25th ABCM International Congress of Mechanical Engineering
October 20-25, 2019, Uberlândia, MG, Brazil

COB-2019-0363
ISOTHERMAL TENSILE PROPERTIES OF POLYAMIDE MONOFILAMENTS

Sarah Silveira Mendes
Carolina Seixas Moreira
Luiz Carlos da Silva Nunes
Universidade Federal Fluminense, Department of Mechanical Engineering, Rua Passo da Pátria 156, Departamento de Engenharia Mecânica, Laboratório de Mecânica Teórica e Aplicada, Bloco E, sala 210, Niteroi, Brazil
smendes@id.uff.br, carolina_moreira@id.uff.br, luizcsn@id.uff.br

Abstract. *This article explores the influence of the temperature in the tensile strength of two types of polyamide monofilament. Tensile tests were conducted, isothermally, in two different PA monofilaments with 0.8mm and 0.66mm diameter. Three different temperatures were tested, the first one, 30ºC; the second one, 50C, which is near the glass transition temperature of the fibers; and the third one, 70ºC. During the tests, the tensile force was monitored using a cell load and images of the specimens were captured. A Digital Image Correlation software was used to attain the displacement fields. As expected there was a decrease of the Young's Modulus in both specimens of polyamide monofilament. The measured Young's Modulus for Specimens 1 and 2 were (214.5 ± 3.3) GPa and (282.4 ± 3.2) GPa, respectively. For Specimen 1 there was a decrease of 23.6% of the Young's Modulus at 50ºC and 42.81% at 70ºC, for Specimen 2 the decrease in both temperatures was smaller, 12.12% at 50ºC and 17.35% at 70ºC.*

Keywords: *polyamide, glass transition temperature, DIC Method, tensile properties*

1. INTRODUCTION

For decades, polymeric materials have been studied because of their favorable characteristics, such as ease of forming, durability, and relatively lower cost and weight. Polymers are the most important viscoelastic systems. At room temperature the polymer chains do not have sufficient energy to provide mobility, preferably responding elastically to the mechanical requests. The viscous component (plastic deformation) exists, but its contribution is minority. The polymer is rigid and brittle (Canevarolo, 2006, Khanna, 1992, Van Krevelen, 2009). When the semi crystalline polymer is heated, above a certain temperature, its chains acquire mobility only to the amorphous phase, keeping the crystalline phase rigid (Lewin, 2006). The flexibility of the polymer mass is a function of the mobility generated by the amorphous phase, restricted by the stiffness of the crystalline phase. The temperature, in which the polymer chains acquire mobility and time to respond to the mechanical demands, is called glass transition temperature (Tg). Above the glass transition temperature, the response of these materials to a mechanical perturbation field involves several types of molecular motion (Cerrada, 2005). Therefore the mechanical properties of a polymer changes above the Tg. By heating above it, the oriented part of the polymer will tend to retract, and in the amorphous part, the retractive force is even a direct measure of the degree of orientation obtained (Lewin, 2006).

The polyamides belong to the largest group of polymeric materials, semi crystalline polymers, which constitute a blend of ordered crystalline and randomly oriented amorphous phases (Arabnejad et al., 2014, Sedighiamiri et al., 2010). Several studies observed the two constituent phases of polyamides—amorphous and crystalline— having different mechanical properties (Kagan et al., 2000, Peeters et al., 2002, Arabnejad et al., 2014, Choy et al., 1981). These two constituent phases have different stiffness and strengths, and plastic deformation of them is dominated by deformation of the crystalline portion (Choy et al., 1981). The mechanical properties of the polymers are also dependent on the molecular mobility which is influenced by the chemical nature of the macromolecules, the molecular mass, the presence or absence of branching and crosslinking, the degree of crystallinity, presence or absence of plasticizer, temperature, moisture, orientation and other aspects related to the thermal history of the particular sample (Wasilkoski, 2006, Choy et al., 1981, Lewin, 2006).

Since for semi crystalline polymers, processing methods and history define the temporal, mechanical and thermal environment, which in turn determine the mobility, relaxations and extent of chain extension, as well as nucleation and orientation of polymer crystallites. The process and use history dictate the final morphology and thus ultimate thermomechanical properties of semi crystalline polymers (Lincoln et al., 2001). It was observed that the Young's modulus of polyamides depends not only of the temperature of the polymer and the other factors cited, but also by the number of monomer units in the body (Peeters et al., 2002) and its form (Raz et al., 2017, Shan et al., 2007). Studies

(Peeters et al., 2002, Kagan et al., 2000) reported large ranges of the Young's modulus, Polyamide 6 being between 270- 377GPa and the Polyamide 6.6 being 173- 508GPa at room temperature (Peraro, 2000, Wypych, 2016, McIntyre, 2005). Specifying the values of thermomechanical properties of monofilaments of polyamides becomes relevant due to the discovery of their application as thermomechanical actuators (Haines et al., 2014).

This study adopted the Digital Image Correlation method for the estimation of the deformations fields during the tensile isothermal tests of polyamide fibers. Two types of polyamide monofilaments blends (polyamide 6 and polyamide 6.6) were submitted to tensile tests. All tests were conducted under the same velocity, 8mm/min. Three different temperatures were tested, the first one, 30ºC; the second one, 50ºC, which is near the glass transition temperature of the fibers; and the third one, 70ºC.

2. EXPERIMENTAL PROCEDURE

2.1 Materials

In this study two types of polyamide fiber were investigated, one from Ekilon Crystal with 0.8 mm diameter (named Specimen 1 in this study) and the second, from Daiyama Max Force with 0.66 mm diameter (named Specimen 2 in this study). Recent study (Mendes et al., 2018) has determined thermal properties of the materials. The thermogravimetric analysis indicated the presence of two types of crystal regarding the form γ that melts at lower temperatures, and another for the crystalline form α that melts at higher temperatures in Polyamide 1. For the Polyamide 2 was observed a single crystalline melting peak assigned to crystalline form α (Mendes *et al.*, 2018 and Liu *et al.*, 1999). Both monofilaments are characterized by its manufactures as a blend of polyamide 6 and polyamide 6,6. The differential scanning calorimetry analysis of the specimens indicated the glass transition temperature (Tg) of the two types of polyamide were close, being 48ºC for Specimen 1 and 49ºC for Specimen 2.

2.2 Experimental Setup

Experiments were conducted using an in-house-produced temperature-controlled chamber fabricated from balsa wood, Teflon, glass wool and tempered glass. This chamber was connected to a universal machine and a 20Kgf cell load. The front part of the camera was manufactured with tempered glass transparent to capture images of the specimen using a CCD camera (Charged Couple Device) Sony high-resolution model XCD-SX900 (1280 x 960 pixels) with C-Mount lenses10xZoom fixed at a constant distance from the apparatus. The temperature during the tests was increased with the aid of Omega's resistance SRFG Model 809/10. The temperature in the chamber was controlled with Arduino free electronic prototyping platform and two K-type thermocouples connected to it. All data were stored in the computer. The polyamide fiber was attached to the cell load (Fig. 1 a)) and positioned in the heating zone of the chamber, as seem on Fig. 1 b).

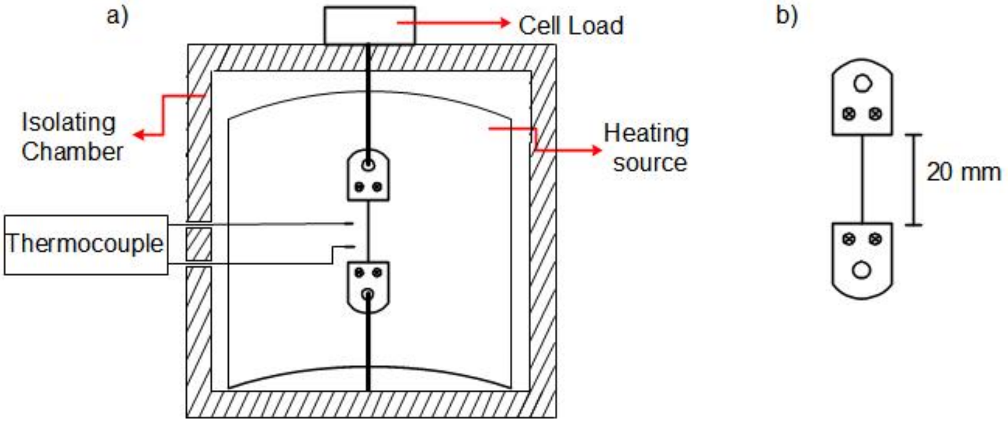

Figure 1. Experimental setup.

The chamber was linearly heated from room temperature (23ºC) until the test temperature (Fig.2). Three different temperatures were investigated, the first one, 30ºC; the second one, 50C, which is near the glass transition temperature of the fibers (Mendes et al., 2018); and the third one, 70ºC. The tests were performed after the temperature in the chamber stabilized. Five specimens were tested in each temperature condition. All tests were conducted under the same velocity, 8mm/min.

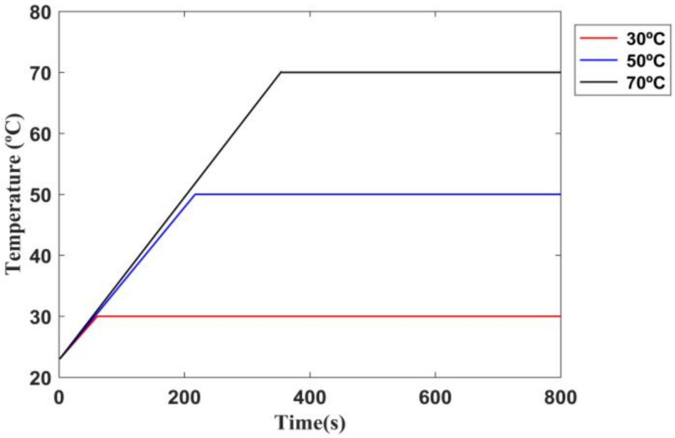

Figure 2. Temperature versus time during tensile tests.

Captured images were processed using a program of digital image correlation, aiming to attain the fields of displacement of the bodies during the isothermal tests. This program consists in the comparison of images before and after a deformation being imposed on a body. More information about the method can be found at Schreier *et al.,* 2009. In this work the sub-reference image had 21 x 21 pixels and the sub-image search 41 x 41 pixels.

3. RESULTS AND DISCUSSION

The experimental results of the polyamide fibers are presented in this section. In the Fig. 3 and Fig. 4 the results of the tensile tests are shown for Specimens 1 and 2, respectively. Five specimens were tested for each condition of temperature. After a certain value, 70MPa – 100MPa, of the traction load was observed sliding of the specimens due to the smooth nature of its surface.

According to the literature the elastic behavior of polymeric materials is more difficult to describe than that of metals or ceramics, since it is strongly dependent on both temperature and time (McIntyre, 2005), thus being called viscoelastic material. Above de glass transition temperature, semi crystalline polymers do not melt, unlike crystalline polymers, but undergo a change in their structure from rigid to flexible (Lewin, 2006). Their polymer chains of the amorphous phase acquire mobility, that is, they acquire possibility of conformation, of change. This change implies a drastic change on almost all of its properties. Hence, as expected, the specimens 1 and 2 presented relaxation when tensile tests above the glass transition temperature were performed, thereby; the tensile force was lower for the same deformation in higher temperatures (Fig. 3 and Fig. 4). Since the Specimen 2 has a higher temperature of glass, 49 ºC, the fibers did not presented a large relaxation in the 50º C tensile tests in contrast to the Specimen 1.

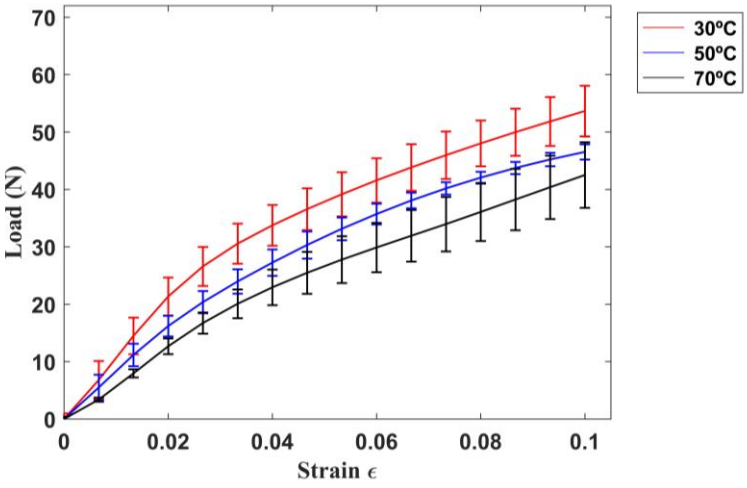

Figure 3 Load (N) versus strain (ε) in Specimen 1

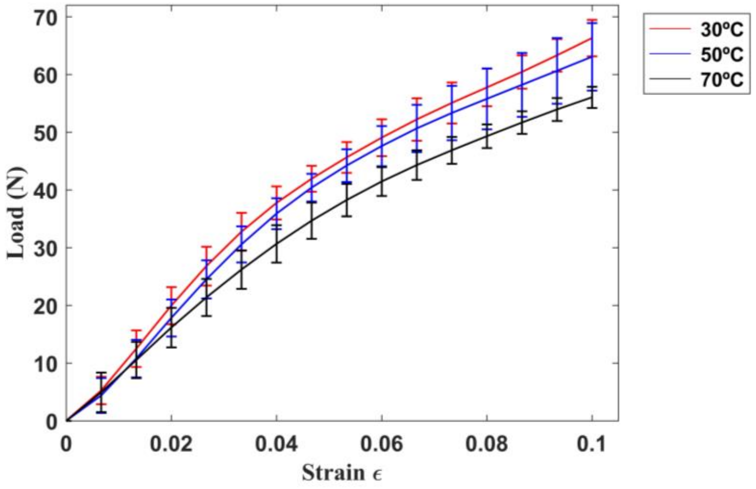

Figure 4 Load (N) versus strain (ε) in Specimen 2

The experimentally measured Young's Modulus of the Specimens 1 and 2 are presented in Tables 1 and 2, respectively. As can be observed, the values obtained for Specimen 2 are superior compared to those of Specimen 1. One reason for that phenomenon is that since, as the thermogravimetric analysis indicated, there are present two types of crystals in Specimen 1, the form γ that melts at lower temperatures, and the crystalline form α that melts at higher temperatures, and in the Specimen 2, it was observed a single crystalline melting peak assigned to crystalline form α, which is also known as being a more stable structure (Mendes *et al.*, 2018 and Liu *et al.*, 1999).

The elastic modulus obtained for specimens 1 and 2 at 30ºC, 214.53 ± 3.308 GPa and 282.43 ± 3.217 GPa, respectively, were persistent with the literature, being between 270- 377 GPa for polyamide 6 and 173- 508 GPa for polyamide 6.6 at room temperature. For Specimen 1 there was a decrease of 23.6% of the Young's Modulus at 50ºC and 42.81% at 70ºC, for Specimen 2 the decrease in both temperatures was smaller, 12.12% at 50ºC and 17.35% at 70ºC.

Table 1. Experimental data of Specimen 1
Strain rate 8mm/min.

Temperature tested	Young's Modulus (GPa)
30 ºC	214.53 ± 3.308
50 ºC	163.91 ± 1.808
70 ºC	122.70 ± 1.372

Table 2. Experimental data of Specimen 2
Strain rate 8mm/min.

Temperature tested	Young's Modulus (GPa)
30 ºC	282.43 ± 3.217
50 ºC	249.08 ± 3.202
70 ºC	234.26 ± 3.429

4. CONCLUSIONS

The objective of this study was to evaluate the tensile properties of different types of polyamide fiber during isothermal tests. Two different fibers were investigated in three temperatures, 30ºC, 50ºC and 70ºC. Images of the actuators were acquired during the test and through post-processing of these images, with the method of Digital Correlation of Images (DIC); it was obtained the fields of displacement of the body. These were used to provide the longitudinal deformation. The method of correlation of Digital Images has the advantage of generating these fields of displacement of the material without the need to be in contact with the specimen inside the heating chamber.

The data obtained for the isothermal tensile tests and Young's Modulus for the specimens of Specimens 1 and 2 are consistent with large range presented by the literature. It was observed a greater loss in the elastic modulus in the

Specimen 1, 23.6% at 50ºC and 42.81% at 70ºC, compared with Specimen's 2 decreases, 12.12% at 50ºC and 17.35% at 70ºC.

As mentioned before the data reported for plastics are not sufficient relevant in contrast with data sheets for metals. This research has shown that other factors, such as temperature and form, must be taken into account while investigating plastics' properties. This work has preliminary character; more tests will be carried out in the future in order to evaluate the influence of the presence of water in the polyamide fiber properties.

5. ACKNOWLEDGEMENTS

The authors would like to acknowledge the financial support provided by the Brazilian Government funding agencies CNPq, FAPERJ, and CAPES.

6. REFERENCES

Arabnejad, Saeid, et al. "Shear-induced conformation change in α-crystalline nylon6." *Applied Physics Letters* 105.22 (2014): 221910.

Canevarolo Jr, Sebastião V. "Ciência dos polímeros." *Artiliber editora, São Paulo* (2002): 110-115.

Cerrada, María L. "Introduction to the Viscoelastic Response in Polymers." (2005).

Choy, C. L., F. C. Chen, and K. Young. "Negative thermal expansion in oriented crystalline polymers." *Journal of Polymer Science: Polymer Physics Edition* 19.2 (1981): 335-352.

Haines, Carter S., et al. "Artificial muscles from fishing line and sewing thread." *science* 343.6173 (2014): 868-872.

Kagan, Val A., and Nanying Jia. Tensile Properties of Semi-Crystalline Thermoplastics-Performance Comparison under Alternative Testing Standard. No. 2000-01-1319. *SAE Technical Paper*, 2000.

Khanna, Yash P. "Overview of transition phenomenon in nylon 6." *Macromolecules* 25.12 (1992): 3298-3300.

Lewin, Menachem. *Handbook of fiber chemistry*. Crc press, 2006.

Lincoln, Derek M., et al. "Temperature dependence of polymer crystalline morphology in nylon 6/montmorillonite nanocomposites." *Polymer* 42.25 (2001): 09975-09985.

Liu, Limin, Zongneng Qi, and Xiaoguang Zhu. "Studies on nylon 6/clay nanocomposites by melt-intercalation process." *Journal of Applied Polymer Science* 71.7 (1999): 1133-1138.

McIntyre, J. Eric, ed. Synthetic fibres: nylon, polyester, acrylic, polyolefin. Taylor & Francis US, 2005.

Mendes, Sarah S., Melo, Amanda, Moreira, Carolina S., Nunes, Luiz," Investigação do comportamento térmico de monofilamentos de poliamida usando Correlação de Imagens Digital" *CONEM – X Congresso Nacional de Engenharia Mecânica",* 2018

Peeters, Anik, et al. "Ab initio calculation of the Young's modulus of α-polyamides." International journal of quantum chemistry 87.5 (2002): 303-310.

PERARO, James S. Limitations of Test Methods for Plastics. ASTM, 2000

Raz, Karel, and Martin Zahalka. "Tensile strength of various nylon PA6 specimen modes." *AIP Conference Proceedings.* Vol. 1846. No. 1. AIP Publishing, 2017.

Schreier, Hubert, Jean-José Orteu, and Michael A. Sutton. *Image correlation for shape, motion and deformation measurements*. Springer US, 2009.

Sedighiamiri, A., et al. "Micromechanical modeling of the elastic properties of semicrystalline polymers: A three-phase approach." *Journal of Polymer Science Part B: Polymer Physics* 48.20 (2010): 2173-2184.

Shan, Gui-Fang, et al. "Effect of temperature and strain rate on the tensile deformation of polyamide 6." *Polymer* 48.10 (2007): 2958-2968.

Van Krevelen, Dirk Willem, and Klaas Te Nijenhuis. *Properties of polymers: their correlation with chemical structure; their numerical estimation and prediction from additive group contributions*. Elsevier, 2009.

Wasilkoski, CM. Comportamento Mecânico Dos Materiais Poliméricos. 2006. 143f. Diss. Tese (Doutorado em Engenharia Química)-Programa de Pós-Graduação em Engenharia Química-Universidade Federal do Paraná, Curitiba-PR.

Wypych, George. Handbook of polymers. Elsevier, 2016.

7. RESPONSIBILITY NOTICE

The authors are the only responsible for the printed material included in this paper.