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Effect of electron beam irradiation on polyimide film

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Abstract

The effects of electron beam radiation on polyimide films were investigated through dynamic mechanical analysis (DMA), in which the energy of the electron beam was set to a voltage of 1 MeV and a beam current of 4 mA. The β_2 sub-glass relaxation temperature of polyimide decreased with irradiation dose, consisting with formation of free radicals at the ends of the molecular chains. The glass relaxation temperatures are not influenced as a function of increasing electron beam exposure because imide rings prevent structural damage by electron beam irradiation. Furthermore, decomposition temperatures of the irradiated polyimide slightly decrease, from 571.42 to 570.67 °C and the residue weight also decreases.

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Keywords: Electron beam; Irradiation; Polyimide

1. Introduction

Highly heat-resistant polymers are highly useful structural materials for space, aviation, electricity, high-tech electronics, and potential future applications. Polyimides like Kapton are high-performance polymers which combine excellent physical, electrical, and mechanical properties at temperatures as low as −269 °C and as high as 400 °C. In addition, polyimide has an outstanding thermal stability and decomposes before melting. Due to this reason, polyimide is used in many applications such as ion exchange membranes, metal absorbents, gas absorbents, separation filters, and adhesives. In space applications, polyimides are used for thermal blankets, electric insulators, satellite antenna covers, array substrates, and as polymeric membranes for solar sail systems [1–2]. It is important that polymers used in space are able to retain good tensile and optical properties during the expected lifetime of the space vehicle.

Earth-orbiting hardware operates in environments that generally include neutral particles, charged particles such as trapped protons and electrons, solar protons, cosmic rays, vacuum ultraviolet light and dilute low energy plasma. Spacecraft and satellites in geosynchronous Earth orbit also encounter debris from other spacecraft, natural micrometeoroids, direct and reflected solar photons, and thermal radiation and magnetic fields from Earth. These satellites are exposed to charged particles doses of about 1.5–2.0 × 10^4 kGy, or even more in some circumstances. Under these conditions, major structural transformations in the polyimide, such as chain scission and cross-linking events, have been reported. Such transformations can result in changes in the optical, electrical, and mechanical properties of the polymers [3–4], with an associated increase in the likelihood of hardware failure as material properties continually degrade over the course of longer periods in space.

Low energy electrons are the cause of electrostatic discharge. This can be a serious problem for spacecraft in higher altitude orbits where they are exposed to more intense electron populations. Higher energy electrons can penetrate the spacecraft, collect in insulator materials, and damage electronics upon discharge. In addition, the system is not grounded with respect to the space plasma potential. This can give a rise to unstable conditions in which charge can be lost from the spacecraft or satellite to space. The environmental components responsible for polymer degradation in space are ionizing radiation and extreme temperatures.

Previous investigations have investigated electron beam effects on the stability and tensile characteristics of polyimide over temperatures as high as 250 °C and doses up to 1.85 × 10^4 kGy [5]. Polyimides were shown to maintain good optical and tensile properties at temperature up to about 177 °C for a dose of 1.85 × 10^4 kGy, but above this temperature the

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moduli of the polyimide began to deteriorate and there was a small decrease in the transmittance of the exposed polymer films. Also the tensile strength of polyimides has been extensively studied, and has been reported to resist γ rays and electron beam up to $\sim 10.0 \times 10^4$ kGy [6].

The relaxation behavior of ^{238}U ion-irradiated polyimides was investigated using the thermally stimulated discharge (TSD) current technique [7]. This study revealed that carbonyl groups affect the dipolar relaxation process, and that cross-linking of imidic groups and formation of free radicals responsible for deep and shallow energy traps affect the α-relaxation. In addition, the effect of accelerated proton beams has been examined by measuring the glass relaxations of polyimide by using dynamical mechanical analysis [8]. Artiaga and Chipara indicated that the temperature dependence of the loss factor proved the presence of two relaxations; a β_2 sub-glass relaxation located at ∼100 °C, and a glass relaxation located at ∼400 °C. The near-insignificant shift was observed in the glass relaxation temperatures following proton irradiation supports the use of polyimide in environments that combine radiation with extreme temperatures.

In this study, we investigated the effect of electron beam irradiation on the dynamic mechanical properties of polyimide films using dynamic mechanical analysis (DMA). DMA is the most accurate technique for the study of the glass relaxation and relaxation processes in polyimide. These tests focused on the shift of the β_2 sub-glass relaxation peaks and the glass relaxation peak due to electron beam irradiation. The goal of this research is to determine if the electron beam-induced shift in the glass relaxation temperature of polyimide is sufficiently large to harm short-term space missions of geosynchronous Earth-orbiting satellites. Additional thermo-gravimetric analysis (TGA) was performed to evaluate the thermal stability of electron beam-irradiated polyimide.

2. Experimental

2.1. Materials

The polyimide film (Kapton HN, 125 μm thick) was supplied by DuPont Electronics. A strip 50 mm wide was cut from the center. From this strip, rectangular samples (25 mm × 5 mm) were cut and used for DMA testing.

2.2. Electron beam irradiation

Irradiations were carried out in N_2 using an ELV-4 accelerator at an acceleration voltage of 1 MeV and a beam current of 4 mA. The dose rate was 10 kGy/pass. The samples were irradiated at 1.5×10^4 kGy.

2.3. Measurements

The mechanical analysis of polyimide was carried out in tension mode with a DMA Q 800 dynamic mechanical analyzer (TA Instruments, Inc. USA). The temperature range studied was 40–500 °C. Film specimens were heated at a rate of 5 °C/min in air. The temperature dependence of the storage modulus (elastic), loss modulus (viscous), and tan δ was measured at a frequency of 1 Hz. Examination was also performed at frequencies ranging from 0.5 to 2 Hz. Tg was identified as the maximum value of the tan δ curves. The storage modulus (E'), loss modulus (E'') and the tan δ (loss factor) were determined as a function of temperature.

TGA was carried out from 50 to 800 °C, at a heating rate of 10 °C/min under N_2 using a SDT Q600 thermo-gravimetric analyzer (TA instrument, Inc. USA). About 4–5 mg of irradiated Kapton HN film was examined.

3. Results and discussion

A typical response from a DMA shows three curves; storage modulus, loss modulus, and tan δ (loss factor). As the polyimide sample goes through its glass transition, the storage modulus reduces and tan δ passes over a peak because the sample becomes less stiff and molecular reorganization of the relaxation induces less elastic behavior. As can be seen in Fig. 1, the tan δ curve exhibited two at near 100 and 420 °C, indicative of two relaxations. The high-temperature peak originates from the glass relaxation occurring in polyimide. Its position is in accordance with the properties of pristine polyimide, as furnished by DuPont. The low-temperature peak may originate either from the β_2 sub-glass relaxation or the contribution of adsorbed water molecules [9–12]. The β_2 sub-glass relaxation was assigned to the rotation or oscillation of phenyl groups within polyimide's diamine moiety [11,12]. Typically, this relaxation was reported at higher temperatures (ranging between 125 and 190 °C). The β_1 sub-glass relaxation was not observed. The β_1 relaxation may be hidden by the overlap between the glass relaxation and β_2 sub-glass relaxation peaks. It was reported that the β_2 sub-glass relaxation peak is the weakest peak of polyimide [12]. And Fig. 2 shows DMA curves of control (not irradiated) and 1.5×10^4 kGy electron beam-irradiated samples. It was found that the β_2 sub-glass relaxation peak points decrease, as the electron beam dose increases. The tan δ values of electron beam-irradiated sample are higher than them of the control sample.

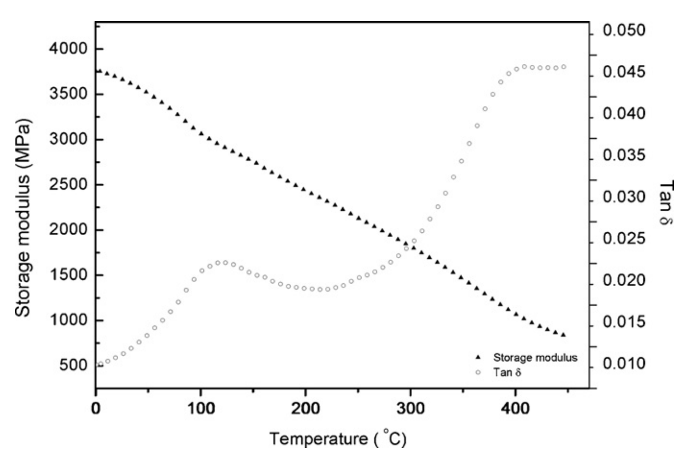

Fig. 1. DMA curves of polyimide.

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