# Analysis of Damage in Metallic Materials by X-ray Tomography

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#### 1. ABSTRACT

In situ tensile test coupled with X-ray tomography is a powerful tool for 3D reconstruction and non destructive observation of microstructure and damage of materials, in particular for the comprehension of nucleation, growth and coalescence of voids in metal alloys. A set of tests has been carried out on dualphase steels but also ferrite and martensite. These tests were carried out in order to compare the performance of the dual phase material with a good knowledge of the behaviour of its constituents separately. two **Oualitative** analysis of the carried damage events was out at many deformation steps, on a same 3D region in the reconstructed volumes. This technique allows a deep insight into the material behaviour and provides experimental support to damage analysis, previously carried out by SEM and optical microscopy. The 3D reconstruction also provides the opportunity for building an accurate geometry for FE analysis.

Keywords: steel, dual phase, damage, tomography

## 2. INTRODUCTION

Dual-phase (DP) steels, composed by ferritic ductile matrix (89%) and martensitic hard islands (11%), are the high strength materials more widely used in modern automotive industry. Understanding their properties for industrial applications requires the knowledge of the role of the microstructure on the mechanical behaviour and damage mechanisms. In particular, according to Kumar [1] the hard phase is responsible of the effect on the yield stress and work hardening, but also the brittleness of the martensitic phase is likely to promote damage [2] and reduces the ductility. Predictive models for damage have been carried out from the simplest Rice and Tracey (RT) approach [3] to the popular Gurson approach [4]. Several authors agree on the fact that the best method for quantifying damage (this is required to validate the different models) is X-ray tomography [11,18]. This non destructive technique can be used like a simple microscopy technique with a slightly lower resolution than conventional microscopes (of the order of 1 micron) and provides three dimensional (3D) images of samples of different materials during in situ tensile tests.

## 3. MATERIALS AND METHODS

The DP steel used for this study is cut from a 3 mm thick sheet obtained by hot rolling and thermal treated. X-ray microtomography have been used to quantify damage during in situ tensile tests. Although never directly applied to DP steels, the method can be used for the imaging and quantification of the microstructure of materials. The tomography setup at the ID15 beam line of the European Synchrotron Radiation Facility (ESRF) in Grenoble (France) has been used to achieve a very fast radiography acquisition speed by combining a high efficiency scintillator screen, a reflecting microscope objective and a fast charge coupled device (CCD) detector (1024x1024 pixels) with an intense high-energy white beam radiation produced by the source. The spatial resolution achieved is 2 microns and the voxel size is 1.6x1.6x1.6µm<sup>3</sup> [7]. A dedicated in situ tensile machine was mounted on the rotation stage of the tomography setup. The following experimental conditions were used: white x-ray radiation with a peak energy set to 60 keV, a number of projections of 500 and a time for recording one projection of 150 ms. The stage rotates at a constant speed during data acquisition.



Figure 1 – the experimental materials: tomography device and DP specimen geometry

Figure 1a shows the tomographic device on the beam line with the cylinder uncovered, ready for specimen mounting. Figure 1b and c show the DP specimen mounted in the cylinder, with the displacement of the top clamp mechanically controlled with a stepping motor, and in Figure 1e the geometry of specimens is sketched. The raw data for each deformation step has been represented by a stack of gray level images, analysed using the software ImageJ. The gray level images have been filtered and binarized using a connected threshold grower algorithm to differentiate the voxels belonging to the cavities from those belonging to the solid phase. Minimal section for each step has been identified and a 200x200x200 voxels cube centred on the minimal section has been considered for further investigations (see Figure 1e). The reason for such a restriction of the analysed volume is that the triaxiality and deformation when the sample starts to neck become heterogeneous. If the analysed volume is small (i.e. a cube of side 0.3mm in the center of the neck), the variation of triaxiality and deformation can be assumed to be small.

## 4. RESULTS

Figure 2 shows a sample of dual phase steel raw tomographic data after filtering. The outer shape of the sample has been used to determine an approximation of the local value of the tensile strain.



Figure 2 – dual phase steel tomographic data at different strain values

The ratio of the value of the surface S(z) of the sample in a given section after deformation over the value of the same surface in the initial state  $S_0$  allows to estimate the local value of the true tensile strain in each slice, using Eq. 1:

$$\varepsilon = \ln \frac{S_0}{S_{(z)}}$$
 Eq.1

By knowing the value of true strain, it is possible to express all the information obtained by data analysis as a function of strain. In Figure 2 it is possible to observe that the necking occurs at  $\varepsilon = 0.17$ . This has been verified for all samples. The material shows already a few defects before necking. Around  $\varepsilon = 0.43$  first evidences of nucleation appears. Nucleation is concentrated in the minimal section. At  $\varepsilon = 0.78$  the biggest voids are easily recognisable and they are mostly responsible of the fracture after  $\varepsilon = 0.91$ . It is interesting to note how the biggest voids centre of mass is not consistently changing from one deformative state to the following. This allows rebuilding the history of biggest voids along the deformation. For this population, with the support of tomographic data, it is reasonable to use the Rice and Tracey model for predicting growth.

Seperately, and in order to understand the behaviour of dual phase steels, tomographic tests have been carried out also on the two phases constituting the composite, i.e. the martensitic phase and the ferritic phase. Figure 3 shows the tomographic image of a purely martensitic sample. This sample was aged so it exhibits a non negligible ductility.



Figure 3 – martensite (hard phase) tomographic data at different strain values

As expected, the martensite presented a minor necking at a strain higher than  $\varepsilon = 0.17$ . Also, the brittleness of the hard phase promotes an unpredictable fracture after few deformative steps, as reported by Bouaziz et al. [8], and with a lower

necking, compared to dual phase steel. Figure 3 shows a scattered nucleation at  $\varepsilon = 0.33$ , and only at the latest steps of deformation the voids show a concentration around the minimal section ( $\varepsilon = 0.57$ ).

Figure 4 shows the purely ferritic sample. This sample is representative of the ductile phase in the dual phase steel analysed by tomography.



Figure 4 – ferrite (ductile phase) tomographic data at different strain values

Since the early states of deformation the ferrite shows a pronounced necking, the voids numerical density stays relatively low, compared to martensite, due to the high ductility of the ferrite (low nucleation). High deformed states show big voids nucleation after  $\varepsilon = 1.6$ , while at the latest steps, coalescence leads to the fracture (after  $\varepsilon = 2$ ). The sum of contributions due to ferritic matrix and hard martensite islands suggest that a quantitative investigation has to be carried out on a reduced portion of material. The 200x200x200 voxel cubes, centred on minimal section have been chosen as representative of the more damaged portion of the sample. Figure 5 show a tomographic image of dual phase steel, where minimal section is recognisable. The sketch also shows the portion of material considered. The cubes have been processed by commercial software for 3D rendering. Bigger voids in tomographic images (marked in blue) are recognisable in the 3D rendering image. These images show the complete void morphology, and offer fundamental investigations on voids orientation.



Figure 5 – 200x200x200 voxel cubes in filtered data and 3D rendering

Preliminary quantitative analyses are shown in Figure 6. The plot show the number of voids per cubic millimetre as a function of strain for martensite and ferrite. The analysis has been carried out considering the cubes at the centre of the sample. Figure 6 demonstrate the martensite, due to the high nucleation rate, is likely to promote damage in dual phase steels, also according to the qualitative analysis.



Figure 6 – comparison between martensite and ferrite in the number of voids per cubic millimtre

## 5. CONCLUSION AND FURTHER RESEARCH

Using an in situ tensile test during X ray tomography experiments on a high strength steel, it has been shown in the present study that it is possible to qualify and quantify damage in 3D in the bulk of steels. Investigation methods have been

demonstrated to be valuable for qualitative analysis and show great potential for quantitative analysis, currently ongoing. Quantitative analysis in a central 200x200x200 voxel region will include the measurement of : the number of voids, the voids average diameter, the voids average and minimal distance, and their orientation. X ray tomography can be used for selecting voids by class of volume, and offer the chance of analysing nucleation and growth of each population of voids. Using the Bridgeman hypothesis on axisimmetrical samples, it will be possible to estimate the stress triaxiality T, the ratio between hydrostatic stress  $\sigma_m$  and Von Mises stress  $\sigma_{ea}$ , as shown by Eq. 2

$$T = \frac{\sigma_m}{\sigma_{eq}} \qquad \text{Eq.2}$$

Stress triaxiality is fundamental in modelling the growth of the equivalent radius (R), such as shown by the Rice and Tracey expression for the prediction of void growth [3] (Eq.3).

$$\frac{dR}{R} = 0.238 \exp\left(\frac{3}{2}T\right) \qquad \text{Eq.3}$$

The RT model can be successfully applied to the biggest voids in the volume considered. Also the X ray tomography represents the main experimental method for validating a growth model.

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