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# Characterization and modeling of void nucleation by interface decohesion in dual phase steels

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In situ tensile tests have been carried out during X-ray microtomography imaging of dual phase steels. Void nucleation has been quantified as a function of strain and triaxiality using the obtained three-dimensional images. The Argon criterion of decohesion has been used in a model for nucleation in the case where martensite plays the role of inclusions. This criterion has been modified to include the local stress field and the effect of kinematic hardening present in such a heterogeneous material.

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Ductile damage is characterized by a three-step process: first cavities nucleate, then grow until coalescence leads to ductile fracture. The first step of nucleation has been extensively studied and modeled. Void nucleation is often inhomogeneous, e.g., cavities appear at inclusions or at second phase particles [1]. In the latter case void formation occurs either by inclusion fracture, by separation of the inclusion/matrix interface or by cavitation near the inclusion.

Dual phase (DP) steels, which contain hard martensite islands embedded in a ductile ferritic matrix, are such a material promoting inhomogeneous nucleation. In DP steels the main nucleation mechanism is interface decohesion, as experimentally observed by Steinbrunner et al. [2] and Avramovic-Cingara et al. [3]. To model this debonding an energy criterion [1,4,5] is necessary for the creation of new surfaces and a stress criterion [6,7] or a strain criterion [8,9] is required to break the bonds. To combine the two criteria numerical models using cohesive zones have also been developed [10–12].

In order to be validated these models have to be compared with key experiments. Currently, X-ray absorption microtomography is one of the most reliable ways to obtain quantitative three-dimensional (3D) information on damage [13,14]. In the present paper, damage

in a DP steel has been studied by in situ tensile tests during X-ray microtomography imaging. Quantitative data has then been used to validate a model of void nucleation based on the Argon criterion.

The DP steel used for this study was cut from a 3 mm thick sheet obtained by hot rolling and thermal treatment (intercritical annealing following by water quenching). The chemical composition of the steel is given in Table 1 and its mechanical properties in Table 2. Image analysis of optical micrographs of polished surfaces showed that the steel contained about 11% martensite, in the form of islands.

Four axisymmetric specimens with a minimal diameter of 1 mm were machined from the original sheet. Two kinds of specimen shapes, inspired by Bron et al. [15], were cut: two smooth samples and two samples with a 1 mm radius notch. Each shape induced a different initial triaxiality. This allows us to study the effect of this important parameter on damage.

X-ray microtomography has been used in the present study to quantify damage during in situ tensile tests. The method can be used to image and quantify the microstructure of materials. Applications in the study of damage to ductile materials can be found in the literature [13,14,16]. The tomography set-up used is that located in the ID15 beam line at the European Synchrotron Radiation Facility (ESRF) in Grenoble, France (for more information, see Maire et al. [17]). Tomography acquisition was carried out with a voxel size of

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**Table 1.** Chemical composition of the DP steel studied (wt.%).

| C    | Mn   | Si   | Cr   | P, S   |
|------|------|------|------|--------|
| 0.08 | 0.80 | 0.23 | 0.68 | Traces |

**Table 2.** Mechanical properties in tension of the DP steel studied.

| $R_e$ (MPa) | $R_m$ (MPa) | $A_g$ (%) | $A$ (%) |
|-------------|-------------|-----------|---------|
| 366         | 603         | 17.7      | 26.6    |

$1.6 \mu\text{m}^3$ . With such a resolution the smallest observed voids had a diameter of almost  $2 \mu\text{m}$ . Smaller voids, not accounted for in the quantification, existed in the sample but may not play a major role in damage.

Fractured samples were subsequently polished down to their central plane and etched with a 2% nital solution. The samples were dipped in a solution of ethanol and placed into an ultrasound cleaner for 30 min after polishing to eliminate the possibility of fragments obscuring the cavities. Light optical micrographs were then acquired in order to observe the nucleation sites.

The same procedure as that presented in Maire et al. [17] was used in this study. Initial volumes were median filtered and simply thresholded to differentiate material from voids. The minimal section area  $S$  was then measured in order to calculate the local strain  $\varepsilon_{\text{loc}}$  at each step using the following equation:

$$\varepsilon_{\text{loc}} = \ln \left( \frac{S_0}{S} \right) \quad (1)$$

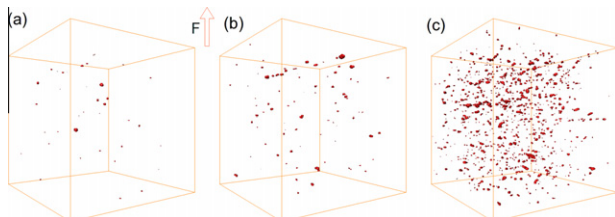
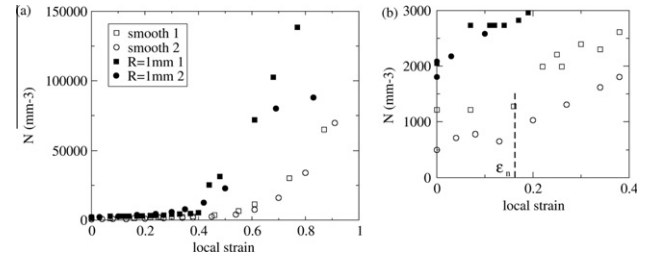
$S_0$  being the initial section of the sample. This equation implies that the effect of porosity in the volume change of the sample is neglected in our analysis.

The curvature radius  $R_{\text{notch}}$  was also measured in order to determine the stress triaxiality  $T$  using the Bridgman formula, Eq. (2) [18]

$$T = \frac{1}{3} + \ln \left( 1 + \frac{a}{2R_{\text{notch}}} \right) \quad (2)$$

$a$  being the radius of the minimal section.

As in Maire et al. [17], damage was only quantified in the central part of the tensile specimen. This sub-region was chosen to be a cubic volume of  $300 \mu\text{m}^3$ . Figure 1 shows the studied sub-region of a smooth specimen of DP steel at several steps of deformation. This qualitative figure clearly shows that the number of cavities increases (nucleation) and that the size of the nucleated cavities also increases (growth) with increasing strain. It is noticeable in this image that nucleation is a quantita-

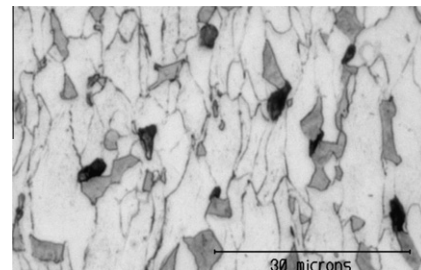
**Figure 1.** 3D views of damage at the center of a smooth strained specimen at various steps of deformation: (a)  $\varepsilon_{\text{loc}} = 0.05$ ; (b)  $\varepsilon_{\text{loc}} = 0.4$ ; (c)  $\varepsilon_{\text{loc}} = 0.75$ . The side of the cube is about  $300 \mu\text{m}$ .**Figure 2.** Evolution of  $N$ , the number of cavities per cubic mm, in the four studied samples measured during the in situ tensile tests. (a) Global view; (b) focus on the low strain region of (a).

tively important part of the damage progression in these materials, as already shown in Maire et al. [17]. Each pore of the volume was then labeled using a dedicated image processing plug-in implemented in ImageJ [19] freeware. The labeling plug-in uses a binary image as input. It simply detects 3D clusters of connected voxels and gives a label to each separate connected cluster of voxels. The void density was calculated as the number of cavities per cubic millimeter in the sub-volume. Figure 2 shows the evolution of  $N$ , the number of voids per unit volume (expressed per cubic mm), in several smooth and notched DP steel samples. A very low level of porosity (0.03%) could be detected before the tensile test, possibly due to the fabrication process. The experimental results show that triaxiality had a straightforward impact on the nucleation kinetics: void nucleation occurred earlier in notched samples, inducing higher triaxiality, than in smooth samples.

Optical micrographs performed on the fractured specimens and given in Figure 3 show that most cavities were localized between the ferritic matrix and martensite islands and thus nucleate by decohesion of the ferrite/martensite interface, as previously observed [2,3].

As demonstrated by Tanaka et al. [4], the energy criterion necessary for the creation of new surfaces at the inclusion/matrix interface is satisfied at the onset of plastic deformation in materials containing inclusions bigger than about  $25 \text{ nm}$  in diameter. Only a stress criterion will therefore be used to model interface decohesion in DP steels. The Argon criterion [6] is a critical stress criterion: void nucleation occurs when a critical stress state necessary to interface decohesion is reached in the material. This stress state involves a contribution of the hydrostatic stress  $\sigma_m$  and the equivalent stress  $\sigma_{\text{eq}}$

$$\sigma_{\text{eq}} + \sigma_m = \sigma_C \quad (3)$$

**Figure 3.** Micrograph of a fractured specimen. Voids appear in black, ferrite in light gray and martensite in dark gray.

where  $\sigma_C$  is the interface strength, e.g., the maximum shear stress that the interface can support without breaking.

The interest in using the Argon criterion lies in the fact that it accounts for the triaxiality  $T$  ( $T$  being the ratio between  $\sigma_{eq}$  and  $\sigma_m$ )

$$T = \frac{\sigma_{eq}}{\sigma_m} \quad (4)$$

Combining Eqs. (3) and (4), the criterion is expressed as:

$$\sigma_{eq}(1 + T) = \sigma_C \quad (5)$$

In the original Argon criterion the triaxiality used is the macroscopic triaxiality. However, decohesion is a local phenomenon which occurs at the interface of ferrite and martensite islands. In Helbert et al. [20] it was demonstrated that local triaxiality is higher at the interface because of the kinematic hardening  $X$  generated by the difference in mechanical behaviors of the two phases. In DP steels the difference between the mechanical behavior of ferrite and martensite is quite high. Therefore, it would be better to use the local triaxiality at the interface  $T_{loc}$ .  $T_{loc}$  can be estimated using the following expression from Helbert et al. [20]:

$$T_{loc} = T \left( \frac{\sigma_{eq}}{\sigma_{eq} - X} \right) \quad (6)$$

The modified expression of the Argon criterion to obtain local decohesion then becomes:

$$\sigma_{eq} \left( 1 + T \left( \frac{\sigma_{eq}}{\sigma_{eq} - X} \right) \right) = \sigma_C \quad (7)$$

The left side of the equation in this local version of the Argon criterion is termed  $\chi$  below.

By calculating the value of  $\chi$  at the nucleation strain  $\varepsilon_n$ , an average value of  $\sigma_C$  can be estimated in the case of the ferrite/martensite interface.  $\varepsilon_n$  is determined from the cavity measurements on the smooth samples. The value is taken at the point when the void density started to increase. Focusing on the low strain region in Figure 2b, this shows a value for nucleation strain of 0.18. Table 3 gives the values for some mechanical parameters needed to determine the value of  $\sigma_C$  for this particular value of strain.  $\sigma_{eq}$  is taken from the experimental true stress tensile curve and  $T$  is calculated with the Bridgman formula [18]. The value of the kinematic part of hardening  $X$  is estimated from the formula given by Allain and Bouaziz [21] and expressed as a function of the respective hardness of ferrite  $HV_f$  and martensite  $HV_m$ :

$$X = 3(1 - f_m)f_m|HV_m - HV_f| \quad (8)$$

where  $f_m$  is the volume fraction of martensite. Assuming that all the carbon present in the DP steel studied is in the martensitic phase, the carbon content of the martensite was 0.73 wt.%. Based on the work by Grange on the prediction of martensite hardness [22] a  $HV_m$  va-

**Table 4.** Value of ferrite particle interface strength.

| Particle                      | Interface strength (MPa) | Ref.    |
|-------------------------------|--------------------------|---------|
| Y <sub>2</sub> O <sub>3</sub> | 1000–1600                | [23]    |
| MnS                           | 1100–1400                | [24]    |
| Fe <sub>3</sub> C             | 1200–2000                | [25,26] |

lue of about 830 MPa was assumed. The hardness of the ferritic phase was taken as 150 MPa. The interface strength found here for this particular value of strain when nucleation started to increase was about 1200 MPa. A compilation of values for the strength of the interface between other particles and ferrite found by different authors is listed in Table 4. The order of magnitude is the same as that calculated using our approach, the case of ferrite/martensite being situated in the lower range of these values. Note that our estimation is valid for the very beginning of nucleation (i.e., when the number of voids started to increase as seen in the tomography images). The other values were obtained with other methods and probably other definitions, however, a direct comparison is not the aim of the table.

As seen by Avramovic-Cingara et al. [3], void nucleation in DP steels occurred during the entire deformation process, i.e., each single interface probably exhibits a different value of  $\varepsilon_n$  and each interface is possibly subjected to one of a scatter of values of  $\chi$ . Interface decohesion is thus a progressive phenomenon, starting for a strain of 0.18 but continuing after this value of strain, and evolution of the cavity density has to be modeled as a function of the local strain. Figure 2 shows two different nucleation regimes at low and high strains. Firstly, the number of cavities increases slowly and linearly. In a second regime many voids appear exponentially. This experimental observation led us to propose the following empirical equation based on the local criterion of decohesion and involving the parameters  $\chi$  and  $\sigma_C$

$$\frac{dN}{d\varepsilon_{loc}} = A \frac{\chi}{\sigma_C} \left( 1 + \frac{N}{N_0} \right) \quad (9)$$

$A$  and  $N_0$  being two constants (expressed in the same unit as  $N$ , for instance, mm<sup>-3</sup>).

The two extreme regimes are well described by this empirical expression. When  $N \ll N_0$  the following approximation is applicable:

$$\frac{dN}{d\varepsilon_{loc}} \approx A \frac{\chi}{\sigma_C} \quad (10)$$

Interface decohesion is then linearly controlled by the local stress  $\chi$ , which increases with the applied strain. In the second step, when  $N \gg N_0$  the approximation becomes:

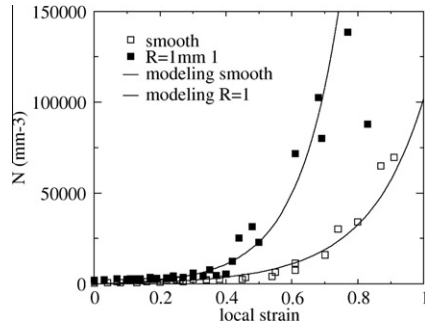
$$\frac{dN}{d\varepsilon_{loc}} \approx A \frac{\chi}{\sigma_C} \frac{N}{N_0} \quad (11)$$

The evolution rate of  $N$  with strain is proposed to depend on  $N$  itself, thus describing a self-catalytic effect, and thus the exponential acceleration of the number of cavities.

We now have a means to integrate the value of  $N$ , by accounting for local triaxiality at the interface. Assessment of the model was first done using experimental

**Table 3.** Estimated mechanical parameters at the nucleation strain.

| $\varepsilon_n$ | $T$  | $\sigma_{eq}$ (MPa) | $X$ (MPa) | $\sigma_C$ (MPa) |
|-----------------|------|---------------------|-----------|------------------|
| 0.18            | 0.35 | 816                 | 199       | 1194             |



**Figure 4.** Comparison of the prediction of the nucleation model and experimental data.

data from smooth specimens. The values of  $A$  and  $N_0$  giving the best fit between the model and experimental data for the smooth sample were  $A = 4500 \text{ mm}^{-3}$  and  $N_0 = 1250 \text{ mm}^{-3}$ . These values, when applied to the notched samples, also showed satisfactory agreement, as shown in Figure 4. This validates the use of a local value for triaxiality as the driving force in the interface fracture criterion.

Using in situ tensile tests during X-ray tomography, the present study has shown that it is possible to obtain quantitative information about damage, in particular about void nucleation. With regard to the sites of nucleation, optical micrographs of fractured samples have shown that most cavities appear by decohesion of the ferrite/martensite interface. A value for the critical interface strength (1200 MPa) has been estimated for the very first nucleation events. Evolution of the void density was then modeled according to an analytical approach based on a local version of the Argon decohesion criterion and accounting for triaxiality. The model was fitted to the experimental data for the smooth samples. The parameters identified were then applied to the notched samples, also leading to satisfactory agreement with the predicted evolution of the number of nucleated cavities. Some improvements to the present approach can be foreseen, particularly concerning the value of the interface strength of DP steels. This strength probably depends on the carbon content in the martensite and on eventual tempering. These effects have to be investigated in more detail before being modeled.

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