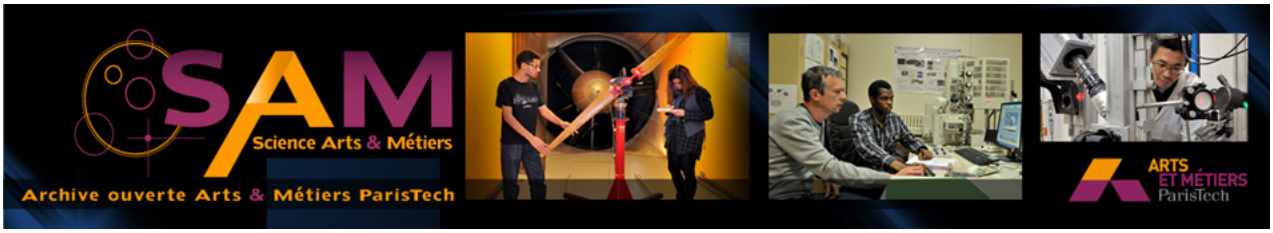

Influence of temperature on stress distribution in bainitic steels - Application to 16MND5-A508 pressure vessel steel

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Abstract. In this study, the internal stress evolution of the ferrite phase of 16MND5-A508 has been determined using X-Ray Diffraction (XRD). The results of in situ tests combined with XRD analyses and performed at different temperatures (-150°C and 22°C) exhibit a difference of about 200MPa between the macroscopic stress and the ferrite one. The stress state in the cementite is determined by a mixture law; it reaches very high values up to 9000MPa. These results highlight the need to analyze the stress directly in the cementite phase by using appropriate tools, since its volume fraction does not allow it using XRD.

Introduction

In complex assemblies such as nuclear reactors, sources of radiation can induce changes in the mechanical properties of materials. When considering the resilience curve of the 16MND5-A508 pressure vessel, the ductile-to-brittle transition region is therefore shifted to high temperatures, leading to an increase in the risk of cleavage brittle fracture in case of rapid cooling [1].
To bring a better comprehension of failure mechanisms in this low alloy steel at different scales (macroscopic, phase, intragranular), especially those involved in the cleavage fracture, a series of experiments have been performed at room and low temperatures (-150°C) to promote this kind of damage; in situ tests combined to laboratory XRD analyses enabled to follow the stress evolution in ferrite phase.

Studied materials

The 16MND5-A508 steel provided by EDF company is a low alloy steel used for the conception of Pressurized Water Reactors (PWR). Table 1 gives its chemical composition in weight percentage.

C	S	P	Mn	Si	Ni	Cr	Mo	V. Cu. Co. Al et N
0.159	0.008	0.005	1.37	0.24	0.7	0.17	0.5	<0.1

Table 1: Chemical composition of 16MND5 steel [weight % - iron balance]

The steel underwent three stages of heat treatment: two austenitizations at 850°C followed by water quenching, a tempering between 630°C and 645°C to improve the toughness and finally a stress relieving treatment at 610°C. At the end, an upper bainite with two phases was obtained: ferrite (Fe), which forms the matrix and precipitates of cementite (Fe_3C), which reinforce the matrix [2].
Before crystallographic observations, the samples were mechanically polished and etched with 2% Nital. Fig. 1-a shows a typical microstructure of 16MND5 steel obtained with FEG-SEM.

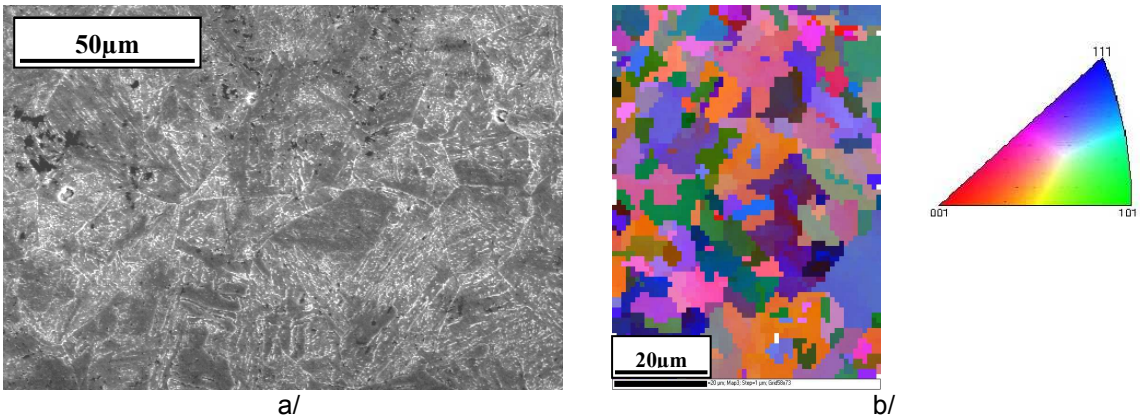


Fig. 1: a/-FEG-SEM micrograph showing the microstructure of the 16MND5 steel – b/-EBSD mapping highlighting the complex structure of the steel

The size of the bainite grains is relatively homogeneous (~50µm). Ferrite is composed of thin laths, the observation of which is difficult with SEM. The EBSD (Electron BackScattered Diffraction) technique can be more appropriate to make evident the real size of each element of the microstructure. The examination reveals a size of lath from 5 to 15µm, oriented perpendicularly to the former austenitic grain boundaries (Fig. 1-b). Fe_3C precipitates are distributed non-homogeneously: they can be intragranular (located between ferrite laths or dispersed in the structure) or intergranular with an average carbide size less than 0.1µm. A phase dosing was performed by neutron diffraction: the volume fraction of cementite was estimated to 2.1% ± 0.1. The presence of MnS inclusions with a low volume fraction (<1%) is also observed. They are mainly elongated in the direction of forging (~60µm) or spherical (~8µm), and can be isolated or grouped in clusters of 3 to 8 inclusions.
A second steel type XC40 supplied by ARCELORMITTAL was also studied in order to show a possible size effect on phase stress distribution and brittle fracture. Its chemical composition is given in table 2.

C	Mn	Si	P et S
0.4	0.7	0.10-0.40	<0.035

Table 2: Chemical composition of XC40 steel [weight %- iron balance]

This steel was heat treated in order to obtain a two-phased microstructure composed of ferrite and cementite (Fig. 2) with a carbide size of about 1µm and a volume fraction of about 5.7%. The grain size is smaller than in 16MND5 steel: it is about 15µm. The heat treatment will not be specified in this paper as it's a confidential data to the industrial.

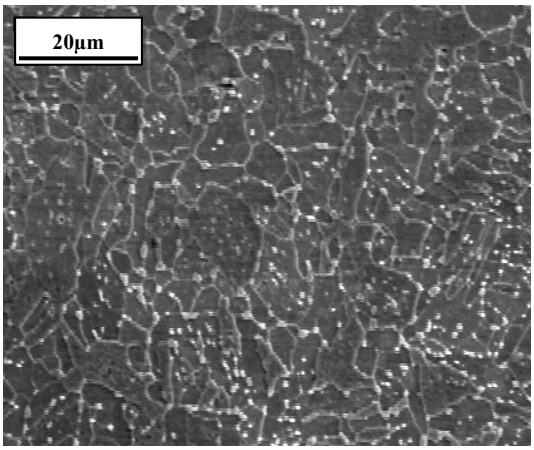


Fig. 2: SEM micrograph showing the microstructure of the XC40 steel

The carbide size distribution determined by image analysis reveals an average size of 0.73 µm: intergranular carbides are the largest while smaller carbides are predominantly intragranular.
For the work presented here, the evolution of internal stress in the two phases during macroscopic loading has been determined for both steels described above.

Stress analyses by XRD in ferrite phase

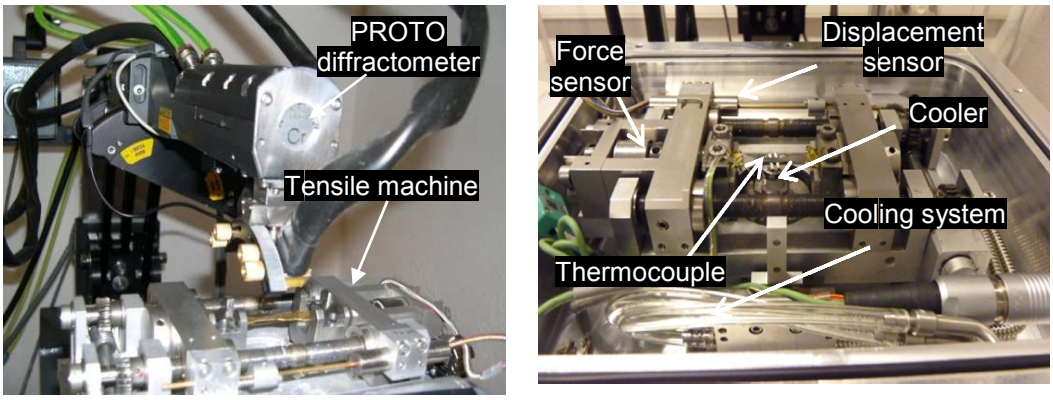
XRD allows the determination of stresses in each phase of a polycrystalline material. For stress analysis, the $\sin^2\psi$ method is used to determine the macroscopic or pseudo-macroscopic strain-stress in the ferrite phase considering lattice plane distance as a strain gauge. More details about this method are given in [3].
The experimental work concerns only the ferrite phase, since the volume fraction of cementite in 16MND5 steel is too low to make any analyses; we use the following elastic constants S_1=-1.277*10^{-6}MPa^{-1} and $\frac{1}{2}S_2$=5.919*10^{-6}MPa^{-1} which are determined from de Young's modulus and Poisson's ratio of the material (Eq.4):

$$S_1=-1\frac{\upsilon}{E} \;\&\; \frac{1}{2}S_2=\frac{1+\upsilon}{E} \tag{4}$$

For direct measurements in cementite, the radiation flow or diffracted volume must be increased using synchrotron radiation or neutron diffraction. Therefore, the stress state in cementite is first estimated using a mixture law which takes into account the volume fraction of each phase (Eq. 5):

$$\sigma^I=f_{Fe}\sigma^I_{Fe}+f_{Fe_3C}\,\sigma^I_{Fe_3C} \tag{5}$$

where σ^I is the macroscopic stress, (σ^I_{Fe}, $\sigma^I_{Fe_3C}$) are the stresses in the ferrite and cementite phase, and (f_{Fe}, f_{Fe_3C}) are the volume fractions of ferrite and cementite.
In order to determine the stress evolution of the ferrite phase during loading, XRD analyses are carried out in situ using a small tensile machine placed directly under a Proto goniometer (Fig. 3):



a/ b/

Fig. 3: Experimental setting up - a/ Proto diffractometer - b/ Small tensile machine allowing in situ analyses

The influence of temperature on the mechanical behavior of the two studied steels is investigated. The small tensile machine is kept at low temperature with a cooling system using liquid nitrogen and isolated in a thermal enclosure to limit ice formation on the samples during cooling operation. This allows maintaining intense diffraction peaks.
For all the tests, flat specimens (dimensions in mm: 60x2x1) were loaded in uniaxial tension to determine the evolution of internal stresses at room temperature and -150°C. Analyses were made during loading considering the {211} crystallographic planes of ferrite and using CrK_{α} radiation (wavelength $\lambda = 2.2897$Å).

Results and discussion

The stress results determined in situ through tensile tests on the two materials are presented below. The curves give the evolution of internal stress in the ferrite phase during macroscopic loading with an average uncertainty of about ±20MPa.
Fig. 4 shows the ferrite stress evolution (isolated dots) and the macroscopic stress (continuous line) obtained for the 16MND5 steel at room temperature and -150°C. The specimens were not equipped with extensometer; the macroscopic stress and the strain were calculated from a force and displacement sensors directly attached to the tensile machine. The macroscopic stress represents the bainite one and the ferrite stress is determined by XRD.

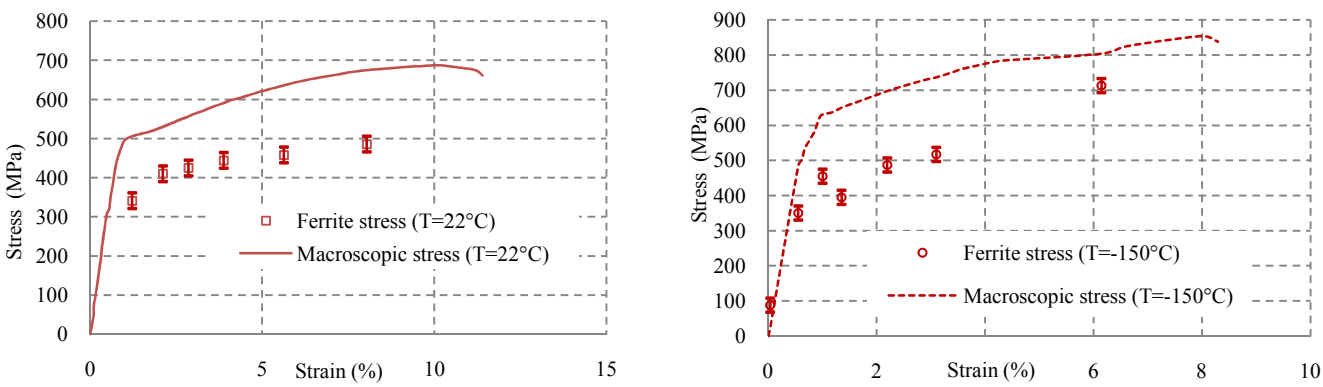


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Fig. 4: Stress evolution in ferrite phase, 16MND5 steel - a/ T=22°C - b/ T=-150°C

At room temperature, the yield strength of 16MND5 steel is about 500MPa, which is in agreement with literature data [4]. It is observed that the stress state in the ferrite remains close to the macroscopic one. The difference between the two stresses increases slightly with the plastic

deformation: it is approximately 150MPa. At low temperature, there is a classical increase in the macroscopic yield strength that is about 620MPa. The ferrite stress also slightly increases. If one compares the ferrite and bainite stresses, the gap between the two is greater than at room temperature since it reached 200MPa. These results are also observed in Mathieu's works [5].
The same tests were conducted on XC40 steel that differs mainly to 16MND5 steel by its cementite volume fraction. Fig. 5 shows the curves obtained for this steel at room temperature and -150°C with the evolution of the stress in ferrite.

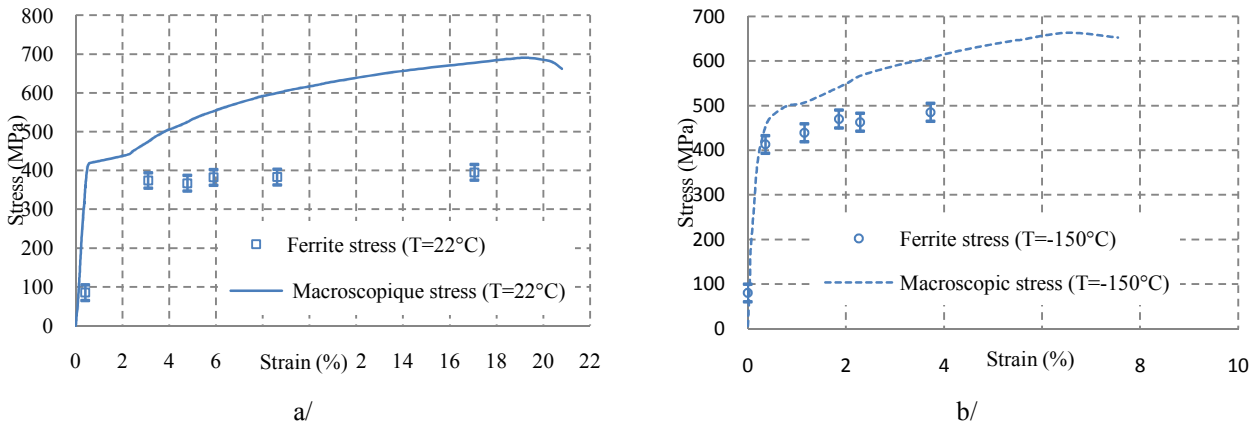

Fig. 5: Stress evolution in ferrite phase, XC40 steel - a/ T=22°C - b/ T=-150°C

XC40 steel is more ductile than 16MND5; its yield strength is about 420MPa at room temperature and reached 500MPa at -150°C. The evolution of internal stresses in ferrite during loading seems to be similar for both steels.
At room temperature, the gap between the macroscopic stress (bainite) and ferrite one is 200MPa for XC40 steel. Unlike 16MND5 steel, this gap seems to be less important at low temperature, since it is only 100MPa. This behavior might be explained by the difference in the volume fraction of cementite that characterizes the two steels. Likewise, the different grain size and carbide distribution may also provide an explanation since they could compensate the macroscopic stress level reached [6]. However, more works must be performed to confirm these results.
On the other hand, XRD analyses reveal that the macroscopic stress in both steels is governed by ferrite phase due to its high volume fraction in both steels (97.9% for 16MND5 steel and 94.3% for XC40 steel).
Considering now the stress in cementite calculated from the XRD measurements in ferrite and mixture law indicated in paragraph 3, the values obtained at 22°C and -150°C for both studied steels are presented in Table 3 with an average uncertainty of about ±930MPa for 16MND5 steel and ±330MPa for XC40 steel. This uncertainty is linked to the mixture law that is very sensitive to errors in main phase stresses when being applied to a material with phases of very low volume fraction.

	Stress in 16MND5 steel [MPa]			Stress in XC40 steel [MPa]		
	(σ) bainite	(σ) ferrite	(σ) cementite	(σ) bainite	(σ) ferrite	(σ) cementite
T=22 [°C]	530	410	6130	540	390	3020
	680	490	9540	610	420	3750
	(σ) bainite	(σ) ferrite	(σ) cementite	(σ) bainite	(σ) ferrite	(σ) cementite
T=-150 [°C]	630	460	8560	510	440	1670
	740	520	11000	570	465	2310

Table 3: Stress distribution in both studied steels at T=22°C and T=-150°C

In the case of 16MND5 steel, we observe that cementite stress state increases with increasing plastic deformation; the reached values are about 9000MPa. Cementite stress also increases when the temperature drops: the maximum value is 11000MPa.
In the XC40 steel, the values of stress in cementite are much lower than in the 16MND5 steel; it does not exceed 3700MPa. We note however that in this steel, the cementite stress state appears to decrease with decreasing temperature.
When comparing these values with the macroscopic or ferrite stresses in both steels, we can see that they are much higher due to the low volume fraction of cementite. Therefore, this highlights strong stress heterogeneity by phase. These values are comparable to those predicted by models that take into account even more important volume fractions [7]. But since many people wonder if it is realistic to think that this phase can really stand such loadings, it is crucial to estimate experimentally the level of stress in cementite to validate or refute these various models developed to predict the mechanical behavior of the PWR steel. This underlines the need to determine directly the stress in the cementite phase during loading using diffraction techniques more appropriate than XRD (neutron diffraction and synchrotron radiation).

Conclusion

The present experimental works were carried out in order to characterize the brittle mechanical behavior of 16MND5 steel through in situ tensile tests combined to X-ray diffraction. The obtained results of per phase stress analysis are consistent with literature. The macroscopic strength tensile of 16MND5 steel is about 700MPa at room temperature and 820MPa at -150°C. According to the stress distribution presented above, the breaking strength of the ferrite phase is estimated to 550MPa at 22°C and 600MPa at low temperature. However, more tests are necessary to confirm XC40 steel results.
The volume fraction of cementite in the 16MND5 steel (2.1%) and its low carbide size require the use of tools, for stress determination, more powerful than XRD (neutron diffraction and synchrotron radiation).
First stress analyses were conducted directly in the cementite phase at the Institute Laue Langevin in Grenoble; they showed that for this low volume fraction of carbides, it is necessary to use a maximum neutron flux in order to achieve useful results. New experimental tests are therefore projected in this way and tests performed at the European Synchrotron Radiation Facility in Grenoble are still under examination.

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