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Dielectric relaxation of liquid crystalline dendrimers and liquid crystalline polymers with pendant nitro groups

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Abstract

The dielectric relaxation was determined in a time-domain spectrometer between 100 and 295 K and from 0.1 to 1000 Hz for series of dendritic liquid crystalline polymers with scaffolds based on 2,2-bis(hydroxymethyl)propionic acid (*bis*-MPA) of generations 0–3 with 4″-((*R*)-1-methylheptyloxy)phenyl 4-{4′-[10-(hydroxy-carbonyl)decyloxy]-phenyl} benzoate as mesogen and for the second generation of dendrimers with 4″-[10-(hydroxycarbonyl)decyloxy]-biphenyl 4-[4′-(2-(*R*)-octyloxy)-3′-nitrophenyl] benzoate as mesogen. Dielectric data are also reported for a side-chain liquid crystalline polymer based on {4″-[(*R*)-(−)-2-octyloxy]-3″-nitro-phenyl 4-(4′-(11-acryloyl-oxyundecyloxy)phenyl}benzoate. The low temperature subglass process (γ) assigned to the spacer group was essentially insensitive to molecular architecture and to the presence of a pendant nitro group in the mesogen. The high temperature sub-glass process (β) assigned to a 180° flip of part of mesogen was only moderately sensitive to the molecular architecture. The introduction of a pendant nitro group in the mesogen led to a substantial increase in both activation energy and relaxation strength. The results suggest that the phenylene ring with the pendant nitro group participates in the β process. © 2001 Published by Elsevier Science Ltd.

Keywords: Dielectric relaxation; Liquid crystalline polymers; Dendrimers

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1. Introduction

Materials with second-order non-linear optical properties based on liquid crystalline polymers with non-linear optical chromophores in the mesogens may have different molecular architectures, e.g. side-chain polymers, networks with mesogens as side-chains or, as discovered more recently, dendrimers with mosogens as terminal groups. Chiral liquid crystalline monomers with smectic C^* mesomorphism were surface-stabilised, poled and photo-polymerised to form stable non-linear optical materials [1–5]. The pendant dipoles (e.g. nitro groups attached to a phenylene ring in the mesogen) in the parallel orientation are a prerequisite for obtaining second-order non-linear optical properties. It is essential to know the segmental mobility of the mesogenic unit because a decrease in the orientation of the dipoles will lead to a decay in the second-order non-linear optical susceptibility. Dielectric spectroscopy is one of the few methods capable of capturing these features.

The dielectric relaxation of side-chain polymers, including polyacrylates, polymethacrylates, polysiloxanes and polyvinylethers, has been extensively studied [6–20]. An excellent review of the dielectric relaxations of liquid–crystalline polymers is given by Simon [21]. These polymers exhibit five dielectric relaxation processes: the glass–rubber transition, α, three subglass processes referred to as β, γ and δ′ and a high temperature process also often denoted δ. The low temperature subglass process (δ′) has been assigned to torsion about bonds in the tail group [9,18], and the γ process is assigned to local motions in the spacer group [9,18]. The β process was assigned to the reorientation of dipoles in the mesogen, e.g. the ester group in phenylbenzoate unit [9,10,18,19]. The high temperature process denoted δ involves a reorientation of the entire side-group by rotation about the backbone chain [11–13,16,17]. Side-chain and network liquid–crystalline polyacrylates with a pendant nitro group displayed only a slight indication of the high temperature subglass (β) process [20]. This is particularly important for the stability of the second-order non-linear optical susceptibility. The polar order would decay with time by the β process, i.e. by coordinated torsions about σ bonds near or in the mesogens causing reorientation of the pendant dipoles of the mesogens.

This paper presents new dielectric data that clearly reveals the β process in a side-chain liquid–crystalline

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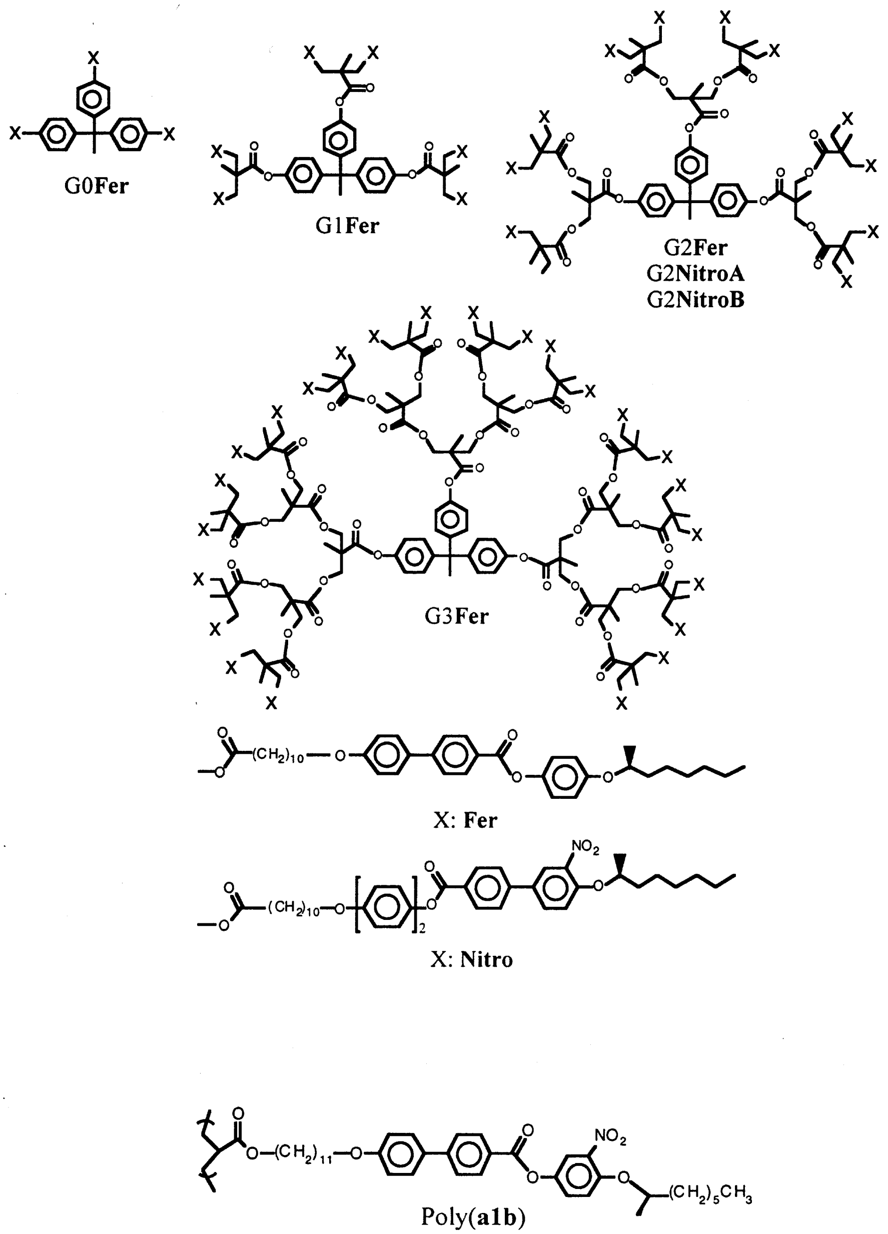

Fig. 1. Structure of polymers studied. The terminal groups (denoted X) of the dendrimers were either Fer or Nitro. Poly(**a1b**) is a side-chain liquid–crystalline polymer.

polyacrylate with a pendant nitro group in the mesogen. Dielectric data for dendrimers of different generations with terminal mesogens with and without pendant nitro groups in the mesogens are also presented, and these data substantiate the important role of the pendant nitro group.

2. Experimental

2.1. Materials

The structures of the studied polymers are shown in Fig. 1. The dielectric cells were filled with the heated polymer liquids (above the isotropisation temperature) using capillary forces, and were cooled at a rate of 2 K min^{-1} to 298 K while being subjected to a 10 MV m^{-1} electric field. Polarized light microscopy confirmed that the electric field aligned the smectic C* structure of the liquid–crystalline dendrimers (G0Fer, G1Fer, G2Fer, G3Fer and G2Nitro), whereas the side-chain liquid crystalline polymer [poly(**a1b**)] regained its smectic C* helical structure after removal of the electrical field. Details about the synthesis of the polymers have been reported earlier [22,23]. The glass transition temperatures of the dendrimers have been reported earlier [24]: G0Fer: 321 K; G1Fer: 330 K; G2Fer: 333 K; G3Fer: 337 K; G2Nitro: 343 K. Poly(**a1b**)

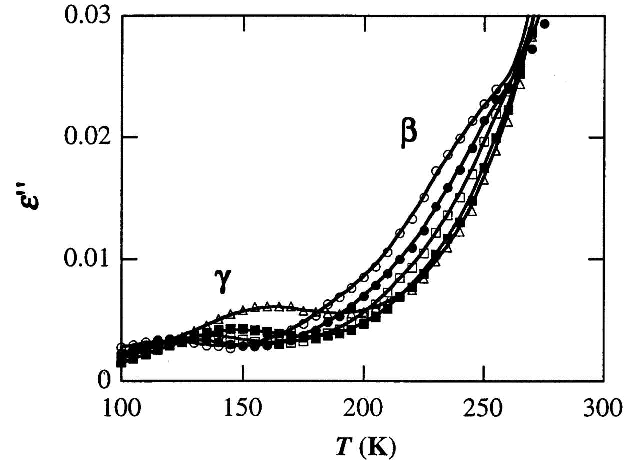

Fig. 2. Imaginary part of the dielectric permittivity (ϵ'') as a function of temperature for poly(**a1b**). Isochronous curves at the following frequencies: ○ 0.1 Hz; ● 1 Hz; □ 10 Hz; ■ 100 Hz; △ 1000 Hz.

showed a weak glass transition at 300–310 K according to DSC.

Thermal transition data in the dendrimers have been reported by Busson et al. [22–26] and the polymers are in the following states at the temperature range studied by the dielectric measurements: poly(**a1b**): smectic C*;G0Fer, G1Fer, G2Fer and G3Fer showed tilted crystalline structure; G2Nitro: smectic C*.

2.2. Dielectric measurements

The dielectric measurements were made on 4 or 10 μm thick samples with planar dimensions of 10 × 10 mm. The samples were held between two glass plates coated with conductive indium–tin-oxide. Rubbed polyimide films of submicron thickness were placed between the conductive indium–tin-oxide layers and the polymer (Y type electric cell, E.H.C. Co. Ltd, Japan). The indium–tin-oxide layers shows a dielectric loss peak at high frequencies, $>10^4$ Hz [27]. This is however outside the frequency range of the data reported in this paper. The polyimide film showed no measurable dielectric loss in the temperature and frequency ranges used in this study. The dielectric apparatus was an IMASS TDS time domain spectrometer equipped with a Hewlett Packard Series 300 computer and a Lakeshore cryostat (330 Autotuning Temperature Controller). The time domain spectrometer is based on a design by Mopsik [28]. At time $t = 0$, a step voltage of 10 or 100 V was applied between the indium–tin-oxide layers. This induced a charge $Q(t)$ to flow through the sample and the complex capacitance was obtained as a function of frequency by a numerical Laplace transform, based on a cubic spline, of the time domain capacitance data. The latter covered a time period of 1 min and frequency domain data were obtained from 10^{-2} Hz to 10 kHz. High quality data were in this case obtained only between 0.1 and 1000 Hz. The samples were cooled to 100 K and then heated while measurements were made at progressively higher temperatures. Temperature equilibrium was established during a 30 min rest period prior to each measurement. The measurement was repeated 16 times at each temperature and an average dielectric permittivity was obtained at each frequency and temperature.

3. Results and discussion

3.1. General features of the dielectric data

The dielectric data were represented in terms of the Havriliak–Negami equation [29,30]:

$$\epsilon^* = \epsilon_u + \frac{\epsilon_r - \epsilon_u}{(1 + (i\omega\tau_0)^\alpha)^\beta} \qquad (1)$$

where ϵ^* is the complex dielectric permittivity at the radian frequency ω, ϵ_u and ϵ_r are the unrelaxed (at high frequency) and relaxed (at low frequency) dielectric permittivies, τ_0 is the central relaxation time, α is the symmetric broadening factor and β is the asymmetric broadening factor. The dielectric data showed only symmetrical broadening, i.e. $\beta = 1$, which is demonstrated by the circular arcs in the Argand diagrams (real part of the dielectric permittivity (ϵ') as a function of the imaginary part of the dielectric permittivity (ϵ'')) presented in Sections 3.2 and 3.4. The dielectric data thus conformed to a special case of the general Havriliak–Negami equation ($\beta = 1$), namely the Cole–Cole equation [31] with only one broadening factor (α). The dielectric relaxation processes were at each temperature (T) characterised by the following quantities: ϵ_u, $\Delta\epsilon = \epsilon_r - \epsilon_u$ ('the relaxation strength'), α and the frequency ($f_{max} = \omega/2\pi$) associated with the maximum in the dielectric loss (ϵ''). These quantities were obtained by fitting Eq. (1) to the experimental isothermal dielectric data; ϵ_u and ϵ_r were obtained as the intersections of the circular arc with the ϵ' axis in the Argand diagram. Only the part of the complex plane data corresponding to frequencies between 0.1–0.7 Hz (minimum) and 300–700 Hz (maximum) were used for the fitting of the Havriliak–Negami equation (see Figs. 3, 4 and 11). The temperature dependence of f_{max} in all the studied relaxation processes was in accordance with the Arrhenius law, i.e.:

$$f_{max} = A\, e^{-\frac{\Delta E}{RT}} \qquad (2)$$

where A is a constant, ΔE is the activation energy and R is the gas constant. The validity of the Arrhenius law is demonstrated in Sections 3.2–3.4. This is an expected result because only sub-glass processes were observed [32] and it is in accordance with earlier reported data on other and similar liquid–crystalline polymers [6–21].

3.2. Poly(*a1b*)

Fig. 2 shows isochronous curves for the dielectric loss as a function of temperature. Two dielectric relaxation processes are clearly visible: the γ process at low temperatures

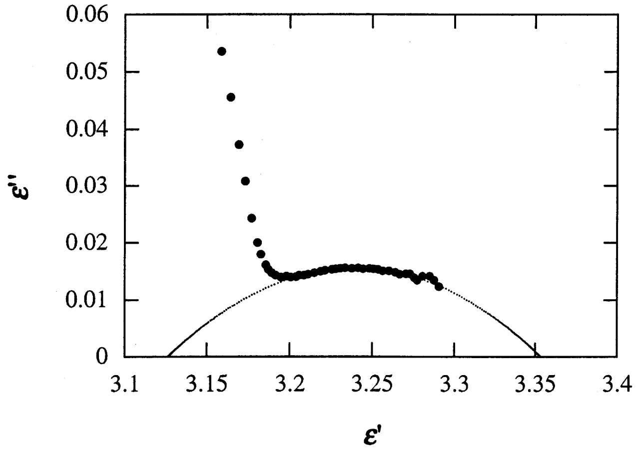

Fig. 3. Argand diagram of the γ process at 125 K for poly(**a1b**).

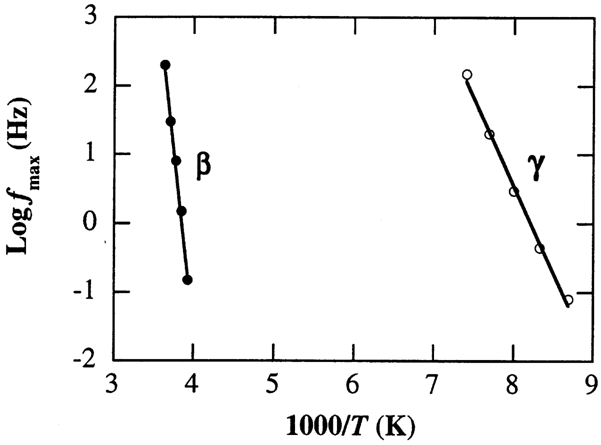

Fig. 5. Temperature dependence of the dielectric relaxation processes in poly(**a1b**).

and the β process at higher temperatures. Both these relaxation processes were symmetrically broadened as is clearly seen in the Argand diagrams (Figs. 3 and 4). The γ process exhibited from 115 to 135 K constant values of the following quantities (average ± standard deviation): $\alpha = 0.163 \pm 0.007$; $\Delta\epsilon = \epsilon_r - \epsilon_u = 0.246 \pm 0.021$; $\epsilon_u = 3.11 \pm 0.02$. The β process showed the following characteristics between 255 and 275 K: (average ± standard deviation): $\alpha = 0.134 \pm 0.009$; $\epsilon_u = 2.97 \pm 0.07$; $\Delta\epsilon$ increased from 2.0 at 255 K to 2.4 at 275 K.

The frequency shift of the dielectric loss peak with temperature obeyed the Arrhenius law (Fig. 5) with the following activation energies: 57 (γ) and 240 kJ mol^{-1} (β). The β process had a higher activation energy in poly(**a1b**) than in liquid crystalline polymers with a similar structure, but without the pendant nitro group; typical values for polymers with phenylbenzoate units are 60–100 kJ mol^{-1} [18–20]. It is known that the β process involves a 180° rotation of the ester dipole in the mesogen [18–20] and the high activation energy found for the polymer with pendant nitro group suggests that the phenylene ring with pendant nitro group is involved in the β process. This supposition is further substantiated by the high relaxation strength of the β process for poly(**a1b**), 2.0–2.4; the corresponding values for polymers with phenylbenzoate units are 0.3–1.4 [18]. The flip of the phenylene ring with the pendant nitro group involves a significant swept-out volume and it is suggested that a large part of the activation energy is of inter molecular origin. A close examination of dielectric data for poly(**a1b**) and for polymers with phenylbenzoate units [18] shows that the β process appeared at 20–30 K higher temperatures in poly(**a1b**).

It may be concluded that, by introducing a pendant nitro group in the mesogen, the β process becomes more intense, because it involves both ester and nitro groups. Furthermore, the β process is shifted towards higher temperatures and increased in activation energy because the pendant nitro group increases the swept-out volume involved in the 180° flip.

3.3. GnFer polymers

Figs. 6–8 shows the imaginary part of the dielectric

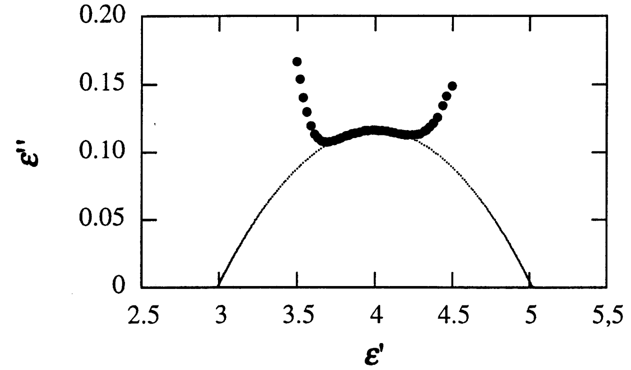

Fig. 4. Argand diagram of the β process at 265 K for poly(**a1b**).

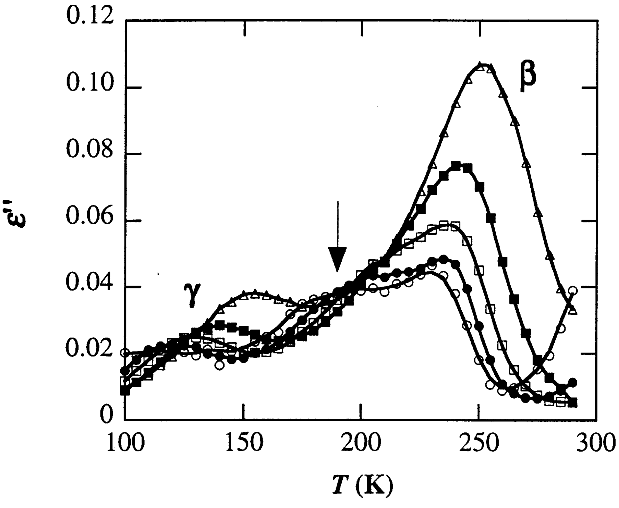

Fig. 6. Imaginary part of the dielectric permittivity (ϵ'') as a function of temperature for G0Fer). Isochronous curves at the following frequencies: ○ 0.1 Hz; ● 1 Hz; □ 10 Hz; ■ 100 Hz; △ 1000 Hz.

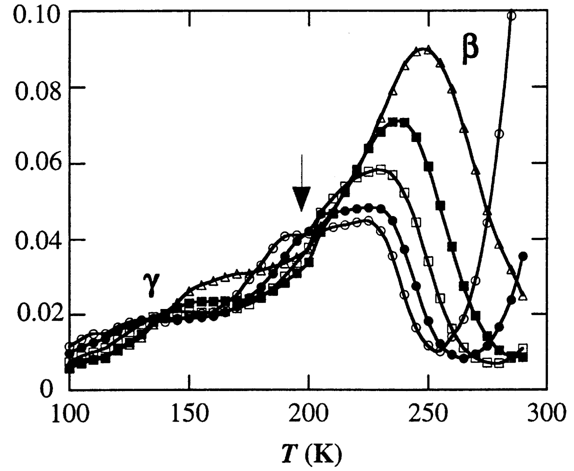

Fig. 7. Imaginary part of the dielectric permittivity (ϵ'') as a function of temperature for G1Fer). Isochronous curves at the following frequencies: ○ 0.1 Hz; ● 1 Hz; □ 10 Hz; ■ 100 Hz; △ 1000 Hz.

permittivity as a function of temperature for three generations of the dendritic liquid–crystalline polymers. All three polymers clearly displayed γ and β processes. The low frequency curves (0.1 and 1 Hz) showed a broadening of the β peak towards lower temperatures. A shoulder at ~200 K appeared at these frequencies. Malmström et al. [33] found a sub-glass processes in this temperature–frequency range in hyperbranched polymer with the same branch unit (based on 2,2-dimethylolpropionic acid, *bis*-MPA) as used in the GnFer polymers, but this process was assigned to the terminal hydroxyl groups not present in GnFer. Furthermore, G0Fer which contained no *bis*-MPA branch units exhibited exactly the same shoulder as the polymers of higher generation with the *bis*-MPA branch units. At present, we have no good explanation of the shoulder appearing at 200 K.

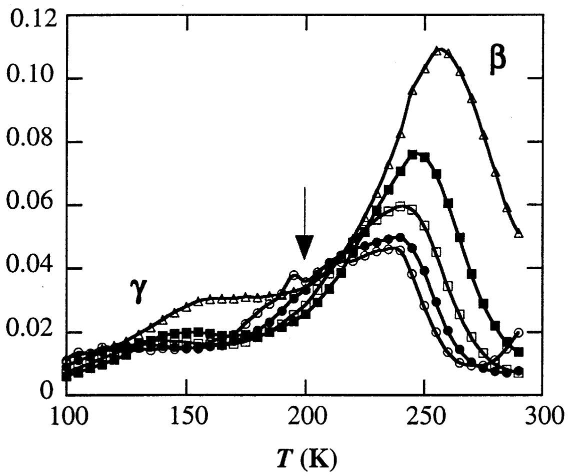

Fig. 8. Imaginary part of the dielectric permittivity (ϵ'') as a function of temperature for G3Fer). Isochronous curves at the following frequencies: ○ 0.1 Hz; ● 1 Hz; □ 10 Hz; ■ 100 Hz; △ 1000 Hz.

Malmström et al. [33] found a high-temperature sub-glass process in hyperbranched polyesters assigned to reorientation of the dipoles in the *bis*-MPA branch units. This process appeared at 260 K and 0.1 Hz for a series of hyperbranched polymers with the same branch unit and different terminal groups: hydroxyl, acetate and benzoate groups [33]. The relaxation strength of this process for the hyperbranched polymers with bulkier terminal groups (acetate and benzoate) was less than 0.1. It may thus be concluded that the influence of the dendritic part (i.e. *bis*-MPA branch units) on the dielectric relaxation of the GnFer polymers should be negligible.

Fig. 9 and Table 1 show that the activation energies of the γ process were approximately the same for the dendrimers of different generation. The γ process for the zero generation polymer (G0Fer) was shifted towards a lower temperature than for the higher generation polymers (Fig. 9). The activation energy of the β process increased with increasing number of generations from 81 kJ mol^{-1} for G0Fer to ~130 kJ mol^{-1} for G2Fer and G3Fer (Table 1). The dielectric data from both the γ and β regions were adequately described by the Havriliak–Negami equation with $\beta = 1$ (symmetric broadening). The results of fitting Eq. (1) to the experimental data are shown in Table 1.

The characteristics of the γ process were essentially independent of the number of generations of the dendrimer (Table 1). This is in accordance with earlier results on a variety of liquid–crystalline polymers [18–21] suggesting that the γ process is insensitive to changes in morphology. It is more surprising that most of the characteristics of the β process remained constant, independent of the number of generations (Table 1). Only the activation energy displayed a clear trend with respect to the number of generations. However, the activation energy range (81–132 kJ mol^{-1}), the relaxation strength range (0.46–0.99) and the symmetric broadening factor are very similar to those of conventional side-chain liquid–crystalline polymers [18–20].

3.4. G2Nitro and comparison with data obtained for G2Fer

G2Nitro displayed both γ and β relaxation processes (Fig. 10) and indeed it resembled the behaviour of poly(**a1b**). Both sub-glass processes exhibited symmetrical broadening as shown in Fig. 11 for the β process.

The γ process in G2Nitro displayed basically the same features as in G2Fer, viz: $\alpha = 0.19$ (G2Nitro) and 0.18 (G2Fer); $\Delta\epsilon = 0.27$ (G2Nitro) and 0.26 (G2Fer); $\Delta E =$ 58 kJ mol^{-1} (G2Nitro) and 65 kJ mol^{-1} (G2Fer). These features were also very similar to the characteristics of the side-chain liquid crystalline polymer poly(**a1b**). The β process showed different characteristics in G2Nitro and G2Fer; the former showed significantly higher values in activation energy and relaxation strength: $\Delta E =$ 219 kJ mol^{-1} (G2Nitro) and 132 kJ mol^{-1} (G2Fer); $\Delta\epsilon =$ 2.5 (G2Nitro) and 0.46–0.76 (G2Fer). The symmetric

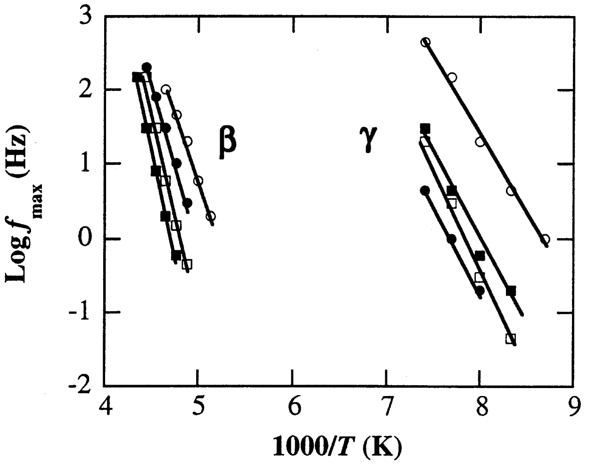

Fig. 9. Temperature dependence of the dielectric relaxation processes in the following polymers: ○ G0Fer; ● G1Fer; □ G2Fer; ■ G3Fer.

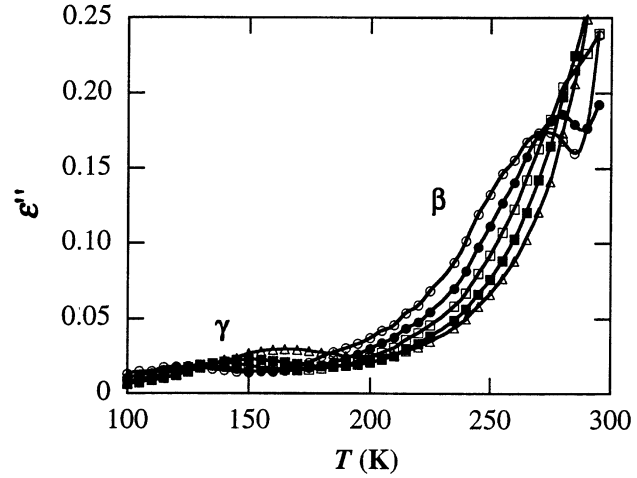

Fig. 10. Imaginary part of the dielectric permittivity (ϵ'') as a function of temperature for G2Nitro). Isochronous curves at the following frequencies: ○ 0.1 Hz; ● 1 Hz; □ 10 Hz; ■ 100 Hz; △ 1000 Hz.

broadening factor was essentially the same for the two polymers.

The resemblance between G2Nitro and poly(**a1b**) regarding the characteristics of the β process and the pronounced differences in these characteristics between the second generation dendrimers strongly suggests that the structure (packing of mesogens) is relatively independent of the molecular architecture. The relaxation behaviour is very sensitive to local structure of the flipping unit, i.e. whether or not it has a pendant nitro group.

4. Conclusions

The dielectric relaxation behaviour was recorded below the glass transition temperature for a series of dendritic polymers of generations 0–3 with mesomorphic terminal groups with and without a pendant nitro group attached to a phenylene ring in the mesogen. In addition, a side-chain liquid crystalline polymer with a pendant nitro group attached to a phenylene ring in the mesogen was studied. The polymers exhibited two sub-glass processes denoted γ (low temperature) and β (high temperature). The γ process, which is assigned to conformational changes in the spacer, proved to be insensitive to changes in molecular architecture and to the presence of the pendant nitro group in the mesogen. The β process, earlier assigned to a 180° flip of a part of the mesogen, was greatly influenced by the presence of pendant nitro groups in the mesogen and to a lesser extent by the number of generations in the dendrimer. The high activation energy (>200 kJ mol^{-1}) and high relaxation strength (>2) of the β process in polymers with attached pendant nitro groups suggest that the phenylene ring with the nitro group participates in the β process. The pendant nitro group increases the swept-out volume of the 180° flip motion and this explains the high activation energy. The fact that two strong dipoles participate in the process explains the high relaxation strength.

Table 1
Characteristics of the dielectric relaxation processes of GnFer polymers

	G0Fer	G1Fer	G2Fer	G3Fer
γ Process				
T-range (K)	115–135	120–140	120–135	120–135
α[a]	0.20 ± 0.01	0.18 ± 0.03	0.17 ± 0.02	0.17 ± 0.02
ϵ_{u}[a]	2.77 ± 0.03	2.88 ± 0.02	2.43 ± 0.02	2.72 ± 0.03
$\Delta\epsilon$[b]	0.27–0.35	0.26 ± 0.04	0.17–0.21	0.20–0.29
ΔE (kJ mol^{-1})	48	52	65	54
β Process				
T-range (K)	195–215	195–220	205–225	210–235
α[a]	0.18 ± 0.00	0.21 ± 0.01	0.17 ± 0.01	0.19 ± 0.01
ϵ_{u}[a]	2.95 ± 0.02	3.02 ± 0.05	2.45 ± 0.06	2.77 ± 0.09
$\Delta\epsilon$[b]	0.56–0.72	0.56–0.82	0.46–0.76	0.56–0.99
ΔE (kJ mol^{-1})	81	95	132	130

[a] Average ± standard deviation.

[b] Temperature-dependent value; minimum and maximum values correspond to values obtained at the low and high temperature limits, respectively.

The orientation of the relaxing dipoles with respect to the electric field will influence primarily the dielectric strength. It would be very useful to establish the relationship between the relaxation parameters (e.g. the relaxation strength) and

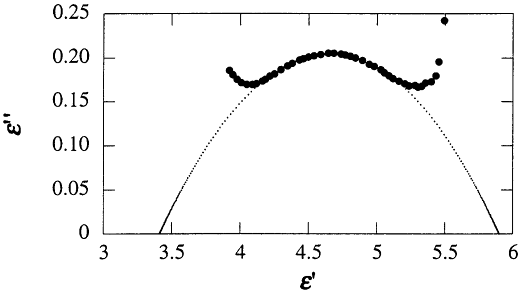

Fig. 11. Argand diagram of the β process at 280 K for G2Nitro.

the order parameter. This still remains to be done on the polymers reported in this paper.

Acknowledgements

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