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 up to 180 °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: **Thermal and rheological characterization of PIB 1 and PIB 2 up to 180 °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 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.

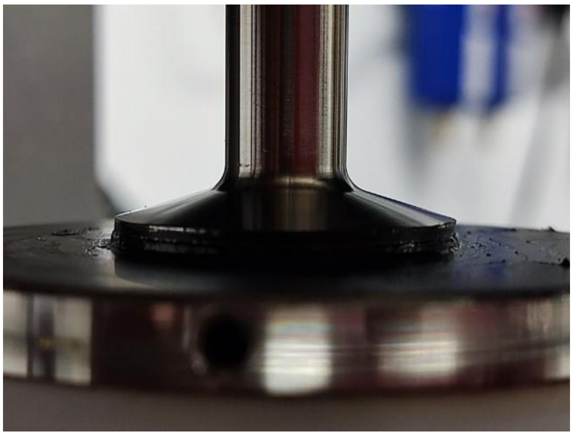

Figure 1: Trimmed cylindrical sample at 60°C.

2. MEASUREMENT

2.1. DSC: phase transition temperatures

Instruments: DSC2500, Ta Instruments

Procedure details:

Samples: PIB 1 and PIB 2

Temperature range: -80°C – 200°C

Heating and cooling rate: 10°C/min

Pan type: Al

Sample geometry: finger kneaded granule

Repetitions: 2 repetitions per material

Figure 1 shows DSC thermograms of PIB 1 and PIB2 indicating glass transitions at ~ -65°C

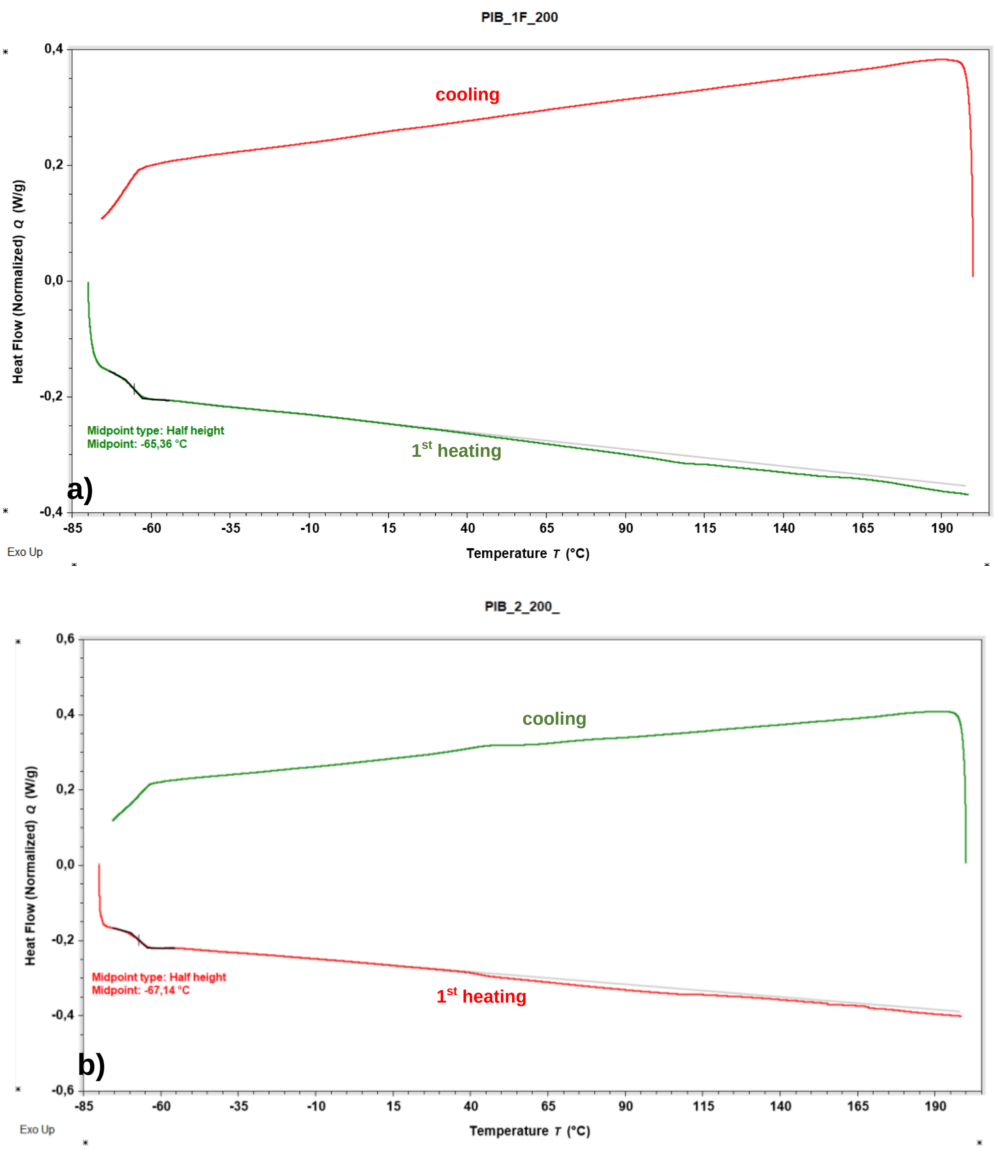

***Figure 2**:* DSC thermograms of a) PIB 1 and b) PIB 2 (only 1. repetition is shown).

2.2. Rheology: determination of LTVE range @ 180°C

Instruments: Modular rotational rheometer MCR302, Anton Paar

Procedure details:

Temperature: 180°C

Shear stress: 10 - 3000 Pa

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 the results of amplitude sweep tests, which were used to determine shear stress limit of linear viscoelastic range LTVE for PIB 1 and PIB 2 at constant temperature of 180°C.

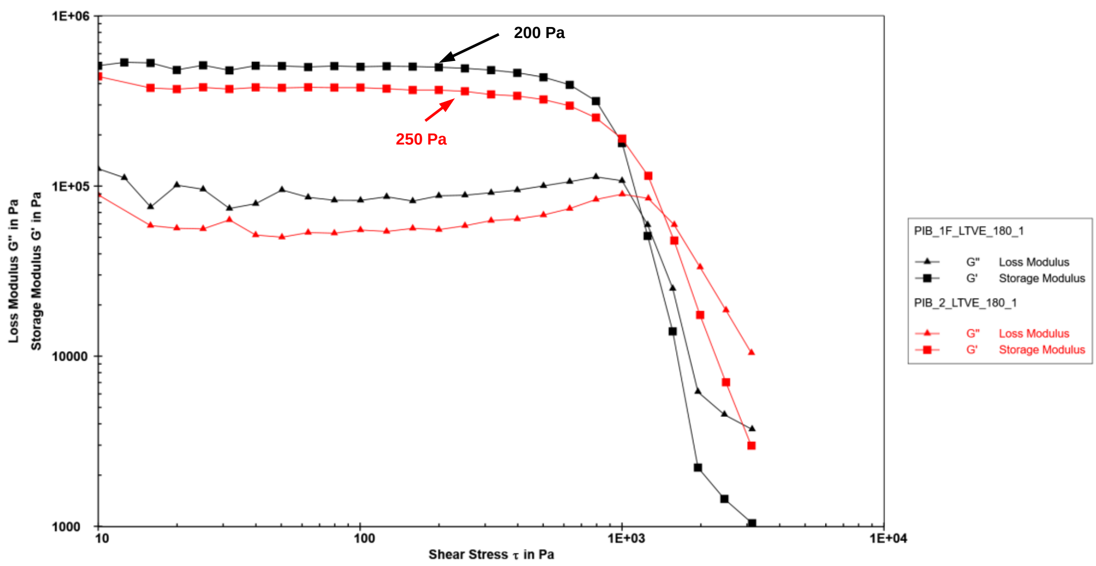

Figure 3: Storage G' and loss G'' modulus as a function of shear stress for PIB 1 at 180°C.

2.3. Rheology: temperature sweep between 15 – 180 °C

Instruments: Modular rotational rheometer MCR302, Anton Paar

Procedure details:

Samples: PIB 1 and PIB 2

Temperature: 15-180°C

Shear stress: 200 Pa (for PIB 1F) and 250 Pa (for PIB 2)

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 4 shows the results of temperature sweep tests for PIB 1 over a wide temperature range during three cycles of heating.

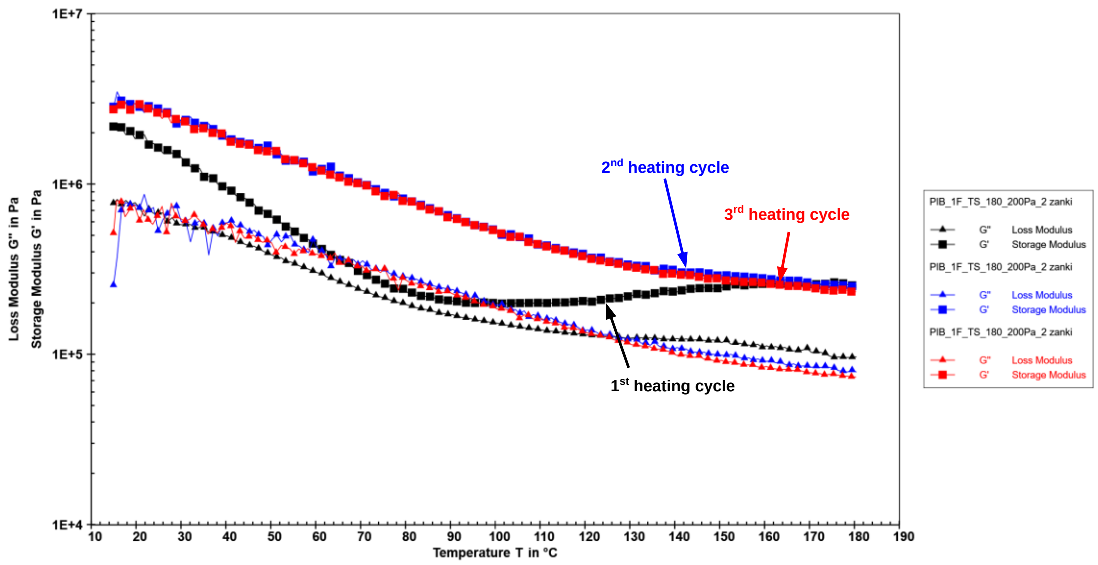

***Figure 4**:* Storage G' and loss G'' modulus as a function of temperature during three heating cycles.

Figure 5 shows the results of temperature sweep tests for PIB 2 over a wide temperature range during three cycles of heating.

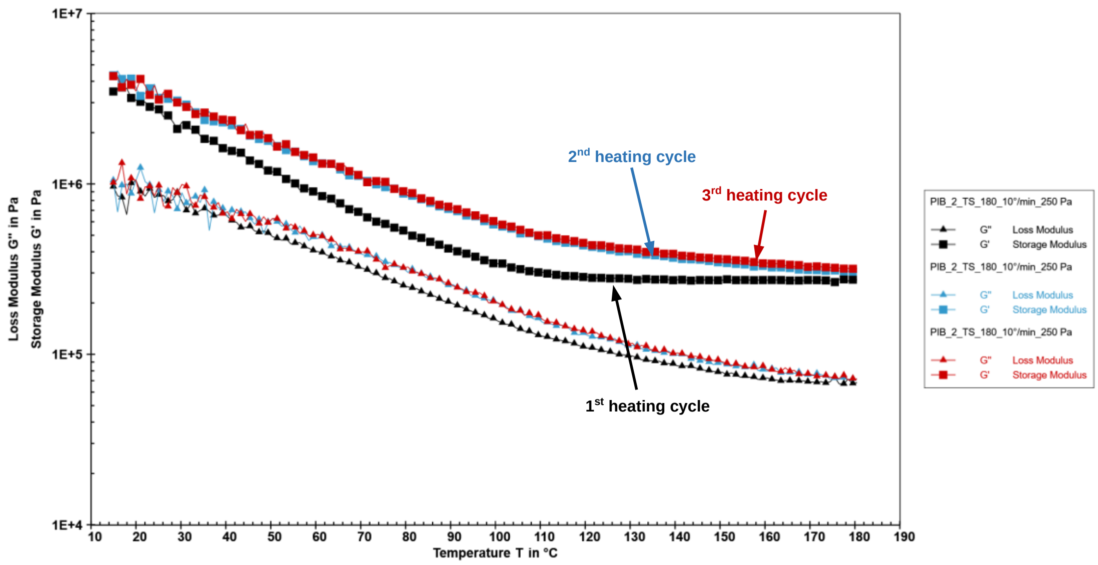

Figure 5*:* Storage G' and loss G'' modulus as a function of temperature during three heating cycles.

Results show that at temperatures above ~60°C smaller molecules (or components) start to evaporate or degrade, which changes the rheological behavior of PIB. However, after the first heating cycle the material becomes thermo-rheologically stable.

2.4. Rheology: Comparison with previous measurements

Instruments: Modular rotational rheometer MCR302, Anton Paar

Procedure details:

Samples: PIB 1 and PIB 2

Temperature: 10-180°C

Shear stress: 200 Pa (for PIB 1F) and 250 Pa (for PIB 2)

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 6 shows the comparison of temperature sweep tests of current measurements (from 15°C to 180°C) with previous measurements, which were performed to lower temperatures, i.e. from -20°C to 60°C) for PIB 1 and PIB 2.

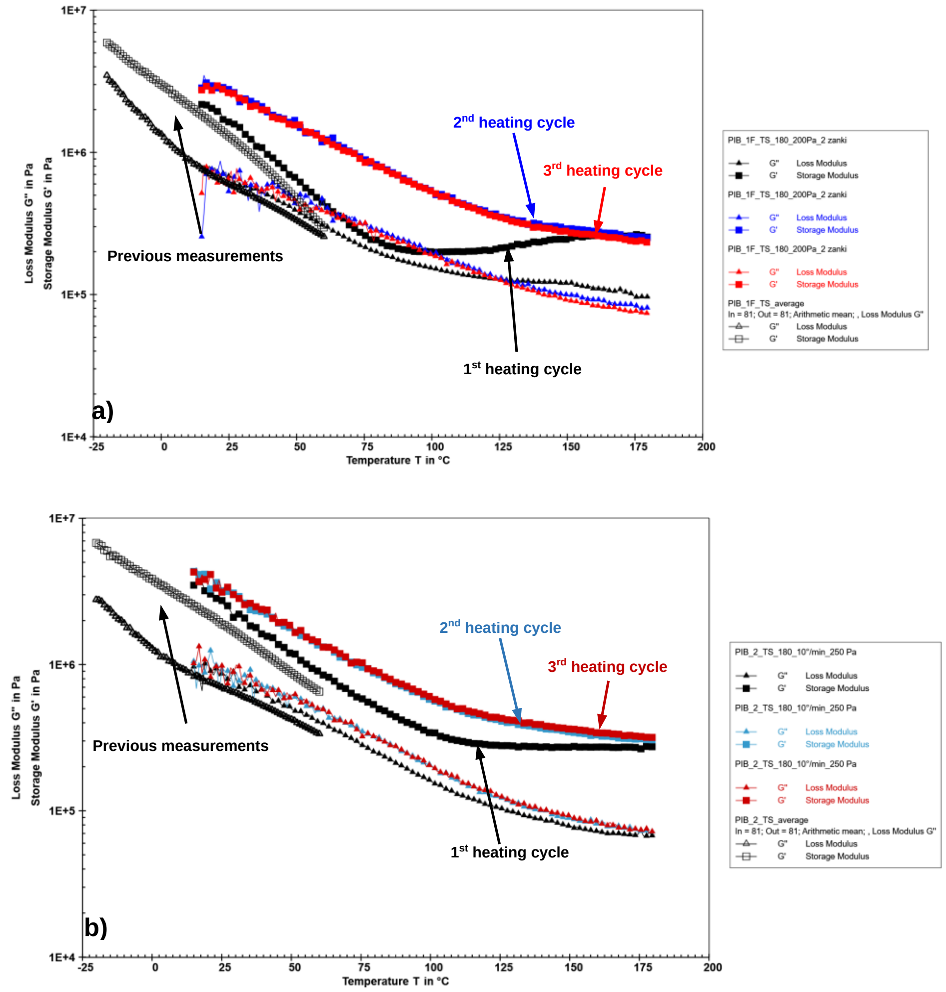

***Figure 6**: Storage G' and loss G'' modulus as a function of temperature for a) PIB 1 and b) PIB 2.*

2.5. Rheology: Stability at 60 °C

Instruments: Modular rotational rheometer MCR302, Anton Paar

Procedure details:

Samples: PIB 1 and PIB 2

Temperature: 60°C

Shear stress: 400 Pa

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 7 shows rheological stability tests for PIB 1 and PIB 2, which were performed at constant temperature of 60 °C for 8 hours. From the results it is clearly visible that viscoelastic properties (storage G' and loss G'' modulus) change over time and reach the plateau of constant values after ~400 min.

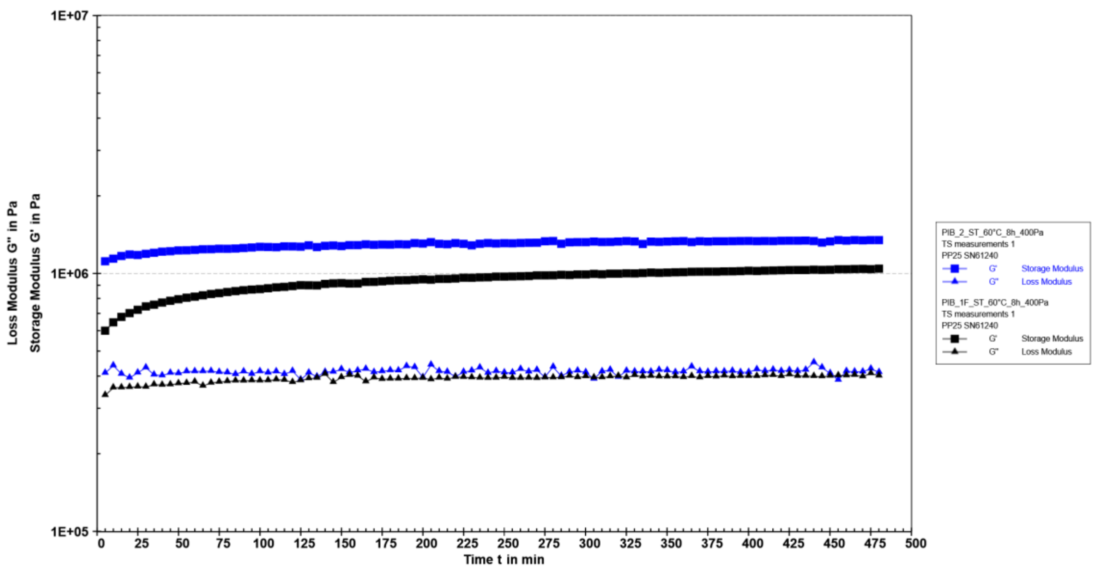

***Figure 7**:* Storage G' and loss G'' modulus as a function of time at constant temperature of 60°C for PIB 1 and PIB 2, respectively.

3. CONCLUSION

After first heating cycle the PIB materials become thermo-rheologically stable. The results show that viscoelastic properties (storage G' and loss G'' modulus) at constant temperature of 60 °C change with time. At temperatures above ~60°C, smaller molecules (or components) start to evaporate or degrade, which changes the rheological behavior of PIB materials.