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Physical modelling and identification of polymer viscoelastic behaviour above glass transition temperature and application to the numerical simulation of the hot embossing process

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Abstract. The experimental processing parameters, such as applied pressure and forming temperature have been analysed during polymer hot embossing of micro-cavities. The viscoelastic characteristics of polymer above the glass transition temperature have been investigated with the classical viscoelastic models. Generalized Maxwell Model has been used to describe polymer behaviours in the glass transition temperature range. The parameters include relaxation time, storage modulus and loss modulus of the Generalized Maxwell Model that have been introduced. The identification of polymer characteristics has been carried out through Dynamic Mechanical Analysis (DMA). The storage modulus, the loss modulus and the damping factor of the selected polymer have been obtained with different imposed frequencies. The master curve of complex modulus has been obtained by applying the time temperature superposition principle. The experimental data has been identified with optimized fitting parameters of Generalized Maxwell Model. A proper agreement between the experimental measurement and the identification of viscoelastic model is observed. The resulting constitutive equations have been implemented in finite element software in order to achieve the numerical simulation of the hot embossing process.

Introduction

Hot embossing process is growing fast during recently years, especially for polymers based micro- and nano- component fabrication in a variety of industries [1]. The mechanical behaviours of polymer during hot embossing process depend strongly on the compression temperature that is always lightly above the polymers' glass transition temperature. The important change in the physical properties is observed during the glass transition temperature range. The polymer state changes from solid to relatively liquid displayed by a smooth increase in the viscosity of polymer. The polymer exhibits an elastic behaviour and a viscous behaviour in the same time. The viscoelasticity of polymer needs to be investigated in order to achieve the modelling of polymer's behaviour during hot embossing process.

Song et al. [2] have proposed a biphasic model to analyse the linear dynamic viscoelasticity of polymer melts filled with nano-sized fillers. The storage modulus and the loss modulus of nanofilled polymers have been analyzed under the consideration of different nanocomposites. The effect of the structural difference of the filler phase has been also discussed on basis of the proposed model. The biphasic model has been applied to rubbers filled with nanoclay and conventional fillers to study the mechanisms of dynamic rheology behaviours beyond the terminal flow region [3].

The linear thermo-viscoelasticity of polypropylene has been studied by Drozdov [4]. A constitutive model for the linear viscoelastic response of semi-crystalline polymers has been developed. The tensile relaxation tests in a temperature range from -20°C to 120°C have been carried out in order to identify the material constants of the model.

The linear viscoelasticity of polymer blends with co-continuous morphology has been set up by Yu et al. [5]. A quantitative connection between the structural information of co-continuous morphology and the corresponding linear viscoelasticity has been established through comparison with the experimental data.

Although the viscoelasticity properties of polymers have been deeply investigated during the recent years, the application of the viscoelastic material models for polymers in the hot embossing process has not been deeply investigated. Hot embossing process of polymers in the micro and nano scales has been widely developed in the experimental domains, but the lack of physical modelling and numerical simulation for hot embossing process makes it difficult to extend diffusion in micro-manufacturing industry [6].The paper is aimed to propose a viscoelastic model (Generalized Maxwell model) to describe thermo-mechanical behaviours of polymers (PMMA) during hot embossing process. Dynamic Mechanical Analysis (DMA) is carried on to identify the viscoelasticity properties of the polymers used in the process. The storage modulus, the loss modulus and loss factor are illustrated as a function of imposed frequency and temperature. The viscoelastic model is identified by fitting parameters and compared with the experimental data. A proper agreement between the physical modelling and experimental investigation has been observed

Description of hot embossing process

Hot embossing has been considered as a much more prominent polymer micro-fabrication technology compared with injection moulding [7] over the last years. The hot embossing process is based on the replication of a master structure into the polymer material [8]. A polymer substrate is heated up to the mould temperature, which is lightly above Tg of polymer. The mould moves down and then upsets the polymer substrate with an imposed pressure or an imposed displacement at the mould temperature. The polymer substrate is cooled down to the demoulding temperature range while maintaining the pressure. Finally the mould is opened to obtain the polymer component with micro structured cavities.

The compression temperature and pressure during hot embossing cycle will determine the replication accuracy of microstructures [9]. The hot embossing processing parameters have been optimized for the manufacturing of COP-based micro-fluidic chips [10]. The processing conditions depend on the material properties of the substrate, the shape and dimension of the substrate and the depth and width of the micro-cavities. The variation of the temperature and pressure during the hot embossing process is related in Fig. 1 [11].

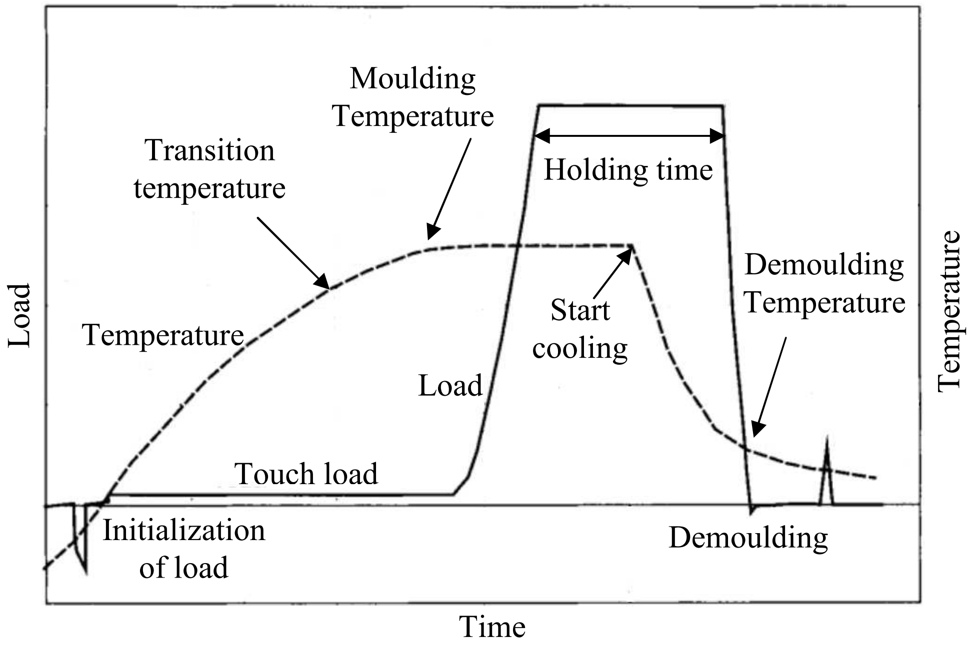

Figure. 1 Typical process parameters and related moulding load and temperature during hot embossing cycle

Viscoelastic models for polymers in hot embossing

The physical behaviours of polymers depend not only on temperature, especially for the polymers above their glass transition temperature, but also on processing time, which could be observed in the creep or stress-relaxation tests. Time-temperature superposition principle [12] implies that the response of polymers at high temperature with short time is equivalent to the response of polymers at low temperature with longer time. The Williams–Landel–Ferry equation (or WLF Equation) [13] is widely used to identify the time-temperature equivalence of polymers in the temperature range from glass transition temperature (Tg) to Tg + 100°C. The viscoelastic models are capable to describe the time dependent behaviour of polymer in hot embossing processing temperature range. The viscoelatistic models for polymers have been largely studied in the literature:

A physically based, three-dimensional constitutive model has been developed for the characteristic features of amorphous polymer behaviours near Tg. [14]. The model accounts linear elasticity, as Eyring viscous flow, the Edwards-Vilgis entropy function, Vogel-Tammann-Fulcher and Arrhenius equations to express glassy-rubber response of polymers. The essential features includes bond stretching stresses act in parallel with conformation entropic stresses, bond stretching stresses relaxation by local self-diffusion of molecular segments and conformational entropy of the entangled molecules.

Five viscoelastic models composed of elastic and viscous components have been accounted for modelling of nonlinear tensile behaviour of PMMA foams [15]. The developed constitutive equations have been expressed in terms of strain, strain rate, elastic modulus, relaxation of time constant and relative density of foam, but without considering the temperature effect. The constitutive models have been verified by the uniaxial tensile tests data with PMMA microcellular foams manufactured using batch process method.

In this paper, a linear viscoelastic Generalized Maxwell Model has been employed to describe the polymer's behaviour during hot embossing process. Generalized Maxwell Model consists of a linear Hookean spring and a number of Maxwell models connected in parallel, as is showed in Fig. 2.

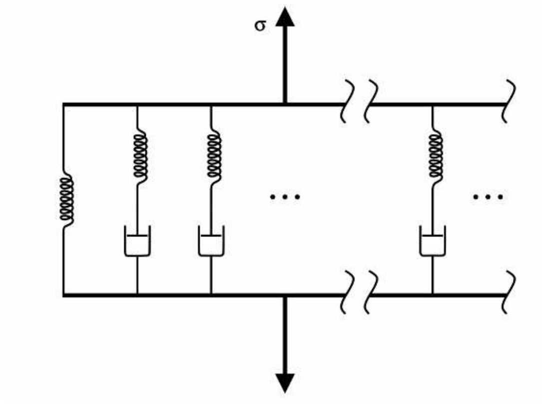

Figure. 2 Schematic of Generalized Maxwell Model

The behaviour laws described by the spring and the dashpot are expressed as:

$$\sigma = E\varepsilon \text{ (Hooke's law)} \quad (1)$$

$$\sigma = \eta \frac{d\varepsilon}{dt} \text{ (Newtonian law)} \quad (2)$$

where, respectively E is elastic modulus and η is viscosity.

The spring stands for the elastic response and the dashpot represents the viscous response (or time-dependent response). The elasticity in the model reflects its ability to conserve energy after deformation and the viscosity reflects it ability to dissipate energy.

The relaxation time constant is defined as:

$$\tau = \frac{\eta}{E} \tag{3}$$

The relaxation time constant couples both most important parameters of the viscoelastic models: elastic modulus and viscosity. It indicates that the viscoelasticity is the result of viscosity and elasticity existed together in matter. It is dependent on their relative values instead of their absolute values. It is an internal time scale characterized relaxation phenomena in polymer [16].

The Generalized Maxwell takes into account that the fact that the relaxation does not occur at a single time, but distributed at different specific times. The relaxation modulus of Generalized Maxwell model could be represented as follows:

$$E_{(t)} = E_{\infty} + \sum_{m=1}^{N} E_m \exp\left(-\frac{t}{\tau_m}\right) \tag{4}$$

where, N represents the number of branches in the model, E_m represents the relaxation modulus and τ_m represents the relaxation time in the m-branch.

$$E_{\infty} = \lim_{t\to\infty} E_{(t)} \tag{5}$$

corresponds to the relaxation modulus when t becomes infinite.

Dynamic mechanical analysis (DMA) tests

Polymer viscoelastic behaviours depend strongly on temperature and time. A number of experimental methods for measuring viscoelastic properties of solids have been extensively studied, such as time domain measurement (creep and stress relaxation tests); in frequency domain (resonant and sub-resonant tests, piezoelectric ultrasonic composite oscillator technique, Resonant ultrasound spectroscopy, wave methods) [17]. Most individual methods permit the analysis of the viscoelasticity of materials in a narrow range of frequency or temperature. The time-temperature superposition is often used to extend the measurement of viscoelastic properties over a wider range, especially in the study of polymers. This principle indicates that a shift factor could be defined to extend the range of effective time or frequency of measure properties by exploiting the temperature dependence of properties.

Dynamic mechanical analysis (DMA) has been used to study the viscoelastic behaviour of polymers. Dynamic mechanical analysis is a powerful tool for developing insight into viscoelastic behaviour of polymer while simultaneously enabling a high level of sensitivity in the detection of changes in mobility of the polymer chain segments [18]. The DMA experimental tests are always used to identify the transition of the polymer state from solid to liquid. The DMA technique applies an oscillating force or displacement to a sample and the resultant stress and strain measured as functions of both oscillatory frequency and temperature [19].

Viscoelastic response in DMA test

A sinusoidal displacement is applied to the sample, which results a sinusoidal strain in the form of:

$$\varepsilon = \varepsilon_0 \sin \omega t \tag{6}$$

where ε_0 is the amplitude of the oscillating strain, ω stands for the angular oscillation frequency expressed through the following equation:

$$\omega = 2\pi f \tag{7}$$

where f is the oscillating frequency.

The stress response is also sinusoidal with the same frequency, which is presented by a phase lag caused by the hysteresis property in the viscoelastic system:

$$\sigma = \sigma_0 \sin(\omega t + \delta) \tag{8}$$

where δ is the phase angle between the stress and strain.

The stress response could be expressed in the following form by introducing the storage modulus E' and the loss modulus E'' :

$$\sigma = \varepsilon_0 E' \sin \omega t + \varepsilon_0 E'' \cos \omega t \tag{9}$$

where

$$E' = \frac{\sigma_0}{\varepsilon_0} \cos \delta, E'' = \frac{\sigma_0}{\varepsilon_0} \sin \delta \tag{10}$$

The ratio of the loss modulus and the storage modulus is called the loss tangent or damping factor:

$$\tan \delta = \frac{E''}{E'} \tag{11}$$

The storage modulus represents the real or elastic component of viscoelastic behaviour, which expresses the ability to maintain the mechanical energy of the material. The loss modulus represents the imaginary part or viscous component of viscoelastic behaviour, which expresses the energy dissipation ability of the material. The storage and loss modulus are associated through the friction between the molecular chains of the material. The phase angle corresponds the phase shift between the strain and the stress. The loss tangent or damping factor indicates the proportion of the storage modulus and the loss modulus, which is a common parameter to characterise the polymer glass transition zone. The significant change of the polymer state could be observed in the glass transition zone. The loss tangent or damping factor reflects the energy dissipated by friction in the molecular chains, so it is sometimes called internal friction [20].

Description of DMA test device

Dynamic Mechanical Analysis test device is composed mainly by five parts presented in Fig. 3. Mechanical excitation equipment is used to apply an oscillating displacement to the sample, which is placed in an insulated chamber. The displacement, acceleration and force sensors are installed to identify the mechanical parameters during the excitation, as it is presented in Fig. 4. Control and measuring equipment are used to initiate the excitation with different frequencies, control the amplitude of the excitation displacement and measure the stiffness as well as phase angle of the sample. The lab-VIEW is equipped for data acquisition and instrument control, along with a computer for the post treatment of experimental data. The temperature regulator controls the thermal condition in the chamber. The power amplifier consists to increase the power of the signals by an external energy. In this test, a sinusoidal displacement is applied to a cylindrical sample and the amplitude of the oscillating displacement is invariable for every imposed frequency. Seven imposed frequencies have been selected: 7.8 Hz; 15.6 Hz; 31.2 Hz; 62.5 Hz; 125 Hz; 250 Hz; 500 Hz. The responses of the oscillation, such as the oscillating force, the phase angle between stress and strain have been captured for each imposed frequency. The test temperature varies from the room temperature to 40 °C above the glass transition temperature of selected polymer.

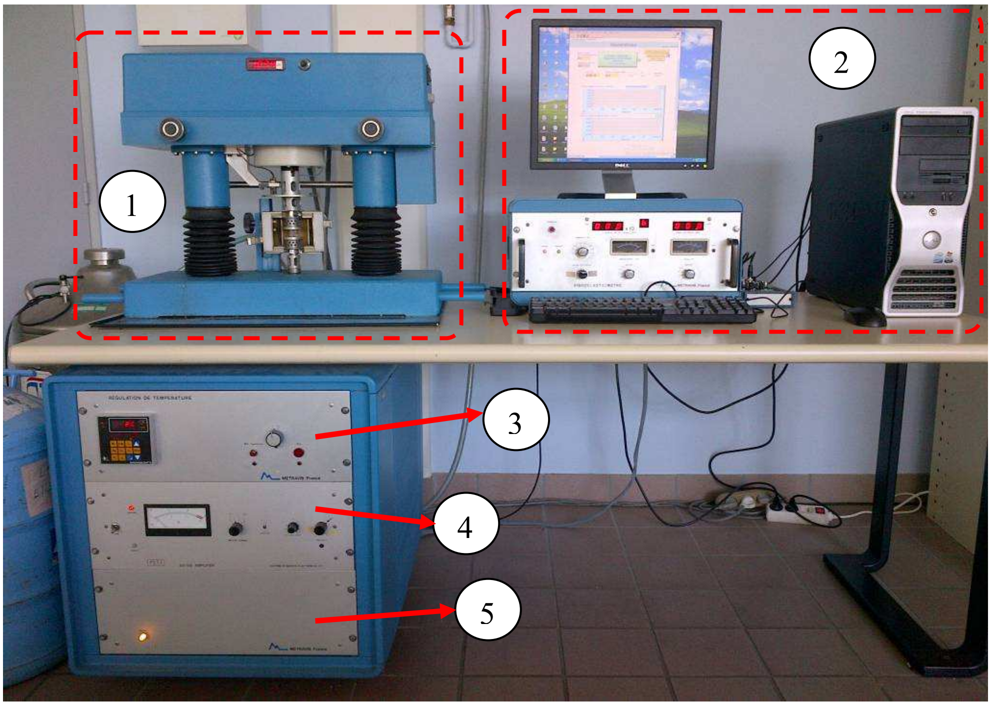

Figure. 3 Description of Dynamic Mechanical Analysis (DMA) test device: ①Mechanical excitation equipment ② Control and measuring equipment ③ Temperature regulator ④ Power amplifier system ⑤ Power supply system

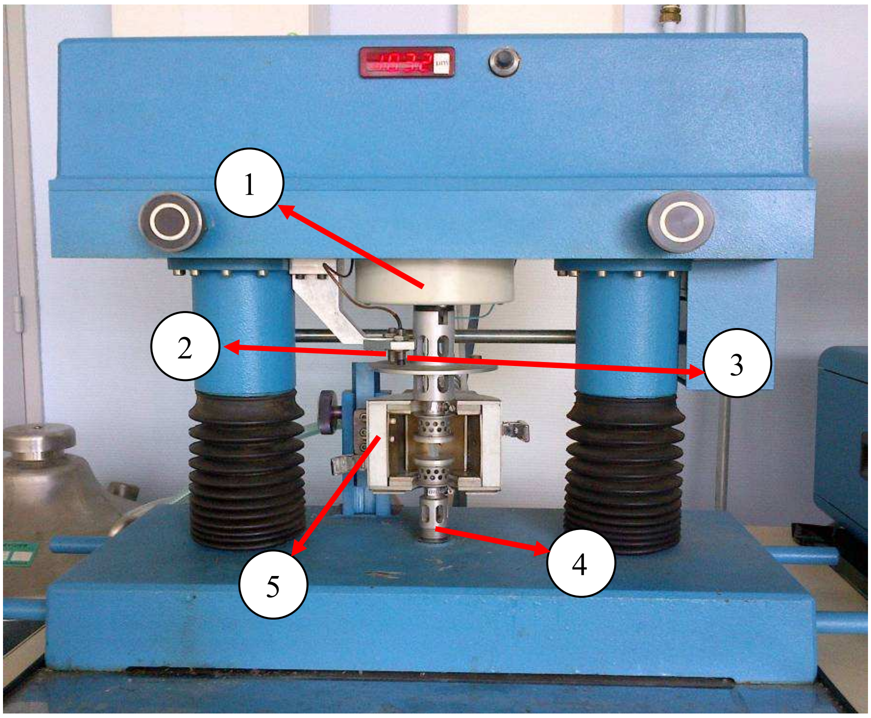

Figure. 4 Description of the mechanical excitation equipment of the DMA test device: ① Electrodynamics exciter ② Displacement sensor ③ Acceleration sensor ④ Force sensor ⑤ Insulated chamber

The sample is in the form of cylinder with a diameter equal 18mm and a length equal 10mm, showed in Fig. 5. There are two plates used for applying the compression to the sample. The sample is pasted in the middle of these two plates in order to keep the connection between the plates and the sample. Because the sample becomes softer when the temperature reaches the glass transition temperature of the polymer, and the two surfaces of the sample are possible to be disconnected with the plates. The sample used in the test is made of Poly (methyl methacrylate) (PMMA).

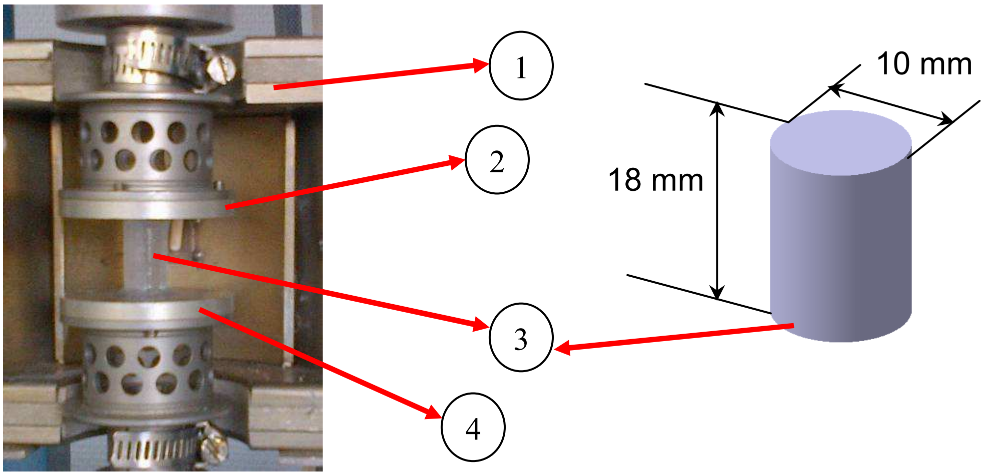

Figure. 5 The set up of the sample in DMA test device and the dimension of the sample: ① Insulated chamber ② Top plate ③ Polymer sample ④ Bottom plate

Results of DMA tests

The PMMA sample that is used in the DMA tests in the temperature range from room temperature 20°C to 150°C. The seven oscillating frequencies are applied to the sample for each testing temperature with the interval of 5°C. The amplitude of the oscillating displacement is fixed at 2.5μm and remains constant for every oscillation frequency. The storage modulus, loss modulus and damping factor vs. temperature with different imposed frequency are related in Fig. 6-8.

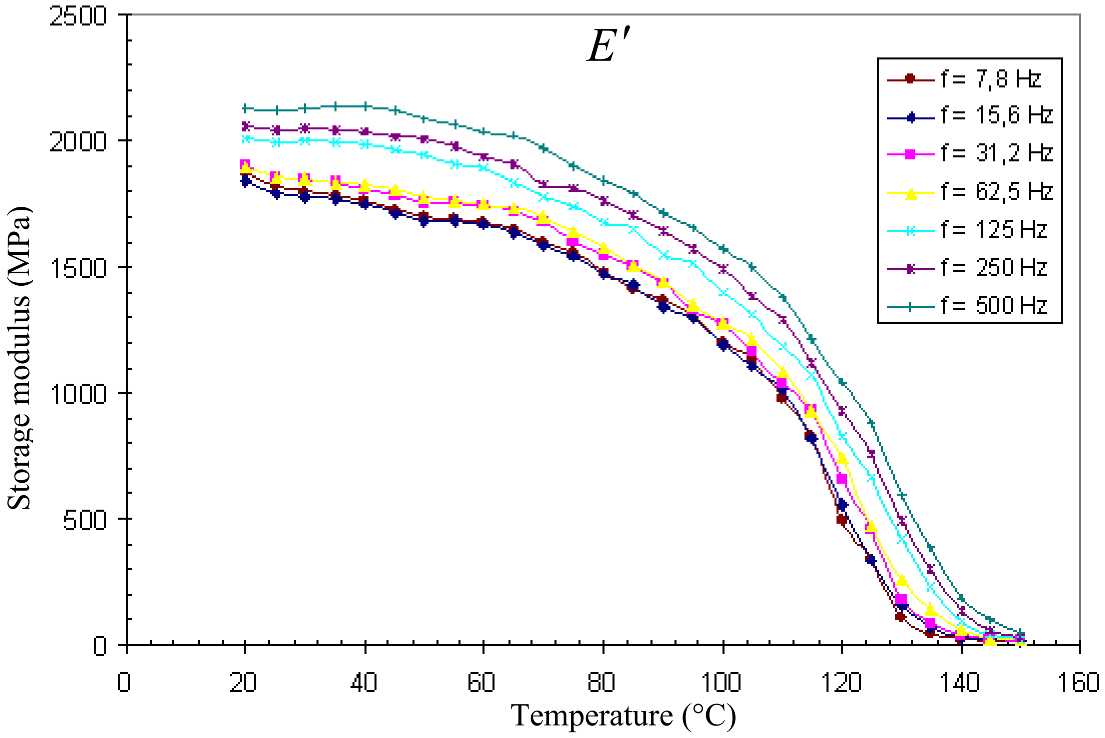

Figure. 6 Storage modulus vs. temperature with different imposed frequencies for PMMA sample in DMA test

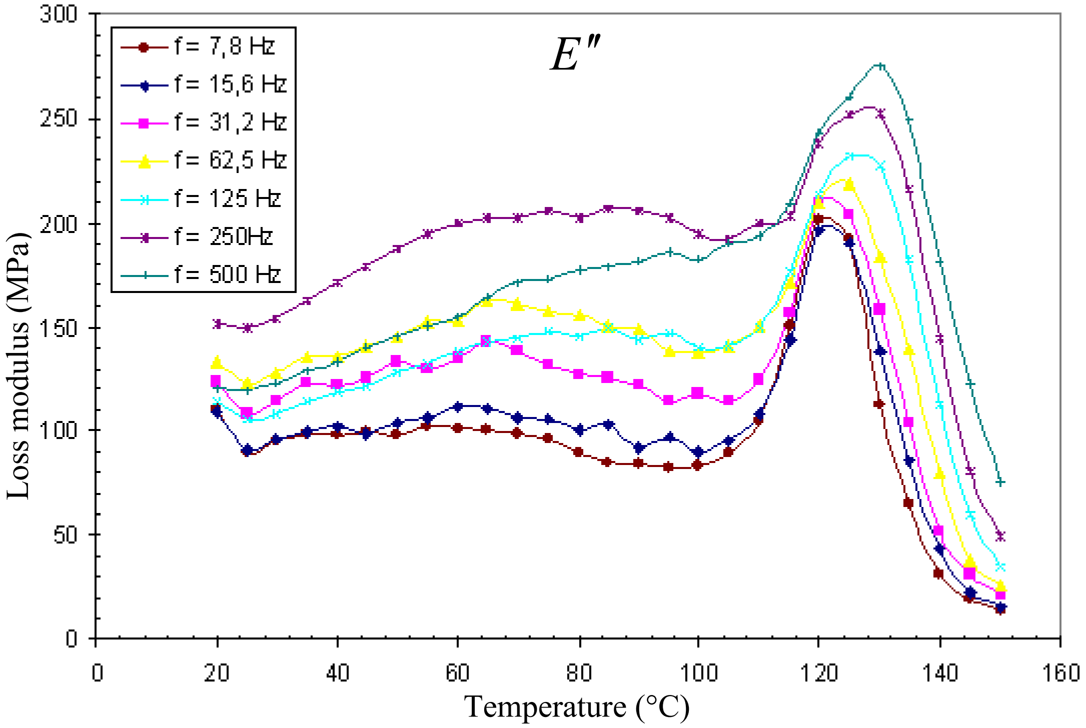

Figure. 7 Loss modulus vs. temperature with different imposed frequencies for PMMA sample in DMA test

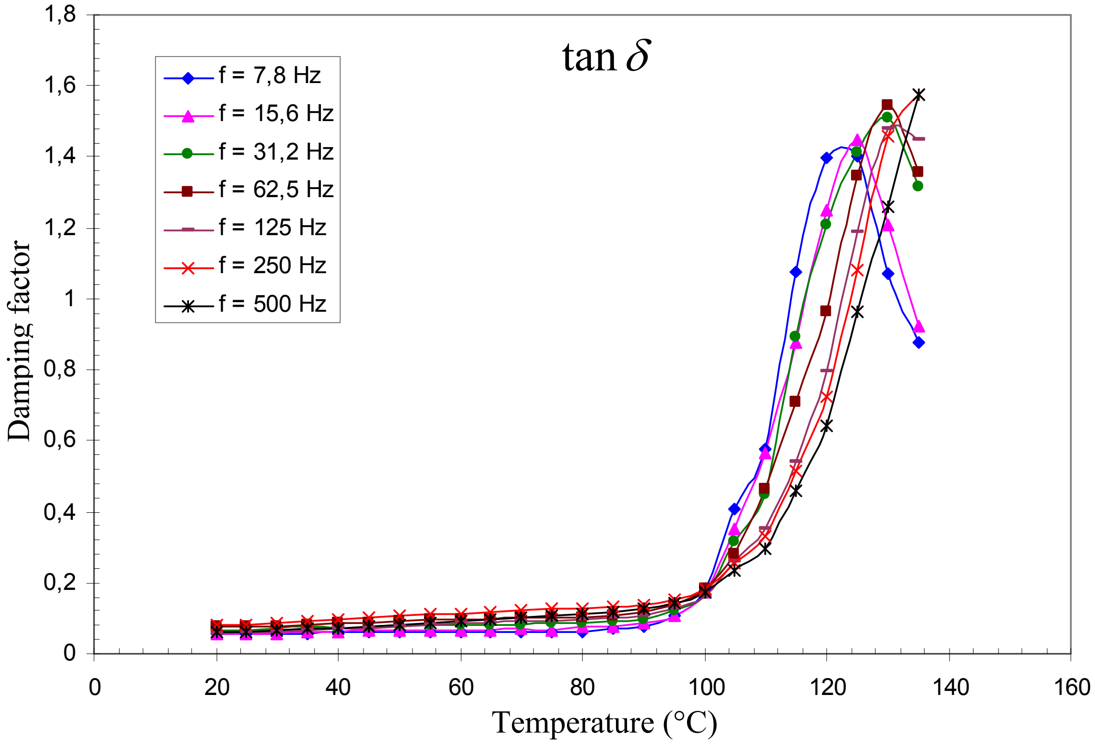

Figure. 8 Damping factor vs. temperature with different imposed frequencies for PMMA sample in DMA test

The storage modulus decreases as the temperature rises, and decreases more significantly when the temperature near and above the glass transition temperature of the polymer under consideration about 110°C for PMMA. All the curves of storage modulus with different imposed frequencies have almost the same form, but the value of the storage modulus at the same temperature increases slightly with the rise of the imposed frequency. These curves imply that the state of the polymer is transformed from solid state to nearly liquid state, indicating that the transition from the elastic behaviour to viscous behaviour during the temperature range.

The loss modulus does not have significant change at low temperature, but a sudden augment occurs around the glass transition temperature. It presents the converse tendency compared with the storage modulus during this temperature range. The value of the loss modulus increases when the imposed frequency augments in the glass transition temperature range. The augment of the loss modulus implies that the polymer becomes more and more viscous, and the viscous behaviour becomes dominant above the glass transition temperature.

All the curves of the damping factor present a peak above the glass transition temperature. The damping factor begins to increase almost at the same temperature for all imposed frequencies. The damping factor is an important parameter to determine the glass transition temperature of the polymer. Because it is much more sensitive vs. temperature compared with the other dynamic parameters: the storage modulus and the loss modulus.

It shows in Fig. 9 that the storage modulus, loss modulus and damping factor vs. temperature with the imposed frequency of 7.8 Hz. The tendency of the dynamic parameters vs. temperature with the other imposed frequency is almost the same with that of 7.8 Hz. The value of the storage modulus is much more important than that of the loss modulus at low temperature range. The storage modulus decreases and the loss modulus increases around the glass transition temperature, which is characterised by the increase of the damping factor.

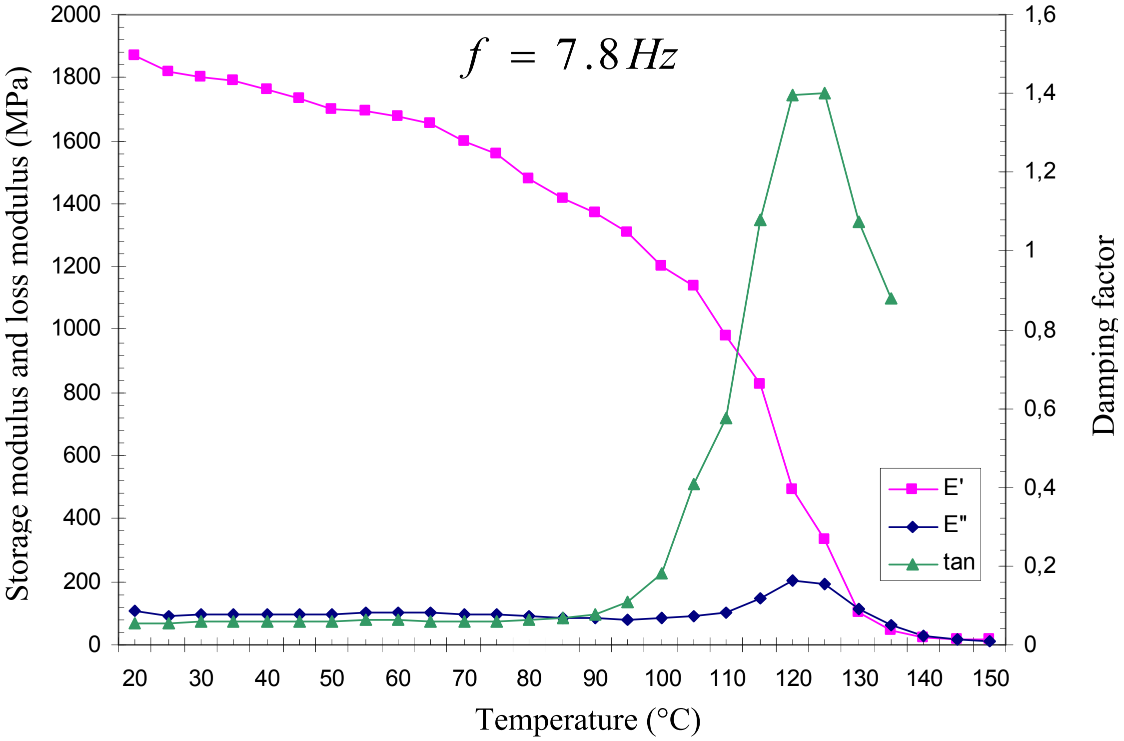

Figure 9 Dynamic parameters of viscoelastic polymer (PMMA): storage modulus, loss modulus and damping factor vs. temperature under the 7.8 Hz imposed frequency of in DMA test

Rheological characterisation

The complex modulus may be obtained through the following equation:

$$\left|E^*\right| = \sqrt{E'^2 + E''^2} \tag{12}$$

The complex modulus vs. imposed frequency at different temperatures is showed in Fig. 10. The values of complex modulus are almost constant at lower temperatures. The significant decrease of the complex modulus can be observed when the temperature arrives the glass transition temperature. The value of the complex modulus at the same temperature (higher than Tg) increase with the increasing of the imposed frequency.

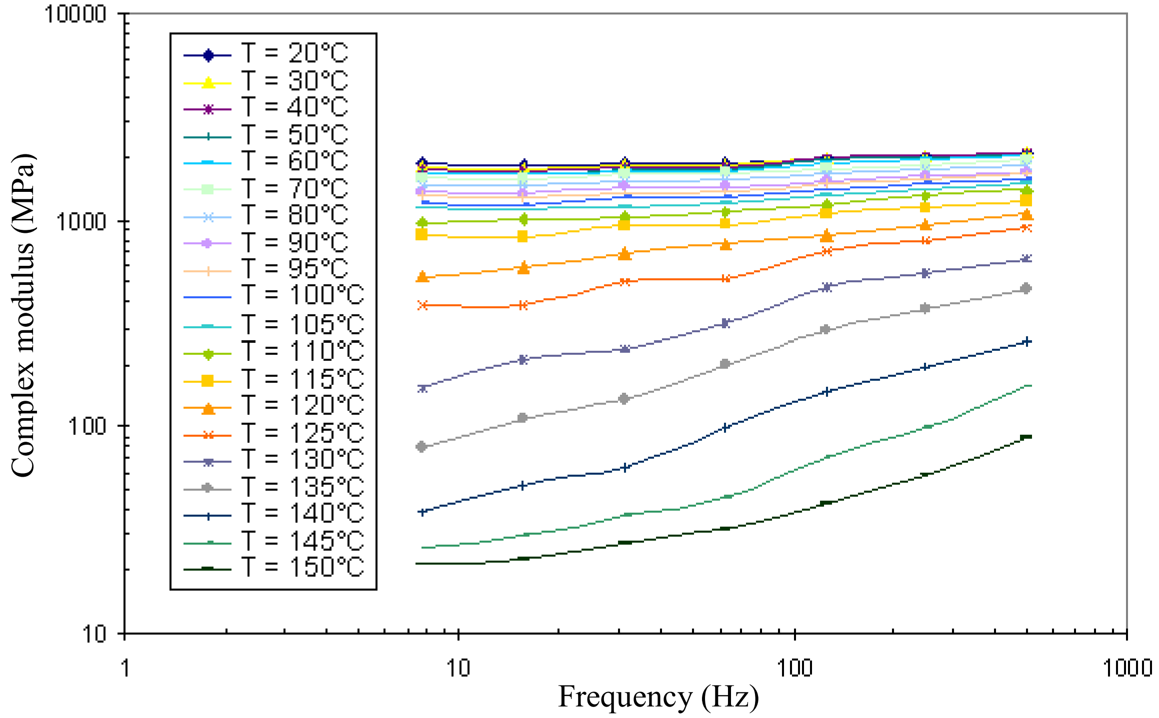

Figure 10 Complex modulus of viscoelastic polymer (PMMA) vs. frequency at different temperature in DMA test

The polymers will be heated to the temperature above Tg in hot embossing process. The viscoelastic behaviour in this temperature range has to be identified. In this study, the Tg of the PMMA polymer could be considered as 110°C due to the variation of the damping factor vs. temperature showed in Fig. 8. But the imposed frequency in DMA test is limited to seven levels from 7.8Hz to 500Hz. The time-temperature superposition principle [21] has been used to extrapolate the viscoelastic properties of polymer over a wide range of frequency beyond the measurement in DMA test.

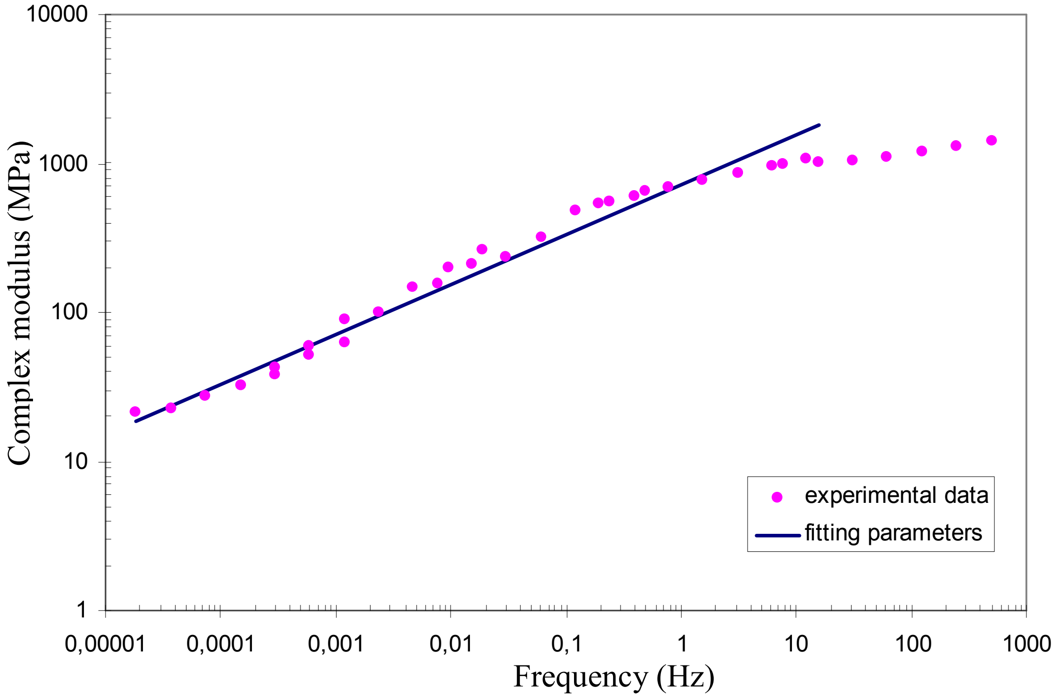

Figure 11 Master curve of polymer (PMMA) complex modulus of vs. frequency at 110°C obtained through test along with the fitting parameters for Generalized Maxwell Model

Material properties over a wide range are determined by superimposing the curves measured in the test by shifting them along the log frequency axis to construct a master curve that spans a much greater window of frequency. The master curve could be obtained from the curves measured, such as showed in Fig. 10. The master curve of complex modulus for the polymer PMMA at 110°C over a wide range of frequency is presented in Fig. 11. The complex modulus of polymer PMMA increases with the augmentation of the imposed frequency.

The Generalized Maxwell model according to Eq. 4 and Eq. 5 has been used to describe the viscoelastic behaviour of polymer PMMA. The fitting parameters in the model have been obtained by first selecting a set of relaxation times and then fitting with experimental data. The optimized fitting parameters for the model are listed in Table 1.

Table 1 Optimized fitting parameters of the Generalized Maxwell model for polymer PMMA at 110°C

Temperature (°C)	E_∞ (MPa)	E^* (MPa)	τ (s)
110	5	2,31E+01	1,00E+05
		2,82E+01	1,00E+04
		8,00E+01	1,00E+03
		1,76E+02	1,00E+02
		3,34E+02	1,00E+01
		6,82E+02	1,00E+00
		1,04E+03	1,00E-01
		1,11E+03	1,00E-02
		1,85E+03	1,00E-03

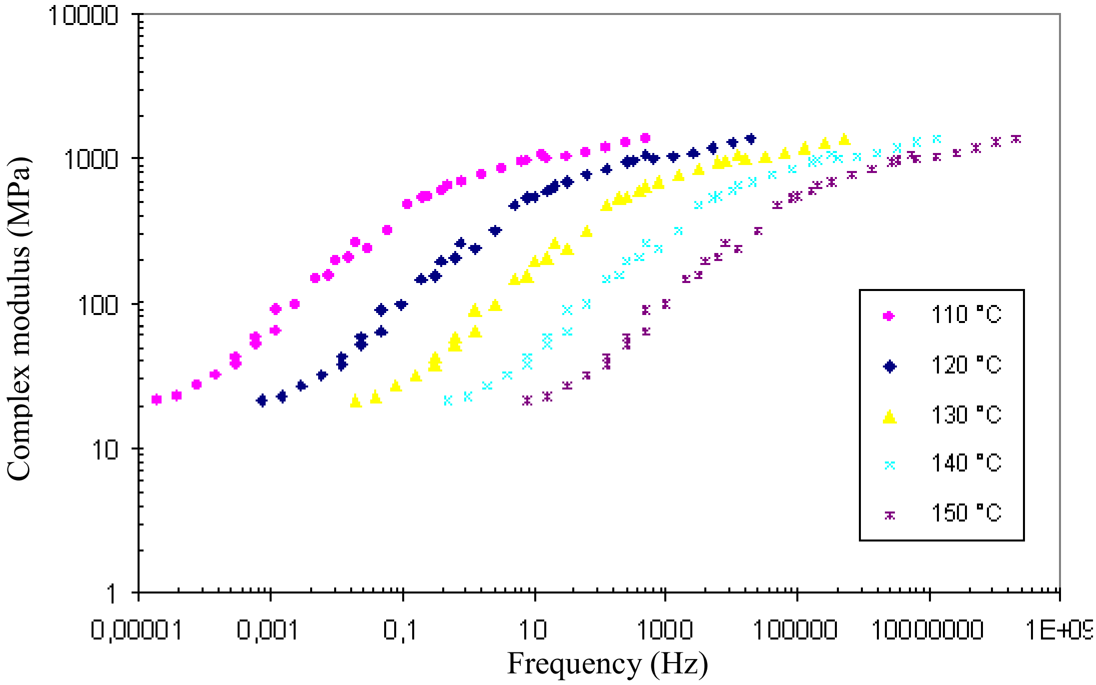

Figure 12 Complex modulus of viscoelastic polymer (PMMA) vs. frequency at different temperature by shifting the master curve

A good agreement has been obtained by using the corresponding fitted Generalized Maxwell parameters and the measured complex modulus master curve of polymer PMMA, as related in Fig. 11. The complex modulus in low frequency could be well fitted by selecting an appropriate value of the infinite relaxation modulus E_∞, which is defined in Eq. 5. That's why the experimental data in low frequency range has been well covered by the identification curve. In addition, the experimental data is obtained by the DMA test from 7.8 Hz to 500Hz, which is relatively low. The data in higher frequency are obtained by translating the master curve with an approximate shift factor. So the values of complex modulus at higher frequency are less accurate than the ones in lower frequency.

The time-temperature superposition principle has been used in order to obtain the master curve of complex modulus at other temperatures. The master curve at 110°C is considered as the reference curve and a shift factor has been selected for each temperature. The shift factors for the temperature 120°C, 130°C, 140°C and 150°C are presented in Fig. 12. The reference curve is shifted horizontally with the shift factor and the master curve at other temperatures could be obtained, as it is showed in Fig. 13.

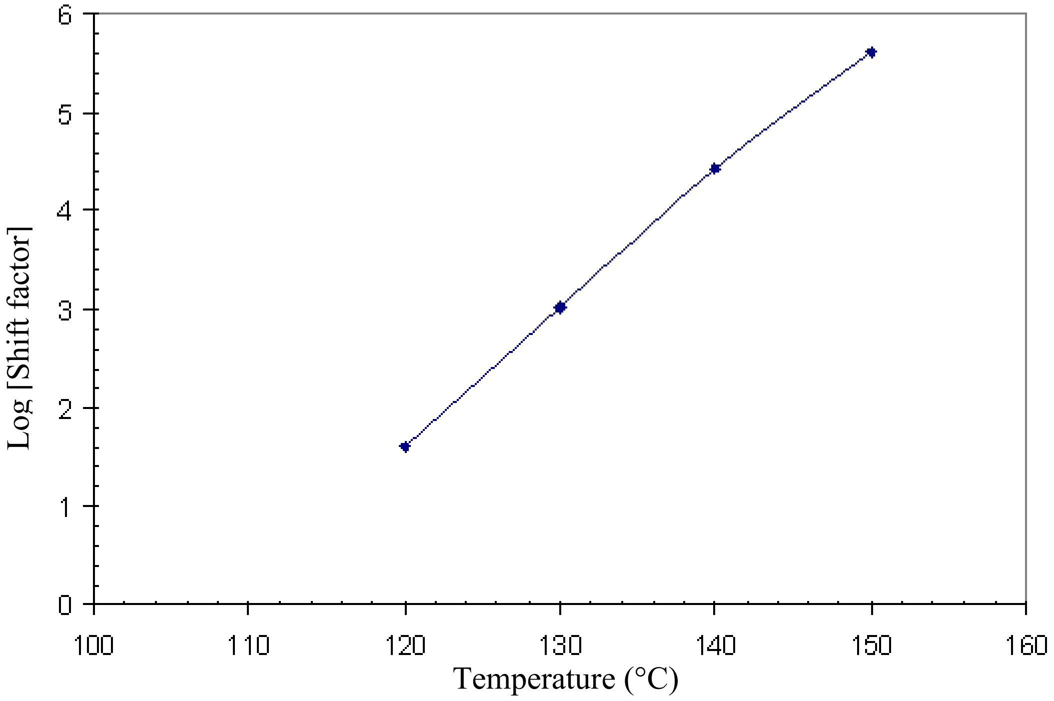

Figure 13 Shift factor used to obtain the complex modulus at other temperatures

Conclusions and perspectives

The viscoelastic models have been investigated in order to develop appropriate model for the polymer behaviour during the hot embossing process. Generalized Maxwell Model has been selected to describe the polymers viscoelastic property and the related parameters involved in the model have been studied, such as relaxation modulus and relaxation time. PMMA's viscoelastic properties have been measured by dynamic mechanic analyse tests. The DMA tests are carried on with a cylinder PMMA sample in viscous analyse equipment. The sample has been oscillated by a series of imposed frequencies and the dynamic mechanical parameters could be obtained. The DMA tests have been carried out in the temperature range from 20°C to 150°C, which spans PMMA's glass transition temperature by taking into account the temperature dependant properties of polymer.

Generalized Maxwell Model has been used to characterize the viscoelastic property of the test polymer PMMA. A good agreement has been observed between the experimental data and the fitting model parameters. The temperature effect has been taken into account by applying the time temperature superposition principle. The complex modulus of the test polymer PMMA above the glass transition temperature has been obtained. The constituted viscoelastic model could be implanted in the simulation software and the numerical simulation of the hot embossing could be achieved.

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