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Estimation of the Tensile Behavior of Polyamides at different Temperatures

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Abstract: The present paper proposes a unified expression to estimate the mechanical behaviour of polyamides in tensile tests at different temperatures. The goal is to present an algebraic equation able to describe the stress-strain curves that would be obtained using the ASTM D638-14 standard for a wide range of temperatures (roughly from -30ºC to 90ºC, even above the Glass Transition Temperature). This expression is valid for different kinds of polyamides. It is shown that with a minimum of three tests performed at different temperatures, it is possible to obtain a reasonable approximation of the stress-strain curve for this range of temperatures. Examples are presented considering different types of polyamides – PA11, PA12 and PA66-GF30 (PA66 reinforced with 30 wt% of short glass fibres). Although the mechanical behaviour is strongly affected by temperature variation, the estimates of the material behaviour are in good agreement with experiments in all cases studied in this paper. Such a procedure can be a practical tool for designers, used to obtain a reasonable approximation of tensile tests and thus reducing the need of performing many complex experiments.

Keywords: Polyamides, Tensile Tests, Temperature, Mechanical Behavior, Modeling

INTRODUCTION

Thermoplastic polymers are being used throughout the years in infrastructures exposed to harsh environments involving high and low temperatures or marine environments. Polyamides or nylons are a type of thermoplastic materials. They are being adopted in engineering applications due to their high stiffness/weight ratio, high strength/weight ratio and corrosion resistance. Industrial applications include automotive, aeronautical and Oil and Gas industries. There are many studies regarding the effects of temperature and strain rates on the tensile behaviour of polymers (Reis, 2015; You, 2018; Şerban, 2013; Acharya, 2017; Zhang, 2009). Low and high temperatures induce changes in mechanical properties. It is necessary, therefore, to carry out a mechanical characterization over the complete range of temperatures that can be reached during the operational life of components made of these materials.

The goal of the present paper is to present a unified algebraic expression able to describe the stress-strain curves up to a 5% elongation obtained using the ASTM D638-14 (ASTM D638, 2014) standard for a wide range of temperatures (from temperatures below 0ºC to 90ºC, above the Glass Transition Temperature). The fact of dealing with small strains allows the use of very simple expressions. The study is motivated by engineering applications in which the maximum admissible strain is small, but the temperature can vary from below zero to above the glass transition temperature, such as in the case of internal pressure sheath of flexible pipes. In applications such as in in ultra-deep reservoirs in offshore exploitation, the oil temperature can be very high and the temperature of the pressure sheath in a flexible pipe vary a lot depending on the depth and can be above the glass transition temperature. Polyamide is commonly used as a pressure sheath material and understanding the mechanical behaviour of this polymer material is crucial to a safe and reliable operation.

A simple exponential type equation in which two parameters are quadratic functions of the temperature is proposed. The goal is not to discuss a general model for polyamides, but to approximate the stress strain curves with a minimum number of tests. These approximations can be used as an input to any constitutive model. It is shown that with a minimum of three tests performed at different temperatures it is possible to obtain a reasonable approximation of the stress-strain curve for this range of temperatures.

Tensile tests at different temperatures following ASTM D638-14 (ASTM D638, 2014) have been performed using samples of three types of polyamides: PA12 and PA11 used in inner polymeric pressure sheath layers of flexible subsea pipes for deep-water and ultra-deep-water offshore oil exploration and PA66-GF30, a composite reinforced with 30 wt% of short glass fibres used in different industrial application such as water drain valves and car intakes, use of reinforced thermoplastics in many sectors has led to exigency of higher mechanical properties for injection-moulded components (Lafranche, 2007). The experimental identification of the material constants necessary to use this unified expression is discussed, and the constants obtained for the three materials are compared. The estimates of the stress-strain curves are in good agreement with experiments, showing that engineers and designers can use this simplified procedure as a preliminary approach to understand how the stress-strain curve obtained using the ASTM D638-14 (ASTM D638, 2014) standard is affected by temperature without the need of performing many experiments.

MATERIAL AND EXPERIMENTAL PROCEDURES

Three different polyamides were considered in the study: PA66-GF30 used in different industrial application such as water drain valves and car intakes and PA11, PA12 used in inner polymeric pressure sheath layers of flexible subsea pipes for deep-water and ultra-deep-water offshore oil exploration and.

PA66-GF30 is a composite material manufactured with polyamide 66 reinforced with 30 wt% of short glass fibres (PA66-GF30) and provided by RADICI® under the commercial name of RADILON® AN6630HSL. This composite material is used in different industrial application such as water drain valves, car intakes and oil pipes. Specimens were injection moulded from this grade. Polyamide 66 reinforced with 30% short glass-fibre was injected in a mould according to ASTM D 638-14 (ASTM D638, 2014). The tensile tests in this case were performed at 12 different constant isothermal temperatures from -30ºC to 80ºC in 10ºC steps. 12 batches were used at 12 different constant temperatures from -30ºC to 80ºC with 5 specimens for each batch. Tensile test specimens for PA11 and PA12 were machined from an internal pressure sheath of flexible pipes used in the oil industry. 12 batches were used at 5 different constant temperatures (0ºC, 20ºC, 50ºC 70ºC and 90ºC) with 5 specimens for each batch.

All tests were performed using a thermostatic chamber attached to a Shimadzu® AG-X universal testing machine according to ASTM D638 (ASTM D638, 2014) at a prescribed elongation rate of 5 mm/min. This chamber allows to perform tests from -50ºC to 320ºC.

RESULTS AND DISCUSSION

Tensile tests

From now on, the classical uniaxial engineering stress and engineering strain will be noted, respectively, σ and ε will be called simply stress and strain Eq. (1).

$$\sigma(t) = \frac{F(t)}{A_0} \quad ; \qquad \varepsilon(t) = \frac{\Delta L(t)}{L_0} \tag{1}$$

$F(t)$ is the axial force necessary to impose an elongation $\Delta L(t)$ of the useful portion of the specimen at a given instant *t*. L_0 is the gauge length and A_0 the cross-section area. Specimens for the PA 11, PA 12 and PA66-GF30 are tested until a deformation of 5% ($\varepsilon < 0.05$) at different temperatures.

A unified expression for the stress - strain curve at different temperatures

The goal of this section is to propose a simple analytical expression to estimate the stress - strain curves in region I at different temperatures within a given range, using a minimum number of experiments. This expression is supposed to be adequate for very different types of polyamides such as the superplastic specimens of PA 11 and PA 12 and the fibre reinforced PA66-GF30 specimens.

Based on previous experimental works in the Laboratory of Theoretical and Applied Mechanics, such as (da Costa Mattos, 2016), the following $\sigma = a(\theta)[1-\exp(-b(\theta)\varepsilon)]$ unified expression is proposed to describe the stress-strain curve in a monotonic test under controlled strain following the ASTM standard D638-14 (ASTM D638, 2014)

$$\sigma = a(\theta)[1-\exp(-b(\theta)\varepsilon)] \tag{2}$$

with

$$a(\theta) = a_o + a_1\theta + a_2\theta^2 \tag{3a}$$

$$b(\theta) = b_o + b_1\theta + b_2\theta^2 \tag{3b}$$

The parameters $a(\theta)$ and $b(\theta)$ are quadratic functions of the temperature θ and a_o, a_1, a_2, b_o, b_1, b_2 are material constants that characterize a particular polyamide. These parameters have a precise definition from the stress-strain curve. From Eq. (2) it is possible to conclude that

$$\lim_{\varepsilon \to \infty} (\sigma) = a \text{ and } \left.\frac{d\sigma}{d\varepsilon}\right|_{\varepsilon=0} = a\ b \tag{4}$$

$a(\theta)$ is the maximum stress and $a(\theta)b(\theta)$ is the initial slope of the stress-strain curve at a given temperature θ if Eq. (2) is considered.

The coefficients a_o, a_1, a_2, b_o, b_1, b_2 can then be obtained by another curve fitting from the experimental curves $a \times \theta$ and $b \times \theta$ if an adequate number of tests are performed. However, these can be considered as particular cases of a second order polynomial. This expression is conceived for a given range of temperatures $\theta_{min} \leq \theta \leq \theta_{max}$. It is difficult to present a precise definition of the limiting temperatures θ_{min} and θ_{max}. In the absence of a precise physical definition, it is suggested that a range from -30ºC to 90ºC (as yet commented, in some applications such as in the pressure sheath in a flexible pipe, the polymers can be subjected to a temperature that is above the Glass Transition temperature T_g, hence $\theta_{max} > Tg$).

The model can be considered adequate if a good prediction of the stress-strain curve in all the temperature range is obtained from a minimum number of tests. Since a quadratic expression was suggested for the parameters a and b in Eq. (3a) and Eq. (3b), the minimum possible number of tests is 3.

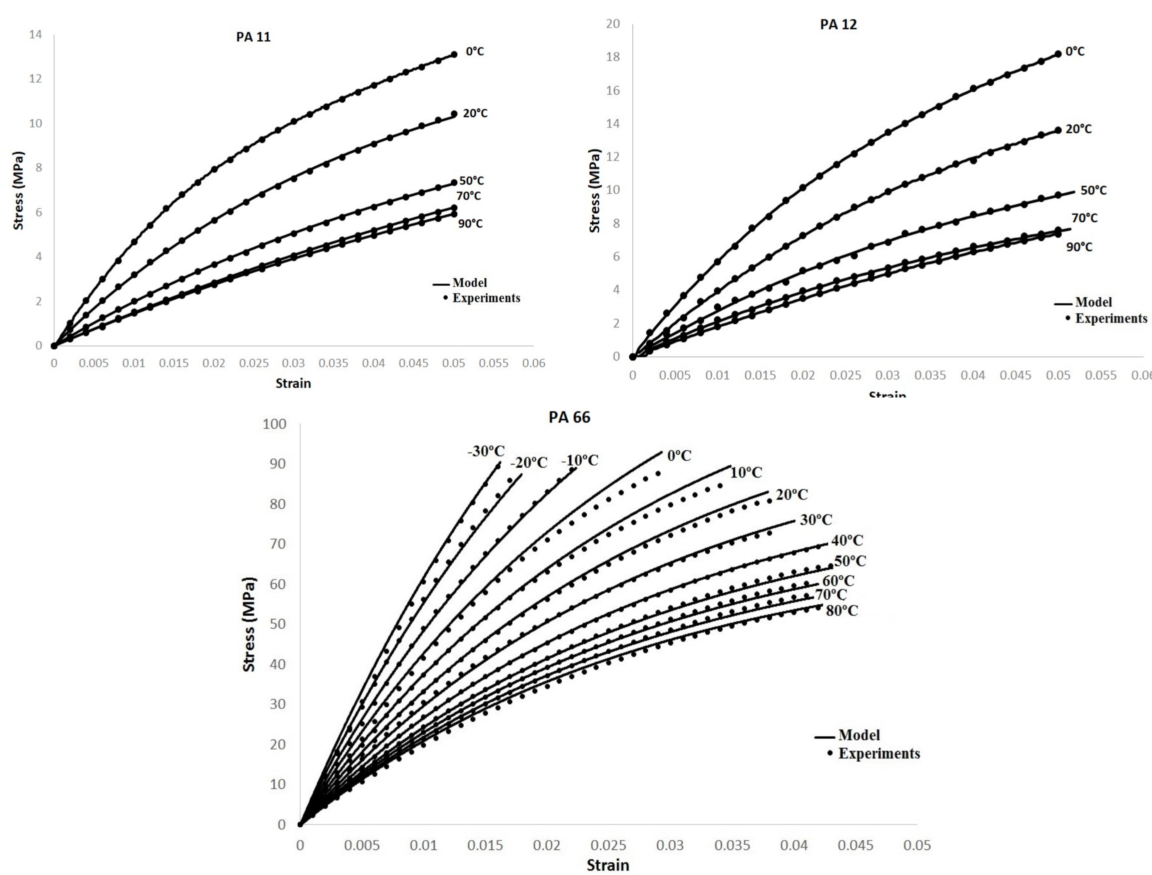

Figure 1 – Typical stress vs. strain curves of PA11, PA12 and PA66 at differente temperatures

Figure 1 shows the behaviour of the parameters a and b as functions of temperature, respectively, for the polyamides PA 11, PA 12 and PA66-GF30. A second-degree (maximum) polynomial gives an adequate fitting in all cases. It is interesting to observe that the parameter b is temperature insensitive in the case of PA66-GF30. Table 1 presents the values coefficients a_o, a_1, a_2, b_o, b_1, b_2 identified experimentally.

Table 1: Coefficients for Eqs. (2), (3a) and (3b) considering θ_{max} = 90 °C.

Material	a_o (MPa)	a_1 (MPa/°C)	a_2 (MPa/(°C)^2)	b_o	b_1 (°C)$^{-1}$	b_2 (°C)$^{-2}$
PA 11	15.472	-0.056	0.006	34.942	-0.5519	0.0031
PA 12	25.479	-0.3549	0.0028	25.117	-0.0524	-0.001
PA 66	145.07	-1.8098	0.0112	35	0	0

CONCLUSIONS

In this study, the temperature dependency of PA 11, PA 12 and PA66-GF30 is investigated. A unified expression is proposed to describe the mechanical behaviour of polyamides under small strains in tensile tests performed at different temperatures using the ASTM D638-14 (ASTM D638, 2014) standard. This unified expression can be used as an auxiliary tool by engineers and specialists for a quick estimate of the stress-strain curve. Temperature influences notably tensile strength, ductility and stiffness. This expression is found to be adequate to predict the temperature dependent responses of nylons in tensile tests under isothermal conditions at various temperatures. Second-degree polynomials give an adequate modelling of the temperature dependent parameters a and b that arise in the theory. Thus, all the material constants in the model can be identified experimentally using a minimum of three tensile tests. The expressions proposed in this paper allow a simple, but adequate, estimate of the stress strain curves for a wide range of temperatures. If the Glass Transition Temperature is adopted as the maximum operation temperature, the parameters a and b vary almost linearly with the temperature, what allows including other effects, such as the ageing due to hydrolysis

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