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

# EFFECT OF ION BEAM IRRADIATION ON HARDNESS OF POLYETHYLENE TEREPHTHALATE

N. L. Singh[a]; C. F. Desai[a]; N. Shah[a]; P. H. Soni[a]; V. Shrinet; D. K. Avasthi
[a] Physics Department, The M.S. University of Baroda, Vadodara, India

Online publication date: 24 June 2002

**To cite this Article** Singh, N. L. , Desai, C. F. , Shah, N. , Soni, P. H. , Shrinet, V. and Avasthi, D. K.(2002) 'EFFECT OF ION BEAM IRRADIATION ON HARDNESS OF POLYETHYLENE TEREPHTHALATE', Journal of Macromolecular Science, Part A, 39: 3, 231 — 235

**To link to this Article: DOI:** 10.1081/MA-120013262
**URL:** http://dx.doi.org/10.1081/MA-120013262

J. MACROMOL. SCI.—PURE APPL. CHEM., A39(3), 231–235 (2002)

NOTE

# EFFECT OF ION BEAM IRRADIATION ON HARDNESS OF POLYETHYLENE TEREPHTHALATE

**N. L. Singh,[1] C. F. Desai,[1,]* N. Shah,[1] P. H. Soni,[1] V. Shrinet,[2] and D. K. Avasthi[3]**

[1]Physics Department, The M.S. University of Baroda, Vadodara 390002, India
[2]Insulation Division, ERDA, Makarpura, Vadodara 390010, India
[3]Nuclear Science Center, Aruna Ashaf Ali Marg, New Delhi 110067, India



## ABSTRACT

Ion beam irradiation as one of the effective methods of polymer modification has been used on the PET films. The ion beam used was $Li^{+3}$(50 MeV) of various fluences. The Vickers' hardness of the films irradiated at different ion fluences was measured. The hardness tests on each film were carried out at different loads. The true bulk hardness of the film is obtained at loads greater than 400 mN. The irradiation has been found to significantly increase the hardness of the film. The increase follows a linear trend with the fluence and has been explained in terms of crosslinking effected by electronic stopping mode of the ions.

*Key Words*: Ions; Irradiation; Hardness; Scission; Crosslinking; Fluence

*Corresponding author.

## INTRODUCTION

Ion beam irradiation is one of the well-established tools for modifying the structural and physical properties of materials. It has been widely used particularly in the field of polymer modifications. There have been numerous reports on radiation induced electrophysical and electrochemical changes of polymers [1–4]. That the strength or hardness also changes as a result of suitable irradiation is no less significant than the other modifications. PET together with other important polymers like PMMA, PI, PES, etc., have been a subject of interest in this regard.

Metallized polymers and especially PET, are widely used for different applications like packaging, protective coating, capacitors, magnetic tapes etc. In such applications a good adhesion between the polymer and the metal is required. In packaging, for example, the polymer films provide the mechanical strength, whereas the metal layer, generally aluminum, acts as the gas diffusion barrrier.

Hardness as an important strength measure may play a crucial role when it comes to usage of the polymer in the actual application field. It is also a general macroprobe for assessing the bond strength apart from being a measure of the bulk strength. The authors have not come across any report on hardness modification of PET by ion beam irradiation. We report here the results of hardness tests on pristine and $Li^{+3}$ (50 MeV) irradiated PET films. The hardness variations with ion beam fluence and also with a applied test load have been studied.



## EXPERIMENTAL

The PET films of thickness 230 μm, used in this study were obtained from a commercial sheet. The irradiation of samples was carried out in a general-purpose scattering chamber (GPSC) using 50 MeV $Li^{+3}$ beam. For hardness tests, the indentor employed was the Vickers' pyramidal diamond indentor supplied with the microhardness testing accessory of Vickers' Projection Microscope. The hardness indentations were carried out on the surfaces of pristine and irradiated films at room temperature under different applied loads from 10 mN to 1000 mN and constant loading time of 30 seconds. The measurement of the indentation diagonal length was made to an accuracy of 0.190 μm (using a micrometer eyepiece precalibrated against the standard graticule supplied with the hardness tester), and the load was determined to any accuracy of 2.5 mN.

The Vickers' diamond pyramidal hardness, often designated as $H_v$ is the quotient of the applied load divided by the pyramidal surface area of the impression and is given by the formula:

$$H_v = \frac{1854 \times P}{d^2}$$

where; P = load in mN and d = diagonal length in μm.

The Vickers' hardness $H_v$ is obtained in MPa. The tests were repeated on films irradiated with different fluences of the ions.

## RESULTS AND DISCUSSION

Since hardness has to be tested with an applied load, it is important to note that the hardness may be a function of load though it should not be so as implied by the above equation, giving $P = d^2$ which is known as Kick's law. The load dependence of hardness amounts to the law modified to $P = d^n$ where n, known as Meyer index, may differ from 2. There have been numerous studies [5–7] reporting $n > 2$, particularly in the so-called low load range. Most of these reports are on metals, alloys, semiconductors or inorganic compounds in either single crystalline or polycrystalline forms. No such reports on polymer have so far been found. Hence, it is desirable that for the material under test, the tests should cover a sufficient load range appropriate to the material.

Figure 1 shows the plots of measured Vickers' hardness $H_v$ as a function of applied load P for the pristine and irradiated PET films, the irradiations being of different fluences indicated in the figure. As can be seen from the graph, the hardness becomes independent of load for loads more than 400 mN. The values obtained from the saturation region, therefore, represent the true hardness values of the bulk material since at high loads the indentor penetration depth is also high and surface effects become insignificant. With an increase in fluence, the hardness is found to increase from 335 MPa for

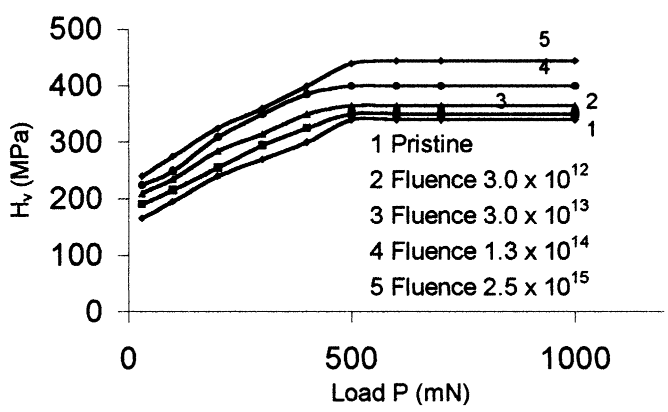


***Figure 1.*** Plot of $H_v$ vs. applied load P at various fluences mentioned (in ions/sq. cm).

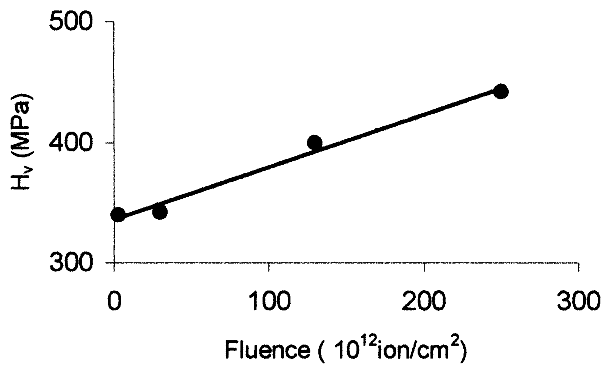


***Figure 2.*** The plot of $H_v$ vs. fluence.



pristine film to 442 MPa (about 40% increase) for films irradiated at the fluence of $2.5 \times 10^{14}$ ions/cm$^2$. This increase shows nearly a linear dependence on the fluence as shows in Fig. 2.

The ion irradiation of a polymer is known to affect its hardness. The incident ion can undergo energy loss upon its passage through the polymer by two mechanisms, namely, electronic stopping and nuclear stopping. The nuclear stopping involves the energy loss by displacing atoms in the medium as a result of nuclear collisions. These collisions are most effective when the incident ions are heavy. The nuclear collision causes the release of pendant atoms in the polymer resulting into bond breakage. This phenomenon is known as chain scission. The scission has been reported to degrade mechanical strength [8]. However, in the present study the ions used are relatively light having small nuclear collision cross section and hence the nuclear stopping would be insignificant. The other important mechanism is the electronic stopping in which the stripped ion re-acquires its orbital electrons as it also creates a large number of secondary electrons. These energy loss processes contribute to the electronic stopping. While the nuclear stopping causes more scissions, the electronic stopping causes more crosslinking that occurs whereby two free dangling bonds on neighboring chains unite [9].

Hence, the observed increase in hardness as a result of increasing fluence can be assigned to the crosslinking phenomenon affected by electronic stopping.

## CONCLUSION

$Li^{3+}$ (50 MeV) irradiation has been found to increase the Vickers' hardness of the PET films showing a linear dependence on the fluence.

## REFERENCES


1. Picq, V.; Balanzat, E. Ion Induced Molecular Emission of Polymers: Analytical Potentialities of FTIR and Mass Spectroscopy. Nucl. Instru. Methods Phys. Res. B **1999**, *151* (1–4), 76–83.
2. Picq, V.; Ramillon, J.M.; Balanzat, E. Swift Heavy ions on Polymers: Hydrocarbon Gas Release. Nucl. Instru. Methods Phys. Res. B **1998**, *146* (1–4), 496–503.
3. Apel, P.; Sphor, R.; Trautmann, C.; Vutsadakis, V. Track Struc. in PET Irradiated by Heavy Ions: LET Dependence of Track Diameter. Radiat. Meas. (UK) **1996**, *31* (1–6), 51–56.
4. Mishra, R.; Tripathi, S.P.; Sinha, D.; Dwivedi, K.K.; Ghosh, S.; Khathing, D. T.; Miller, M.; Fink, D.; Chung, W.H. Optical and Electrial Properties of Some Electron and Proton Irradiated Polymers. Nucl. Instru. Methods Phys. Res. B **2000**, *168*, 59–64.
5. Boyarskaya, Yu. S.; Grabko, D.Z.; Dintu, M.P. Correlation Between Microhardness and Dislocation Mobility in Crystals with a Different Type of Bond. Cryst. Res. Tech. **1981**, *16*, 441–448.
6. Buckle, H. The Science of Hardness Testing and its Research Applications. Westbrook, J.H., Conrad, H., Eds.; ASM: Ohio, 1973; p. 453.
7. Desai, C.F.; Soni, P.H.; Bhavsar, S.R.; Microhardness of Sb-, Sn- and Se-Doped $Bi_2Te_3$ Single Crystals. Ind. J. Pure and Appl. Phys. **1999**, *37*, 119–121.
8. Cowie, J.M. *Polymers: Chemistry and Physics of Modern Materials*; Billing & Sons: Worcester, GB, 1973; p. 283.
9. Lee, E. H.; Rao, G.R.; Mansur, L.K. Hardness Enhancement and Crosslinking Mechanisms in Polysterene Irraidiated with High Energy Ion-Beams. Mater. Sci. Forum **1997**, *248–249* (9) 135–146.


Received April 20, 2001