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Evolution of stress in the cementite phase of the 16MND5 pressure vessel steel during a tensile test at very low temperatures (-150°C)

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Aims of the experiment and scientific background

Reactor safety is a critical area. Under normal use conditions, pressure vessel steels display a ductile behavior, but neutron irradiation ageing causes a temper embrittlement which shifts their ductile to brittle transition range to higher temperatures. Therefore, to assess how the integrity of these bainitic steels may be compromised during a pressurized thermal shock (in case of urgent cooling or an accident involving loss of coolant, for instance), it becomes necessary to consider their brittle behavior. It is thus very important to characterize the mechanical properties of such un-irradiated materials at very low temperatures and to define relevant criteria in order to predict their service life: irradiation leads to a shift of the resilience curve to higher temperatures so that the behavior of the un-irradiated material at -150°C is equivalent to the one of the irradiated material at 30°C, temperature reached in case of a dramatic cooling (and it is forbidden to study irradiated material.

A bainitic steel is composed of two phases, ferrite and cementite that don’t have the same mechanical properties. It is relatively easy to determine the stress state in ferrite using X-Ray Diffraction (XRD), but it is absolutely impossible to determine the stress in cementite, due to its low volume fraction. As a result, a much higher X-ray flux/energy is needed to observe and subsequently exploit diffraction peaks. Usually, to work around those practical difficulties, we use more or less “classical” models (mixture law, Mori-Tanaka or self-consistent models…) that permit to estimate the stress in cementite from the macroscopic stress and/or the stress determined in ferrite [1]. Now, while the usefulness of such models is obvious and undeniable, it is however necessary to calibrate and validate them through experimental testing (X-rays, synchrotron emission), all the more so as they unfortunately tend to reach their limit fairly quickly, especially when it comes to predicting per-phase stress distribution when volume fractions are low from the start, which is the case for the 16MND5 steel (about 2%). A very small variation in the volume fraction of cementite is enough to increase stress tremendously in this phase; for example, a simple deviation of a few tenths of percent can cause a variation of several hundreds of MPa in the stress state and can therefore have a dramatic influence on the stress fracture criteria used. With volume fractions of the order of 1.5 to 2 %, the models show that the stress state can quickly reach values around 3000-4000MPa or even more (figure 1b); at the moment, many people (including us) wonder if it is realistic, relevant (or even correct!)

to think that this phase can actually stand such loadings, and to focus therefore only on criteria (maximum stress, fracture stress) determined in the ferritic phase.

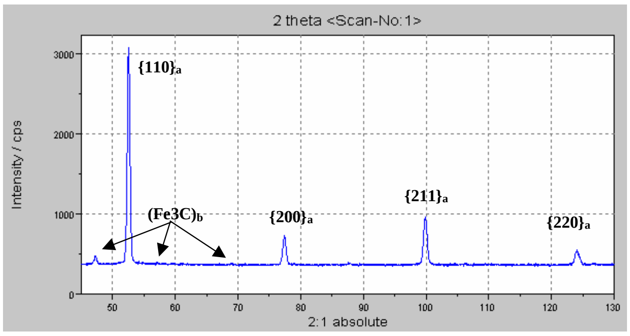

Figure 1a - Diffraction peaks (ferrite ‘a’ and cementite ‘Fe_3C’) obtained using for example a cobalt anticathode (XRD)

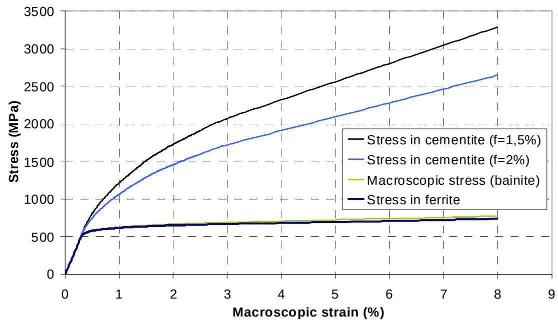

Figure 1b - Stress distribution in the 16MND5 bainitic steel at -150°C (model)

Experimental method

A small micromachine was used for in situ tensile tests on ID11 beamline, with high-energy X-rays. Ring diffraction measurements were conducted with a 60keV (λ=0.207Å) monochromatic X-ray beam in transmission mode to follow the evolution of the bulk stress in both ferrite and cementite phases simultaneously, Figure 2. The micromachine was placed so that the tensile axis was always vertical, and the different samples were previously covered with a thin layer of vacuum grease and a nanocrystalline CeO_2 cerium dioxide powder as calibrant. The 50*50µm incident beam entered normally to the specimens forming complete Debye-Scherrer rings from ferrite, cementite and the CeO_2 calibrant. These resulting 2D diffraction rings were recorded by a Frelon 2D CCD camera, with a resolution of 2048*2048 pixels and a 48.1*46.8µm pixel size. The sample-to-camera distance was 340mm in order to mainly focus on the {110} planes of ferrite and the {122} planes of cementite.

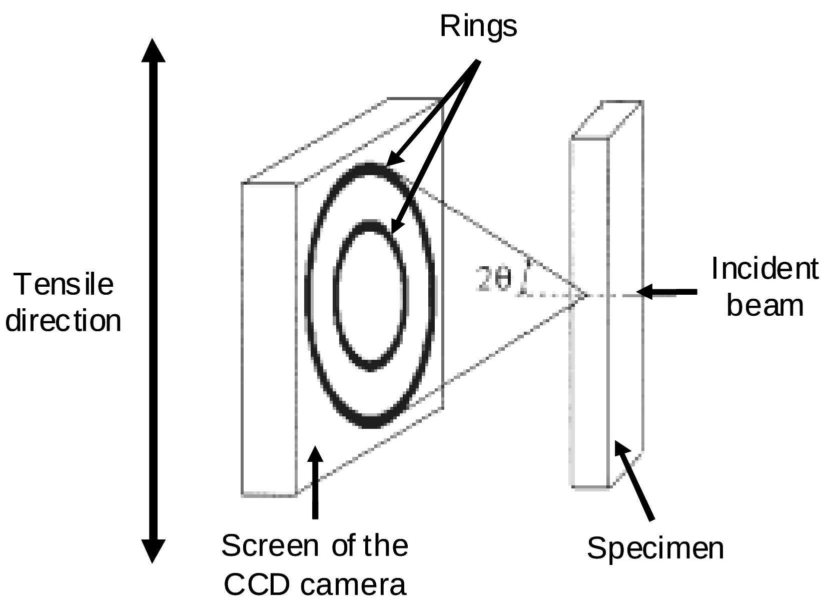

Figure 2 - Ring diffraction device in the ESRF, ID11 beamline

Results

The obtained diffraction rings (figure 3) were analyzed using the FIT2D software. A complete procedure was developed in order to obtain the ε_{11} and ε_{22} elastic lattice strains in the tensile and transverse directions.

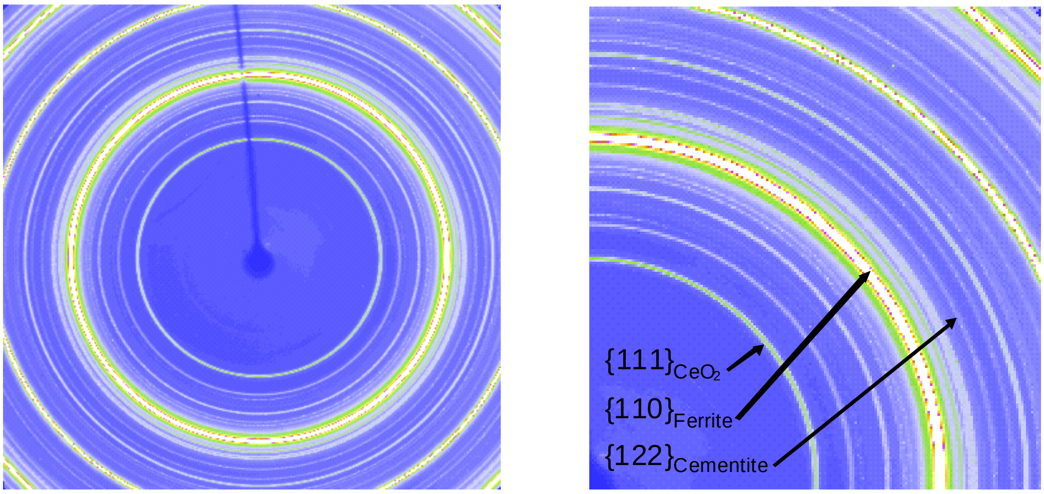

Figure 3 - Ring diffraction pattern with different rings corresponding to the two phases of the material analysed and the CeO_2 calibrant - 20°C

The integration of the rings therefore lead to the evolution of ε_{11} and ε_{22} elastic strains vs. macroscopic strain applied for both ferrite and cementite presented in figure 4. As expected, since the 11 direction is the tensile direction, ε_{11} and ε_{22} strain values were positive and negative, respectively. Moreover, in the plastic range, the level of strain in cementite was about five times higher than in ferrite; this can be explained by the difference in mechanical properties between the two phases, and in particular the yield stress.

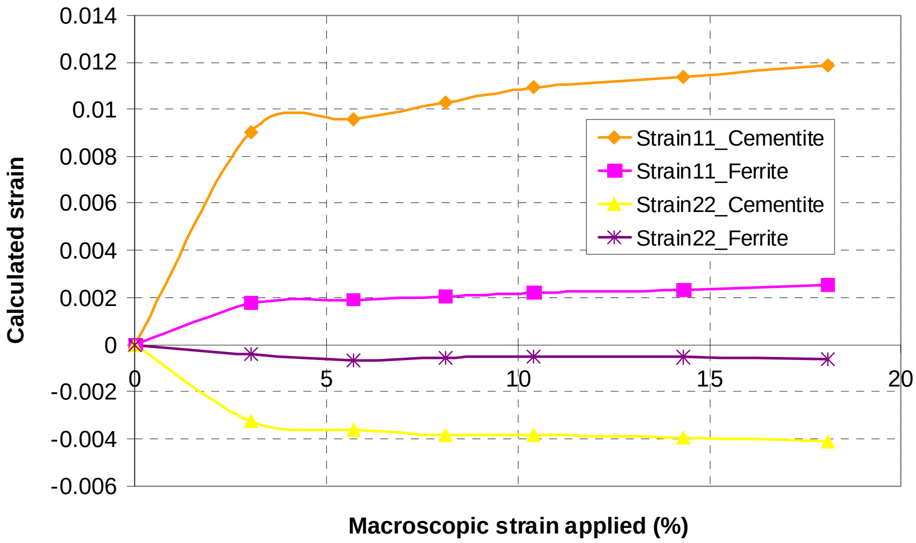

Figure 4 - Evolution of ε_{11} and ε_{22} elastic strains for both ferrite and cementite during loading - 20°C

The stress values in the tensile direction were then estimated in each phase using linear elastic formulation with the hypothesis $\varepsilon_{22} = \varepsilon_{33}$:

$$\sigma_{11} = \frac{E}{1+\nu}\varepsilon_{11} + \frac{E.\nu}{(1+\nu).(1-2\nu)}.(\varepsilon_{11} + 2\varepsilon_{22})$$

where E and ν were the Young modulus and Poisson's ratio of each phase, respectively, considering the {110} planes for ferrite and the {122} planes for cementite. There results are presented in figure 5.

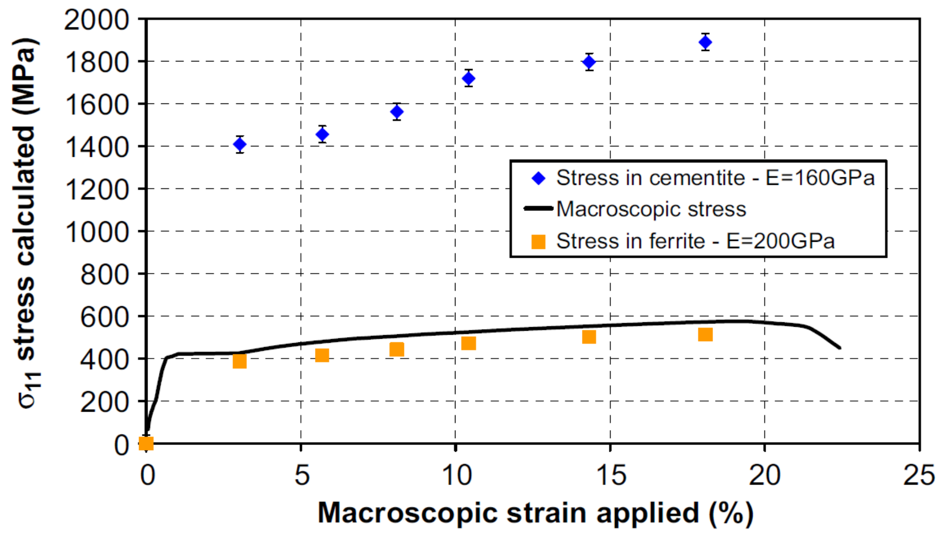

Figure 5 - Evolution of the σ_{11} stress for both ferrite and cementite during loading - 20°C

All the results will be published in international scientific journals. For room temperature, see [2].

References

[1] K. Inal, R. Pesci, J.L. Lebrun, O. Diard and R. Masson, « Grain and phase stress criteria for behaviour and damage in duplex and bainitic steels », Fat. & Fract. of Eng. Mat. & Struc. 29 (9), 2006, 685-696.

[2] V. Taupin, R. Pesci, S. Berbenni, S. Berveiller, R. Ouahab and O. Bouaziz, « Lattice strain measurements using synchrotron diffraction to calibrate a micromechanical modeling in a ferrite-cementite steel », Mat. Sci. Eng. A 561, 2013, 67-77.