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# Methodology of Accelerated Characterization for long-term Creep Prediction of polymer structures to ensure their service life

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**Abstract:** Predicting long-term creep behaviour (10 or 20 years) is important in the development and design of thermoplastic structures to ensure their service life. But providing robust material data to engineers is not easy, as testing is expensive and time-consuming. **The purpose of this paper is to propose a new Methodology of Accelerated Characterization for long-term creep Prediction (MACcreeP) which gives more material information much faster and at a lower cost, than Classical Methodology (CM) used today by engineers. In order to prove the effectiveness of this new method, the material used for the study is a well-known high density polyethylene grade (HDPE). More precisely MACcreeP is a characterization protocol based on creep tests performed in true tensile stress conditions over short time periods (24h) and at different temperatures combined with the Time-Temperature Superposition Principle to construct master curves. In addition to the material data obtained over a short period of time, the data generated by the behaviour laws and the use of the CREEP law in a FEA software allows a more predictive numerical response than in CM. Finally, this methodology, which implies very few tests, allows to compare and choose the appropriate material properties to design a thermoplastic structure which will comply with end-use product requirements.**

**Keywords**

Creep test / Modulus Creep / Polyethylene / True stress / Standard test / Long-term

## 1 Introduction

Predicting the long-term creep behaviour of thermoplastic materials and particularly of semi-crystalline materials has been a major issue for several decades. This problem concerns not only the academic world but also the industry producing these materials as well as those who design structures for a life span of 10 or 20 years according to specifications. There are many articles in the literature covering this problem [1-11], different materials [12-18] and different experimental approaches.

There are mainly two standards defining the tests to characterize the creep behaviour of plastics. These are ISO 899-1 [19] and ASTM D 2990 [20]. Both standards are commonly used by material suppliers to provide the creep modulus at 100, 1000 and/or 5000 hours. In these creep tests, dumbbell-shaped samples are loaded using a dead weight [19]. Thus, the samples are subjected to a constant nominal stress ( $\sigma_N = F/S_0$ ) where  $F$  is the applied load and  $S_0$  is the initial cross-section of the sample. The curves obtained from these tests are expressed by the evolution over time of the nominal strain,  $\varepsilon_N = (L-L_0)/L_0$ , where  $L_0$  and  $L$  are the initial and current values of the sample gauge length, obtained with an extensometer. Obviously, nominal stress and strain represent the intrinsic behaviour of the material only if the strain is homogeneous all over the gauge length. This is the reason why fundamental research has been often limited to very small strains. In previous studies on polyethylene [17,21], authors have shown that, for  $\varepsilon_N < 0.05$ , the creep strain was evenly distributed, controlled by viscoelastic processes and largely reversible. Conversely, for tests performed under higher stresses with polyethylene and polypropylene [5,22], it was observed that the strain becomes more and more unstable (necking) and irreversible.

In addition, standards ISO 899-1 and ASTM D 2990 provide guidance on the use of long-term creep data and long-term relaxation data for the prediction of mechanical properties beyond the duration of the experiment. In general, to extrapolate the moduli to 10 or 20 years, these tests have a minimum duration of 5000h. Then, using different fitting methods, an extrapolation is performed to determine the long-term modulus. Extrapolation of the creep is done by fitting a power law curve to the modulus versus time data over a time period typically from 100 to 5000 hours and extracting the value at 10, 20 or 50 years. Generally the design engineer is satisfied with the creep modulus determined at a given nominal stress and a given point in time to perform a static numerical calculation by FEA (normally using a geometric nonlinear analysis). By doing so, the law of material behaviour is reduced to linear elasticity with an elastic modulus but takes into account large displacement effects. In principle, for technical applications, a complete set of creep tests should be performed over a wide range of loads and temperatures. As the duration of these tests is long, a full creep campaign is very expensive. Therefore, it is useful to have an alternative methodology able to predict long-term creep kinetics based on robust behaviour law determined from tests performed over shorter time periods.

This other approach consists in carrying out tests over short time periods and then extrapolating over long time periods by methods described in the literature. Different techniques have been used in many of the reported attempts to measure the true stress–strain relation of polymers [15,23]. In this study, the optical technique developed by G'Sell et al [23] where four markers placed on one face of the specimen are followed. The displacement of these markers is used to calculate the actual axial stress (true stress). The true stress was calculated using the measured transverse strain and assuming transverse isotropy.

This characterization method is interesting because it has been shown that the non-homogeneous decrease in cross-section during creep tests causes a progressive increase in real stress that cannot simply be deduced from the nominal strain.

For many years, the Time-Temperature Superposition Principle (TTSP) [1,2] has been used to predict the long-term behaviour of polymeric materials based on short-term experimental data. This principle is commonly applied with Burgers [1,2,8] and Findley [7,8] models.

Since higher temperatures and stress levels cause an acceleration of creep strain and shorten material relaxation times, there is a time and stress analogy similar to the time and temperature analogy described by TTSP. This time-stress analogy is mostly referred as Time-Stress Superposition Principle (TSSP) [24,25]. For example, Eftekhari et al [25] used this principle with Findley's power law to represent non-linear viscoelastic creep curves and to predict long-term creep behaviour from short-term test data of neat, talc-filled, and short glass fiber reinforced injection molded thermoplastic composites. Still later, the Time-Temperature-Stress Superposition Principle (TTSSP) was proposed by many researchers and used as a more potential accelerated prediction procedure for long-term performance of viscoelastic materials [6,11,16,26]. As pointed out in [11], the TTSP and TSSP can be considered as two reduced forms of the TTSSP. Many efforts have been made to use TTSP or TSSP to model and predict the long-term viscoelastic behaviour of polymeric materials, such as low-density polyethylene [18], high-density polyethylene [11,17], polypropylene [12,24], polycarbonate [13,14] and many other composite materials [25,27,28]. These methods allow the construction of a creep master curve with tests performed over short times (24 hours). More recently, a new accelerated creep test method called Stepped Isothermal Method (SIM) has been proposed by Thornton et al [29] to predict the long-term creep behaviour of geogrids used in soil reinforcement applications. Later, Alwis [30,31] applied this technique to characterize the creep of Kevlar 49 yarns. This method is a derivative of the classic TTSP method. Recently, a few studies [32,33] considered the Stepped isoStress Method (SSM), a derivative technique of the SIM method. The approach in SSM testing is similar to that of the SIM method but the acceleration of the creep is obtained by increasing the stress level in steps rather than stepping up the temperature. While this technique was developed and used

to predict the creep behaviour of technical yarns, Hadid et al. [9] showed that it could be used to predict the long-term creep of a thermoplastic sample (polyamide 6) with a high thickness.

At the present time, this research work has not led to an alternative standard proposed to design offices to establish the behavior over long periods of time by minimizing the tests and by defining an efficient model to make the predictive calculation of structures.

In this framework the aim of this paper is to compare two approaches, in order to carry out numerical simulations to design polymer structures subjected to low loads in the long term (10, 20 years). The first approach, called "Classical Methodology" (CM), is commonly used by engineers in design offices. It therefore consists in fitting experimental data from standardized creep tests [19,20] performed on 1000 or more generally 5000h from a power law. Once the parameters of this power law have been found, it is then possible to extrapolate and to identify the modulus at 10 or 20 years (as per specifications) according to the stress required for dimension design. For example, according to standard [34] for tank design, this value is 4MPa for a majority of thermoplastic materials. In the end, the numerical simulation is carried out statically, with a material whose behaviour is linearly elastic and whose modulus is that obtained as described above.

The second method proposed in this paper, called the **Methodology of Accelerated Characterization for long-term creep Prediction (MACcreeP)**, has been developed by the authors over the past fifteen years. The objective was, based on peer-reviewed techniques and methods, to provide long-term creep data for engineers in the shortest possible time and at the lowest cost. In order to provide long-term creep data, this MACcreeP methodology is based on tensile tests at constant strain rate and creep tests under constant true stress [23] (gauge length surface) for 24 hours **and a different temperatures**. Using the Time-Temperature Superposition Principle (TTSP), the most commonly used extrapolation technique, and a creep behaviour law available in most FEA software, it provides the detailed data that engineers need to undertake a creep analysis **over more or less time (from a few tens hours to several years)**.

Section 2 describes material, both creep characterization methods and experimental systems. In the first two parts of section 3, the two methodologies (Classical and MACcreeP) describing the characterization and the identification of the behaviour law are presented. Then, the results of the tensile creep test derived from the standard test method and then the actual stress control method are discussed. The initial creep curves are presented as well as the construction of the master curves for each stress level. The third part is concerned with the comparison of both methodologies for creep in the medium and long-term. Then the fourth part presents the numerical part and compares the two methodologies on a simple model of a tank subjected to internal pressure. The last part of section 3 presents two materials characterized by the MACcreeP methodology for comparison and evaluation. The interest of the methodology presented in this paper in comparison with the Classical Methodology (CM) commonly used in design offices is discussed in the conclusion.

## 2 Material and Experimental

### 2.1 Material

The characterization methodology has been performed on a high density polyethylene grade (PE1) supplied by TOTAL. The Melt Flow Rate and the density of PE1 are 3.8g/10min. and 0.947 cm<sup>3</sup>/g, respectively, according to ISO 1133/D (190°C and 2.16kg) and ISO 1183 standards. PE1 is characterized by a glass transition temperature (T<sub>g</sub>) of -120°C. Some test have been performed on a second high density polyethylene grade (PE2) supplied by TOTAL and will be compared to PE1. The

Melt Flow Rate and the density of PE2 are 4.0g/10min. and 0.940 cm<sup>3</sup>/g, respectively, according to ISO 1133/D (190°C and 2.16kg) and ISO 1183 standards. PE2 is characterized by a glass transition temperature (T<sub>g</sub>) of -120°C.

PE2 polyethylene has a slightly lower density than PE1 polyethylene. If the modulus of PE1 obtained from a tensile test at 23°C and a strain rate of 10<sup>-3</sup> s<sup>-1</sup> is chosen as a reference ( $E_{23}^{PE1}$ ), then the modulus of PE2 under the same conditions is 21.3% lower. As the material data are confidential, the moduli will be expressed by % in this publication and calculated from the PE1 modulus at 23°C (100%) as reference. The PE2 modulus therefore has a relative value of 78.7%.

## 2.2 Standard creep test – ISO 899-1

In industrial tensile creep tests used for engineering purposes, the dumb-bell shaped specimens are loaded by means of a dead weight [19]. The tests are generally performed at 23°C. As such, the samples are subjected to a constant nominal stress, or Kirchhoff stress,  $\sigma_N = F/S_0$ , where  $F$  is the applied load and  $S_0$  the initial cross-section of the sample. The curves obtained from these tests are expressed as the evolution of the nominal strain or Lagrangian strain, with time,

$$\varepsilon_N = \frac{(L-L_0)}{L_0} \quad (1)$$

where  $L_0$  and  $L$  are the initial and current values of the gauge length of the sample, obtained with an extensometer.

For standard creep tests, the specimen dimensions (Fig. 1) are defined by ISO527-1 [35] or alternatively by ISO527-2 [36]. **The useful length is 100-110mm and the minimum cross-section is a rectangle with 13mm width and 3mm thickness.** Generally, they are punched out of compression moulded plates.



**Fig. 1. Creep tensile test specimen – Standard 527 [1, 2]**

## 2.3 Constant true stress creep test

Tensile tests and creep tensile tests are performed in an Instron Testing machine 1195 which is equipped with a 500N load cell. Temperature is regulated at a constant value ( $\pm 1^\circ\text{C}$ ). Since the tests are performed at isothermal temperature and the specimens are conditioned at the set temperature until stabilization, there is no thermal strain to be taken into account. Strains are computed from the displacement of four markers on the surface of the specimen using the Videotraction® software (distributed by ProViSys Engineering, France). The details on the strain calculation can be found in the publication by G'Sell [23,37,38]. During the test, a CCD camera (resolution 800 x 600 pixels and frame rates 1.875–15 fps) is following the four markers in real time. The logarithmic longitudinal ( $\varepsilon_l$ ) and transverse ( $\varepsilon_t$ ) strains are calculated following equations:

$$\varepsilon_l = \ln\left(\frac{L}{L_0}\right) = \ln\left(1 + \frac{\Delta L}{L_0}\right) \quad (2)$$

$$\varepsilon_t = \ln\left(\frac{W}{W_0}\right) = \ln\left(1 + \frac{\Delta W}{W_0}\right) \quad (3)$$

where  $L$  and  $L_0$ ,  $W$  and  $W_0$  respectively stand for the current and initial distance between longitudinal and transverse (in width direction) markers,  $\Delta L$  and  $\Delta W$  the increments of these values.

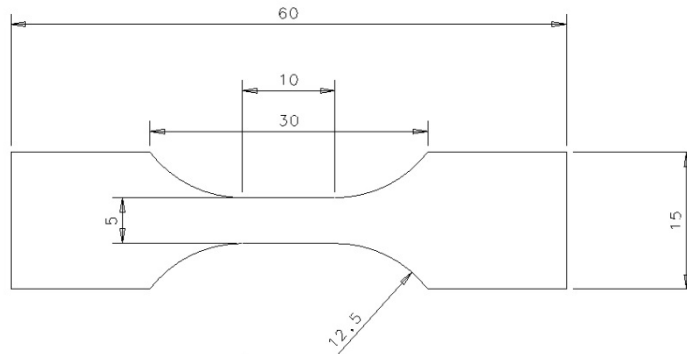
The longitudinal Cauchy stress (or True stress  $\sigma_v$ ) is calculated following equation (4), in an isochoric framework and assuming isotropy of transverse strains [38]:

$$\sigma_v = \frac{F}{S} = \frac{F}{S_0} \frac{1}{\exp(2\varepsilon_t)} \quad (4)$$

where  $S_0$  is the initial cross-section area,  $S$  is the actual cross-section area and  $F$  is the current axial load.

The tensile tests are carried out at one constant strain rate  $10^{-3} \text{ s}^{-1}$  with the Videotraction system and performed at different temperatures. In the creep procedure, the axial true stress is kept constant in the cross-section during the experiment. The actual cross-section is measured in real time and loading  $F$  is adapted to keep constant true stress by using the relation (4).

In order to locate the deformation in the region where all mechanical variables are determined, the dimensions of the ASTM D638M [39] specimens have been modified (Fig. 2). **The useful length is 10 mm and the minimum cross-section is a rectangle with 5 mm width and 4 mm thickness.** Samples are generally extracted from structures resulting from the final process (compression molded, injection molded, rotational molded, etc.) because depending on the process, the mechanical characteristics may be different for the same base material. Here, specimens are extracted from rotomoulded structures. The specimens for the creep tests are the same as those utilized for the tensile tests at constant true strain rate.



**Fig. 2. Creep tensile test specimen – ASTM D638M [3]**

#### 2.4 Time-Temperature Superposition Principle (TTSP)

Numerous creep experiments have been conducted to make long-term creep predictions from short-term creep tests. The most commonly accepted methods to achieve this involves the use of TTSP, which implies that viscoelastic behaviour at one temperature can be related to that at another temperature by a change in the time scale only. The detailed procedure is that creep tests performed at high temperatures for short periods can mimic creep behaviours performed at low temperatures on a long-term time scale [1,2]. In this study, creep experiments at various constant true stresses on polyethylene samples were performed at four temperatures (23, 40, 50 and 60°C) to predict behaviour up to 20 years. The creep data were then shifted horizontally along the logarithmic time axis until they overlapped to form a continuous master curve at a reference temperature. This master curve can be used to predict creep performance over a long time period. The corresponding shift factor  $a_T$  was described by Williams, Landel and Ferry [2] as:

$$\varepsilon_0(t, T_{\text{ref}}) = \varepsilon\left(\frac{t}{a_T}, T\right) \quad (5)$$

Where  $\varepsilon_0$  is the strain at reference temperature ( $T_{\text{ref}}$ ),  $t$  the time,  $\varepsilon$  the strain at the elevated temperature  $T$ . Compliance is commonly used to build the master curve and therefore determine the shift factor  $a_T$ . Thus, compliance is given by the following relationship:

$$D(t, T_{\text{ref}}) = \frac{\varepsilon_0(t, T_{\text{ref}})}{\sigma} = \frac{\varepsilon\left(\frac{t}{a_T}, T\right)}{\sigma} = D\left(\frac{t}{a_T}, T\right) \quad (6)$$

They also defined the WLF equation, which provides an empirical description of the shift factor  $a_T$  for amorphous polymers above the glass transition temperature [ $T_g$ ,  $T_g + 100$  °C],

$$\log a_T = -\frac{C_1(T - T_{\text{ref}})}{C_2 + (T - T_{\text{ref}})} \quad (7)$$

where  $C_1$  and  $C_2$  are arbitrary material constants whose values depend on the material and choice of reference temperature  $T_{\text{ref}}$  and  $T$  is in Kelvin. However, the WLF equation is not valid for semi-crystalline polymers at temperatures below their melting point. Seitz and Balazs [3] have shown that in this case, the description of the shift factor  $a_T$  follows an Arrhenius equation:

$$\log a_T = \frac{\Delta H}{2.303R} \left( \frac{1}{T} - \frac{1}{T_{\text{ref}}} \right) \quad (8)$$

where  $R$  is the universal gas constant and  $\Delta H$  is the activation energy. The activation energy can be obtained from the slope of the curve of  $\log a_T$  against  $1/T$ .

From the independent creep curves obtained under different stress and temperature levels, the conventional TTSP procedure is used. Then, the construction of the master curve requires some horizontal offsets in time of the rescaled creep curves.

## 2.5 Constitutive law

Total strain is defined as the sum of elastic strain (the relationship between stress  $\sigma$  and elastic modulus  $E$ ), creep strain and thermal strain:

$$\varepsilon_{\text{total}} = \varepsilon_{\text{elastic}} + \varepsilon_{\text{creep}} + \varepsilon_{\text{thermal}} \quad (9)$$

Since the tests are performed at isothermal temperature and the specimens are conditioned at the set temperature until stabilization, there is no thermal strain to be taken into account ( $\varepsilon_{\text{thermal}} = 0$ ). Creep strain can have linear (stress independent) or non-linear (stress dependent) viscoelastic behaviour.

In FEA software, creep behavior can be described by either viscoelastic laws (Prony series) or by viscoplastic laws (CREEP). As long as the release of the load of structure is not considered in the simulation, both laws will give the same results if the sign of load doesn't change. The creep behavior law used here is the viscoplastic law which is available in industrial computational codes and more particularly in Abaqus [40]. In addition, this law gives the possibility to describe the nonlinear behaviour by power law in the stress. The "time hardening" form is the simpler of the two forms of the power model:

$$\dot{\varepsilon}_{\text{creep}} = A \tilde{q}^n t^m \quad (10)$$

where  $\dot{\bar{\epsilon}}_{creep}$  is the uniaxial equivalent creep strain rate ( $\sqrt{\frac{2}{3} \dot{\epsilon}_{creep} : \dot{\epsilon}_{creep}}$ ),  $\tilde{q}$  is the uniaxial equivalent deviatoric stress,  $A$ ,  $n$  and  $m$  material parameters.

Uniaxial equivalent creep strain can be obtained by integrating the uniaxial equivalent creep strain rate with time:

$$\bar{\epsilon}_{creep} = \int_{t_0}^t \dot{\bar{\epsilon}}_{creep} dt \quad (11)$$

In a tensile creep test:

- the uniaxial equivalent deviatoric stress is equal to uniaxial stress in the direction of loading

$$\tilde{q} = \sigma, \quad (12)$$

- the uniaxial equivalent creep strain is equal to uniaxial creep strain in the direction of loading

$$\bar{\epsilon}_{creep} = \epsilon_{creep}. \quad (13)$$

When considering a linear load under stress as a time dependence, the creep strain is calculated by:

$$\epsilon_{creep} = \frac{A}{n+m+1} \left( \frac{\sigma}{t_0} \right)^n t_0^{m+1} + \frac{A}{m+1} \sigma^n [t^{m+1} - t_0^{m+1}]. \quad (14)$$

If an instantaneous load is considered i.e.  $t_0 = 0$ , then the creep strain is calculated by:

$$\epsilon_{creep} = \frac{A}{m+1} \sigma^n t^{m+1}. \quad (15)$$

which is identical to Findley's [7,8] with the modification made by Hadid [26].

It is important to note here that this creep law (Eq. 10) in finite element analysis software should be used with caution. As shown in equations 14 and 15, for the same set of parameters ( $A$ ,  $n$  and  $m$ ), the creep response will be different. Thus, the procedure of identifying these parameters depends on the imposed loading rate (instantaneous or temporal). In the case of standard creep tests (ISO899-1), only equation 15 is true. In the case of constant true stress creep tests, equation 14 can be used since the load increase to achieve the imposed true stress is controlled at a strain rate. Equation 15 can be used if the assumption of a fast loading is assumed. Finally, this creep law must be used only in the case of constant and/or increasing load but not in the case of mechanical discharge.

### 3 Results and Discussion

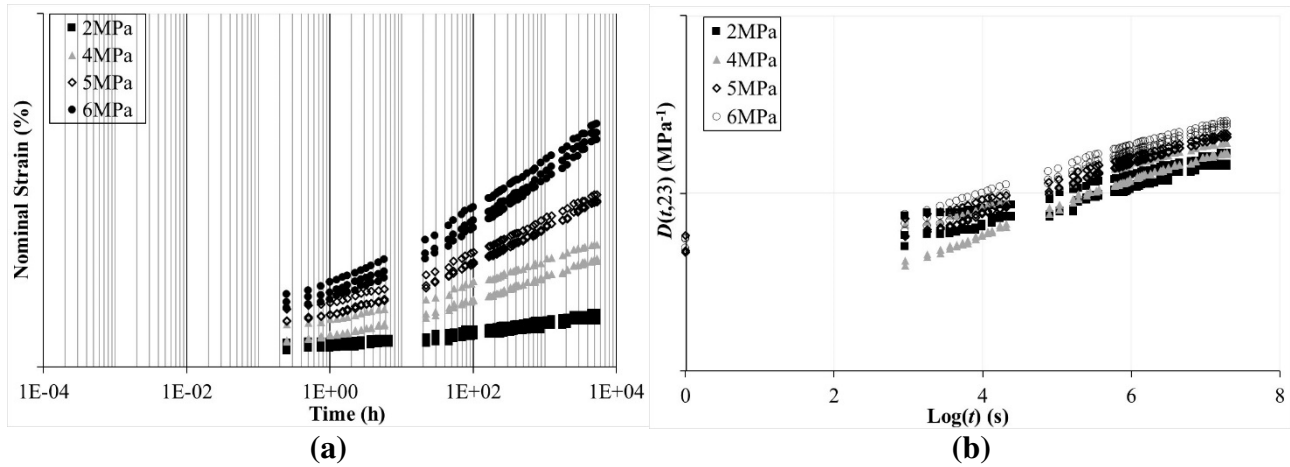
#### 3.1 Classical methodology

##### 3.1.1 Standard creep test – ISO 899-1

Creep tensile tests were performed according to standard ISO 899-1 [19] using the specimens from standard ISO527 [35,36] presented in Fig. 1. They are made by imposing a weight, therefore with constant load, so as to have a nominal stress ( $\sigma_N = F/S_0$ ) defined in the specimen at the start. Four stresses are imposed in this protocol. Tensile creep tests were undertaken at 2, 4, 5 and 6 MPa. Thus, three samples were tested at each nominal stress level over a period of approximately 5000 hours. Fig. 3a shows the responses for 5000 hours of PE1 under the four stresses. Another way of presenting



the creep results is represented as on Fig. 3b which shows compliance as a function of  $\log(\text{time})$ . The material PE1 therefore has a nonlinear (stress dependent) behaviour.



**Fig. 3. Creep curves under constant nominal stress (a) nominal strain (%) vs time (h) – (b) Compliance creep as a function of time (s).**

### 3.1.2 Fitting

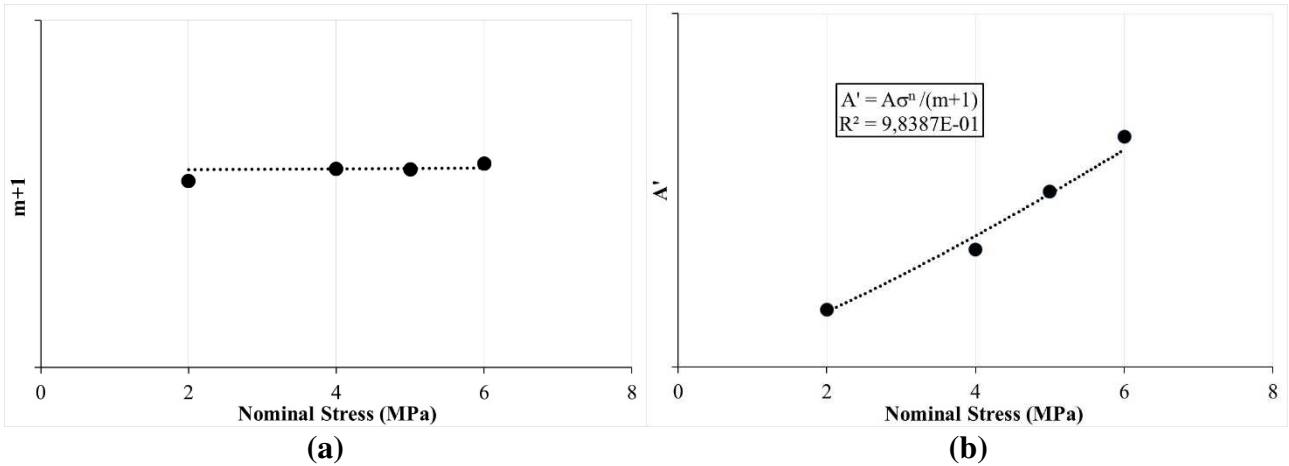
From these results, elastic and creep strains can be extracted. The first step of application of the load is important to identify the elastic part. As for this type of test, the load is almost instantaneous and the time scale for measuring the strain is small, consequently it is not easy to correctly identify the elastic modulus. It is better to, if a tensile test has previously been carried out, calculate the elastic strain by dividing the nominal stress imposed by the modulus. After separating the elastic strains and creep strains, the methodology consists in adjusting creep strains  $\varepsilon_{creep}$  as a function of time by the following expression:

$$\varepsilon_{creep} = A't^{m+1} \quad (14)$$

where  $A'$  is stress-dependent parameter,  $m$  a material parameter and  $t$  the time.

The fitting of the experimental data for the four stresses by the model given by equation 14 is performed over a period between 160 and 5000 hours. Since the load is considered instantaneous, equation 13 allows calculation of the three material parameters  $A'$ ,  $m$  and  $n$ . The Fig. 4a shows the value of  $m+1$  as a function of the stress. This term being independent of the stress, it is possible to select a constant value. Then knowing this value  $m$ , it is easy to determine  $n$  and  $A$ . It is sufficient to represent the values of  $A'$  of the equation (13) as a function of the stresses (Fig. 4b). The adjustment of the curve by a power law (Fig. 4b) gives directly  $n$ , and allows the determination of  $A$ .

Thus, with the creep data generated during the tests according to standard 899-1, the creep law (eq. 10) is identified over the range 160 - 5000 hours. Now, by extrapolating this law (eq. 13), it is possible to obtain the modulus at 10 and 20 years at a given stress. This is what the engineer uses to perform a geometric nonlinear stress analysis based on the derived long-term creep modulus. Alternatively, using equation 8, a nonlinear viscoelastic creep analysis can be carried out over the designated creep period which assumes the creep law based on engineering stress and strain is valid for an analysis based on true stress and strain.

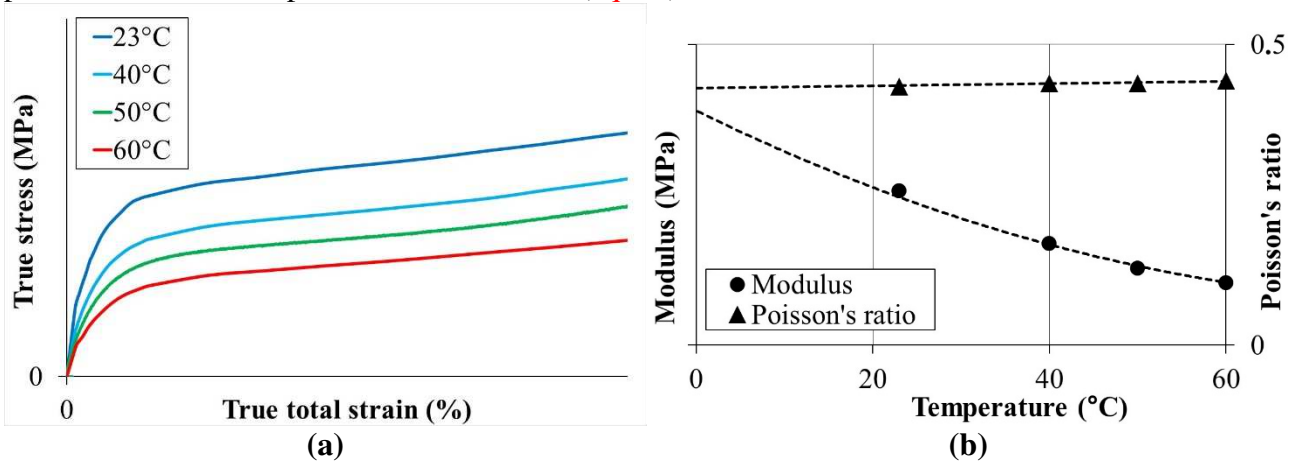


**Fig. 4. Identification of the parameters of the creep law within the framework of the classical methodology (a) Coefficient  $m$  – (b) Coefficients  $A$  and  $n$ .**

### 3.2 Methodology of Accelerated Characterization for long-term creep Prediction (MACcreeP)

#### 3.2.1 Tensile tests

In the protocol, tensile tests at a constant **strain rate** of  $10^{-3} \text{ s}^{-1}$  are performed at four temperatures (23, 40, 50 and  $60^\circ\text{C}$ ) with the system presented in 2.3 **and using the specimens from ASTM 638M [39] defined in Fig. 2**. From these tests presented in Fig. 5a, it is possible to identify the modulus (Fig. 5b) and the Poisson's ratio (Fig. 5b) as a function of temperature. These parameters will be used during the numerical simulation to define the elastic behavior of the material considered for the studied structure. Depending on the specifications, it will then be possible to establish the behavior law at different temperatures. In addition, the knowledge of the static modulus at different temperatures will allow to build a master curve at the desired temperature and to identify the specific parameters of the creep via the behavior law (eq. 10).

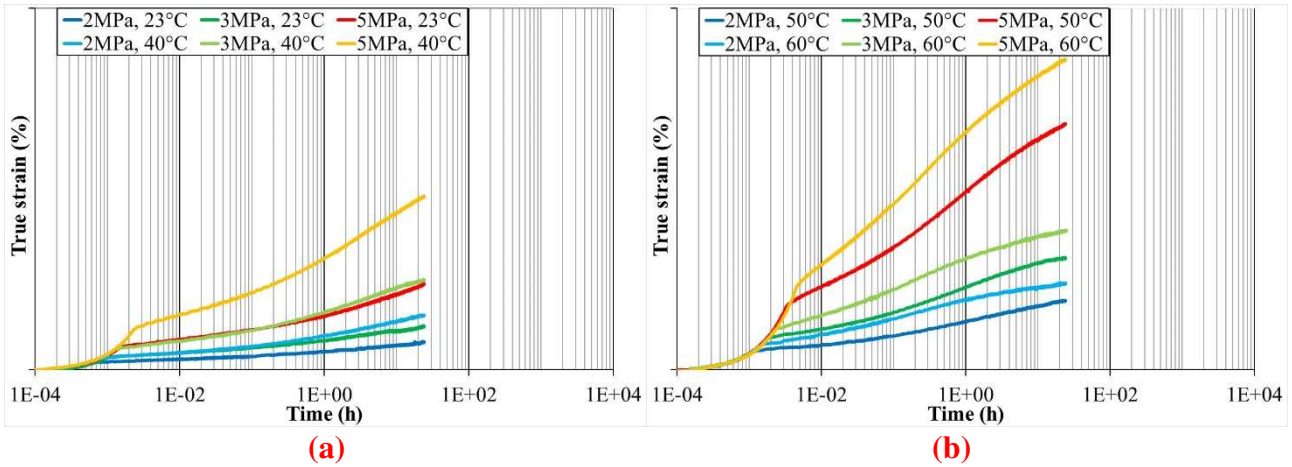


**Fig. 5. (a) Tensile curves at different temperatures – (b) Modulus and Poisson's ratio as a function of temperature**

#### 3.2.2 Constant stress creep test

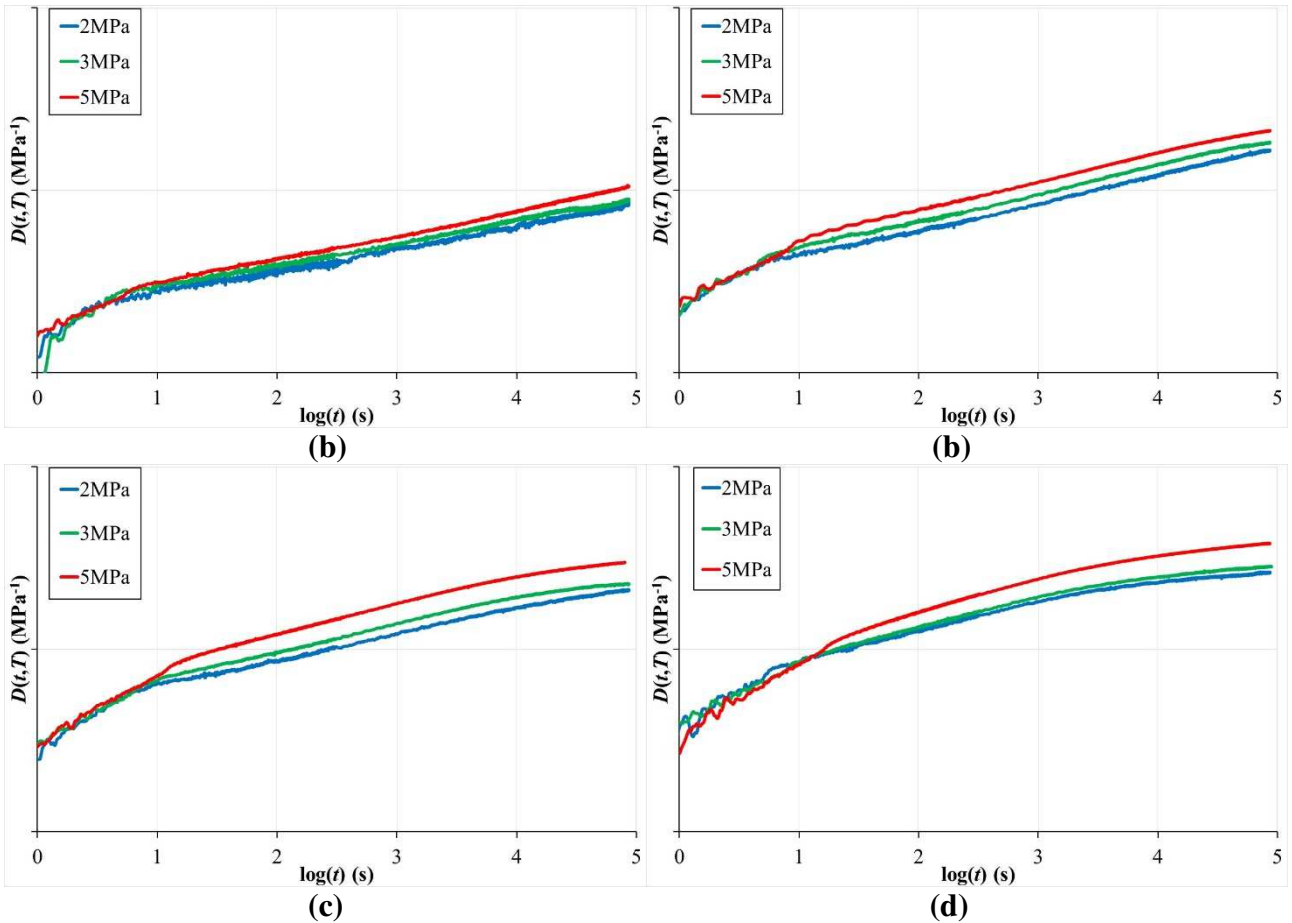
In the protocol, creep tests at a constant true stresses are performed at four temperatures (23, 40, 50 and  $60^\circ\text{C}$ ) for 24 hours with the system presented in 2.3 **and using the specimens from ASTM 638M [39] defined in Fig. 2**. Three constant true stresses are imposed: 2, 3 and 5 MPa. Fig. 6a and

Fig. 6b show the 24-hour responses of PE1 under the three constant true stresses at 23-40°C and 50-60°C respectively. The true strain axis of Fig. 6a and Fig. 6b are the same as for Fig. 3a.



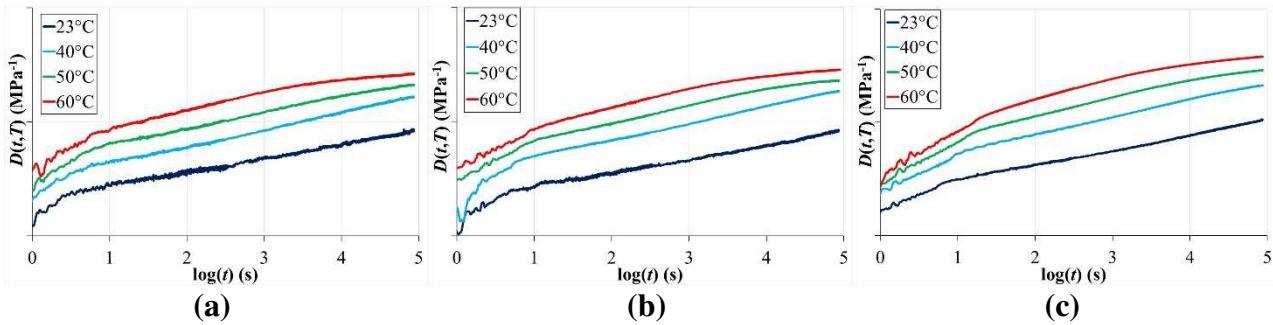
**Fig. 6. Creep curves under constant true stress (a) true strain (%) vs time (h) at 23 and 40°C– (b) true strain (%) vs time (h) at 50 and 60°C.**

The effects of the applied true stress level on compliance are examined using PE1 in the temperature range from 23 to 60°C. The observed variations of the compliance against the logarithmic time measured under different stress levels at different temperatures are plotted in Fig. 7. The positive dependence of the compliance on stress shows a typical nonlinear viscoelastic behaviour. Fig. 8a-b-c shows the influence of temperature on the compliance curves as a function of time for each of the stresses (2, 3 and 5MPa).

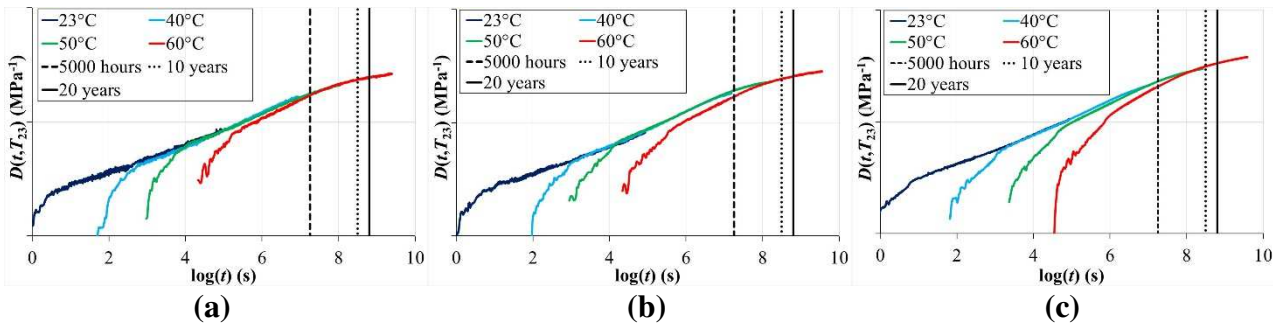


**Fig. 7. Stress dependence of creep curves for a given temperature (a) 23°C – (b) 40°C – (c) 50°C – (d) 60°C**

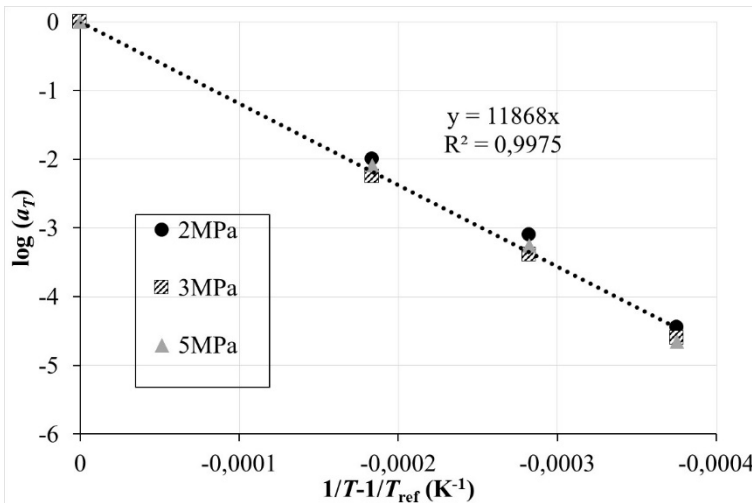
Using the Time-Temperature Superposition Principle (TTSP), it is therefore possible to build master curves for each stress. Fig. 9 a-b-c show these master curves at different temperatures for stresses 2, 3 and 5MPa respectively. On these graphs, the black vertical line, the black dotted vertical line and the black solid vertical line represent 5000h, 10 and 20 years respectively on the time axis. Fig. 10 shows the horizontal shift factor in function  $1/T-1/T_{ref}$ . The shift function checks the Arrhenius law (eq. 8) and is independent of the stress on the range 0-5MPa. This relationship allows to determine the activation energy which is 227.3 kJ/mol for PE1. This result falls within the range of Arrhenius parameters reported in the literature for polyethylene, whose activation energy ranges from 160 to 320 kJ/mol [41].



**Fig. 8. Temperature dependence of creep curves under a true stress (a) 2MPa – (b) 3MPa – (c) 5MPa**



**Fig. 9. The master curves for creep compliance at reference temperature 23°C for constant true stresses (a) 2MPa – (b) 3MPa – (c) 5MPa.**



**Fig. 10. Shift factor obtained for the PE1 specimens at various temperatures.**

### 3.2.3 Long-term identification

From the master curves at 23°C and the different stresses obtained previously (Fig. 9), it is now possible to estimate the modulus at any time over the 0-20 year range. And more particularly, for the long-term dimensioning of polymer structures, it is simple to know the 10 and 20 year modulus at 23°C. As shown in Fig. 9a-b-c, the 10-year modulus corresponds to one point of the experimental data obtained at 50°C over the 0-24 hour range. The 20-year modulus (black solid vertical line) corresponds to a point of the experimental data obtained at 60°C over the 0-24 hour range.

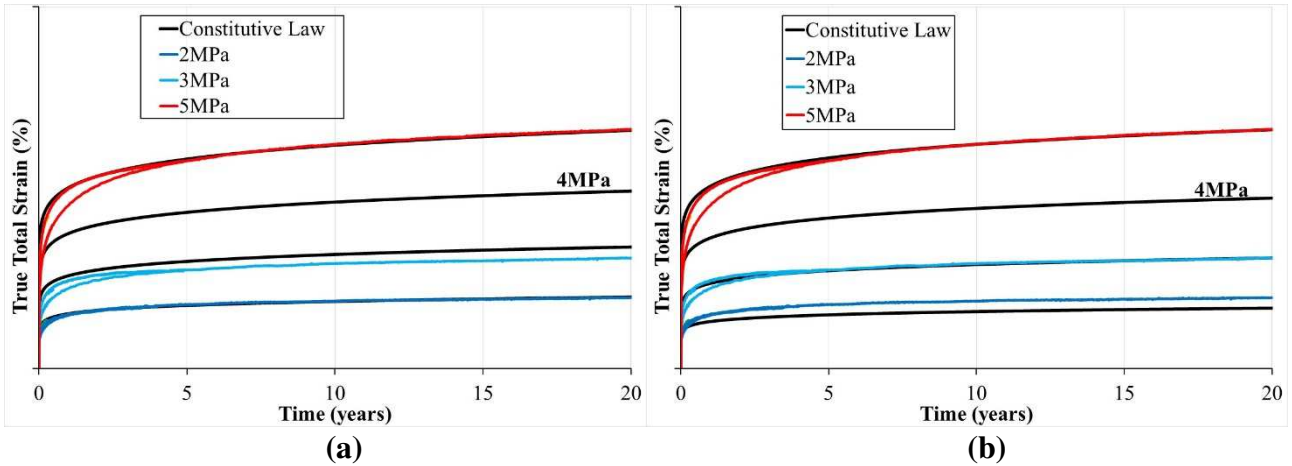
In order to have a creep behaviour law that can be used in FEA software, it is necessary to identify the parameters of this law (eq. 10) from the constructed master curves. The elastic modulus has been determined by tensile tests. It is presumptuous to think that with only one set of parameters of the standard creep law, it is possible to be predictive over the 0-20 year range. Thus, although the widest possible range is sought, we will always limit ourselves in order to identify the parameters as well as possible. Thus, to know here the behaviour at 10 and 20 years, the parameters of the creep law will be determined precisely over the range of 5 to 20 years. In this configuration, the loading and start of the master curves are secondary. Generally, during creep simulations on structures, loading is imposed instantly. The reason is that the first loading step is considered to have no impact on long-term creep. However, the use of the creep law (eq. 10) in FEA software can be of concern. Because as specified in 2.3, the result for a given set of parameters of the creep law will be different if an instantaneous load or a load is imposed over a defined period of time. To take into account the loading speed, it would then be necessary to have creep tests with different load increases and to ensure that the law as it is written today is able to account for this behaviour. In order to avoid this point, it is therefore considered that the rise is purely elastic, and that creep begins as soon as the real stress is reached and is applied instantly.

The identification of the parameters of the creep law consists in finding a set of parameters passing through the three master curves constructed at 2, 3 and 5MPa. By accumulating the uncertainties of experimental measurements of the strain and the shift of the curves to construct the master curves, it is illusory to have a set of parameters allowing a perfect fit to the three curves. Thus, two protocols are possible: either by imposing for identification that the law must fit the extreme curves (2 and 5 MPa) or between 3 and 5MPa.

If the value of  $m$  does not change, the other two parameters of the law  $A$  and  $n$ , which correspond respectively to the initial amplitude and the stress deviation, are slightly modified. In Fig. 11a and Fig. 11b, the master curves are superimposed on the different stresses, the behaviour law identified by imposing the master curves at 2 and 5MPa, and 3 and 5MPa respectively. In Table 1 are given the modulus for the different stresses and at 10 and 20 years, obtained from the behaviour law identified from the curves of 2 and 5MPa and 3 and 5MPa. These modulus are standardized with respect to the tensile modulus at 23°C ( $E_{23}^{PE1}$ ).

In the first case (identification via the 2 and 5MPa curves), the 10 and 20 year modulus at 3MPa are underestimated by 8% and 4.5% respectively compared to the modulus identified via the master curves. In the second case, the 10- and 20-year-old modulus at 2MPa are overestimated by 18% and 17% respectively. Finally, for these two protocols, this leads to a modulus difference at 10 and 20 years of 5.2% and 4.4% respectively for the 4 MPa stress.

As part of the MACcreeP methodology, the final parameters of the creep law will be those obtained from the identification of the experimental curves at 2 and 5MPa. Because these data allow to be predictive over a stress range between 2 and 5MPa. In the other case (3-5MPa), the 17-18% overestimation of the response to a 2MPa stress is far too high for the design of a structure with a stress field between 0 and 4MPa.



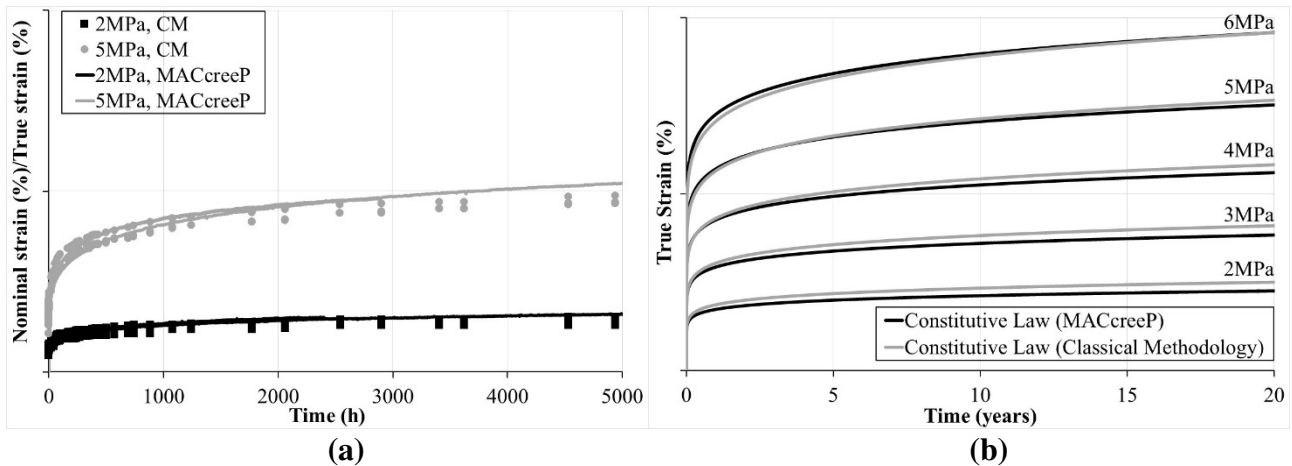
**Fig. 11.** Superposition of experimental data and the law of behavior identified (a) by taking the curves at 2 and 5MPa – (b) by taking the curves at 3 and 5MPa.

**Table 1.** PE1 moduli at 10 and 20 years identified according to the two different protocols

Methodology		MACcreeP			
Stresses (MPa)	Constant True Stress Law between 2 and 5MPa	Constant True Stress Law between 3 and 5MPa			
		10	20	10	20
Years	10	20	10	20	
2	13.2%	12.4%	15.6%	14.7%	
3	11.6%	10.9%	12.8%	12.0%	
4	10.6%	10.0%	11.1%	10.4%	
5	9.9%	9.3%	9.9%	9.3%	

### 3.3 Comparison of the two approaches for medium-term and long-term creep

By comparing the two approaches over the time range 150 - 5000 hours, Fig. 12a shows that up to 1000h the experimental data from the ISO 899-1 standard tests and the master curves at each stress from the constant true stress tests are almost superimposed. Between 1000 and 5000 hours, the creep rate seems to be slightly different in the two configurations. It is not possible here to conclude on this difference, because the differences in terms of tests are so important. It should be remembered that the dimensions of the samples are different, the measurement length of the samples is a factor 10 between both test methods. Finally, in the MACcreeP test method, the transverse strain measurement controls the machine to ensure that a constant real stress is maintained. For tests according to ISO899-1, the tests are at constant weight. Although the deformations are small and moderate, the actual true stress changes over time. Fig. 12b presents the creep curves over 20 years for the two methodologies constructed from the behaviour law and the identification of the parameters of the creep law (eq. 10). It is clear that over the 5-20 year stress range, the classical method underestimates behaviour.



**Fig. 12. (a) Superposition of experimental data (standard 899-1) and the master curves (MACcreeP) at constant true stress (2 et 5MPa) between 0 and 5000 hours – (b) Comparison of the behavior laws identified according to the two methodologies.**

Table 2 groups the different modulus of PE1 at 10 and 20 years obtained according to the two methodologies. For the Classical Methodology, these modulus are extrapolated after fitting the experimental data with the power law. For the MACcreeP methodology, they are determined via the behaviour creep law (eq. 10). As already mentioned, the reference modulus is that of PE1 at 23°C obtained from the tensile test.

Now, by comparing the two methodologies, except at 2 MPa, where the gaps at 10 and 20 years are more than 10%, the table shows that this gap decreases to be less than 4% for a stress of 4MPa at 10 and 20 years. In view of the possible measurement errors, the fitting by a theoretical law and the extrapolation, it can confirm that both methodologies lead to moduli at 10 and 20 years being almost identical.

Finally, it should be noted that although the polyethylene studied PE1 does not have the same density as compared to the density reported by Findley [42], the levels of strains appear to be in the same order of magnitude over the 0-20 year range.

**Table 2. PE1 modulus at 10 and 20 years identified by the two different methodologies**

Methodology	Classical		MACcreeP	
Stress (MPa)	Standard (extrapolation, fitting)		Constant True Stress Law between 2 and 5MPa	
Years	10	20	10	20
2	12.0%	11.2%	13.2%	12.4%
3	11.0%	10.2%	11.6%	10.9%
4	10.2%	9.6%	10.6%	10.0%
5	9.8%	9.1%	9.9%	9.3%
6	9.4%	8.7%	9.3%	8.7%

### 3.4 Illustration numerical simulation

Let's illustrate with an example. The objective in this paragraph is to determine the thickness of a rotomolded storage tank (Fig. 13a) with a diameter of 2340mm and a height of 3677 mm subjected to a hydrostatic pressure (Pink arrow on the Fig. 13a) of water over 99% of its height and to its own weight (Yellow arrow on the Fig. 13) during 20 years. **The determination of the thickness is based on the standard [34] which is listing the requirements for the design of storage tanks. This design**

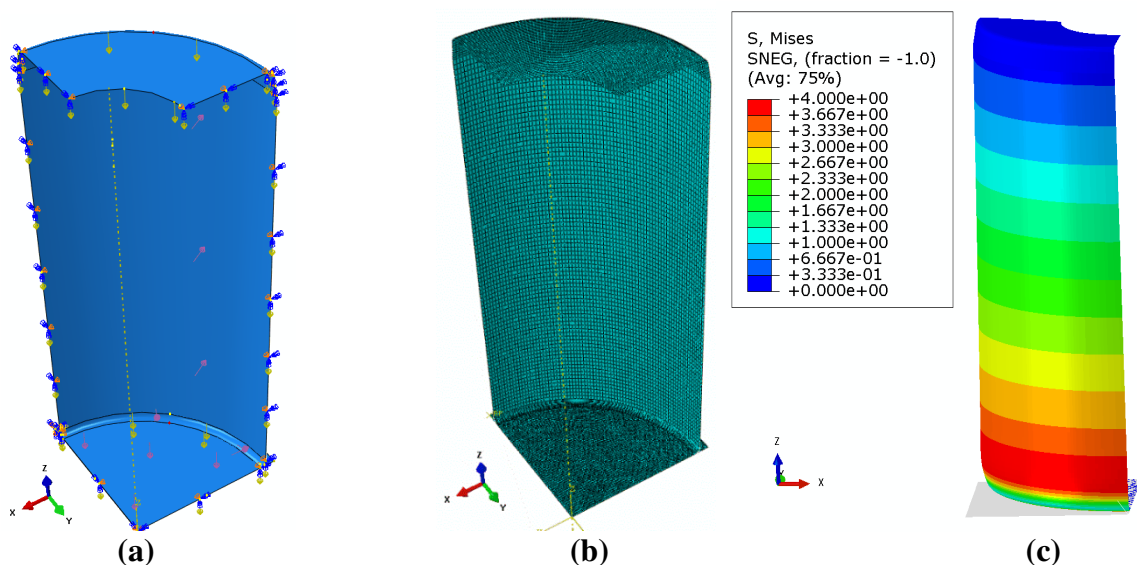
requirement has a value of 4 MPa for PE1 as a majority of thermoplastic materials used in this context. A model is meshed with 16458 nodes and 16189 elements (S4) in Abaqus (Fig. 13b). The two methodologies CM and MACcreeP are compared using the FEA ABAQUS© software [40].

For the Classical Methodology, a purely static linear calculation is carried out. The geometric nonlinearity option is activated in order to capture a possible buckling mode. If this were the case with this option, the numerical calculation would diverge. The behaviour of the PE1 material is defined by a linear elastic law; the creep modulus at 20 years ( $E_{20Y}^{CM}$ ) obtained by extrapolation (as described above) and a Poisson's ratio.

As part of the MACcreeP methodology, a calculation is performed using the VISCO procedure. The elasto-viscoplastic behavior of the PE1 material is defined by the elastic modulus ( $E_{23}^{PE1}$ ) and the identified parameters of the creep law (eq. 10) from the 2 and 5 MPa stress curves.

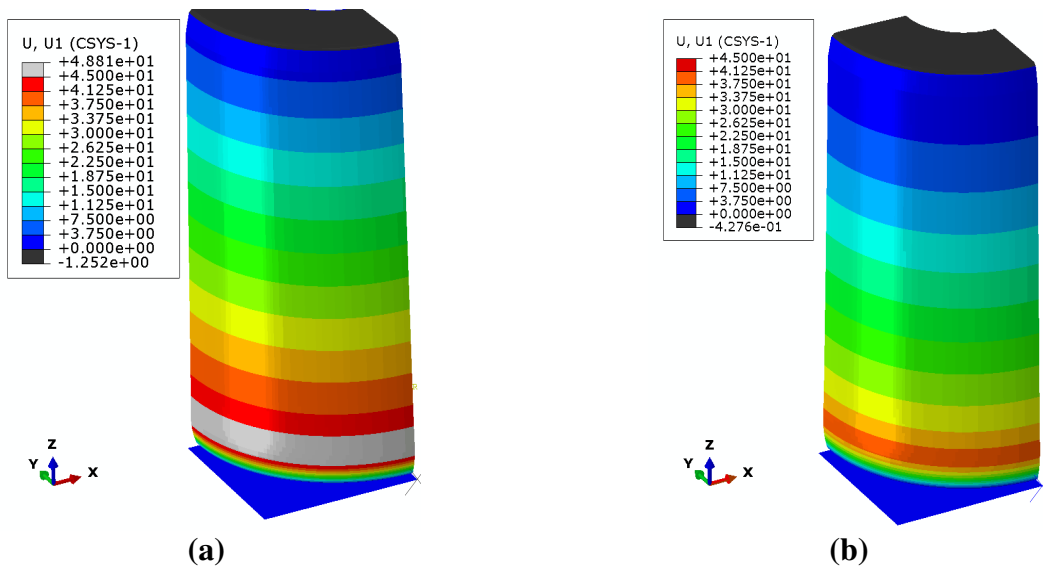
With the Classical Methodology, carrying out a linear static numerical simulation with a modulus ( $E_{20Y}^{CM}$ ), one obtains a minimum thickness of 16.1mm required to stay below 4MPa stress in the structure (Fig. 13c). The numerical response in this configuration gives a maximum radial displacement of 48.81mm in the lower zone of the tank. This causes a local deformation of 4%. By imposing the same thickness, 16.1 mm, with the MACcreeP methodology the levels of stress and displacement are lower. Thus, Fig. 13b shows that the maximum stress is slightly less than 4MPa, exactly 3.95MPa in the most stressed area. Fig. 14a and Fig. 14b show the radial displacement field of the tank subjected to internal pressure for both methods. The Classical Methodology gives the case where the tank is most radially deformed (Fig. 13a). In addition, Fig. 14b shows that the displacement field is slightly different than in the first calculation is that the maximum radial displacement is 37.07mm. By tracing the radial displacement of a vertical section of the tank (Fig. 15a), within the framework of both methodologies, it is clear that there is a relatively large deviation in response (Fig. 15b). This result is quite logical, because in the Classical Methodology the use of a static modulus (here 4MPa) leads to overestimation of deformations in the stress range between 0 and 4 MPa. While using the creep law (MACcreeP), the modulus is different depending on the constraint. Thus, by using the creep law, we obtain a better kinetics and therefore a more realistic displacement field. In this case, it is possible to further reduce the theoretical thickness. Finally, if the tank thickness is optimized and therefore has a variable thickness for example, then the gain will be even greater using the MACcreeP methodology.

On the other hand, on a classic desktop machine with two processors for our example, the STATIC (CM) and VISCO (MACcreeP) computation times are respectively 481s and 2900s, i.e. a ratio of 6. But this remains compatible for the development of polymer structures, and is very low compared to the time required to generate the creep data.

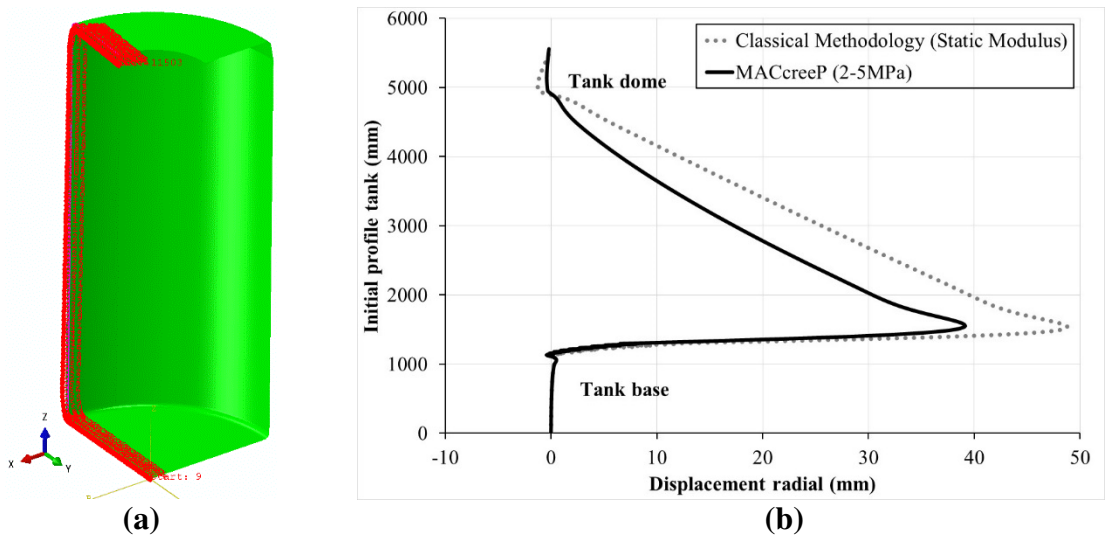




**Fig. 13. (a) Boundary conditions and loading on the rotomolded tank (b) Tank mesh – (c) Von Mises stresses field**



**Fig. 14. Radial displacement field (a) Classical Methodology (Modulus  $E_{4MPa-20Y}^{CM} = 9.6\% E_{23}^{PE1}$ ) – (b) MACcreeP (Identified with curves of 2 and 5MPa)**



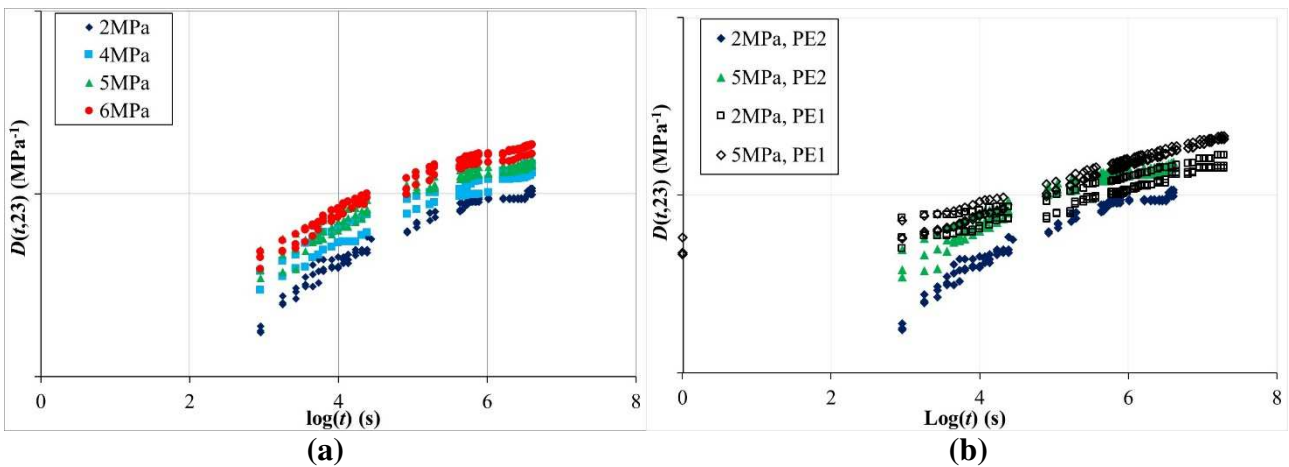
**Fig. 15. (a) Path – (b) Displacement radial**

### 3.5 Development and material selection

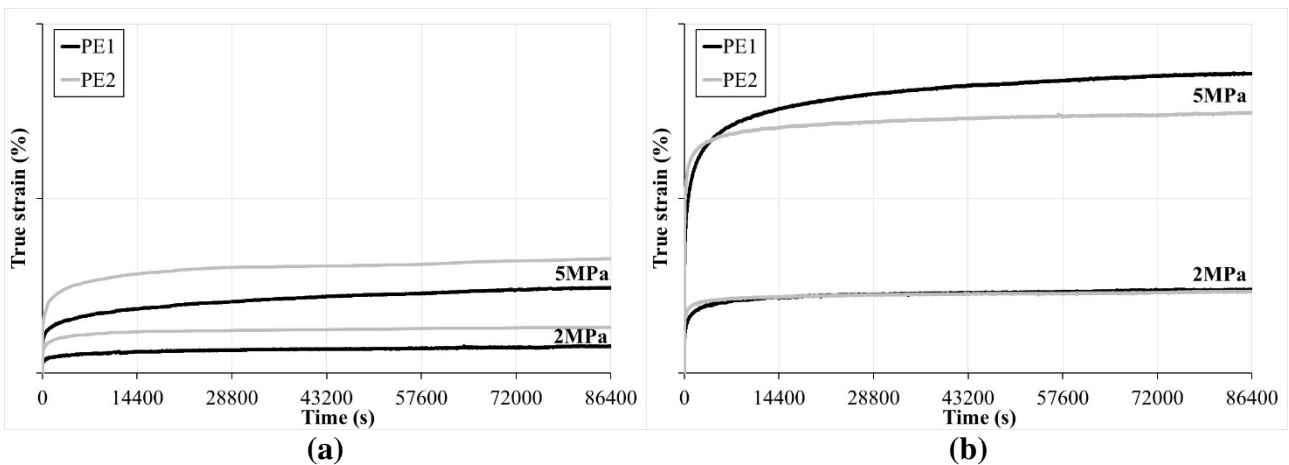
In the previous paragraph, it was shown the interest of the MACcreeP methodology to dimension a polymer structure in the long-term. Another advantage of this methodology stands also in the choice of materials and in the development of new materials.

Fig. 16a and Fig. 16b show the creep curves at different stresses obtained by the Classical Methodology for PE2 material and a comparison with PE1 for 2 and 5MPa respectively. The tests on PE2 having been carried out only over 1000 hours. By making the stress curves for PE2 and extrapolating to 10 or 20 years, the basis of fitting is different. It is therefore not reasonable to compare them.

However, by performing only 24-hour tests at the two extreme stresses (2 and 5MPa) and four temperatures, i.e. eight days of tests, it is then possible to evaluate the materials against each other. Fig. 17a and Fig. 17b show the creep curves at the two limit stresses (2 and 5MPa) for PE1 and PE2 materials at 23 and 60°C respectively. With these 8 creep tests, it is possible to build the master curves and to know the law of the shift factor for PE2, to compare them with those of PE1. However, with only the curves at 23 and 60°C one can already choose the appropriate material. Fig. 17a shows that PE2 material has a higher deformation response than PE1 at 23°C. As a reminder, the static modulus of PE1 and PE2 are 100% and 78.7% respectively. This justifies the difference in deformation on the curves. However, the creep rates are different and PE1 has a higher creep rate because the slope is more pronounced than for PE2. Fig. 17b shows that at 60°C, the curves of PE1 and PE2 are reversed, slightly to 2MPa but significantly to 5MPa. It is then easy to estimate that the PE2 material will be more appropriate for a polymer structure to be creep dimensioned at 20 years.



**Fig. 16. Creep curves under constant nominal stresses (a) PE2 Compliance creep vs log(time) – (b) Comparison of PE1 and PE2's compliant responses vs log(time).**



**Fig. 17. Comparison of the creep curves at two stresses (2 and 5MPa) over 24 hours for PE1 and PE2 (a) 23°C – (b) 60°C.**

## 4 Conclusion

It is important to underline that the objective of this work is not to compare two types of characterization tests or data extrapolation, but rather to compare methodologies ranging from characterization, through the identification of a behaviour law to numerical simulation. The Classic Methodology that has already proved its worth in design offices and a new methodology (MACcreeP)

that is proposed in this article. Indeed, it is not possible to compare the experimental measurements of the two methodologies because too many parameters are different. First of all, the samples, for the classical method, are extracted from plates made by compression injection, whereas with the proposed method, they systematically come from rotomoulded structures. Although it is not always easy to extract these samples, the important thing is to characterize the material in the final process. However, it could be required in standard tests that samples are taken from rotomoulded parts. In addition, in standard tests, nominal deformations and stresses are used, while in the second method, actual deformations and stresses are used. The measurement lengths have a factor of 10 between the two methods. Finally, the measuring means (mechanical extensometer against optical extensometer therefore without contact), loads, operators, etc. are different.

As presented, the identification of the parameters of the behavior law is also a source of deviation, which depends on the technique used, the behaviour law, the restrictions that can be imposed (choice of curves) and finally on the operator. In the Classical Methodology, the experimental data are fitted over a range between 100 and 5000h by a power law, then the modulus at 10 or 20 years are obtained via this law by extrapolation. Finally, a module with the imposed dimensioning stress defines the elastic behavior of the material. In the proposed methodology (MACcreeP), the identification of long-term creep moduli is based on the time-temperature principle that has already been tested and validated on other semi-crystalline polymers and on a standard creep behavior law in industrial FEA (finite element analysis) codes.

The interests of the proposed methodology are multiple. It allows data to be obtained more quickly: three tensile tests performed at four temperatures, i. e. 12 tests, last approximately 50 hours. For the 24-hour creep tests, at three different stresses and two four-temperature stress tests, the 576-hour test machine is used. A complete characterization of a material in less than 700 hours compared to 5000 hours in the conventional method at the same cost. In addition, this methodology provides much more material data. In addition to the modulus and Poisson's ratio, it is possible to identify all the modulus and therefore the creep behavior laws in the short, medium and long term. Only the creep parameters will be dependent on the time range taken for identification. Taking into account the evolution of stresses and specifications, it is possible with these data to determine the long-term creep moduli at 40°C, conditions that rotomoulded structures may undergo. Finally, the data generated from the behavior laws and therefore the use of the CREEP law in FEA software allows a more predictive numerical response than the use of a single elastic modulus.

Moreover, this methodology allows with very few tests (and therefore time) to compare and choose the appropriate material for the structure to be dimensioned.

In order to improve prediction at different stresses and potentially manage mechanical discharges, the creep law could be replaced by a nonlinear viscoelastic law [43] that has already been proven for high density polyethylene.

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