Analysis by X-ray diffraction of the mechanical behaviour of austenitic and ferritic phases of a duplex stainless steel

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ABSTRACT

This paper deals with the X-ray diffraction characterisation of the mechanical behavior of a cast duplex stainless steel, containing 30% ferrite and 70% austenite. The structure of solidification leads a coarse grain material with grain size of the order of millimeters. The aim of the study is to identify the mechanical behavior for both phases by X-ray diffraction and to correlate it to the microstructure. The classical sin^2ψ method for stress determination can not be applied to this material because of the large grains single size. The stresses are determined using a method resulting from an adaptation of the single crystal measurement method to large grains materials. So stress are determined using a method resulting from an adaptation of the single crystal measurement method to coarse grain materials.

The measurement in each phase has successfully be applied to follow the stress state evolution during an in-situ tensile test. Three grains, with different crystallographic orientations were studied. For each one, the stress tensor was determined in the two phases under different macroscopic loading in elastic and plastic domains. For all grains, stress state in the ferritic phase is higher than the applied macroscopic stress and compared to the austenitic one where the stress is lower. This can be explained by ageing embrittlement of the ferrite which makes it much harder than the austenite. The important heterogeneity stress level is getting worse because of the crystallographic orientation of each grain.

At each loading, micrographic observation are made to correlate the mechanical state determined by X-ray diffraction with the microstructure. Visible glides and cracks are noted and related to the stress state. This coupling of methods has been applied to identify the yield stress of each phase and the critical stress that leads to cracking of ferrite. The yield stresses so determined are in agreement with those deduced from the θ-2θ peak broadening analysis.

INTRODUCTION

Cast duplex stainless steels, composed of austenite and ferrite phases are frequently used in the nuclear industry, because of the mechanical properties of this material due to the presence of the ferritic phase. In particular some components of pressurised water reactors such as pipes, elbows... are manufactured with this material. In use, 280°C to 320°C water is flowing in the primary circuit. In that case, ferrite of the duplex material undergoes to the well known phenomenon of 475°C embrittlement of ferritic stainless steels [Fischer & al, 53] [Miller & al, 86] [Bonnet & al, 90].

Many studies have been done to characterise behavior of these materials [Charles, 91]. Experimental and numerical approaches were performed to define macroscopic mechanical properties [Besson & al, 95]. In situ tensile tests in the SEM (Scanning Electron Microscopy) have shown that local strain and damage mechanisms are heterogeneous at the scale of phases and grains, above all in the case of damaging. Crystallographic orientation of the grain has a major influence on the damaging for this material. A polycrystalline approach can take into account this aspect, by consideration of the behavior of each phase at the microscopic level.
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More generally, polycrystalline representations integrate the behavior of the material components at different scales (microscopic, mesoscopic) with the aim to reproduce its mechanical properties at the macroscopic level. Different order stresses which are related to this different scales can be analysed by X-ray diffraction. These are represented in figure 1.

First order stresses concern the whole polycrystalline material. The large number of grains in the diffracting volume is related to macroscopic mechanical state. First order stresses are an average of second order stresses which correspond to the stress in each grain (mesoscopic scale). Second order stresses are also an average of third order stresses which correspond to the stresses fluctuation into a grain due to the heterogeneous distribution of dislocations, precipitations or intragranular phases (microscopic scale).

For a better understanding of the polycrystalline material mechanical response, mesoscopic mechanical behavior and microscopic parameters must be clearly identified. The mechanical state can be determined using X-ray diffraction technique which enables us to measure strains in each phase separately. These mechanical states are associated to metallurgical mechanisms by coupling X-ray diffraction and optical observations. The mechanical analysis by X-ray diffraction at the grain scale is possible because the studied material exhibits coarse grains of up to one millimeter. In this case, only few crystals are irradiated by the incident X-ray beam. An adaptation of the single crystal measurement method to large grains materials is used [Gergaud & al, 97]. In this case, determined stresses correspond to second order stresses.

**STUDIED MATERIAL**

In the present work, an austeno-ferritic stainless steel containing volumic fraction from 30 % ferrite and 70 % austenite is investigated. Optical micrography (fig. 2) shows coarse two phase grains (a, b, c...). These two phases are morphologically connected each other, corresponding to a Widmanstätten microstructure. Figure 3, where pole figures performed in the same grain, of austenite and ferrite are presented, confirms the single crystal appearance of our material. Effectively, pole figure analysis shows that a single crystal of ferrite and one of austenite with some light disorientation.
Moreover, in each grain, both phases are linked by Kurjumov Sachs crystallographic orientation relationships: \{111\}_γ // \{110\}_α and \{110\}_γ // \{111\}_α.

It is easy to notice in figure 3, where \{111\}_γ austenite pole and \{110\}_α ferrite pole are represented, that the pole figures are identical, so the relationships are verified. This is caused by the elaboration mode, where austenite appears at the solid state to the detriment of ferrite. For the same reason, austenite presents some mosaicity.

![Figure 3: Pole figure of each phase for the same grain](image)

The material studied was treated at 400°C for 1000 hours in order to reproduce the natural ageing. This treatment lead to a ferrite embrittlement whose microhardness has increased from about 350 Hv for the received material to 700-800 Hv. This important difference of mechanical properties between ferrite and austenite must be considered. It leads to heterogeneous distribution stress in the two phases during solicitation process.

**SINGLE CRYSTAL STRESS MEASUREMENT**

**Principle**

The classical sin²ψ method cannot be applied because of the grain size in comparison with the beam size. A specific analysis is used; the adaptation of the single crystal measurement to large grain material has been initiated by [Reimers, 89] and then applied by [Gergaud & al, 97] and developed in the case of a two phase materials [Lebrun & al, 97]. The measurement is carried out in two parts:

- Orientation of the crystal to determine the orientation matrix in the laboratory axis. The orientation matrix is deduced from the analysis of one pole figure. The \{hkl\}<uvw> orientation of one grain provides the position of any (hkl) planes of this grain, which is useful for the second part of the measurement. Since the precise determination of the position of the plane (hkl) is not always easy, some iterative process is used for this [Eberl, 98].

- Measurements of the 2θ shift of different (hkl) planes, which lead to the determination of the strained metric tensor. In order to minimise the influence of diffractometer misalignment, it is interesting for stress determination to use (hkl) planes at high 2θ angles and to try to use only a single \{hkl\} family. The calculation of the strains requires to know accurately the lattice parameter of the unstrained material. It is almost impossible indeed to measure experimentally the parameter of the crystal considered as
totally unstrained because any treatment made to relax the stress is bound to induce physical and chemical changes which could modify the lattice parameter. However, as X-ray diffraction only concerns a very thin layer of matter near the surface, it is reasonable to assume that no stresses are applied on the free surface sample, i.e. $\varepsilon_{33}=0$. This assumption allows a direct determination of the lattice parameter of the stress free cubic crystal. This point will be discussed later.

**Practical aspects**

The single crystal orientation is deduced from pole figure analysis. The analysis of a pole figure without ambiguity requires the use of low multiplicity family (but with enough planes), like in our case {022} family.

For stress determination, diffraction experiments were performed on a four circle diffractometer Seifert or Philips. With the first one, the radiation MoK$_\alpha$ is used, which permits working with the {246}_α plan family for the ferritic phase ($2\theta=136^\circ$) and {139}_γ for the austenitic one ($2\theta=140^\circ$). And with the other, the radiation CuK$_\beta$ is used because it allows to work with the {123}_α family for ferrite ($2\theta=129^\circ$), {024}_γ and {224}_γ for austenite ($2\theta=120^\circ$ and $142^\circ$ respectively). High multiplicity families were choose in order to analyse a maximum of planes (14 to 17 plans with $\psi$ lower than $70^\circ$). The high number of strain measurement allows a good information all over the space.

**EXPERIMENTAL RESULTS AND DISCUSSION**

In situ uniaxial tensile tests have been performed. The results of two samples will be presented. Figure 4 gives the macroscopic loading applied on sample one. A load cell allows the measurements of the force applied on the sample and thus knowing sample geometry, one can determine the applied macroscopic stress. Measurement during in situ loading were performed by fixing the small tensile device on the sample holder of the diffractometer. At each loading step, stresses are determined for both phases in three grains, their orientations are given in table 1 (for the sample 1). The loading process is in the elastic and plastic range.

**Table 1 : Crystallographic orientation of the studied grains**

<table>
<thead>
<tr>
<th>Grain</th>
<th>Austenite</th>
<th>Ferrite</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>$h k l$</td>
<td>$u v w$</td>
</tr>
<tr>
<td>Grain 1</td>
<td>6 4 7</td>
<td>0 2 -1</td>
</tr>
<tr>
<td>Grain 2</td>
<td>1 3 1</td>
<td>-3 2 -3</td>
</tr>
<tr>
<td>Grain 3</td>
<td>0 1 0</td>
<td>-1 0 0</td>
</tr>
</tbody>
</table>

**Figure 4 : Sample loading**
As already stated, all stresses were calculated considering that the normal stress component to the sample ($\sigma_{33}$) is equal to 0. This assumption is justified by this kind of experiment where X-ray penetration is only a few µm.

The calculated free lattice parameters are reported in figure 5 to verify this hypothesis. A variation of $10^{-4}$ Å induces a variation of about 15 MPa on components $\sigma_{33}$ and thus on the other $\sigma_{ii}$ terms.

As figure 5 shows for the two phases, the calculated free lattice parameter is almost constant; and we can observe only the experimental scatter. We can also interest to the $\sigma_{33}$ component evolution considering the free lattice parameter determined at the first loading and introduced for all other steps (figure 6).

This figure shows that the considered hypothesis for the stress calculation is valuable. Even if the free lattice parameter is unknown with a good accuracy, the component $\sigma_{33}$ is constant for each loading and for mechanical equilibrium reasons can not be far from zero.

**Second order stresses analysis**

Figure 7 shows the stress distribution of component $\sigma_{11}$ corresponding to the tensile direction. The macroscopic loading is also represented for comparison. The stress level in ferrite is higher than the macroscopic loading and lower in austenite. This is due to the difference of mechanical properties of ferrite and austenite. Ferrite is harder and more fragile than austenite which has a ductile behavior, even for the unaged material.
From this figure, it appears that one parameter that determines the crystal behavior is the crystallographic orientation. In each phase, a different stress state evolution can be observed for the three considered crystallographic orientations. This is normally due to the Schmid law (see next paragraph).

Let's now analyze each crystal orientation (fig. 8). A similar behavior is observed between ferrite and the macroscopic loading.

Actually, the system of loads in ferrite is equivalent to the applied macroscopic one, i.e. in our case an almost uniaxial tensile loading. This remark is supported by calculating the triaxiality rate, as seen in figure 9. For ferrite in the three studied grains, the triaxiality rate is constant and about 0.3-0.4 which is close to 0.33 value corresponding to the theoretical triaxiality rate in the case of an uniaxial tensile.
In the case of austenite, the loading is more complex: the orientation of the crystal has a major effect on its response to the macroscopic loading. As we can see, grain 1 has a biaxial stress state, grain 3 has an important shear stress component. The grain 2 is in a configuration of a rigid behavior.

The complex loading observed in austenite is explained by the fact that this phase is the soft one and has to accommodate the local strains.

**Characterisation of the parameter for the modelisation of the crystal behavior**

Coupling of techniques such as optical observations and X-ray diffraction allows the correlation of the different mechanical states evolution with plasticity and damage mechanisms identification. So, for each loading step, optical observations were done to note the apparition of glide and crack.

**Plastic activity**

The different steps which have been observed in the mechanical state evolution are:

- a first step where austenite and ferrite have an elastic behavior,
- in the second step, ferrite is still elastic and austenite is plastic. We can effectively observe, as shows for example figure 10 in the case of the grain 1, glide from active slip system,
- then, both phases are plastic. Glide is observed in the two phases, and in particular, pencil glide in ferrite (bcc).
This metallurgical characterisation is illustrated in figure 10. This approach permits one to
determine the beginning of plasticity and to correlate it with the determined stress state in order to
deduce the yield stress of the crystal. For that, we consider crystalline plasticity is determined by
the Schmid criteria. Plasticity occurs when the resolved shear stress $\tau^s$ is equal to the critical shear
stress $\tau^c$ in one or more systems. The resolved shear stress $\tau^s$ is defined by,

$$\tau^s = R^s_{ij} \sigma_{ij},$$

where $R^s_{ij} = \frac{1}{2} (n_i^s m_j^s + n_j^s m_i^s)$, $n^s$ and $m^s$ are the plane and the direction of the slip system $s$.

The critical shear stress $\tau^c$ is one of the major parameters in polycrystalline modelisation that uses
this kind of plasticity description. This parameter is determined using the coupling of techniques:
as we can see in figure 10, plastic activity appears at the $4^{th}$ loading in the austenite phase of grain
1. The stress determined at this loading is then projected on the glide plane and direction.

Plastic activity starting point can also be determined by qualitative study of the peak broadening
evolution.

As we can see in figure 11, the peak breadth is constant in the elastic domain and increases with
the plastic activity which allows one to identify the loading that causes the plastification of the
crystal.

From this figure, we deduce the yield stress is given by the stress state at the $4^{th}$ loading. This
is in good agreement with the micrographical observations.

**Damage**

The elastoplastic flow is followed by damaging stage. Material ageing induces the initiation of
microcracks in the ferrite leading to its rupture by cleavage. The final rupture occurs by cracks
coalescence through the austenitic phase.
This stage is illustrated in figure 12; the crack initiation occurs on the grain whose orientation is not far from \(\{3 1 9\}<0 1 0>\). These results concern a second sample, in which we follow the evolution of residual stress in some grains after different prestrain. We can see that cracks are normal to the tensile direction, which confirms that it is a cleavage crack. The residual stress has been determined in the ferrite of this damaged grain for 5 preloadings. They are taken back in figure 13. We can observe that the residual stress decreases after 0.8% macroscopic strain. This stress relaxation is directly related to the crack apparition.

CONCLUSION

Stress single crystal measurements were successfully applied to characterise the stress state in several grains of a duplex stainless steel. It was observed that during the elastoplastic tensile test, ferrite was submitted to an uniaxial stress state and austenite which is the soft phase and accommodates the local strains, is in a complex stress state that depends on its crystallographic orientation. The coupling of techniques allows to link the stress state to the metallurgical state, which permits identifying microscopic parameters that characterise the mechanical behavior of the crystal such as glide and cracks.

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