University *of Ljubljana*
Faculty *of Mechanical Engineering*

University *of Ljubljana*
Faculty *of Mathematics and Physics*

Time-temperature superposition (tTs) and spectral analysis of PIB 1 and PIB 2 (temperature range: -20 °C to 60 °C)

Partners: **University of Ljubljana, Faculty of Mechanical Engineering**
Laboratory for Numerical Modelling and Simulation
Center for Experimental Mechanics
Laboratory for Sustainable Technologies in Buildings
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Slovenian National Building and Civil Engineering Institute
University of Ljubljana, Faculty of Mathematics and Physics

No. of ARRS project: L2-3172

ARRS project title: Development of technical guidelines for quadruple glazing

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Report title: **Time-temperature superposition (tTs) and spectral analysis of PIB 1 and PIB 2 (temperature range: -20 °C to 60 °C)**

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1. SAMPLE PREPARATION

Instrument:	Modular rotational rheometer MCR302, Anton Paar
Procedure details:	Temperature: 60 °C Sample geometry: Cylindrical disk (PP25/S sensor geometry): d=25 mm; h=1mm (gap) System configuration: upper plate PP25/S (sandblasted); lower plate P-PTD 200; H-PTD 200 SN83000362-804264

Figure 1 shows cylindrical disk sample (example of PIB 2), trimmed around PP25/S sensor geometry at 60°C.

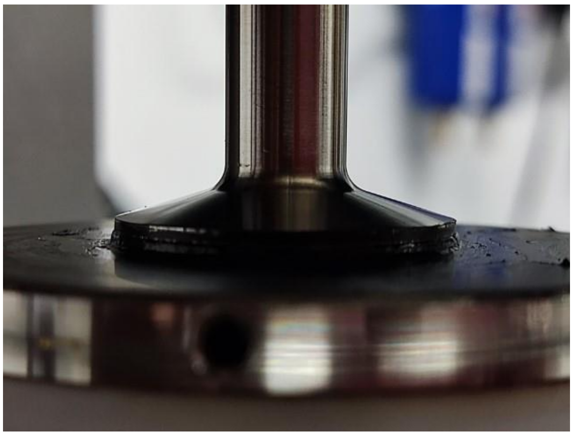

Figure 1: Trimmed cylindrical sample at 60°C.

2. MEASUREMENT

2.1. Rheology: temperature sweep

Instruments: Modular rotational rheometer MCR302, Anton Paar

Procedure details:

Shear stress: 1000 Pa (within LTVE)

Temperature range: -20°C - 60 °C

Heating: 3°C/min

Frequency: 1Hz (constant)

System configuration: upper plate PP25/S (sandblasted); lower plate P-PTD 200; H-PTD 200 SN83000362-804264

Gap: ~1 mm (changing due to temperature to sustain normal force of 0N)

Repetitions: 2 repetitions per material

Figure 2 shows results of standard temperature sweep measurements used to determine viscoelastic response of PIB 1 and 2 over a wide temperature range (experimental error ±4,8%).

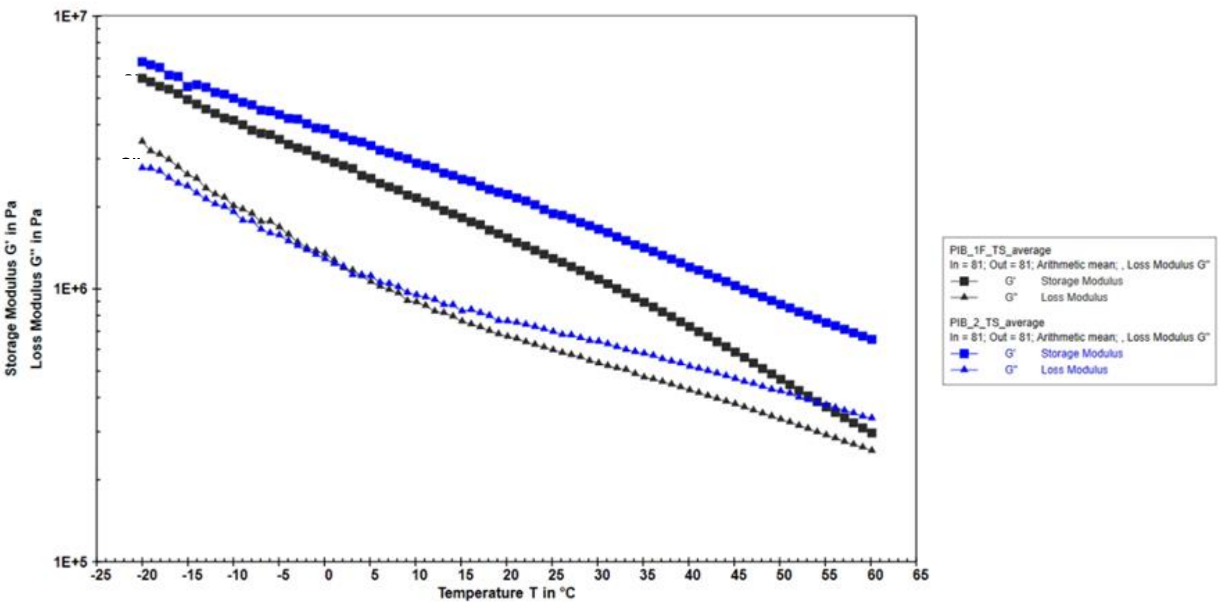

***Figure 2**: Storage G' and loss G'' modulus of PIB 1 and 2 over a wide temperature range at constant frequency of oscillation and constant shear stress (in LVTE) (average values are presented).*

2.2. Rheology: segments for tTs (-20 °C to 60°C)

Instruments: Modular rotational rheometer MCR302, Anton Paar

Procedure details: Shear stress: 1000 Pa (within LTVE)

Temperature of segments: -20°C, 10°C, 0°C, 10°C, 20°C, 30°C, 40 °C, 50 °C, 60 °C

Frequency range (experimental window): 0,01Hz – 100 Hz

System configuration: upper plate PP25/S (sandblasted); lower plate P-PTD 200; H-PTD 200 SN83000362-804264

Gap: ~1 mm (changing due to temperature to sustain normal force of 0N)

Repetitions: 2 repetitions per material

Figures 3a and 3b show the results of frequency sweep measurements at constant shear stress (in LVTE) at different temperatures, i.e. isothermal segments of storage G' and loss G'' modulus, for PIB 1 (experimental error ±6%).

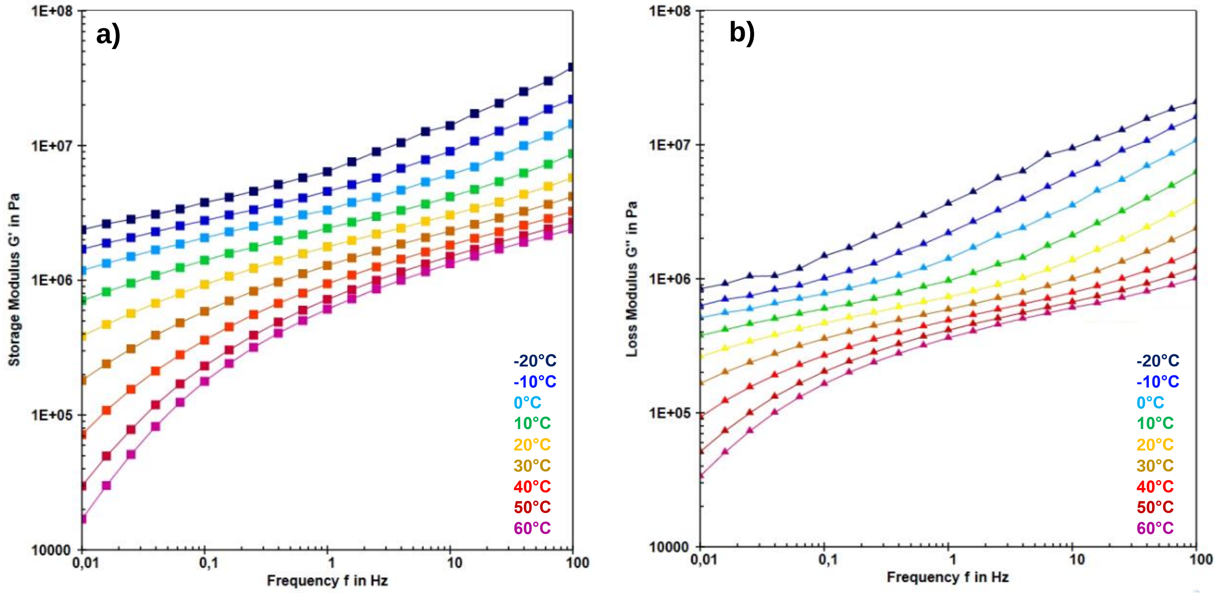

***Figure 3**:* a) Storage G' and b) loss modulus G'' as a function of frequency at different temperatures for PIB 1 (average values are presented).

Figures 4a and 4b present the results of frequency sweep measurements at constant shear stress (in LVTE) at different temperatures, i.e. isothermal segments of storage G' and loss G'' modulus, for PIB 2 (experimental error ±8%).

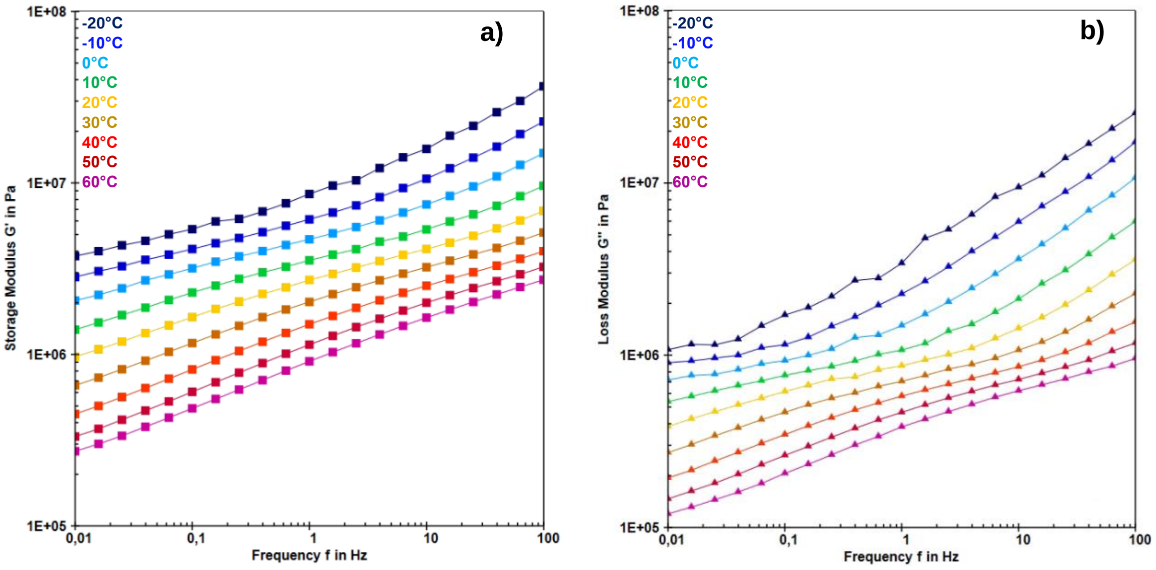

***Figure 4**: a) S*torage G' and b) loss modulus G'' as a function of frequency at different temperatures for PIB 2 (average values are presented).

3. ANALYSIS

Software: RheoCompass, Anton-Paar

Procedure details: tTs: standard (horizontal shifting)

Spectral and inverse spectral analysis: Edge preserving method

3.1. tTs and spectral analysis

Figure 5a shows master curves of storage G' and loss G'' modulus which were obtained by shifting isothermal segments (black curve) and calculated from shear relaxation time spectrum (red curve) for PIB 1 material. Figure 5b presents corresponding shift factors (WLF included), while Figure 5c presents relaxation time spectrum. The relaxation modulus in time domain calculated from the spectrum is presented in Figure 5d. All results were determined at reference temperature of 20°C. Please note that for tTs analysis the segments at following temperatures were used: -20°C, 0°C, 20°C, 40°C and 60°C.

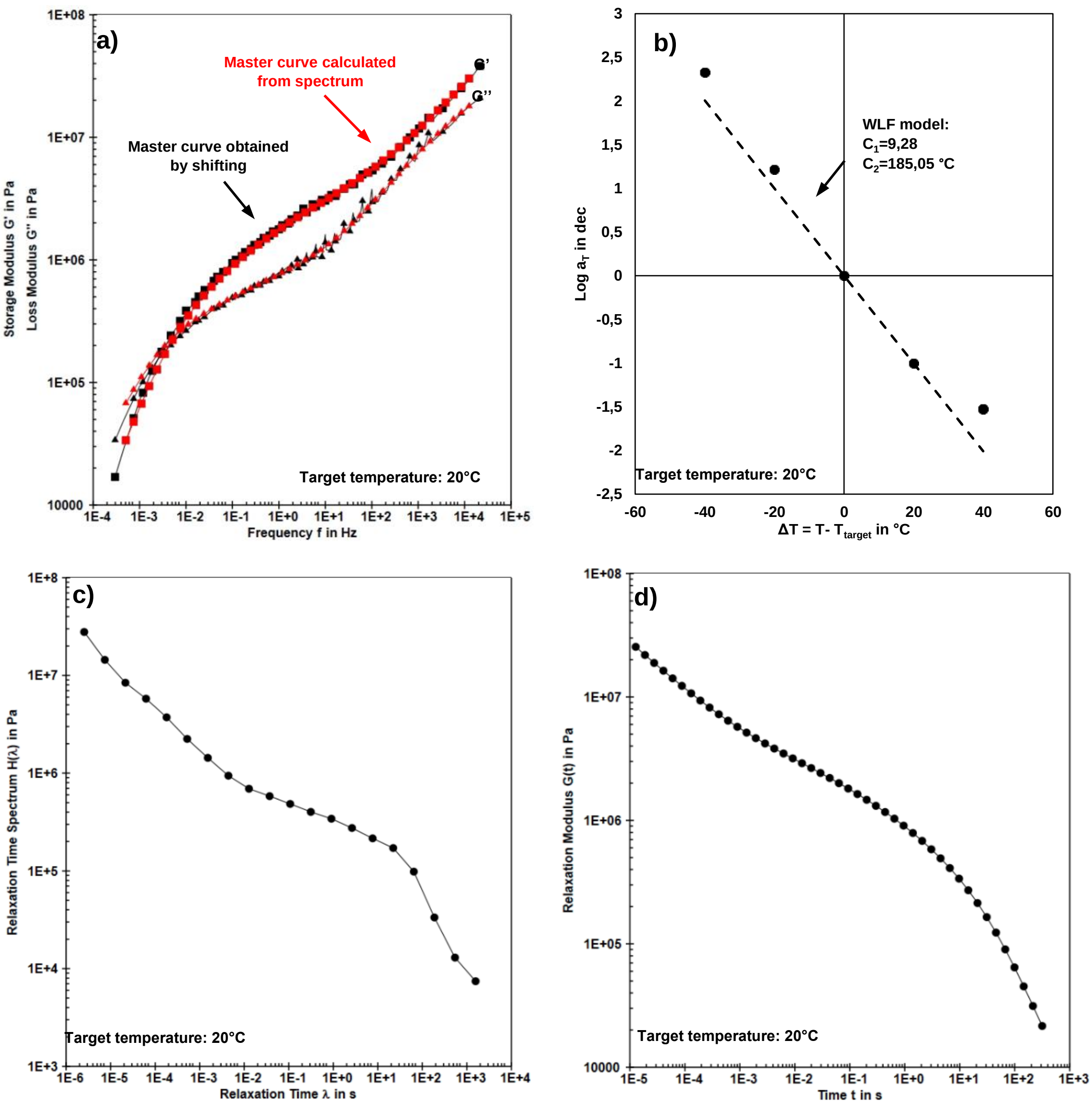

***Figure 5**:* a) Master curves of storage G' and loss G'' modulus, b) horizontal shift factors (WLF included), c) relaxation time spectrum and d) relaxation modulus in time at target temperature of 20°C for PIB 1.

Figure 6a presents master curves of storage G’ and loss G’’ modulus obtained by shifting isothermal segments (blue curve) and calculated from shear relaxation time spectrum (red curve) for PIB 2 material. Figure 6b presents corresponding shift factors (WLF included), while Figure 6c presents relaxation time spectrum. Relaxation modulus in time domain calculated from the spectrum is presented in Figure 6d. All results were determined at target temperature of 20°C. Please note that for tTs analysis the segments at following temperatures were used: -20°C, 0°C, 20°C, 40°C and 60°C.

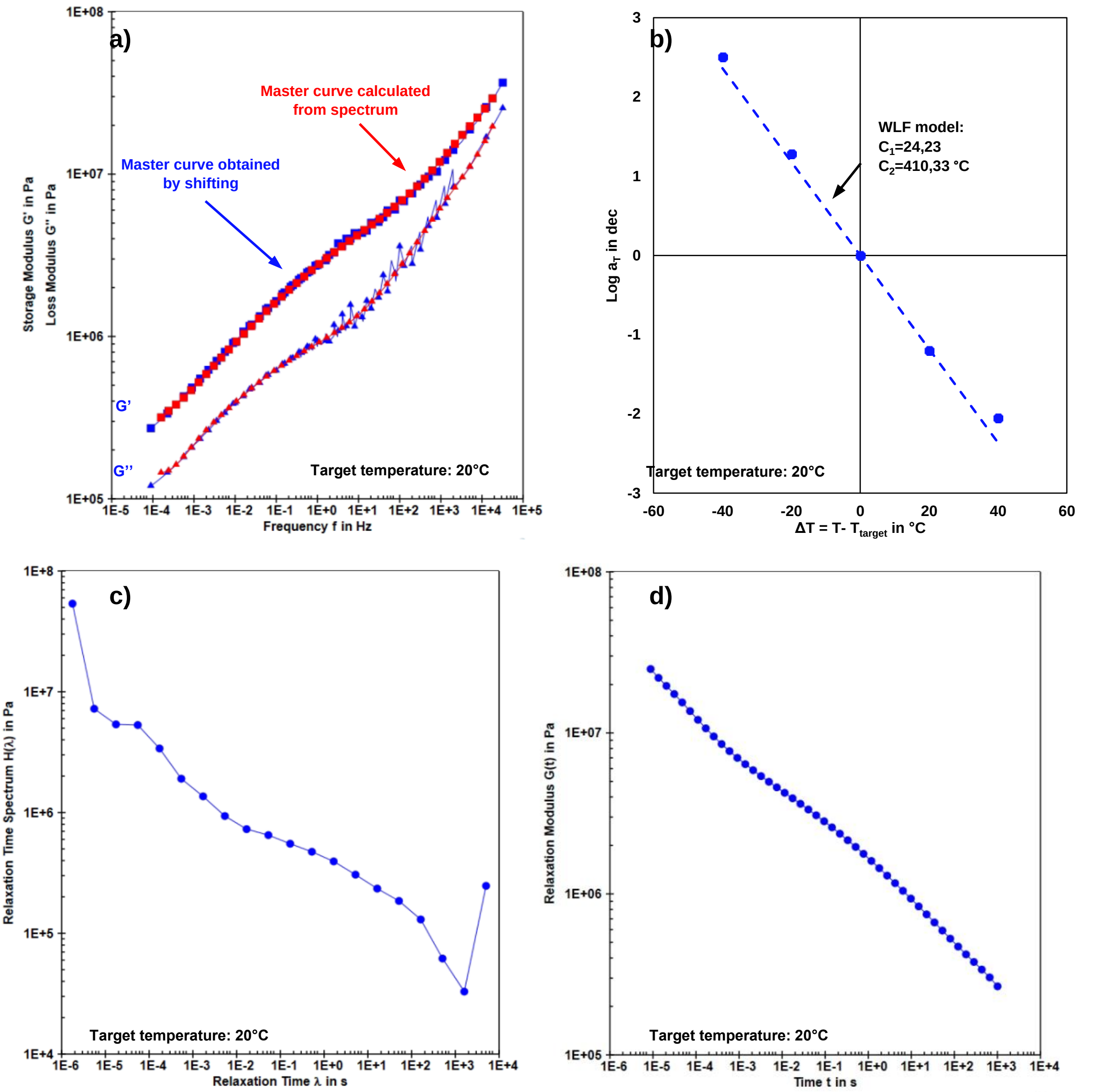

Figure 6: a) Master curves of storage G’ and loss G’’ modulus, b) horizontal shift factors (WLF included), c) relaxation time spectrum and d) relaxation modulus in time at target temperature of 20°C for PIB 2.

Figures 7a and 7b present the comparison of viscoelastic properties and shear relaxation time spectrum. The results indicate higher molecular weight of PIB 2 compared to PIB 1, resulting in slower relaxation at higher temperature / low frequencies / long relaxation times.

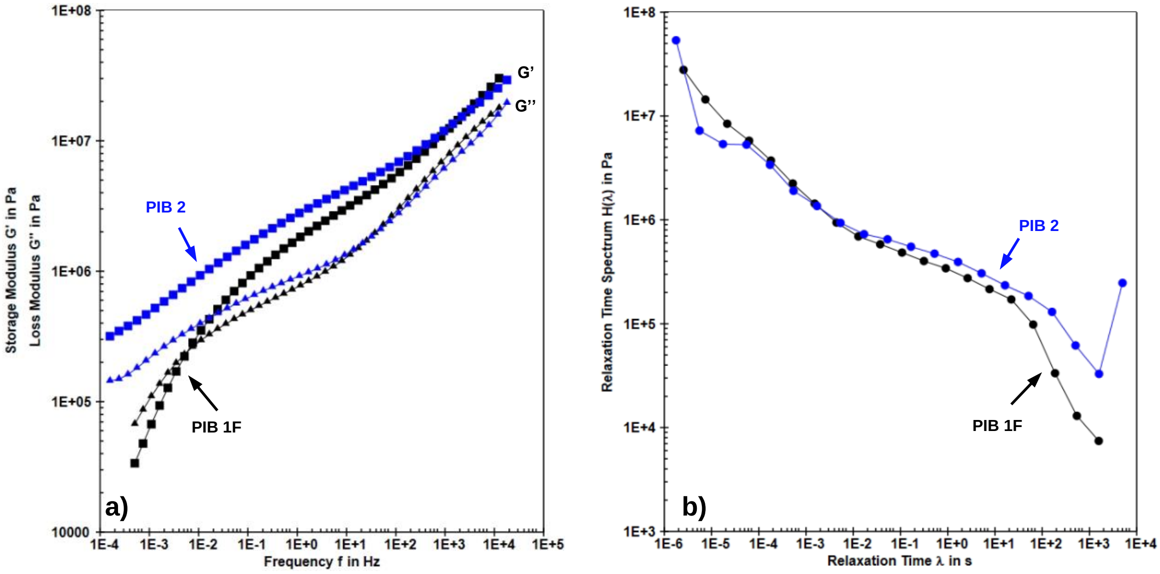

***Figure 7**:* Comparison of a) viscoelastic properties (*s*torage G' and loss modulus G''), b) shear relaxation time spectrum for PIB 1 and PIB 2.

4. CONCLUSION

Time dependent properties were measured and master curves of storage G' and loss modulus G'' were obtained by shifting isothermal experimental segments and calculated from shear relaxation time spectrum. The curves were constructed at reference temperature of 20°C. Moreover, the results indicate higher molecular weight of PIB 2 compared to PIB 1, resulting in slower relaxation at higher temperature / small frequencies / long relaxation times.