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SIMULTANEOUS MEASUREMENT OF OPTICAL AND DYNAMIC MECHANICAL PROPERTIES OF PLASTIC OPTICAL FIBERS

Dynamic mechanical analysis (DMA) is one of the most powerful tools to study the behavior of plastic and polymer composite materials and it is a potentially very useful tool to simulate behavior of plastic optic fibers (POF) in real applications. The possibility of simultaneous measurements of some optical properties during DMA would significantly upgrade investigations of POF alone or embedded in some materials. In this work, single cantilever DMA of the POFs that was done simultaneously with measuring the transmitted optical signal intensity is described and discussed. In order to compare mechanical results of the same material for cylindrical and rectangular specimens, rectangular plates were prepared by melting POFs and the same kind of tests were performed. It is shown that changes of the optical signal intensity correspond to the changes of storage modulus of the POF during DMA, and the maximums in optical signals intensity indicate the beginning of glass transition processes in the POF material.

Key words: plastic optical fibers; dynamic mechanical analysis; storage modulus; glass/transition temperature; loss modulus; simultaneous measurement.

Plastic optical fibers (POF) have various applications: in home and local area networks (LAN), lighting technology, for fiberoptic sensors and sensors networks, for fiber light amplifiers and as scintillating fibers [1]. They have some great advantages over glass multimode optical fibers such as: easy installation owing to large diameter, efficient light coupling owing to large numerical aperture (NA) (typically 0.5), high ductility or low modulus, resistance to impact and vibrations and low cost. The main disadvantage is higher optical loss [2]. During their applications POF are exposed to different kinds of static and dynamic stresses and environmental conditions. It is of great importance that they keep their optical properties unchanged when they are exposed to different influences in LAN and lighting applications, but on the other hand in sensors applications they should have significant, regular and repetitive change of some optical property.

The most important property of POF is the optical transmission which mainly depends on core materials, drawing process and the conditions employed in the manufacturing process. Poly(methyl methacrylate) (PMMA), polystyrene (PS) and polycarbonate (PC) are the polymers most used as core materials in commercially available multimode step and graded index POF. PMMA has the highest transparency among them [2,3]. The major source of optical loss in POF are intrinsic factors such as absorption (higher harmonics of C-H absorption and electronic transitions absorption) and Rayleigh scattering. During their practical operations environmental factors and mechanical stresses bring both physical and chemical changes of core material and cause change in optical transmission [4-6].

Dynamic mechanical analysis (DMA) is one of the most powerful tools for studying the behavior of plastic and polymer composite materials by applying an oscillating force to a sample and analyzing the material deformation response as a function of frequency, time or temperature [7,8]. It is a potentially very useful tool for simulating behavior of POF in case of versatility of mechanical and environmental conditions, although DMA of cylindrical rods and fibers is

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up today not very common compared to other geometries [9]. Possibility of simultaneous measurements of some optical properties during DMA would significantly upgrade investigations of POF alone or embedded in some materials.

In this work, DMA of the POF that was done simultaneously with measuring the transmitted optical signal intensity is described. In order to compare mechanical results of the same material for cylindrical and rectangular specimens, rectangular specimens, with optimal dimensions for single cantilever DMA, were prepared by melting POFs and the same kind of tests were repeated with them.

EXPERIMENTAL

Two kinds of DMA were performed on POF: single cantilever tests with constant amplitude and frequency and on constant temperature and the dynamic temperature scan tests. During both types of tests the storage modulus (E') was determined. At the same time light was launched into the POF under test and the output light intensity (I) was measured in real time.

The experimental set up consisted of two parts, mechanical and optical. The DMA instrument (TA Instruments Q800) and its personal computer (PC) were used for mechanical measurements and data acquisition. The investigated POF (ESKA™ GK-40) with output diameter 1mm (core diameter 0.98 mm) was clamped inside the DMA instrument between the movable and stationary fixtures and enclosed in the thermal chamber. The length of a fiber between clamps was 18.14 mm, but the whole length of a fiber was about 2.5 m. The clamped parts of the optical fiber were protected with Teflon sleeves in order to prevent its damage during testing. The ends of a POF were carefully brought out from the temperature chamber of the DMA instrument through a hole on its top. Those two outlet ends were connected to the light source and the photodetector (PD), the optical parts of the experimental set up. The light source was a light emitting diode (LED), and the used LEDs had peak wavelengths 840 or 650 nm. The PD was a phototransistor based circuit. The light from the LED was launched to the POF and the intensity of the propagated light was measured by PD. The output signal from the PD was connected to the acquisition system (USB type A/D converter (A/D) and personal computer (PC)). The schematic view of the experimental set-up is presented in Figure 1.

Single cantilever test was performed at constant frequency of 3Hz, constant amplitude of 30 μm and at constant temperature. The number of oscillations at one temperature was about 6400. Each test lasted from 35-40 min because the time for temperature stabilization was not the same at each temperature and that is why the net number of oscillations for the same biased parameters except temperature varied a little from one to another temperature. This kind of DMA was performed successively on 50, 60, 70, 80 and 90 °C on the same POF without opening the temperature chamber. So, a set of five successive single cantilever tests on five temperatures could be considered as an accelerated fatigue test, and the POF is considered as fatigued after those tests.

The dynamic temperature scan test was performed approximately from 40-120 °C at one fixed POF. The experiment was done in standard single cantilever mode using a ramping rate of 3 °C/min, with an oscillation frequency of 1 Hz and amplitude of 20 μm.

A set of tests was performed with the launched light at one wavelength. The first of them was a dynamic temperature test at one POF considered as not fatigued. Than that POF was removed from the instrument and a new POF of the same type was fixed. Five successive single cantilevered tests were performed at five constant temperatures, followed by the dynamic temperature test on the same POF, considered as fatigued, without opening the temperature chamber.

The performed single cantilever tests were originally provided for the rectangular specimens. In order to investigate their applicability to fibers the com-

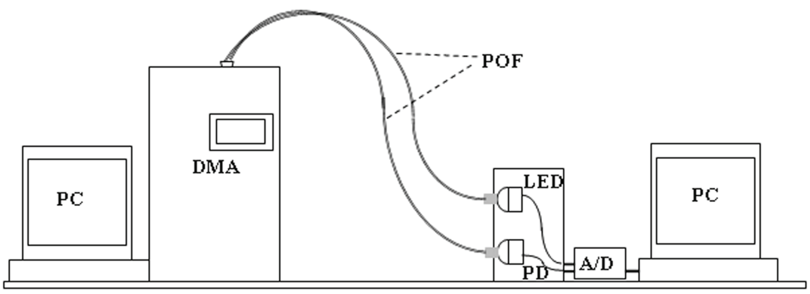

Figure 1. The experimental set up.

parison of mechanical results on the same material for cylindrical and rectangular specimens was done. The rectangular plates of dimensions 3 mm×12 mm×30 mm were prepared by melting the POFs and the same set of mechanical measurements as for the POFs was performed on them with the DMA instrument.

RESULTS AND DISCUSSION

Measurements with POF

Single cantilever tests

Before all mechanical tests, the transmission spectral characteristic of the investigated POF was measured with a spectrometer (Carl Zeiss Jena 384824), and it is presented in Figure 2. Two wavelengths, 650 and 840 nm, are chosen for further measurements. From the spectral characteristics, it is obvious that 650 nm is in the middle of the near constant part of spectral characteristics with maximum transmission and the other chosen wavelength, 840 nm, is at the near infrared descending slope and transmission is about half compared to the maximum. Both these wavelengths are frequently used in fiber optic applications.

The first set of measurements was at wavelength 840 nm, and the second at 650 nm. The number of the performed oscillations on each temperature and at each wavelength is presented in Table 1.

The measuring results for single cantilever tests with optical signal wavelength 840 nm are presented in Figures 3 and 4. The changes of *E'* of POF *versus* time are presented in Figure 3 for five constant temperatures. The measured changes in optical signal intensity during the same tests are presented by normalized signal intensities (I/I_0) versus time in Fig. 4.

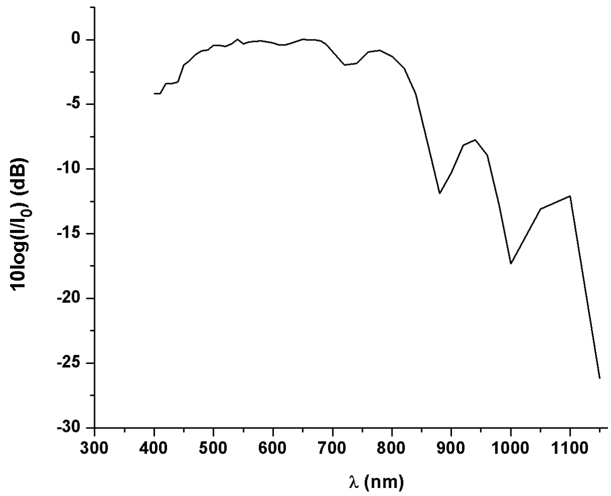

Figure 2. Transmission spectra of POF.

Table 1. Number of performed oscillations, N, during single cantilever test on 5 different temperatures and at two different wavelengths of launched light

t / °C	*N* (λ = 840 nm)	*N* (λ = 650 nm)
50	6405	6409
60	6393	6583
70	6680	6391
80	6440	6409
90	6393	6294

malized signal intensities (I/I_0) versus time in Fig. 4. Parameter I is the measured signal on the PD, and I_0 is the measured signal on the PD at the beginning of the single cantilever test at that measuring temperature. Comparing the graphs in Figures 3 and 4 it is obvious that after some period of temperature stabilization the *E'* is slightly rising, while I/I_0 is descending during single cantilever tests. The most significant changes of *E'* and I/I_0 has happened at 90 °C.

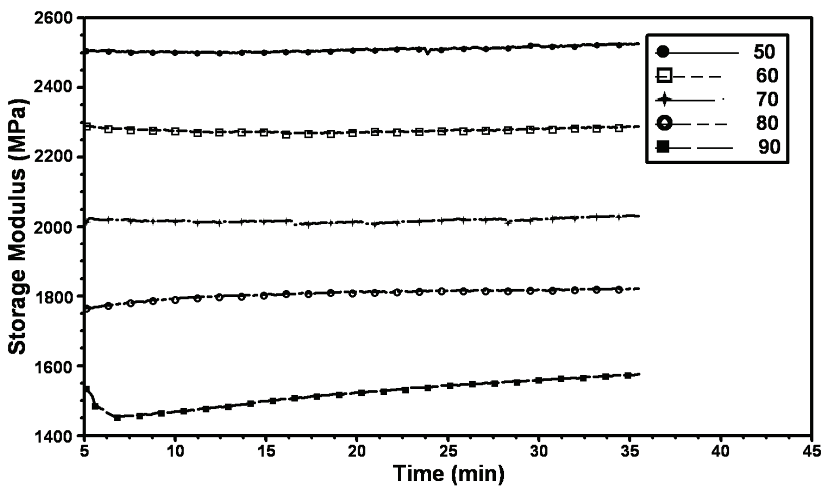

Figure 3. The changes of E' versus time of POF during single cantilever tests (wavelength: 840 nm).

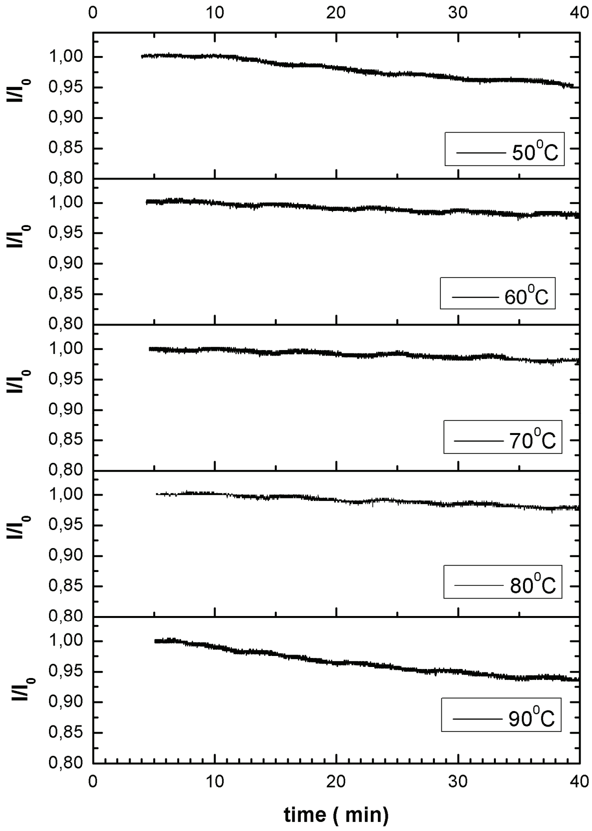

Figure 4. The change of I/I_0 of POF versus time during single cantilever tests (wavelength: 840 nm).

In order to quantify and compare changes in E', and I, their relative changes are calculated and presented in Table 2. The relative changes of E' denoted as $\Delta E'/E'$, and of optical normalized signal intensity denoted as $\Delta I/I_0$ are obtained from the values of E' and I/I_0 at t_1 = 15 min and t_2 = 35 min after temperature stabilization.

Similar graphs of E' and I/I_0 *versus* time, but for wavelength 650 nm are presented in Figures 5 and 6. The calculated values of $\Delta E'/E'$ and $\Delta I/I_0$ for measurements on 650 nm are also presented in Table 2. During the tests at 80 and 90 °C the measurements were disturbed before t = 35 min which could be seen from irregular changes of E' in Figure 5, as well as, from the change of slope of optical signals in Figure 6. So, the relative changes $\Delta E'/E'$, and $\Delta I/I_0$, for 80 °C were calculated from the values at t_1^* = 15 min, and t_2^* = 32 min, and for 90 °C at t_1^{**} = 5.65 min and t_2^{**} = 13.25 min. This irregular behavior was emphasized in the Table 2 with values marked by "*" and "**".

The measurements at both wavelengths show similar relative changes of E' and I/I_0 and the most significant changes were at 90 °C.

Dynamic temperature scan test

Two POFs are examined at each wavelength, one not fatigued and the other, fatigued. The changes

Table 2. Relative changes $\Delta E'/E'$ and $\Delta I/I_0$ for POFs and $\Delta E'/E'$ for rectangular specimen after single cantilever tests at various temperatures

t / °C	POF (λ = 840 nm)		POF (λ = 650 nm)		Rectangular specimen
	$\Delta I/I_0$ (%)	$\Delta E'/E'$ (%)	$\Delta I/I_0$ (%)	$\Delta E'/E'$ (%)	$\Delta E'/E'$ (%)
50	-2.39	0.94	-3.81	0.99	0.56
60	-1.74	0.88	-1.02	1.56	0.46
70	-1.01	0.81	-0.65	0.80	0.60
80	-1.53	0.82	-1.47*	1.51*	0.77
90	-4.76	4.81	-1.35**	1.96**	1.00

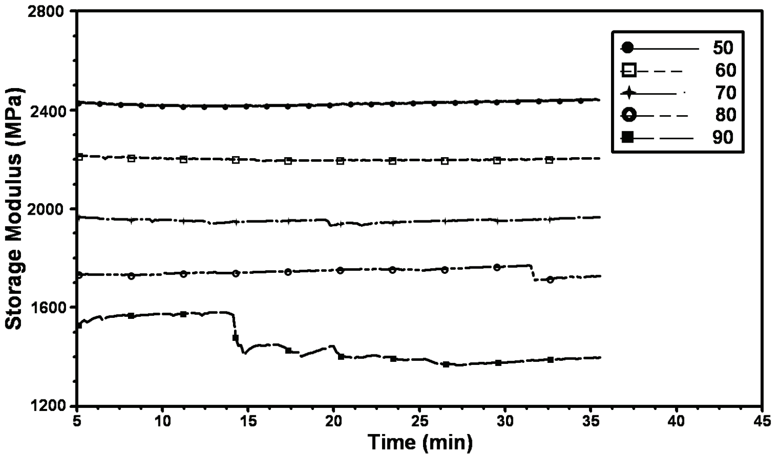

Figure 5. The changes of E' of POF versus time during single cantilever tests (wavelength 650 nm).

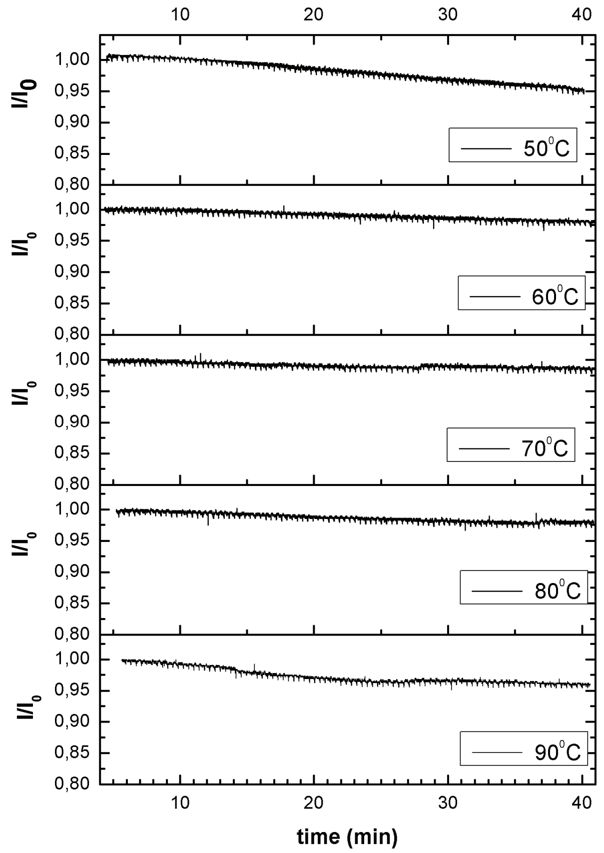

Figure 6. The change of I/I_0 of POF versus time during single cantilever tests (wavelength: 650 nm).

of E' versus temperature of the both POFs with launched light wavelength 840 nm are presented in Figure 7. The E' descended linearly from 40 to 80 °C for both fatigued and not fatigued POF. Nonlinear behavior started at temperatures higher than 80 °C. The values of I/I_0 for the not fatigued and fatigued POF at 840 nm are presented in Figure 8. The intensity of optical signal was almost constant from 40 to 50 °C, and ascended almost linearly from 50 to 80 °C. At temperatures higher than 80 °C the dependence of I/I_0 *versus* temperature had non linear behavior, reached their maximums and then constantly descended. The maximum I/I_0 for not fatigued POF was at 89 °C and for fatigued was at 104 °C.

Similar changes in E' and I/I_0 had occurred during the measurements at wavelength 650 nm. The changes of E' *versus* temperature are presented in Figure 9, and the changes of I/I_0 are presented in Figure 10 both for fatigued and not fatigued POFs. The maximum I/I_0 of not fatigued POF was at 88 °C. For fatigued POF the maximum I/I_0 was between 95 and 105 °C, The relative changes $\Delta E'/E'$ and $\Delta\ I/I_0$ at 650 nm are presented in Table 3.

The relative changes $\Delta E'/E'$ and $\Delta\ I/I_0$ for fatigued and not fatigued POFs at both wavelengths are presented in Table 3. Two kinds of relative changes were calculated: one for the temperature range from 40-80 °C, and the other from 40 °C to the temperatures at which I/I_0 was maximum.

The changes of I/I_0 *versus* temperature for not fatigued POFs are bigger than for not fatigued. This is more significant for signals at 840 nm. Maximums of I/I_0 for not fatigued POF are almost at the same temperature (88-89 °C) for both wavelengths as well as for the fatigued POF (104 °C).

Measurements on rectangular specimens

Single cantilever test mode used for described measurements is originally provided for rectangular specimens. The calculated values of E' obtained by DMA are based on expressions for rectangular specimens divided by π [9]. In order to compare results of mechanical characteristics on the same material for

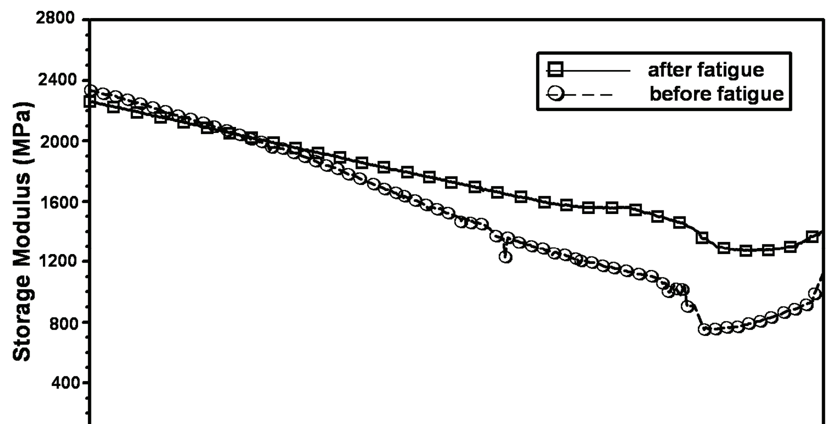

Figure 7. The changes of E' versus temperature for fatigued and not fatigued POF (wavelength: 840 nm).

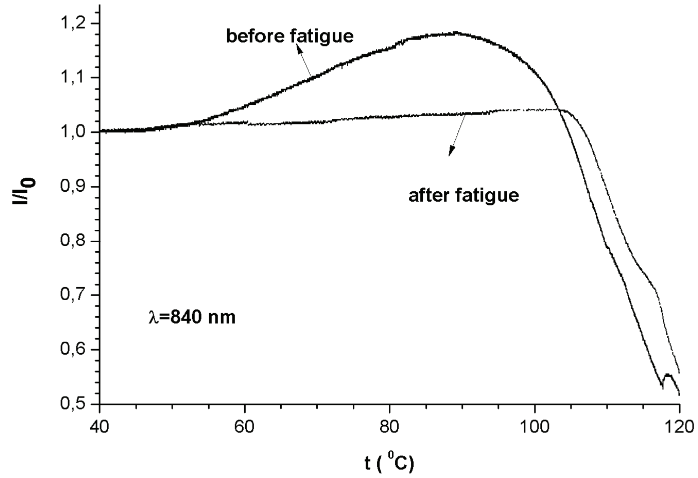

Figure 8. The changes of I/I_0 versus temperature for fatigued and not fatigued POF (wavelength: 840 nm).

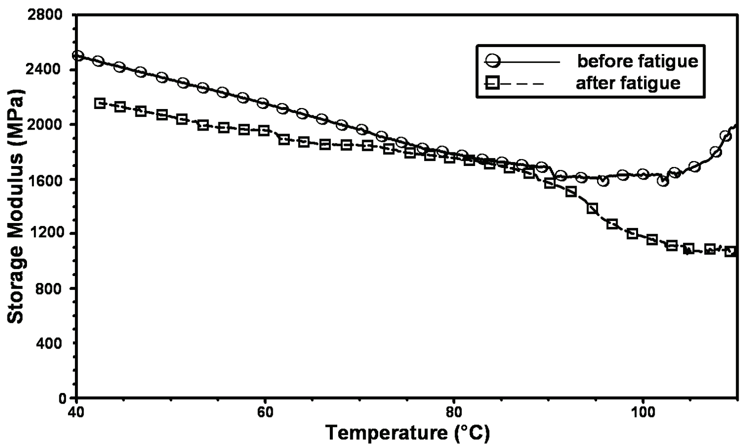

Figure 9. The changes of E' versus temperature for fatigued and not fatigued POF (wavelength: 650 nm).

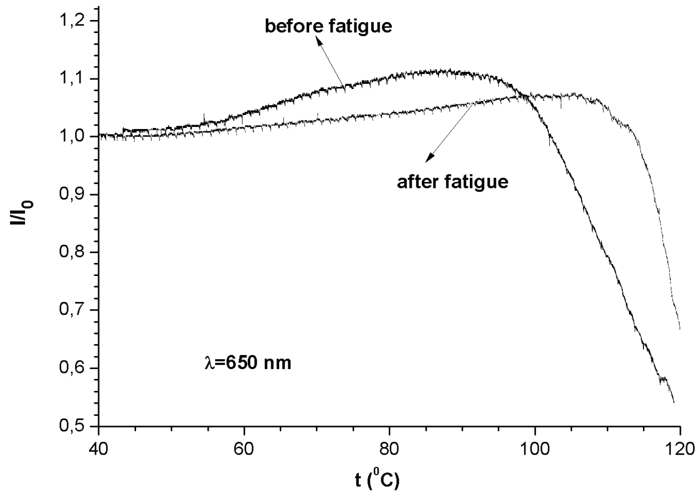

Figure 10. The changes of I/I_0 versus temperature for fatigued and not fatigued POF (wavelength: 650 nm).

cylindrical and rectangular specimens, two rectangular specimens, are prepared by melting POFs. The dynamic temperature scan test was performed on the first one in order to compare *E'* of not fatigued POF with it. Then, the single cantilever tests on five temperatures and the dynamic temperature test were performed on the second rectangular one.

Table 3. Relative change of I/I_0 and E' of POFs and ΔE'/E' of rectangular specimens in dynamic temperature scan tests

Temperature interval, °C	POF (λ = 840 nm)				POF (λ = 650 nm)				Rectangular specimen	
	Not fatigued POF		Fatigued POF		Not fatigued POF		Fatigued POF		Before fatigue	After fatigue
	$\Delta E'/E'$ %	$\Delta I/I_0$ %	$\Delta E'/E'$ %	$\Delta I/I_0$ %	$\Delta E'/E'$ %	$\Delta I/I_0$ %	$\Delta E'/E'$ %	$\Delta I/I_0$ %	$\Delta E'/E'$ %	$\Delta E'/E'$ %
40-80	-41.9	15.4	-27.4	2.4	-28.7	9.9	-18.7	3.5	-28.9	-26.9
40-89	-50.4	18.3	-31.2	3.0	-34.1	10.6	-25.8	5.4	-36.3	-34.1
40-104	-	-	-43.7	3.6	-	-	-50.8	7.9	-49.7	-47.8

The single cantilever test

The curves representing storage modulus *versus* time at five constant temperatures are presented in Figure 11. The $\Delta E'/E'$ for single cantilever tests are presented in Table 2. From those results it is obvious that they are of the same order for temperatures from 50-80 °C and for 90 °C the value is higher than for the other temperatures.

The dynamic temperature scan test

The results of dynamic temperature scan tests for the not fatigued, and the fatigued rectangular specimen are presented in Figure 12. The curves represent the change of E', the loss modulus (E'') and the loss factor (tanδ) *versus* temperature. From these curves glass transition temperature (T_g) was derived and it is 118.7 °C for fatigued and 121.6 °C for not fatigued plate.

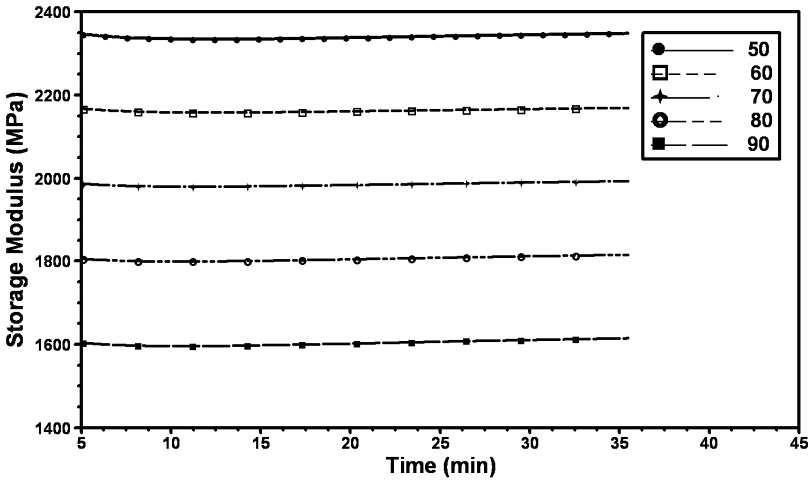

Figure 11. The changes E' of the rectangular specimen versus time for single cantilever tests on five constant temperatures.

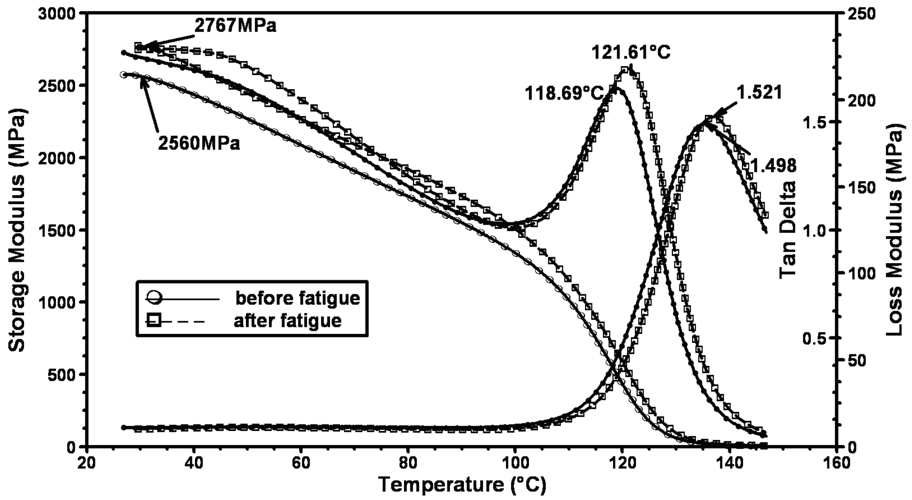

Figure 12. Changes of E', E" and tanδ versus temperature for the rectangular specimens during single cantilever temperature scan test.

The values of *E'* at 40, 80, 90 and 104 °C are obtained from the curves in Figure 11, and $\Delta E'/E'$ for fatigued and not fatigued specimen from 40-80 °C, 40-90 °C and 40-104 °C are calculated and presented in Table 3.

If the results for dynamic temperature scan test for POFs and rectangular plates are compared it could be noticed that the temperatures at which optical signals had maximums for not fatigued and fatigued POFs are lower than T_g of rectangular specimens. The temperatures of optical signal maximums were close to the temperatures when *E"* had their minimums (95 °C for not fatigued rectangular specimen, and 103 °C for fatigued). So, maximums in optical signals did not indicate the T_g of the material of the POF, but the beginning of the glass transition processes. This indication is more sensitive for not fatigued POF at 840 nm, and is generally less sensitive for fatigued POFs. The glass transition process is at higher temperatures for fatigued material and the maximum of optical signals are at higher temperatures for fatigued POF as well.

CONCLUSIONS

Single cantilever tests on POFs at constant frequency and amplitude and on five constant temperatures showed that *E'* slightly increased with the number of performed oscillations at each temperature. Relative changes of *E'* are similar for temperatures from 50-80 °C and significantly bigger at 90 °C. During those tests considered as a kind of fatigue tests the values of I/I_0 were decreasing and $\Delta\ I/I_0$ were similar at both wavelengths of the optical signal. The biggest relative decrease of optical transmission was at 90 °C which corresponded to the most significant change in *E'*.

For dynamic temperature scan tests two kinds of comparison were done: one between not fatigued and fatigued POFs, and the second for the same measurements at two wavelengths 840 and 650 nm. In general, the storage modulus of POF decreases significantly with rising temperature from 40-110 °C. From 40-80 °C, the decrease of storage modulus is linear *versus* temperature, and for higher temperature the behavior is nonlinear. The optical signals are almost constant from 40-50 °C, and in the temperature interval from 50-80 °C the transmission of POF is increasing linearly *versus* temperature. At higher temperatures, the optical signal is ascending, reaching its maximum and then descends on both wavelengths. Optical transmission change versus temperature at 840 nm is significantly higher for not fatigued fiber than for fatigued. Measurements at 650 nm showed that transmission changes versus temperatures for fatigued and not fatigued POFs are less different.

The maximum intensity of optical signals are almost at same temperatures at both wavelengths (88-89 °C for not fatigued and 104 °C for fatigued POF).

Comparing the dependence of *E'* and *E"* *versus* temperature of rectangular specimens with the optical signals of POF during temperature scan tests it is obvious that maximums in optical signals transmission of POFs are at temperatures close to loss modulus minimums. They are lower than T_g, both for fatigued and not fatigued POF. So, optical signal changes in POF could indicate the beginning of glass transition process, but not the T_g.

It is showed that simultaneous DMA and optical signal intensity measurements could be done on POF. In the temperature interval from 40-80 °C the relative changes in storage modulus are opposite to the change of the intensity of optical signal, *i.e.* its transmission. It is possible to choose optical wavelengths with higher and lower sensitivity to temperature, and in general fatigue of the POF decrease its temperature sensitivity. The measuring modes for rectangular specimens could be used for POF, but the calculations should be multiplied with appropriate factor.

The DMA of POF is potentially useful for fiber optic sensors development, especially for their temperature sensitivity investigations during various types of mechanical measurements.

Nomenclature

Abbreviations

DMA - Dynamic mechanical analysis
LAN - Local area network
LED - Light emitting diode
NA - Numerical aperture
PMMA - Poly(methyl methacrylate)
PD - Photodetector
POF - Plastic optical fiber
PS - Polystyrene
Tg - Glass transition temperature

Symbols

E' - The storage modulus
E" - The loss modulus
I - Optical signal intensity
I_0 - Optical signal intensity at the beginning of the measurement
$\tan\delta$ - Loss factor

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NAUČNI RAD

ISTOVREMENO MERENJE OPTIČKIH I DINAMIČKO--MEHANIČNIH SVOJSTAVA PLASTIČNIH OPTIČKIH VLAKANA

Dinamičko-mehanička analiza (DMA) je jedna od najefikasnijih metoda za proučavanje ponašanja plastičnih i polimernih kompozitnih materijala i potencijalno može biti veoma korisna za simulaciju ponašanja plastičnih optičkih vlakana (POV) u realnim primenama. Mogućnost istovremenog merenja nekih optčkih svojstava za vreme DMA može značajno da unapredi proučavanje POV, samih ili ugradjenih u neki materijal. U ovom radu je opisano i diskutovano merenje mehaničkih svojstava POV pomoću "single cantilever" DMA koje je izvršeno istovremeno sa merenjem intenziteta propuštene svetlosti kroz POV. Kako bi se uporedili rezultati DMA koji su dobijeni za pravougaone i cilindrične uzorke od istog materijala, ista vrsta ispitivanja vršena je i na pravougaonim pločicama dobijenim topljenjem POV. U radu je pokazano da promene intenziteta optičkih signala odgovaraju promenama modula sačuvane energije POV za vreme DMA, a da dobijene maksimalne vrednosti optičkih signala označavaju početak procesa prelaza u staklasto stanje u materijalu od koga je napravljeno optičko vlakno.

Ključne reči: plastična optička vlakna; dinamičko-mehanička analiza; modul sačuvane energije; temperatura staklastog prelaza; modul gubitaka energije; istovremeno merenje.