Local approach of fracture on semi-crystalline polymers: contribution of X-ray laminography technique

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Abstract Damage mechanisms in a PolyAmide 6 semi-crystalline polymer were characterized by using Synchrotron Radiation Laminography technique on CT like specimen. Damage appeared as multiple penny shaped crazes. The maximum damage occurred at mid-thickness and located at a small distance from the notch root. An attempt was made to apply local approach of fracture concept thanks to finite element analysis using damage-based constitutive model. FE simulation successfully captured the aforementioned micro-mechanisms of crack initiation, by void coalescence. Further work is carried out to determine the crack dimension corresponding to the maximum net stress: the criterion being used for the global approach of fracture.

Keywords Polymers, X-ray laminography, Local approach of fracture, Finite element

1. Introduction

The use of fracture mechanics concepts for polymers is subjected to controversial discussions. It is often reported that many requirements are not fulfilled to obtain fracture mechanics characteristics such as the toughness. For instance, plane strain conditions together with tunneling effect (curved crack front), crack advance measurement, crack initiation/growth determination…are more difficult to obtain for polymers than in metallic materials. In the recent years, growing interest for X-ray tomography technique was observed. Indeed, this is a non destructive method allowing for construction of digital image in 3D. Therefore, virtual cuts and local observations of the microstructure can easily be carried out. The present contribution aims at highlighting the crack initiation concept from CT like specimen on a semi-crystalline polymer: PolyAmide 6 (PA6). The evolution of the microstructure during an increasing load could be observed thanks to laminography technique. The local coalescence of voids at mid-thickness was evidenced. The increase in macroscopic net stress was computed by Finite Element (FE) analysis, with a dedicated damage based model. The crack initiation criterion at macroscopic scale, based on the maximum net stress, was then assessed in terms of critical size of the principal damage within the microstructure.

2. Experimental procedure

2.1. Material - Specimen

The material of interest is a PolyAmide 6 (PA6) [1], selected among several semi-crystalline polymers. Indeed, for this polymer void morphology and distribution were already studied [2] for axi-symmetrically notched specimens, together with a dedicated constitutive model for FE analysis. In the present work, the approach consisted of application of the local approach of fracture which takes the damage evolution in the microstructure into account. Here a novel technique, known as laminography is utilized [3]. The method takes advantage of the Synchrotron Radiation Tomography (non destructive and 3D reconstructed images) but applied to plate like specimen. In situ test is carried out on CT like specimen. The geometry is described in fig.1a. The specific dimensions were: thickness B = 2mm, width W = 40mm, crack depth ratio a/W = 0.5, notch root radius ρ = 250μm, half height H/2 = 20mm. For the following FEA (section 3), figs.1b-c respectively illustrate the CT specimen overall meshing and refined meshes (25μm) in the vicinity of the crack tip.

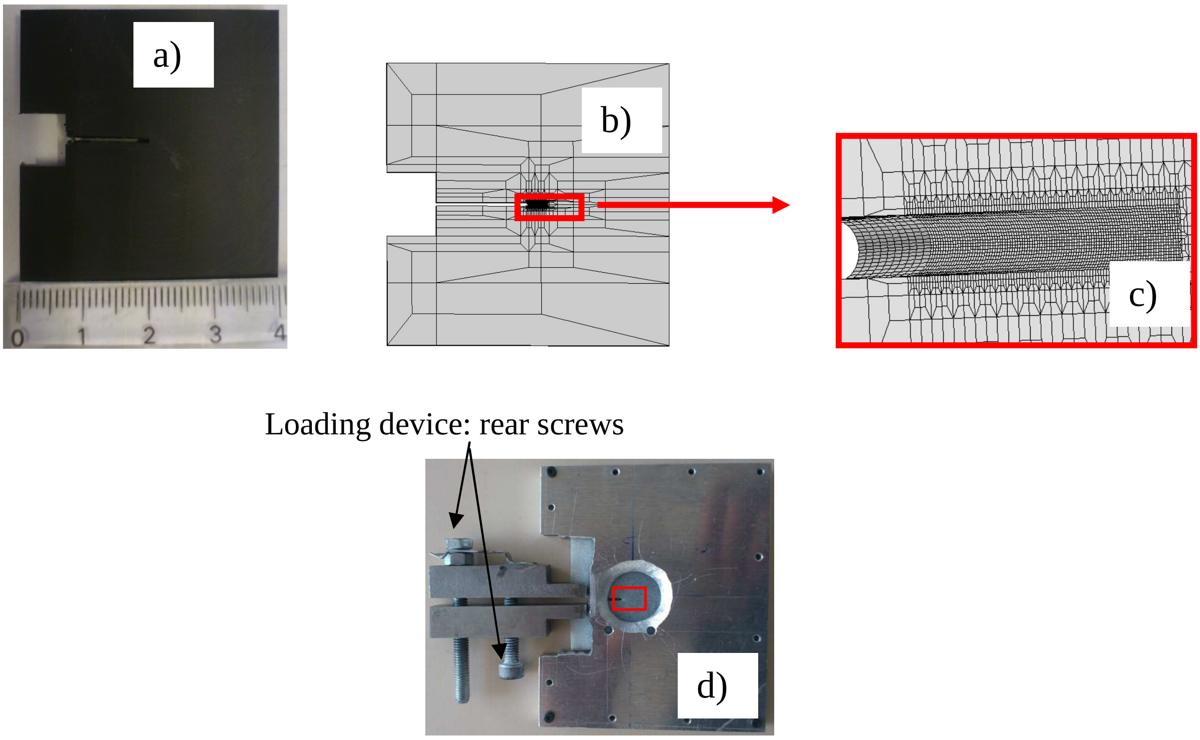

Figure 1. CT specimen geometry with the experimental setup: a) CT specimen sketch; b) Overall view of the sample meshing; c) Fine meshing near the notch root (mesh size 25μm); d) Experimental setup.

The experimental setup is presented in fig.1d. The CT specimen was inserted between two aluminum plates to avoid buckling in the compressive part of the remaining ligament. The opening displacement is applied thanks to the turns of both rear screws. A circular window was designed within the anti-buckling aluminum system allowing for inspection of the region near the notch root, at the macroscopic scale (pictures), as well as at the microscopic scale (laminography scans).

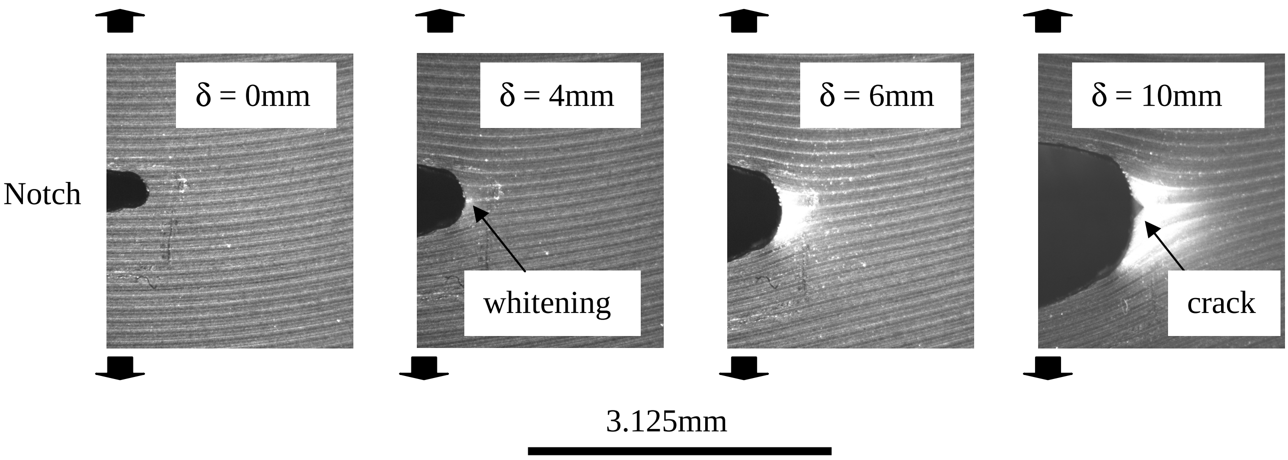

Figure 2. Evolution of the geometry observed at the external face of the CT specimen. Black arrows indicate the loading direction.

Stepwise loading was applied between laminography observations. Fig.2 shows a series of pictures made at the side view of the specimen before the laminography scans. The corresponding applied displacement δ is indicated in each viewgraph, starting from the initial material without any deformation, up to δ = 10 mm. For this latter, a crack is visible at the external surface of the sample. For δ = 4mm, a localized whitening at the notch root is observed. Being the first event appearing at the surface, it was considered that this corresponded to an internal crack initiation. For δ = 6mm, whitening is spreading out, surrounding the notch root. It should be mentioned that no scan was done between δ = 6mm and 10mm. Moreover, the present experimental setup was not equipped with a load cell. The load level was therefore estimated by using FEA with a dedicated constitutive model.

2.2. Laminography technique

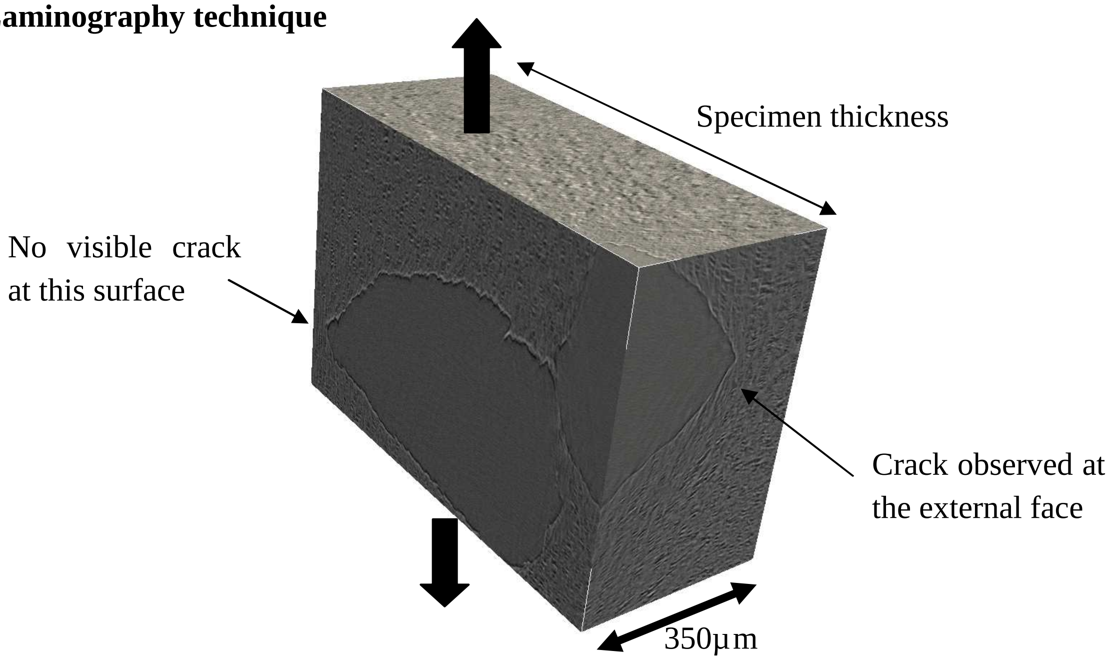

Figure 3. Volume corresponding to δ = 10mm: two cracks through the thickness

Synchrotron Radiation Laminography (SRL) technique is detailed elsewhere [3]. It has already been applied on both metallic and laminate composite materials. To the authors' best knowledge, it is the first time that this technique is utilized for semi-crystalline polymer investigations. To this end, for each value of the applied opening displacement δ the whole experimental setup (fig.1c) was mounted on adapted sample support of the laminography device at the ID19 beamline of the European Synchrotron Radiation Facility (ESRF) in Grenoble (France). The scanned volume was about 1 mm^3, with a voxel size of 0.7µm. Fig.3 shows a trough thickness microstructure around the crack tip in 3D. Apart from multiple crazes observed within the whole volume, two large cracks were evidenced. One of them appeared at the surface (fig.2, δ = 10 mm) whereas the opposite face did not show any crack. Attention is now paid on the scans corresponding to δ = 4mm and δ = 6mm to observe the microstructure, especially at mid-thickness.

Fig.4 illustrates such views in 3D where the plane at mid-thickness is shown in forefront. Multiple penny shaped crazes [2, 4] were observed around the notch root. These features could be linked to the whitening observed at the macroscopic scale.

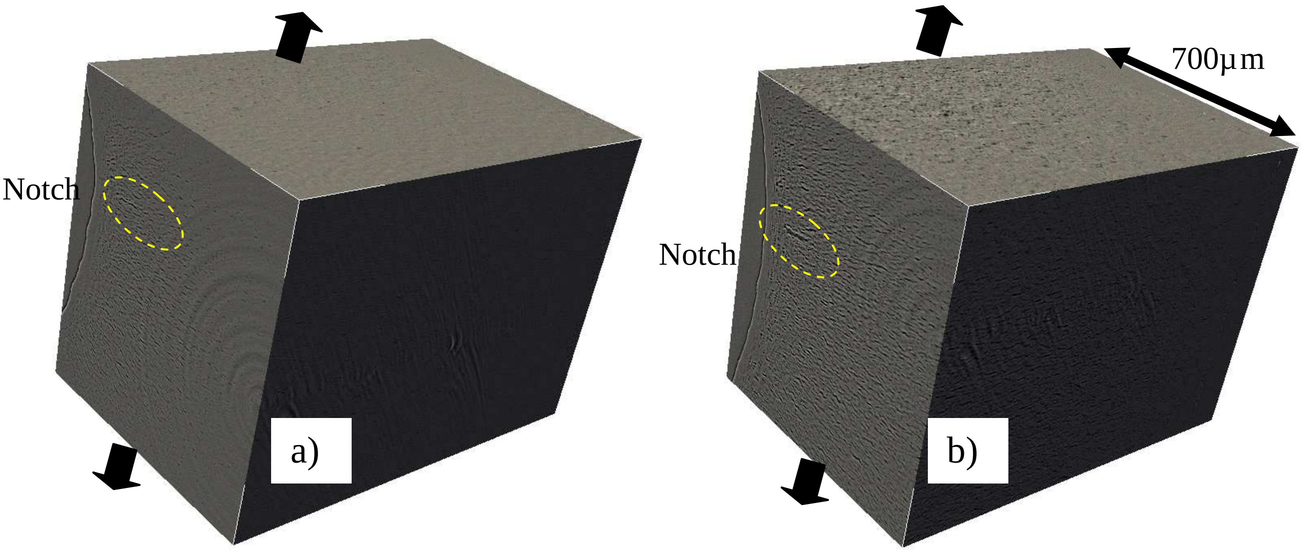

Figure 4. Observations at mid-thickness plane: a) δ = 4mm; b) δ = 6mm

A significant damage, surrounded by the dashed lines was evidenced at mid-thickness, located at a small distance (~100µm) from the initial notch root.

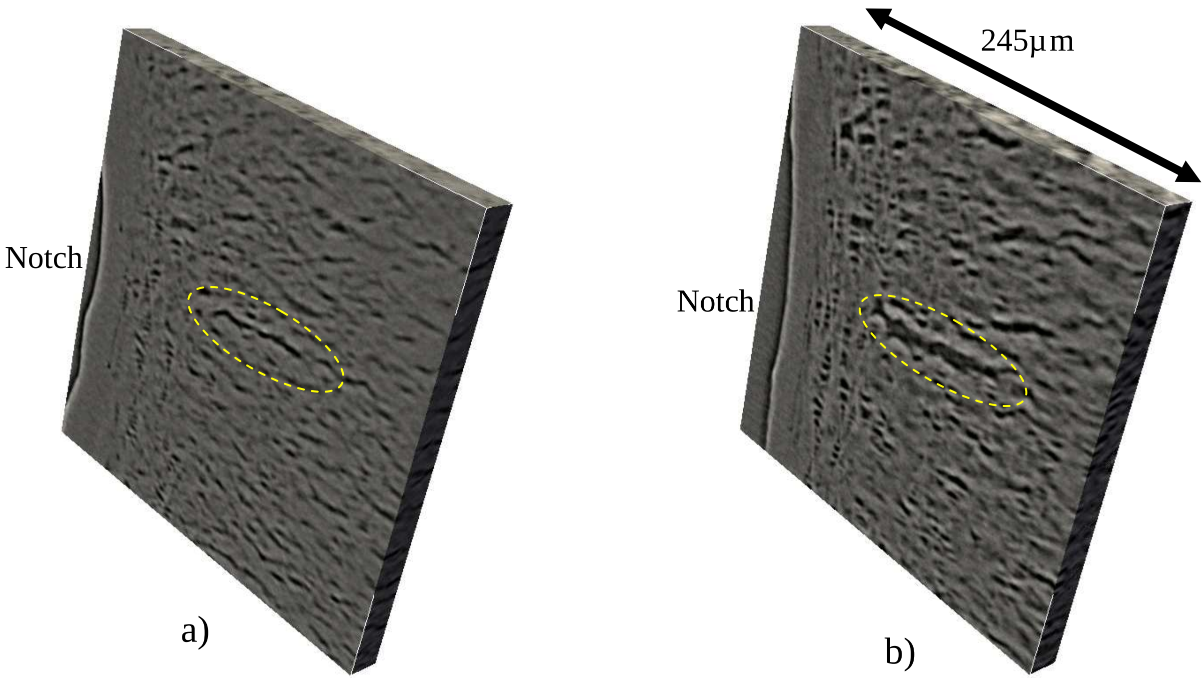

Figure 5. Maximum damage at mid-thickness surrounded by dashed lines: a) δ = 4mm; b) δ = 6mm

Fig.5 details the morphology and the size of this damage. To this end, a 19µm thick slice of PA6 scan was extracted and analyzed for both δ values. It was observed that the damage consisted of crazes (presence of fibrils within the voids) aligned (coalesced) following the crack remaining ligament plane. This damage increased in height from δ =4mm to 6mm. Its average values of the characteristic dimensions were summarized in Table 1.

Table 1. Characteristic dimensions of the damage located at mid-thickness

	δ = 4mm	δ = 6mm
Length	60μm	90μm
Height	5μm	10μm
Width	20μm	30μm

3. Finite element modeling

	Broken elements in red	Broken element removed Contour map of the damage
a)		
b)		
c)		

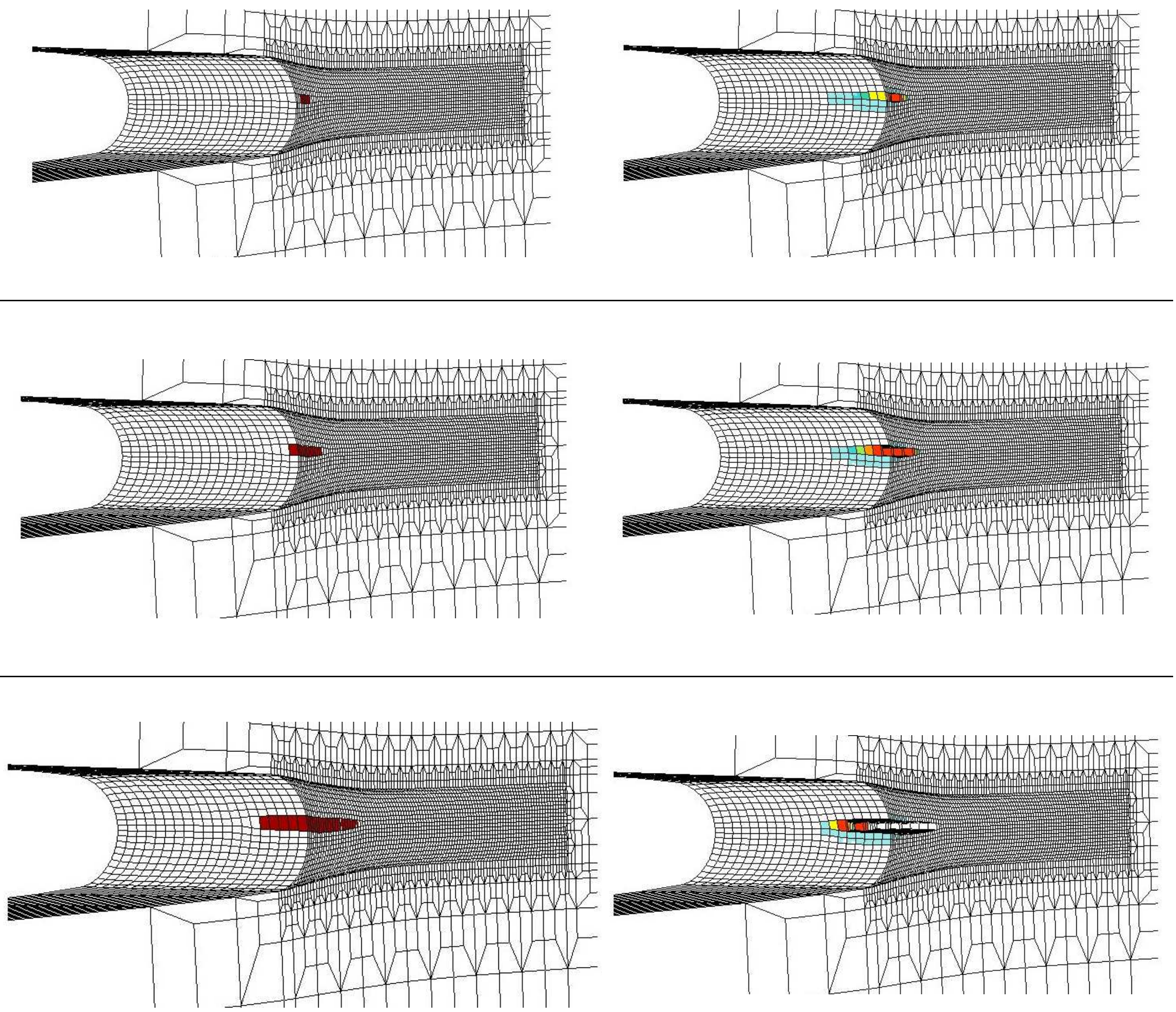

Figure 6. FE simulations of the crack initiation by using damage based constitutive model.

The local approach of fracture requires FE computation with a damage based constitutive model to better assess the stress/strain tensors distribution within the material. For the PA6 under study, a multi-mechanisms model was proposed [5-6] where the void volume fraction is considered as an internal variable. By using the same damage based model, simulation of laminography CT test was carried out. A procedure of “kill/remove element” was applied when a critical porosity value was reached within this element. The following analyses focus on the refined meshes (fig. 1c) in the vicinity of the notch root. Constant mesh size of 25µm was set for 2.5mm along the ligament.

Fig.6 summarizes the evolution of damage (broken element) during the increasing load. The forefront face corresponds to the plane at mid-thickness. The first column of deformed meshes shows the broken elements in red. The second column illustrates the contour map of the void volume fraction over the deformed mesh where the broken elements were removed.
Fig.6a shows that four elements simultaneously broke for the first time (length ~100µm; width ~25µm). They appeared at mid-thickness and located at 3 elements away from the notch root (distance ~75µm). These characteristic values correspond to those of δ = 6mm. Fig.6b indicates that broken elements propagate into two opposite directions: ahead of and back to the notch root. The width seems to remain constant (one element size). Fig.6c evidences that the broken elements propagates through the thickness as well, but did not reach the external surface. These latter steps were not observed with SRL technique since no scan was done between δ = 6mm and δ = 10mm when the crack appeared at the external face. However, although fine tuning of damage parameters is still ongoing, these figures demonstrate that the FE analysis with the damage based model was able to reproduce the micro-mechanisms of crack initiation. At present time, computation is still running to obtain the configuration where the crack reaches the external surface.
Next investigation consists of using FE modelling to estimate the load evolution during the crack propagation. The aim is to determine the crack size allowing the crack initiation criterion at the macroscopic scale to be reached (maximum load). To this end, in fig.7 the load divided by the net section of the remaining ligament is plotted as a function of the applied opening displacement.

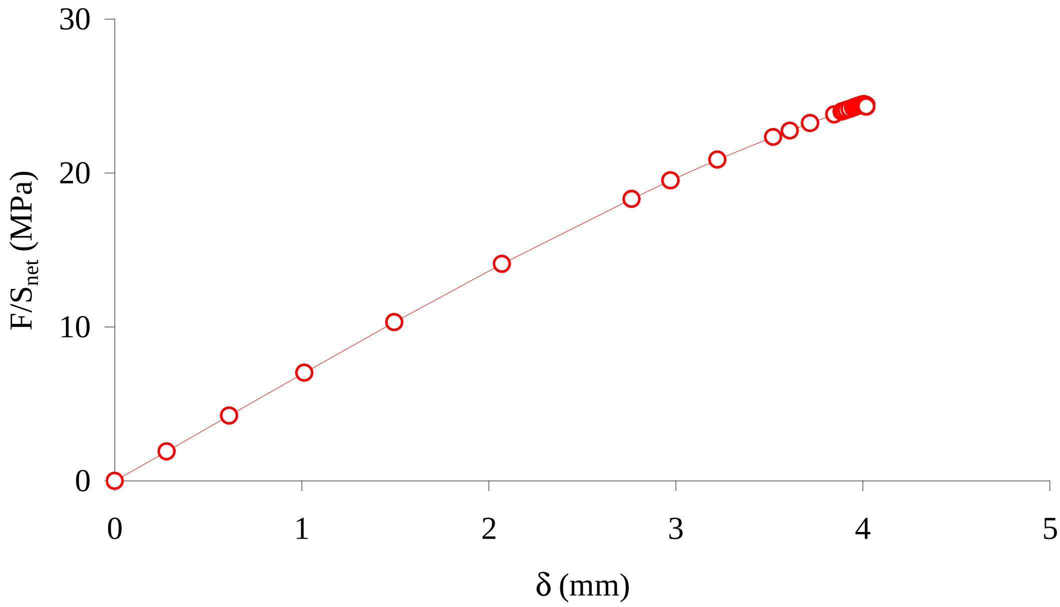

Figure 7. Net stress as a function of the opening displacement by FE analysis

Contour maps presented in fig.6 correspond to the last loading steps of the numerical simulations (δ ≥ 4mm). The first results shown at this stage indicate that although a non linearity appears in the last part of the plot (fig.7), the net stress is still increasing. The maximum net stress is not yet reached. Final results will be shown later.

5. Conclusion

The micro-mechanisms of damage and crack initiation were investigated thanks to Synchrotron Radiation Laminography (SRL) technique. In situ tests were carried out on CT-like specimen. The local coalescence of voids leading to crack initiation occured at mid-thickness of the specimen located at a small distance from the initial notch root. Finite Element analysis, with a damage based constitutive model was able to capture the location of the crack initiation, as well as the damage mechanisms observed by SRL. The evolution of the macroscopic net stress was also computed by FEA. At present time, the crack initiation criterion based on the maximum net stress has not still been reached. Computation is still ongoing to compare this criterion with the critical size of the crack at the microscopic scale.

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