

Damage threshold determination of bulk polymer samples using pulsed photothermal deflection technique

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Abstract. Photothermal deflection technique was used for determining the laser damage threshold of polymer samples of teflon (PTFE) and nylon. The experiment was conducted using a Q-switched Nd-YAG laser operating at its fundamental wavelength ($1.06\ \mu\text{m}$, pulse width 10 ns FWHM) as irradiation source and a He-Ne laser as the probe beam, along with a position sensitive detector. The damage threshold values determined by photothermal deflection method were in good agreement with those determined by other methods.

Keywords. Photothermal deflection technique; damage threshold; polymer, Nd-YAG laser; He-Ne laser.

1. Introduction

Damage threshold studies of polymers have gained considerable significance in recent years because of their applications in photolithography (Dyer and Sidhu 1985; Srinivasan *et al* 1986; Sell *et al* 1989; Srinivasan and Braren 1989), in the choice of optical components in laser systems (Milam 1977; Dyumaev *et al* 1983) and in the selection of polymer-based nonlinear optical elements (Lipscomb *et al* 1981). There are various methods like surface morphological studies, visual observation of plasma emission from the target and reflectivity variation studies from the target to evaluate the laser-induced damage threshold of materials. Techniques based on photoacoustic effect have proved to be very effective in determining the laser damage thresholds of both transparent and opaque samples (Rosencwaig and Willis 1980). The present paper describes the use of transverse photothermal deflection technique (TPTD) to evaluate damage threshold of bulk polymer samples of teflon (PTFE) and nylon.

2. Photothermal deflection process

Absorption of laser radiation (pump beam) by a sample surface generated heat due to various non-radiative de-excitation processes occurring in the sample. The heat thus generated was transferred to the surrounding medium in close vicinity of the irradiated surface resulting in a temperature increase of the former. This increase in temperature led to density variations which brought about a refractive index gradient in the medium adjacent to the surface. A probe laser beam propagating through this refractive index gradient perpendicular to the direction of the pump beam suffered refraction and consequently deviated from its original path (figure 1) corresponding to ambient condition. The effect was termed as transverse photothermal deflection (TPTD). The magnitude of the beam deflection depended on the amount of heat transferred from the sample to the medium. Such heat transfer depended strongly on

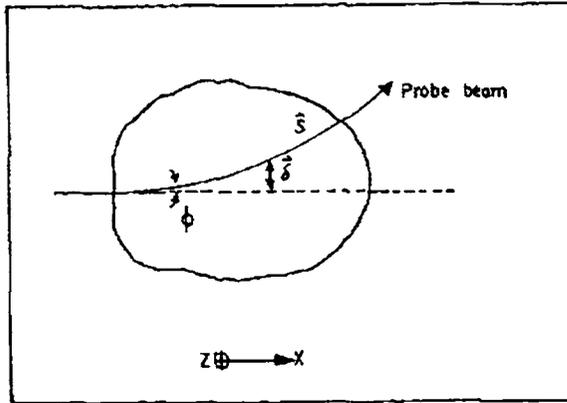


Figure 1. Deflection of the probe beam propagating in an inhomogeneous medium due to mirage effect.

the thermal processes induced on the surface by laser beam and generally it was found to increase for pump energies above the optical breakdown. As a result, at laser fluences above the damage threshold a noticeable enhancement in PTD was found.

The propagation of an optical beam (probe) in an inhomogeneous medium was governed by the equation

$$d/ds(n_0 d\delta/ds) = n(r, t), \quad (1)$$

where s was the beam path, δ the perpendicular displacement from the original path which depend on the angle of deflection ϕ and the position of the detector and $n(r, t)$ the gradient of refractive index perpendicular to the beam path. If n_0 , T_A and ρ_A were the refractive index, temperature and density respectively under ambient conditions, the perturbed refractive index was written as

$$n(r, t) = n_0 + \partial n / \partial T |_{T_A} T(r, t) + \partial n / \partial \rho |_{\rho_A} \rho(r, t) \quad (2)$$

and

$$d\delta/ds = 1/n_0 \partial n / \partial T \int_{\text{path}} \nabla_{\perp} T(r, t) ds. \quad (3)$$

For small deflections we can write $\phi(x, y, t) = d\delta/ds$ where ϕ is the angle of deflection. Therefore (3) may be written as

$$\phi(x, t) = 1/n_0 \partial n / \partial T \int \partial T / \partial x(x, y, t) dy, \quad (4)$$

where $T(x, y, t)$ was the temperature distribution created by the heated sample surface. The details of solving (4) were given by Tam (1986) and Rose and Gupta (1986). Since the probe beam profile was gaussian, the beam deflection was measured using a position sensitive detector (PSD).

To compute the thermal energy of a heated region by processing a detector signal one has to correlate the temperature distribution of the investigated region with the optical beam propagation through the adjacent non-homogeneous medium and the detector response. The theoretical calculation of the probe beam deflection was verified using quadrant detector as the PSD (Jackson et al 1980). In the present investigation

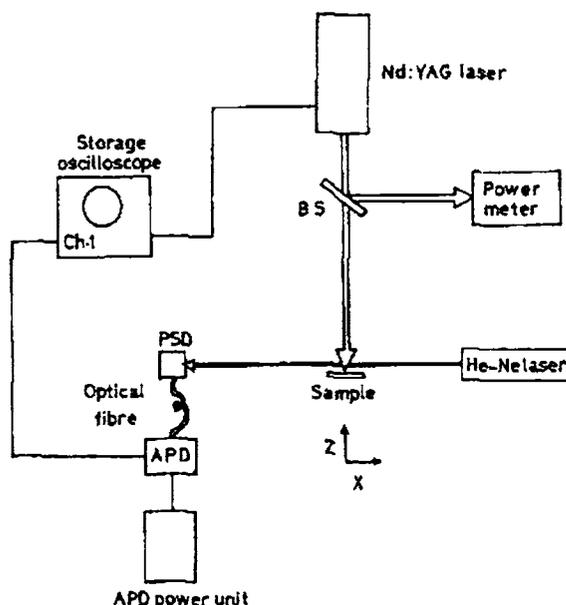


Figure 2. Schematic diagram of the experimental set-up.

the polished tip of an optical fibre coupled to an avalanche photodiode (APD) acts as the position sensitive detector (Rajasree *et al* 1990).

3. Experimental technique

The schematic diagram of the experimental set-up for the determination of the damage threshold of the polymer samples using the PTD technique is given in figure 2.

The sample in the shape of a disc of 205 cm dia and 0.55 cm thickness was mounted on a micropositional XYZ translator. The pump beam used to irradiate the surface was 1.06 μm radiation from a Q-switched Nd-YAG laser (Quanta ray DCR11). A short focal length convex lens focussed the pump laser beam on to the sample surface. The laser fluence incident on the sample surface was varied by adjusting the position of the lens in front of the sample. A 5 mW He-Ne laser beam passing parallel to and grazing the sample surface was used as the probe beam. A fibre optical sensor which acts as the position sensitive detector (PSD) (Rajasree *et al* 1990) located at about 50 cm away from the sample measured the magnitude of the probe beam deflection. A 100 MHz storage oscilloscope (Tektronix 466) coupled to the PSD recorded the transient deflection. The laser energy was monitored for each pulse using a pulsed energy meter (Delta developments) triggered in synchronization with the laser pulse.

4. Results and discussion

A typical oscilloscope trace of the PTD signal recorded for nylon is shown in figure 3. The peak-to-peak value of the signal was taken as the signal amplitude. Damage threshold values evaluated for nylon and teflon using PTD technique are given in

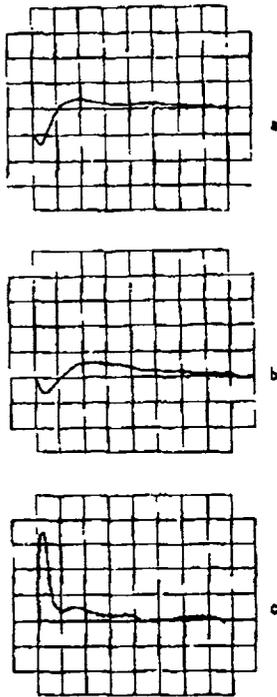


Figure 3. Oscilloscope trace of the signal from teflon at (a) 1.884 mJ (0.2 V/div.; 1 ms/div.), (b) 2.3 mJ (0.1 V/div.; 1 ms/div.) and (c) 3.35 mJ (0.1 V/div.; 1 ms/div.).

Table 1. Energy densities at regions A and B for nylon and teflon.

| Sample | Region (A) J/cm ² | | Region (B) J/cm ² | |
|--------|---------------------------------|-------------------|---------------------------------|-------------------|
| | Present method | Alternate method* | Present method | Alternate method* |
| Nylon | 1.8 | 1.53 | 2.5 | 2.25 |
| Teflon | 2.2 | 1.78 | 3.25 | 2.85 |

Estimated Error ~ 20%. *Ravi Kumar et al (1991)

table 1. For comparison the results obtained with alternate methods (Ravi Kumar et al 1991) are also included here. These values showed close agreement with the results obtained from the PTD measurement.

Figures 4 and 5 show the plot of the measured signal amplitude against the energy density of the pump beam incident on the sample and these graphs exhibit two distinct regions of different slopes corresponding to two different kinds of thresholds for the laser-induced surface damage (Harada et al 1989; Ravi Kumar et al 1991). An abrupt increase in signal amplitude was found to occur in the regions near the threshold in agreement with earlier observations (Rosencwaig and Willis 1980; Srinivasan and

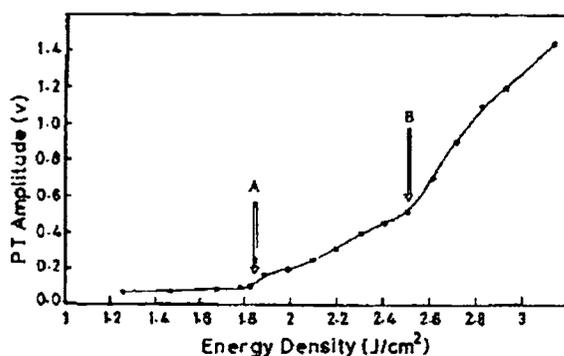


Figure 4. Plot of laser energy density vs PT amplitude for nylon in air.

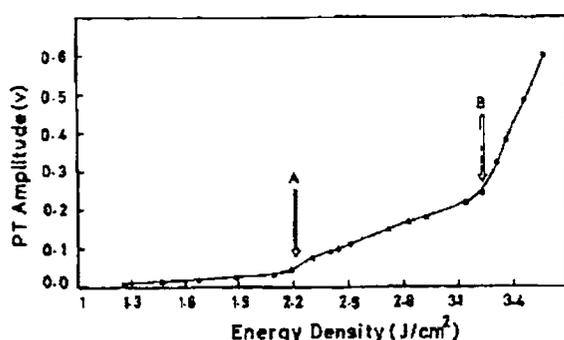


Figure 5. Plot of laser energy density vs PT amplitude for teflon in air.

Braren 1989; Ravi Kumar *et al* 1991). The two distinct threshold values explained in terms of different mechanisms operative in producing the damage-like impurity initiated damage, bond breaking and melting processes took place at different laser energy densities.

Although the mechanism of laser-induced damage in polymeric materials is not well understood, it has been observed that the damage is very sensitive to sample surface condition. The dependence of damage threshold on possible absorptive inclusions and surface polishing of the sample was earlier reported (Golberg *et al* 1983). Results showed that polymers with rough or opaque surfaces had lower values for damage threshold than those for the same material having polished or transparent surfaces at the same wavelength. Therefore, of the two distinct regions observed, the first region (A) corresponded to the damage due to inclusions, impurities and surface inhomogeneity, while the second (B) occurring at higher fluences were assigned to initiation of bond breaking process in the sample. It must be mentioned here that in the second case (B), in addition to the surface layer, the bulk of the material immediately below the surface also got affected in a substantial way due to the action of the laser pulse.

5. Conclusions

The suitability of PTD technique to estimate the damage threshold of a polymer sample has been illustrated. Damage threshold values evaluated by this method for nylon and teflon are in agreement with previous results but tend to be higher.

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References

- Dyumaev K M, Manenkov A A, Maslyukov A P, Matyushin G A, Nechitsilo V S and Prokhorov A M 1983 *Sov. J. Quantum Electron.* **13** 503
 Dyer P E and Sidhu J 1985 *J. Appl. Phys.* **57** 1420
 Golberg S M, Matyushin G A, Pilipetsky N F, Yu Savanin S, Sudarkin A N and Triblesky M I 1983 *Appl. Phys.* **B31** 85
 Harada Y, Kanemitsu Y, Tanaka Y, Nakano N, Kuroda N and Yamanaka K 1989 *J. Phys.* **D22** 569
 Jackson W B, Amer N M, Boccara A C and Fournier D 1980 *Appl. Opt.* **20** 1333
 Lipscomb G F, Grito A F and Narang P S 1981 *J. Chem. Phys.* **75** 1509
 Milam D 1977 *Appl. Opt.* **16** 1204
 Rajasree K, Ravi Kumar A V, Radhakrishnan P, Nampoori V P N and Vallabhan C P G 1990 *J. Acoustic Soc. India* **18** 24
 Ravi Kumar A V, Padmaja G, Radhakrishnan P, Nampoori V P N and Vallabhan C P G 1991 *Pramana - J. Phys.* **37** 345
 Rosencwaig A and Willis J B 1980 *Appl. Phys. Lett.* **36** 667
 Rose A and Gupta R 1986 *Opt. Commun.* **56** 303
 Sell J A, Heffelfinger D M, Ventzek P and Gilgenbach R M 1989 *Appl. Phys. Lett.* **55** 2435
 Srinivasan V, Smertic M A and Babu S V 1986 *J. Appl. Phys.* **59** 3681
 Srinivasan R and Braren B 1989 *Chem. Rev.* **89** 1303
 Tam A C 1986 *Rev. Mod. Phys.* **58** 381