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Stress Distribution and Cleavage Analysis in a 16MnNiMo5 Bainitic Steel

X-ray Diffraction and Multiscale Polycrystalline Modelling

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Many tensile tests have been realized on the 16MnNiMo5 bainitic pressure vessel steel at low temperatures [-196°C , -60°C]. The damaging processes (ductile/fragile) are observed with a scanning electron microscope (SEM), while X-ray diffraction (XRD) is used to determine the internal/residual stresses within the ferritic phase during loading (*in-situ*) and after unloading: stress states are lower in ferrite than in the bulk material due to cementite particles, the difference never exceeding 150 MPa. A polycrystalline modelling with a two-level homogenisation is also developed concurrently with the experimental characterization. It correctly reproduces the stress distribution in each phase, the intergranular strain heterogeneity as well as the macroscopic fracture stress and strain in relation to temperature, considering a constant number of grains (7%) reaching an experimentally identified crystallographic criterion of cleavage.

KEY WORDS: Internal stresses; $\varepsilon_{\phi\psi}$ intergranular strains; Polycrystalline modelling; Fracture criterion

1. Introduction

Nuclear energy has developed tremendously in the last fifty years, thanks to the great progress in mechanics of materials (control and measuring systems) and the capital improvement of numerical technology. However, its safety depends on the thorough knowledge of the materials involved.

The 16MnNiMo5 bainitic pressure vessel steel here studied is mainly used at high temperatures, but irradiation causes a temper embrittlement. It becomes necessary therefore to characterize its mechanical properties and to define the mechanisms leading to crack initiation and propagation at low temperatures, in order to predict its behaviour and its service life.

A series of tensile tests is thus performed at low temperatures: the damaging processes are observed with a SEM, while XRD is used to determine the internal stress distribution within the material as well as the heterogeneity of the intergranular strains^[1]. All these measurements supply a polycrystalline model of behaviour and damage with data, so that it can be compared with experimental results and validated.

2. Experimental Procedures

2.1 Material

The studied material is a 16MnNiMo5 bainitic pressure vessel steel (ferritic matrix containing about 5% of cementite precipitates), which has undergone several heat treatments leading to a microstructure composed of many packets within former austenite grains (Fig.1). The metallographic examination also reveals the presence of a few spherical and elongated manganese sulphide inclusions (MnS: less than 1%).

2.2 Tensile tests

Many sequenced and *in-situ* tensile tests are thus

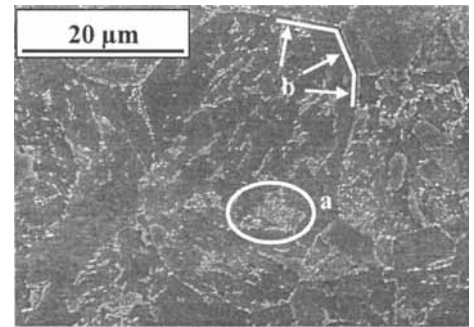


Fig.1 SEM micrograph showing the microstructure of the 16MnNiMo5 bainitic steel
a-ferrite with cementite precipitates,
b-former austenitic grain boundaries

realized between -196°C and -60°C using a small tensile machine equipped with a temperature regulating system, which is placed right into the SEM and onto a diffractometer. At each step of the loading, the specimens tested are observed with the SEM, to determine the evolution of the damaging processes (slip lines, crack initiation) and in particular the nature of the sites responsible for cleavage brittle fracture.

XRD enables to determine the stress state distribution in each phase of the material ($\sin^2\psi$ -method) in relation to temperature, during loading and after unloading. The stress analyses as well as the $\varepsilon_{\phi\psi} = f(\sin^2\psi)$ strain measurements^[1] (strains of the diffracting planes: the ψ angle characterizes the crystallographic orientation of the grains) are realized in ferrite with a linear detector (Set-X), considering the $\{211\}$ planes in the tensile direction. The volume fraction of cementite being too small to take some measures, the values of the internal stresses in this phase (I order stresses) are deduced by using the following hypothesis on the macroscopic scale:

$$\sigma^I = f_{\text{ferrite}} \cdot \sigma_{\text{ferrite}}^I + f_{\text{cementite}} \cdot \sigma_{\text{cementite}}^I$$

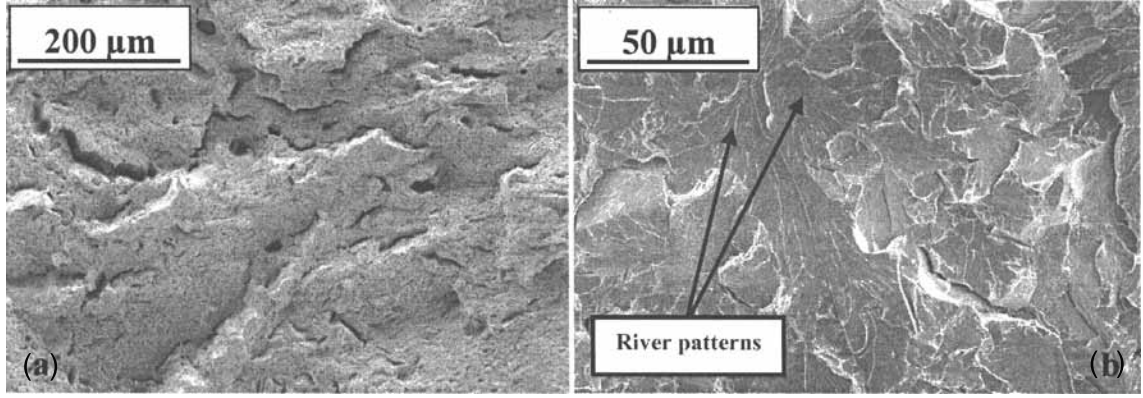


Fig.2 Fracture mechanisms of the 16MnNiMo5 steel (a) ductile fracture at -60°C , (b) brittle fracture at -196°C

3. Results and Discussion

3.1 Fracture mechanisms

The influence of temperature is very important, since the fracture mechanisms depend on it: indeed, during testing, the fracture is ductile at -60°C (plastic strain, microvoids around particles) and becomes brittle below -150°C , with many cleavage microcracks (Fig.2). Besides, the MnS inclusions are cracked in several places and they undergo a strong decohesion that can lead to the ejection of some of them out of the material.

The *in-situ* tensile tests enable to situate with accuracy the crack initiation sites in the specimens tested (SEM observations), as well as the cleavage facets (on the breaking patterns) where the main cracks germinate. The electron back-scattered diffraction (EBSD) is then used to determine the crystallographic orientation of the grains, thereby confirming that the cleavage planes (from which the main cracks start) are $\{100\}$ ones, as it has already been shown in ferrite^[2].

3.2 Stress distribution and $\varepsilon_{\phi\psi}$ strain analysis (XRD)

The stress analyses in ferrite are realized at each temperature tested. At -150°C under loading for example, cementite reaches values of the order of 2600 MPa, while ferrite does not go beyond 665 MPa (Fig.3): so, $\sigma_{\text{cementite}}^I > \sigma_{\text{ferrite}}^I$. The ferritic phase is therefore in compression while the cementite is in traction, after unloading. The difference between the macroscopic stress and the stress in ferrite increases with the applied strain and also with decreasing temperatures, never exceeding -150 MPa (it is maximum at -196°C): it can be more important in other materials such as duplex steels^[3] (it can thus reach more than 400 MPa). Besides, the fracture stress in ferrite is close to 700 MPa, and seems to be constant in relation to temperature (-60°C , -80°C , -120°C and -150°C).

The $\varepsilon_{\phi\psi} = f(\sin^2\psi)$ strain measurement ($\{211\}$ planes, tensile direction) shows that the average slope is negative after unloading (it also increases at low temperatures), because it is linked to the compressive state of ferrite (Fig.4). The $\sin^2\psi$ relation is not linear: ripples characterize the heterogeneity of the elastic strain and reveal that the intergranular stresses in the ferritic phase are different according to the crystallographic orientation of the considered grains^[3]. These ripples are emphasized if the material has a crystallographic texture and/or if its yield stress increases (which is particularly the case at

Table 1 Tensile test at -150°C

Macroscopic strain/%	σ^I/MPa	$\sigma_{\text{Fe}}^I/\text{MPa}$	$\sigma_{\text{Fe}_3\text{C}}^I/\text{MPa}$
0.04	78	74	80
0.22	394	391	440
0.95	733	662	2082
1.99	757	663	2543
3.77	786	664	2584
4.36 (failure)	727	631	2546

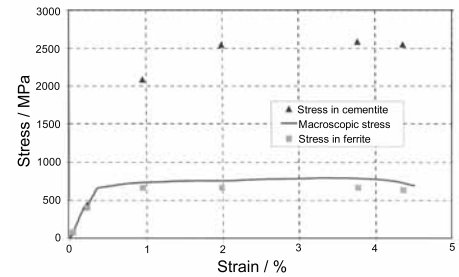


Fig.3 Stress distribution in the 16MnNiMo5 bainitic steel during a tensile test at -150°C

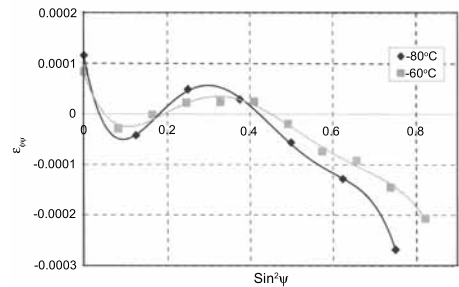


Fig.4 Influence of temperature on the $\varepsilon_{\phi\psi} = f(\sin^2\psi)$ strains measured after unloading (8% strain rate)

low temperatures).

4. Polycrystalline Modelling

A polycrystalline modelling is developed concurrently with the experimental measurements. It aims at predicting the stress states in each phase as well as the cleavage fracture, in relation to temperature. The problem is considered on a crystallographic scale, using a two-level homogenisation.

First, a Mori-Tanaka^[4] formulation enables to describe the elastoplastic behaviour of a bainitic single crystal (stress distribution in each phase). Plastic activity is controlled by an energetic criterion^[5], which regulates

the activation of slip systems. The behaviour law can be written:

$$\dot{\sigma} = l_{\text{Fe}/\text{Fe}_3\text{C}} \cdot \dot{\varepsilon},$$

where $l_{\text{Fe}/\text{Fe}_3\text{C}} = [l_{\text{Fe}} + f \cdot (C_{\text{Fe}_3\text{C}} \cdot T - l_{\text{Fe}})] \cdot [(1 - f) \cdot I + f \cdot T]^{-1}$ is the bainitic elastoplastic tangent modulus. $\dot{\sigma}$ and $\dot{\varepsilon}$ are the stress and the strain rate of the bainitic single crystal, respectively. f is the volume fraction of cementite, $C_{\text{Fe}_3\text{C}}$ and l_{Fe} are the elastic and elastoplastic characteristics of each phase, $T = [I + S^{\text{Esh}} \cdot l_{\text{Fe}}^{-1} \cdot (C_{\text{Fe}_3\text{C}} - l_{\text{Fe}})]^{-1}$ comes from the solution of the elastic inclusion problem^[6] and S^{Esh} is the Eshelby tensor calculated from the bainitic elastoplastic tangent modulus $l_{\text{Fe}/\text{Fe}_3\text{C}}$.

The transition to polycrystal is then achieved by an elastoplastic self-consistent approach^[7]. For the elastoplastic heterogeneous 16MnNiMo5 steel, the constitutive relation is defined on the macroscopic scale by:

$$\dot{\Sigma} = L_{\text{Fe}/\text{Fe}_3\text{C}} \cdot \dot{E}^t,$$

where $L_{\text{Fe}/\text{Fe}_3\text{C}} = \frac{l_{\text{Fe}/\text{Fe}_3\text{C}} \cdot [I + S^{\text{Esh}} \cdot L_{\text{Fe}/\text{Fe}_3\text{C}}^{-1} \cdot (l_{\text{Fe}/\text{Fe}_3\text{C}} - L_{\text{Fe}/\text{Fe}_3\text{C}})]^{-1}}{(l_{\text{Fe}/\text{Fe}_3\text{C}} - L_{\text{Fe}/\text{Fe}_3\text{C}})]^{-1}}$ is the macroscopic elastoplastic tangent modulus. This last equation is an implicit one: it is resolved by a succession of iterations until the solution converges.

This Mori-Tanaka/self-consistent modelling is very efficient and well adapted to the material, since the stress difference between each phase in relation to temperature agrees with the one measured by XRD. It also reproduces correctly the $\varepsilon_{\phi\psi} = f(\sin^2\psi)$ intergranular strains (Fig.5), by projecting the elastic strain tensor normally to the considered diffracting plane:

$$\varepsilon_{\phi\psi} = \langle n_i \cdot \varepsilon_{ij}^e \cdot n_j \rangle_{\phi\psi}$$

It enables to determine as well the stress states within the material for any grain orientation: since cleavage occurs normally to the $\{100\}$ planes in ferrite, it is able to predict in particular the evolution of the $\sigma_{\{100\}}^g$ stress (stress normal to the $\{100\}$ planes) in each bainitic single crystal. In this case, $\sigma_{\{100\}}^g$ is defined by the following relation:

$$\sigma_{\{100\}}^g = \sum_{i,j} n_i \cdot \sigma_{ij} \cdot n_j$$

where n is the normal to the $\{100\}$ planes.

A threshold for cleavage propagation has been identified^[8]. It corresponds to a critical value of the $\sigma_{\{100\}}^g$ stress in the ferritic grain: $\sigma_{c\{100\}}^g = 465$ MPa. Some grains will attain it before others, depending on their crystallographic orientation: therefore, failure will be supposed to take place when a sufficient number of them, which remains the same whatever the temperature considered, reaches this value (7% of the grains in the results presented). This failure criterion is reached sooner at low temperatures, because of the increase in yield stress when temperature decreases (Fig.6), the fracture stress in ferrite remains constant, which is not the case in bainite and cementite, as experimentally observed. Therefore, the model is coherent, it would not be relevant to take the failure criterion in bainite, since it is in

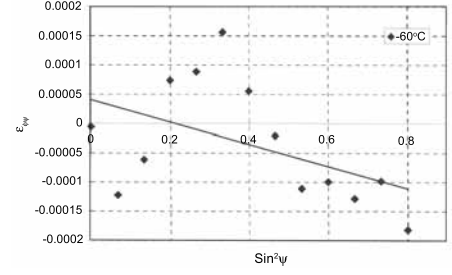


Fig.5 $\varepsilon_{\phi\psi} = f(\sin^2\psi)$ strains calculated with the model after unloading (tensile test, 8% strain rate)

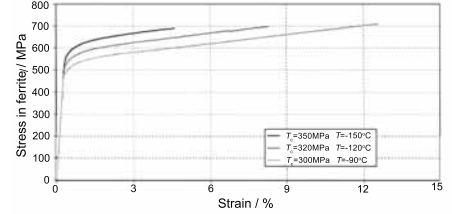


Fig.6 Influence of temperature on the material failure (7% grains reach the cleavage criterion)

the ferritic phase that the cracks leading to the global failure of the material germinate.

5. Conclusions

- (1) Fracture becomes brittle below -150°C , thus, cleavage occurs normally to $\{100\}$ planes identified by EBSD.
- (2) The influence of temperature on stress distribution in the 16MnNiMo5 bainitic steel is very important, the stress difference between ferrite and bainite remains inferior to 150 MPa.
- (3) The polycrystalline modelling is efficient, since it correctly predicts the stress states in each phase as well as the fracture stress and strain in related temperature.
- (4) Many *in-situ* tensile tests remain to be done, in order to understand all the fracture mechanisms taking place between -196°C and -60°C , and to provide new crystallographic damage criteria. The model will be further improved by considering a hardening which depends on temperature, the behaviour of all the grains having the same crystallographic orientation, the formation of dislocation structures.

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