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Electron Paramagnetic Resonance Studies of the Effect of Temperature **on** Polymeric Radiation Dosimeters

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Electron Paramagnetic Resonance (EPR) and infrared spectroscopy were employed to study the effect of the temperature at which a polymeric radiation detector operates during electron irradiation process. The results demonstrate that the rate at which the free radicals created in the polymer by the irradiation recombine, changes with the temperature applied during the actual irradiation.

Keywords: Radiation detectors; polymeric; temperature effect

INTRODUCTION

Solid State Nuclear Track Detectors (SNTD) have become one of the main technologies employed nowadays for detecting, in many fields, heavy charged particles by electric irradiation. It is known that one of the most important effects of radiation on polymeric materials, which

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constitute a good part of all the SNTD commercially used, is the molecular chains scission, which in turn produces free radicals and degradation of the materials themselves. Those effects constitute the basis of the detection mechanisms in polymeric SNTD. However, experimental evidence $[1-3]$ demonstrates that some environmental parameters, in particular temperature, during the actual measurement, strongly affects the detection efficiency and performance of polymerbased SNTD. More specifically, it has been found that the temperature at which the polymeric SNTD is kept during the experiment determines how reliable the figure obtained can be. Nevertheless, there are very few quantitative and systematic studies on the effect of temperature on SNTD of any type. Accordingly, this work is aimed to analyse the changes induced on commercial SNTD (CR 39) by temperature, through Electron Paramagnetic Resonance (EPR) and Infrared Spectroscopy (FTIR) techniques.

EXPERIMENTAL

Commercial polycarbonate-based polymeric SNTD (trademark CR39, 1.5 cm² of exposed area) were irradiated with a 1.3 MeV electron beam **[4],** for 60 minutes each. During the irradiation, the temperature of the detector was controlled and the range of temperatures studied was from room temperature up to 100° C (25, 33, 40, 50, 80 and 100 $^{\circ}$ C) and the corresponding dose at which each sample was subjected was 0.3341 kGy/s. The experimental set-up for the irradiation process is schematically depicted in Figure 1 where the power supply for heating the specimens and the digital thermometer which records the temperature, are shown. Control samples were heated to the same temperatures during the same 60 minutes but without irradiation, to compare the relative effect heating-irradiation.

All the samples were analysed in an EPR spectrometer working at the following conditions:

Magnetic Field: $H = 3400$ gauss $\Delta H = 500$ gauss Modulation of the signal: 10 Reading time: 4min.

FIGURE 1 Experimental set-up for the irradiation procedure of the samples.

The IR spectra were obtained in a FTIR Nicolet 910 bench from powdered samples mixed with KBr.

RESULTS AND DISCUSSION

The EPR signal was located at a frequency of 9.2GHz for the experimental conditions described above. Table I summarises the relative EPR intensity per unit mass produced by the specimens heated at different temperatures. There, t_c represents the cooling time, that, the time required to cool down the specimens back to room temperature.

T_i $(^{\circ}C)$	I_e (min)	Intensity per unit mass (I)
25	138	0.5123
33	133	0.2973
40	136	0.3548
50	138	0.2471
60	138	0.1522
80	137	1.02×10^{-3}
100	129	5.64×10^{-4}

TABLE I Relative EPR intensity per unit mass from specimens heated at different temperatures

Figures 2(a) through (2g) show the corresponding EPR spectra obtained from the CR39 samples heated at the temperatures indicated there, In Figure *3* these results are summarised by plotting the maximum intensity per unit mass of the EPR signal, as a function of temperature. The results clearly show that the maximum EPR response is attained at room temperature and decreases as the temperature at which the sample has been irradiated decreases. This effect is due to the effect of heating temperature, since the control specimens, irradiated at the same conditions, did not show the above EPR behaviour.

To understand the EPR results, one has to realise that the EPR intensity is basically related to the amount of free radicals, molecules in triplet states *(ie.,* having two unpaired electrons) and certain transition metal ions and complexes *[5].* Thus, the observed change in EPR

FIGURE 2 EPR spectra from CR39 specimens irradiated with a dose of 0.3341 **kBy/s** of **electrons** while heated at: **(a)** *25°C;* (b) 33°C; (c) 40°C; (d) 50°C; (e) *60°C;* **(f)** 80°C; **(g)** 100°C.

FIGURE 2 (Continued).

FIGURE 2 (Continued).

FIGURE 2 (Continued).

FIGURE **3** for the CR39 samples. Maximum EPR intensity per unit mass *vs.* temperature during irradiation

FIGURE **4** FTIR spectrum of a sample irradiated while heated at 33°C.

intensity indicates that free radicals are formed and recombined in different fashion in the various samples analysed. The FTIR results showed that the main effect of the irradiation was the formation of free OH radicals, *[6]* as can be observed in the spectrum of Figure **4,** which corresponds to the case of the sample heated at *33°C.* It is interesting to point out that the FTIR spectra for all temperatures were basically equivalent, indicating that temperature has little effect, as compared to that of the irradiation, on the formation of free radicals. However, temperature does not affect the mobility of those

FIGURE *5* EPR relative intensity as a function of the time elapsed after the actual irradiation, for a sample at room temperature.

free radicals created by the irradiation process, thus increasing the kinetics of recombination to form new products.

According to this scheme, the temperature at which the specimens are subjected during the irradiation increases the recombination rate of the free radical created which would be then more short living as the temperature increases. This hypothesis is supported by the plot of Figure 5, which shows the change in EPR relative intensity as a function of the time elapsed after the actual irradiation has taken place, for a sample irradiated at room temperature. As observed there, if one allows the material to set by itself, all the free radicals created by the irradiation would eventually recombine. This is accelerated strongly if the temperature is increased, since the free radicals would have higher mobility, as mentioned.

CONCLUSIONS

The results indicate that EPR is an interesting tool to study in further detail some of the radiolitic processes already reported in SNTD materials. The temperature at which the detector operates was found to play an important role on the mean life of the free radicals created during the irradiation. Some studies on the precise kinetics of the recombination process and on the presence of other chemical functional groups, by using techniques such as Raman spectroscopy, capable of finding groups invisible to FTIR, are required if a deeper understanding of the radiation-polymetric matter interaction is to be achieved.

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