Available online at www.sciencedirect.com
SCIENCE DIRECT®

Polymer 45 (2004) 3789–3796

polymer

www.elsevier.com/locate/polymer

Measurements of the viscoelastic moduli of an acrylate polymer in bulk and film form using a contact method

E. Gacoin, A. Chateauminois*, C. Fretigny

Laboratoire PCSM, Physicochimie des Polymères et de la Matière Dispersée, UMR CNRS 7615, Ecole Supérieure de Physique et de Chimie Industrielles (ESPCI), 10, rue Vauquelin -F- 75231 Paris Cedex 05, France

Received 5 December 2003; received in revised form 11 March 2004; accepted 23 March 2004

Abstract

This paper describes a new method for the measurement of the viscoelastic properties of polymers using a contact mechanics approach. The latter is based on the determination of the tangential response of a macroscopic contact between a polymer specimen and a rigid sphere under small amplitude cyclic micro-motions. Using an acrylate polymer, it was found possible to achieve contact conditions where the tangential behaviour of the contact is strictly linear. Under such conditions, the measurement of the contact stiffness allowed to determine the viscoelastic moduli of the bulk polymer through the glass transition zone. In addition, it was also found to be possible to measure the damping properties of thin films (30 μm) using the same method. The results indicated a shift of the glass transition temperature of the films as compared to bulk specimens. This result was interpreted as an indication of the sensitivity of the glass transition of amorphous polymers to the hydrostatic pressure.

Keywords: Polymer films; Viscoelastic behaviour; Contact stiffness

---

1. Introduction

Polymeric materials are widely used as thin layers (coatings, adhesive or tribological layers…) in applications where their mechanical properties are of great importance. In many practical situations, the corresponding polymer materials cannot be obtained in bulk form or, if this is possible, their mechanical response is often not representative of that of the film. Since the fabrication process and film/substrate interactions may affect the mechanical properties, it is highly desirable to directly characterise the layers themselves.

Standard mechanical dynamic methods are generally difficult to apply on such samples. Nano-indentation [1–4], micro-indentation relaxation measurements [5,6], Brillouin scattering [7], supported dynamic mechanical thermal analysis [8], force microscopy derived techniques [9–11] and quartz resonator [12], are among the specific methods developed in order to mechanically characterise thin layers.

When considering the use of contact methods such as micro or nano-indentation to measure mechanical properties of thin films, one has to face the difficult problem of eliminating the substrate contribution within the context of the large strain elasto-plastic stress fields associated to the use of sharp indenters [1,13]. This problem is further complicated in the case of viscoelastic materials where creep effects combined with time and temperature dependent changes in the modulus can also affect the determination of the contact area, which is an essential prerequisite for any measurement of the viscoelastic modulus. Strojny et al. [3] have also shown that the high hydrostatic pressures exerted by the indenter influenced modulus and yield strength measurement [3].

In this paper, we describe an alternate measurement technique of the viscoelastic moduli of polymers which uses a contact mechanics method. The approach relies upon the determination of the tangential mechanical response of macroscopic contacts between a flat polymer substrate and a rigid glass sphere under predominantly elastic loading conditions.

This method is validated using a bulk acrylate polymer, below and above its glass transition temperature. It is also

---

* Corresponding author. Tel.: +33-1-40-79-47-87; fax: +33-1-40-79-46-86.

E-mail address: antoine.chateauminois@espci.fr (A. Chateauminois).

0032-3861/$ - see front matter © 2004 Elsevier Ltd. All rights reserved.
doi:10.1016/j.polymer.2004.03.067

Nomenclature

a	contact radius
G^*	complex modulus $G^* = G' + iG''$
P	normal load
R	radius of the glass lens
T	tangential load
δ	applied tangential displacement
K_T^*	complex tangential stiffness of the contact
ν	Poisson's ratio

shown that it applies to a thin film of the same acrylate polymer. The presented work constitutes the first part of a more complete study on thin films, where the evolution of the material properties under hydrostatic pressure or severe cyclic deformations is analysed.

2. Materials and experimental techniques

2.1. Materials

The contact method described below applies, in its principle, to any viscoelastic polymer specimen obtained either by polymerisation or conventional melt processing techniques. As an example, a crosslinked acrylate was selected whose chemical composition was adjusted in order to achieve a glass transition temperature below 100 °C and a highly dissipative behaviour in the α transition zone (tan $\delta \approx 1.5$). It was obtained from the copolymerisation of *n*-butylmethacrylate (Acros Organics, purity 99%) and isobutylmethacrylate (Acros Organics, purity 99%) in a 1.2:1.0 molar ratio. The crosslinking agent was butanediol-diacrylate (Lancaster, 85%) with a concentration of 4 mol l^{-1}. Irgacure® 819 (Ciba Specialty Chemicals) was used as an initiator for the radical polymerisation of the mixture under the action of UV light.

Specimens 10 mm in thickness were polymerised from the monomer mixture between two float glass plates which were treated with dichloromethylsilane as a release agent. The reacting species were exposed to UV light during 9 h. In order to ensure an homogeneous polymerisation through the thickness of the specimens, the mould was continuously rotated under the UV lamp. An additional heat treatment at 120 °C under vacuum was carried out for 12 h in order to increase the extent of reaction and to eliminate residual unreacted monomers. The glass transition temperature of the resulting polymer was 53 °C, as measured by DSC at 10 °C/mn. This value remained unaffected when DSC specimens were taken from different locations though the thickness of the acrylate plate, which indicates the absence of any significant gradient in crosslinking density. The polymer gel fraction was found to be greater than 99% after swelling in chloroform and subsequent drying of specimens. The specimens were stored in a desiccator prior to use.

The films were realised using a similar procedure. The specified film thickness (i.e. 33 μm) was obtained using PET spacers which were inserted between the two glass plates. Prior to polymerisation, one of the glass plates was treated with dichloromethylsilane as a release agent; the other one was exposed under nitrogen to a 1% solution of 3-methacryloxy-propyl-dimethyl chlorosilane in toluene in order to promote a chemical bonding between the glass substrate and the polymer film during polymerisation. The use of such a coupling agent was found to be necessary in order to avoid any debonding of the film during the mechanical tests.

After polymerisation, the glass transition of the film was found to be 52 °C by means of DSC at 10 °C/min. The gel fraction of the film was greater than 90%.

2.2. Contact viscoelastic measurements

The viscoelastic properties of the acrylate material were measured from the mechanical response of macroscopic contacts between the polymer surface and a rigid glass lens. A specific device was developed in order to apply tangential cyclic micro-displacements to these contacts under a constant applied normal load. It consisted of the following main parts (Fig. 1):

(i) a moving specimen holder attached to the actuator of a servo-hydraulic MTS 810 machine (MTS, Minneapolis) by means of two thin (0.1 mm) parallel steel blades which ensured a low stiffness (8×10^4 N/m) in the normal load direction and a high stiffness (7×10^6 N/m) in the tangential load direction. This set-up was designed in order to provide a satisfactorily decoupling of the normal and tangential forces during the cyclic loading. Polymer specimens $50 \times 30 \times 10$ mm^3 were glued to the specimen holder on their back faces and mechanically clamped on their lateral faces in order to prevent any relative displacement between the polymer and the specimen holder during the tests.

(ii) a smooth glass lens (rms roughness below 2 nm) was attached to a fixed specimen holder. Two parallel steel blades located behind this holder were used to ensure a high rigidity of this arrangement in the normal load direction and a low stiffness in the tangential load direction. As a result, it was possible to measure the tangential load applied to the contact by means of a piezoelectric transducer fixed to the lower part of the lens holder. The radii of the selected glass lenses were equal to 21 and 78 mm for the bulk acrylate specimens and the films, respectively.

During the tests, a constant normal load was applied to the contact by means of a spring attached to a linear stage located behind the moving specimen holder. A strain gauge transducer in serial with the spring was used to measure the normal load.

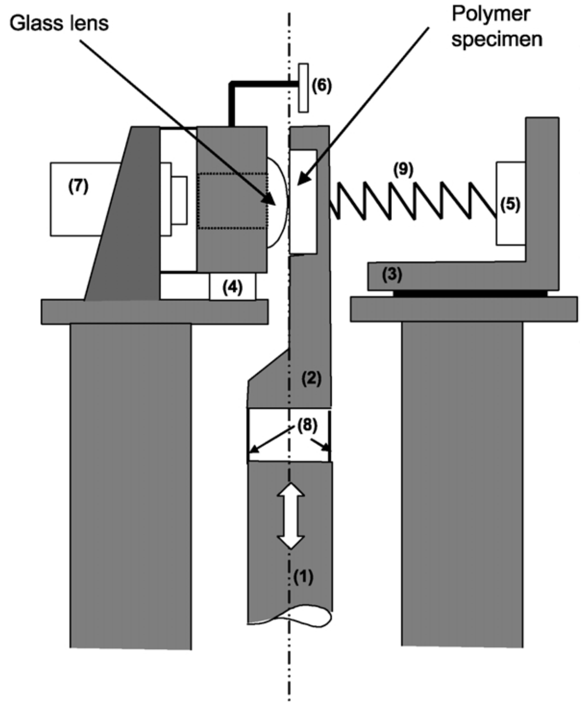

Fig. 1. Schematic description of the contact device. (1) Rod actuator; (2) specimen holder; (3) linear stage; (4) tangential load transducer; (5) normal load transducer; (6) displacement transducer; (7) microscope and CCD camera; (8) leaf springs; (9) spring used to apply the normal load.

The relative displacement between the glass lens and the moving specimen holder was continuously measured using an optical fiberoptic sensor located as close as possible to the contact. This displacement signal was used as an input in the hydraulic actuator feedback loop, which controlled the tangential displacement applied to the contact. This procedure was developed in order to minimize any effects of the machine compliance to the applied contact displacement.

A microscope objective equipped with a CCD camera and an image digitalisation system also allowed the in situ video recording of the contact area through the glass lens during the tests. The whole test device was enclosed within an environmental chamber which allowed controlling the temperature between RT and 100 °C within ± 1 °C. A thermocouple embedded within the bulk polymer specimen close to the contact region was used to monitor the test temperature. For the tests using polymer films, the thermocouple was glued on the specimen holder.

In the experiments described here, the normal load was fixed to 6N. Depending on the temperature, the resulting radius of the contact varied between 0.436 and 2.2 mm. The peak to peak magnitude of the imposed tangential displacement ranged from 0.2 μm at low temperature to 18 μm for the higher ones.

Two kinds of loading procedures were used in order to investigate the frequency dependence of the contact response:

(i) A simple sinusoidal sweep in order to investigate the in-phase and out-of-phase tangential force response at the imposed frequency.

(ii) In order to save time when analysing the lowest frequencies, we also applied another displacement law to the sample which consisted of a linear superposition of harmonic terms. The form of the signal was calculated from the following expression:

$$\delta(t) = \delta_0 \sum_{i=1}^{5} \frac{1}{2^{i-1}} \sin(2^i \pi \nu_0 t) \quad (1)$$

where $\nu_0 = 0.1$ Hz. The value of δ_0 was adjusted as a function of the temperature and the nature of the specimens (bulk or films) in order to obtain a linear response (see below).

The frequency content of this signal was chosen in order to give equally distributed frequencies in a logarithmic scale. The corresponding amplitudes allow a constant signal to noise ratio for each of the harmonics.

As expected from the linearity of the mechanical response of the contact (see below), we have verified that the stiffness obtained through a Fourier analysis of the data was identical to the one which is obtained using procedure (*i*) at each of the signal harmonics (0.1, 0.2, 0.4, 0.8 and 1.6 Hz). This verification has been made at several temperatures and was systematically used in the presented experiments. Moreover, at each of the temperatures, we checked the consistency of data obtained from both procedures at 1.6 Hz. This also validates the linearity of the response (see below).

The servo-loop dynamics limits the high frequency range of the experiments to about 30 Hz.

2.3. Dynamic mechanical thermal analysis (DMTA) of bulk polymer specimens

The conventional bulk viscoelastic properties of the acrylate polymer were measured using a TA Q800 DMTA (TA Instruments, France). Rectangular specimens $17.6 \times 9.15 \times 2.33$ mm^3 were strained under flexural conditions using a single cantilever configuration. Viscoelastic measurements were repeated at temperatures ranging from 40 to 120 °C; the selected frequencies were 0.1, 0.4, 1.6, 6.4, 12.8 and 25.6 Hz. At each temperature step, the measurements were carried out after a dwell time (2 min) in order to ensure that the samples were at the thermal equilibrium.

3. Results

3.1. Linearity of the viscoelastic response

When an elastic sample in contact with a fixed sphere is laterally displaced, friction forces occur. For large displacements, sliding friction takes place. However, when low amplitude cyclic displacements are applied, the situation is different: a central region of the contact interface remains unaffected—no relative displacement of both surfaces takes place. According to Mindlin's theory [14] micro slip only occurs in an annulus region in the outer part of the contact area. More refined models, which take into account adhesion between both surfaces, have been derived by Savkoor and Briggs [15] and Johnson [16]. In the limit of the small cyclic displacements however, one can neglect the partial slip within the outer region and describe the cyclic friction experiment as the drag of a circular region of the sample surface by the slider. The amplitude of the load response T is then linearly related to the displacement amplitude δ through the tangential stiffness K_T [14,17]:

$$T = K_T \delta \tag{2}$$

where

$$K_T = 8\frac{G}{2-\nu}a \tag{3}$$

G is the shear elastic modulus of the sample, ν its Poisson ratio and a is the contact radius. In Eq. (3), the sphere is considered as a perfectly rigid body, which is justified in the present study by the ratio of the glass modulus to polymer modulus (>30).

A direct generalisation can be derived for viscoelastic samples using the correspondence principle [18,19]. Since the domain where the boundary conditions apply is not modified during the experiment, it is sufficient to replace the elastic modulus of the sample with its complex modulus G^* in order to define a complex stiffness, K_T^*, which describes the linear response T^* to a harmonic displacement $\delta \exp(i\omega t)$:

$$T^* = K_T^* \delta \tag{4}$$

where

$$K_T^* = 8\frac{G^*}{2-\nu}a \tag{5}$$

Thus, if the contact size is experimentally determined, the contact stiffness measurement allows the determination of the reduced complex modulus of the sample $G^*/(2-\nu)$. Such a methodology has been used for atomic force microscopy measurements of mechanical properties of surfaces [9,20–23].

The validity of the above analysis relies on a linear response of the contact, which implies that the contact size remains almost unmodified by the cyclic displacement. This condition can only be fulfilled for the smaller displacements.

In order to check that a linear response can be obtained when the displacements are small enough, Fig. 2(a) shows a Lissajous representation of the lateral force for different displacement amplitudes. Averages of 128 cycles at 3.2 Hz are presented in the figure. The experiments are performed at 21.5 °C and the contact radius is 426 μm. Displacement amplitudes were varied in the range 0.22–4.12 μm. When the abscissa and ordinates of these curves are normalised to the excitation amplitude, they become nearly superimposed (Fig. 2(b)). This confirms that, in the considered range of displacement amplitude, the contact lateral mechanical response is linear. For higher amplitudes, a deformation of the cycles takes place; it indicates that some micro-sliding events occur in the contact area.

At this stage, some questions also arise regarding the effects of stress singularities at the edge of the contact. According to acknowledged contact mechanics approaches [24], some stress concentration can take place at the periphery of a contact upon tangential loading. In the case of polymers, such stress concentrations could involve some non-linear viscoelastic response of the highly stressed material domains. However, the fact that a linear response is unambiguously identified at low displacement amplitudes clearly indicates that the contribution of such non-linear effects to the measured contact viscoelastic response is negligible.

3.2. Determination of the viscoelastic moduli from contact stiffness measurements

This section describes the determination of the viscoelastic moduli from the contact response within the above determined linear regime. At each temperature step, the complex contact stiffness and the contact radius were simultaneously measured after a dwell time, which ensured that the specimen was at the thermal equilibrium. In the α transition zone, creep and adhesive effects resulted in the progressive increase in the radius of the contact area under the action of the normal force. In the low temperature range of the transition, these creep effects occurred over an extended timescale, which exceeded largely the time allowed for the experiments. As a consequence, the measured contact radii were not the equilibrium values corresponding to a fully relaxed material under normal loading. It was, however, verified that the size of the contact did not change appreciably during the acquisition time, which is a prerequisite for the accurate measurement of the viscoelastic modulus using Eq. (5).

The complex contact stiffness, K^*, was obtained from a Fourier decomposition of the contact response according to a procedure fully described elsewhere in the context of AFM measurements [21]. A fast fourier transform (FFT) of the tangential force response under the considered excitation spectra was used to determine the in-phase and the out-of-phase components of the contact stiffness. In the case of an excitation signal resulting from the superposition of sine components (cf Eq. (1)), the different harmonics of the FFT spectra provided a separate measurement of K^* at each of the considered frequencies.

The determination of the viscoelastic moduli from Eq. (5) requires knowledge of the Poisson's ratio, ν, at the

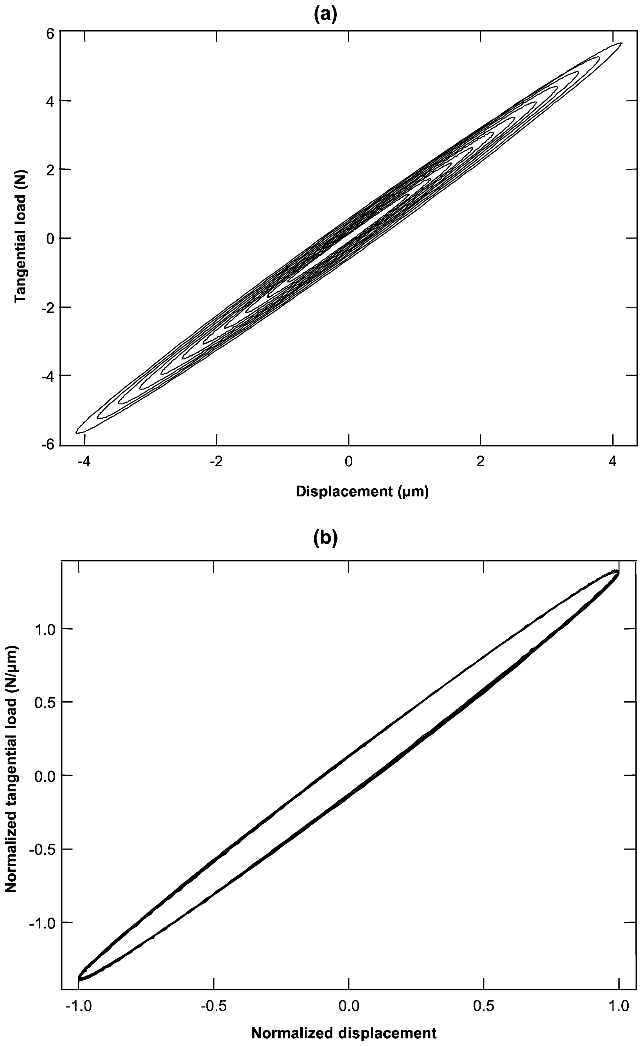

Fig. 2. (a) Tangential force response plotted vs. the sinusoidal displacement, in a Lissajous representation, for 11 different amplitudes (in the range 0.22–4.2 µm). 128 cycles are averaged in order to increase the signal to noise ratio. Experiment is made at 21 °C, normal load is 6N and frequency is 3.2 Hz. (b) Normalized response of (a). The ordinates and the abscissa of the plot have been divided by the amplitude. All the curves are nearly superimposed.

various temperatures under consideration. These values were not known, but it was noted that a change of ν from about 0.3 (minimum value within the glassy range for such amorphous polymers) to 0.5 (i.e. the rubbery value) only results in a 10% change in the calculated modulus, G^*. This uncertainty is very limited when ones take into account the three decades changes in the modulus which occurred within the α transition zone. As a result, all the G^* values reported in this study were calculated assuming that $\nu = 0.5$.

Fig. 3 shows the variation of the conservative and dissipative moduli together with tan δ as a function of temperature at 12.8 Hz. For a comparative purpose, the values obtained from conventional DMTA measurements were also reported in the figure. The very good agreement between both sets of data supports the validity of the contact viscoelastic measurements. When the frequency was varied, the spectra were shifted horizontally along the temperature axis (Fig. 4) according to the linear viscoelastic response of the polymer. Whatever the considered frequency, the measured moduli were in good accordance with the DMTA values.

4. Discussion

When applied to bulk samples, the above described contact method provides results which are in a good agreement with a classical DMTA analysis. Some remarks can be made on the technique. First, it is worthwhile to notice that, even if the modulus changes by about three decades in the glass transition region, the tangential load changes by a much smaller factor. Indeed, the lateral stiffness scales like the contact radius, while the latter scales like the inverse of the cubic root of the modulus in a hertzian contact [24]. As a consequence of the competing effects of the decreased modulus and the enhanced contact size

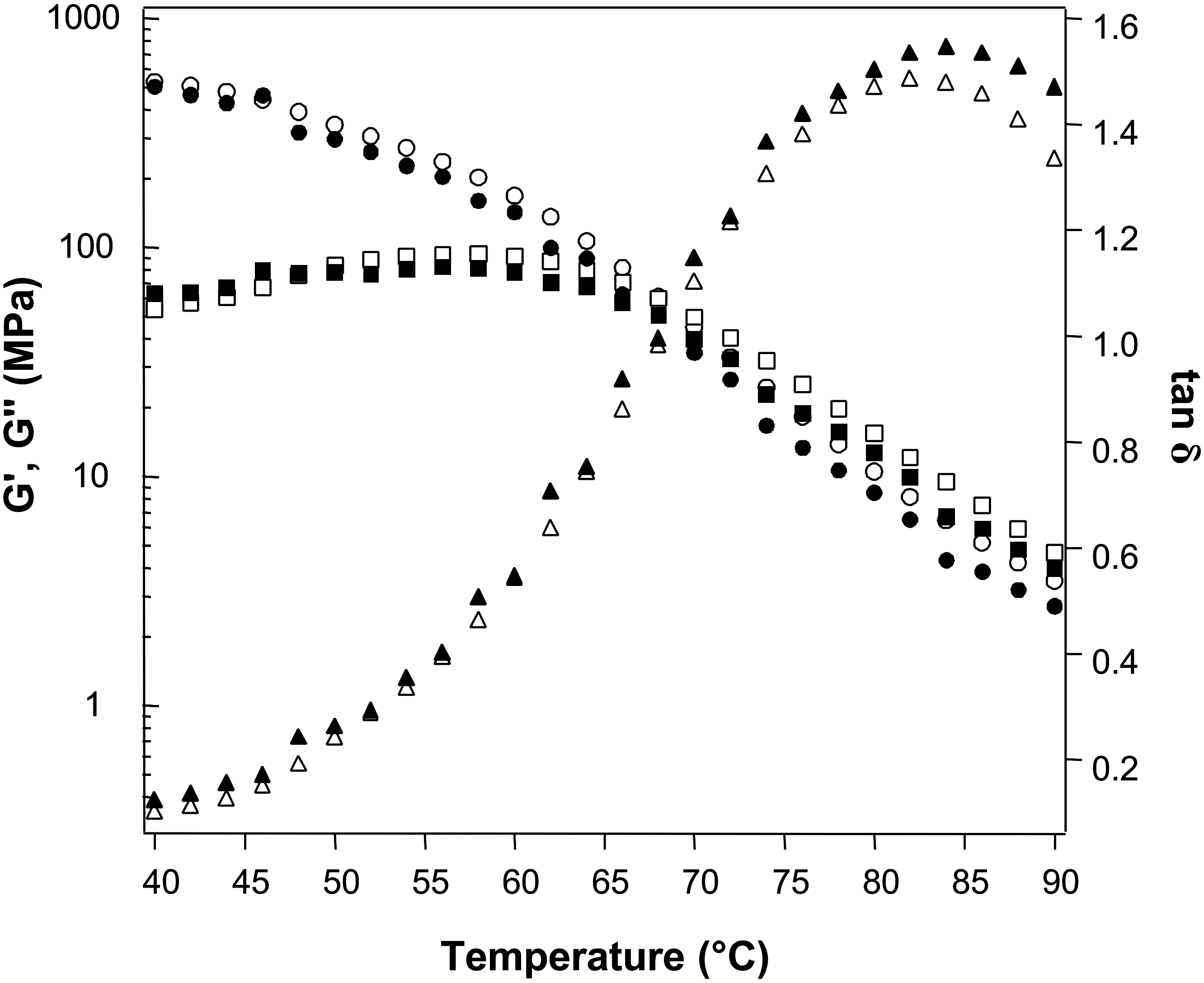

Fig. 3. Variation of the conservative and loss moduli and tan δ as a function of temperature for the bulk acrylate polymer at 12.8 Hz. (●) G'; (■) G''; (▲) tan δ. Open symbols correspond to the DMTA experiments carried out at the same frequency.

through the glass transition, the tangential load only changes by a factor of 10 while the modulus is divided by 1000, for a given normal load and displacement amplitude. The dynamical range for the lateral load through the glass transition is thus rather reduced, which allows the use of a conventional force transducer despite the strong decrease in the specimen modulus.

Moreover, in these experiments, the contact size was continuously monitored in order to get absolute values for the modulus. If one is only interested in the material loss angle, it can easily be obtained from the phase angle. This kind of measurement only requires a simpler version of the experimental set-up without the imaging system for the contact area and it does not necessitate a calibration of the displacements nor of the loads.

It is generally assumed the domain which participates to the mechanical response in a contact experiment has a size which is several times larger than the contact size. Then, in the experiments presented here, on can suppose that the spatial resolution of the measurement is about a few millimetres at the surface of the sample. Then, the method directly applies on samples thicker than a few millimetres. In such a situation, the present contact method can be viewed as a macroscopic equivalent of the shear force modulation measurements which are carried out at the sub-micrometer range using AFM [9,10, 22]. In this study, we have shown that such contact methods can be used to measure viscoelastic properties of polymers through the glass transition zone, which was not previously validated using AFM techniques.

However, as it is discussed below, the contact method may also be used on thinner films. As for the bulk specimens, a prerequisite for the measurements of the viscoelastic properties of thin films is the identification of a linear regime at low displacement amplitudes. The shear deformation of a thin polymer layer within a contact between rigid surfaces can only accommodate a limited part of the imposed tangential

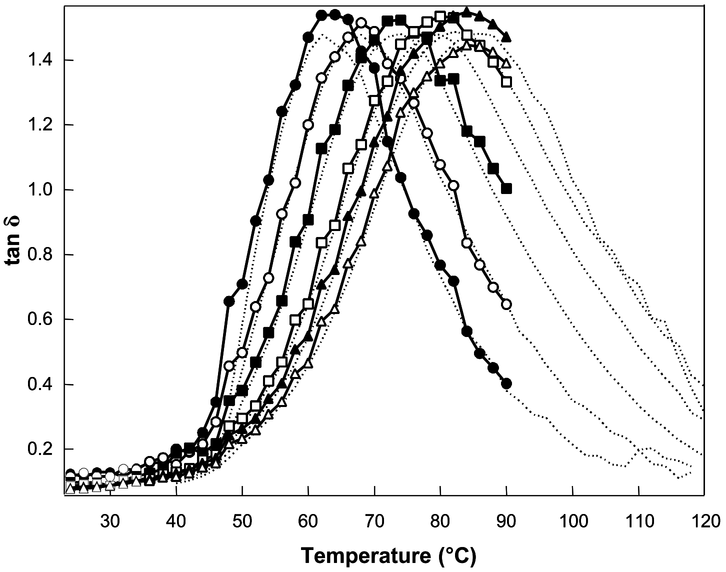

Fig. 4. Variation of the tangent of the loss angle, tan δ, as a function of temperature for the bulk polymer at various frequencies. (●) 0.1 Hz; (○) 0.4 Hz; (■) 1.6 Hz; (□) 6.4 Hz; (▲) 12.8 Hz; (△) 25.6 Hz. Dotted lines correspond to the DMTA measurements carried out at the same frequencies.

displacement. This means that displacement threshold for the occurrence of micro-slip at the contact interface is greatly reduced as compared to contacts involving bulk polymer samples. As a result, the loading conditions associated to a linear response are more critical in the case of thin films. As an example, it was found necessary to apply tangential displacements less than ± 0.5 μm in order to achieve a linear response using a film 33 μm in thickness with a contact with a radius of 700 μm.

In addition, the contribution of the deformation of the glass substrate to the measured contact stiffness can no longer be neglected. This means that Eqs. (4) and (5) which relate the tangential stiffness to the complex modulus of the polymer no longer hold for film systems. In such a situation, a determination of the modulus of the polymer layer would require that the elastic deformation of the glass substrate is taken into account via an appropriate contact model, which is out of the scope of this paper. However, an order of magnitude estimation of the relative contributions to the measured load of the film and of the substrate shows that, except maybe in the purely vitreous regime, the measured phase shift is directly the material loss angle. Fig. 5 shows the changes in the tangent of the phase shift angle as a function of temperature for a film 30 μm in thickness. A damping peak is clearly identified in the glass transition zone. Interestingly, this peak is located at increased temperatures as compared to the bulk polymer specimens, whereas DSC experiments showed that the glass transition

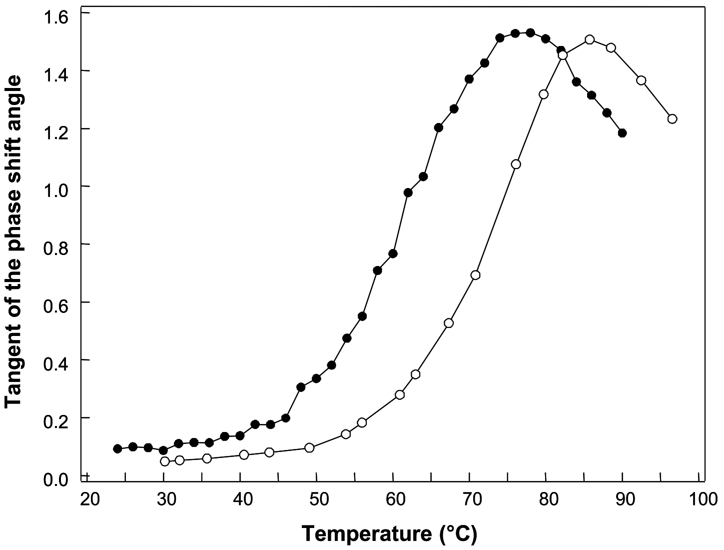

Fig. 5. Variation of the tangent of the phase shift for (●) a bulk specimen and (○) a film 30 μm thick as a function of temperature (3.2 Hz). For the bulk specimen the mean contact pressure decreased from 10 to 0.5 MPa through the glass transition zone as a result of creep effects. For the film specimen the mean contact pressure ranged from 65 to 20 MPa.

did not change appreciably with thickness. This latter effect could be related to known sensitivity of the glass transition to hydrostatic pressure [25–28]. Due to the high level of confinement of the film within the contact (the ratio of the contact radius to the film thickness was greater than 20), the ability of material to flow out of the contact under the action of shear forces is considerably restricted as compared to bulk polymer specimens. As a result, the contact stresses induced within the film are of an essentially hydrostatic nature. Further investigations are currently carried out in order to quantify these effects in relation to reported results for bulk polymer materials.

5. Conclusion

In this study, the potential of contact methods to measure the viscoelastic properties of polymers in their glass transition zone has been shown. Under low amplitude cyclic micro-motions, a complete description of the linear viscoelastic behaviour of bulk polymers as a function of temperature and frequency was provided from the simultaneous measurement of the complex contact stiffness and the contact radius. This contact method was also extended to polymer films on elastic substrates. Within the macroscopic contacts under consideration, it is worth to note that the mechanical properties of the films are determined under highly confined conditions by virtue of the elevated ratio of the contact radius to the film thickness (greater than 20). One of the consequences of this confinement is to induce significant hydrostatic pressure levels (in the order of 10^8 Pa) within the film, which is difficult to obtain using bulk materials. The proposed contact method therefore appears as a privileged tool to investigate the effects of pressure on the viscoelastic properties of polymers. In addition, confined contact conditions could also be used to investigate the mechanical properties of polymers under large shear strain conditions, which are also difficult to achieve using conventional mechanical tests with bulk materials. Within this context, the above detailed linear viscoelastic measurements could be used as a diagnostic in order to investigate the microstructural changes associated with the development of plasticity within the confined film.

Acknowledgements

The authors are pleased to acknowledge the help of Philippe Sergot for the development of the experimental set-up. Hélène Montes is also thanked for her help in the preparation and the characterisation of the samples as well as for many fruitful discussions.

References

[1] He JL, Li WZ, Li HD. Hardness measurement of thin films: separation from composite hardness. Appl Phys Lett 1996;69(10):1402–4.
[2] Nowicki M, Richter A, Wolf B, Kaczmarek H. Nanoscale mechanical properties of polymers irradiated by UV. Polymer 2003;44(21): 6599–606.
[3] Strojny A, Xia XY, Tsou A, Gerberich WW. Techniques and considerations for nanoindentation measurements of polymer thin film constitutive properties. J Adhes Sci Technol 1998;12(12):1299–321.
[4] Loubet JL, Oliver WC, Pharr GM. Measurement of the loss tangent of low-density polyethylene with a nano-indentation technique. J Mater Res 2000;15(5):1195–8.
[5] Shinozaki DM, Lu Y. Micro-indentation relaxation measurements in polymer thin films. J Electron Mater 1997;26(7):852–8.
[6] Krupicka A, Johansson M, Hult A. Viscoelasticity in polymer films on rigid substrates. Macromol Mater Engng 2003;288(2):108–16.
[7] Forrest JA, Dalkoni-Veress K. Effect of free surfaces on the glass transition temperature of thin polymer films (vol 77, pg 2002, 1996). Phys Rev Lett 1996;77(19):4108.
[8] Carlier V, Sclavons M, Legras R. Supported dynamic mechanical thermal analysis: an easy, powerful and very sensitive technique to assess thermal properties of polymer, coating and even nanocoating. Polymer 2001;42(12):5327–35.
[9] Fretigny C, Basire C, Granier V. Determination of complex modulus by atomic force microscopy. J Appl Phys 1997;82:43–8.
[10] Basire C, Fretigny C. Determination of viscoelastic moduli at a submicrometer scale. Eur Phys J Appl Phys 1999;6:323–9.
[11] Oulevey F, Burnham NA, Cremaud G, Kulik AJ, Pollock HM, Hammiche A, et al. Dynamical mechanical analysis at the submicron scale. Polymer 2000;41:3087–92.
[12] Domack A, Johannsmann D. Plastification during sorption of polymeric thin films: a quartz resonator study. J Appl Phys 1996; 80(5):2599–604.
[13] Pharr GM, Oliver WC. Measurement of thin film mechanical properties using nanoindentation. Mater Res Soc Bull 1992;17(7): 28–33.
[14] Mindlin RD. Compliance of elastic bodies in contact. ASME Trans J Appl Mech Ser E 1953;16:327–44.
[15] Savkoor AR, Briggs GA. The effect of tangential force on the contact of elastic solids in adhesion. Proc R Soc London A 1977;356:103–14.
[16] Johnson KL. Adhesion and friction between a smooth elastic spherical asperity and a plane surface. Proc R Soc London A 1997;453:163–79.
[17] Mindlin RD. Compliance of elastic bodies in contacts. J Appl Mech Trans ASME 1949;71:259–68.
[18] Lee EH, Radock JRM. The contact problem for viscoelastic bodies. J Appl Mech Trans ASME 1960;82:438–44.
[19] Graham GAC. Q. Appl Math 1968;26:167.
[20] Fretigny C, Basire C, Granier V. J Appl Phys 1997;82:43.
[21] Basire C, Fretigny C. Determination of viscoelastic moduli at a submicrometric scale. J Appl Phys 1999;6(3):323–9.
[22] Ge S, Pu Y, Zhang W, Rafailovich MH, Sokolov JC, Buenviaje C, et al. Phys Rev Lett 2000;85:2340–3.
[23] Zhang Y, Ge S, Rafailovich MH, Sokolov JC, Colby RH, Ngoepe PE, et al. Surface characterization of cross-linked elastomers by shear modulation force microscopy. Polymer 2003;44(11):3327–32.
[24] Johnson KL. Contact mechanics. Cambridge: Cambridge University Press; 1985.
[25] Briscoe BJ, Smith AC. The shear properties of thin organic films. Reviews on the deformation behaviour of materials III Freud Publication House Ltd, Israel. 1980. p. 151–91.
[26] Jones-Parry E, Tabor D. Pressure dependence of the shear modulus of various polymers. J Mater Sci 1974;9(2):289–92.
[27] Ngoepe P, Lambson EF, Saunders GA, Bridge B. The elastic behaviour under hydrostatic pressure of poly(methyl methacrylate) and its fully deuterated form. J Mater Sci 1990;25(11):4654–7.
[28] Matsushige K, Baer E, Radcliffe S. The mechanical behavior of poly(methyl methacrylate) under pressure. J Polym Sci Polym Phys 1976;14(4):703–21.