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Tailoring of optical band gap and refractive index of heat treated Kapton-H Polyimide

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ABSTRACT

The samples of Kapton-H polyimide were heated at temperatures ranging from 325 to 400 °C in air. The heat treatment induced changes in optical band gap and refractive index of Kapton-H have been studied through UV-visible spectroscopy. The reduction in optical band gap while an increase in refractive index have been observed after heat treatment. The observed optical changes have been tried to be correlated to the structural changes, revealed through FTIR spectroscopy. The present study is quite important for tailoring the optical response of Kapton-H polymer as per specific requirements.

Keywords: Kapton-H, Heat treatment, Optical band gap, Refractive index.

INTRODUCTION

In recent days, the polymeric materials have been progressively substituted the expensive inorganic materials for integrated-optical components due to their exclusive advantages such as light weight, versatile electronic properties, low manufacturing cost and simple handling and processing. Further, the optical properties like optical band gap and refractive index of the polymers can be tuned as per specific requirements for various optical applications through several treatments like doping, irradiation, heat treatment etc. [1-6]. In our previous work [7], we have studied the heat treatment induced changes in the optical band gap and refractive index of CR-39 polymer. Motivated from our previous study, in the present work, we have planned to carry out the similar studies on Kapton-H.

Kapton-H is a high performance material having the cyclic imide and aromatic groups in the main chain (Figure 1). This polymer is widely acknowledged for its plenty of applications in microelectronics (in packaging and fabrication of integrated circuits), aerospace and optoelectronic devices [8-13]. Low out-gassing rate makes it suitable insulator for ultra high vacuum environment. With a good combination of thermal conductivity, dielectric strength and availability in thin films, it is well suited material in cryogenics.

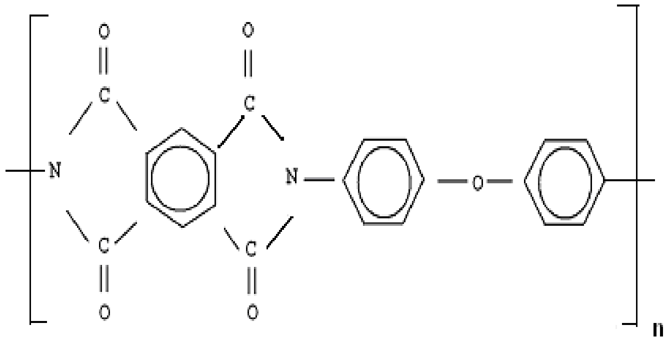

Figure 1. The chemical structure of Kapton-H polyimide.

MATERIALS AND METHODS

Experimental

Kapton-H (DuPont) samples were cut (2 cm x 2 cm) from the flat sheet of thickness 100 μm. These samples were subjected to heat treatment in air for one hour at various temperatures (325, 350, 375 and 400 °C) in a specially designed oven within an accuracy of ±2 °C. All these samples were subjected to UV-Visible transmission and reflection studies by using Shimadzu Double beam Double Monochromator UV-VIS Spectrophotometer (UV-2550) in the wavelength range 190–900 nm with a resolution of 0.5 nm. All these spectra were recorded by mounting the samples appropriately in the Integrated Sphere Assembly ISR-240A attached to the spectrophotometer. FTIR spectra were recorded using Shimadzu IR-Affinity instrument equipped with the Specular Reflectance attachment.

RESULTS AND DISCUSSION

3.1 UV-Visible studies

In order to investigate the optical changes in the virgin and heat treated samples of Kapton-H, the UV-visible transmission and reflection spectra were recorded in the wavelength region 190 – 900 nm. Suitable correction was made in order to eliminate the contribution of the sample holder. As the attenuation of the transmitted light through the sample involves the absorption within the sample and the reflection losses at the sample-air interfaces (top and bottom), therefore the measured transmission (T) and reflection (R) values have to be corrected for the reflection losses at these interfaces [7, 14]. Accordingly, the transmission coefficient (t) for light for a single pass through the sample and reflection coefficient (r) at sample-air interface can be determined through the following expressions

$$t = \frac{2T}{(1-r)^2 + \sqrt{(1-r)^4 + 4T^2 r^2}} \qquad (1)$$

$$r = \frac{2R}{1 + t^2 + \sqrt{(1+t^2)^2 - 4t^2 R(2-R)}} \qquad (2)$$

After substituting the values of T and R from the recorded spectra in these equations and following the iterative process, the values of 't' and 'r' have been determined for virgin and heat treated samples of Kapton-H.

3.1.1 Determination of absorption coefficient

The values of absorption coefficient (α) for virgin and heat treated Kapton-H samples have been determined using the relation

$$t = \exp(-\alpha d) \qquad (3)$$

where d is the thickness of the sample.

The values of absorption coefficient (α), so determined, have been plotted as a function of wavelength (λ) and presented in Figure 2. It is clearly observable from this figure that for virgin sample a strong absorption upto a wavelength ~ 500nm followed by a considerable tailing exist. The observed strong absorption may be attributed to the presence of chromophores like aromatic rings, C=C and C=O double bonds [15] in this polymer. Further, it is evident from this figure that there is a continuous red shift of the absorption edge with rise in heating temperature.

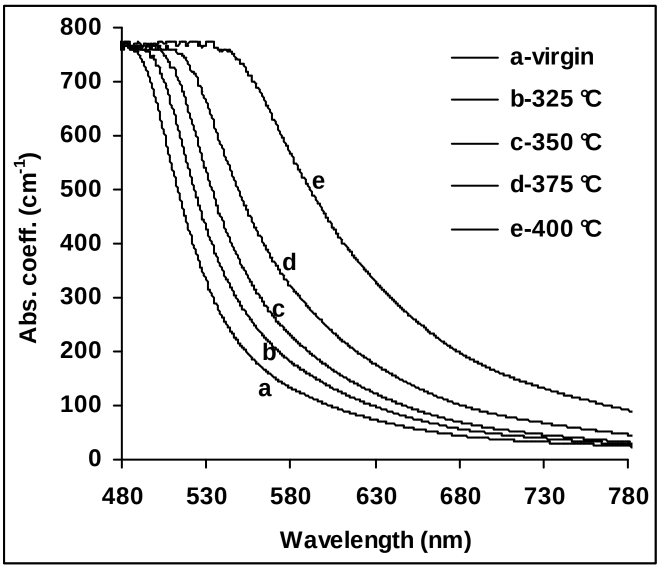

Figure 2. Plots of absorption coefficient (α) vs. wavelength (λ) for virgin and heated treated Kapton-H.

3.1.2 Determination of optical band gap

In order to determine the optical band gap (E_g) of virgin and heat treated samples of Kapton-H, the absorption data corresponding to the fundamental absorption edge as a function of wavelength (λ) have been considered [16]. These data have been plotted in the form of $(\alpha h\upsilon)^{1/2}$ as a function of photon energy (hυ). The band gap values, determined from the intercepts of the plots of $(\alpha h\upsilon)^{1/2}$ versus (hυ) on hυ axis (Figure 3), have been presented in Table 1. It is obvious from this table that the optical band gap decreases continuously from ~2.04 eV in virgin sample to ~1.52 eV for the sample heated at 400 °C.

The observed red shift (Figure 2) and the reduction in optical band gap (Table 1) with increase in heating temperature may be attributed to the formation of a conjugated structure having delocalized π-bonded electrons as a result of heating of Kapton-H [17].

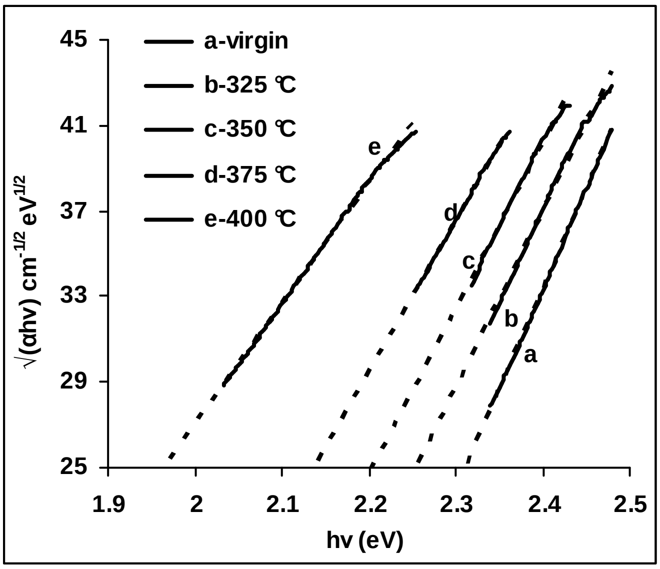

Figure 3. Plots of $(\alpha h\nu)^{1/2}$ vs. energy (hν) for virgin and heated samples of Kapton-H.

Table 1. Optical band gap (E_g) values for virgin and heat treated Kapton-H polyimide samples at different temperatures

Heating temperature (°C)	E_g (eV)	R^2
virgin	2.04 ± 0.01	0.99
325	1.95 ± 0.01	0.99
350	1.88 ± 0.01	0.99
375	1.78 ± 0.01	0.99
400	1.52 ± 0.01	0.99

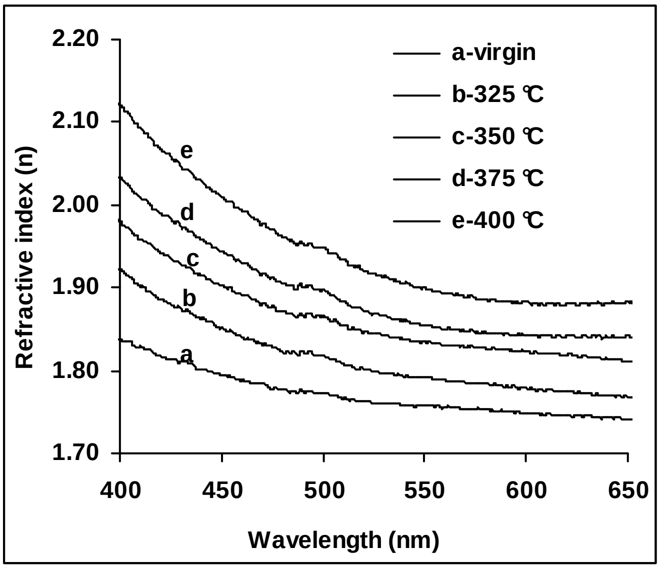

Figure 4. Plots of refractive index (n) vs. wavelength (λ) for virgin and heat treated samples.

3.1.3 Determination of refractive index

The refractive index (n) values for virgin and heat treated Kapton-H samples at different wavelengths have been determined [17] using the relation

$$n=\left\{\left[\frac{4r}{(r-1)^2}-K^2\right]^{\frac{1}{2}}-\frac{r+1}{r-1}\right\} \quad (4)$$

where $K=\dfrac{\alpha\lambda}{4\pi}$, is the extinction coefficient with α as the absorption coefficient at wavelength λ. The values of the refractive index (n) so determined at different wavelengths have been presented in Figure 4. From this figure, it is clear that the values of n rise continuously with increase in heating temperature, which may be attributed to the compaction of the material due to removal of some species [17, 18] like hydrogen, oxygen etc. after heat treatment.

3.2 Structural Studies
3.2.1 FTIR Analysis
The FTIR spectra of virgin and samples heated at 350° C and 400° C have been presented in Figure 5. The identification of the characteristic peaks in virgin sample (Table 2) made on the basis of literature [15, 19-20], confirms the monomer structure of Kapton-H (Figure 1).

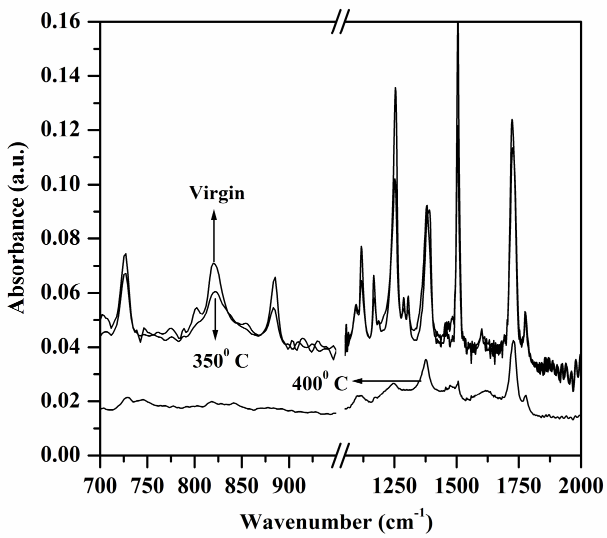

Figure 5. FTIR spectra of virgin and heat treated samples of Kapton-H.

In the FTIR spectrum of the sample heated at 350° C, the decrease in the intensity coupled with the broadening of the various characteristic peaks have been observed. For sample heated at 400° C, most of the characteristic peaks in the region 700-1100 cm^{-1} have disappeared indicating the removal of hydrogen like species. The drastic reduction in the intensities of the bands at 1776, 1722 (C=O, stretching vibrations), 1390 (C-N, stretch), 1505 cm^{-1} (C=C, stretch in phenyl rings) may be due to the fact that the imide and aromatic structures of Kapton are destroyed. The appearance of a broad band in the region 1200-1500 cm^{-1} may be attributed to the formation of a compact carbonaceous structure in the polymer matrix after heat treatment [20].

Further, the broadening and shifting of the peak observed at 1600 cm^{-1} (aromatic C=C stretching) in virgin sample to 1620 cm^{-1} indicates the increase in conjugation as a result of heating. Such structural changes as revealed through FTIR spectroscopy corroborate the observations from optical measurements.

Table 2. Identification of IR bands of virgin Kapton-H

Band Position (cm^{-1})	Identification of Bands
1776, 1722	s
1600, 1505	Aromatic C=C stretching
1463	Skelton vibration of phenyl ring
1390	C-N stretching
1254	Asymmetric stretching of aromatic ether C-O-C
1169, 1117	The in plane hydrogen rocking of para-substituted phenyl
882	Out of plane wagging vibration of the isolated hydrogen in (1, 2, 4, 5)- tera substituted phenyl between the imide rings
820	Out of plane wagging vibration of two adjacent hydrogen of para-substituted phenyl
727	Aromatic imide ring band

CONCLUSION

The observed variation in optical parameters such as optical band gap and refractive index of Kapton-H may lead to tailor the optical properties of this polymer as per specific requirements after heat treatment. To our knowledge, this is probably the first systematic study to report the variation in refractive index of Kapton-H in almost complete visible range as a result of heat treatment.

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REFERENCES

[1] E.H. Lee, *Nucl. Instrum. Methods B,* **1999,** 151, 29.
[2] M.A. El-Shahawy, *Polym. Deg. Stab.*, **1997**, 57, 157.
[3] M.F. Zaki, *J. Phys. D: Appl. Phys.*, **2008**, 41, 175404.
[4] A.M. Shaban, *Mater. Lett.*, **1995**, 25, 179.
[5] W. Wang, X. Yang, Y. Fang, J. Ding, J. Yan, *Appl. Energy,* **2009**, 86, 1479.
[6] A. M. Visco, L. Torrisi, N. Campo, A. Picciotto, *Int. J. Polym. Anal. Charact.*, **2010**, 15, 73.
[7] V. Kumar, P. K. Goyal, S. Mahendia, R. Gupta, T. Sharma, S. Kumar, *Radiat. Eff. Defects Solids*, **2011**, 166, 109.
[8] S. J. Lee., C. S. Ha, J. K. Lee, *J. Appl. Polym. Sci.*, **2001**, 82, 2365.
[9] V. Malba, V. Liberman, A. F. Bernhardt, *J. Vac. Sci. Technol. A,* **1997**, 15, 844.
[10] T. Miwa, *J. Photopolym. Sci. Technol.,* **2001**, 14, 29.
[11] C. P. Wong, *Mater. Chem. Phys.*, **1995**, 42, 25.
[12] S. Xie, Z. Zhang, W. Wei, *J. Korean Phys. Soc.*, **2007**, 51, 1536.
[13] R. Yokota, *J. Photopolym. Sci. Technol.*, **1999**, 12, 209.
[14] X. W. Zhang, *J. Mater. Sci.*, **2001**, 36, 1957.
[15] Y. Sun, Z. Zhu, Y. Jin, C. Liu, Z. Wang, J. Liu, M. Hou, Q. Zhang, *Nucl. Instrum. Methods B*, **2002**, 193, 214.
[16] D. Fink, (Ed.); Fundamentals of Ion-Irradiated Polymers, Springer-Verlag Berlin Heidelberg, Germany, **2004**.
[17] M. D. Migahed, H. M. Zidan, *Current Appl. Phys.*, **2006**, 6, 91.

[18] J. P. Biersack, R. Kallweit, *Nucl. Instrum. Metods B,* **1990**, 46, 309.
[19] J. Megusar, *J. Nucl. Mat.,* **1997**, **245**, 185.
[20] K. Sahre, K. –J. Eichhorn, F. Simon, D. Pleul, A. Janke, G. Gerlach, *Surf. Coat. Technol.,* **2001**, 139, 257.