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DETERMINATION OF DUAL GLASS TRANSITION TEMPERATURES OF A PC/ABS BLEND USING TWO TMA MODES

Mojtaba Haghighi Yazdi and Pearl Lee-Sullivan[*]

Department of Mechanical and Mechatronics Engineering, University of Waterloo, Waterloo, ON, N2L 3G1, Canada

An inherent challenge with polymer blends is the difficulty in resolving the glass transition, T_g, for the smaller mass fraction component. The objective of this work was to determine the practical scanning conditions for identifying the dual T_g's for a 75:25 polycarbonate/acrylonitrile-butadiene-styrene (PC/ABS) blend using a thermomechanical analyzer (TMA). Scanning rates up to 20°C min^{-1} using dilatometer and expansion modes were studied. Heating and cooling rates were found to affect both T_g values but the effects were not simple relationships. T_g values could either increase or decrease depending on the scanning rate applied. Higher rates resulted in large thermal lags which opened the accuracy of measurements to question. Generally, higher rates tended to display only one T_g but the duality of T_g's can be detected with scanning rates between 0.5 and 5°C min^{-1} for both modes.

Keywords: *dilatometer, expansion probe, glass transition temperature, polycarbonate/acrylonitrile-butadiene-styrene (PC/ABS) blend, thermomechanical analysis (TMA)*

Introduction

Polycarbonate/acrylonitrile-butadiene-styrene (PC/ABS) blends are increasingly replacing polycarbonate because they offer better thermal and mechanical properties, improved physical aging properties, easier processability, and are more cost-effective [1, 2]. To date, the two components of the blend have been found to be partially immiscible [1, 3], and hence the presence of two glass transition temperatures (T_g) are important design considerations.

Thermal analysis techniques are known to be convenient means of determining the T_g of polymer blends. However, there is very limited work in the open literature on the use of thermomechanical analysis (TMA) on polymer blends [4–7]. Ragosta *et al.* [5] applied the penetration and extension modes of TMA on polyolefin/polystyrene blends and polyolefin/polyolefin mixtures to obtain the coefficient of thermal expansion (CTE) of these materials in rubbery and glassy regions. Their results showed that the addition of small quantities of a polymer to a different polymer matrix modifies the thermomechanical properties of the blend. Belaribi *et al.* [7] evaluated the compatibility level of PC/MPC blends by determination of T_g with penetration mode of TMA. They obtained only one T_g for blends containing less than 75% PC and two T_g's for blends with higher PC contents. They also presented a microscopic model which explains the data obtained from TMA. Using a heating rate of 2.5°C min^{-1} and the penetration mode of TMA, Minkova and Nikolova [4] studied the effect of irradiation on temperature of melting as well as Young's modulus of LDPE-iPP and EVA-iPP polymer blends. Nandan *et al.* [6] measured CTE of binary blends of PES and Nylon-6 using the penetration mode of TMA on cylindrical specimens with heating rate of 4°C min^{-1}. They found that the blend CTE decreases with PS concentration resulting in higher dimensional stability.

The presence of dual T_g's for the PC/ABS blend has already been reported using differential scanning calorimetry (DSC) and dynamic mechanical analyzer (DMA) [1–3, 8], but there are no studies in the open literature using the TMA method to study this blend. TMA remains an important technique today due to its capability to measure both T_g and CTE, as well as several other physical characteristics depending on the operating mode [9]. TMA has several operational modes, namely expansion, penetration, flexure, and tension [10, 11], which give different types of thermophysical data.

It is widely known that heating rates affect the reliability of glass transition measurements due to thermal lags. A review of the open literature going as far back as 30 years revealed a number of reports [4–7, 9, 12–35] on the different heating rates used for characterizing polymers by different methods including TMA, as shown in Table 1. However, there are only a limited number of reports [9, 12] on the influence of different heating rates on T_g measured using TMA. Schwartz [12] studied the influence of heating

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[*] Author for correspondence: pearls@uwaterloo.ca

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Akadémiai Kiadó, Budapest, Hungary
Springer, Dordrecht, The Netherlands

and cooling rates on the T_g and the CTE of double-base propellant and carboxyl-terminated polybutadiene. He used the expansion and penetration modes of TMA to calculate T_g and CTE below and above T_g at four different heating rates. He found that T_g is dependent on heating and cooling rates but not on sample thickness. The experiments also showed that T_g increased with increasing heating rate and decreased with increasing cooling rate. Brazier and Nickel [9] also investigated the effect of heating rate on T_g of elastomer vulcanizates using three modes of TMA. They found out that over the scanning rate range of 2–20°C min^{-1}, the peaks of the DTMA curve remained constant.

In studies involving polymeric composites, the effect of heating rate on T_g was also studied for epoxy composites using TMA [22, 34]. O'Neal *et al.* [34] studied the effect of heating rate on T_g of a graphite epoxy composite using three different modes of TMA (penetration, flexure and expansion) and compared the results. They found that heating rate has a significant influence on T_g of the tested material.

Table 1 Range of heating rates used in TMA studies

Min. heating rate/°C min^{-1}	No.	Authors	Year	Details
0.5–1	1	Schwartz [12]	1978	expansion and penetration modes, heating rates: 1.04, 2.08, 4.16, 8.32°C min^{-1}
	2	Mano and Cahon [13]	2004	3-point bending tests, heating rates: 0.5, 1, 2, 4, 8°C min^{-1}
2	3	Brazier and Nickel [9]	1978	rapid evaluation of elastomer vulcanizates, heating rates: 2–50°C min^{-1}
	4	Seyler and Earnest [14]	1991	heating rates: 2–10°C min^{-1}
	5	Paroli and Penn [15]	1994	T_g of EPDM roofing materials, comparison of DMA, TMA and DSC
	6	Skuban *et al.* [16]	2003	T_g and CTE of non-crystalline chalcogenides
	7	Sencadas *et al.* [17]	2006	TMA, DSC and DMA studies of a copolymer film
	8	Kim *et al.* [18]	2007	CTE measurement, expansion mode
2.5	9	Minkova and Nikolova [4]	1989	blend, penetration mode
4	10	Ragosta *et al.* [5]	1982	blend, penetration mode
	11	Nandan *et al.* [6]	2001	blend, penetration mode
5	12	Matsumori *et al.* [19]	1991	correction methods for CTE measurement by TMA
	13	Lee *et al.* [20]	1991	asphalt cement characterization
	14	Cassel and Twombly [21]	1991	epoxy composite, comparison of TMA, DSC and DMA
	15	Jankowsky *et al.* [22]	1994	composites, heating rates: 5, 10, 20°C min^{-1}
	16	Gupta [23]	1994	automotive coating, penetration mode
	17	Sircar and Chartoff [24] Sircar *et al.* [25]	1994 1999	T_g of elastomers by TMA, DMA and DSC
	18	Bikiaris and Karayannidis [26]	1996	penetration mode
	19	Shi and Economy [27]	1998	measuring CTE by TMA
	20	Jurkowski *et al.* [28]	2002	penetration mode
	21	Pesetskii *et al.* [29]	2005	effect of cooling rate on structure of a molten polymer by TMA
	22	Wang and Jiang [30]	2007	dilatometer mode
	23	Backfolk *et al.* [31]	2007	T_g measured by TMA, DSC, DMA and SPM and then compared
10	24	Wiedemann *et al.* [32]	1994	comparison (TMA, DSC, TOA)
	25	Boztuğ *et al.* [33]	2004	blend, tensile mode, measuring T_g and CTE
	26	O'Neal *et al.* [34]	2005	T_g of a graphite epoxy composite by TMA, DSC and DMA, heating rates: 2, 5, 10°C min^{-1}
20	27	Belaribi *et al.* [7]	1986	PC/MPC blend, penetration mode
	28	Gaddy *et al.* [35]	1991	TMA of EPDM roofing membrane material

Jankowsky *et al.* [22] used expansion mode of TMA to obtain T_g of a toughened epoxy composite under three different heating rates. However, they could not get satisfactory results for this type of resin with TMA.

The T_g values found by TMA have also been compared with those determined by other thermal analysis techniques, which were applied to both polymers and composites by several investigators [21, 24, 25, 31, 32]. Cassel and Twombly [21] performed three different methods of TMA (flexure, expansion and stress relief) on an epoxy composite and compared the obtained results with those of DSC and DMA experiments. They obtained similar T_g for the material by applying different T_g assigning methods on the obtained curves. Wiedemann [32] used four runs to determine the T_g of polystyrene (PS) and PET using the methods of TMA, DSC and TOA (thermooptical analysis). He compared the obtained results and found good agreement between them. Sircar *et al.* [24, 25] discussed some of the difficulties in determination of T_g of elastomers and employed three thermal analysis techniques of DSC, TMA and DMA. They used expansion and penetration modes of TMA to find T_g of elastomers and concluded that T_g determined from penetration mode is very close to that found from DSC experiments. They also presented some recommendations to obtain consistent data for T_g of elastomers using different thermal analysis techniques. Recently, Backfolk *et al.* [31] applied the penetration mode of TMA on two styrene/butadiene lattices and compared the results with those measured by DSC, DMA, process rheometer (PR), and SPM probe resonance frequency method. They reported that the T_g of the latex with low styrene content obtained from TMA agreed with the thermal SPM data but for the second latex, TMA gave lower T_g with respect to the other methods.

A number of reports on comparison of different thermal analysis techniques, including TMA, which were performed on polymer materials for construction applications were also found in the literature [15, 20, 23]. Lee *et al.* [20] used DSC and expansion mode of TMA to characterize the T_g of aged asphalts at heating rate of 5°C min^{-1}. They found reasonable agreement between T_g determined by DSC and TMA. The T_g of EPDM roofing membranes was determined by Paroli and Penn [15]. They used DMA, DSC and expansion mode of TMA for this purpose and found the same T_g from TMA and DSC experiments within experimental error. DMA, DSC and TMA were also employed by Gupta [23] to determine the T_g of automotive organic coatings on metal substrates. They used a heating rate of 5°C min^{-1} and found TMA very successful for the measurement of T_g for this type of material.

The current work is part of a wider experimental study which applies thermal analysis as a tool for characterizing a PC/ABS blend. This investigation is focused on comparing the values between a dilatometer and expansion mode using a TMA. The secondary objective is to determine the practical heating rates for studying PC/ABS. This is particularly important for the dilatometer probe as there is a need to measure the volumetric expansion or contraction of the blend for understanding mechanisms such as structural relaxation during physical aging.

Experimental

Materials and methods

The material investigated in this work was a 75:25 polycarbonate/acrylonitrile-butadiene-styrene (PC/ABS) blend. The blend is commercially known as Cycoloy C6600, which is produced by GE Plastics (Pittsfield, MA, USA) and has a melt flow rate of 21.5 g/10 m at 260°C with 2.16 kg_f in accordance with ASTM D 1238 [36] standard. The mass average molecular mass (Mw) of the blend was found to be 45000 by Jordi FLP (Bellingham, MA, USA) using Gel Permeation Chromatography with RI (GPC-RI) and GPC-FTIR techniques. The size of the molecules in solution was found to be the same and could not be resolved using GPC. Moreover, GPC-FTIR confirmed that they were eluting at the same time.

Thermomechanical analysis was performed on a TA Instruments 2940 TMA using two modes: an expansion probe and a dilatometer. The temperature measurement reproducibility in the TMA chamber was ±2°C while machine sensitivity for deformation measurement was ±100 nm [37]. In both of testing modes, each sample was weighed to 0.1 mg.

The temperature was ramped from room temperature to 135°C under a nitrogen purge gas with flow rate of 100 mL min^{-1}. An isothermal treatment at 135°C for 10 min was used to remove the thermal history of the sample, followed by a cooling scan to 60°C. Subsequent to this, a second heating scan was applied to find the glass transition temperature after thermal history removal [32]. In total, seven heating (q_1) and cooling rates (q_2) were investigated, that is, $q_1=q_2=0.5, 1, 2.5, 5, 10, 15, 20$°C min^{-1}. Four samples were tested at each of the heating and cooling rates to increase the power of statistical evaluation and to maintain the statistical normality assumption. The calibrations and methodology specific to each probe are described below.

Expansion mode

An expansion probe with a circular flat tip was used to measure the displacement change of the polymer samples under constant thermal heating and cooling rates, in accordance with the ASTM E 1545-05 [38] standard. Calibrations for temperature and length change were done according to ASTM E 2113-04 [39] and E 1363-03 [40] standards, respectively. Samples with dimensions of 5×5 mm were cut from a 152×305 mm sheet of 3 mm nominal thickness. The samples had an average mass of 100.5 mg (with standard deviation of 3.8 mg).

Dilatometer mode

The dilatometer accessory kit for the TMA consisted of a dilatometer probe, sample vial, filling medium and aluminium balls. The filling medium was used to convert the volume change of the sample inside it into displacement recorded by the dilatometer probe. The samples were in the form of small granules with average mass of 56.7 mg (with standard deviation of 1.8 mg).

Baseline and displacement calibrations were conducted before the tests. The baseline calibration consisted of a thermal scan from room temperature up to 160°C at a heating rate of 1°C min^{-1} under compression loading of 0.01 N [41]. This load value of 0.01 N was also used in testing. The displacement calibration is similar to temperature calibration in DSC, in which the melting point of a standard material (usually indium) is used for calibration. For this calibration, three aluminium balls were placed into the vial with 3/4 of the vial filled with the filling medium. The cell constant in the software, referred to as vial constant in TMA, was set to 1. The thermal procedure was the same as what was done for baseline calibration.

Results and discussion

The glass transition temperature can be specified using different methods, either the onset temperature or the inflection point on the curve [42]. Since the slope of length change *vs.* temperature shows the CTE and there is a change in CTE at T_g [34], the glass transition temperature can be defined at a point where the curve slope changes. Assigning glass transition temperature to this blend was, therefore, done using two tangent lines drawn on the curve at the points where the slope changes, as proposed by Earnest [41] for an expansion probe test. The same method is also applicable to the curves obtained from dilatometer. Figure 1 shows how two T_g's were typically assigned from dilatometer mode results at the heating rate of 0.5°C min^{-1}. The first point shows the T_g of ABS in the blend at 100.30°C and the second point assigns 127.57°C to the T_g of PC in the blend. The same method was also used for the expansion probe analysis.

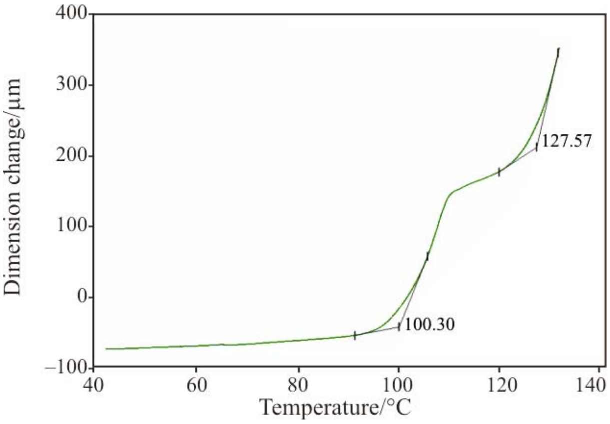

Fig. 1 Assignment of T_g on the blend curve in dilatometer mode at heating rate of 0.5°C min^{-1}

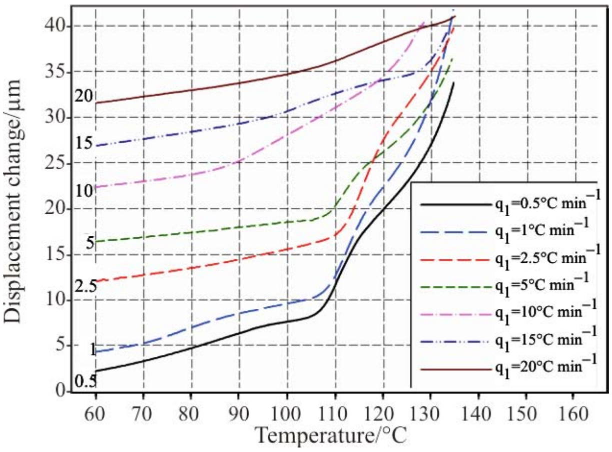

Fig. 2 Displacement change *vs.* temperature at different heating rates from expansion probe mode experiments

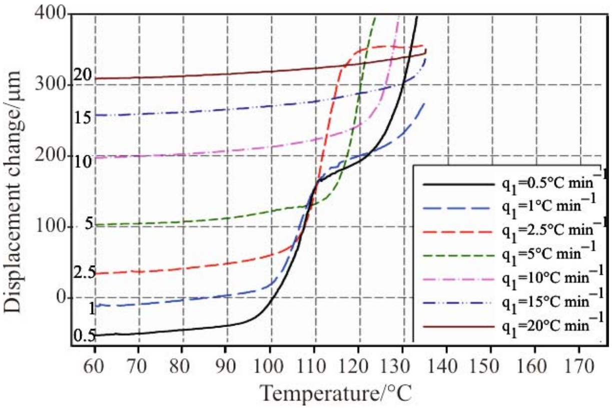

Fig. 3 Displacement change *vs.* temperature at different heating rates from dilatometer mode experiments

Figures 2 and 3 show the typical curves obtained at different heating rates from expansion probe and dilatometer modes, respectively. The superimposed curves demonstrate that the shape of the curves was strongly dependent on applied heating rates.

As seen in Figs 2 and 3, the resolution of the two T_g's of the blend components and confidence in assignment accuracy become increasingly more challenging with increasing heating rates. High heating rates produce significant thermal lag errors in the results, which are well known for the TMA technique since the sample mass is relatively large [1, 21]. High heating rates cause high thermal gradients in the sample and in the furnace, from which thermal lag errors increase [9]. Lower heating rates are therefore recommended in order to avoid erroneous T_g values [42]. Our results suggest that lower heating rates ranging from 0.5 to 5°C min^{-1} are most practical for TMA experiments at the two expansion and dilatometer modes.

Figures 4 and 5 show the T_g of PC and ABS obtained during heat-up and cool-down scans in the expansion probe and dilatometer modes, respectively. The obtained glass transition temperatures seem reasonable as they fall within the temperature range between T_g of pure PC and that of pure ABS for this partially immiscible blend. However, dilatometer and expansion probe tests fail to detect $T_{g,PC}$ on cooling scan.

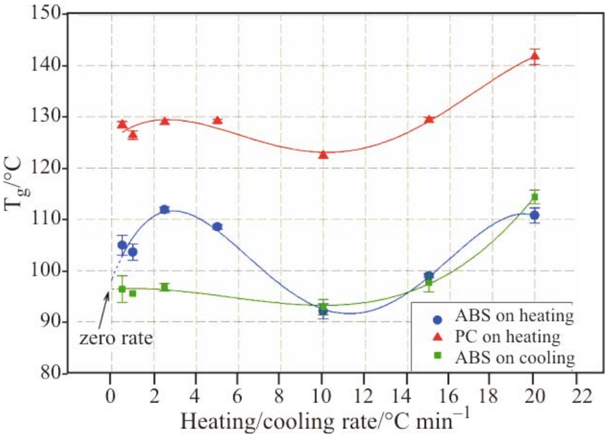

Fig. 4 T_g's of the blend components obtained using the expansion probe for various heating and cooling rates

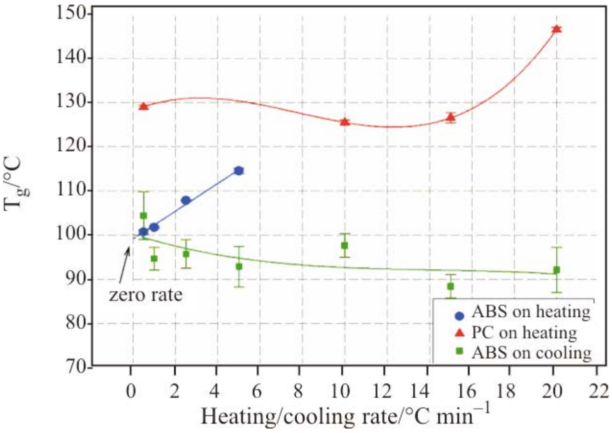

Fig. 5 T_g's of the blend components obtained using the dilatometer probe for various heating and cooling rates

Figures 4 and 5 show a general trend in T_g values with increasing heating rates but the effect of cooling rate is less obvious. One explanation for the latter observation is the larger error bars in the cooling rate curve for ABS which suggests that cooling rate experiments are more prone to thermal lag errors. Another parameter that can be obtained from the curves in Figs 4 and 5 is the T_g at zero heating/cooling rate by extrapolating to zero scan rate (dashed lines). Table 2 shows the T_g of ABS at zero heating/cooling rate from dilatometer and expansion probe tests.

It can be seen that the T_g's at zero rate are within the experimental error on heating and cooling scans for both expansion probe and dilatometer tests. In fact, T_g at zero rate can be considered to be the same if examined within the maximum experimental error of 3.8%. This observation is consistent with earlier suggestion by Schwartz [12] that T_g at zero rate could be considered a material constant.

In order to check whether there is a statistical difference between the T_g's measured at various heating rates, an Analysis of Variance (ANOVA) was performed. ANOVA showed that, in general, there is a statistical significance between the results of each heating rate group with significance level of 0.05, which means that the heating rate influences the values of T_g. This was expected because the glass transition is a relaxation process and is influenced by heating rate change [43]. This is also similar to the findings of Schwartz [12], in which heating and cooling rates influenced T_g. However, there was no difference in the results for heating rates between 0.5 and 1°C min^{-1}, and between 2.5 and 5°C min^{-1} measured by the expansion probe.

It is also known that the sample mass influences the obtained T_g [32, 44]. In order to compare the data within each heating rate group the effect of sample mass should be accounted for. This was accomplished using Analysis of Covariance (ANCOVA), in which the sample mass was taken as a covariate, thus enabling us to compare the T_g obtained from both TMA modes together. The difference between masses (100.5 mg for

Table 2 T_g of ABS at zero scan rate from expansion probe and dilatometer tests measured in °C

Mode	Heating	Cooling
Expansion probe	98±2.5*	96±2.1
Dilatometer	99±0.9	100±4.1

*95% Confidence interval

the case of expansion mode and 56.7 mg for the dilatometer case) was considered and the results were compared. ANCOVA showed that there is no statistical significance in T_g data due to the sample mass change and therefore, the results could be compared.

In order to compare the values of T_g's between expansion probe and dilatometer results, the 95% confidence interval was calculated for each of them and checked whether the T_g of the other group falls within this interval or not. Using this method, it was found that T_g's of PC and ABS obtained from expansion probe and dilatometer are equal for heating/cooling rate of 0.5°C/min. However, there was no agreement between the T_g's obtained from expansion probe and dilatometer modes at the other heating rates.

The second heating scan after the cool-down scan was performed to measure the T_g after thermal history removal during the isothermal step. The comparison of the curves found from the first and second heating scans, Fig. 6, shows the effect of thermal history removal.

Figure 6a illustrates the difference between T_g's of both PC and ABS obtained from the expansion mode before and after thermal history removal step. The thermal history removal had greater effect on PC than ABS. Figure 6b shows that the dilatometer was able to detect the glass transition of ABS in the blend during first heat-up but not the second. This is very likely due to the low ABS content in the blend. This was also observed in DSC and DMA studies of this blend [45].

Table 3 shows the average values obtained for the coefficient of thermal expansion (CTE) before and after T_g, which are denoted by α_g (CTE in glassy region) and α_r (CTE in rubbery region), respectively. These values were obtained for both of the blend components.

As it is seen in Table 3, the ratio of α_r to α_g for ABS is almost twice that for PC (2.3 for expansion probe results and 1.6 for dilatometer ones). Apparently, the change of CTE at the glass transition is greater for ABS than PC.

Finally, if the current glass transition temperature values are compared with those determined from DSC and DMA experiments already reported by our group [45], the TMA values are the highest as shown in Table 4. It is noted, however, that the values of DSC have been obtained using a heating rate of 10°C min^{-1} while TMA results were obtained using a heating rate of 0.5°C min^{-1}. The values of DMA were also obtained at a frequency of 1 Hz and a heating rate of 1°C min^{-1}.

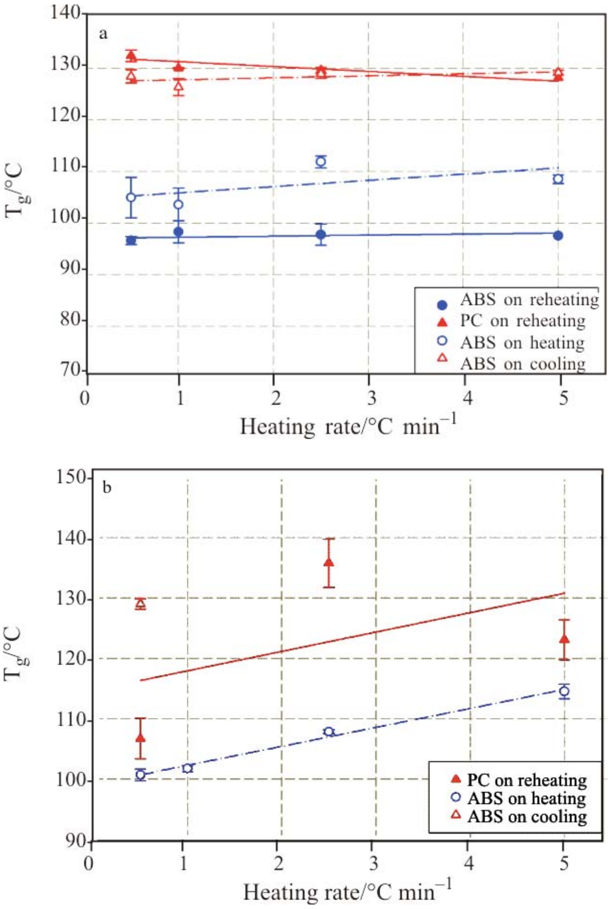

Fig. 6 Comparison of T_g's of the blend components obtained on heating and reheating scans for a – expansion probe mode and b – dilatometer mode

As was expected, there are differences in the T_g values since each of these methods measure a different thermal property. Specifically, the DSC measures heat capacity, the TMA measures thermal expansion (length or volume), while the DMA measures loss tangent (tanδ) or loss modulus (E'') as a function of temperature [25].

The difference in the glass transition temperatures recorded by expansion probe and dilatometer is due to the fact that displacement of the sample is recorded in one dimension by the expansion probe, whereas the dilatometer kit makes it possible to convert the volume change of the sample into displacement. Therefore, the displacement of other directions is not accounted for in the expansion mode.

Table 3 Average values of CTE before and after T_g obtained from dilatometer and expansion modes

Mode	ABS			PC		
	α_g/μm °C^{-1}	α_r/μm °C^{-1}	α_r/α_g	α_g/μm °C^{-1}	α_r/μm °C^{-1}	α_r/α_g
Expansion probe	0.14	1.31	9.36	0.81	3.26	4.02
Dilatometer	1.00	23.43	23.43	1.36	20.07	14.76

Table 4 Comparison of glass transition temperatures obtained using DSC, DMA and TMA

Method			$T_{g,ABS}$/ °C	$T_{g,PC}$/ °C
DSC [45]			92	112
DMA [45]			100	112
TMA	Expansion Probe	heating	105	129
		cooling	98	–
		reheating	97	132
	Dilatometer	heating	101	129
		cooling	104	–
		reheating	–	130

It should also be pointed out that although TMA has its own advantages, particularly for measuring volume relaxation of polymers or measuring coefficient of thermal expansion, it has some disadvantages that may cause differences between the results. One of its drawbacks is that the thermocouple is not in direct contact with the sample, which results in inaccuracy in temperature measurement of the sample. This is also another cause of thermal lag errors mentioned earlier.

It is seen that T_g values obtained from DSC are lower than those obtained from DMA and they are all lower than those obtained from TMA. Therefore, the increasing trend of the methods is DSC→DMA→TMA, which is similar to the trend presented for butadiene rubber by Sircar *et al.* [25].

Conclusions

A dilatometer and an expansion probe were used in a thermomechanical analyzer to detect the dual glass transition temperatures of constituent components of a 75:25 PC/ABS blend. The dilatometer probe measures volumetric changes while the expansion probe measures length changes. Using the statistical method of Analysis of Variance, it was found that heating and cooling rates affected both glass transition temperatures. However, the effect was not simple since T_g values can either increase or decrease depending on the scanning rate range studied. Generally, the duality of T_g's for the blend can be reliably detected with scanning rates ranging between 0.5 and 5°C min^{-1}. Thermal history removal treatment also changed the glass transition temperatures of the blend components – more so for PC than ABS. The results of expansion probe and dilatometer experiments were statistically equal only for heating/cooling rate of 0.5°C min^{-1}. In order to compare the results of the two modes, statistical Analysis of Covariance was used to account for sample mass difference. It was found that the different sample masses had minimal effect on the T_g value. When T_g results were compared with those obtained from DSC and DMA techniques, the glass transition temperatures obtained from TMA were shown to be greater than those of DMA and DSC.

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