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ORIGINAL PAPER

The effects of ion irradiation on the dielectric properties of poly(ether ether ketone)

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Abstract Temperature-dependent dielectric relaxation spectra of ion irradiated (11.2 MeV H^+ and 25.6 MeV He^{2+}) poly(ether ether ketone) (PEEK) were measured at frequencies between 50 and 10^5 Hz. The data were analysed by both Cole–Cole and Kohlrausch–Williams–Watts equations. β_{KWW} values were observed to decrease with irradiation, which was interpreted in terms of the Ngai et al. model as due to the increase of inter-chain coupling between the relaxing units caused by the additional constraints introduced by the cross linking. The effect of irradiation was also discussed in terms of local motion by studying the low temperature β-relaxation in PEEK. Polar groups were attached to the phenyl rings by irradiation as evident from the progressive increase in the β-relaxation peak intensity with dose.

Keywords Ion irradiation · PEEK · β-Relaxation · α-Relaxation · Cole–Cole · KWW · LET effect

Introduction

Irradiation of polymers as a route to improving their material properties has been the subject of numerous papers [1–4]. The various active species generated by irradiation of a polymer can react further and result in cross linking or chain scission. The mechanism of the reaction is being dependent upon irradiation source, and experimental conditions such as temperature and composition of the atmosphere as well as the chemical structure of the target materials [5].

Poly(ether ether ketone), PEEK, is an aromatic high performance engineering polymer with superior properties including good processability, high thermal

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stability, mechanical strength, excellent resistance to irradiation and a wide range of chemical reagents. This combination of desirable properties has resulted in the use of PEEK as a structural material for aerospace applications and in nuclear reactors [1, 2] as well as in fuel cells as a polymer electrolyte membrane, PEM [3, 4].

Chemical modifications produced by ion irradiation of amorphous PEEK have been extensively studied using structural, thermal and mechanical analytical techniques [1, 6–8], and it has been agreed that the main mechanism which accounts for property changes is that of cross linking. To date, the dielectric relaxations of irradiated PEEK have not been given full attention, and only few studies have been published [9–11] on the effect of γ-rays and electron beam irradiation. It has been reported that the glass transition temperature, taken as the peak corresponding to the maximum in the dielectric loss, increased with radiation dose indicating cross link formation. The results of the frequency dependency of the dielectric loss and dielectric constant have been analysed by the Cole–Cole empirical function from which it was concluded that the magnitude of the induced dipoles and ions as well as the relaxation intensity increased with increasing the radiation doses.

Studying the effects of ion irradiation on the dielectric properties of PEEK is, however, of prime importance. This is partly due to the fact that ions have higher LET value [5], the energy deposition rate per track length, and more complex reactions may proceed on ion irradiation. On the other hand, such a detailed study provides useful information in understanding the various polarisation mechanisms in the irradiated polymer and throws light on the mechanism of properties changes on ion irradiation.

In this article, the effects of cross linking/chain session introduced by ion irradiation (11.2 MeV H^+ and 25.6 MeV He^{2+} ions) on the dielectric behaviour of amorphous PEEK were studied by the use of dielectric relaxation spectroscopy, DRS, operating in both dynamic and isothermal modes. The effect of cross linking on the segmental motions was analysed by the change in dielectric response with frequency in terms of Cole–Cole and Kohlrausch–Williams–Watt equations. The fitting parameters were used to interpret the changes in term of the coupling model proposed by Ngai et al. The effect of cross linking/chain session was also discussed in terms of local motion by studying the effect of ion irradiation on the low temperature β-relaxation of PEEK.

Experimental

Material, irradiation and dosimetry

Amorphous PEEK was obtained from Good fellow Ltd., UK, as a 100-μm thick film with density of 1,260 kg m^{-3}. Irradiation with ions was carried out using the University of Birmingham's Scanditronix MC40 Cyclotron operating at 12.5 MeV for protons (H^+) and 33 MeV for helium ions (He^{2+}). In each case, the beam passed through a 30-μm Havar window, then about 40 mm of air, and a 25-μm copper foil followed by a stack of PEEK films (4–10 foils). This setup gave energies of 11.2 and 25.6 MeV at the surface of the first PEEK foil on irradiation with H^+ and He^{2+} ions, respectively. The dose, D, absorbed by each foil in a stack was calculated using the following relationship [1],

$$D = S \times \frac{Q}{Z_{\text{inc}}} \tag{1}$$

where S is the mass stopping power of PEEK (MeV m^2 kg^{-1}), Q is the fluence (C m^{-2}) and Z_{inc} is the charge number of the incident ion. The stopping powers (averaged over the energy range corresponding to each foil) of PEEK for H^+ and He^{2+} ions were calculated using the TRIM89 code (version 92.12) and are published elsewhere [7, 8], along with the energy on entry into each foil.

In the present work the ^{65}Zn activity produced in the copper monitor foil was used to determine the fluence. The ^{65}Zn yield was evaluated by detecting the characteristic 1,115 keV γ-rays using an HPGe detector whose efficiency has previously been determined. The yield Y (atoms) in mass M of copper can be converted to a fluence of incident ions using the relation

$$Y = \frac{QM}{em}\sigma \tag{2}$$

where m is the mass of each copper atom, e is the charge carried by each ion and σ is the cross-section for the nuclear reaction. Based on the evaluated cross sections published by the IAEA [12], values of $\sigma = 200$ mb (2.0×10^{-29} m^2) for the proton reaction and 650 mb (6.5×10^{-29} m^2) for the helium reaction were used.

Initially, the average flux over the whole area (32 mm × 32 mm) was determined. The sample was then cut into 16 separate squares, each approximately 8 mm × 8 mm, and the flux determined for each. Any differences in area were corrected by weighing the individual copper squares to determine their masses M. Table 1 summarises the results obtained using this procedure.

The dose absorbed by the first PEEK layer was calculated based on the incident energy and that of the other PEEK layers was calculated based on the inlet energy after the ions passed through the previous layer. The irradiation doses for samples used in the present work are listed in Table 2, along with the glass transition temperature, T_g, and the corresponding cross linking density.

Dielectric relaxation spectroscopy (DRS)

A dielectric thermal analyser, DETA, manufactured by Polymer Laboratories Ltd was used to follow the change in dielectric constant ε', dielectric loss ε'' and tan δ

Table 1 Irradiation dose evaluation

Irradiation	Proton	Helium
Individual fluence range (C m^{-2})	7.8–10.9	2.6–4.6
Average fluence over the whole irradiated area (C m^{-2})	9.4	3.4
Standard deviation	0.9	0.7
Dose at first PEEK layer (MGy)	37.9 ± 3.6	42.9 ± 8.8

Table 2 Dose, glass transition temperature, T_g, and cross linking density of PEEK samples

Irradiation	Dose (MGy)	T_g^a (±1.0 °C)	Proportion of units cross linked[b] (±0.005)
As received	0	145.4	0.000
Proton, H^+	39.3	152.7	0.168
	50.5	154.2	0.216
	59.1	156.0	0.253
	74.9	158.2	0.320
Helium, He^{2+}	42.9	155.4	0.220
	54.4	158.1	0.280

[a] As measured by DSC-7 in the first heating scan

[b] As evaluated from sol–gel fraction analysis following a previous procedure [8]

with applied frequency and temperature. The DETA consisted of a measuring cell, temperature controller and analyser. The measuring cell was equipped with circular parallel plate electrodes of 20 mm in diameter and placed in a thermostated furnace with a temperature range from −150 to 300 °C. A fixed voltage of 1.0 V was used with a set of frequencies in the range of 50 to 10^5 Hz. Step isothermal experiments were carried out for all PEEK samples and the loss and dielectric constant were measured at 20 frequencies and recorded at temperatures from 120 to 240 °C in steps of 2 or 5 °C with a soak time of 10 min between each step. To ensure good electrical contact between the electrodes and the sample the polymer films were sputter coated with a thin layer of gold, using a Polaron E5000 sputter coating unit.

Nonlinear fits to data were carried out using the Plus V5.44 software supplied by Rheometric Scientific™, UK. This program enables the isothermal data to be fitted to the Cole–Cole equation. Fitting was also carried out using a graphical method described elsewhere [13]. Fitting to the KWW function was done using Relax software.[1]

Results and discussion

The dielectric relaxation spectrum of amorphous PEEK

The dynamic dielectric behaviour of all PEEK samples was investigated at a heating rate of 1.0 K min^{-1} over the temperature range from −130 to 240 °C. Figure 1a shows the variation of both the dielectric constant, ε' and dielectric loss, ε'', of amorphous PEEK with temperature and highlights the presence of two relaxations at −50 and 150 °C. These are accompanied with an increase in dielectric constant and a peak in dielectric loss and were assigned to the sub-ambient β- and α-relaxations, respectively. Above the α transition, the dielectric constant decreases sharply corresponding to the onset of crystallisation, and then recovered at higher temperatures. The relaxation of the newly formed crystal portion was evident as a broad but distinct shoulder in dielectric loss located on the high-temperature side of

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[1] The dielectric data were fitted to the KWW function using Relax software prepared by Prof J Matecki.

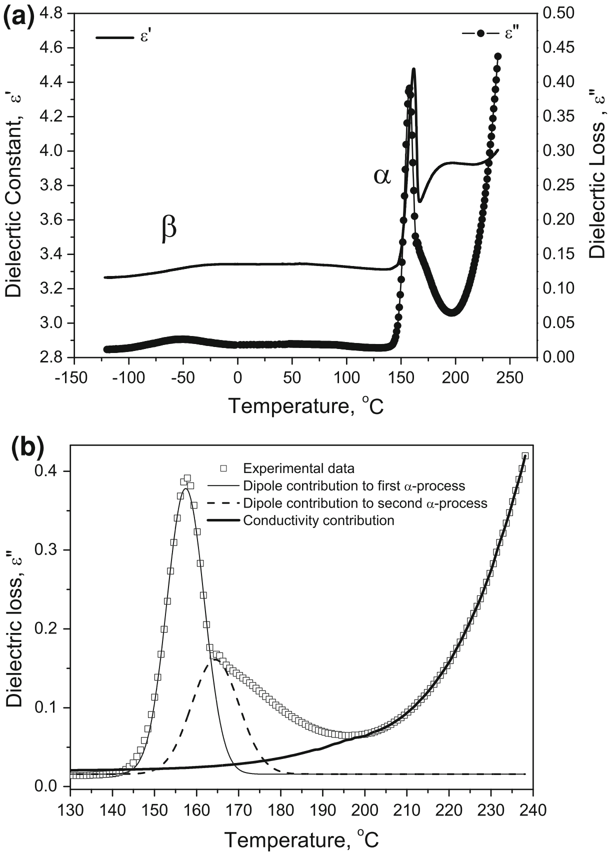

Fig. 1 **a** The temperature dependence of the dielectric constant, ε' and the dielectric loss, ε'', at 1.0 kHz for amorphous PEEK. **b** The dielectric loss, ε'' as a function of temperature at 1.0 kHz for amorphous PEEK; showing the dipole and conductivity contributions

the glass transition loss peak [14]. Finally, there was a large increase in the dielectric loss, ε'' due to increasing ionic conductivity with temperature. The overlapping in the temperature response of the dielectric loss in the region 130–240 °C was resolved using appropriate procedure [15] and assuming Gauss shape for the α-relaxation. As can be seen from Fig. 1b, three distinct regions associated with the mobility of the molecular chains are clearly present.

The dielectric relaxation spectrum of irradiated PEEK samples

On irradiation, as shown in Fig. 2, similar behaviour was observed with the two transitions (α and β) occurred at slightly higher temperatures depending on radiation dose. Differences also existed above the α transition in that there was a single peak in ε'' followed by a rapid increase with temperature. As previously observed [7, 8] crystallisation of the amorphous polymer is progressively suppressed by ion

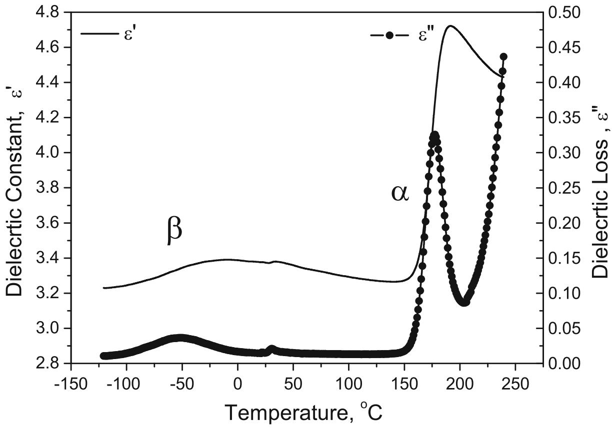

Fig. 2 Temperature dependence of the dielectric loss, ε'' and the dielectric constant, ε', at 1 kHz for 56.1 MGy helium irradiated PEEK

irradiation and at a sufficiently high degree of cross linking is completely inhibited. This appears to be the case with the irradiated samples.

Details of change in the dielectric properties

The sub-ambient β-relaxation

The temperature dependence of the dielectric loss of amorphous and irradiated PEEK in the temperature range −120 to 20 °C is shown in Fig. 3a, corresponding to the region of the sub-ambient β-relaxation of PEEK. The dielectric loss increased with radiation dose but no change in the temperature corresponding to the maximum in ε'' was observed for samples that were allowed to equilibrate with water vapour in the laboratory.

Equilibration with water was necessary as the amount present in the PEEK sample had a marked effect on the intensity and temperature of the sub-ambient β transition, as defined by the maximum in ε''. Amorphous PEEK samples scanned immediately after drying in vacuum at 80 °C for 48 h, equilibrating in the laboratory for 24 h and after soaking in water at 50 °C for 24 h exhibited very different responses, as shown in Fig. 3b. There was an increase in dielectric loss intensity and the peak maximum temperature decreased with increasing moisture content, from −57 °C for the water equilibrated sample to −42 °C for the vacuum dried sample. These results have been explained by the water acting as a plasticiser for the sub-ambient β-relaxation in PEEK [16].

The activation enthalpies, ΔH_β was determined from the frequency dependence of the transition temperature, T_β, assuming an Arrhenius dependence, such that

$$f = A\exp\left(-\Delta H_\beta / RT_\beta\right) \tag{3}$$

The values of ΔH_β were very similar at 45 ± 5 kJ mol^{-1} and were not markedly dependent on irradiation dose.

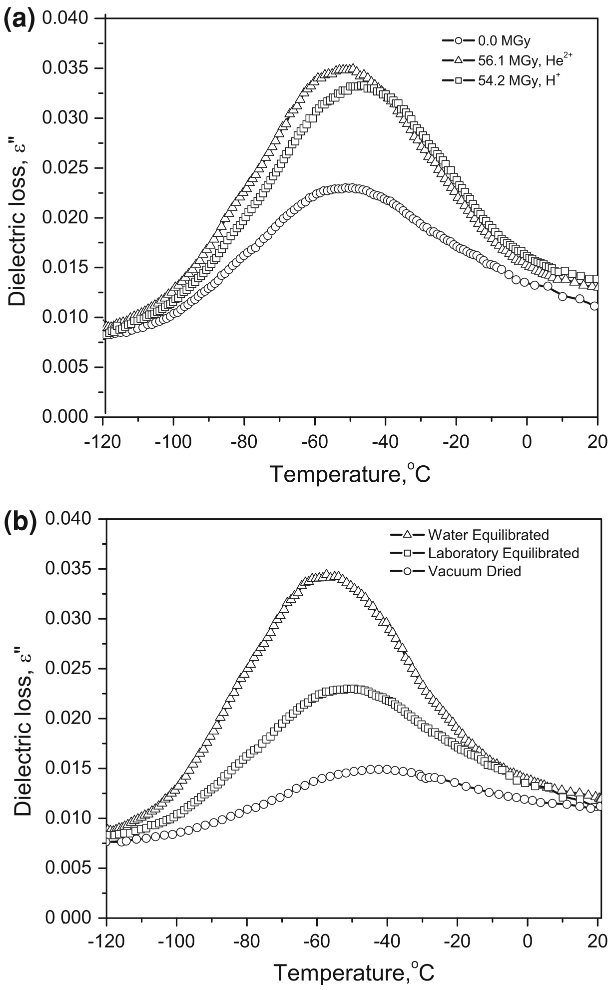

Fig. 3 **a** The effect of irradiation dose on the β-relaxation of laboratory equilibrated PEEK samples and **b** the effect of moisture content on the sub-ambient β-relaxation of amorphous PEEK

The sub-ambient β-relaxation is associated with phenyl ring motions, and while it is influenced by a number of factors such as water content, ageing history and morphology [16], it is reasonable to attribute the increase in intensity with irradiation to the introducing polar groups via oxidation as previously observed on the irradiation of polyethylene [17–19]. This is also consistent with the increase of sulphonation degree with irradiation observed in a previous article [4].

It has also been reported from a dielectric study of model aromatic compounds and related polymers by Schartel and Wendorff [20] that the sub-ambient β-relaxations in aromatic polymers are associated with inter and intra-molecular motions on a length scale equivalent to a single repeat unit, and accordingly cross linking does not have a marked effect on the mobility of such a small localised group.

The glass α-relaxation

Irradiation had a profound effect on the dielectric response of PEEK, in raising the α-relaxation associated with the glass transition to higher temperatures as shown in Fig. 4. This is in agreement with the calorimetric results reported earlier [7, 8] and indicated that the main mechanism of ion irradiation effects in PEEK is that of cross linking.

Different models have been suggested to analyse the dielectric dispersion curve of a broad relaxation process such as the α-relaxation in polymers.

The Cole–Cole expression is given by [13, 21],

$$\varepsilon^* = \varepsilon_\infty + \frac{\varepsilon_0 - \varepsilon_\infty}{1 + (iw\tau)^{\beta_{cc}}} \tag{4}$$

where w is the angular frequency ($w = 2\pi f$), τ is an average relaxation time and $\varepsilon^* = \varepsilon' - i\varepsilon''$ is the complex dielectric. ε_0 and ε_∞ are the low and high frequency limiting values of the dielectric constant, respectively. β_{cc} characterises symmetrical broadening and vary in the range $0 < \beta_{cc} \leq 1$. When β_{cc} is equal to 1, a simple Debye process is implied.

Plotting the dielectric loss against the dielectric constant for PEEK, see Fig. 5, a skewed semi-circular arc was obtained and as shown the agreement between the experimental data and data calculated using the Cole–Cole model is obtained for all samples indicating the validity of the model to describe the dielectric response in this region. Subsequently β_{cc} was determined from Eq. 4 for PEEK and irradiated PEEK as a function of irradiation dose and temperature, see Tables 3 and 4.

Figure 6a, b shows the dependence of the Cole–Cole broadening parameter at the normalised temperature, taken as $T = T_g + 15$, on the cross link density and on the irradiation dose, respectively. It is evident that the deviation from the Debye behaviour increased with increasing cross link density and more complex systems

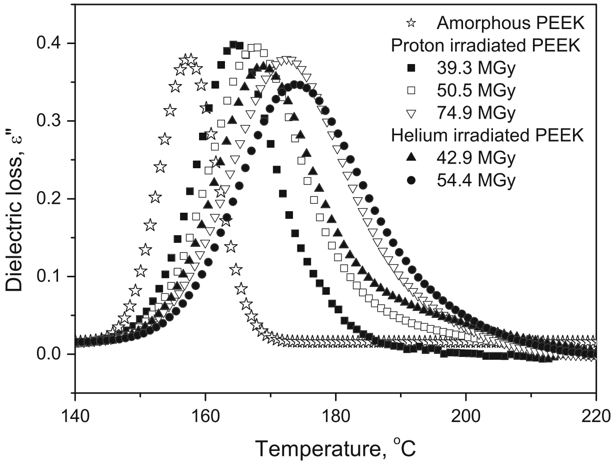

Fig. 4 The corrected (after subtraction of conductivity and second glass transition contributions) dielectric loss, ε'' of PEEK samples as a function of temperature at 1.0 kHz. Heating rate is 2.0 K min^{-1}

Fig. 5 Best fit of the Cole–Cole relationship to the dielectric data of **a** amorphous PEEK at 156 °C, **b** 59.1 MGy proton irradiated PEEK at 175 °C, and **c** 54.4 MGy helium irradiated PEEK at 175 °C

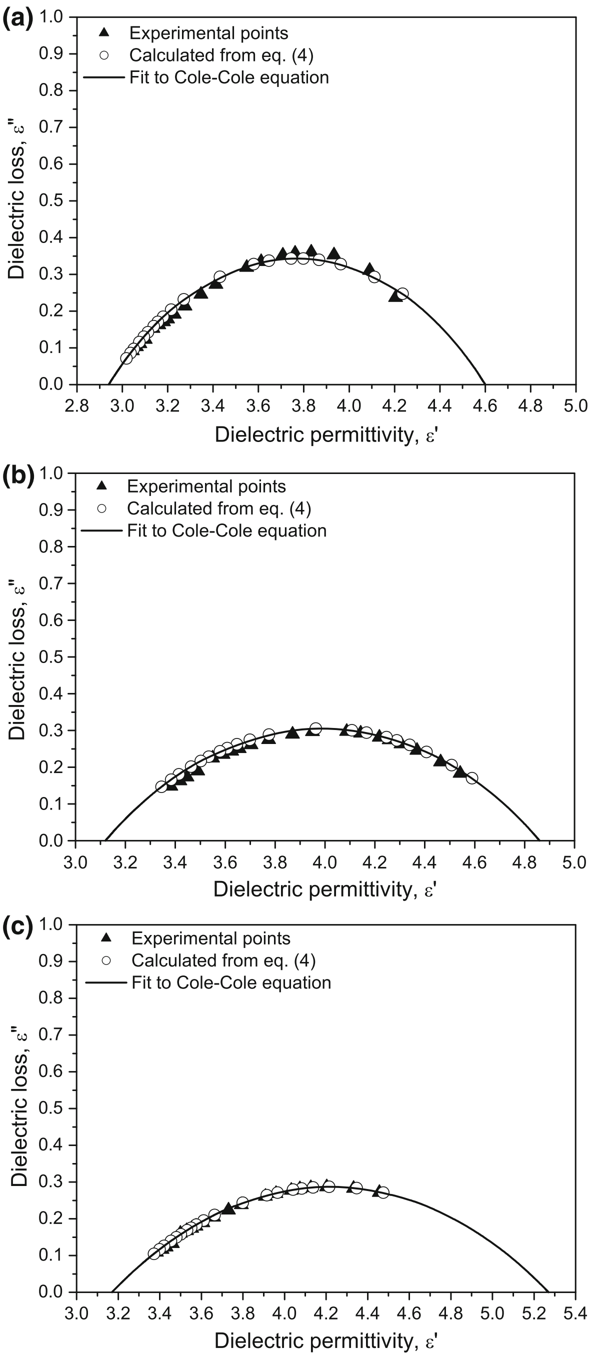

Table 3 The Cole–Cole best fit parameters for amorphous PEEK

References	Temp. (°C)	$\Delta\varepsilon$	β_{cc} (±0.05)	$\tau \times 10^3$ (s)
This work	154	1.98	0.45	2.90
	156	1.66	0.50	0.36
	158	1.44	0.59	0.11
Ref. [22]	154	1.09	0.58	0.38
Ref. [14]	154	1.24	0.64	0.126
	156	1.34	0.66	0.062
	158	1.37	0.63	0.022
Ref. [23]	158	2.21	0.41	0.49

Table 4 The Cole–Cole best fit parameters for irradiated PEEK

Irradiation	Dose (MGy)	Temp. (°C)	$\Delta\varepsilon$	β_{cc} (±0.05)	$\tau \times 10^3$ (s)
Proton, H^+	50.5	170	1.44	0.33	3.95
		175	1.35	0.37	0.82
		180	1.33	0.38	0.095
	59.1	170	1.87	0.39	0.097
		175	1.74	0.43	0.090
		180	1.68	0.43	0.015
	74.9	170	2.41	0.34	5.50
		175	2.03	0.39	0.23
		180	1.94	0.41	0.032
Helium, He^{2+}	42.9	170	1.58	0.34	0.360
		175	1.43	0.38	0.022
		180	1.43	0.38	0.033
	54.4	170	2.31	0.32	17.5.0
		175	2.10	0.34	0.82
		180	1.99	0.37	0.095

are formed on irradiation. The results also show that Cole–Cole formalism can be used to detect the LET effect as evident from Fig. 6b.

Similar conclusion was made from the analysis of the dielectric dispersion data using the Kohlrausch–Williams–Watts equation, KWW [24, 25]. In this model the relaxation function, $\varphi(t)$ exhibits a stretched exponential dependence on time, t, $\varphi(t) = \exp\left[-\left({}^{t}/_{\tau}\right)^{\beta_{\mathrm{KWW}}}\right]$ and,

$$\frac{\varepsilon^* - \varepsilon_\infty}{\varepsilon_0 - \varepsilon_\infty} = 1 - iwL[\varphi(t)] \qquad (5)$$

where L means Laplace transformation, τ is an average relaxation time and $0.0 < \beta_{\mathrm{KWW}} < 1.0$ is a measure of the breadth of the relaxation times of the processes involved in the dielectric relaxation. Figure 7 summarises the results of this

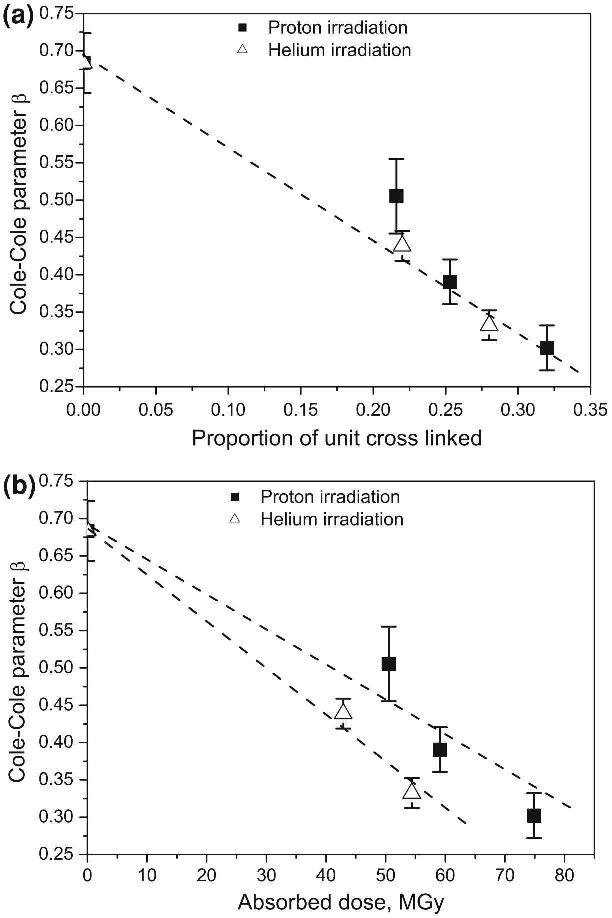

Fig. 6 The Cole–Cole broadening parameter, plotted at the normalised temperature, as a function of the proportion of unit cross linked (**a**) and the absorbed irradiation dose (**b**). *Lines* are guides for the eyes

analysis[2] and shows the variation in β_{KWW} with temperature plotted for different amorphous and ion irradiated PEEK samples.

All the irradiated samples had broader distributions and these also broadened with increasing temperatures. There was a general tendency for the value of β_{KWW} to decrease, and so the relaxation to broaden, with irradiation dose.

The importance of network formation in lowering the observed β_{KWW} values can be seen from Fig. 8 in which the effect of cross link density in broadening the distribution of life time can be seen to be independent of the irradiation ion used, although this was not the case with dose. Differences existed between the efficiencies of the two ions used in cross linking PEEK, in having different LET values.

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[2] See Footnote 1.

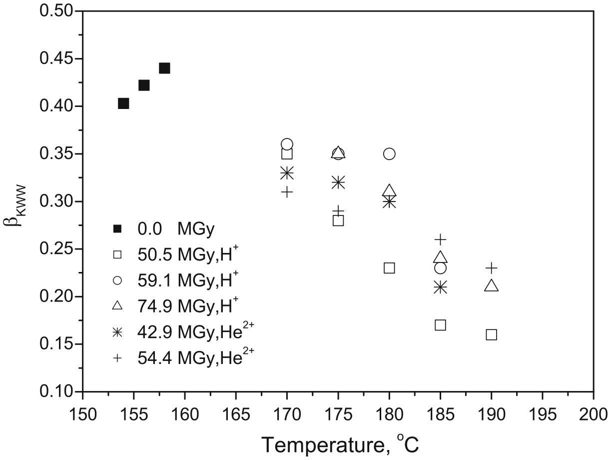

Fig. 7 Temperature variation of the breadth, β_{KWW} of the dielectric relaxation (error is ±0.03)

According to the coupling model proposed by Ngai et al. the deviation from Debye behaviour is due to the intermolecular coupling of relaxing species with neighbouring non-bonded ones [26, 27]. The degree of coupling (cooperativity), n increased with decreasing the experimentally observed β_{KWW} value (since $n = 1 - \beta_{KWW}$). This is the case with the irradiated samples studied, the observed decrease of β_{KWW} is the result of the increase of inter-chain coupling between the relaxing units caused by the additional constraints introduced by cross linking. These results are in accordance with previous investigations in networks based on a model heterocyclic polymer [28].

The characteristics of ion irradiation

Irradiation effects on polymers depend upon a number of experimentally related factors such as the temperature, atmospheric composition, dose and the linear energy transfer (LET) for the radiation source. The latter has been shown to be significant when considering the effect of ion irradiation on the structural and thermal properties of PEEK [8].

The dielectric properties are reported to exhibit changes under radiation due to the production of ions, permanent dipoles, space charges [29], nonreactive gases, and to oxidation by-products when carrying out irradiation on air [30, 31]. However, when considering two different radiation sources the so-called LET effect plays the major role in inducing these changes in the polymer. As can be seen from Fig. 4 the helium irradiated samples, although to lower doses than those of the proton irradiated ones, have the higher loss peak temperature. Figure 6b indicated that the decrease in Cole–Cole parameter with dose is much steeper in the case of helium irradiation. The same observation can be deduced from Fig. 8b, which represents the relation between the KWW broadening parameter and the irradiation dose.

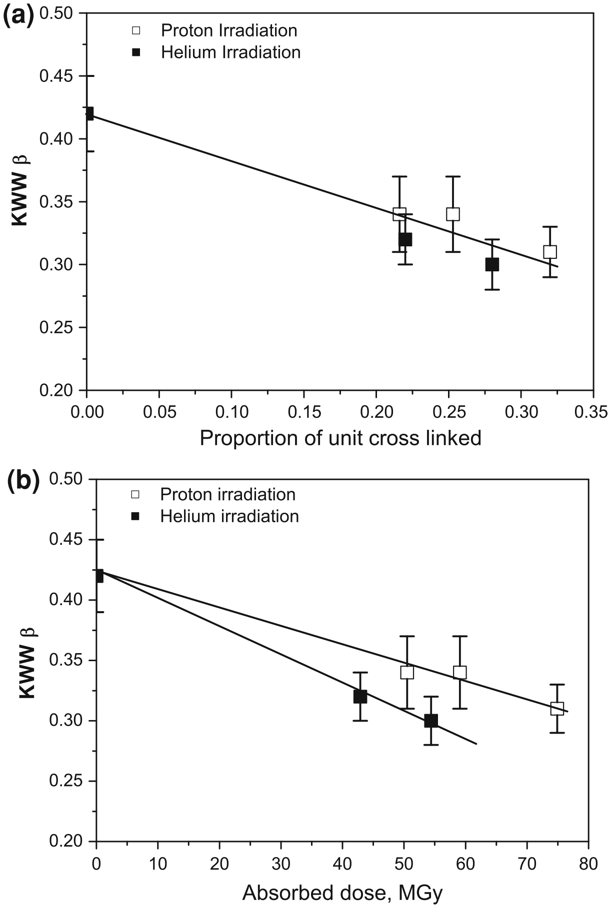

Fig. 8 The KWW breadth parameter, plotted at the normalised temperature, as a function of the proportion of unit cross linked (**a**) and the absorbed irradiation dose (**b**). *Lines* are guides for the eyes

The other effect of irradiation being the production of polar groups due to the presence of air and this is evident from the analysis of β-relaxation.

Conclusions

The specific influence of ion irradiation on the relaxations in PEEK has been investigated by DRS. Changes in the intensity of the β-relaxation of PEEK with ion irradiation are mainly related with oxidation, but cross linking does not have a marked effect on the mobility of small localised groups in this region. In contrast the α transition is extremely sensitive to the presence of cross links and the glass transition temperature increases progressively with absorbed dose. The results also showed that the Cole–Cole and the Kohlrausch–Williams–Watts formalisms could well describe the dielectric spectrum for both amorphous and irradiated PEEK in the frequency and temperature range studied.

With the KWW function, it was possible to describe the results in light of the coupling model. The observed decrease in the β_{KWW} parameter resulted from the increase of inter-chain coupling between the relaxing units caused by the growing hindrance with increasing degree of cross linking. Although the general irradiation effects are similar for both ion, but there were some indications that the dielectric properties of polymers are influenced by the LET effect of the irradiated ion used.

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