University of Ljubljana
Faculty of Mechanical Engineering

University of Ljubljana
Faculty of Mathematics and Physics

Spectral analysis of PIB 1 and PIB 2 after thermal stabilization

Partners: **University of Ljubljana, Faculty of Mechanical Engineering**
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Center for Experimental Mechanics
Laboratory for Sustainable Technologies in Buildings
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No. of ARRS project: L2-3172
ARRS project title: Development of technical guidelines for quadruple glazing

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Report title: **Spectral analysis of PIB 1 and PIB 2 after thermal stabilization**

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1. SAMPLE PREPARATION

Instrument: Modular rotational rheometer MCR302, Anton Paar

Procedure details: Temperature: -20 °C -180 °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.

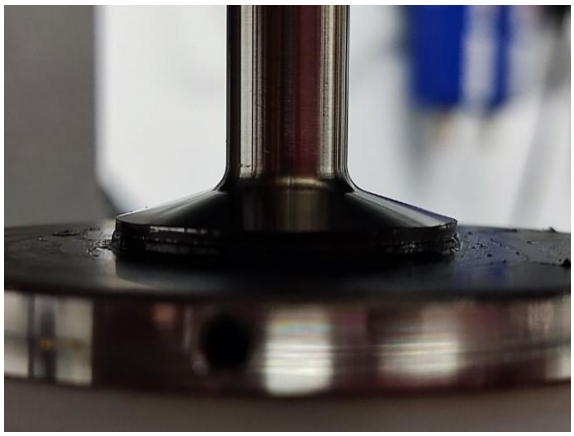

Figure 1: Trimmed cylindrical sample at 60°C.

After the sample preparation process at 60 °C, the sample was cooled to the temperature of 15 °C. Thermal stabilization of both PIB samples was achieved by annealing the material at 180 °C (temperature gradient 3 C/min) for 10 minutes at shear stress of 200 Pa.

2. MEASUREMENT

2.1. Rheology: segments for tTs

Instrument: Modular rotational rheometer MCR302, Anton Paar

Procedure details:

Shear stress: 200 Pa (within LTVE)

Temperature of segments: -20°C, 0°C, 20°C, 40°C, 60°C, 80°C, 100 °C, 120 °C, 140 °C, 160 °C, 180 °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

Figure 2 shows the results of frequency sweep measurements at different temperatures, i.e. isothermal segments of storage G' and loss G'' modulus, for PIB 1 (a, b) and for PIB 2 (c, d) (experimental error ±6%).

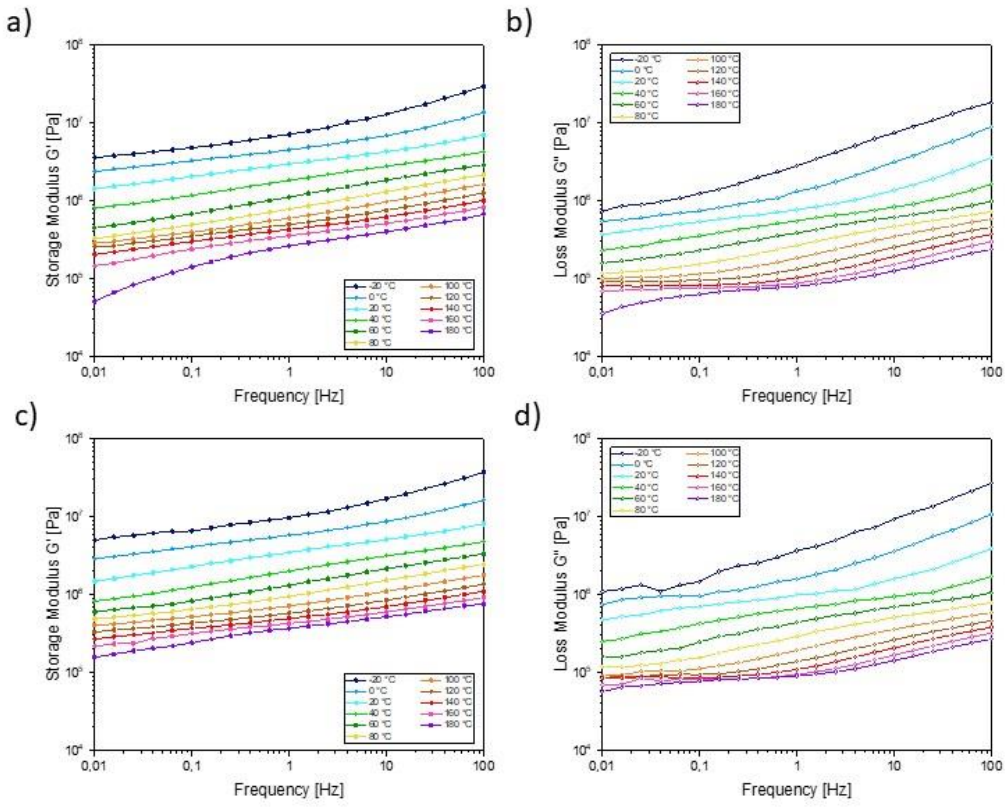

***Figure 2**: Storage G' and loss G'' modulus as a function of frequency at different temperatures for PIB 1 (a, b) and PIB 2 (c, d) after thermal stabilization (average values are shown).*

3. ANALYSIS

Software: RheoCompass, Anton-Paar

Procedure details: tTs: standard (horizontal shifting)

Spectral and inverse spectral analysis: Edge preserving method

3.1. Time-temperature superposition (tTs) and spectral analysis after thermal stabilization

Figure 3a shows the master curves of storage G' and loss G'' modulus, which were calculated from shear relaxation time spectrum for PIB 1 (blue) and for PIB 2 (red) material, respectively. Figure 3b presents the corresponding shift factors (WLF included), while Figure 3d presents the relaxation time spectrum. Relaxation modulus in time domain, which were calculated from the spectrum are presented in Figure 3c. All results were determined at target temperature of 100 °C. Please note that for tTs analysis segments at following temperatures were used: -20°C, 0°C, 20°C, 40°C, 60°C, 80 °C, 100 °C, 120 °C, 140 °C, 160 °C and 180 °C.

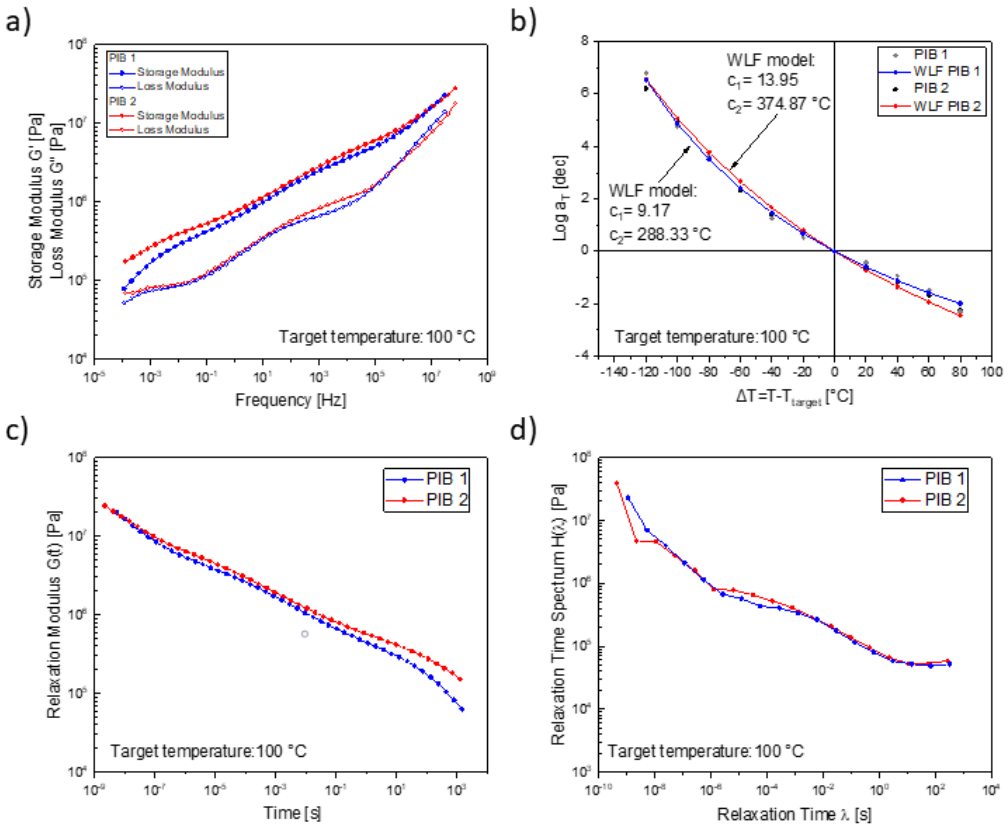

***Figure 3**:* a) Master curves of storage G' and loss G'' modulus, b) horizontal shift factors (WLF included), c) relaxation modulus, d) relaxation time spectrum at target temperature of 20°C for PIB 1 and PIB 2.

Figure 4 presents the comparison of master curves of storage G' and loss G'' modulus, which were obtained by shifting isothermal segments (black curves) and calculated from shear relaxation time spectrum (blue-PIB 1 and red-PIB 2).

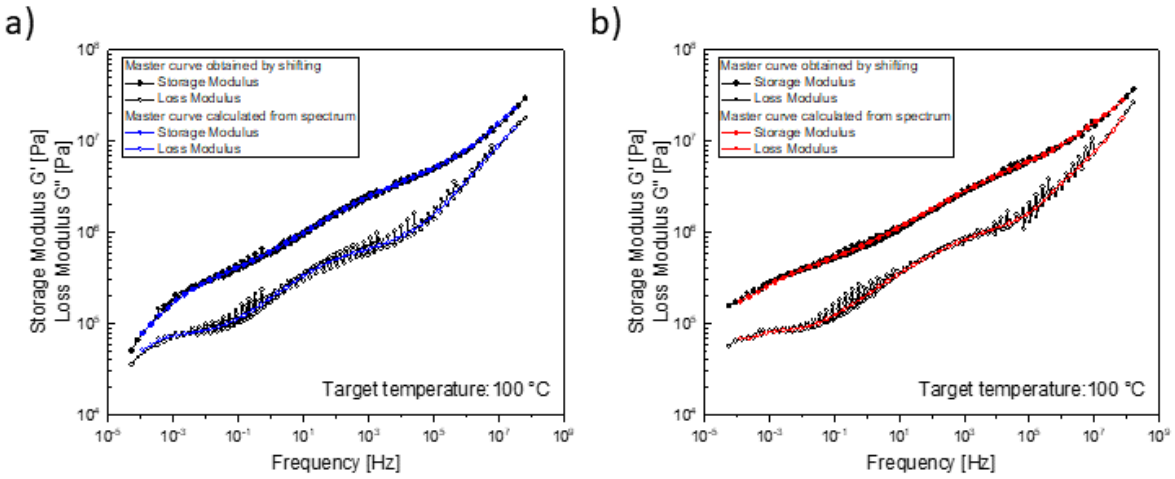

Figure 4: Master curves of storage G' and loss G'' modulus, obtained by shifting isothermal segments (black curve) and calculated from shear relaxation time spectrum (blue/red curve) for a) PIB 1 and b) PIB 2 material.

4. CONCLUSION

The samples were thermally treated at 180 °C for 10 min which led to irreversible structural changes of PIB. During the thermal treatment, the chemical structure of the PIB changed and the mechanical properties became constant.