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Voiding mechanisms in semi-crystalline polyamide 6 during creep tests assessed by damage based constitutive relationships and finite elements calculations

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ABSTRACT

Notched round bars made of a semi-crystalline polymer, polyamide 6, were submitted to creep tests. In order to study the microstructural evolution of the material during creep deformation, these tests were stopped at two characteristic creep times: i) the end of the secondary creep stage, ii) the onset of the failure during the tertiary creep stage, allowing 3D through thickness Synchrotron Radiation Tomography (SRT)-inspections. This SRT-technique allowed features of the damage to be assessed at a micrometre resolution within the notched region, such as spatial distributions of void volume fraction and void heights and diameters. These deformation mechanisms have already been observed and studied during steady strain rate loading and a multi-mechanism model coupled with damage formulation proposed to simulate notched specimens submitted to tensile loads. This model, based on porous visco-plasticity, was used here to simulate the creep behaviour of this PA6 material. The optimization procedure has led to a set of material coefficients capable of reproducing both macroscopic behaviour (creep curves) and damage micro-mechanisms. The level of maximum damage within the material and the spatial distributions of void volume fraction obtained numerically were compared successfully to experiments and the Cauchy stress tensor has been proved to be related to the void deformation mechanisms.

1. Introduction

The durability of engineering structures such as pressure vessels or pipes made of semi-crystalline polymers is of major interest because of the increasing demand for these structures over the last few decades. They are subjected to internal pressure for tens of years (50 years) and therefore to long term creep loading under a multiaxial stress state (complex geometries). The development of efficient predictive simulation tools is of prime importance so as to assess the lifetimes of these components in terms of constitutive relationships (mechanical behaviour) and of damage and fracture mechanics. Different approaches can be found in the literature to model the mechanical behaviour of semi-crystalline polymers. Micromechanical modelling, based on physical mechanisms involved in degradation, is one of them (Bédoui et al., 2006; Gueguen et al., 2008; Lee et al., 1993; Li and Shojaei, 2012; Nikolov and Doghri, 2000; Nikolov et al., 2002; Shojaei and Li, 2013). Based

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on these approaches, several linear visco-elastic visco-plastic rheological models have been developed (Ayoub et al., 2011; Khan et al., 2006). Authors have also proposed phenomenological models such as a visco-plasticity theory based on the overstressing of the material (VBO) (Colak, 2005; Colak and Krempl, 2005; Krempl and Ho, 2002; Krempl and Khan, 2003) later used by Dusunceli and Colak (2007). These phenomenological and micro-mechanical models have been used to simulate the macroscopic behaviour of semi-crystalline polymers under loading–unloading, creep and relaxation tests and take into account the degree of crystallinity. However, the effects of damage on the behaviour of such materials were not included in these models which were based on isochoric assumptions or limited to reversible volume changes. Recently, several studies have been carried out to incorporate the permanent volume change as a damage variable (Besson, 2009; Boisot et al., 2011; Cayzac et al., 2013; Ponçot et al., 2013; Saï et al., 2011; Uchida and Tada, 2011; Voyiadjis et al., 2011, 2012).

The present paper treats the assessment under creep loading of the multi-mechanisms model coupled with damage proposed by Cayzac et al. (2013). Experimental investigations on notched round bars made of PA6 semi-crystalline polymer subjected to interrupted creep tests have been performed by Selles et al. (2016). The macroscopic creep behaviour of this material has been studied based on the creep curves and Synchrotron Radiation Tomography (SRT) used to assess damage features. This non-destructive technique does not require any sample preparation and allows visualization of voids in 3D with their precise location within the sample. Finally, spatial distributions along the revolution axis (axial distribution) and in the net section (radial distribution) of cavitation descriptors (void volume fraction and void characteristic lengths) have been established. During tertiary creep, voids that are considered as cylinders underwent a change in shape from oblate (flattened disks oriented perpendicularly to the drawing direction) to prolate (arrangement in columns of cylindrical voids with lower aspect ratio).

Regrain et al. (2009a, 2009b) had already proposed modelling the PA6 creep behaviour with a multi-mechanisms approach but did not incorporate damage behaviour, in contrast to Saï et al. (2011). The constitutive model developed by Cayzac et al. (2013) succeeded in reducing the number of material coefficients used by Saï et al. (2011). The aim here is to assess the ability of this model to reproduce the coupled mechanical and damage behaviours in a Polyamide 6 (PA6) subjected to creep loading under a moderate triaxiality ratio.

2. Material and methods

2.1. Material description

The material studied in the present paper was a thermoplastic polyamide 6 (PA6). The properties of this semi-crystalline polymer have been published elsewhere (Laiarinandrasana et al., 2012; Cayzac et al., 2013). The crystallinity index $\chi = 43\%$, the glass transition temperature $T_g = 53\ ^\circ\mathrm{C}$ and the melting point $T_f = 219\ ^\circ\mathrm{C}$ have been obtained by Modulated Differential Scanning Calorimetry (MDSC) technique. A spherulitic microstructure has been identified using Scanning Electron Microscopy (SEM) inspections of chemically etched surfaces (Regrain et al., 2009a). The identified spherulites were characterised by a spherical shape and a mean diameter of about $\varphi_s = 5\ \mu\mathrm{m}$. Other microscopic observations by SEM of samples broken in liquid nitrogen (cryofractography technique) have been performed to reveal the presence of pre-existing voids on PA6 fractured surfaces (Laiarinandrasana et al., 2010). An initial porosity, assumed to be the initial void volume fraction, was estimated at about 1% and the void mean diameter determined to be around 0.1 μm obtained from the analysis of these SEM images.

2.2. Creep tests on notched round bars

The notched round bar geometry utilized in the present study has already been reported in previous studies (Cayzac et al., 2013; Laiarinandrasana et al., 2012). This geometry enhances void growth and a macroscopic uniaxial tensile load applied to the specimen produces a triaxial stress state in the cross section of the notched region. The notch root radius was 4 mm for the three samples (NT4 specimens) studied here (noted samples A, B and C). Geometrical characteristics of these specimens (undeformed and deformed), together with the experimental procedure of creep tests, have been published by Selles et al. (2016). The stress triaxiality ratio in the centre of the notched region under tensile load was considered as moderate (about 0.54). It should be recalled here that:

- Specimens have gauge section diameter of 7.2 mm and a gauge length of 65 mm;
- Initial net section diameter ($2 \times R_0$) was 3.5 mm for Samples A and C and 4 mm for Sample B.

These samples, in a dried state (0% of humidity within the material), were subjected to creep tests. The engineering net stress (σ_{net}) is defined as F/S_{net} where F is the load and S_{net} is the initial net section of the notched round bar. The applied σ_{net} on samples A, B and C were respectively 71, 75.4 and 71.5 MPa. The deformed notch opening (δ) is defined as the distance between the two notch shoulders and represented in Fig. 1a for Sample A. Table 1 summarizes these experimental data, together with the numerical results that will be discussed in Section 5.

Fig. 1b displays the creep displacement (Δu_c), defined as the difference between the applied crosshead displacement during the test and at the end of the loading stage, as a function of the time for the three samples. The experimental creep