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# Stress distribution correlated with damage in duplex stainless steel studied by synchrotron diffraction during plastic necking

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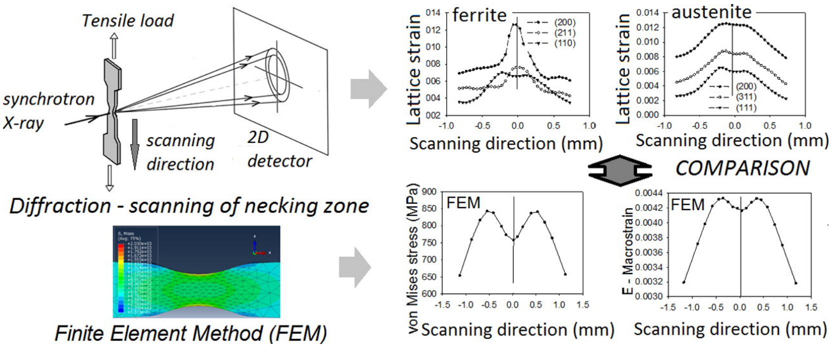
## HIGHLIGHTS

- Relaxation of stress in ferrite indicates damage initiation in this phase for a large deformation of duplex steel.
- The stress relaxation in ferrite depends on the grain orientation and position in the neck.
- Heterogeneous lattice strains and three-axial heterogeneous stresses are present in the necking zone.
- The softening/damage process in ferrite depends on the value of equivalent von Mises stress.

## GRAPHICAL ABSTRACT



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## ABSTRACT

The goal of this work was the determination of lattice strains distribution in two phases of duplex steel during plastic necking. Subsequently, the stress heterogeneity in the neck was studied in order to determine the reason for the damage initiation and to verify the hypothesis that the damage begins in the ferritic phase. To do this, X-ray synchrotron radiation was used to scan the 'in situ' variation of the interplanar spacings along the necking zone for samples subjected to tensile loading. A self-consistent model and FEM simulation were applied for the experimental data interpretation.

It was found that for advanced necking the phase lattice strains, especially those measured at some distance from the neck centre, show a large inversion of the loads localised in both phases compared to the undamaged state (the lattice strains in the ferrite become smaller than in the austenite). This effect indicates stress relaxation in the ferrite which is connected with the damage phenomenon. Correlation of the experimental results with the modelling shows that the value of von Mises stress is responsible for the initiation of the ferritic phase softening.

## 1. Introduction

Duplex stainless steel was discovered by Hochmann [1] during intergranular corrosion tests. This type of steel combines an excellent

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resistance to corrosion with high mechanical strength due to the high content of chromium and nitrogen. The duplex steel studied in this work consists of ferrite (α phase) having a bcc crystal structure and austenite (γ phase) exhibiting an fcc structure. Both phases possess good corrosion resistance. While the ferritic phase increases the value of yield stress, the austenitic phase improves the ductile response of the material.

Several studies of cast duplex stainless steel used in the cooling system of a nuclear power plant have been published [2,3]. This steel is subjected to a slow thermal ageing at a working temperature of 320 °C, which causes an embrittlement of the ferritic phase [4,5]. It is well-known that at temperatures below 475 °C, the spinodal decomposition of ferrite occurs [6–8].

During the deformation of duplex steel, the plastic strains are homogenous in the austenitic and more localised in the ferritic phase, where fewer slips, but with a higher magnitude, are observed [2,9–11]. SEM measurements performed for cyclic loadings show that the slip systems in the austenitic phase are activated first i.e. before the glides in the ferrite [11]. In the case of the aged duplex steel, Bugat et al. [2] observed the slip lines in the ferrite to be tortuous (following the {112}⟨111⟩ slip system and twinning along the same slip systems).

Due to the above-mentioned mechanisms, the damage process in aged duplex stainless steel is initiated mainly in ferrite, where cleavage micro cracks perpendicular to the loading direction have been shown in previous works [2,3,10]. The damage appears mainly in the regions where the strain incompatibles are high (double or triple grain boundaries in the austenite) and/or in the intersection of the strain modes (slipping and twinning in the ferrite). The nucleation of cleavage cracks is a continuous and stable phenomenon that accelerates with the strain. The study of damage in polycrystalline materials is usually based on a direct observation of the cracks' initiation and evolution using electron microscopy [12–14] or X-ray tomography [14]. Using electron microscopy (EBSD, TEM and SEM techniques) the initiation of cracks due to a cyclic loading of duplex steel was seen in the ferritic phase [12,14]. Recently, the effect of significant lattice strain release in damaged ferrite grains was confirmed by Istomin et al. [13] using X-ray synchrotron diffraction.

To observe the mechanical behaviour inside both phases during the damage process, diffraction measurements combined with an in situ tensile test were performed [15–25]. The main advantage of the diffraction methods is that the measurements are performed selectively only for the crystallites contributing to the measured diffraction peak. When several phases are present in the sample, measurements of a separate diffraction peak allows for investigating the behaviour of each phase independently [15–25]. A comparison of the diffraction data with micromechanical models is very convenient for the study of elastoplastic properties at micro and macro scales. Both selectivity and scale considerations determine the use of diffraction technique in the present study. An analysis of the experimental data using model predictions enables understanding of the physical phenomena which occur during a sample's deformation. Moreover, the micro and macro parameters of the elastoplastic deformation can be experimentally identified.

The damage processes initiated on slip planes or particular crystallographic planes, as suggested in many works [3,26–29], may be responsible for the relaxation of the grain stresses, depending on the orientation of the crystallographic lattice. This effect was also observed by measuring lattice strains in both phases of duplex steel using the neutron diffraction method [17,20].

In this work, the effect of stress partition between phases inside and outside the necking area in plastically deformed duplex steel is studied for the first time using synchrotron radiation. An interpretation of the results was performed with help of the Finite Element Method (FEM) and elastoplastic self-consistent modelling. The FEM simulation was applied to determine the influence of the neck shape on the stress distribution, while the self-consistent model was used to predict elastoplastic deformation of the undamaged state of the material and to show the difference between this state and the behaviour of the damaged material. In particular, a tendency of lattice strains evolution was studied and then compared with the experimental results obtained for the deformation neck.

The present work is a continuation of our previous investigation [17,20] in which the damage process occurring in the neck was studied using neutron diffraction. Previously, the measurements of lattice strains were performed for both phases and compared with a self-consistent model accounting for the damage process. It was found that relaxation of stresses in the ferritic phase and the load transfer to the austenite occurred in the deformation neck. The observed phenomenon was explained by a model, but due to relatively large gauge volume the stress/strain heterogeneity was not studied. In the previous study [29] the preliminary results of synchrotron measurements before the sample fracture under the applied load were also presented, but the strains heterogeneity in the neck was not investigated.

The present experiment was performed using synchrotron diffraction during a tensile test for which the sample strain was controlled. Due to a small gauge volume enabling high spatial resolution of the measurements, the lattice strains were scanned along the deformation neck created during the tensile test. The main goal of this work was to observe the stress partitioning between the phases at various positions in the necking zone. As a result, the relaxation of the stress in the ferritic phase was studied and the heterogeneity of the damage within the deformation neck was described.

## 2. Experimental

### 2.1. Material characterisation

The present study examined an austenitic-ferritic duplex stainless steel UR45N, which was previously investigated in [17,29]. The microstructure of the studied material is constituted of a ferritic phase and austenitic phase in approximately equal volume proportions (50% austenite and 50% ferrite). EBSD maps show the elongated grains of the austenite (γ phase) evenly embedded in the ferritic (α phase) matrix (see [17,29]). The grains of the austenite are divided into smaller crystallites with different orientations, while the grains of the ferrite possess nearly the same crystal orientation resulting from the rolling process [29]. The Orientation Distribution Function (ODF, shown in [30]) of each phase was determined from the experimental pole figures {110}, {200}, {211} and {111}, {200}, {220} measured respectively for the ferrite and the austenite, using Cr radiation on a Seifert four circles diffractometer [29].

### 2.2. Measurement configuration and treatment of the 2D diffraction patterns

The tensile tests combined with in-situ X-ray synchrotron diffraction measurements were performed in a transmission mode on the beamline ID15B at ERSF (Grenoble, France), using a monochromatic X-ray radiation with the wavelength $\lambda = 0.14256$ Å, which was determined from a preliminary calibration performed on the $CeO_2$ reference powder [29]. The configuration of the experimental setup is shown in Fig. 1a. The diffraction pattern, recorded by a Thales 2D Pixium 4700 CCD detector [31], is presented in Fig. 1b. A Pico1 detector was placed in front of the outgoing beam in order to measure the intensity of the beam passing straight through the sample (Fig. 1a). The measurements were performed using a beam size of 100 μm × 100 μm for 'dog-bone' shape samples having a cross section of 1.5 mm × 1.5 mm in the region studied by diffraction. The samples were named respectively 'sample TD' and 'sample ND'. Sample TD was manufactured so that the transverse direction (TD) was perpendicular to the sample surface, which means that the incident X-ray beam was parallel to the TD direction during the tensile test [29]. Sample ND was machined with the normal direction (ND) perpendicular to its surface, allowing measurement with