University of Ljubljana
Faculty of Mechanical Engineering

University of Ljubljana
Faculty of Mathematics and Physics

Thermal and rheological characterization of PIB 1 and PIB 2

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: **Thermal and rheological characterization of PIB 1 and PIB 2**

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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 INSET/pp25/SS/S D:25 mm, SANDB; HETD 400

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

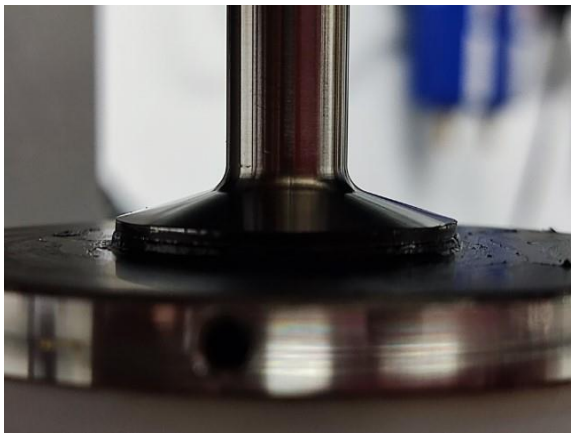

Figure 1: Trimmed cylindrical sample at 60°C.

2. MEASUREMENT

2.1. DSC: phase transition temperatures

Instruments: DSC2500, Ta Instruments

Procedure details: Temperature range: -80°C – 100°C
Heating and cooling rate: 10°C/min
Pan type: Al
Sample geometry: finger kneaded granule
Repetitions: 2 repetitions per material

Figure 2a and 2b show DSC thermograms of PIB 1 and PIB2, respectively, indicating glass transitions at - 65°C.

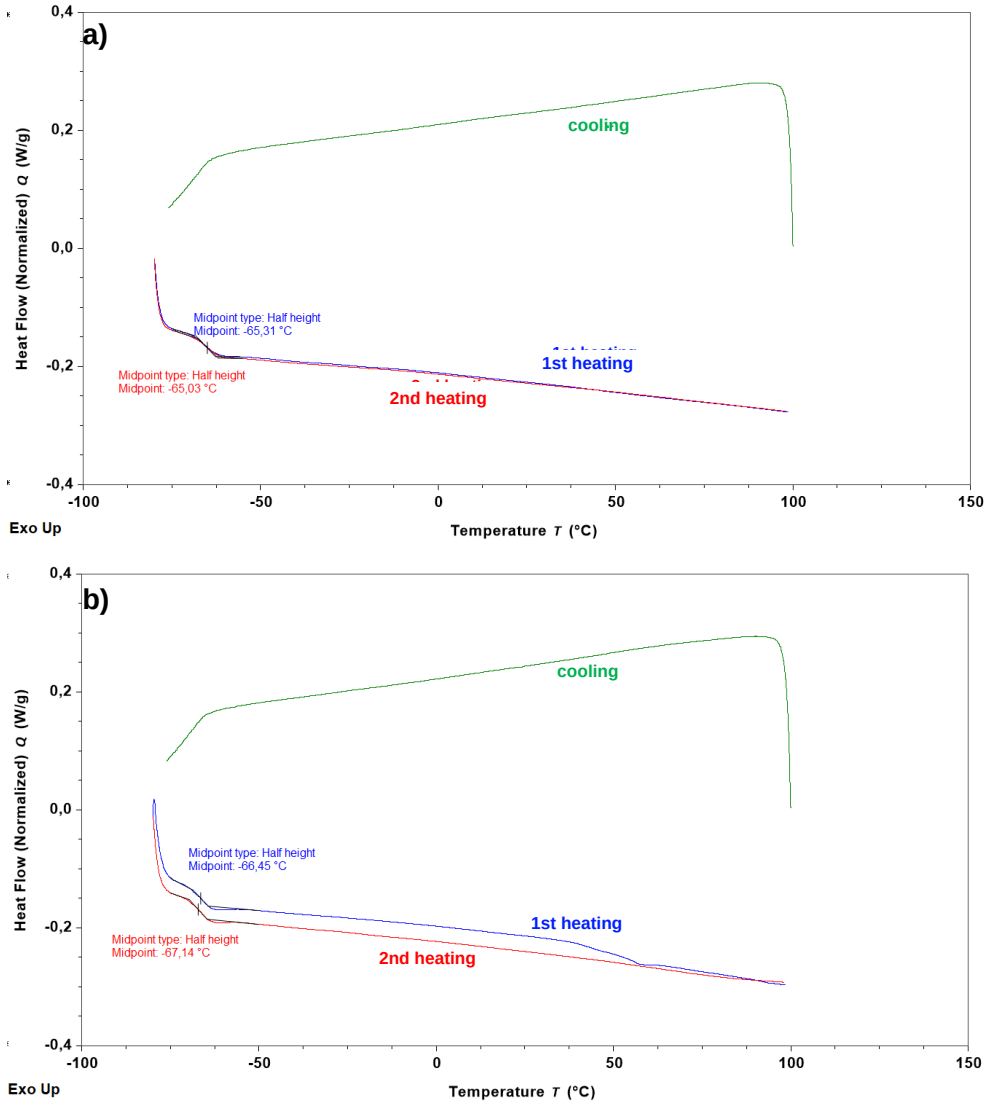

***Figure 2**: DSC thermograms of a) PIB 1 and b) PIB 2 (only 1. repetition is shown).*

2.2. Rheology: determination of LTVE range @ 60°C

Instrument: Modular rotational rheometer MCR302, Anton Paar

Procedure details:

Shear stress: 10 - 10000 Pa

Temperature: 60°C

Frequency: 1 Hz

System configuration: upper plate PP25/S (sandblasted); lower plate INSET/pp25/SS/S D:25 mm, SANDB; HETD 400

Gap: 1 mm

Repetitions: 1 repetition per material

Figure 3 shows rheological amplitude sweep tests, which were performed in order to determine shear stress limit of linear viscoelastic range (LTVE) for PIB 1 and PIB 2, respectively. The results showed that for PIB the LVTE ended at 1000 MPa, while LVTE for PIB 2 was determined at 1650 MPa.

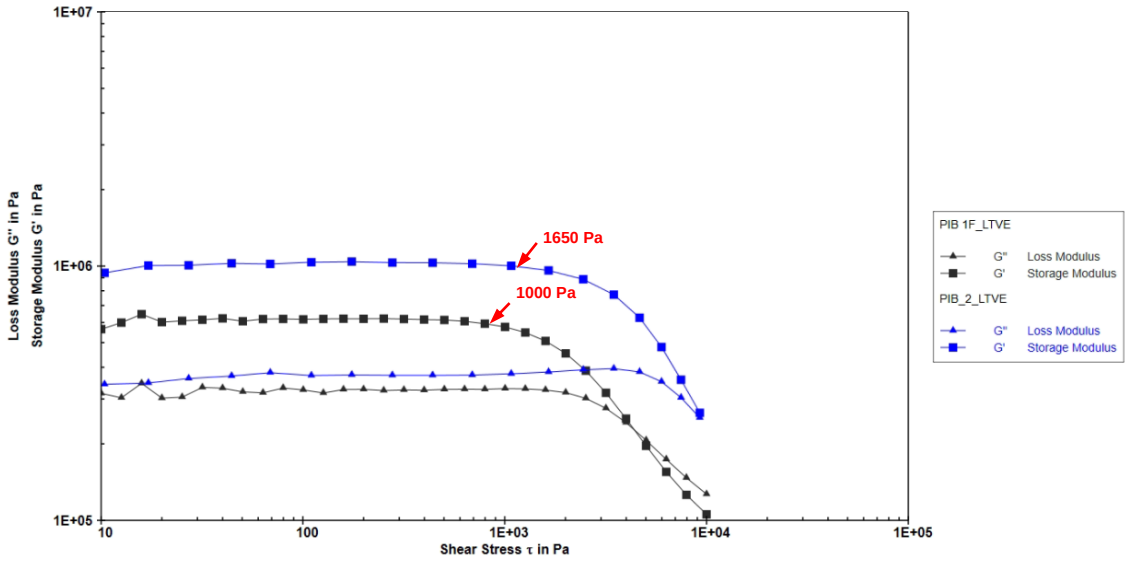

***Figure 3**: Determination of LVTE - storage G' and loss modulus G'' as a function of shear stress at 60°C.*

2.3. Rheology: frequency sweep @ 60°C

Instruments: Modular rotational rheometer MCR302, Anton Paar

Procedure details:

Temperature: 60°C

Shear stress: 1000 Pa (within LTVE)

Frequency: 0,01 - 100 Hz

System configuration: upper plate PP25/S (sandblasted); lower plate INSET/pp25/SS/S D:25 mm, SANDB; HETD 400

Gap: 1 mm

Repetitions: 2 repetitions per material

Figure 4 shows viscoelastic behavior (storage G' and loss G'' modulus) of PIB 1 and 2 over a wide frequency range at constant temperature of 60°C (isothermal segment).

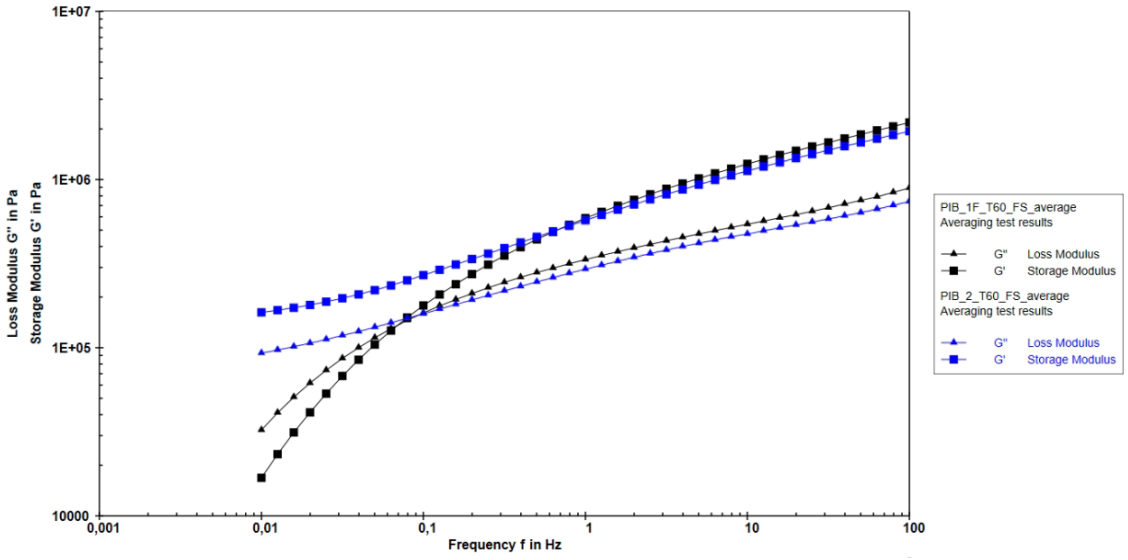

***Figure 4**: Storage G' and loss G'' modulus as a function of frequency at constant temperature of 60°C (average values are presented).*

3. CONCLUSION

Differential dynamic calorimetry was used to determine the glass transition temperature which was for PIB 1 determined at -65 °C and -66.8 °C for PIB 2. At 60 °C, the storage modulus of PIB 1 dominated over the loss modulus up to the frequency of 0.1 Hz, where the flow point could be observed. At low frequencies, the storage modulus of PIB 1 decreased rapidly, indicating that the material at these conditions behaves as a liquid. On the other hand, the results for PIB 2 show that the storage modulus dominated the viscous one in the whole frequency range. For this sample, the flow point was not observed in the frequency range examined.