Experimental investigations and modeling of volume change induced by void growth in polyamide 11

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Polymers are known to be sensitive to hydrostatic pressure. The influence of stress triaxiality ratio on cavitation and damage has been highlighted in numerous studies. This paper proposes experimental investigations allowing the control of both the stress triaxiality ratio and the void distribution via microscopic observations of microtome-cut surfaces from interrupted tests. With the help of a finite element code, the Gurson–Tvergaard–Needleman model was calibrated by using these multi-scale experimental data. Then comparison between both numerical and analytical models and experimental data was performed. Bridgman formulae were reported to be valid up to the peak load. Moreover, a better understanding of the time evolution of significant parameters such as the porosity (volume change) and the stress triaxiality ratio (hydrostatic pressure), was highlighted.

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1. Introduction

Polymers are more and more used in a wide range of structural applications. Thus, it becomes fundamental to better understand and predict their mechanical response to assess their durability. Polymers exhibit a complex nonlinear behavior depending on external factors such as strain rate, temperature, hydrostatic pressure (stress triaxiality ratio), but also on morphological parameters, e.g., the molecular weight or the degree of crystallinity. This requires scientific investigations in order to develop predictive tools able to capture the mechanisms of deformation, damage and fracture.

Numerous studies have been carried out to develop constitutive equations for polymeric materials. Beyond the abovementioned external factors, modeling the mechanical response of polymers requires to account for large strain. Many papers dealing with mechanical response of polymers were reported in the literature but generally all these conditions were not totally fulfilled. Indeed, most of studies dealt with uniaxial tensile test, obviously modeled under one dimensional (1D) conditions where rheological schemes were often proposed. This first class of model was studied under small strain (up to necking) for rigid polymers. Exception can be noticed for elastomers that generally require finite strain assumption. Furthermore, visco-elastic/visco-plastic strains were investigated in such models by numerous authors. Popelar et al. (1990) and Zhang and Moore (1997) proposed viscoelastic models polyethylene. Khan and Zhang (2001) set up a viscoelastic–viscoplastic model to describe the inelastic response of polytetrafluoroethylene. Through a similar approach, Khan et al. (2006) captured the inelastic response at large strains of adiprene-L100. Other studies were devoted to the modeling of the viscoplastic behavior of polyamide 66 (Krempl et al., 1984; Krempl and Ho, 2000). To take the multiaxial stress-state under visco-elasticity and/or visco-plasticity constitutive models were recently developed by Van Domellen et al. (2003) under monotonic loading, by Drozdov (2010) and Ayoub et al. (2010, 2011) under strain reversal loading and by Ben Hadj Hamouda et al. (2007) and Reigrain et al. (2009) for creep loading. These models generally take the degree of crystallinity into account but not the volume change.

The influence of hydrostatic pressure on polymeric materials was found to be worthy to investigate in these conditions. From experimental viewpoint, tensile tests under hydrostatic pressure or compressive tests were performed to evidence this influence (see for instance, Ghorbel, 2008; Hasanpour et al., 2009; Zairi et al., 2005; Zairi et al., 2008). Whitening of polymers is related to cavitation occurring within the material by void nucleation and growth which generates volume change (Schirrer et al., 1996). For elastomers, works of Ball (1982), Dorfmann et al. (2002), Gent and Lindley (1958), Hou and Abeyaratne (1992) depicted hydrostatic pressure vs. volume change plots in spite of the assumed incompressibility of such a material.
For thermoplastics, when necking appears during the uniaxial tensile tests, multiaxial stress state takes place in the cross section, without any measurement technique to estimate this stress triaxiality. Only deformation can be measured, at least for both axial and transverse directions. This transverse deformation may lead to an indirect volume variation measure when isotropy is assumed. Concerning the triaxial stress state, Bridgman (1952) and Kachanov (1974) developed analytical and experimental analyzes on “idealized necked specimens” – that are, axi-symmetrically notched specimen. The theory, dedicated to metal, was formulated with perfect plastic material and thus ignored the volume variation. But the approach is considered to be relevant in terms of stress distribution. Combining the stress triaxiality (hydrostatic pressure) with the volume change can be handled with the help of finite element (FE) analysis. In these last two decades, constitutive models dealing with porosity evolution – considered as damage for polymers – were developed. Especially, essential works were focused on rubber toughened glassy polymers (for instance: Fond et al., 1996; Jeong and Pan, 1995; Kuroda et al., 2004; Lazzeri and Bucknall, 1995; Seelig and van der Giessen, 2002; Steenbrink and van der Giessen, 1999). In these models, void growth is due to cavitation of rubber particles and promotes a significant volume change. Calibration of the material coefficients related to the porosity is performed by using unit cells that are built to be as much representative as possible of the rubber particles distribution.

This paper is devoted to the modeling of the mechanical properties and void growth of a semi-crystalline neat PolyAmide 11 (PA11) tested at 0 °C. It follows the work of Challier et al. (2006) and Laiarinndrasana et al. (2009a) concerning experimental investigation and numerical modeling of semi-crystalline polymers. In these previous works, experimental results consisted of data obtained from tensile tests on axi-symmetrically notched specimen, with a macroscopic measure of the volume variation. The mechanics of porous media via the Gurson–Tvergaard–Needleman (GTN) model (Gurson, 1977; Tvergaard, 1982; Tvergaard and Needleman, 1984) has been set up to give an account of the elastovisco-plastic behavior and damage of the PVDF material. The volume change, measured at the macroscopic scale, was utilized to calibrate the damage parameters of this modified GTN model, implemented in a FE software. The novelty in the present paper is that a series of interrupted tests on NT specimens allowed comprehensive Scanning Electron Microscopy (SEM) observations of the distribution of porosity in the net cross section. Instead of volume change at macroscopic scale, these local data were used to calibrate damage parameters during the optimization procedure of the modified GTN model. Discussions were then based upon the comparison of simulated data with experimental ones at the macroscopic scale. A particular interest is devoted to the analysis of local parameters such as the evolution of the stress triaxiality ratio (hydrostatic pressure) and the porosity (volume change) in time and space. This also allowed a quantification of the deviation from Bridgman formulae.

2. Material and experiments

2.1. Material

Produced from a renewable source (castor oil), PolyAmide 11 (PA11 or also known as Rilsan®) is a semi-crystalline polymer used in a large number of applications thanks to its properties: excellent resistance to chemicals, ease of processing, a wide range of working temperature (−40/+130 °C), a low density, to name a few. Among the semi-crystalline polymers, PA11 stands for one of the most used. However few references exist in the literature and most of them are more focused on its chemical properties than its mechanical behavior. Thus, despite its wide presence in the industry, PA11 resistance to fracture is not well established. The grade investigated in this work is used in pipes devoted to natural gas distribution. To go further in the use of polyamide 11 such as toughened polyamide 11 for instance and thus to create a more relevant material for specific uses, it is necessary to study mechanical behavior as well as damage process of this material.

The material under study is a PA11 BESNO grade provided by ARKEMA. It was supplied as pieces of pipe, extruded without plasticizer. Specimens were extracted at mid-thickness of the pipe to minimize the effects of anisotropy. The PA11 under study is a semi-crystalline polymer for which the degree of crystallinity was estimated at 25% (Boisot, 2009). An initial amount of porosity has been evidenced and evaluated thanks to microscopic observations of samples broken in liquid nitrogen. The initial porosity, assumed to be the initial void volume fraction, was estimated at about 1%. Some key properties are summarized in the Table 1. Dynamic mechanical analysis (DMA) has been performed on uniaxial tensile (UT) specimens to confirm glass transition temperature of 50 °C obtained by differential scanning calorimetric (DSC) technique.

2.2. Mechanical tests (parameters at macroscopic scale)

Tensile tests were performed at 0 °C on two kinds of geometry: uniaxial tensile (UT) and notched tensile (NT) specimens. UT specimens were manufactured from 6 mm extruded sheets with a gauge length of 100 mm and a cross section of 10 × 4 mm². A testing machine is used to carry out the tests with four prescribed strain rates (0.001, 0.01, 0.1 and 1 s⁻¹). An extensometer with a gauge length of 25 mm is also used. Symbols in Fig. 1 show the experimental engineering stress–strain curves that clearly exhibit strain rate effects. All tests were conducted at a fixed temperature of 0 °C, a critical temperature for the use of the pipe. The humidity rate was not controlled but it was estimated at about 50% in all cases.

Regarding the NT tests, four notch radii were concerned: 4, 1.6, 1.2 and 0.8 mm (Fig. 2a). These geometries are devoted to the study of stress triaxiality ratio effects in the minimal cross section (Bridgman, 1944; 1952) as it has also been reported by Castagnet and Debureck (2007). The nomenclature used in the document for these geometries is as follows: for a given notch root radius ρ, the corresponding geometry is noted as NT₉. For instance NT₄ stands for a notch root radius equal to 4 mm. NT specimens have a length of 85 mm. For all specimens, diameters of both nominal and minimum cross section are unchanged and equal to 4 and 7.2 mm, respectively. A strain gage is attached to the notch root in order to record the reduction in diameter of the minimal cross section. The nominal diametrical strain is thus defined as Δφ/φ₀ with φ₀ = 4 mm the initial diameter (φ₀ = 2b). Additionally, the axial strain Δh/h₀ is defined as the cross head displacement divided by the initial notch height (see Fig. 2a) h₀ equal to 1.6, 2.4, 3.2 and 6.4 mm for NT0.8, NT1.2, NT1.6 and NT4, respectively. The net stress F/S₀ is defined as the load F divided by S₀ that is the initial minimal section (S₀ = πφ₀²/4). NT tests were carried out at 0 °C, by controlling the crosshead speed at 0.05 mm s⁻¹. Each loading condition was repeated at least twice in order to

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Melting point</td>
<td>184−188 °C</td>
</tr>
<tr>
<td>Index of crystallinity</td>
<td>20–25%</td>
</tr>
<tr>
<td>Glass transition</td>
<td>50 °C (measured by DSC)</td>
</tr>
<tr>
<td>Young modulus (at room temperature)</td>
<td>1500 MPa</td>
</tr>
<tr>
<td>Poisson ratio</td>
<td>0.42</td>
</tr>
</tbody>
</table>
check the reproducibility of the experimental results. Fig. 2(b) displays the net stress versus diametrical strain average curves under these conditions. Only symbols in Fig. 2(b) are discussed in this section. It can be observed that by decreasing the notch root radius \( r \), the peak stress increases, that is a classical result related to the stress triaxiality ratio increase inversely to the notch radius of NT specimens. It seems that the peak stress appears around a diametrical strain of \( \Delta \Phi/\Phi_0 = 0.1 \) for all geometries, whereas the stress softening observed after the peak stress stops at about \( \Delta \Phi/\Phi_0 = 0.3 \).

The maximum net stress normalized by the equivalent stress \( \sigma_{eq} \) can be plotted with respect to a normalized geometrical parameter \( b/\rho \) in Fig. 3. UT results, corresponding to \( b/\rho = 0 \), were superimposed in this Fig. 3 as open squares symbols to define \( \sigma_{eq} \). Indeed, the normalized peak stress level depends on the choice of \( \sigma_{eq} \).

Here, \( \sigma_{eq} \) is defined as the mean value of peak stress for UT tests at various strain rates, that is \( \sigma_{eq} = 57 \) MPa. When \( b/\rho \) decreases, increase in the normalized peak stress is clearly shown in Fig. 3. It can be observed that there is an abnormal large scatter for NT4 tests (full squares). In particular, NT4 interrupted tests that will be discussed later, gave the two lowest peak stress values in Fig. 3.

The aim being to characterize voids distribution and evolution, an innovative experimental method was set up to assess voids within the samples. To this end, some of the tensile tests performed on NT specimens were interrupted well after the peak stress. Fig. 4 shows experimental data produced for typical interrupted tests on NT4, NT1.6 and NT0.8. Thanks to strain gage positioned at the notch root, the evolution of both \( \Delta \Phi/\Phi_0 \) and \( F/S_0 \) (second Y-axis) with respect to the axial strain \( \Delta h/h_0 \) could be plotted. The same scale was set to all graphs for comparison purposes. Although a constant crosshead speed was applied, the diametrical strain exhibited non linearity. In particular, a fast increase of \( \Delta \Phi/\Phi_0 \) is observed when the net stress gradually drops down after the peak stress. Following Challier et al. (2006) and Laiarinandra-sana et al. (2009a), \( \Delta \Phi/\Phi_0 \) can be used to approximately estimate the volume change. However, it can be also connected to the amount of voids in the vicinity of the minimum cross section. It seems that the larger the notch radius, the lower the value of the diametrical strain at stop, but this is only an effect of neck height \( h_0 \). From Fig. 4, it can be confirmed that the diametrical strain at peak stress is about 0.1. Furthermore, tests were interrupted when the diametrical reduction approximately reached \( \Delta \Phi/\Phi_0 = 0.3 \). As shown in Fig. 4, it was obtained only for NT0.8 whereas for both NT4 and NT1.6 the diametrical strain at stop was about 0.4. It can also be noted that the fast increase of volume change coincides with the stress softening stage.

### 2.3. Microscopic examinations: voids distribution

Deformed specimens obtained from interrupted tests were first cut longitudinally, in order to only focus on the whitened notched part where the damage by void growth was expected to be the largest. Then, the sample was plunged into liquid nitrogen before a second cut to minimize the use of the microtome. The sample is then micromotomed along the tensile direction from the surface to the core of the specimen, layer by layer, with a glass knife. Then scanning electron microscopy (SEM) observations of the longitudinal cross sections are performed on Pd–Au coated surfaces. The amount of voids, defined as the ratio of cavities area over the observed surface is determined by digital image processing (ImageJ software). These values were assumed to be the void volume fraction, or porosity, representative of the damage parameter. Voids...
maximum void volume fraction is located in the minimal cross section mainly in the core of the specimen. Additionally, the smaller the notch radius, the higher the amount of voids. Fig. 6 clearly emphasizes the influence of the notch root radius on the damage distribution, as well as the void volume fraction gradient in the net section.

It is worth noting that the abovementioned procedure probably underestimates the damage values. Indeed, the unloading step at the end of the interrupted tests and the microtome cut operations may close voids. Conversely, what is observed can be considered as a permanent set in the sense that “reversible void expansion” was removed during unloading. However, Fig. 6 possesses significant information allowing damage coefficients calibration, in constitutive models. It should also be mentioned, at this stage, that although elongation of voids was clearly observed in Fig. 5 (NT0.8), this will not be discussed in this work.

3. Modeling

3.1. Bridgman formulae

According to Bridgman (1952) and Kachanov (1974) (see Appendix A), the axial stress $\sigma_{zz}$ and the stress triaxiality ratio $\tau_\sigma$ distributions consist of inverted parabolas. $\sigma_{zz}$ is composed of a structural term (function of $b(\rho)$) and a constitutive term (the equivalent stress $\sigma_{eq}$). Strains are assumed to be homogeneous within the cross section. Furthermore, Bridgman theory assumes isochoric deformation which can be questionable for polymeric materials.

In terms of global parameter, Bridgman stress distribution enables to estimate the net stress $F/S_0$ (Eq. (A2) in Appendix A). In Fig. 7, solid line corresponds to the geometrical term (function of $b(\rho)$ of Eq. (A2) second part, that is the ratio between $(F/S_0)_{max}$ and $\sigma_{eq}$ Although the trend of experimental normalized peak stresses is reproduced, Bridgman formula overestimates both stress level and slope of the curve. The dependency of the normalized peak stress on $\sigma_{eq}$ was studied. It was shown that Bridgman simulations always predicted steeper slope of the Fig. 7 plot. The discrepancy is presumably due to both viscous strain and volume change effects since they are not accounted for by Bridgman formulae. However, one can assume that in first approximation, Bridgman formula can be used up to the peak stress.

Attention was now paid to the distribution that follows the trend of porosity in Fig. 6. As mentioned previously the strains were assumed to be homogeneous in the minimal cross section. The porosity distribution in Fig. 6 rather fits with that of the stress or the stress triaxiality ratio. Indeed, the maximum values of $\sigma_{zz}$ (Eq. (A1)) and $\tau_\sigma$ (Eq. (A3)) are located in the center of the specimen. Moreover, the higher value the notch radius, the lower the axial stress and the triaxiality, respectively. One can then deduce that the stress or $\tau_\sigma$ are the leading parameters to damage evolution.

Once again, Bridgman formulae being only valid for small strain and incompressible material, they are questionable especially for polymers that exhibit finite strain, strain rate effects and volume change. The aim here below is to check at what extent these formulae remain valid, with the help of finite element (FE) modeling where the constitutive relationships were calibrated in such a way that all these previous effects were accounted for.

3.2. FEA with modified GTN model

3.2.1. Model description and optimization strategy

To go further in modeling the mechanical response including damage evolution of the PA11, the modified Gurson–Tvergaard–
Needleman (GTN) model, detailed in Appendix B, was used here. This model was already developed in Challier et al. (2006) and Laiarinandrasana et al. (2009a). The only difference from the present model concerns the expression of \( q_2 \) parameter recalled in Eq. (B7) (Appendix B).

The abovementioned modified GTN model was implemented in an in-house FE code, Zset (Besson and Foerch, 1997). Integration of the constitutive model is achieved with an implicit scheme and the consistent tangent matrix is got by using the description of Simo and Taylor (1985). An updated Lagrangian finite strain formulation was used associated with Jaumann stress rate (Sidoroff and Dogui, 2001). NT specimens were meshed by using axi-symmetrical quadratic elements with reduced integration elements. Deformed meshes are illustrated in Fig. 8. Furthermore, the analysis of the mesh size effects showed no significant influence in the results here below.

Notch was paid on the determination of material’s parameters. The Young’s modulus \( E \) and the Poisson ratio \( \nu \) are assumed to be equal to 1500 MPa and 0.42, respectively. To take the visco-plasticity of the material into account a Norton power law function is used, with \( K \) and \( n \) as material coefficients.

The plastic hardening is described by \( Q, b, A, B \) parameters that account for initial hardening stage assumed to be isotropic and the
rheo-hardening due to the large stretching of the fibrils (Challier et al., 2006; Laiarinandrasana et al., 2009a). Unlike hyperelastic based constitutive equation, $A$ and $B$ material parameters were added to take the rheo-hardening into account.

Material parameters were identified by using an optimizer routine of Zset code (Besson et al., 1998) based on least square function minimization. First, only the UT tests up to the peak stress were considered. This enables to get a first approximation of $K$, $n$, $Q$ and $b$. The second step accounted for the notched specimens allowing further tuning of previous values of the coefficients, but rheo-hardening parameters $A$ and $B$ were also adjusted.

Unlike Challier et al. (2006), where “macroscopic” volume change based on diametrical strain was used to fit damage parameters, the present identification utilizes the comprehensive observations of microtome-cut surfaces from interrupted tests, especially on Fig. 6. $q_1$ parameter was set to a rather high value to prevent the numerical failure of the specimen, which is out of the scope of this paper. Furthermore $q_2$ parameter (Eq. (B7)), function of the maximum principal plastic strain $p_1$, was characterized by $M$, $m$, $v$, $p_r$ values corresponding to the distributions of void volume fraction given in Fig. 6. As detailed in Appendix B, the values of these parameters make that $q_2$ continuously decreases from a maximum value of 0.62 to a minimum value of 0.075, when $p_1$ increases. To the authors’ knowledge, it is the first time that this kind of calibration is done, at least for polymeric materials. The optimized set of material parameters is summarized in Table 2.

It should be mentioned that more sophisticated models, explicitly accounting for anisotropy or void shape change (Danas and Ponte Castañeda, 2009a, 2009b; Pardoen and Hutchinson, 2000; Zaïri et al., 2011) could possibly be used at the cost of an increasing complexity.

3.2.2. Comparison between experiments and models with optimized set of material coefficients

Simulations of all interrupted tests were performed, allowing comparison between experimental and simulated data.

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Fig. 8. (a) Porosity contour maps for NT4, NT1.6 an NT0.8 after the stress softening; (b) comparison between measured (symbols) and simulated (solid lines) porosity (%) for the three involved NT specimens interrupted at 40% for both NT4 and NT1.6 and at 30% for NT0.8.
Results obtained by using fitted material coefficients (Table 2) are presented as solid lines in Figs. 1 and 4 in terms of the nominal and net stresses $F_s/n$ against $\Delta l/l_0$ and $\Delta h/h_0$, respectively for UT and NT specimens. Additionally, the reduction in diameter $\Delta D/D_0$ for NT specimens was superimposed in Fig. 4. Good agreement is observed between experimental and simulated curves for both stress and strain. Note that while damage parameters were calibrated with void volume fraction data (Fig. 6), the volume change between experimental and simulated curves for both stress and strain. Indeed, the present modified GTN model, the relationship between the porosity $f$ and the volume change is given in Eq. (B6) (Appendix B). Indeed, $f$ distribution is in Fig. 6 whereas $tr(\varepsilon_p)$ term is linked to the volume change, thus to $\Delta \Phi/\Phi_0$ variable.

In terms of local parameters, Fig. 6 was utilized to adjust the parameters of $q_2$ (Eq. (1)). For the optimized set of material coefficients, contour maps of porosity were displayed in Fig. 8(a) on the deformed meshes at time corresponding to the test stops for all NT specimens. It can be observed that both maximal values at the center of the specimen and evolution of the void fraction along the section are well described. Additionally, a good agreement is obtained between experimental and numerical values of porosity as depicted in Fig. 8(b), for involved NT geometries. These results ensure that the fitted material coefficients are consistent with the particular response of PA11 polymeric material at 0 °C. The constitutive relationships are able to capture viscous deformation and volume variation under finite transformation conditions.

4. Main results

Taking benefits of the aforementioned FE model with the fitted material coefficients in Table 2, full simulations of all tests were performed. This consisted of the stress/strain curves up to rheo-hardening of the tested specimens including NT1.2 specimens for which data were not used in the optimization procedure. The aim of this section is first to compare Bridgman theory with FE simulations at the peak stress. Then, FE results were extrapolated in order to explore the trend up to the rheo-hardening, prior to failure.

Fig. 2(b) summarizes the general trend of net stress versus diametrical strain curves for all tested NT specimens. It is observed that experimental and numerically simulated curves are in good agreement on all NT specimens. Indeed, numerical peak stress, stress softening and stress plateau are in good accordance with the experimental data. Fig. 2(b) was split to plot individual curve for each NT geometry to give details about some characteristic events. Fig. 10 displays experimental and simulated curves plotting the net stress against the diametrical strain. In Table 3, some specific values of $\Delta \Phi/\Phi_0$ were summarized, corresponding to the aforementioned characteristic events during the deformation (noted in Fig. 9):

- $\Delta \Phi/\Phi_0$ around 0.1, corresponding to the peak stress, assumed to be the start of the re-necking phenomenon (Challier et al., 2006; Laiarinandrasana et al., 2009a).

- $\Delta \Phi/\Phi_0$ around 0.3, where the stress softening ends and the rheo-hardening starts. It will be noted as “min stress”; the maximum volume change is supposed to occur during this step.

- $\Delta \Phi/\Phi_0$ corresponding to the end of the considered computation. This was chosen arbitrarily although a little rheo-hardening was supposed to be taken into account. This specific point will be noted as “end” in the following.

4.1. At macroscopic scale

Simulated peak stress were collected and superimposed in Fig. 3 as dashed line. As mentioned in the previous section, experimental and FE simulations are in good agreement. Therefore, the same deviation from Bridgman formula is observed between experimental data and simulated ones. Next sub-sections attempts to better understand the origins of this deviation.

4.2. Microscopic scale: local parameters

In this subsection, all graphs were gathered in Fig. 10 where characteristic parameters were plotted according to the above-mentioned specific events. For each mechanical local parameter with respect to normalized radius absissa, three figures are displayed corresponding respectively to peak stress (first column), min. stress (second column) and end of the simulation (third column). These three figures were obtained with the deformed configuration. They are displayed with the same scale for comparison purposes. Thin vertical arrows indicate the order in which NT geometries are encountered. Figures corresponding to the peak stress (column 1) generally contain dashed lines for Bridgman formula according to equations in Appendix A. The figures of last column can present discrepancies due to the arbitrary choice of the end of the calculation.

4.2.1. Stress triaxiality ratio $\tau_\sigma$

Fig. 10(a) shows the distribution of stress triaxiality ratio $\tau_\sigma$ in the net section. This parameter is considered as the leading one for void expansion. The inverted parabolic trend is observed in Fig. 10(a) (columns 1–2), relevant to the porosity distribution in Fig. 6. For each NT geometry, $\tau_\sigma$ decreases when $\Delta \Phi/\Phi_0$ increases. Volume change during the stress softening stage alleviates the stress triaxiality ratio value. In Fig. 10(a) (column 3), $\tau_\sigma$ is rather flat, with a minimum value close to 0.33 corresponding to uniaxial stress state. The maximum value moved from the center of the net section to the notch root. In all plots (Fig. 10(a)), it is shown that the lower the initial notch root radius, the higher $\tau_\sigma$. Although the general trend is followed by Bridgman formula (dashed lines in Fig. 6) – column 1), it can be noticed that numerical result for NT0.8 is rather flat at the top of the parabola. Bridgman formula overestimates $\tau_\sigma$ for this NT0.8 geometry in the center of the net section. For NT4, NT1.6 and NT1.2, Bridgman formula underestimates $\tau_\sigma$ values near the center of the section. In addition, it assumes that close to the notch root radius ($r/b = 1$), the stress state is uniaxial ($\tau_\sigma = 0.33$). Except for NT4 specimen, numerical simulations predict higher values of $\tau_\sigma$ at this location.

4.2.2. Normalized maximum principal stress $\sigma_{zz}/\sigma_{eq}$

Fig. 10(b) displays the largest principal stress normalized by von Mises equivalent stress $\sigma_{eq}$. Note that numerical $\sigma_{eq}$ was not homogeneous within the net section. Fig. 10(b) – column 1 shows

| Table 2 |
|-----------------------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|
| R0            | K                  | n               | Q               | b               | A               | B               |
| MPa          | MPa s$^{-1/n}$     | –               | MPa             | –               | MPa             | –               |
| 10            | 38.5               | 10              | 20              | 30              | 7.9             | 3               |
| q1           | q2                  | $\nu$           | m               | $\nu$           | m               | $\nu$           | m               | $\nu$           |
| 2             | 0.9                | 0.35            | 0.6             | 6               | –               | –               | –               | –               |

| Table 3 |
|-----------------------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|
| $\Delta \Phi/\Phi_0$ values at characteristic times. |
| NT4           | NT1.6            | NT1.2           | NT0.8           |
| Peak stress (peak)       | 0.07             | 0.07            | 0.09            | 0.08            |
| End of stress softening (min) | 0.31           | 0.34            | 0.28            | 0.28            |
| End of the simulation (end) | 0.48          | 0.40            | 0.39            | 0.37            |
| Minimum local $\tau_\sigma$ | 0.007          | 0.015           | 0.024           | 0.023           |
| Maximum local $\tau_\sigma$ | 0.13           | 0.15            | 0.19            | 0.20            |
that Bridgman formula gives results which slightly differ from the numerical results obtained by the finite element method. Similarly to $\sigma_{\tau}$, the maximum stress moves from the center to the surface of the specimen for increasing $\Delta \Phi/\Phi_0$. Indeed, $\sigma_{zz}/\sigma_{eq}$ is firstly maximal at the center until the peak stress. At this time, cavitation is maximal in the center of the specimen. During the stress softening phase, $\sigma_{zz}/\sigma_{eq}$ decreases. In Fig. 10(b) – column 3, the $\sigma_{zz}/\sigma_{eq}$ is rather flat with a maximum value near to the notch root. Indeed, the stress triaxiality ratio in the center of the specimen drops down and uniaxial stress state is applied to a voided material. Therefore, the material near to the surface is submitted only to the rheo-hardening, and not to the stress softening due to damage (cavitation). During this last stage, $\sigma_{zz}/\sigma_{eq}$ increases more at the surface of the specimen, generating the shift of its maximum value from the center to the surface.

The same discussion as for $\tau_{\sigma}$ can be repeated here for the largest principal stress. In particular, Fig. 10(b) column 1 compares Bridgman formula (dashed lines) with FE results. It clearly shows that the range of maximum stress is larger for Bridgman than that of simulation results. In fact, Bridgman formula underestimates $\sigma_{zz}/\sigma_{eq}$ at low stress triaxiality ratio (NT4) and underestimates it for NT0.8. These differences in terms of stress appear whereas Bridgman formula is likely to be more relevant at peak stress. In particular, Fig. 10(b) column 1 compares the Bridgman formula with FE results (Fig. 10(e)). At peak stress, the maximum mean stress is located in the center of the specimen whatever the notch root radius – the hydrostatic pressure- is to be investigated thanks to FE results (Fig. 10(e)). At peak stress, the maximum mean stress is located in the center of the specimen.

4.2.3. Normalized maximum principal strain $\varepsilon_{zz}/\varepsilon_0$

Normalized strain $\varepsilon_{zz}/\varepsilon_0$ was also investigated to assess its homogeneity within the net section as suggested by Bridgman theory. In Fig. 10(c), $\varepsilon_0$ is considered here as the mean value of the total strain in this net section. According to Bridgman theory (Appendix A, Eq. (A6)), the normalized strain should be homogeneously equal to unity in the whole net section. Fig. 10(c) – column 1 corresponding to the normalized strain at peak stress shows strain gradients. The lower the notch root radius (higher $\varepsilon_0$), the more heterogeneous the normalized strain field. Indeed, in Fig. 10(c) – column 1, NT4 curve is the closest to the Bridgman formula (dashed line with $\sigma_{zz}/\sigma_0 = 1$). At the end of stress softening (minimum stress), Fig. 10(c) – column 2 shows that the normalized strain distribution gets closer to the unity. This is presumably due to stress/strain redistribution after the volume change increase during the stress softening. In terms of strain, it seems that even at peak stress Bridgman formula fails to estimate the strain.

4.2.4. Volume change $\Delta \Phi/\Phi_0$

Fig. 10d investigates the volume change that is supposed to be null by Bridgman formula (Eq. (A6)). Fig. 10(d) – column 1 shows that at peak stress the volume change ranges from 2% to 3%, respectively from NT4 to NT0.8. This clearly indicates the measure of approximation assumed when using Bridgman theory. At minimum stress, Fig. 10(d) – column 2 shows that a great increase in volume change appears. The maximum volume change occurs in the center of NT0.8 specimen and its value may reach 16%. Basically, this figure demonstrates that assuming Bridgman theory at the peak stress implies that 3% volume change is considered as acceptable. Beyond the peak stress, as soon as stress softening takes place, Bridgman formula is no longer valid. Only, FE provided with a constitutive modeling that takes volume change versus hydrostatic pressure into account can correctly handle the deformation and damage mechanisms.

4.2.5. Mean stress (hydrostatic pressure) $\Delta \Phi/\Phi_0$

Once the volume change is demonstrated to be present, the dual parameter – the hydrostatic pressure- is to be investigated thanks to FE results (Fig. 10(e)). At peak stress, the maximum mean stress is located in the center of the specimen whatever the notch root radius – the hydrostatic pressure- is to be investigated.
At minimum stress, the hydrostatic pressure seems to be homogenized within the net section, as depicted in Fig. 10(e) – column 2. At the end of the simulation, that is also during the rheohardening stage, the maximum hydrostatic pressure moves towards the surface (notch root). Additionally, the mean stress may reach values as high as 100 MPa.

### 4.2.6. Porosity versus triaxiality relationship

In Fig. 11, it is proposed to follow the evolution of both maximum stress triaxiality ratio and porosity (open square) at the center of the minimal cross section during the test – that is for increasing diametrical strain. For the sake of clarity, each NT geometry has its own plot and the same scale was set.
4.2.7. Stage I

It can be observed that $\tau_\sigma$ decreases at the early stage of the deformation. This is presumably due to the increase of the deformed notch radius. Note that the lower the initial notch radius, the higher the amount of this decrease. During this early stage of deformation, the porosity, initially equal to 1%, remains stationary. An attempt was made to estimate via Bridgman formula (Eq. (A5)) – which is valid at this stage of deformation – the increase of the notch root radius corresponding to the decrease of $\tau_\sigma$. For NT4, NT1.6, NT1.2 and NT0.8 specimens, the obtained $\Delta \rho$ values were 190, 230, 270 and 290 $\mu$m, respectively. Measuring such a change in notch deformation requires more precise technique. At macroscopic scale, it was evidenced experimentally that for NT4 specimens, immediate re-notching occurs during the first applied deformation.

4.2.8. Stage II

Stage I is then followed by a significant increase of $\tau_\sigma$ up to its maximum value. This latter is all the more so high and delayed as the initial stress triaxiality ratio related to the initial notch radius is significant. This step corresponds to the re-notching phenomenon as already mentioned by Boisot (2009), Boisot et al. (2008), Challier et al. (2006), Laiarinandrasana et al. (2009a,b). Indeed, a new neck appears in the minimal cross section, with a smaller notch root radius. Re-necking is complete when $\tau_\sigma$ reaches its maximum value. The porosity quickly increases to stabilize at its final value. For each NT geometry, the porosity starts increasing during the re-notch process. As expected, maximum void growth rate is obtained when $\tau_\sigma$ is at its maximum value. Furthermore, $\tau_\sigma$ maximum values are attained later than the peak stresses. Indeed, according to first and last lines of Table 3, there is a factor 2 between the two diametrical strains. Once again, by using the Bridgman formula (Eq. (A5)), an estimates of the stabilized notch root radius at the end of the re-necking was attempted. It turned out that it depends on the initial notch root radius: $\rho = 1.93, 0.93, 0.72, 0.49$ mm for respectively NT4, NT1.6, NT1.2 and NT0.8. It can be concluded that around the peak stress, efficient Bridgman calculation needs updating of $b$ and $\rho$.

4.2.9. Stage III

After its maximum value, the stress triaxiality ratio drops down below its initial value and tends to final value $\tau_\sigma \approx 0.44$. The decreasing rate depends on the initial triaxiality. In the beginning, porosity still increases, but at lower void growth rate due to $\tau_\sigma$ decrease. Afterwards, it seems that void fraction stabilizes to its final value when the triaxiality drops below its minimum value reported in subsection “stage I”. Voids are still expected to be spherical at this stage. Next decrease in the stress triaxiality ratio induces low void growth rate. Nevertheless, it is expected that quasi-uniaxial stress rather generates a modification of the shape of the voids consisting of stretching (elongation) in the axial direction. At the macroscopic scale, the necking zone stretches along the direction of the loading, leading to a small quasi-uniaxial specimen constituted of highly voided material (porosity at maximum).

To summarize, Fig. 11 allowed a multi-scale analysis because simultaneous comparison was made between local (stress triaxiality ratio, void volume fraction at the center of the specimens) and global (net stress, diametrical strain) parameters.

4.2.10. Volume change versus hydrostatic relationship

This paper was devoted to better understand the damage mechanisms under multiaxial stress states. FE modeling was proposed to account for these mechanisms. The damage is represented by void volume fraction. Its evolution induces volume change and the stress triaxiality is mainly due to the hydrostatic part of the stress tensor. For the integration point located in the center of the net sections, the local mean stress (hydrostatic pressure $= tr(\sigma)/3$) versus volume variation $\tau_\sigma$ relationships were plotted (Fig. 12). A unique initial compressibility modulus seems to appear, whose value is about 2000 MPa. For high values of the volume change, non linearities appear, for which characteristics strongly depend on the geometry. It can be concluded that modeling the

![Fig. 11. Stress triaxiality ratio and porosity at the central integration point versus diametrical strain curves of NT specimens.](image-url)
mechanical response of the PA11 requires an account for volume change versus hydrostatic relationship.

5. Discussion and further works

The experimental approach developed in this paper has been the opportunity to highlight firstly, the damage by void growth of the investigated material the PA11 and secondly, the influence of the stress triaxiality ratio on the damage evolution. The concluding remarks regarding the second point seems to match the ones described by Challier et al. (2006) in the case of the PVDF but also, generally speaking, with the ones stood up by Castagnet et al. (2000) for semi-crystalline polymers. As the voids found in polymers generally reach the micrometer scale, it is easily possible to follow their development (nucleation, growth and coalescence) as well as their size and shape evolution. Thus, it could be relevant to run interrupted tests at some pre-scribed deformation stages to investigate the latter phenomena mentioned previously. Recent work of Laiarirandrasana et al. (2010) on PolyAmide 6 is worth being mentioned. Indeed, by using X-ray tomography such data in 3D could be collected. Through images analysis technique, it can determined if whether or not nucleation occurs by counting the voids and observe the evolution of the size and shape of voids regarding their location within the specimen (center of the minimal cross section, proximity from the notch root...). These data are crucial to further use the finite element method and to better calibrate constitutive models coefficients devoted to study the evolution of damage within polymers.

From material science viewpoint, the paper developed methodology based on observations at macroscopic scale, that is during the test. The peak stress is the first event that can be noticed. It was demonstrated that this event takes place in the damage growth stage. It does not exactly correspond to the void nucleation which is only observable at microscopic, even nanoscopic scale. Such a study on void nucleation requires a definition of a characteristic size consistent with the observation techniques. At macroscopic scale, it was demonstrated that the neck root radius (occurring at the peak stress) is not an intrinsic parameter of the material. The neck curvature for UT specimens might be regarded like this because it corresponds to NT∞. But, it is well known that necking depends on the defects within the material sample. Therefore, continuum mechanics, even with sophisticated constitutive equations seems to be unable to predict this parameter.

The stage of void elongation and coalescence was not discussed in this paper. In fact, void elongation parameter (shape factor) as well as failure modes by voids coalescence can be determined from microscopic observations. However, only in situ tests are able to capture the real time of coalescence. It is worth noting that according to Challier et al. (2006), two modes of failure were observed for PVDF material: by critical porosity (void coalescence) and by critical plastic strain.

From a numerical point of view, the use of the modified Gurson–Tvergaard–Needleman model accurately depicts the evolution of damage of PA11 material through the q1 and q2 parameters. Indeed, the experimental quantifications of the void volume fraction during deformation in the case of several stress triaxiality ratios corroborate the numerical predictions. From then on, it confirms that GTN model, initially developed for the metallic materials, can be also used in the case of polymeric material and thus for semi-crystalline materials. As mentioned previously, the limitations of the present GTN model deal with of void nucleation, elongation and coalescence. All of these parts (Tvergaard, 1981; Zhang and Hauge, 1999) are already implemented in the present GTN model. Attention should be paid also on the recent works of Danas and Ponte Castañeda (2009a,b) in terms of void elongation modeling. All of these models require to be based on experimental investigations to better calibrate material coefficients.

6. Conclusion

The mechanical properties and void growth of the polyamide 11 have been investigated at 0 °C. Experimental investigations were carried out on both uniaxial and notched specimens to highlight the strain rate and the triaxiality effects. These latter were observed on global variables such as the net stresses, the crosshead displacement and the diametrical strain that reflected the volume variation. SEM observations on specimens subjected to interrupted tests enabled to quantify the amount as well as the distribution of porosity in the net cross section. These local data were used to calibrate damage parameters incorporated in a modified GTN model, implemented in a FE code. The material parameters determination was carried out with the help of an inverse method optimization procedure. Good agreement was obtained between experimental values and corresponding FE simulations by using the optimized set of material parameters.

Comparison of numerical simulations with analytical model proposed by Bridgman was performed. The peak stresses depend on the stress triaxiality ratio via the normalized geometrical parameter b/ρ (b = radius of the net section, ρ = the notch root radius). It was shown that, although the effects of triaxiality were overestimated by Bridgman formula, the peak stress values calculated by both analytical and numerical methods were rather similar. Conversely, the volume variation via the diametrical strain was not compared because Bridgman theory assumed isochoric deformation.

Local variables, within the net section, were also compared at peak stress, at the end of the stress softening and at the end of the computation. At the peak stress, the distribution of the largest principal stress as well as the stress triaxiality ratio in the net section was consistent with that of the porosity measured experimentally. It was concluded that the “driving force” leading to void growth was related to the stress triaxiality ratio that is linked to the hydrostatic pressure. Bridgman formulae were found to overestimate the range of local largest principal stress and stress triaxiality ratio, obtained for the prescribed NT geometries. Moreover, both parameters gradually decreased after the peak stress: at the end of the stress softening and at the end of the computation.

As expected, the main discrepancy between Bridgman formulae and the numerical solution was found by comparing the largest principal strain and the local volume change tr(δ). Indeed, conversely to Bridgman assumption, strains are neither homogeneous.
nor isochoric in the net section. At peak stress, volume changes were numerically estimated at 3%. Applying Bridgman formula at this time reduces to neglect this volume change.

The history of the porosity and the stress triaxiality ratio in the center of the net section was discussed. Triaxiality decreases in the early stage of the deformation. This was attributed to the first deformation of the notch radius that was not detected experimentally. The porosity is unchanged during this first stage. Then, triaxiality increases until the peak stress where it reaches its maximum rate. The porosity follows the same trend. The stress softening changes the distribution of the triaxiality in the net section. It results in a decrease of the triaxiality in the center and decreases the void growth rate. The rheo-hardening stage is done with a quasi-uniaxial stress but with maximum porosity material.

Finally, the local mean stress vs. volume change relationship was analyzed. In the beginning (initial porosity value) it seems that a compressibility modulus of about 2000 MPa is obtained. As soon as porosity differs depending on triaxiality value, the pressure versus volume change relationship is different.

Appendix A. Bridgman formulae

By assuming perfectly plastic material, deforming with no volume change (i.e., isochoric) and with a homogeneous axial strain in the minimal cross section, Bridgman (1952) demonstrated that both radial and axial stresses can be expressed as follows:

\[
\begin{align*}
\sigma_{rr} &= \sigma_{\infty} \ln \left( 1 + \frac{r^2 - r_0^2}{2 \rho} \right), \\
\sigma_{zz} &= \sigma_{eq} \ln \left( 1 + \frac{r^2 - r_0^2}{2 \rho} \right),
\end{align*}
\]

(A1)

where, \( \ln \) is the naepERICAN logarithm, \( r \) is the current radial abscissa in the cross section: \( 0 < r < b, r = 0 \) corresponding to the center of the cross section.

It has to be noticed that Eq. (A1) is composed of a structural term (function of \( b/\rho \)) and a constitutive term (the equivalent stress).

The stress distribution in Eq. (A1) enables to estimate the net stress \( F/S_0 \) as defined previously:

\[
\frac{F}{S_0} = \frac{\sigma_{eq}}{\sigma_0} \left( 1 + \frac{2 \rho}{b} \right) \ln \left( 1 + \frac{b}{2 \rho} \right).
\]

(A2)

The triaxiality of the stress state in the minimal cross section is measured by the stress triaxiality ratio \( (\tau_\sigma) \), defined as the mean stress divided by the equivalent von Mises stress:

\[
\tau_\sigma = \frac{\sigma_m}{\sigma_{eq}} = \frac{1}{3} \ln \left( 1 + \frac{b^2 - r^2}{2 \rho} \right).
\]

(A3)

where \( \sigma_m = \text{trace}(\vec{\sigma})/3 \).

It is then demonstrated that the triaxiality \( \tau_\sigma \) is maximum in the center of the minimal cross section \( (r = 0) \). This value of maximal triaxiality \( \tau_\sigma \) can be expressed as:

\[
\tau_\sigma = \frac{\sigma_m}{\sigma_{eq}} = \frac{1}{3} \ln \left( 1 + \frac{b^2 - r^2}{2 \rho} \right).
\]

(A4)

According to Eq. (A4), \( \tau_\sigma \) only depends on the ratio \( b/\rho \). Therefore, it is often taken as reference triaxiality for NTp specimen. Namely, it can be deduced in the case of a sharp notch, i.e., \( \rho \rightarrow 0 \), \( \tau_\sigma \) tends to infinity. Fracture mechanics approaches are recommended to investigate the stress singularity effect. Conversely, if \( \rho \rightarrow \infty \), the geometry of the specimen tends to an uniaxial tensile specimen \( \tau_\sigma = 1/3 \). Moreover, it is clear from Eq. (A4) that, decreasing the notch root radius leads to an increase in the stress triaxiality ratio.

By inverting Eq. (A4), one can deduce \( \rho \) evolution corresponding to that of \( \tau_\sigma \) in the central part of the NT specimen due to geometrical term can be calculated by updating \( b = \phi_0 (1 + \Delta\phi/\phi_0)/2 \):

\[
\rho = \frac{\rho_0}{4} \left( 1 + \frac{\Delta\phi}{\phi_0} \right).
\]

(A5)

In addition, strains are assumed to be homogeneous within the cross section. They can be approximated by \( \varepsilon_{zz} = \varepsilon_{eq} \) and \( \varepsilon_{rr} = \varepsilon_{eq} = -(1/2)\varepsilon_{zz} \).

(A6)

The second equality is obtained with the isochoric deformation assumption, which can be questionable for polymeric materials.

Appendix B. Modified Gurson–Tvergaard–Needleman model

The Gurson–Tvergaard–Needleman (GTN) model (Gurson, 1977; Tvergaard, 1982; Tvergaard and Needleman, 1984), extended by Besson et al. (2001) and Besson and Guillemer-Neel (2003) was used in this study. Following Challier et al. (2006) and Laiarinandrasana et al. (2009a), the plastic flow potential \( \phi \) is written as:

\[
\phi = \sigma^* - R(p),
\]

(B1)

where \( R \) is the yield stress of the undamaged material and \( p \) an effective plastic strain representative of the matrix hardening. \( R \) is described as follows:

\[
R(p) = R_0 + Q (1 - e^{-bp}) + A e^{bp} - 1,
\]

(B2)

where \( Q(1 - e^{-bp}) \) describes the initial hardening stage supposed to be isotropic whereas \( A e^{bp} - 1 \) allows to simulate the rheo-hardening large stretching of the fibrils (Challier et al., 2006; Laiarinandrasana et al., 2009a).

To take the viscosity of the material into account, a Norton law is used:

\[
\dot{p} = \left( \frac{\phi}{R} \right)^n.
\]

(B3)

where \( K \) and \( n \) are material parameters to be identified.

\( \sigma^* \), introduced by Besson et al. (2001), is an effective scalar stress which is a function of both the macroscopic stress tensor \( \vec{\sigma} \) and the effective porosity \( \varepsilon^* \). \( \sigma^* \) is defined by the following equation:

\[
\Omega(\sigma, \varepsilon^*; \sigma^*) = \frac{\sigma^2}{2} + 2q_1 \varepsilon^* \cosh \left( \frac{q_2 \Sigma_{kk}}{2 \sigma} \right) - 1 - q_1^2 \varepsilon^* = 0.
\]

(B4)

where \( \Sigma_{eq} \) is the von Mises equivalent stress and \( \Sigma_{kk} \) the trace of stress tensor \( \sigma \). \( \varepsilon^* \) is a function of the actual porosity \( \varepsilon \) introduced by Tvergaard and Needleman (1984) to represent the void coalescence.

The plastic multiplier \( \dot{p} \) can be related to the deformation rate tensor:

\[
\dot{\varepsilon}_p = (1 - f) \frac{\partial \vec{\phi}}{\partial \vec{\sigma}}.
\]

(B5)

The evolution of porosity is expressed using the mass conservation:

\[
\dot{f} = (1 - f) \text{trace}(\dot{\varepsilon}_p).
\]

(B6)

Failure of a given integration point occurs when \( f = 1/q_1 \). Furthermore, \( q_2 \) parameter, expressed as a decreasing function of the maximum principal plastic strain \( \dot{p}_1 \), enables to take into
account the slowing down of the damage kinetics due to void elongation (Challier et al., 2006). However, to avoid discontinuity observed in the Challier et al. (2006) $q_2$ function, in the present model, $q_2$ is expressed as:

$$q_2 = \left( M - m \right) \frac{\tanh\left(\frac{p_1 - p_1^c}{k} \right)}{2} + m + \frac{p_1 - p_1^c}{2}$$

(B7)

where $M$, $m$, and $p_1$ are material parameters and $p_1$ a plastic strain value, all adjusted in such a way that:

- at $p_1 = 0$, $q_2$ has its maximum value equals to $q_{2,m} = (M - m)\frac{p_1 - p_1^c}{2} + m$;
- at $p_1 = p_1^c$, $q_2 = m$;
- when $p_1 \to -\infty$, $q_2$ tends to its minimum value equals to $q_{2,m} = -\frac{M - m}{2}$.

Note that for the values of $M$, $m$, $p_1$ parameters given in Table 2, $q_2$ continuously decreases from $q_{2,m} = 0.62$ to $q_{2,m} = 0.075$, when $p_1$ increases.

References


