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Mechanical properties characterization of polymethyl methacrylate polymer optical fibers after thermal and chemical treatments

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ABSTRACT

This paper presents the dynamic mechanical analysis (DMA) in polymer optical fibers (POFs) made of Polymethyl Methacrylate (PMMA) that were submitted to different thermal and chemical treatments, namely annealing and etching processes. The prepared samples were submitted to stress–strain cycles to evaluate the Young's modulus of each fiber. Also, test with constant stress and temperature variation were performed to estimate the thermal expansion coefficient of the fibers submitted to each thermal and chemical treatment. The samples were also tested under different temperature, humidity and strain cycle frequency conditions to analyze the variation of their mechanical properties with these parameters. Results show that the thermal and chemical treatments lead to a reduction of Young's modulus and an increase of the thermal expansion coefficient, which can produce sensors based on intensity variation or fiber Bragg grating with higher dynamic range, stress and temperature sensitivity. Furthermore, the etching and annealing resulted in fiber that presents lower Young's modulus variation with temperature, humidity and strain cycling frequency in most cases. However, the annealing made under water and the combinations of etching and annealing resulted in POFs with higher modulus variation with humidity, which enable their application as intensity variation or FBG-based sensors in humidity/moisture assessment.

1. Introduction

Optical fiber sensors present advantages over the conventional electronic and electromechanical sensors that include compactness, lightweight, multiplexing capabilities, electrical isolation and electromagnetic field immunity [1]. Polymer optical fibers (POFs) share the advantages presented by silica fibers [2]. Additionally, POFs show advantages over silica fibers related to its material features such as lower Young's modulus, fracture toughness, flexibility in bending and non-brittle nature that make it clinically acceptable for in vivo applications [3]. Such advantages enable the application of POFs as sensors for parameters like strain [4], temperature [5], humidity [6], curvature [7], liquid level [8], acceleration [9], refractive index [10] and pressure [11].

Advances in polymer processing and fabrication enabled the growth of POF sensors field, where different POF materials have been proposed. One of these POF materials is Thermoplastic Olefin Polymer of Amorphous Structure (TOPAS), which presents humidity insensitivity due to its chemical structure composition [12]. In addition, if the TOPAS Cyclic Olefin Copolymer (COC) grade 5013 is employed on the fiber fabrication, it will present a glass transition temperature (Tg) of about 134 °C [4]. Another humidity insensitive material is Zeonex that also presents a Tg higher than 130 °C [13]. Furthermore, polycarbonate POFs are employed as an alternative when the application requires higher strain limits [14]. There are also the cyclic transparent amorphous fluoropolymers (CYTOP) fibers that present lower optical losses than the other POF materials due to its amorphous nature and perfluorinated backbone [15]. Despite their lower Tg (about 110 °C) and humidity absorption, polymethyl methacrylate (PMMA) still is the most employed material in POFs manufacturing [3].

Regarding the fiber diameter and cladding structure, there are different options as well. Polymer optical fiber Bragg gratings (POFBGs) are usually inscribed in microstructured POFs (mPOFs) [16] that present a pattern of holes, which generally is hexagonal with a ring of holes [16] through the fiber. The hole diameter and the pitch between them can ensure an endlessly single mode operation i.e. the fiber is single mode or few moded for any wavelength or a wide wavelength region [17]. In addition, mPOFs provide the possibility of holding gas or a biological sample in the holes for evanescent-wave sensing [18]. For these reasons, different PMMA mPOFs cladding structures are reported

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[19,20]. However, mPOFs present some issues such as high dependency of the cleaving parameters, namely angle, temperature and velocity [21]. Also, the microstructure can be degraded by impurities that can get into the holes, light scattering losses at the holes walls and difficulty of connectorization also can inhibit their applications [22]. The mPOFs disadvantages and the wider commercial availability encourage the application of large core diameters PMMA POFs in this work that present advantages over the mPOFs, which are also related to its simplicity on manufacturing [10]. Such fibers also enable the application of low precision connectors and low cost lasers or light emitting diodes (LEDs) due to its higher numerical aperture, which generally results on low cost systems [23]. Although they are less suitable for grating inscription due to its multimode operation, some recent reports of POFBG inscription in multimode fibers have been presented [24,25].

Polymers are viscoelastic materials that do not present a constant response with stress or strain [26]. The polymer response with stress or strain presents an elastic or storage component and a viscous or loss component [27]. The viscoelastic response of a PMMA mPOF under strain cycles with different frequencies was characterized in [26]. Moreover, creep and recovery tests of PMMA mPOFs and long term monitoring of the viscoelastic behavior are presented in [17] and [28], respectively. This polymer viscoelastic behavior can lead to higher hysteresis of POF-based sensors, which can be reduced by compensation techniques on the signal processing [7,29] or with the proper annealing of the fiber [30]. The fiber annealing is a thermal treatment that comprises on keeping the POF on a temperature above the polymer β-transition for some time that leads to the relaxation of the POF internal stress created on its manufacturing process [31]. Furthermore, the fiber annealing can be done with a methanol solution that allows it to be made in room temperature [32] or under water that enables the annealing during some minutes, instead of hours [31]. Besides the hysteresis reduction, the fiber annealing can influence the sensor sensitivity to strain and stress as reported in [31]. In addition, such thermal treatment can reduce the POFBG inscription time when it is made before the grating inscription [33] or it can lead to blue shift of the Bragg wavelength, especially when it is made at high humidity conditions for the case of the fiber annealing after the grating inscription [34].

Etching is a chemical treatment that can be made in POFs, which generally comprises of positioning the fiber in a container filled with acetone. Such chemical treatment can lead to variation of the polymer properties due to the molecular chain relaxation that is related to the solvent absorption [35]. The chemical reaction between the PMMA POF and the acetone leads to a diameter reduction of the fiber that can provide lower response times for POF-based humidity sensors [6]. In addition, the fiber etching can provide higher reflectivity in POFBGs and lower exposure times on the gratings inscription [36].

In order to characterize the effects of such heat and chemical treatment in a PMMA POF, this paper presents the dynamic mechanical analysis (DMA) on a POF that was submitted to such treatments. A POF without any heat or chemical treatment is also tested for comparison purposes. Since the humidity can influence the annealing process [34], the DMA is made with the sample annealed at low humidity and under water (humidity of 100%). Additionally, the etched fibers with two different etching times, namely 4 and 7 min are also tested to evaluate the influence of this parameter on the fiber material response, where the etching time depends on the fiber diameter reduction [36]. Finally, the fibers annealed at low and high humidity (in hot water) are submitted to an etching process for 4 and 7 min. The DMA is made to measure the Young's modulus and thermal expansion coefficient of each fiber tested. Moreover, the variation of the Young's modulus of each sample is characterized with respect to the temperature, humidity and strain cycle frequency variations.

This paper is organized as follows. Section 2 presents the experimental setup, where the annealing and etching treatments are presented as well as the dynamic mechanical analyzer employed. The tests results are presented and discussed in Section 3. Then, conclusions and future works are discussed in Section 4.

2. Experimental setup

The POF employed on the tests presents a PMMA core with a diameter of 980 μm and a cladding of fluorinated polymer with a thickness of 20 μm. The annealing at low humidity of this fiber is made in the climatic chamber 400/1ND (Ethik Technology, Brazil) with an annealing temperature of 70 °C, which is higher than the PMMA β-transition temperature of about 50 °C. At this temperature, the humidity is about 10% and the annealing time is 24 h. The annealing under water is made with the same annealing temperature and time (70 °C @ 24 h). In order to eliminate the influence of any water absorbed by the fiber, the POFs annealed under water are positioned outside the water recipient for about 24 h after the annealing. For the etching process, the samples are placed at the vertical position in a container filled with pure acetone, where one set of five samples is etched for 4 min and the other, for 7 min. The etching is made in about 10 mm of the fiber length, where the POFs presented a diameter reduction to 0.92 mm and 0.88 mm for the etching times of 4 and 7 min, respectively. Such reduction represents a rate of about 0.012 mm/min for the etching made with pure acetone. In addition, a deviation of about 0.01 mm was obtained in all etched samples.

After the samples preparation, they are positioned on the dynamic mechanical analyzer DMA 8000 (Perkin Elmer, USA) presented in Fig. 1, where an oscillatory load with controlled displacement and frequency is applied on the sample. In addition, the analyzer has a climatic chamber that provides temperature and humidity control in the tests. The samples have a length of about 10 mm and are fixed on the DMA supports as presented in Fig. 1. The distance between the supports is 7 mm. Therefore, the etching of 10 mm length is sufficient for the tests employed and each etched sample is cleaved to position only the etched length on the DMA.

The DMA is employed to characterize the Young's modulus variation with respect to the strain cycle frequency, humidity and temperature. Since the PMMA is a viscoelastic material, its Young's modulus presents a storage and a loss component (see Eq. (1)). The storage component is related to the elastic part of the material response, whereas the loss component is related to the viscous part of the response [37].

$$E^* = E' + iE'', \tag{1}$$

where E^* is the Young's modulus, E' is the storage Modulus and E'' is the loss Modulus. If a strain is applied, the stress response will present a phase lag (δ), the same holds true if a stress is applied [27]. In addition,

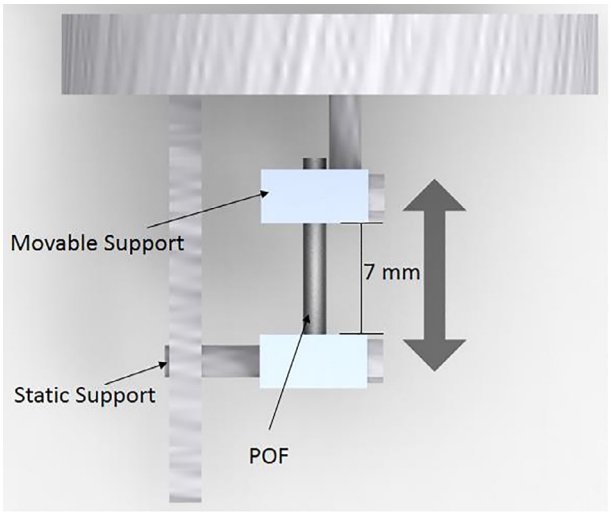

Fig. 1. Experimental setup of the DMA tests on the PMMA POF.