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Damage characterization for particles filled semi-crystalline polymer

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Abstract. Damage evolution and characterization in semi-crystalline polymer filled with particles under various loadings is still a challenge. A specific damage characterization method using Digital Image Correlation is proposed for a wide range of strain rates considering tensile tests with hydraulic jacks as well as Hopkinson's bars. This damage measurement is obtained by using and adapting the SEE method [1] which was developed to characterize the behaviour laws at constant strain rates of polymeric materials in dynamic. To validate the characterization process, various damage measurement techniques are used under quasi-static conditions before to apply the procedure in dynamic. So, the well-known damage characterization by loss of stiffness technique under quasi-static loading is applied to a polypropylene. In addition, an in-situ tensile test, carried out in a microtomograph, is used to observe the cavitation phenomenon in real time. A good correlation is obtained between all these techniques and consequently the proposed technique is supposed suitable for measuring the ductile damage observed in semi-crystalline polymers under dynamic loading. By applying it to the semi-crystalline polymer at moderate and high speed loadings, the damage evolution is measured and it is observed that the damage evolution is not strain rate dependent but the failure strain on the contrary is strain rate dependent.

1. Introduction

Damage characterization in semi-crystalline polymers is still a real challenge. Due to the deformation process, an early necking occurs and leads to a non-homogeneous strain and strain rate field into tensile specimen. This inhomogeneity state is then a problem to determine constant strain rate behaviour law to be used in finite element simulations and consequently to obtain damage evolution as well. Mostly damage studies based on volume variation have been conducted under static conditions and by controlling the in real time the tensile displacement in order to obtain a constant strain rate. A video controlled-system is generally used to measure the volume variation with seven spots painted on the tensile specimen [1,2]. It is also possible to use inverse technique by identifying the parameters of a damage model by comparing and correlating same measurements obtained from experiments and finite element simulations and also taking into account the initial stress triaxiality ratio state [3–5]. Nevertheless, if you want to obtain the damage evolution without numerical simulations and for dynamic loadings, all these methods are not adapted for this particular condition. In consequence, another approach is needed. In this paper, the $SE\dot{E}$ method [6], which was first developed for the polymer behaviour identification on a large strain rate range, is extended to the damage measurement on a large strain rate range. This method is applied to a polypropylene filled with 20% wt of mainly talc particles called Hostacom by LyondellBasell, for various strain rates and on small volumes all along the tensile test specimen. To validate this approach, the method is first applied under quasi static loadings and compared with the loss of stiffness technique. Tensile tests inside a microtomograph are also performed to complete the validation as it is a very powerful technique to visualize the damage inside the specimen [7,8]. As good results are obtained by comparing these various techniques, the $SE\dot{E}$ method for damage measurement is then applied to dynamic tensile tests carried out on a hydraulic jack and Hopkinson bars'. The results obtained for this semi-crystalline polymer filled with mineral particles highlight the independence of the damage evolution to the strain rates.

2. The loss of stiffness technique versus the SEE method for damage

The damage evolution is given by the loss of stiffness technique by measuring the evolution of the Young modulus during a cyclic tensile loading [9] as illustrated Fig. 1 and by calculating the damage variable D by:

$$D = 1 - \frac{\tilde{E}}{E} \qquad (1)$$

with E the initial Young modulus and $\tilde{E}$ the damaged Young modulus.

The corresponding behaviour obtained for the semi-crystalline polymer is presented Fig. 2. This result is obtained with a notched specimen loaded with a

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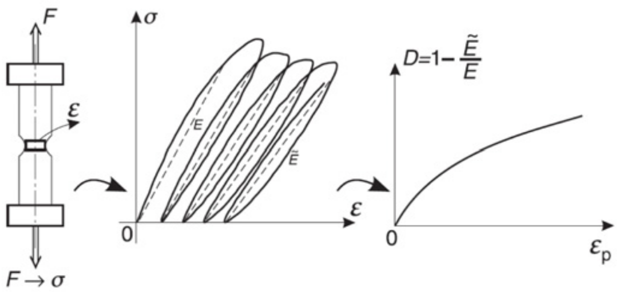

Figure 1. Procedure for damage measurement by loss of stiffness technique: Step1: Longitudinal strain measurement on lighted notched tensile specimen, Step2: measurement of an equivalent Young modulus for each hysteresis, Step 3: calculation of the damage evolution.

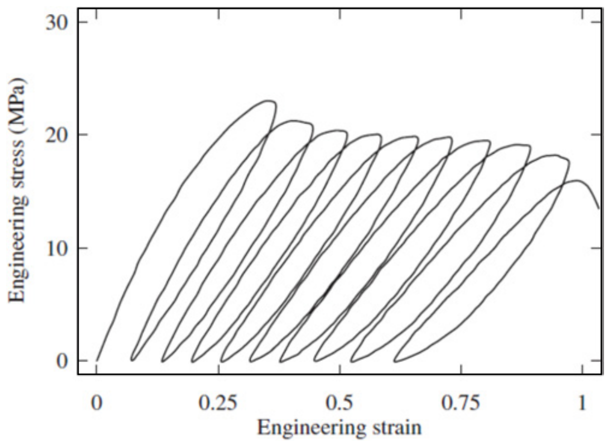

Figure 2. Engineering stress-strain curve obtained by the tensile test with repeated unloadings.

prescribed displacement of 1mm/min for both loadings and unloadings. Viscoelastic behaviour is observed with some hysteresis in the loading-unloading phases. In the classical loss of stiffness technique, the Young modulus evolution is captured by measuring with the unloading slope. Due to the non-linearity of the results for this material, the damage is then calculated with an apparent Young modulus obtained by taking the upper point, at the crossing between loading and unloading curves, and the lower point, at zero stress.

For the $SE\dot{E}$method, the damage evolution is obtained at constant strain rate by comparing the behaviour laws under the assumption of incompressibility and transverse isotropy. The $SE\dot{E}$ method used the digital image correlation to obtain the strains and strain rates in different Zone Of Interest (ZOI) on the tensile specimen during a tensile test. The true stress which takes the reduction of the real cross-sectional area into account in each ZOI, is therefore calculated assuming the compressibility (transverse isotropy) $\sigma_{yy_i}^{com}$ or the incompressibility $\sigma_{yy_i}^{inc}$ hypothesis. The damage evolution is therefore determined by the following relation:

$$D = 1 - \frac{\sigma_{yy_i}^{com}}{\sigma_{yy_i}^{inc}}. \qquad (2)$$

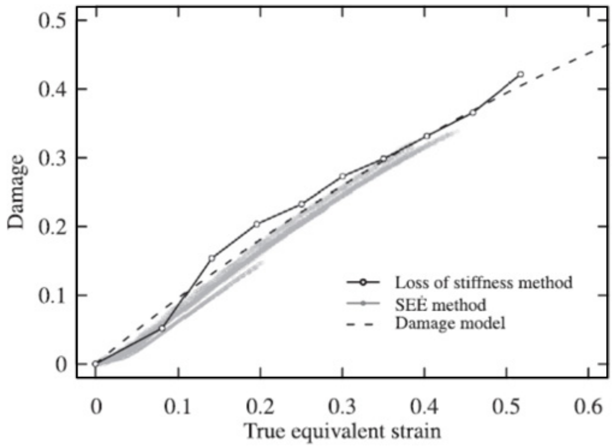

Figure 3. Damage evolution obtained with the loss of stiffness technique and the SEE method and the corresponding damage model.

With

$$\sigma_{yy_i}^{inc} = \frac{F_i}{S_{o_i}} \exp(\varepsilon_{yy_i}); \sigma_{yy_i}^{com} = \frac{F_i}{S_{o_i}} \exp(-2\varepsilon_{xx_i}) \qquad (3)$$

where F_i, S_{o_i}, ε_{xx_i} and ε_{yy_i} are the force, the initial cross section, the transversal and longitudinal strains of the ZOI subscribed i. A damage model (4) is also identified based on the work of Balieu [10] and presented in Fig. 3 with both the damage from the loss of stiffness technique and the $SE\dot{E}$ method.

$$D = 1 - \exp\left(\frac{k}{k_c}\right) \qquad (4)$$

with k the equivalent plastic strain and k_c a material parameter.

Results obtained by the both techniques are close and validate the approach for measuring the damage evolution by the $SE\dot{E}$ method. To conclude this static part of the damage measurement, an in situ tensile test on a small specimen is performed inside a microtromograph (μcT) at three different deformations to observe and quantify the porosity. μCT imaging is performed thanks to a 1172 Skyscan system, with 40 kV and 100 mA settings. The voxel size for all μCT data is adjusted to 3.97 mm. This choice ensures capturing small voids size (around 8 mm) with a reasonable imaging duration. Under these conditions, the acquisition of the sample geometry in each mechanical state takes about 8 h. A first μCT-scan is carried out at the initial state (i.e. undamaged material) and two other μCT-scans are performed to follow the evolution of the specimen microstructure at different deformed states. The reconstruction of tomography data leads to a set of grayscale images (Fig. 4) obtained along the principal direction of the sample.

In order to correctly estimate the damage, an accurate measurement of the cross section of the specimen is necessary. The first use of μCT is to analyse the outer shape of the specimen and how it changed during deformation. The edges of the cross section are detected and the sum of all the pixels leads to the effective cross sectional area S. The real resisting cross sectional area

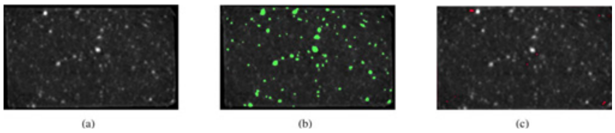

Figure 4. Initial cross section visualization, a) Grayscale image, b) particles distribution in green, c) voids distribution in red.

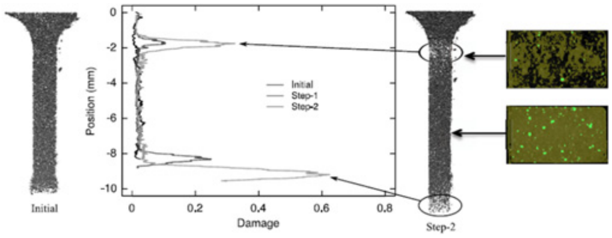

Figure 5. Evolution of the damage measured by μCT.

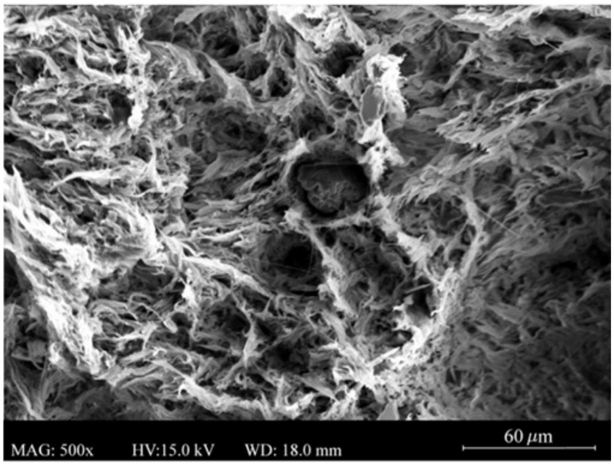

Figure 6. High magnification SEM micrograph of the fracture surface of particles filled semi-crystalline polymer under tensile loading at 1 mm/min.

can also be computed by retrieving the void surface (S_D) from the effective cross section area. The effective cross sectional area along the length gauge of the specimen is presented in Fig. 5 for the initial and two deformed states. In this test, the deformation occurs mainly at the extremities of the length gauge. At the bottom of the specimen, where the displacement is applied, effective cross-sectional area decreases strongly.

The tensile test in-situ μCT is an interesting technical solution to visualize the evolution of the voids during deformation. Unfortunately, the localization of the deformations in two very small parts of the specimen makes the deformation measurement difficult. The calculated damage value is therefore, in this case, not accurately correlated to the deformation. Nevertheless, the three techniques converge to an equivalent measurement of the damage evolution with important volume of porosities at the last stage before as highlighted in the image of the fracture surface in Fig. 6.

After validation of the damage measurement by the $SE\dot{E}$ method in static, this method is then applied on dynamic tensile tests at various speed loadings to evaluate the influence of the strain rate on the damage evolution.

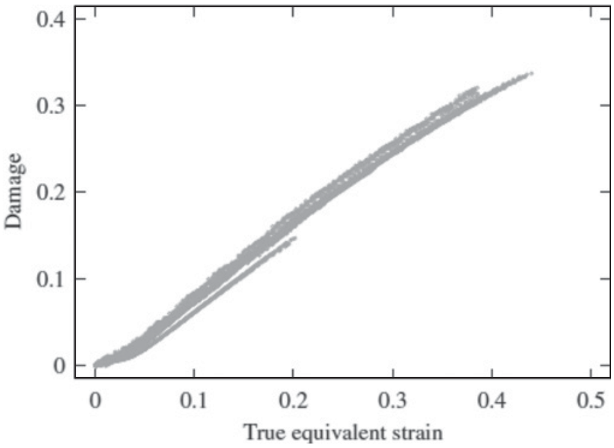

Figure 7. Damage evolution obtained with the SEE method from low to high strain rates.

3. Application to dynamic tensile tests

A high speed hydraulic machine (Instron 65 kN-20 m/s) is used for the dynamic loadings. A piezoelectric load cell-calibrated in the range [0;5] kN with a precision of 25 N is fixed in the upper part of the machine and a LVDT sensor is used to measure the displacement of the jack during the test. The lower part is composed of the clamping system used to load the specimen once the velocity is constant. The strain rate effects are studied at 0.08, 0.8 and 4 m/s which correspond to average equivalent strain rates of 2.6, 26 and 133 s^{-1}, respectively. Due to ringing phenomenon of the assembly (6 kHz), tensile tests are also performed on direct tensile Hopkinson bars over 200 s^{-1}. This equipment is composed of two aluminium bars made of 6060 aluminium alloy with 20 mm in diameter and a hollow striker made of Polyamide 66 with respect to impedance matching issues. The length of the input, output bar and the striker are respectively 5.63, 4 and 1 m. For these tests, two input pressures are used, 9 and 17 bars, which correspond in terms of velocities of the striker to 6.7 and 11.5 m/s. Two approximate strain rates of 350 and 600 s^{-1} can be obtained during the tests with these velocities. High speed imaging is used with digital image correlation in order to obtain the behaviour laws at different strain rates and then calculate the damage corresponding.

The results obtained by using the $SE\dot{E}$ method for damage measurement with all these static and dynamic tests are presented Fig. 7. All the points plotted in this figure are extracted from all the ZOI on the tensile specimens. As consequence, the damage evolution for this mineral filled polypropylene is always the same whatever the strain rate imposed by the tensile conditions. The damage is then not strain rate sensitive. Nevertheless, fracture of the specimens occur at different fracture strain according the speed loading conditions which means that the fracture strain is strain rate dependent.

Average values of the fracture strains according the initial strain rates are plotted Fig. 8. For low strain rates, the fracture strain first increase which could link to the small increase of the temperature due to deformation which helps the molecular chain motion. When the strain rates increase,

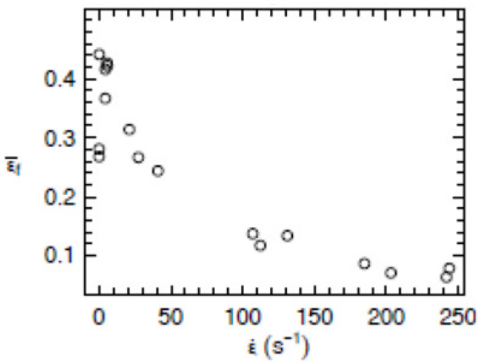

Figure 8. Fracture strain evolution for a tensile test with different strain rates.

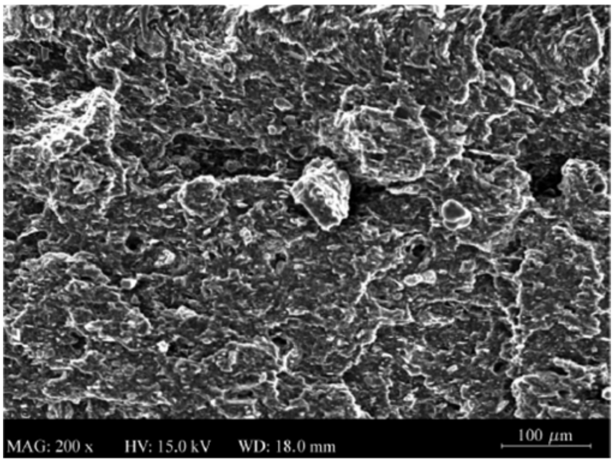

Figure 9. Fracture surface obtained by magnification SEM micrograph under tensile loading at 600 s^{-1}.

this temperature effect disappears and a ductile to fragile transition is observed as the pictures of the fracture surface highlighted it (Fig. 6 and Fig. 9).

4. Conclusions

A method is proposed to determine the damage evolution on a large strain rate range for particles filled semi-crystalline polymers. This method used information obtained by digital image correlation on tensile specimens loaded at different speeds. The damage is then calculated on the basis of the $SE\dot{E}$ method. To validate the damage measurement with this method, results are compared with thus obtained by the loss of stiffness technique in static and correlated to information obtained with an in-situ μCT tensile test. As the results are in agreement, this method is applied to dynamic tensile tests on hydraulic jack and Hopkinson bars. The damage measurement obtained highlights that the damage evolution is independent of the strain rates. Nevertheless, the fracture strains are strain rate dependant with a ductile to fragile transition at high speed loadings.

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References

[1] C. G'Sell, J. Jonas, J Mater Sci, **14**, 583–91 (1979)
[2] F. Zaïri, M. Naït-Abdelaziz, J. Gloaguen, J. Lefebvre, Int. J. Plasticity, **24**(6), 945–65 (2008)
[3] G. Boisot, L. Laiarinandrasana, J. Besson, C. Fond, G. Hochstetter, Int. J. of Solids and Structures **48**, 2642–2654 (2011)
[4] F. Lauro, B. Bennani, P. Croix, J. Oudin, J. Mater. Process Technol., **118** (13), 472–477 (2001)
[5] F. Lauro, B. Bennani, P. Drazetic, J. Oudin, X. Ni, Commun. Numer. Methods Eng., **13** (7), 113–126 (1997)
[6] F. Lauro, B. Bennani, D. Morin, A Epee, Int. J. Impact Eng., **37**(6),715–722 (2010)
[7] L. Laiarinandrasana, T.F. Morgeneyer, H. Proudhon, C. Regrain, J. Polym. Sci. B. Polym. Phys. **48**, 1516–1525 (2010)
[8] R. Balieu, F. Lauro, B. Bennani, G. Haugou, F. Chaari, T. Matsumoto, E. Mottola, Int. J. Impact Eng., **76**, 1–8 (2015)
[9] J. Lemaitre, J. Eng. Mater. Technol., **107**(1), 83–89 (1985)
[10] R. Balieu, F. Lauro, B. Bennani, R. Delille, T. Matsumoto, E. Mottola, Int. J. Plasticity, **51**, 241–270 (2013)