XA0103556

IAEA-SM-366/147

Glass Transition Temperature Determination on Cross-linked Polyethylene by Dynamic Mechanical Analysis.

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ABSTRACT

The Glass Transition Temperature, T_g, was determined on electron beam cross-linked low-density polyethylene, using the dynamic mechanical analysis technique. The measurements were carried out over a temperature range of –170 to –100°C, at frequencies of 0.5, 1.0, 2.0, 5.0 and 10.0 Hz and at an average heating rate of 1.83 °C/min. The radiation dose was 50,100,150,200,300 and 400kGy that corresponded to about a cross-linking degree from 20 to 80%. The experimental results have shown that T_g changed about 10 degrees as a function of frequency but for each frequency the value of Tg remained constant as a function of the irradiation dose. At the fixed frequency of 1.0 Hz Tg had a value of –139.4±0.1°C and the activation energy was 51.2±0.7kJ.mol^{-1}. These experimental results have shown that the presence of a three-dimensional interconnected structures and increase in molecular weight, due to the cross-linking effect, had not affected the T_g value at each frequency. It was expected that these molecular modifications could hinder, in some way, the molecular rotational motion, changing this Tg value.

Key Words: Polyethylene, Glass Transition Temperature, Cross-linking, Radiation.

INTRODUCTION

The glass transition temperature, T_g, is one of the most important physical parameter in the industrial applications of polymeric materials, since it determines the processing conditions and also its in-service properties [1]. Cross-linking, on the other hand, is one of the most important radiation process, for improving physical, chemical and mechanical properties of polymeric irradiated materials. DMA, has become a very useful technique for polymer characterization. Polymers exhibit, in DMA measurements, characteristic modulus changes and energy absorption peaks at specific temperatures and one of this energy absorption peak is the loss factor tanδ [2]. The temperature corresponding to the maximum of this peak, $\tan\delta_{max}$, have been identified as the T_g of the material. The main aim of the present work, was to find out if the change in the molecular structure and the increase of the molecular weight due to the irradiation cross-linking effect, could produce changes in the polyethylene T_g

EXPERIMENTAL

DMA samples of branched-LDPE were cut from a plaque with dimensions 50×10×5 mm^3. These samples were electron irradiated at the irradiation facilities at IPEN-CNEN/SP. The radiation dose was of 50, 100, 150, 200, 300 and 400kGy, that correspond to a cross-linked degree of 20 to 80%. The DMA measurements were carried out in the three point bending mode over a temperature range of –170 to –100°C and at frequencies of 0.5, 1.0, 2.0, 5.0 and 10.0Hz. With the T_g values as a function of the frequencies, the activation energy was estimated for each irradiation dose.

RESULTS AND DISCUSSION

DMA measurements have shown that the position of $tan\delta_{max}$, moves to higher values of temperature with an increase of the frequency. This behavior is similar for all irradiated samples when analyzed under the same conditions. In Figure 1 is shown the effect of the radiation dose and temperature on the values of $tan\delta_{max}$ when the DMA measurements were carried out at a fixed frequency of 1.0Hz. The result has shown that the T_g,of the LDPE remained constant for each dose and at this frequency had a value of – 139.4±0.1°C. In Table 1 are given the obtained values of T_g at 1.0Hz for each radiation dose.

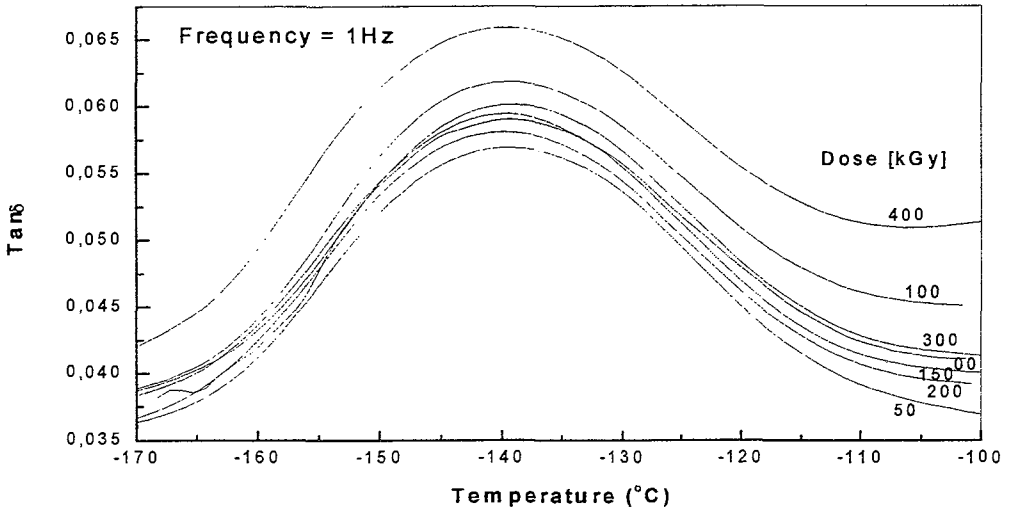

Figure 1 Effect of the irradiation dose on Tanδ at 1.0Hz.

Table 1 T_g as function of irradiation dose[kGy] measured at 1.0Hz.

DOSE	00	50	100	150	200	300	400
Tg (°C)	-139.4	-139.3	-139.3	-139.6	-139.7	-139.0	-139.6

CONCLUSION

In this work, the most important result was that the glass transition temperature of the electron irradiated low-density polyethylene, measured by the DMA technique, remained constant independent of the radiation dose. It was expected that the molecular modifications and gain in molecular weight, due to the irradiation cross-linking effect, could hinder, in some way, the molecular mobility of the polyethylene producing the change of the of Tg value.

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