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Full Length Research Paper

Similar dielectric behaviour of polyphenylglycidyl-ether and diglycidyl-ether of bisphenol-A at glass transition temperature

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In this work, the dynamics of the primary α- and the Johari-Goldstein (JG) β- processes are studied with dielectric spectroscopy. We found the relaxations at glass transition temperature in polyphenylglycidyl-ether and diglycidyl ether of bisphenol-A to be strongly correlated over a wide temperature *T* and pressure *P* range. Analysing the temperature and pressure behaviour of the α- and (JG) β-processes, a clear correlation has been found between the structural relaxation time, the Johari-Goldstein relaxation time and the dispersion of the structural relaxation.

Key words: Dielectric relaxation, nano-size motion, polyphenylglycidyl-ether (PPGE), diglycidyl-ether of bisphenol-A (DGEBA).

INTRODUCTION

In glass former materials close to the glassy state, the relaxation scenario evolves over many times decades, from very fast processes (β-processes) on the scale of nanoseconds to very slow ones (structural process) occurring on the scale of seconds (Sharifi, 2011b). On this respect, it is of fundamental importance to verify the universal occurrence of a non cooperative relaxation of the whole molecule before the cooperative one reflecting in the structural relaxation (Sharifi, 2011a). So, it is challenging to distinguish between intermolecular and intramolecular β-processes, also in systems with a complex molecular structure, even showing more than one β-process. Intermolecular β-processes that is observed in regid molecule called Johari-Goldstein β-processes (JG-processes).Among secondary relaxations, those originated by intra-molecular transitions naturally bear no relation to the structural relaxation. However, some relation may be expected for intermolecular secondary relaxations (Sharifi, 2011a), originated by local motions of the entire molecule. These processes,possibly relevant for the glass transition, are now referred to as the Johari-Goldstein (JG) secondary or JG β-relaxation to honor their discovery of secondary relaxation even in totally rigid molecules without internal degree of freedom. A rational of relaxation dynamics in such terms is for example proposed by the Coupling Model (Ngai, 2003). In this paper, we contribute to such discussion by investigating the relation between the structural and the JG β-relaxation in two systems, namely polyphenylglycidyl-ether (PPGE) and diglycidyl-ether of bisphenol-A (DGEBA), in an interval of pressure ranging from 0.1 to 600 MPa and temperature from 345 to 233K. A great advantage in such a study is provided by the possibility of studying the variation of relaxation dynamics with temperature and pressure. Variation of pressure acts on the density only of the systems while variation of temperature acts on the thermal energy and on the density. By controlling both thermodynamic parameters, it is possible to study the relaxation dynamics of the same system with different density and thermal energy values. In such conditions we can evidence any, if there exist a relation between the dynamics properties of the different relaxation processes.

EXPERIMENTAL

The complex dielectric constant $\varepsilon = \varepsilon' - i\varepsilon''$ was measured in the frequency range from 10^{-2} to 10^{7} Hz at different isothermal and

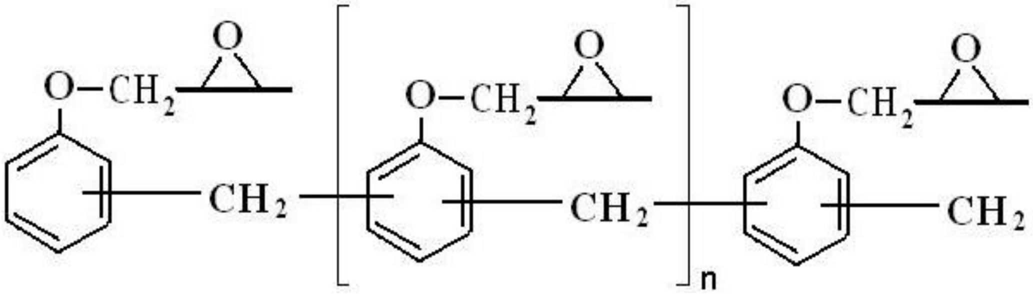

Figure 1. Chemical structure of poly[(phenyl glycidyl ether)-*co*-formaldehyde].

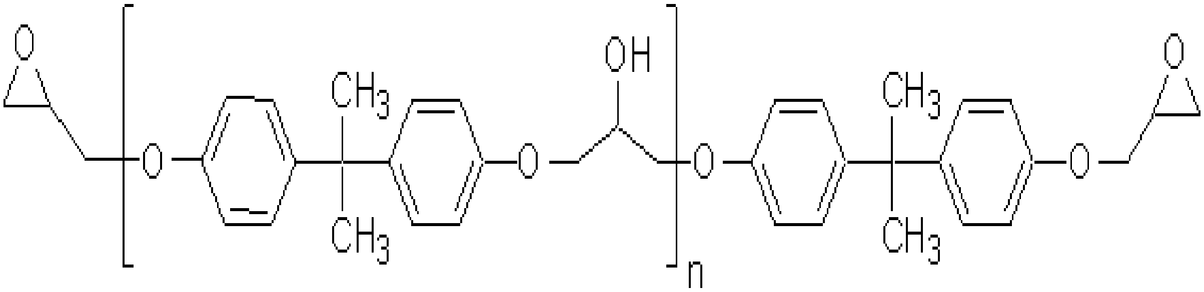

Figure 2. Chemical structure of poly(Bisphenol A-co-epichlorohydrin), glycidyl end-capped.

isobaric conditions using Novocontrol Alpha analyser. The temperature at atmospheric pressure was varied from 100 and 320K by means of a conditioned nitrogen flow cryostat. High pressure experiment was carried out by means of an hydrostatic press and silicon oil as a pressure transmitting medium. A Teflon membrane prevented the oil to contact the dielectric cell. The temperature of the whole pressure chamber was controlled by a thermal jacket connected to a liquid circulator. Poly[(phenyl glycidyl ether)-*co*-formaldehyde] (PPGE) with average molecular weight (MW) = 345 g/mol, T_g = 258 ± 1K, was purchased from Aldrich. Glassy PPGE shows two secondary processes: the slower being of JG type and the faster probably related to local motion of the epoxy subunits (Whalley et al., 1967; Buchenaii et al., 1984; Mosseri et al., 1984; Evstropyev et al., 1972; Anderson et al., 1972; Corezzi et al., 2002).

Poly(Bisphenol A-co-epichlorohydrin), glycidyl end-capped (DGEBA), with average molecular weight ~ 380 g/mol, corresponding to n = 0.14 in the chemical formula (Figures 1 and 2) and glass transition temperature 255 ± 1K was purchased from Shell Co. under the trade name of Epon 828. In the glassy state, two secondary processes are present and the slower one was identified as the JG process (Grzybowska et al., 2006; McKinney et al., 1976; Gunter et al., 2005; Madhusudan et al., 2007).

RESULTS

Dielectric loss spectra, $\varepsilon(\omega)=\varepsilon'-i\varepsilon''$ of PPGE and DGEBA were measured from above to below the glass transition. In Figure 3, the different thermodynamic paths are reported, along which we investigated the relation between the structural and the secondary relaxation close to $T_g(P)$. Dashed lines show isothermal paths and solid lines show isobaric paths followed during the vitrification. For all the systems investigated here, the secondary process was analysed only in the glassy state close and below Tg.

We acquired dielectric spectra along isothermal paths by varying pressure from 0.1 MPa up to the maximum value of 600 MPa, with step of 10 MPa in the supercooled liquid and step of 50 MPa in the glass (Figures 4 and 5). We also measured dielectric spectra by varying temperature at high pressure from 344.5 to 233K with step of 3K in supercooled liquid and 5K in the glass (Figures 4 and 5). The increase of pressure slows down the structural relaxation similarly to the decrease of temperature: the structural relaxation peak shifts to lower frequencies until the structure of the liquid is eventually arrested in a glassy state. At ambient pressure in PPGE and DGEBA only, the α- structural process is visible above T_g, when spectra are collected in the frequency interval 10^{-2} to 10^{6} Hz. In the case of PPGE and DGEBA, for temperatures below T_g, two further relaxation processes, β and γ are visible in the frequency interval (Figures 4 and 5). The entire observed relaxation processes move towards lower frequencies on decreasing temperature or increasing pressure, the α-process being the most sensitive and the γ-processes the less. The γ-process occurs only at very high frequency and in our frequency window, it can be measured only at

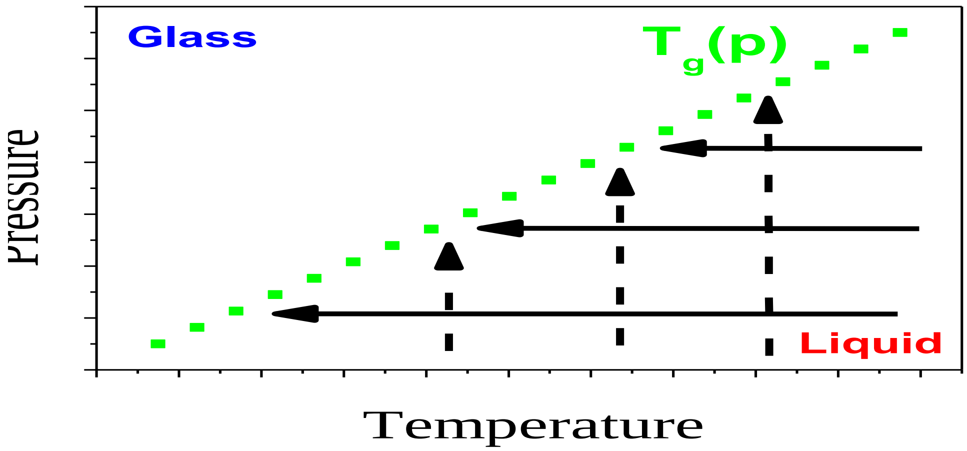

Figure 3. Schematic representation of the thermodynamic paths used during the measurements reported in this chapter. Squares (green points) represent the glass transition temperature at different pressure. Dashed and continuous lines represent isothermal and isobaric paths to the glass transition.

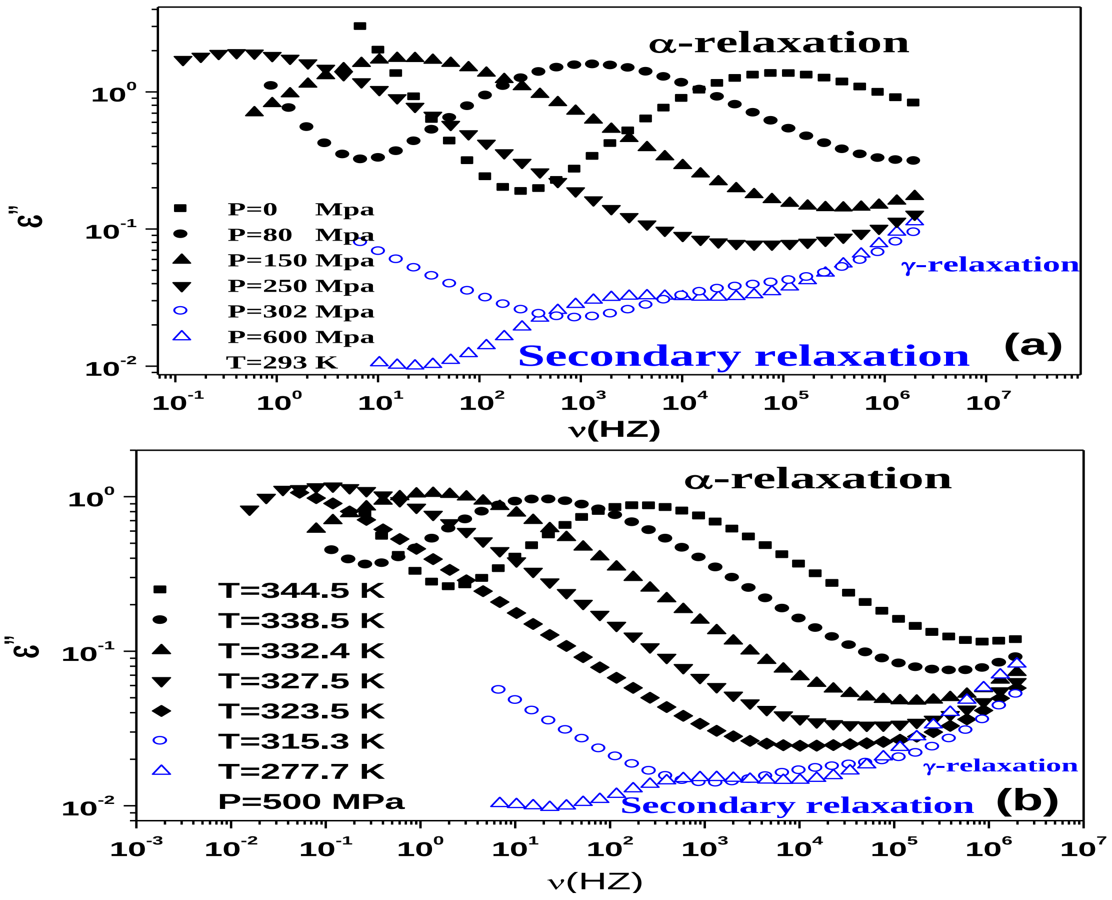

Figure 4. (a) Isothermal (293K) dielectric loss spectra of PPGE, measured at different pressures (reported in the figure). (b) Isobaric (500 MPa) dielectric loss spectra of PPGE, measured at different temperatures (reported in the figure). The black spectra are measured in the supercooled liquid and are dominated by the α-peak and the blue open symbols spectra are measured in the glassy state.

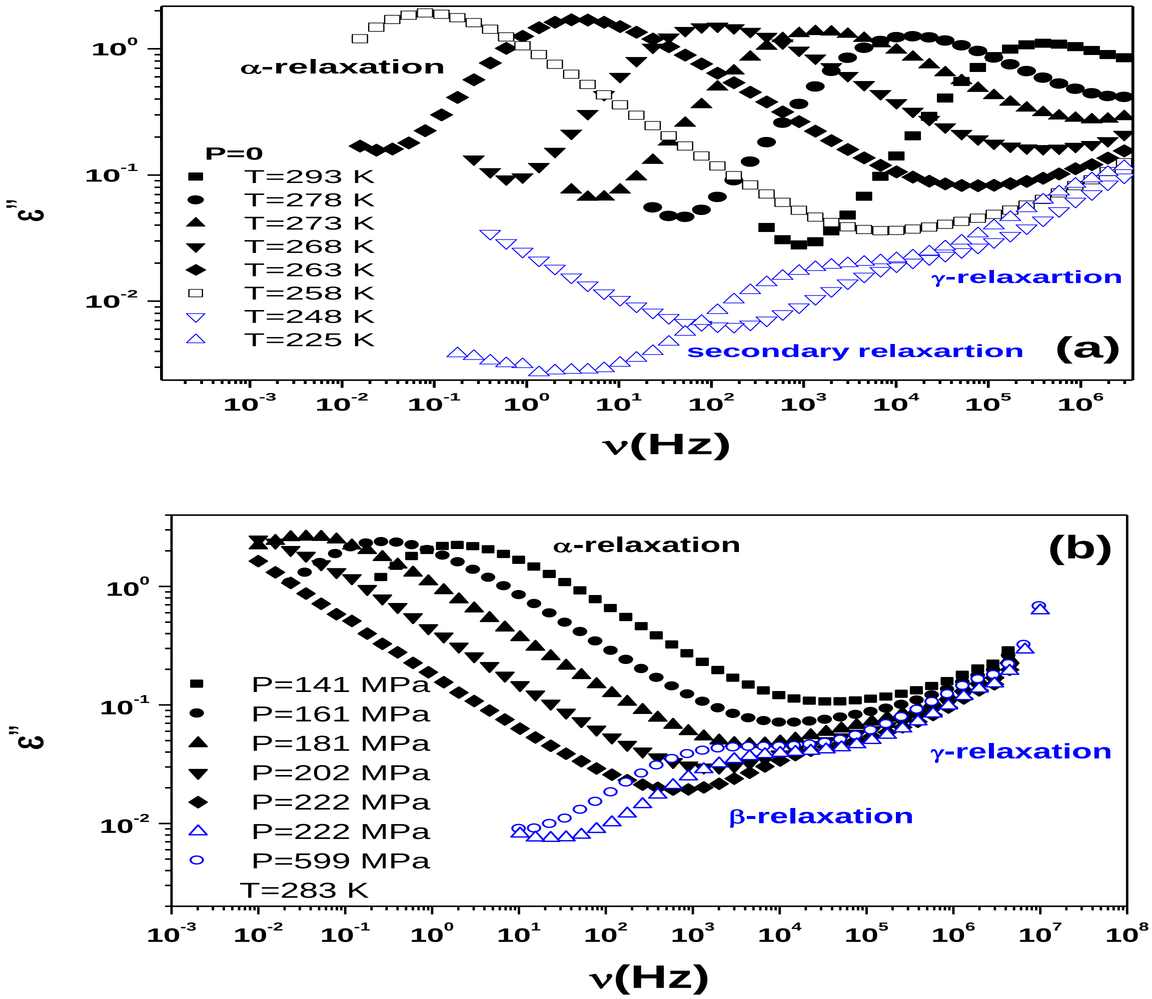

Figure 5. (a) Isobaric (0.1 MPa) dielectric loss spectra of DGEBA measured at different temperatures (values reported in the figure). (b) Isothermal (283K) dielectric loss spectra of DGEBA measured at different pressure (values reported in the figure). The black spectra are measured in the supercooled liquid and are dominated by the α-peak and the blue open symbols spectra are measured in the glassy state.

very low temperature at ambient pressure, but never at high pressure due to limitation of the temperature interval (Figures 4 and 5). Dielectric spectra were fitted with a superposition of different Havriliak–Negami (HN) functions, one for each relaxation process (Sharifi, 2011a):

$$\varepsilon^{*}_{HN}(\omega)-\varepsilon_{\infty}=\frac{(\Delta\varepsilon)}{\left[1+(i\omega\tau_{HN})^{1-\alpha_{HN}}\right]^{\beta_{HN}}} \qquad (1)$$

where the shape parameters α_{HN}, β_{HN} $(0<(1-\alpha_{HN});\ \beta_{HN}(1-\alpha_{HN})\leq 1)$ describe the symmetric and asymmetric broadening of the complex dielectric function.

Since for any value of temperature, not more than two relaxation processes appear; in the fitting procedure, we never use more than two relaxation functions. Moreover, the β-process was usually described by the CC function (β=1), and the contribution of the γ-process at high pressure (for PPGE and DGEBA), was accounted for by an HN function with only $\Delta\varepsilon_{\gamma}$ and τ_{γ} as free parameters, and the α_{γ} and β_{γ} parameters fixed to the values found at ambient pressure and very low temperature. The fitting program, based on a least square minimizing procedure, contemporary fits the real and the imaginary part of the dielectric spectrum and furnishes the values of the parameters and the corresponding errors. Tables 1 and 2, report some representative values of the parameters α and β for the considered systems at different pressure

Table 1. Parameters of superposition of two HN functions of α-(structural) and β-(secondary) relaxation at different pressures and temperatures for PPGE.

P (MPa)	T (K)	α - relaxation		β - relaxation	
		α_{HN}	β_{HN}	α_{HN}	β_{HN}
0	293	0.35	0.49	0	0
76	293	0.36	0.60	0	0
150	293	0.39	0.67	0	0
302	293	0	0	0.59	1.0
600	293	0	0	0.67	1.0
500	338	0.47	0.48	0	0
500	335	0.49	0.44	0	0
500	332	0.49	0.4	0	0
500	315	0	0	0.62	1.0
500	277	0	0	0.54	1.0

Table 2. Parameters of superposition of two HN functions of α-(structural), β-(secondary) relaxation at different pressures and temperatures for DGEBA.

P (MPa)	T (K)	α -relaxation		β - relaxation	
		α_{HN}	β_{HN}	α_{HN}	β_{HN}
240	293	0	0	0.6	1
270	293	0	0	0.59	1
300	293	0	0	0.48	1
340	293	0	0	0.43	1
141	283	0	0	0.51	1
244	283	0	0	0.47	1
303	283	0	0	0.44	1
450	283	0	0	0.42	1
0	273	0.15	0.43	0	0
0	198	0.4	0.5	0.64	1

and temperature.

The structural relaxation times for PPGE were investigated near Tg over a wide range of thermodynamic variables (for example, pressure, temperature and volume) (Paluch et al., 2002). This study showed that temperature is not the dominant control variable for structural relaxation. Broadband dielectric spectroscopy, heat capacity spectroscopy and viscosimetry were used to study PPGE at ambient pressure. In spite of their rather simple molecular structure, the dynamics of these systems are characterized by two well-separated crossover regions where the relaxation times of structural relaxation and the two secondary relaxations β and γ approach each other (Paluch et al., 2002). The structural relaxation in DGEBA was examined with three spectroscopic methods: dielectric spectroscopy (DS), dynamic light scattering-photon correlation spectroscopy (LS) and mechanical spectroscopy (MS) (Paluch and Fisher, 2000; Comez et al., 1999; Paluch et al., 2003).

The DS and LS measurements were carried out as a function of both temperature and pressure. Moreover, pressure-volume-temperature measurements were obtained for the DGEBA. These data allow an assessment of the relative contributions of thermal energy and free volume to structural relaxation in DGEBA. The results clearly show a substantial role for both thermal and free volume in the dramatic slowing down of the dynamics. The combined temperature- and pressure-dependences of the dielectric and light scattering relaxation times were analyzed using the Avramov equation, showing that the fragility (normalized temperature dependence) is pressure independent over the studied range of pressures. Conformance to the time-temperature-pressure superposition principle was also observed for all measurement techniques. As model independent parameter for the characteristic time scale of the process, we considered the maxima of frequency,

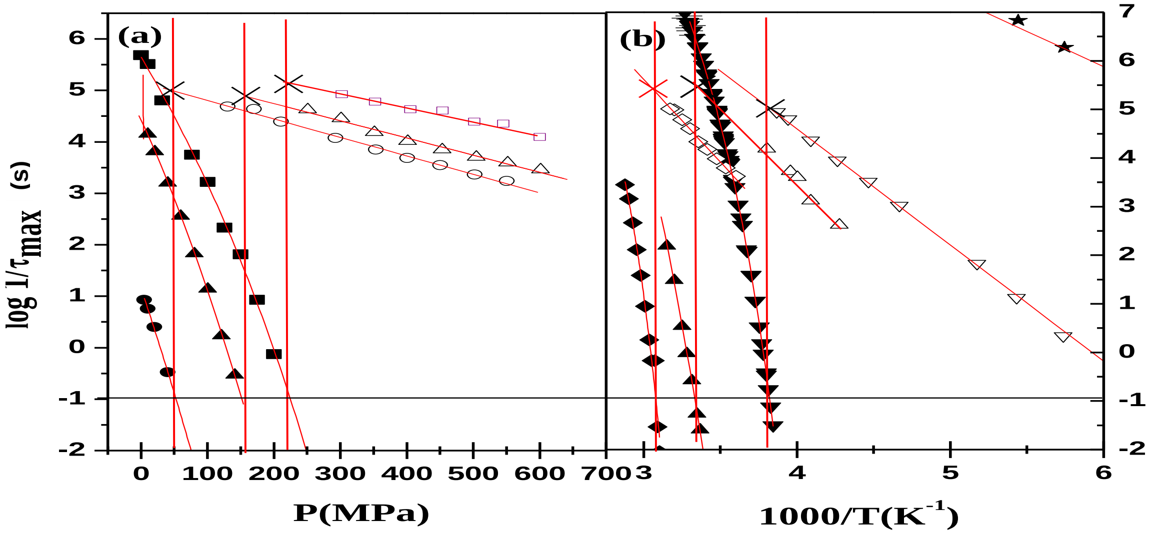

Figure 6. (a) Pressure dependence of the logarithmic of τ_{max} for the α- (full symbols) and the β-process (open symbols), at three different values of T: 293K (squares), 283K (up triangle) and 267.6K (circles) of PPGE. (b) Temperature dependence of the logarithmic of τ_{max} for the α- (full symbols) and the β-process (open symbols), at three different values of pressure: 0.1 MPa (triangles), 268 MPa (up triangle) and 500 MPa (diamonds) of PPGE. In both panels the continuous lines represent fit with the VFT (α- relaxation) and Arrhenius (β-relaxation) equations. The horizontal dotted line show the relaxation time value used to define the glass transition. Crosses represent the values of τ^{β}_{max} at $(T,P)_g$.

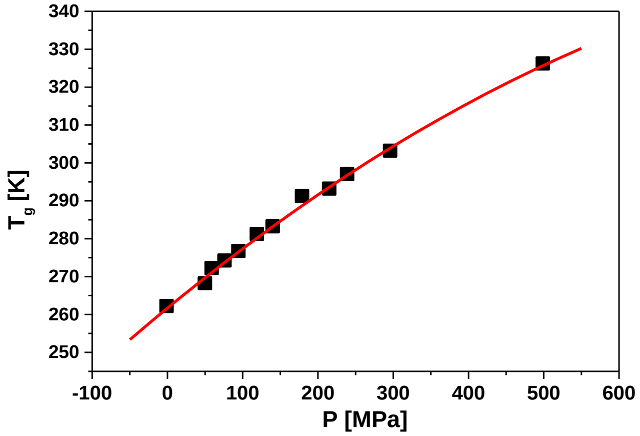

Figure 7. Values (symbols) of T_g as a function of P for PPGE. The continuous line is a fit with a second order polynomial.

$\nu^i_{max}=1/(2\pi\tau^i_{max})$ corresponding to the loss peak maximum frequency, which was calculated by:

$$\frac{1}{\tau^i_{\max}} = 1/\tau_i * \{\sin[((1-\alpha_{HN})*\pi/(2+2*\beta_{HN})]\}^{1/(1-\alpha_{HN})} * \{\sin[((1-\alpha_{HN})*\beta_{HN}*\pi)/(2+2*\beta_{HN})]\}^{-1/(1-\alpha_{HN})} \qquad (1)$$

where the symbols have the same meaning as in Equation 1. Those results of relaxation time as function of temperature and pressure are presented in Figures 6 and 8. The value of the glass transition temperature, T_g, determined by $\tau_{\alpha}(T_g)=10$ s at ambient temperature is (262 ± 1K) for PPGE, (257 ± 1K) for DGEBA. The pressure dependence of T_g can be expressed by polynomial function, $T_g(P) = 261.7 + 0.164*P-7*10^{-5}*P^2$ K for PPGE, Figure 7, $T_g(P) = 259.89 + 0.1*P-5.42*10^{-5}*P^2$ K for DGEBA (Figure 9).

The temperature dependence of υ^{α}_{max} can be well fitted by a Vogel-Fulcher-Tammann (VFT), equation, $\upsilon^{\alpha}_{max} = \upsilon^{\alpha}_0\exp(DT_0/(T-T_0))$ over the entire temperature interval. The pressure dependence of υ^{α}_{max} can be well fitted by a Vogel-Fulcher-Tammann like, PVFT equation, $\log(\upsilon_{max}) = \log(\upsilon_{0max})+[A*P/(B-P)]$ over the entire pressure interval. In the isothermal paths, pressure dependence of υ^{α}_{max} below T_g are well reproduced by the Arrhenius equation, $\upsilon_{max} = \upsilon_0\exp(-P.\Delta V_{\beta}/k_BT)$ (where ΔV_{β} is the activation volume of the secondary β-relaxation) and in isobaric condition the temperature dependence of υ^{β}_{max} below T_g is well reproduced by the Arrhenius equation, $\upsilon^{\beta}_{max} = \upsilon^{\beta}_0\exp(-E_a/k_BT)$. The different VFT and Arrhenius parameters for the isobaric dielectric relaxation spectra of the differences are listed in Table 3.

DISCUSSION

The combined variation of both temperature and

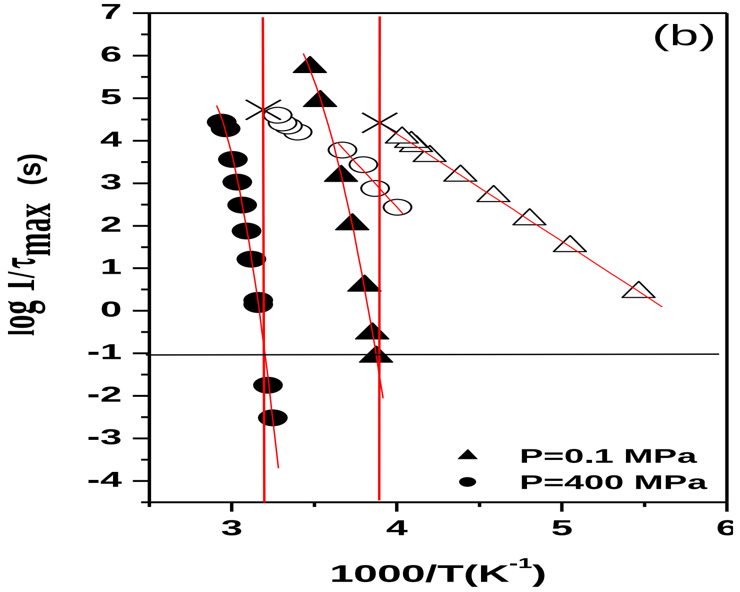

Figure 8. (a) Pressure dependence of the logarithmic of τ_{max} of DGEBA for the α-(full symbols) and the β(Secondary)-process (open symbols), at two different values of T: 293K (squares) and 283 K (circles). (b) Temperature dependence of the logarithmic of τ^{β}_{max} of DGEBA for the α-(full symbols) and the β(Secondary)- process (open symbols), at two different values of pressure: 0.1 MPa (triangles), and 500 MPa (circles). In both panels the continuous lines represent fit with the VFT (α-relaxation) and Arrhenius (β-relaxation) equations. The horizontal continuous line show the relaxation time value used to define the glass transition. Crosses represent the values of τ^{β}_{max} at $(T,P)_g$.

Table 3. The glass transition temperature and the relevant VFT parameter for three different systems, as determined by dielectric relaxation measurement.

	P (MPa)	T (K)	$\log(\upsilon_{0\alpha})$	DT_0 (K)	$T_g(P)$ (K)	$\log(\upsilon_{0max})$	A	B (MPa)	$P_g(T)$ (MPa)
PPGE	#	293	4.8	#	293 ± 1	#	18.6	854	214.5 ± 0.1
	#	283	3.6	#	283 ± 1	#	23	793	132.8 ± 0.1
	#	268	0.2	#	223 ± 1	#	4.8	165	34.3 ± 0.1
	0.1	#	#	3.3	263 ± 1	14.4	#	#	0.1 ± 0.1
	298	#	#	2.6	303 ± 1	12.1	#	#	298 ± 0.1
	500	#	#	1.2	327 ± 1	9.1	#	#	500 ± 0.1
DGEBA	#	293	6.1	15.2	293 ± 1	#	#	#	244 ± 0.1
	#	283	5.0	26.5	283 ± 1	#	#	#	174.3 ± 0.1
	0.1	#	#	#	258 ± 1	2.6	2.6	3.76	0.1 ± 0.1
	400	#	#	#	312 ± 1	3.57	3.57	4.69	400 ± 0.1

Parameter of A and B comes from the equation, $\log(\upsilon_{max}) = \log(\upsilon_{0max}) + A \cdot P/(B-P)$.

pressure allows dynamic states characterized by the same value of structural relaxation time to be reached, but different thermal energies and densities. The relative role of density and thermal energy on the slowing the structural relaxation on approaching the glass transition is a matter of study. Even if a similar behavior can be found for materials belonging to the same class, the details of their relative role vary for each system.

From the dynamic point of view the glass transition is traditionally defined by considering the structural relaxation time being a fixed long value. According to our definition $\tau_{\alpha}(T_g)$ = 10s and the glass transition can be obtained for different couples of values of pressure and temperature, whose relation in our case is expressed by

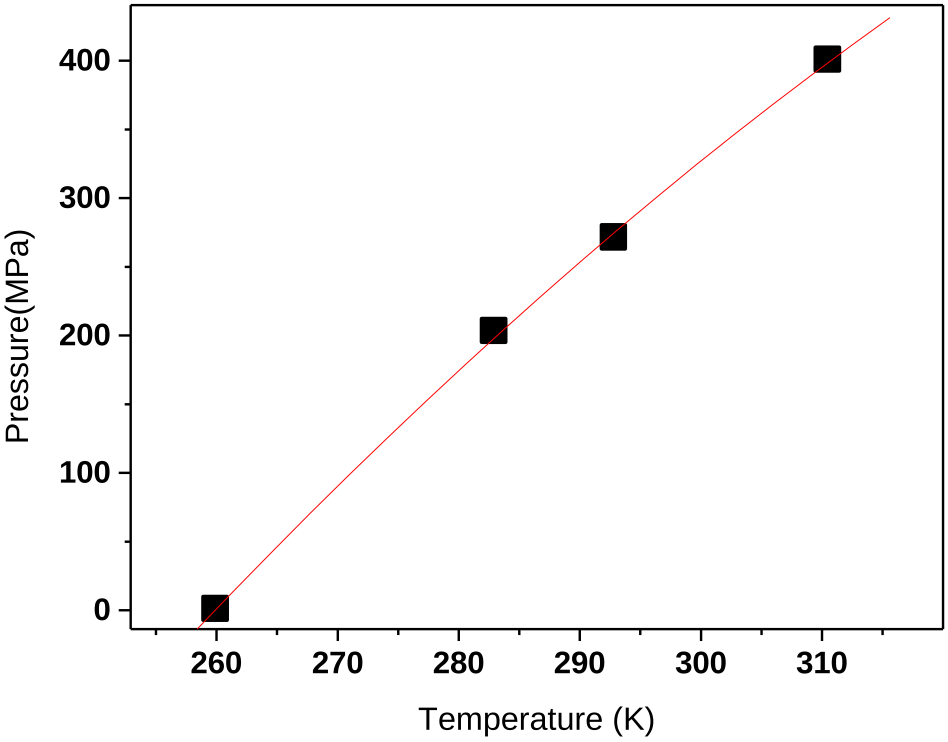

Figure 9. Values (symbols) of T_g as a function of P for DGEBA. The continuous line is a fit with a second order polynomial.

polynomials function for pressure values up to 500 MPa (Figures 7 and 9).

It is interesting to deepen the investigation about the β-JG process, also in systems with complex molecular structure, and its eventual connection with the glass formation phenomenon. Since one of the most prominent aspects related to the glass formation is of dynamic nature, it seems reasonable to find out a dynamic relation between the two processes. If we scrutinize our systems, the behaviour of the β-relaxation for different values of $(T,P)_g$ but the same value τ_α, for example at the glass transition, it appears that the values of τ_{max} is also almost constant (Figures 6 and 8). In fact, for PPGE and DGEBA we observe a maximum variation of τ_{max} of less than half a decade for all the different temperature and pressure conditions. In other words, locating the glass transition line (Tg,Pg) as isochronal locus of the structural process, irrespective of the fact that the conditions of temperature and of pressure are different, the ratio of $\tau_\alpha(P,T)/\tau_\beta(P,T)$ is constant.

We know that pressure and temperature act to a different extent on the structural dynamics. The same is also for the β-process, and a limiting case is represented by those processes that are almost insensitive to pressure variations, but are temperature dependent (Zorn et al., 1995; Tyagi et al., 2007; Alegr et al., 1997). The evidence that at the glass transition, the α and β time scales are correlated suggesting that the dependence of the characteristic times of the two processes on the external parameters must be correlated. This result is compatible with the hypothesis of the complex relaxation scenario as a continuous evolution from localized (almost non cooperative) to fully cooperative motions, both involving the motion of the whole molecule.

It is likely to suppose that the connection between α and β-dynamics is not a universal feature of all β-processes, but only of those local processes intimately connected to the cooperative structural dynamics. PPGE and DGEBA present two β-processes, and it would be important to check this idea not only on the slower β-process but also on the faster γ-process. However, since the experimental limitation of the γ-process was not measured at high pressure, such test cannot be done. However, to support the idea that such type of investigation can distinguish β-processes with a particular origin, we can refer to data of decahydroisoquinoline (Alegr et al., 1997). Different molecular motions are at the basis of the β-process in PPGE and decahydroisoquinoline.The effect of thermodynamic histroy on glass transition was observed in the ionic liquid, for example, 1-Methyl-3-trimethylsilylmethylimidazolium Tetrafluoroborate (Jarosz et al., 2011) and mixture systems (Kessairi et al., 2008) and at PVAc (Sharifi and Jahanbakhsh, 2011).

Conclusions

We investigated the relation between secondary and structural dynamics of PPGE and DGEBA under variations of temperature and pressure.

We propose a model independent analysis for scrutinizing a possible relation between structural and secondary relaxations. The analysis consists in investigating the ratio τ_α/τ_β for different values of pressure and temperature (density and thermal energy), but the same value of structural relaxation time. According to such analysis, we evidenced that the β-process in PPGE and DGEBA is clearly related with the structural dynamics, and then to the glass transition. In fact, for different thermodynamic conditions of pressure and temperature corresponding to $\tau^{\alpha}_{max}\approx 10$ s for PPGE and DGEBA, we observe that the relaxation time of the β-process is almost constant. This results is fully consistent with those contemporarily found for mixtures of glass formers materials.

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