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Microhardness and radiation damage studies of proton irradiated Kapton films

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Abstract

Kapton films were irradiated with a 3 MeV proton beam at different fluences. The microhardness, and electrical properties were studied using microhardness tester and LCR bridge, respectively. It is observed that hardness of the film increases significantly as fluence increases. The true bulk hardness of the film was obtained at loads greater than 400 mN. There is an exponential increase in the conductivity with frequency but effect of irradiation is not significant. The dielectric loss and dielectric constant are observed to change significantly due to irradiation.

Keywords: Kapton; Proton irradiation; Microhardness; AC frequency response

1. Introduction

Kapton (Polyimide) plays an ever-increasing role in microelectronics and aerospace due to its attractive dielectric properties, mechanical strength, excellent thermal stability and chemical properties. It is noteworthy that polymers are included as nuclear materials as well as insulating materials in nuclear-fusion (Thomas and Lindsay, 1991). The high-energy ion beam irradiation effects in Kapton have been investigated by many groups in different aspects. Ion beam-induced electrical conduction of Kapton film could find potential application for encapsulation of microelectronics and IC fabrication. Shrinkage effects of polyimide films under ion beam irradiation have been reported (Xu et al., 1991).There are also reports on physical and chemical aspects of ion irradiated polyimide films including infrared spectral analysis (Virk et al., 2001; Xu et al., 1991; Davenas and Thevenard, 1991).Temperature and frequency dependence of dielectric properties have also been studied using 50 MeV Si^+ ion (Garg et al., 2001). However, the effect of MeV ion beam irradiation on microhardness has not been reported so far. In the present work, we have studied the effects of 3 MeV proton beam irradiation of Kapton films. Vickers' microhardness of the films has been measured at different applied loads and ion fluences. AC electrical properties, viz., conductivity, dielectric constant and dissipation loss were studied in the frequency range 100 Hz to 100 KHz at room temperature.

2. Experimental

Three pieces of Kapton film with composition C_{22} H_{10} N_2 O_5 (density 1.43 gm/cm^3) and each of thickness 70 μm and size 1.5 cm × 1.5 cm were mounted on a metallic target holder. The samples were exposed to 3 MeV proton beam at Department of Physics, Panjab University, Chandigarh. The current density of the proton beam was 35 nA/cm^2. The beam was defocused to 6 mm diameter at the films. The irradiation was carried out at three fluences: 10^{13}, 10^{14} and 10^{15} ions/cm^2. The Vickers' microhardness indentation was carried out on pristine and irradiated films at different loads in the range of 100–1000 mN at room temperature using Carl Zeiss Microscope and its accessories.

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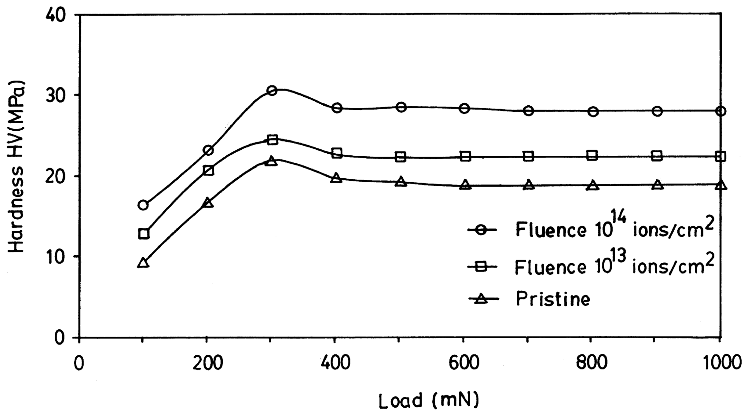

Fig. 1. Plot of hardness (HV) versus applied load (P) at the different fluences.

The capacitance and the dissipation factor (tan δ) of the samples were measured simultaneously with the variable frequency LCR bridge in the frequency range 100–100 kHz (General Radio, USA, model 1689).

3. Results and discussion

The projected range of 3 MeV proton beams in Kapton was calculated to be 110.2 μm using SRIM-97 code (Ziegler, 1997), which is 1.57 times the thickness of the Kapton sample. It is observed that 99.94% of energy lost by a 3 MeV proton in 70 μm thick Kapton is electronic in nature; the electronic stopping power $(dE/dX)_e$ is 1.3×10^{-1} eV/Å and nuclear stopping power $(dE/dX)_n$ is 7.66×10^{-5} eV/Å. The energy deposited in the medium turns out to be 2.3 MeV.

3.1. Microhardness

Fig. 1 gives the plots of the Vickers' microhardness (HV) versus applied load P, at different fluences. It is evident that the value increases with load up to 300 mN and then saturates beyond the load of 400 mN. The increase of HV with load can be explained on the basis of the strain-hardening phenomenon. Applying the load, the polymer is subjected to some strain hardening. Beyond certain load the polymer exhausts its strain hardening capacity and the hardness tends to become constant. The rate of strain hardening is greater at low loads and decreases at higher loads (Singh et al., 2002; Awasthi and Bajpai, 2001; Lee et al., 1997). 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 material, 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.

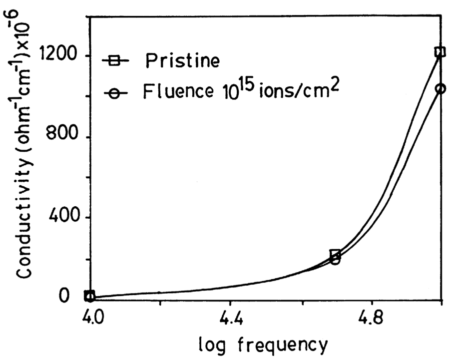

Fig. 2. Variation in AC conductivity with log frequency of pristine and proton irradiated Kapton films.

3.2. AC Electrical Frequency Response

Fig. 2 shows the variation of conductivity with log frequency for the pristine and irradiated samples. A sharp increase in conductivity at 50 KHz has been observed in both the cases, with a lower magnitude for the irradiated sample at the fluence of 10^{15} ions/cm^2. The decrease in conductivity as a result of irradiation may be attributed to charge centres created due to scissioning of polymeric chains. Charge centers oscillate giving rise to circulatory currents under the influence of an externally applied voltage. Slow vibrations arise due to large mass of positive charge carriers whereas free electrons generate circulatory currents. These circulatory currents develop an inductive behavior in the polymeric dielectric, which may be responsible for the observed small decrease in the conductivity (Srivastava and Virk, 2000).

Fig. 3 shows the plot of tan δ versus log frequency for the pristine and irradiated samples. It is observed that loss factor (tan δ) increases moderately as frequency increases up to 10 KHz. It is also observed that the loss factor increases as

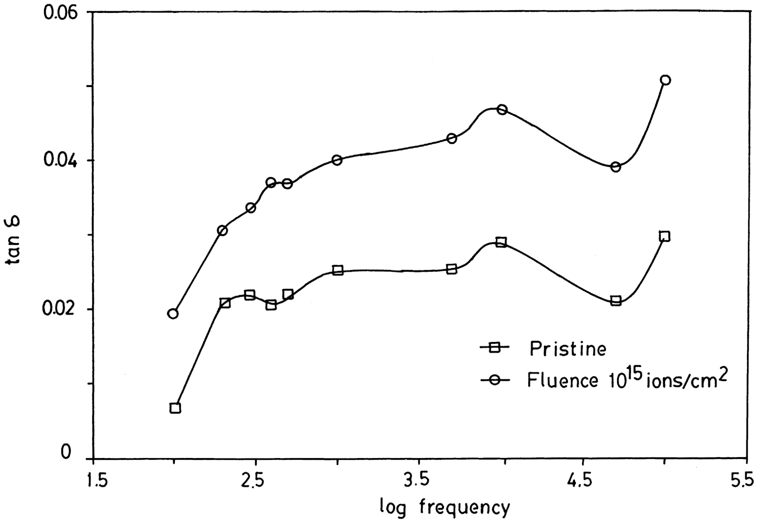

Fig. 3. Variation of tan δ with log frequency for pristine and proton irradiated Kapton films.

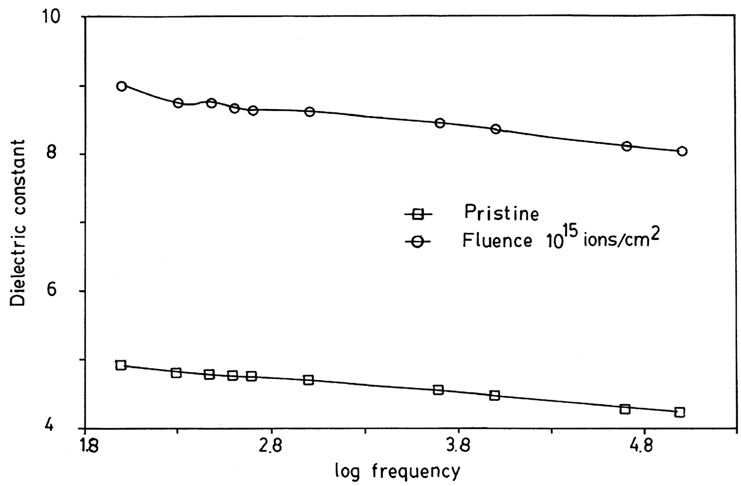

Fig. 4. Plot of dielectric constant versus log frequency for pristine and proton irradiated Kapton films.

the fluence increases. tan δ has positive values indicating the dominance of inductive behavior.

Fig. 4 shows the variation of dielectric constant with log frequency for the pristine and irradiated samples. As evident from the graph the dielectric constant remains almost constant over a wide frequency range and increases as the fluence increases. It is an interesting observation because lot of Kapton-based capacitors are used in nuclear plants and bound to be exposed to nuclear radiation.

4. Conclusion

Proton irradiation has been found to increases the Vickers' hardness of the Kapton films. The decrease in AC conductivity with frequency due to irradiation may be attributed to charge centers created due to scissioning of polymeric chain.

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