A novel testing equipment for measuring the glass transition temperature based on a dynamic mechanical analysis

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Glass transition temperature

The glass transition temperature (T_g) is related to the molecular motion of polymeric chains. Above Tg, the polymer chains move freely which makes the polymer very flexible. Below T_g, the chains have much less mobility and the polymer is effectively ‘‘frozen’’ (Figure 1).

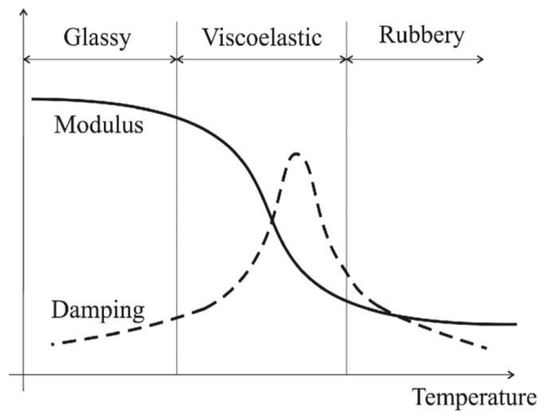

Figure 1 – Variation of polymer properties near the T_g

The glass transition temperature can be affected by many factors such as the degree of the cure and the moisture uptake. While some common methods can be used to measure T_g, they are lengthy, and the corresponding equipment is expensive. A slow test might alter the polymer characteristics by evaporating moisture or by further curing the polymer. From an industrial perspective, it is therefore desirable to develop a method to rapidly measure T_g to minimize the change of the original state of the specimen.

Measurement concept

Using a beam with a magnet at each end, the specimen is supported on cotton threads (Figure 2). The positions of the nodes were found by sprinkling fine dry sand on the surface of the beam. The sand migrated to the nodes when the beam was excited at resonance. The positions of the supports were then readjusted, if necessary, and the frequency for maximum amplitude was found again. This was taken to be the resonant frequency.

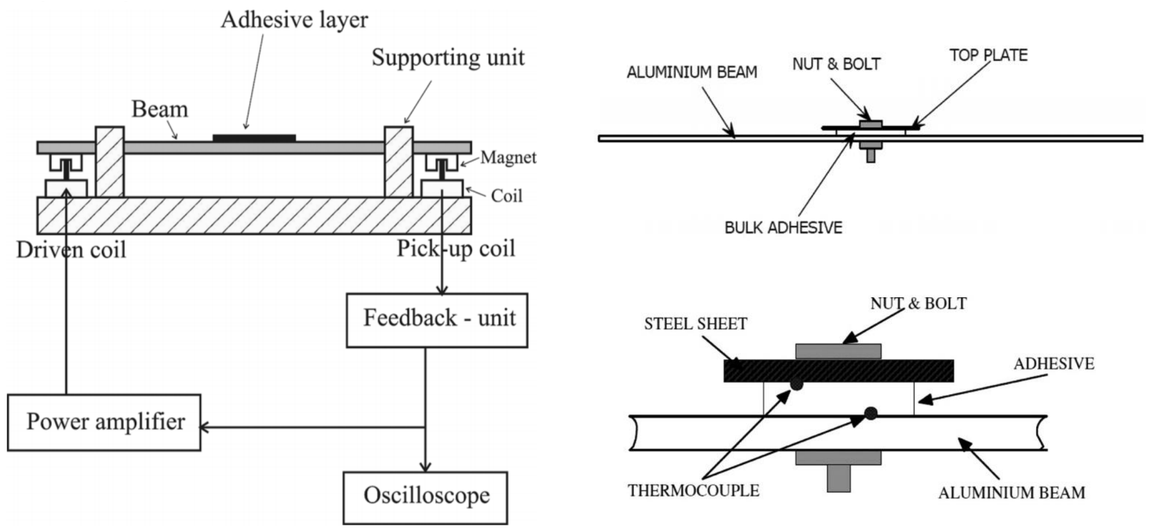

Figure 2 – Testing setup (right) and specimen configuration (left)

A pre-cured sheet of the polymer is attached to the beam and constraining layer via a bolt (Figure 2). This latter is highly useful for investigating the change of T_g with moisture uptake as specimens could be readily removed from the beam and placed in the environmental chamber.

The main problem with the ‘‘quick’’ test used in this investigation was to obtain a rapid but uniform (in the polymeric specimen) temperature rise. This was overcome by using an additional specimen, placed next to the vibrating specimen, which was used to monitor the temperature.

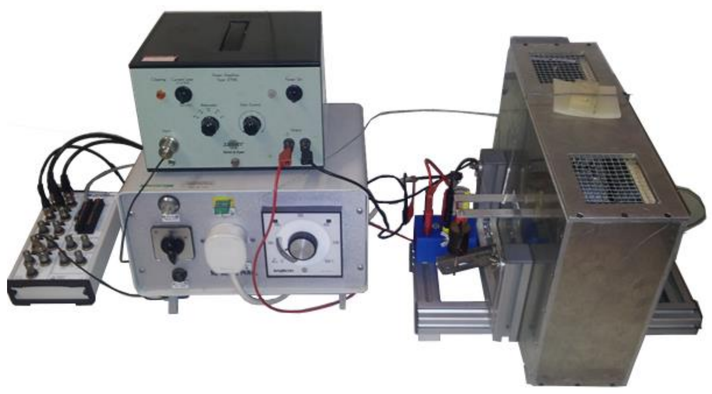

Figure 3 – Testing setup including oven (on the right)

The specimen is placed inside an oven to determine the damping at that specific temperature (Figure 3). The advantage of using an oven was that the temperature could be maintained at a specific level while measuring the damping

Control and software design

The testing concept relies on a feedback process that allows resonant oscillation to be maintained regardless of variations in the resonant frequency. In an initial version, an analogical unit achieved this this by first considerably amplifying the sinusoidal wave received from the pick-up coil. Then, this is ‘‘chopped’’ to yield what is almost a square wave. A phase shift circuit then alters the phase of the signal to correspond with that needed to input the power amplifier to maintain resonance (i.e., it allows for the various phase changes in the electronics between the coil and the input to the power amplifier). This process is shown in Figure 4.

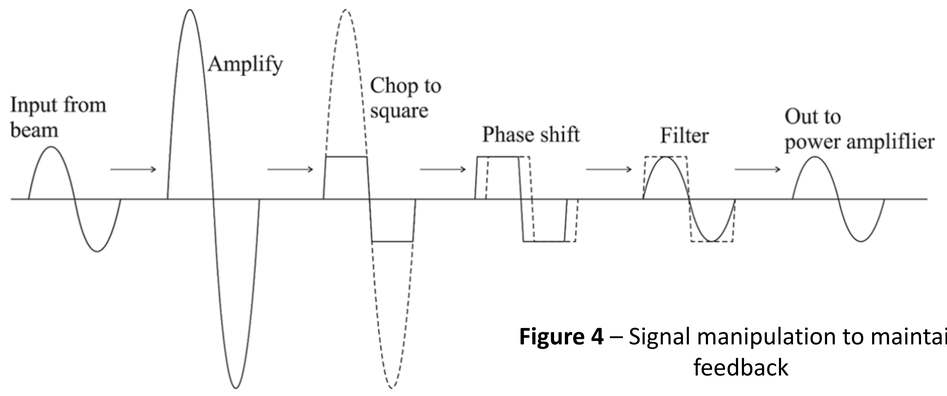

Figure 4 – Signal manipulation to maintain feedback

Finally, a filter with its central frequency approximately at the resonant frequency of the specimen is used to block frequencies that are outside a select bandwidth bracketing the first resonant frequency of the specimen. This transforms the square wave into a sinusoidal wave which is amplified and sent via a power amplifier to the driving coil.

Changes were implemented that led to the elimination of hardware, such as the signal generator, the oscilloscope, the ammeter and the feedback box, and quality of the excitation and feedback signal of the previous system. were replaced by a data acquisition card. This transformation led to a reduction in the amount of equipment used, making the system more compact, simple and easy to use. The use of a data acquisition card facilitated the creation of a graphical interface (Figure 5), which eliminated the need for manual data processing.

The testing equipment was successfully validated for various polymers, being in agreement with data obtained from other sources (Figure 6).

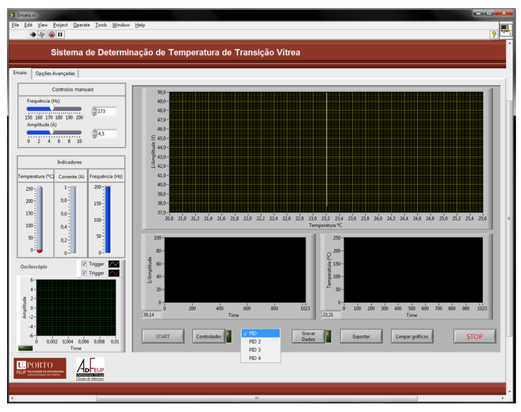

Figure 5 – Custom designed software used for process control and data acquisition

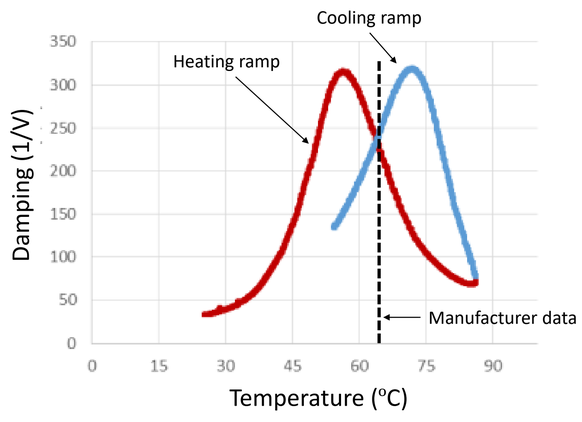

Figure 6 – Example of recorded curves, compared against manufacturer T_g data.

Conclusions

A novel glass transition testing apparatus as well as the accompanying software was designed, which has the dual function of acquiring the test data and controlling the resonance circuit that applies an alternating current to the electromagnetic coils. Then process was validated, demonstrating the accuracy of the novel equipment when compared to commercial glass transition testing equipment.

Acknowledgements

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