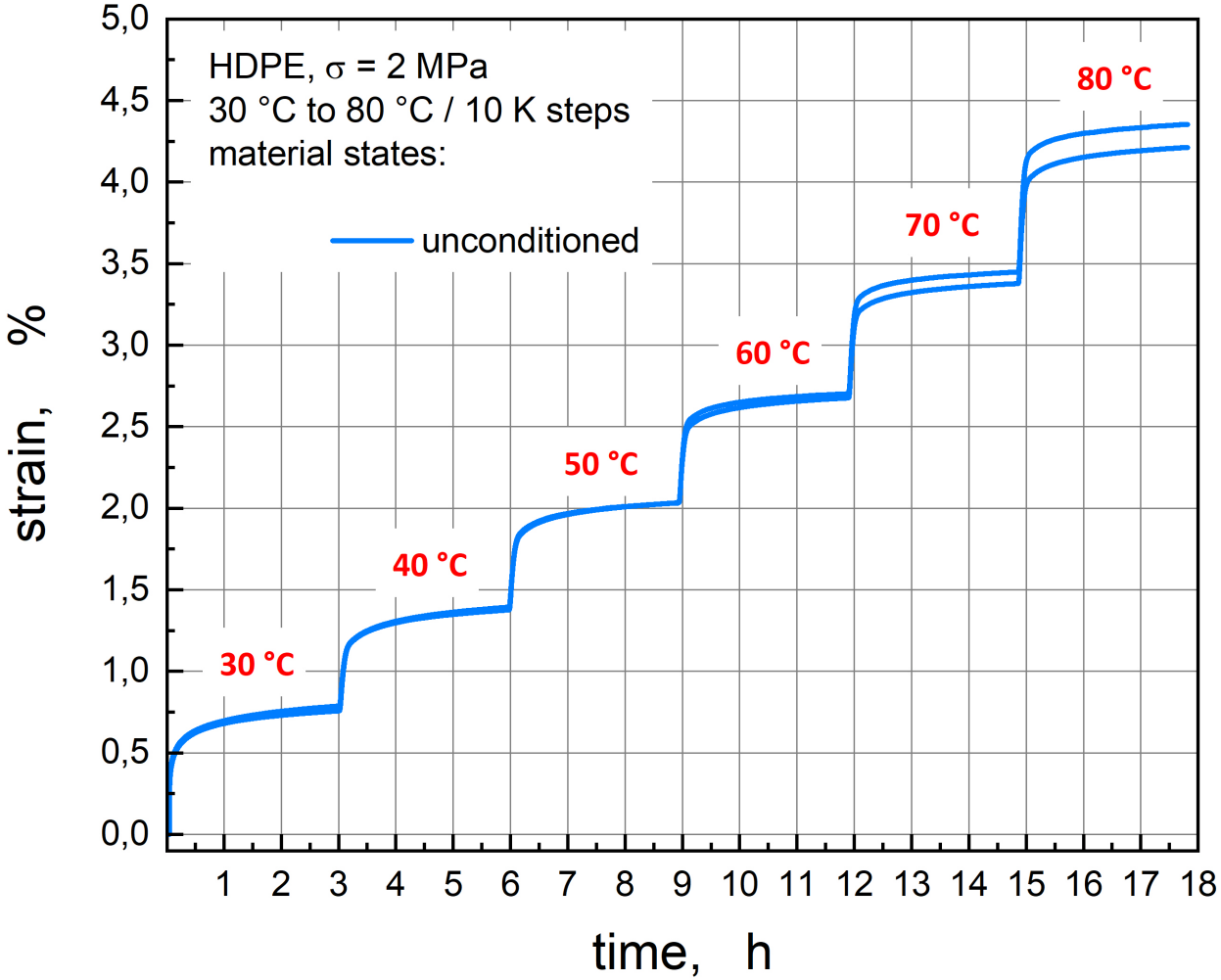
Loading mode:	tensile, bar specimens
Clamping length:	18.5 mm
Loading stress:	1 and 2 MPa
Temperature steps:	30, 40, 50, 60, 70, 80 °C
Dwell time:	180 min

Experimental



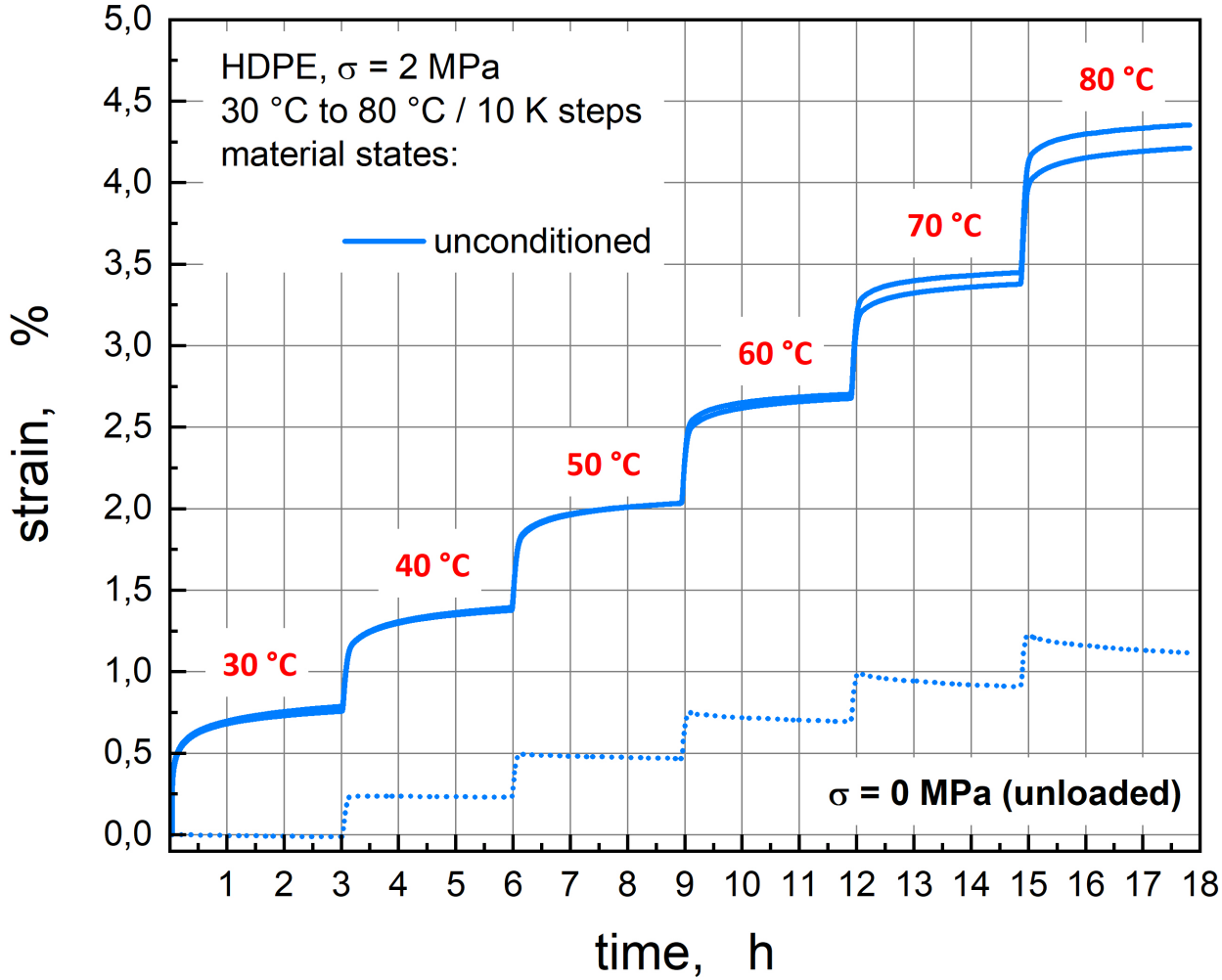
Results – Creep strain curves by SIM

for specimens without preconditioning



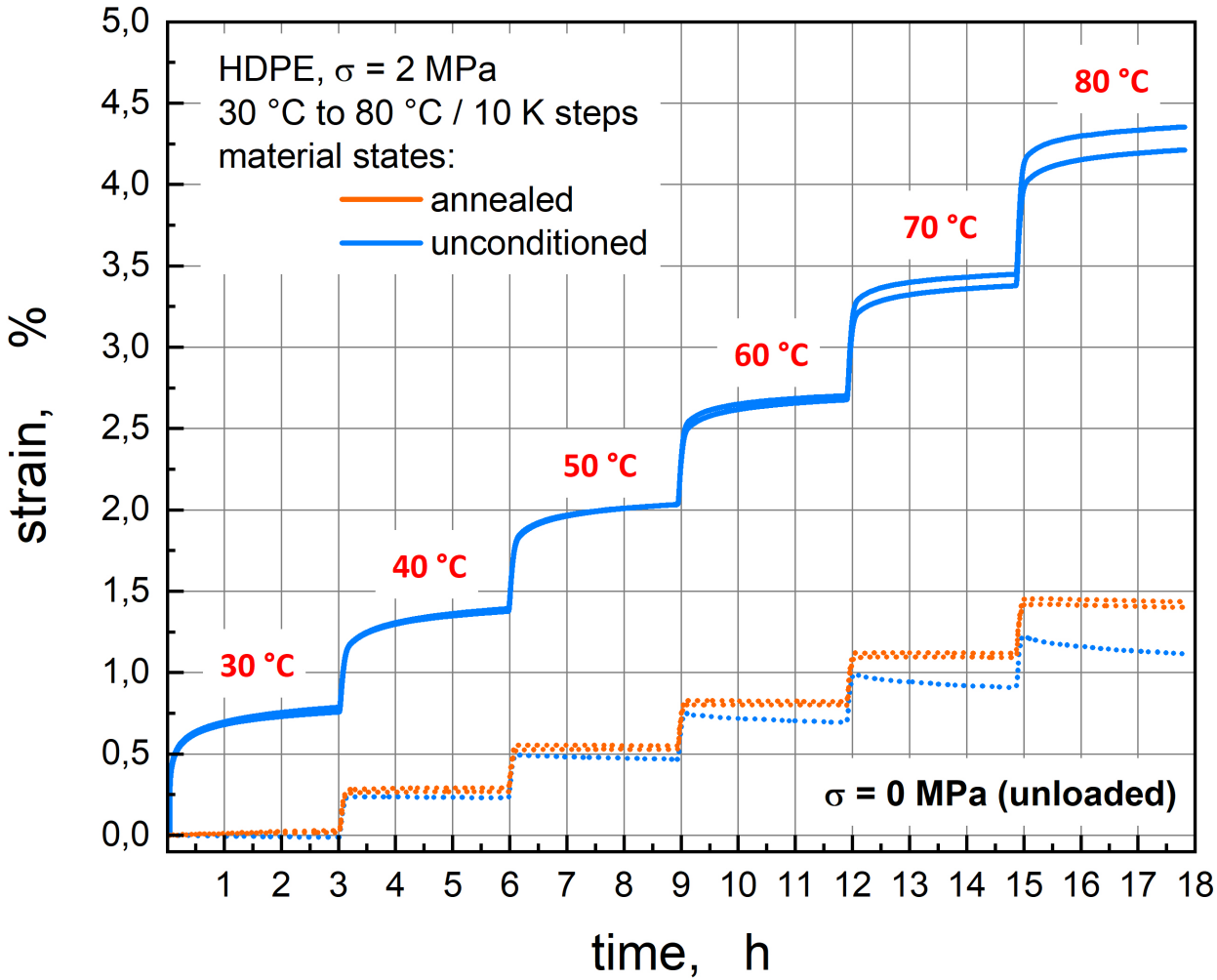
Results – Creep strain curves by SIM

for specimens without preconditioning



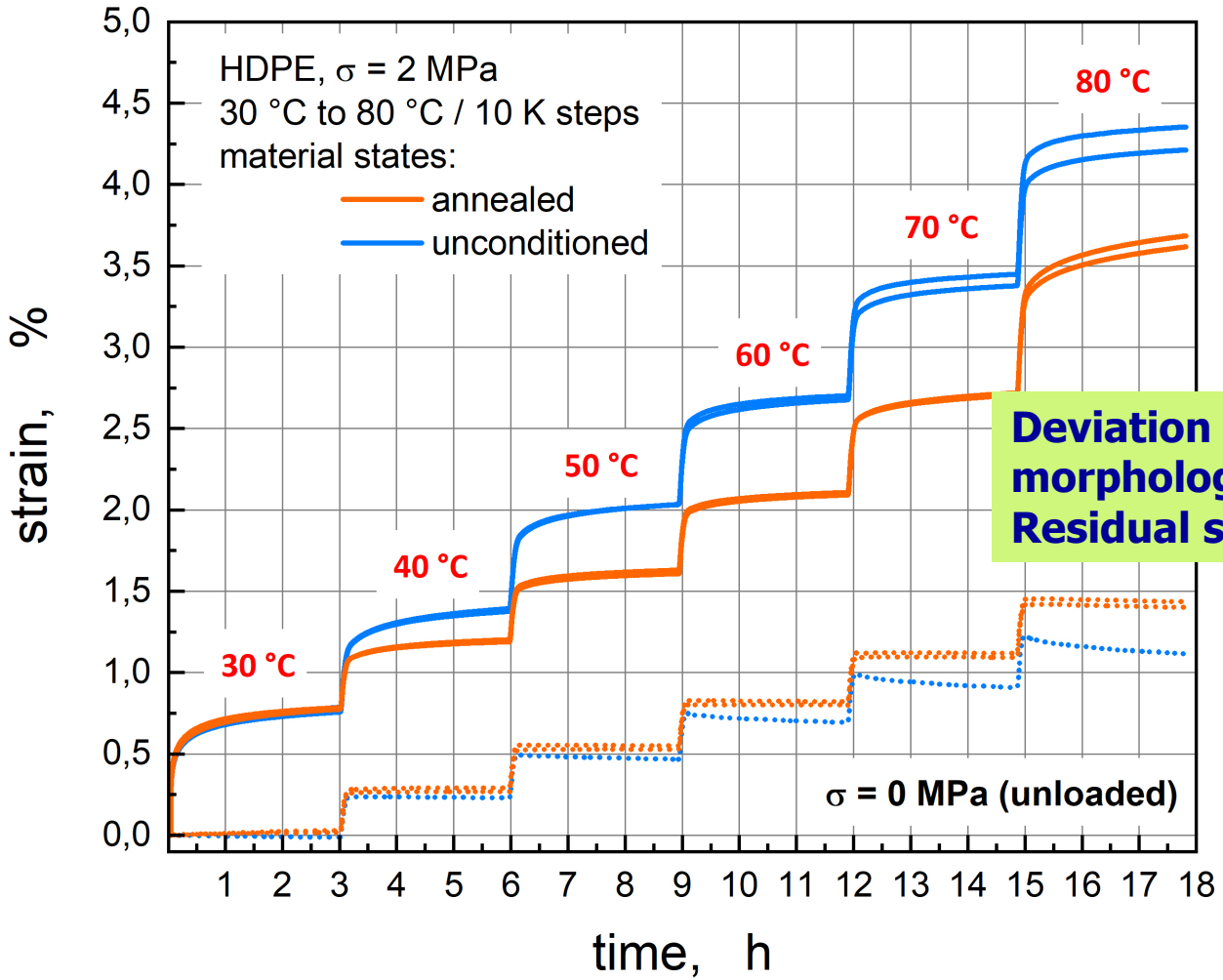
Results – Creep strain curves by SIM

for specimens with thermal pre-treatment



Results – Creep strain curves by SIM

for specimens with thermal pre-treatment



Deviation due to morphological changes? Residual stress?

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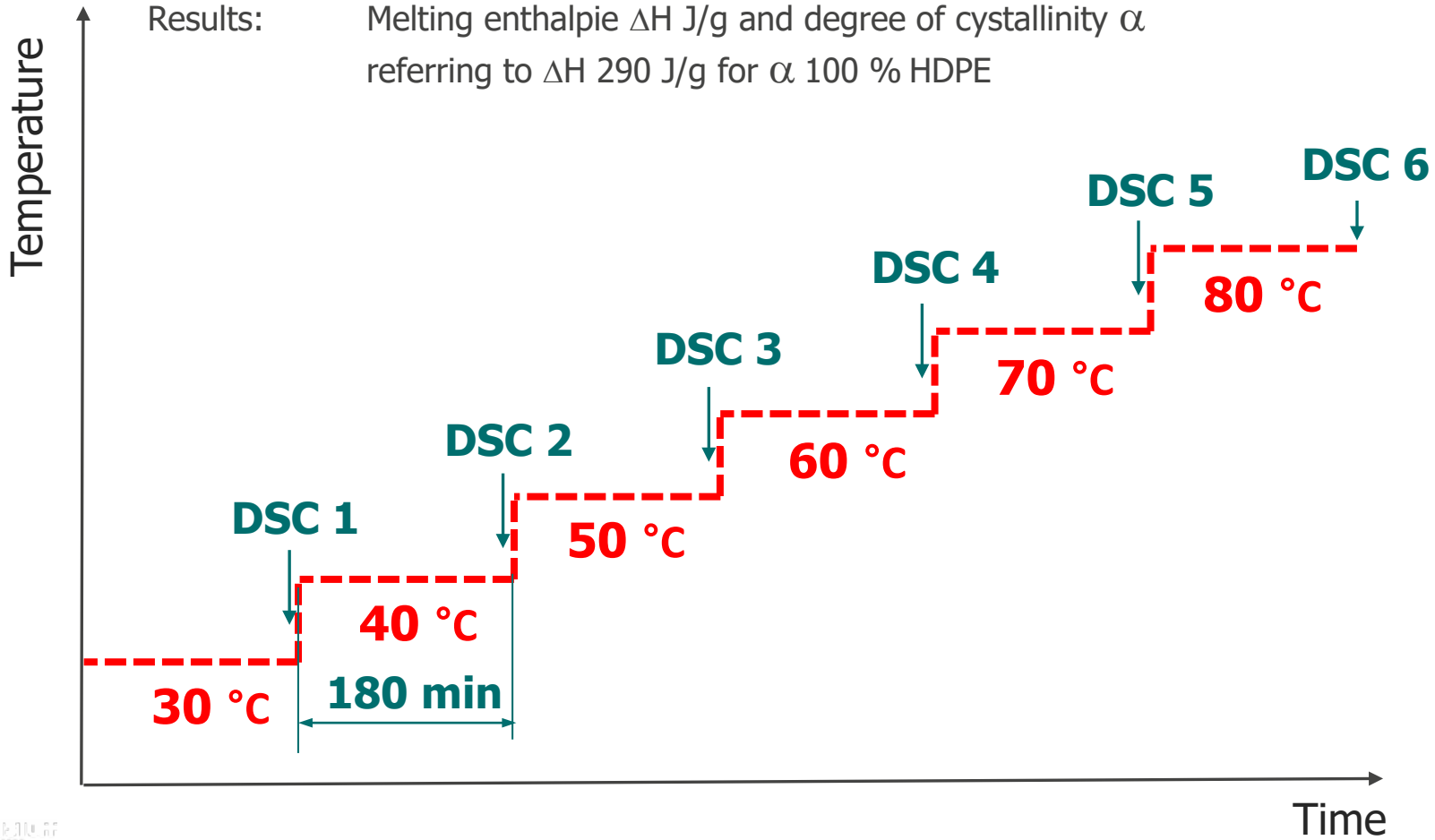
Cystallinity for the various temperature steps

Samples: preparation out of unloaded specimens; cubes ~ 2.2 mm / ~10 mg

Heating: 10 K/min up to 180 °C

Results: Melting enthalpie ΔH J/g and degree of cystallinity α referring to ΔH 290 J/g for α 100 % HDPE



DSC Results

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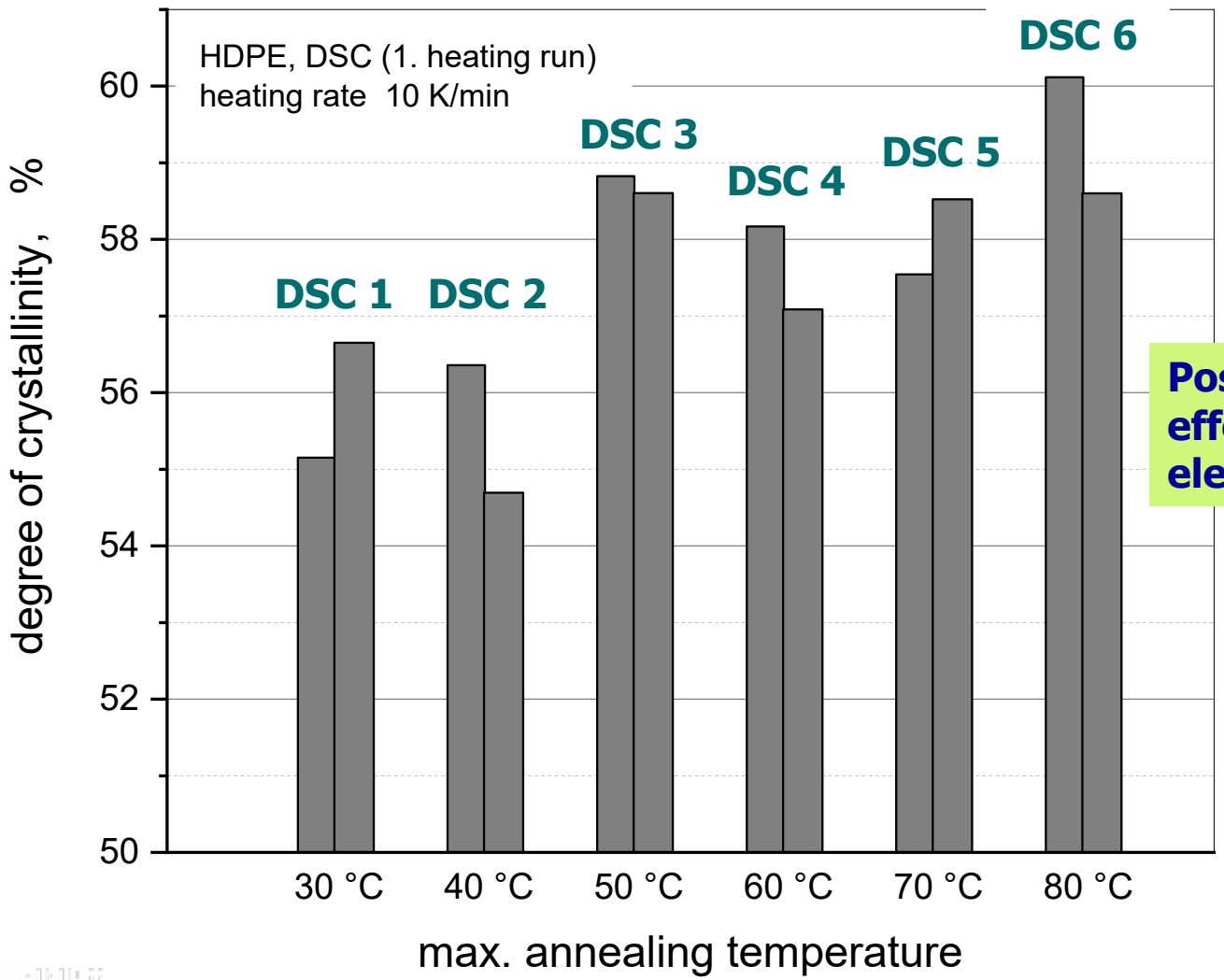
Cystallinity for the various temperature steps



Post-crystallization effect after storage at elevated temperatures

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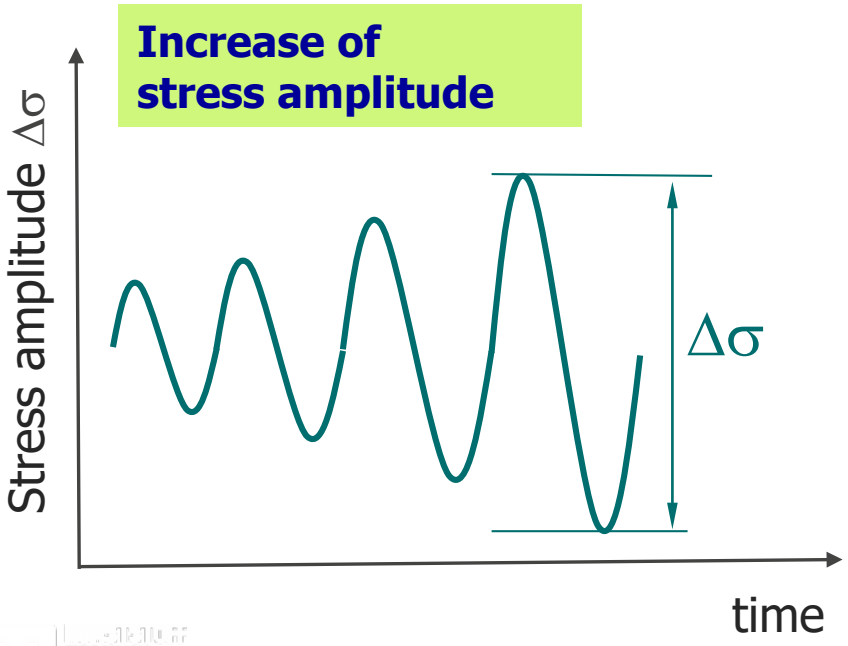
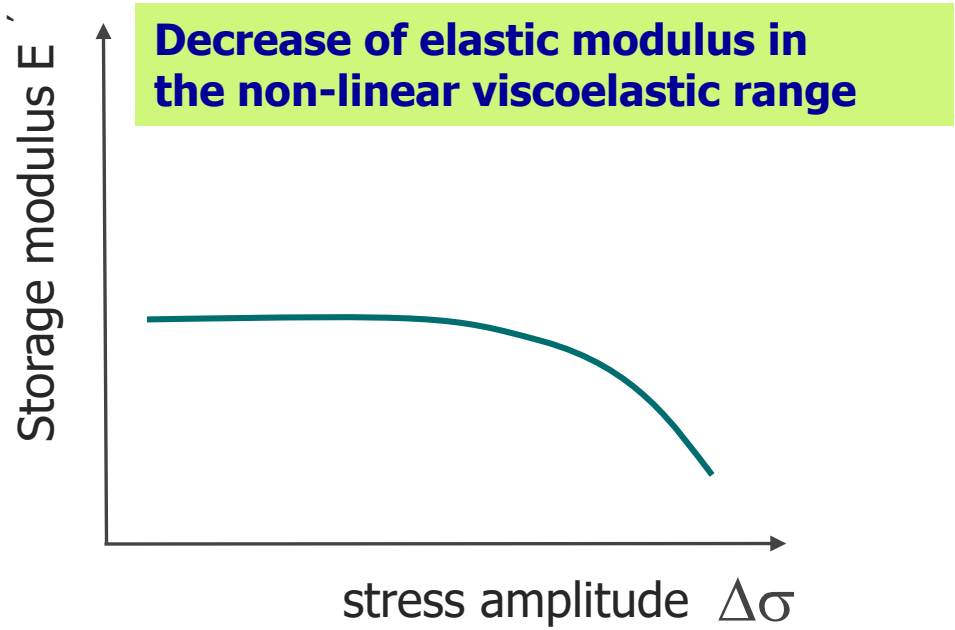
DMA with increasing stress amplitude

Device:	Dynamic Mechanical Analyzer MCR 702 MultiDrive (Anton Paar, GmbH, Austria)
Loading mode:	tensile, bar specimens, cross-sectional area: 2 x 2 mm
Clamping length:	18.5 mm
Loading:	stress amplitude 0.1 MPa to 8 MPa
Temperatures:	30, 40, 50, 60, 70, 80 °C





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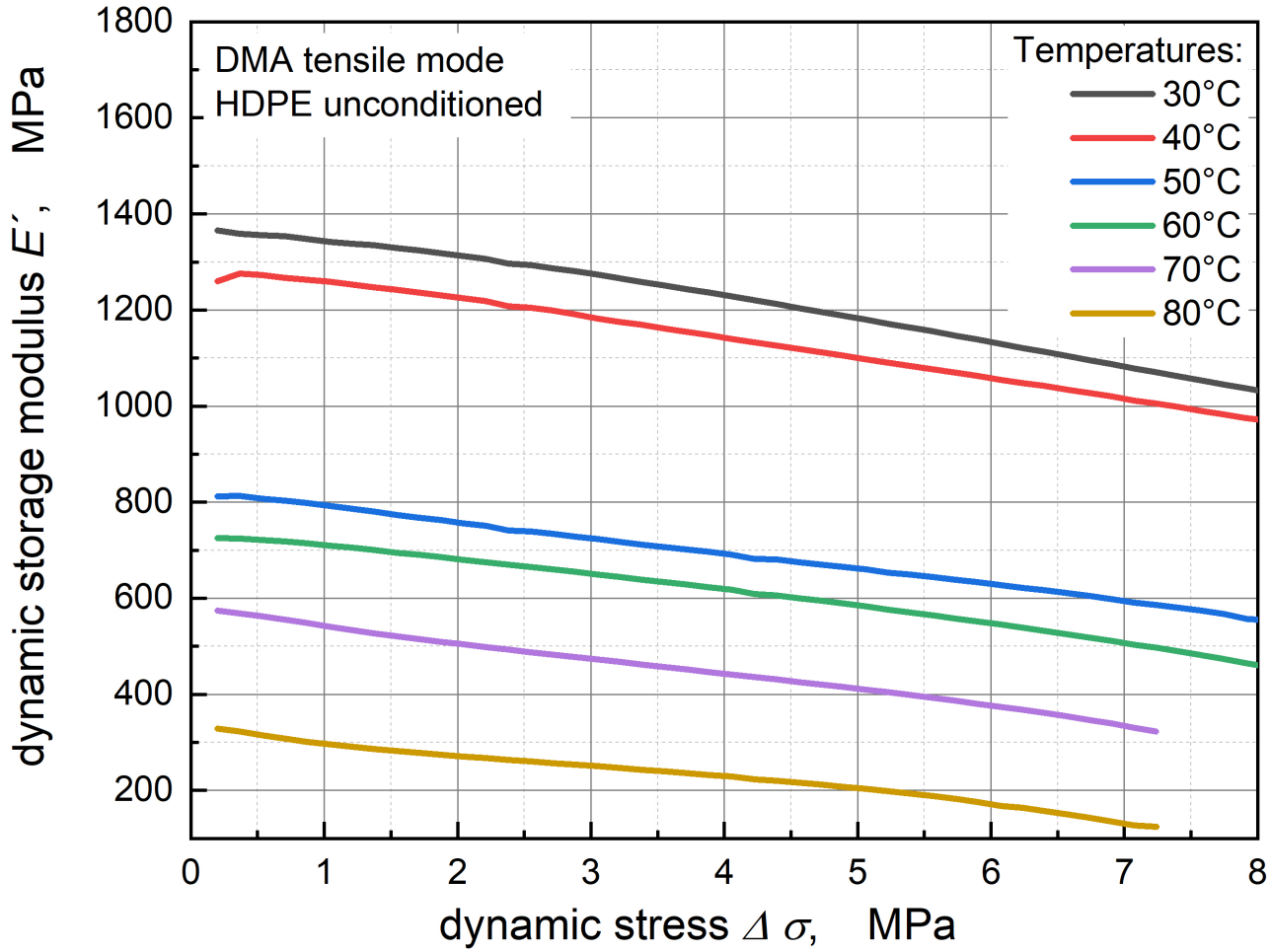
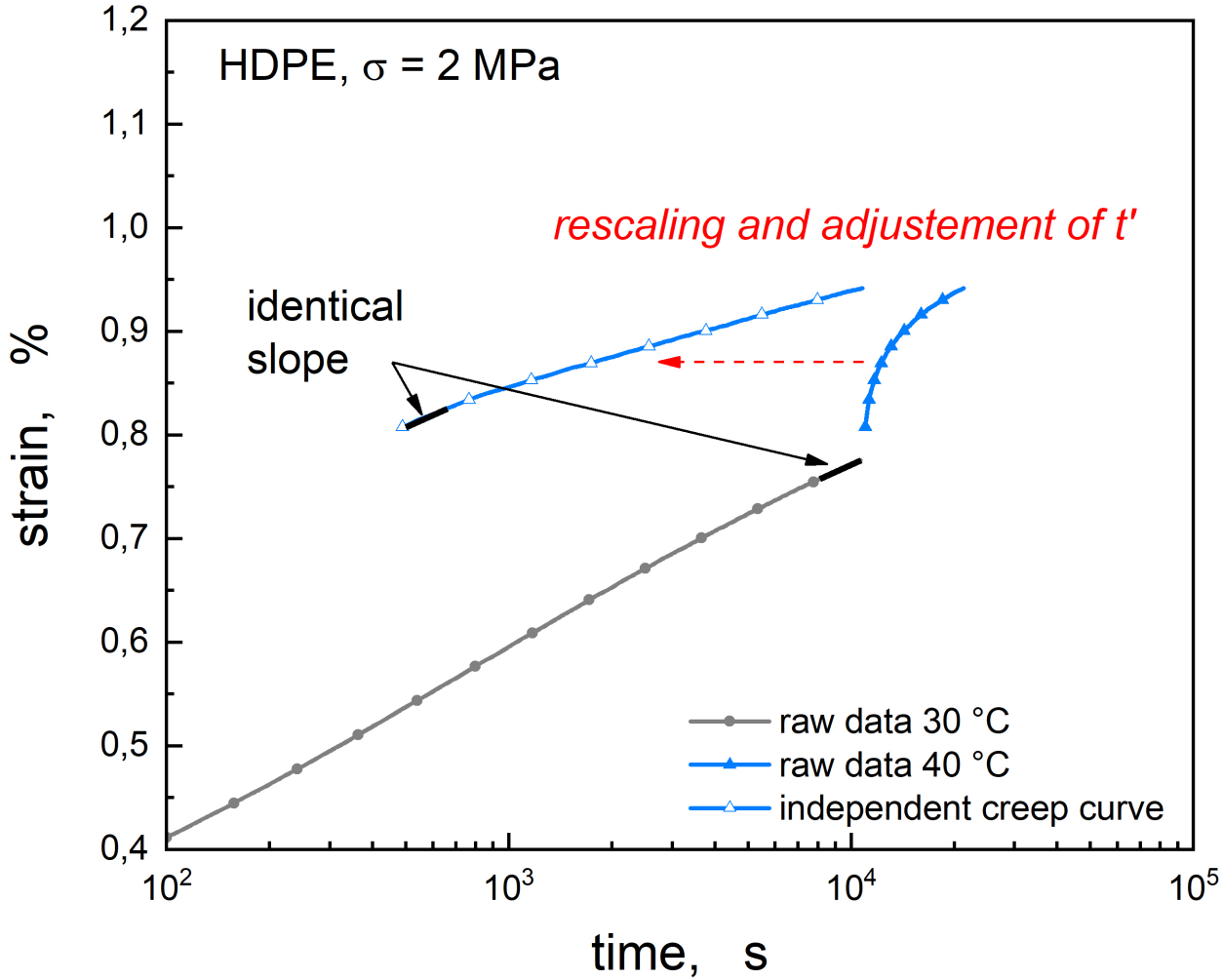
DMA with increasing stress amplitude



No distinct linear viscoelastic range even at low stress level

Results – Creep strain curves by SIM

Definition of virtual starting times



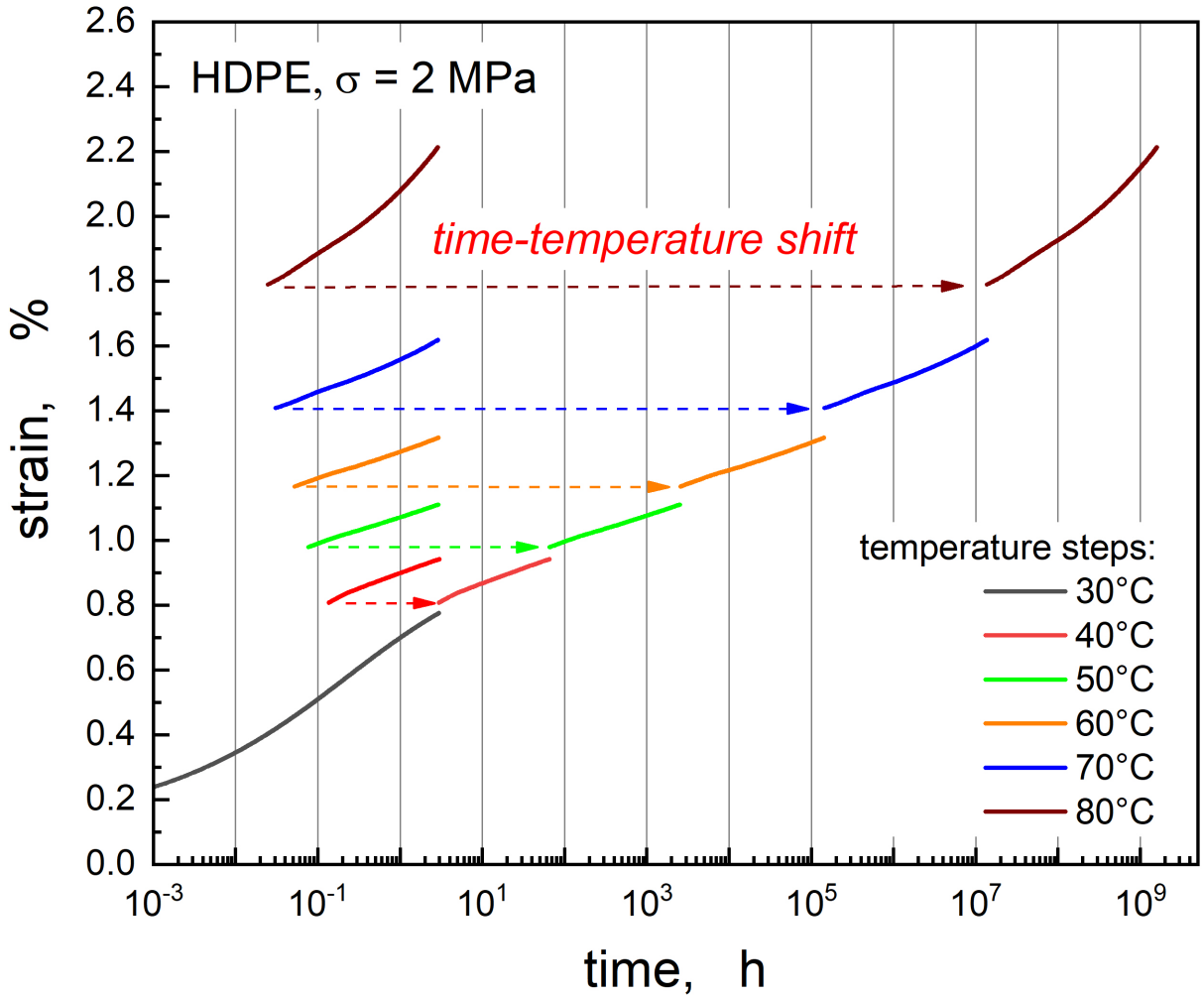
Uncertainties:

Undefined creep in the heating phases

Empirical time adjustment

Results – Master curves by SIM

Time-temperature superposition



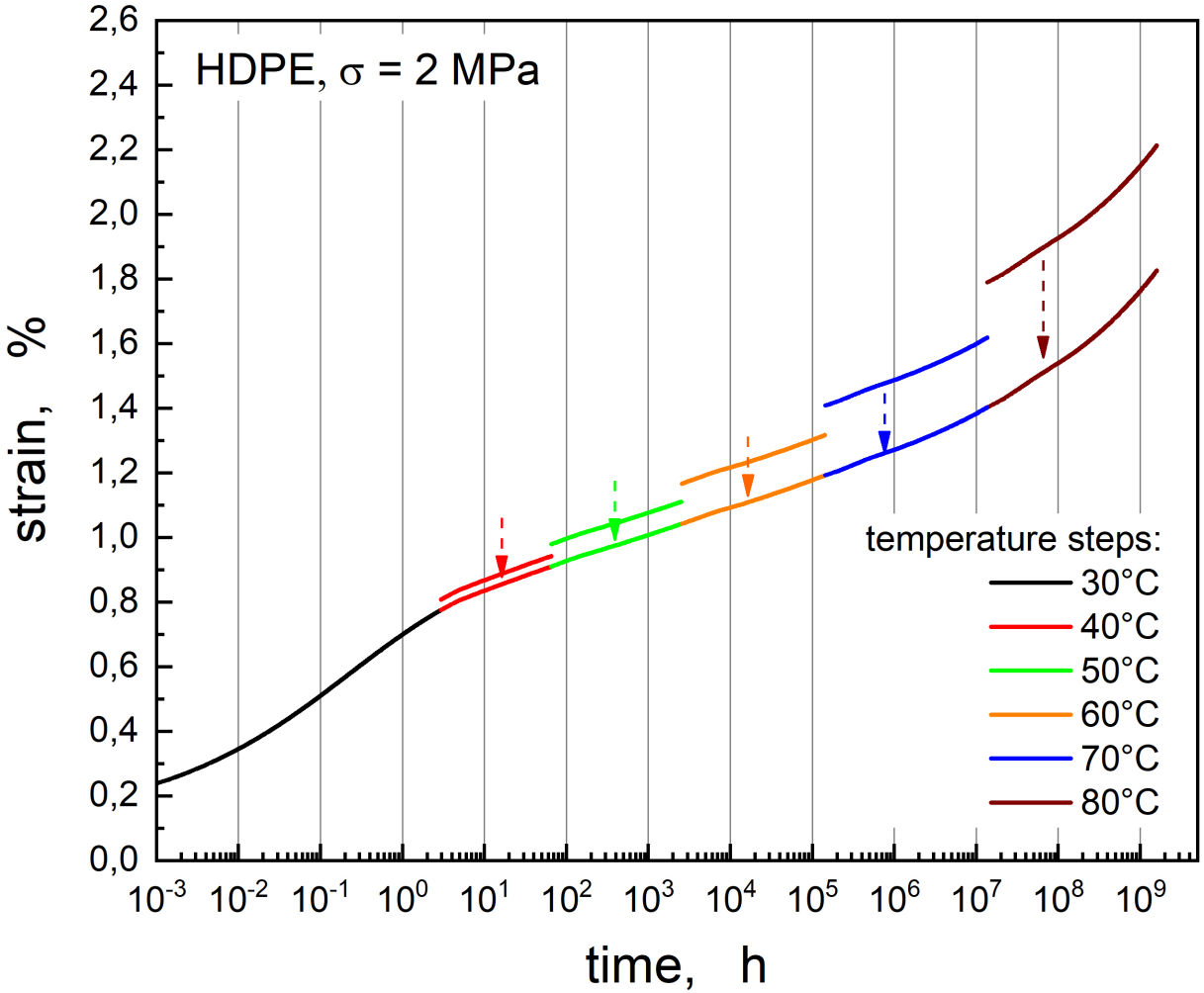
Horizontal curve shift according to standard SIM procedure

Results – Master curves by SIM

Curve adjustment

Vertical curve shift to account for

- **Non-linear viscoelasticity**
- **curve gaps in the heating phases**



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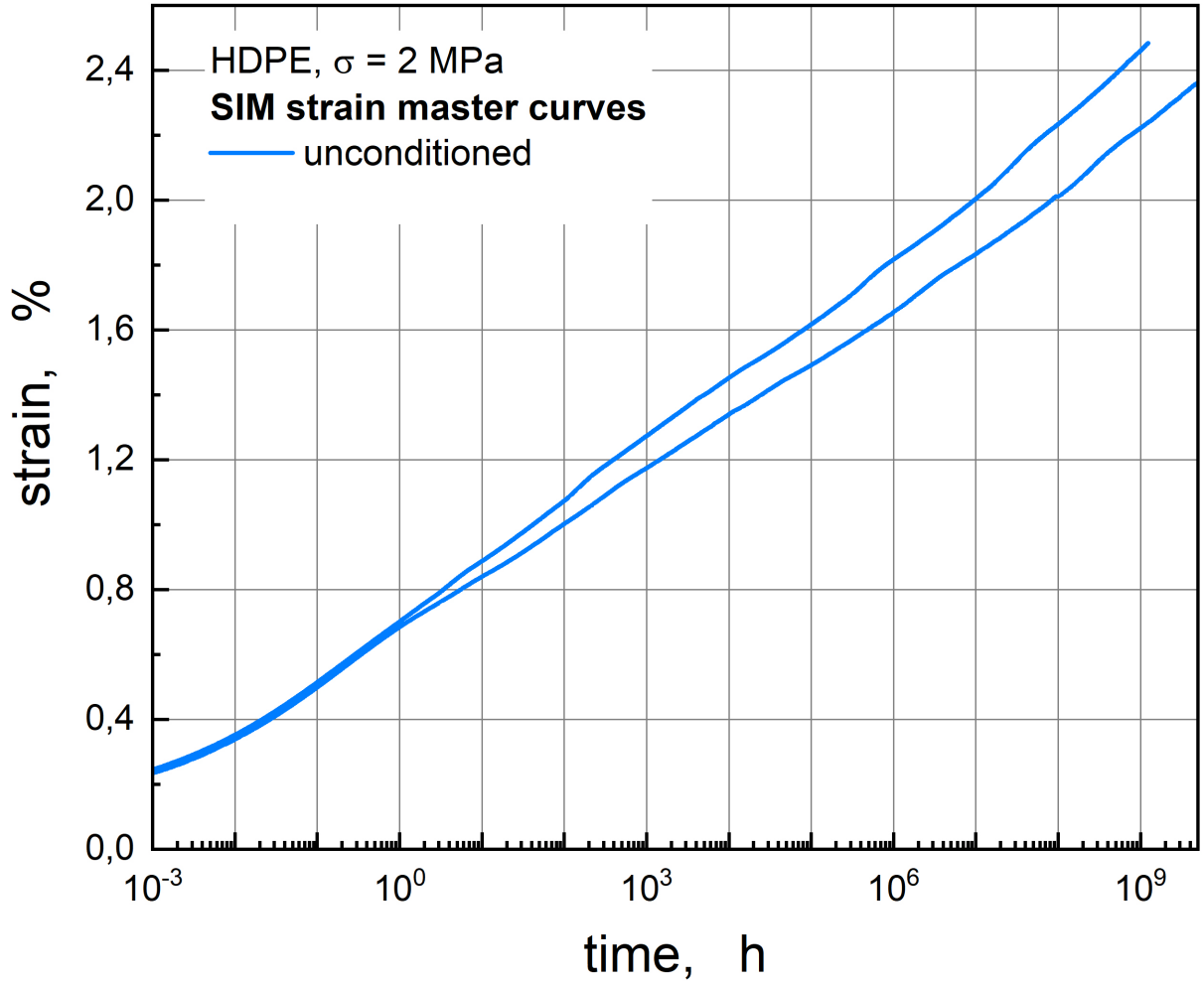
CREEP STRAIN - Influence of thermal pre-treatment

Limited reproducability

due to:

Morphological differences of the various specimens

Morphological changes during the creep tests



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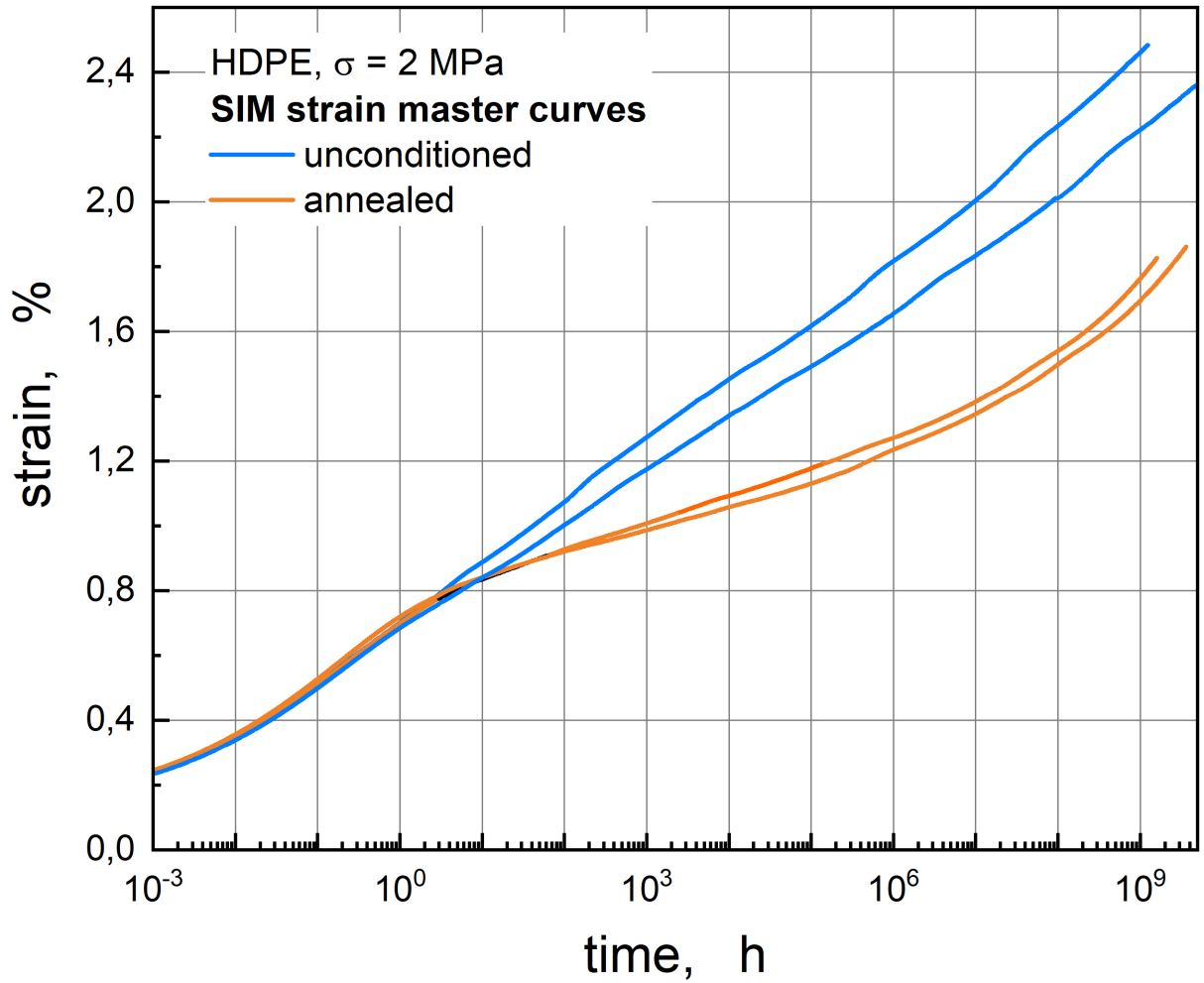
CREEP STRAIN - Influence of thermal pre-treatment

Reduced creep tendency for annealed specimens

due to:

physical ageing effects



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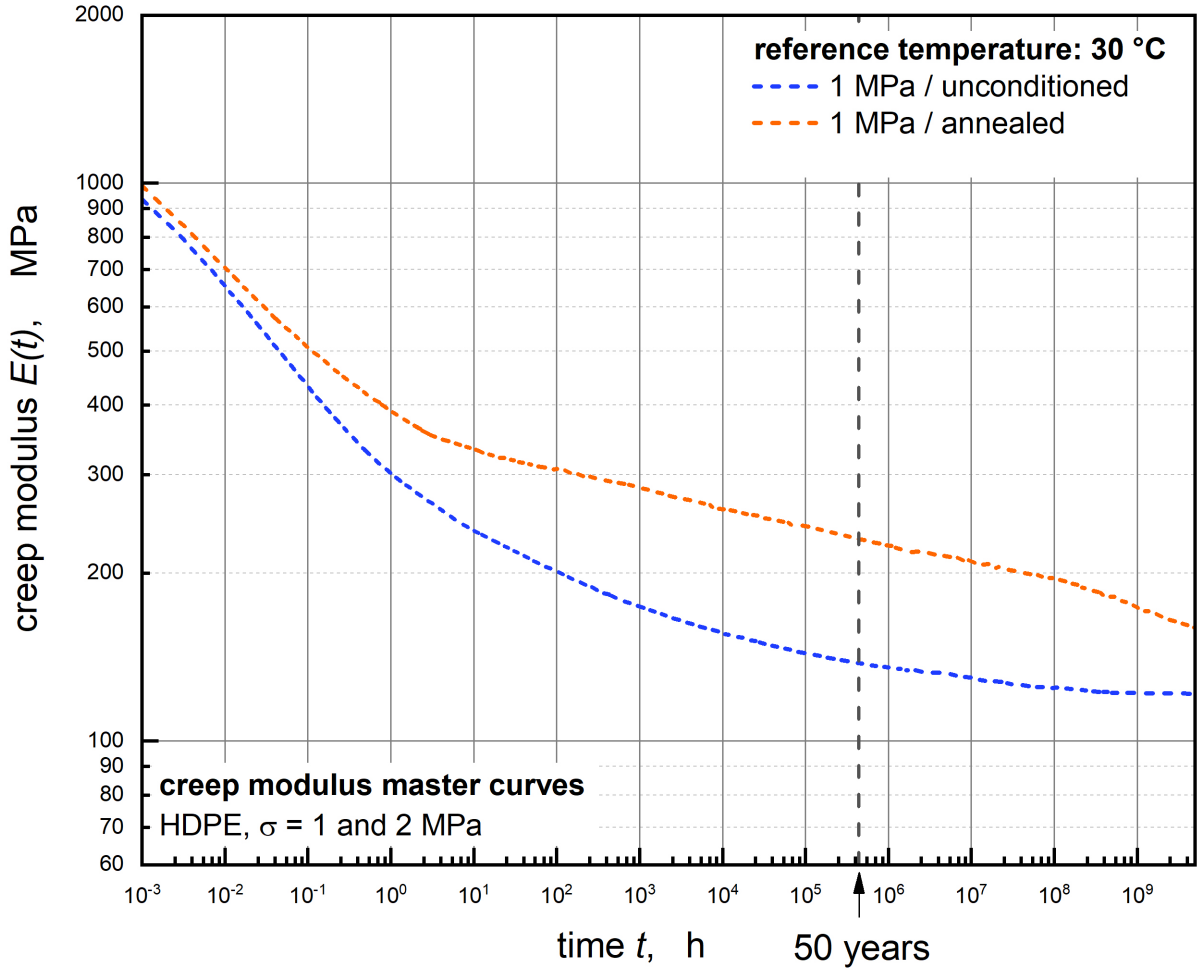
CREEP MODULUS - Influence of thermal pre-treatment

Deviations in creep modulus

due to:

Morphological differences between the annealed an the unconditioned state



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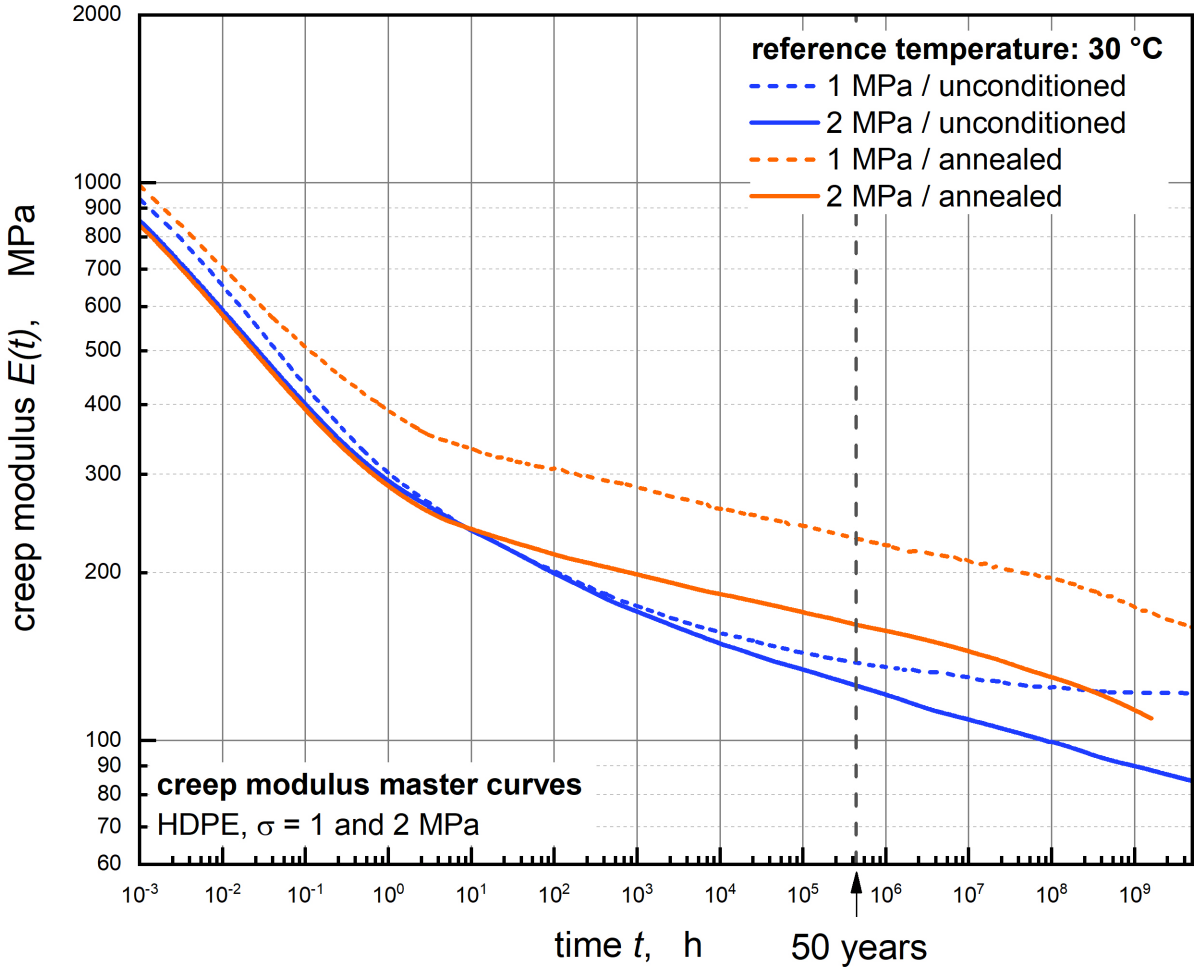
CREEP MODULUS - Influence of thermal pre-treatment

Deviations in creep modulus

due to:

Morphological differences between the annealed an the unconditioned state

Noticeable influence of stress with decreased modulus level at 2 MPa



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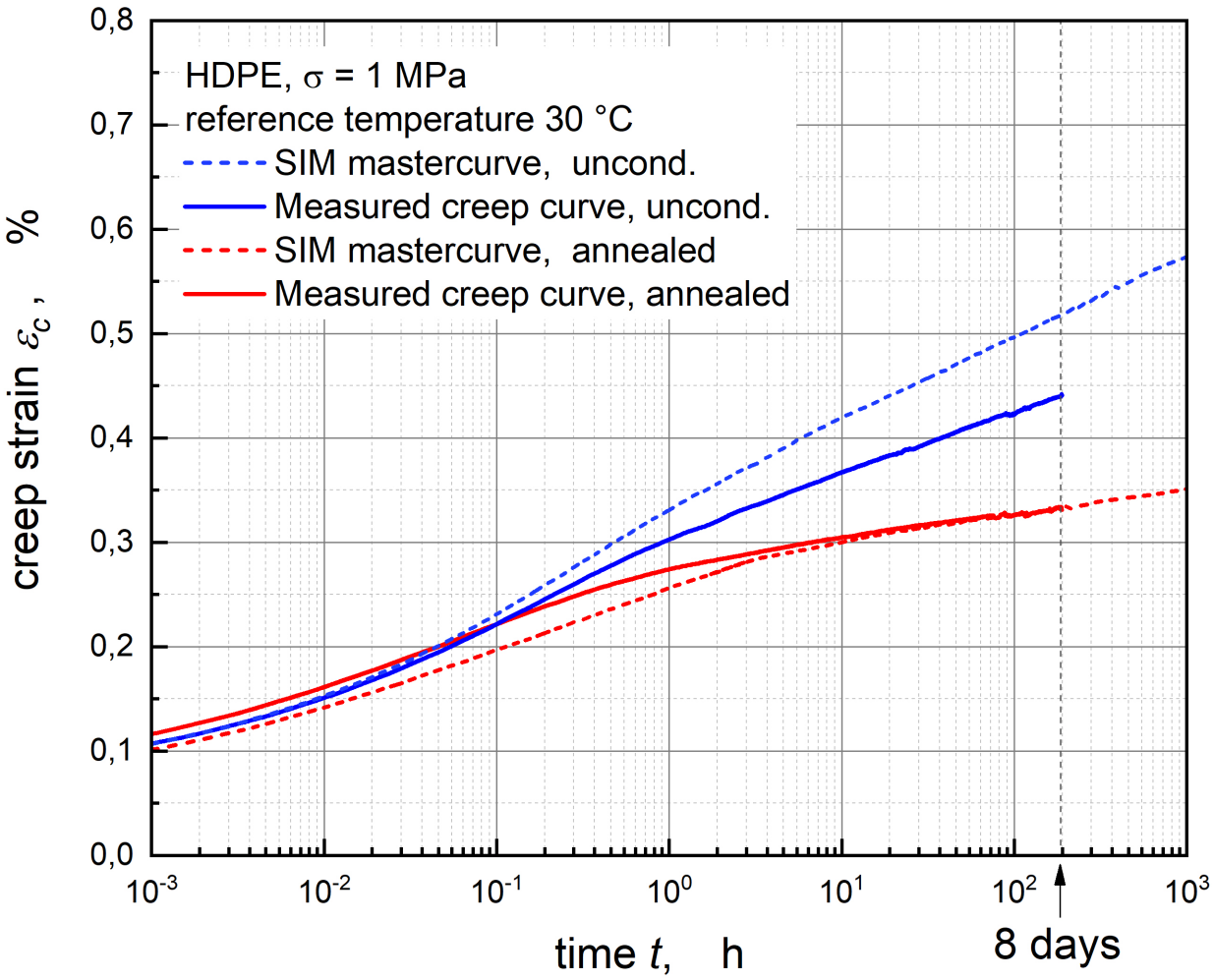
Comparison of SIM results to conventional creep curves

CREEP STRAIN

Acceptable agreement of SIM master curves and measured creep curves

particularly for the annealed material state

at least for a limited time range up to 8 days



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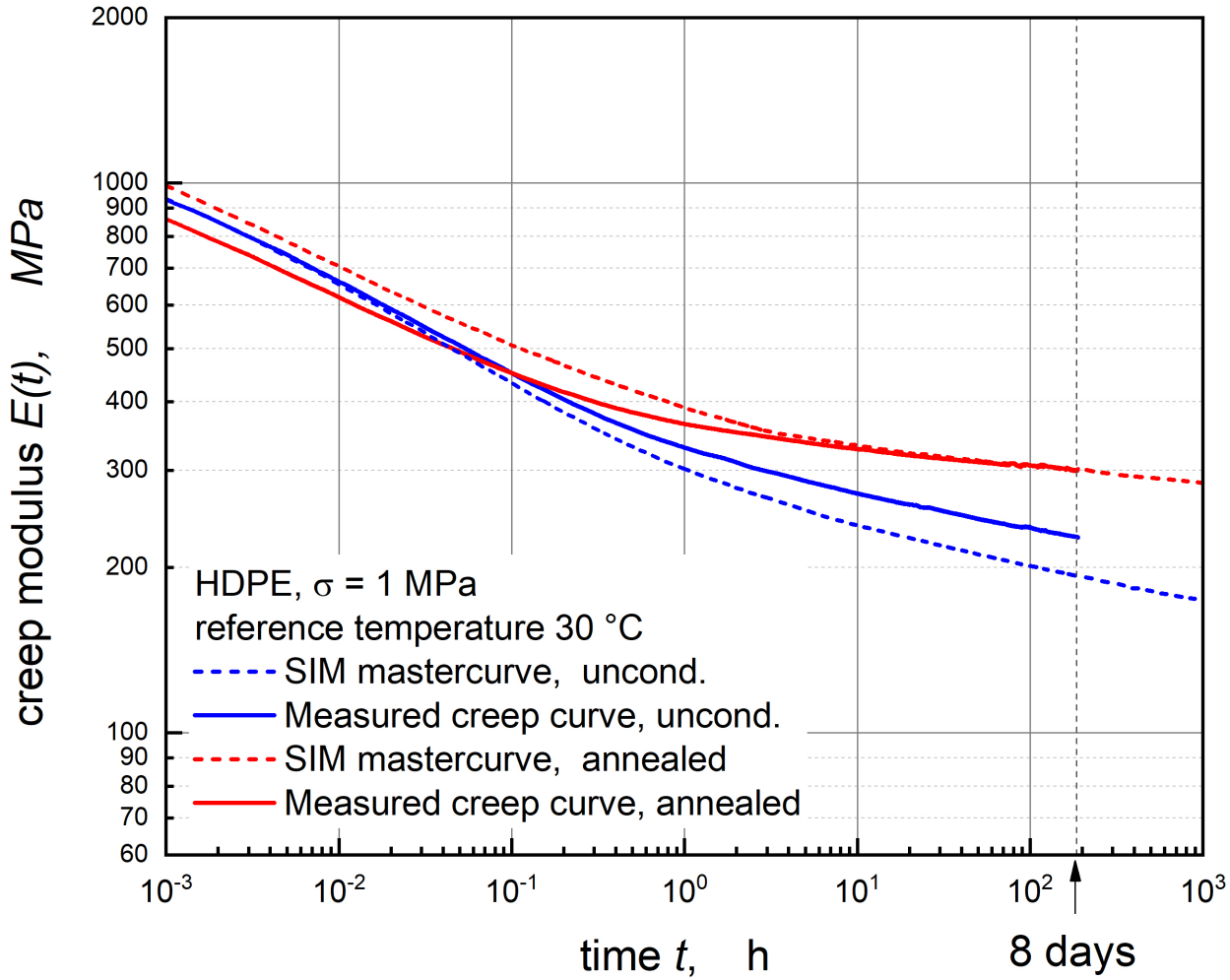
Comparison of SIM results to conventional creep curves

CREEP MODULUS

Corresponding results for creep modulus

Deviating creep tendencies between the various material states „annealed" vs. „unconditioned"



Conclusions

- **SIM provides an efficient method for long-term estimation of the creep behaviour: 1 day measurement to creat a master curve for service oriented long-term prediction.**

- **Reliability of the resulting master curves is dependent on:**
  - ⇨ **Material parameters: morphology, thermo-mechanical properties, range of linear viscoelasticity, thermal conductivity**
  - ⇨ **Experimental parameters: particularly heating rate for temperature steps and the SIM procedure (definition of virtual starting times, curve shift factors)**

- **The current results for HDPE show plausible creep modulus master curves created by SIM in good agreement with corresponding conventional creep measurements.**

Many thanks to…

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