Journal of Macromolecular Science, Part A
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713597274

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To cite this Article Sharma, Anita , Singh, N. L. , Gadkari, M. S. , Shrinet, V. and Avasthi, D. K.(2005) 'Effects of 50 MeV Li^3 Ion Irradiation on Kapton Films', Journal of Macromolecular Science, Part A, 42: 2, 149 — 155
To link to this Article: DOI: 10.1081/MA-200046970
URL: http://dx.doi.org/10.1081/MA-200046970

Journal of Macromolecular Science®Part A—Pure and Applied Chemistry, 42:149–155, 2005

ISSN 1060-1325 print/1520-5738 online
DOI: 10.1081/MA-200046970

Effects of 50 MeV Li^{3+} Ion Irradiation on Kapton Films

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Blisters formation on the surface of kapton film has been observed due to 50 MeV Li^{3+} ion irradiation up to the fluence of 1.04×10^{14} ions/cm^2 at ambient temperature. This investigation provides a foundation for a quantitative evaluation of the FTIR results for thermally stable polymer on the chemical bond deterioration with irradiation fluences. The blistering mechanism is correlated with the internal gases (CO, H_2) released due to Li^{3+} ion induced radiation damage. The Vicker's micro-hardness testing has been carried out to study the mechanical behavior of irradiated films at room temperature. It is observed that the true bulk hardness of the film was obtained at loads greater than 400 mN. The hardness of the film increases significantly as fluence increases.

Keywords 50 MeV Li^{3+} ions, irradiation, blisters, microhardness, kapton

Introduction

Blistering on the surface of metals and alloys due to ion implantation is a well-known phenomena.[1,2] Besides its academic interest, it has great importance in nuclear and space technologies due to the fact that materials used are constantly exposed to radiation environment, which predominantly consists of various types of radiations. The well-established theory for blistering in metals may not remain stable in the case of dielectrics, insulators and polymers due to the fact that they are electrical and thermal insulators. This gives rise to a complex situation as compared to the metals. One of the major differences in the effect of ion bombardment in metals and insulators is the quenching of the deposited charge. In the case of metals, the deposited charge gets quenched immediately together with available free electrons, where as in insulators it does not occur as such. The energy deposited in polymers due to ion bombardment leads to chemical changes and breakage of chemical bonds of polymer thereby releasing gases, which are independent of the nature of ionizing radiation. The electrostatic charge deposited during ion bombardment may play an additional important role in polymers.[3]

In the present investigation, we have utilized Vicker's microhardness testing to study the mechanical behavior of the Kapton film. This testing has been found to be a non-destructive

Received June 2004, Accepted July 2004
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method. Kapton is one of the most important electrically insulating polymers as it has good ultimate tensile strength (UTS) and its working temperature ranges from liquid helium's temperature to 200°C in a continuous range. It is noteworthy that nowadays, polymers are included as nuclear materials as well as insulating materials in nuclear fusion reactors. Glass fiber filled (GFF) polyimide is a very promising material for super conducting magnet coil insulation in radiation environment.[4,5] Furthermore, in the nuclear reactor containment building, there are a large number of electrical components and scales made of polymeric materials, which may get radiation dose as high as 5×10^8 Rad.[6] It is desirable that they should remain unaffected even after exposure to such a high dose.

The present investigation is concerned with the study of the effect of 50 MeV Li^{3+} ions irradiation on the surface of kapton films by means of optical micrograph, scanning electron microscope, FTIR spectroscopy and Vickers' microhardness indentations.

Experimental

Two pieces of kapton [composition: $(C_{22}H_{10}N_2O_5)_n$; density 1.43 gm/cm^3] each of 70 μm thickness and 1.5 cm × 1.5 cm in dimension, were mounted on a metallic holder. The metallic holder was then placed in a general-purpose scattering chamber (GPSC) at a vacuum of 10^{-6} torr. The pieces of sample were exposed with 50 MeV Li^{3+} ion beam at the Nuclear Science Center, New Delhi, India. The current density of the beam was 23 nA/cm^2. The beam was defocused to 6 mm in diameter on the film. The irradiation was carried out at two fluences of 5.2×10^{13} and 10.4×10^{13} ions/cm^2, respectively.

The surface morphology variations on the kapton films due to irradiation were recorded using optical micrograph at 120X magnification and with scanning electron microscope (SEM). Functional group analysis for irradiated and pristine films was carried out by means of Fourier transform infrared (FTIR) spectroscopy (model 104-Bomem, Canada), in the wave number range 500 to 4000 cm^{-1}, with a resolution of 4 wave number. The Vickers' micro-hardness indentations have been carried out on the surface of irradiated and pristine films at room temperature. The hardness tests on each film were carried out at different loads. The Vicker's diamond pyramidal hardness, often designated as Hv is the quotient of the applied load divided by the pyramidal surface area of the impression and is given by the formula.

$$Hv = (189096 \times P)/d^2$$

where, P = load in mN and d = diagonal length in μm. The hardness Hv is obtained in MPa. The tests were repeated for pristine and irradiated films.

Results and Discussion

The projected range of 50 MeV Li^{3+} ion beam in kapton was calculated to be 396 μm using SRIM-97 code,[7] which is 5.65 times the thickness of kapton film. It is observed that 99.94% of energy is lost due to electronic interaction; the electronic stopping power of the beam $(dE/dx)_e$ is 7.224 eV/Å and nuclear stopping power of the beam $(dE/dx)_n$ is 0.0038 eV/Å. The energy deposited in the kapton film comes out to be 18.0 MeV.

Surface Morphology

The optical micrographs of pristine and irradiated kapton films are shown in Fig. 1. The optical micrograph of irradiated samples with a fluence of 5.2×10^{13} ions/cm^2

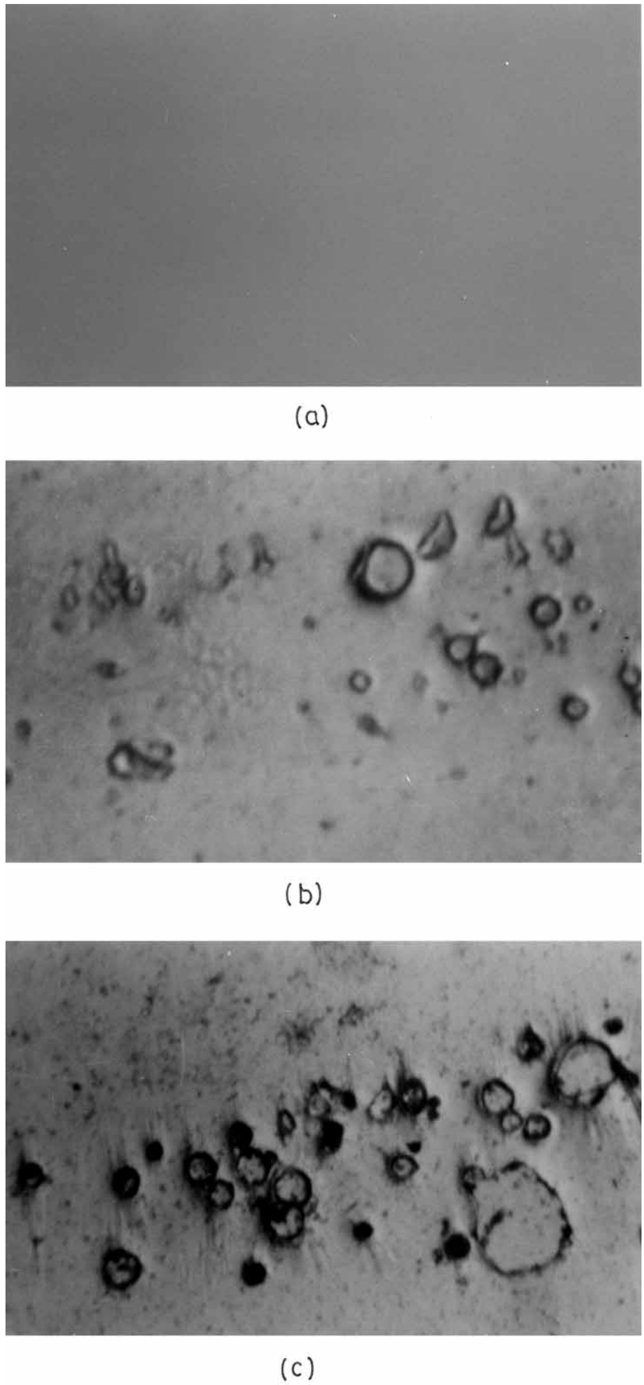
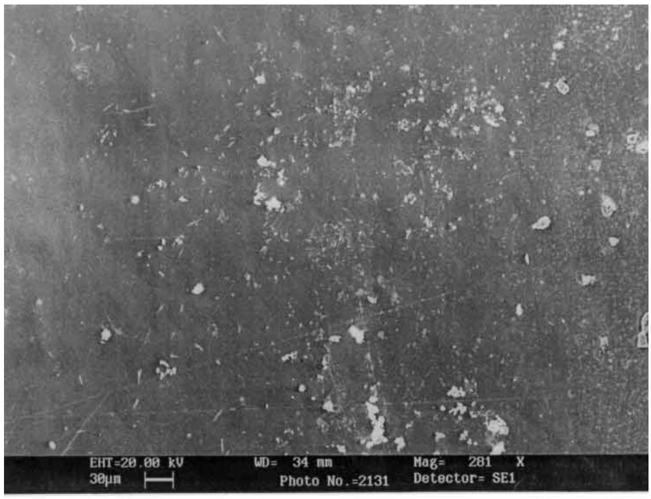
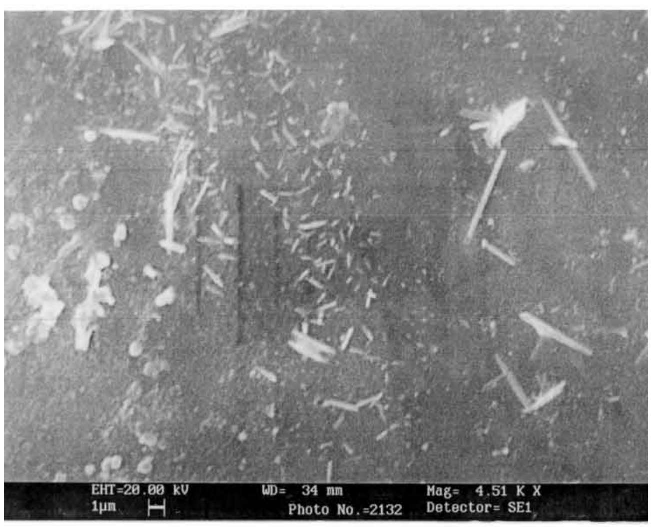


Figure 1. The optical micrograph of pristine and irradiated kapton films. (a) Pristine, (b) at the fluence of 5.2×10^{13} ions/cm^2, (c) at the fluence of 10.4×10^{13} ions/cm^2.

(Fig. 1b) and 10.4×10^{13} ions/cm^2 (Fig. 1c), clearly show blisters formation, whereas a smooth surface was observed on pristine film (Fig. 1a). It is also observed that there are more blisters at higher fluence. Further investigation of irradiated film with enhanced magnification was also recorded using scanning electron microscope (SEM). These micrographs clearly show the surface roughness, due to ion irradiation as shown in Fig. 2.



(a)



(b)

Figure 2. Scanning electron microscope (SEM) of irradiated kapton films.

FTIR Analysis

To investigate the reason for the formation of blisters on kapton surface, the FTIR studies were carried out for irradiated and pristine films in the wave number range 500 to 4000 cm^{-1}. The FTIR spectra of these films in the transmission mode are shown in Fig. 3. It is observed that there is no change in overall structure of the polymer, but minor change in intensities have been observed, which implied that inter-chain separation is not affected much by Li^{3+} ion irradiation. Most of the peak positions were found to be unshifted. Only the absorbance or transmittance value of the following functional group changed. They might be due as a result of a decrease in concentration of the preexisting bonds or groups.

The absorption bands, as obtained from the pristine spectrum, are identified as (A) 1723 cm^{-1}; C=O stretching vibration, (B) 3074 cm^{-1}; aromatic C–H stretching, (C) 3486 cm^{-1}; N–H bending vibration, (D) 3630 cm^{-1}; OH stretching vibration. The absorption bands at 1723, 3074, 3486, and 3630 cm^{-1} are selected for this analysis, since they have a relatively large absorbance. It is found that the absorption bond characteristics of all the above functional group exist even after the fluence of 1.04×10^{14} ions/cm^2. The minor decrease in the intensity of these functional groups (i.e., 1723, 3074, 3486, and 3630 cm^{-1}) are attributed to the emission of adsorbed gases just below the surface. It is inferred that the reduction in specific height is due to deterioration of these groups in the form of H_2, CO, or CO_2 gas. The release of H_2/CO/CO_2 gas results in the blisters formation on the surface of thermally stable kapton film. The minor changes in the peaks of the irradiated sample may be due to the breakage of one or two bonds in the ladder structure, but this will not change the overall structure of the polymer.[8,9]

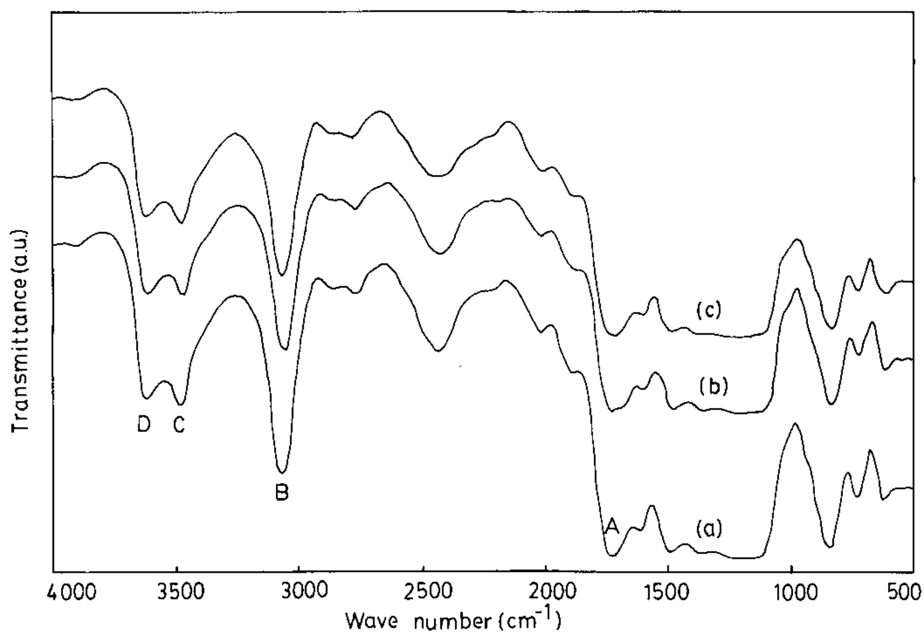


Figure 3. FTIR spectra of pristine (a) and irradiated kapton films at the fluence of 5.2×10^{13} ions/cm^2, (b); at the fluence of 10.4×10^{13} ions/cm^2 (c).

From these observations, it may be concluded that the kapton is highly resistant to radiation degradation.

Microhardness

Figure 4 shows the variation of the Vicker's microhardness (Hv) with a applied load (P) for pristine and irradiated films. It is evident from Fig. 4 that the Hv value increases with a load up to 300 mN and then saturates beyond the load of 400 mN. The increase in Hv with load can be explained on the basis of the strain hardening phenomenon. On applying the load, the polymer is subjected to some strain hardening. Finally, when Hv value tends to become constant, the polymer is completely strain hardened. The rate of strain hardening is greater at low loads and decreases at higher loads.[10–12] As can be seen, the hardness becomes independent of load for more than 400 mN. The value obtained from the saturation region, therefore, represents the true hardness of the bulk materials, since at high loads the indentor penetration depth is also high and surface effects become insignificant. It is also observed that the hardness increases as fluence increases. The increase in hardness, as a result of increase in fluence, can be assigned to the cross-linking phenomena affected by electronic stopping.[13]

Conclusion

The blisters formation on the surface of the kapton is attributed to the breakage of chemical bonds and the formation of low molecule gases. These gases are accumulated inside the polymer at a depth where maximum radiation damage takes place. When the pressure of accumulated gas crosses the mechanical strength of kapton film, it deforms and results as blisters. The emission of low molecular gases (i.e., CO and H_2) is also confirmed with the FTIR analysis of irradiated kapton films. The irradiation has been

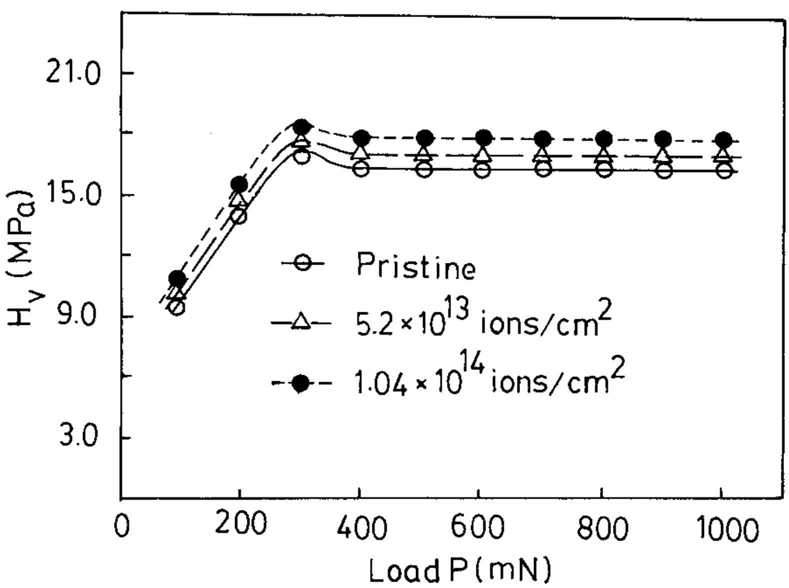


Figure 4. Plot of microhardness (Hv) vs. applied loads.

found to increase the Vickers' hardness of the kapton film significantly. The true bulk hardness of the film was obtained at loads greater than 400 mN.

Acknowledgments

The authors are grateful to Professor G. K. Mehta (Director, Nuclear Science Center, New Delhi, India) for his support and encouragement. The authors are also thankful to Dr. C. F. Desai for his help in this study.

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