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Electron beam irradiated polyamide-6,6 films—II: mechanical and dynamic mechanical properties and water absorption behavior

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Abstract

Electron beam irradiation of poly(iminohexamethylene-iminoadipoyl) (Polyamide-6,6) films was carried out over a range of irradiation doses (20–500 kGy) in air. The mechanical properties were studied and the optimum radiation dose was 200 kGy, where the ultimate tensile stress (UTS), 10% modulus, elongation at break (EB) and toughness showed significant improvement over the unirradiated film. At a dose of 200 kGy, the UTS was improved by 19%, the 10% modulus by ~9% and the EB by ~200% over the control. The dynamic mechanical properties of the films were studied in the temperature region 303–473 K to observe the changes in the glass transition temperature (T_g) and loss tangent (tan δ) with radiation dose. The storage modulus of the film receiving a radiation dose of 200 kGy was higher than the unirradiated film. The water uptake characteristics of the Polyamide-6,6 films were investigated. The water uptake was less for the films that received a radiation dose of 200 and 500 kGy than the unirradiated film. The role of crystallinity, crosslinking and chain scission in affecting the tensile, dynamic mechanical and water absorption properties was discussed.

Keywords: Polyamide-6,6 films; Electron beam radiation; Tensile properties; Dynamic mechanical analysis; Water absorption

1. Introduction

Investigations on the effect of high-energy radiation on polymeric materials started soon after man learnt to harness the power of an atom. The first polymer on which the effect of radiation was studied was polyethylene. Subsequently irradiation studies were carried out on other polymers like polypropylene, polyvinylchloride, polystyrene, poly (methyl methacrylate), polytetrafluoroethylene, polyamide, polyurethane, various rubbers, thermoplastic elastomers, etc. (Böhm and Tveekrem, 1982; Wilski, 1984, 1987; Wundrich, 1985; Banik et al., 1998; Sen Majumder and Bhowmick, 1999; Chattopadhyay et al., 2001).

One of the well-known polyamides is poly(iminohexamethylene-iminoadipoyl) (abbreviated henceforth as PA66), which finds use as an engineering plastic because of its hardness, stiffness, abrasion resistance, good processability and high heat distortion temperature. Radiation crosslinking of PA66 has been reported by several workers (Charlesby, 1953; Lawton et al., 1953; Little, 1954; Valentine, 1957; Zimmerman, 1960; Bernstein et al., 1965; Parkinson and Sisman, 1971; Ueno,

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1990; Lyons and Glover, 1990). However, most of the above workers used extruded films, yarns or fibers or injection molded chips and rods for their studies. But detailed investigations on the correlation between structure and mechanical properties of irradiated PA66 were not done. Only one work (Sengupta et al., 2003) discussed the influence of high energy irradiation on *compression molded* PA66 samples.

Dynamic mechanical analysis (DMA) is one of the most powerful techniques for studying the structure–property relationships of polymers, because the damping behavior of polymers is acutely sensitive to the molecular structure. Numerous DMA studies have been carried out on PA66 (Bell and Murayama, 1969; Turi, 1997). DMA of PA66 reveals three peaks known as α, β and γ peaks at approximately 353, 203 and 123 K, respectively, in the tan δ versus temperature scans. The α peak is believed to result from the rupture of the hydrogen bonds between polymer chains and is a characteristic of the glass transition temperature (T_g) of PA66. The β peak arises due to the amide groups in the amorphous region, while the γ peak is associated with very localized crankshaft rotation of four or more methylene carbon atoms between the amide groups. The dynamic mechanical properties of PA66 irradiated by high-energy radiation (consisting of neutrons and γ-rays) from an atomic pile have been reported (Deeley et al., 1957; Woodward et al., 1957). But the dynamic mechanical properties of electron–beam irradiated PA66 have not been reported so far. In this report (the second in our series), the mechanical and dynamic mechanical properties of *compression molded* PA66 films that had been electron–beam irradiated in air have been investigated. Further, an attempt has been made to analyze these results in light of the crystallinity and gel content. In this paper, water uptake behavior of irradiated films has also been studied.

2. Experimental

2.1. Materials

PA66 pellets [chemical name: poly(iminohexamethylene-iminoadipoyl)] (manufactured by DuPont, USA) which contained very small amounts of proprietary antioxidant, lubricant, etc., were kindly supplied by M/s. Black Burn & Co. Pvt. Ltd., Kolkata. It has a melting point of $\sim$536 K as determined from Differential Scanning Caloriemetry (DSC).

2.2. Preparation of films and subsequent irradiation

The PA66 pellets were vacuum dried at a temperature of 353 K for 6 h prior to compression molding. Compression molding was carried out at 563 K under 5 MPa pressure for 8 min (480 s) in between Teflon sheets to form rectangular films ($180 \times 150\,\text{mm}^2$) of $\sim$0.1 mm thickness, which were then cooled under pressure by passing water through the mold platens and were taken out when the platen temperature reached 303 K. The films kept in polyethylene zipper packets were then subjected to electron beam irradiation (RDI Dynamitron) in air at ambient temperature at NICCO Cables, Shyamnagar. The irradiation doses used were 20, 50, 100, 200 and 500 kGy. 10 kGy dose was given in each pass and the samples in the packets were cooled in air with a blower subsequently before the next pass. The frequency of irradiation of each pass was of 18 min (1080 s). The sample designations are given in Table 1. The specification of the electron beam accelerator was reported in our previous communication (Sengupta et al., 2003).

Table 1
Designation and irradiation dose of the films

Sample code	PA66 (parts)	Radiation dose (kGy)
N0 (Control)	100	0
N2	100	20
N5	100	50
N10	100	100
N20	100	200
N50	100	500

2.3. Tensile studies

The films were punched out by an ASTM D638-98 Type IV die and the tensile properties of the dumbbell specimens were carried out in a Zwick 1445 Universal Testing Machine at a crosshead speed of 50 mm min^{-1} at 296$\pm$2 K and 50$\pm$5% relative humidity. Since the Dry-As-Molded (DAM) condition was difficult to maintain, the films were exposed to a temperature of 303$\pm$2 K and 70$\pm$5% relative humidity for 120 h (432,000 s) after irradiation. The average of three tests is reported here.

2.4. DMA studies

DMA 2980 Dynamic Mechanical Analyzer of TA Instruments was used in tension mode in the temperature range 303–473 K at a frequency of 1 Hz and heating rate of 2 K min^{-1}. The dimensions of the films were $25 \times 6.3 \times 0.1\,\text{mm}^3$. The data were analyzed by using Thermal Advantage software of TA Instruments.

2.5. Water absorption

The irradiated films were conditioned in an oven for 24 h (86,400 s) at 323$\pm$2 K, weighed and then the