

Internal stresses and carbon enrichment in austenite of Quenching and Partitioning steels from high energy X-ray diffraction experiments

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ABSTRACT

Quenching and Partitioning (Q & P) process permits to produce innovative microstructures containing large fraction of carbon enriched retained austenite. The present study highlights that austenite undergoes significant internal stresses generated during such thermal cycle. Both mechanical and chemical contributions are likely to affect its stability at room temperature and thus the resulting mechanical properties of the steel. The experiments carried out by High Energy X-Ray Diffraction (HEXRD) show unambiguously that internal stresses in austenite originate from martensitic transformation strain and from additional hydrostatic stresses induced during both reheating to partitioning temperature and final cooling. These eigenstrains are attributed to the difference in Coefficients of Thermal Expansion (CTE) between martensite and austenite and are predicted successfully with a purely elastic mean field approach. In the present study, retained austenite is shown to be in compression at room temperature. As a consequence, this state of stress contributes to stabilize retained austenite against a possible strain induced transformation at room temperature and affects the way to determine the carbon content in austenite.

1. Introduction

Q & P is a new annealing route proposed in 2003 to produce 3rd Generation of Advanced High Strength Steels for automotive applications [1–3]. This two-step process leads to nanostructured duplex martensite/austenite microstructures with excellent balance between strength and formability. The high flow stresses of these steels are explained by the fine lamellar structure of the martensitic matrix (typical thickness of martensite blocks of laths around 200 nm). They also show a high work-hardening rate and thus a good drawability thanks to a TRIP effect (Transformation Induced Plasticity), i.e. a dynamical hardening due to the strain-induced transformation of austenite into martensite. The stability of austenite against a strain-induced transformation at room temperature is thus one of the key factors controlling the mechanical properties of these steels and must be designed with great care [4–6].

The stability of retained austenite in Q & P steels, as in more conventional ferritic TRIP steels, depends on its local composition (mainly carbon content in the studied steels) [4] but also on its mechanical state

(hydrostatic stresses) and its local micromechanical neighborhood (size and shape effects) [5]. Xiong et al. [6] have investigated using HEXRD a Q & P steel containing two populations of retained austenite islands with high and low carbon contents respectively. When straining such steel, the strain induced martensitic transformation is surprisingly shown to occur in carbon rich films instead of films showing lower carbon content [6]. The authors have thus suspected a stabilizing effect of internal stresses due to the phase transformations to explain this unexpected result, when compared to other studies [4].

HEXRD [7] or neutron diffraction [8] have been used in the past to study in situ also phase transformations and carbon enrichment in austenite during Q & P processes. Nevertheless, these prior studies are restricted to the partitioning step. In a previous paper [9], the authors have shown, using in situ HEXRD experiments, that austenite is not only enriched in carbon due to both partitioning and carbide-free bainitic transformation, but is also affected by successive mechanisms inducing internal stresses in austenite, confirming the views of Xiong et al. Along the Q & P cycle, austenite undergoes first compressive hydrostatic stresses after the primary martensitic transformation, and tensile

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hydrostatic stresses due to eigenstrains at phase scale during final cooling. These eigenstrains of thermal origins are induced by the difference between the coefficients of thermal expansion (CTE) of martensite and austenite. They will be designated as thermal eigenstrains in the following. In this previous analysis, retained austenite was shown to undergo tensile hydrostatic stresses which favor a possible TRIP effect at room temperature (RT). On the other hand, carbon enrichment leads to austenite stabilization (against thermal and strain-induced transformation). These three contributions (phase transformation, carbon enrichment and thermal eigenstrains) affect the austenite lattice parameters and can solely be discriminated from in situ analysis. As a corollary, it must be admitted that the austenite carbon content of retained austenite should not be estimated solely from its lattice parameter at RT in these steels.

In this paper, the thermal eigenstrains are derived from mean field models, in order to predict the internal stresses during reheating and final cooling. The analysis conducted introduces for the first time the temperature dependence of the coefficient of thermal expansion (CTE) of unconstrained austenite [10,11]. The results achieved are based on new in-situ HE XRD experiments conducted at DESY synchrotron beamline in Hamburg [12]. Compared to our previous studies at ESRF, qualities of the thermal cycles have been improved while keeping the time-resolved quantification.

2. Material and methods

The studied steel is a Fe–0.313C–2.44Mn–1.52Si (wt%, as for all compositions given in this paper) model alloy, a chromium free version of the alloy studied previously by the authors [9,13]. The alloy has been prepared by Vacuum Induction Melting (VIM). 30 mm thick ingots were homogenized at 1250 °C for 16 h followed by hot rolling until 10 mm sheets were obtained. Finally, cylindrical samples ($\Phi = 4$ mm; 10 mm height) were machined in the central part of the sheets. The Ms value of the studied steel is 320 °C according to our in situ investigations. This value is consistent with Van Bohemen's model (313 °C) [14].

The HEXRD experiments were carried out on the DESY PETRA P07 beamline (Hamburg, Germany). The high-energy monochromatic beam ($E = 100$ keV, $\lambda = 0.13$ nm) allowed working in transmission mode. The association with a fast 2D Perkin-Elmer detector enabled high acquisition rates (10 Hz) needed to study time-resolved processes on bulk samples, especially during initial quenching and reheating. The detector was positioned about 1 m behind the sample, giving access to full Debye-Scherrer rings with a maximum 2θ angle of 12°. The experimental set-up is similar to the one used in [9,13].

The 2D diffraction patterns produced during the experiments were integrated circularly using Fit2D software (<http://www.esrf.eu/computing/scientific/FIT2D/>). The deduced 1D diffractograms (intensity vs 2θ) were analyzed with a full Rietveld refinement procedure. Diffraction peaks were modeled by pseudo-Voigt functions using FullProf software (<https://www.ill.eu/sites/fullprof/>) with 20 degrees of freedom for each record (background, phase fraction, lattice parameters, shape of peaks, and temperature effects).

On the experimental diffraction patterns, two phases can be identified and calibrated for certain. The first one is a face-centered cubic (fcc) phase corresponding to austenite. The second one is a body centered phase, close to a body centered cubic (bcc) lattice. This phase correspond either to martensite transforming during the initial and the final quench or to bainitic ferrite transforming during partitioning. The better resolution of the experiments conducted at DESY compared to our previous ones carried out at ESRF [9,13] have permitted to measure the mean tetragonal distortion of the lattice considering a body centered tetragonal (bct) lattice for the refinement procedure (better indexing criteria than using a bcc lattice). Nevertheless, the distinction has not been made between martensite and bainite contributions. As a consequence, only two phases have been taken into account in the Rietveld refinement procedure ($Fm\bar{3}m$ and $I4/mmm$ space groups). As in

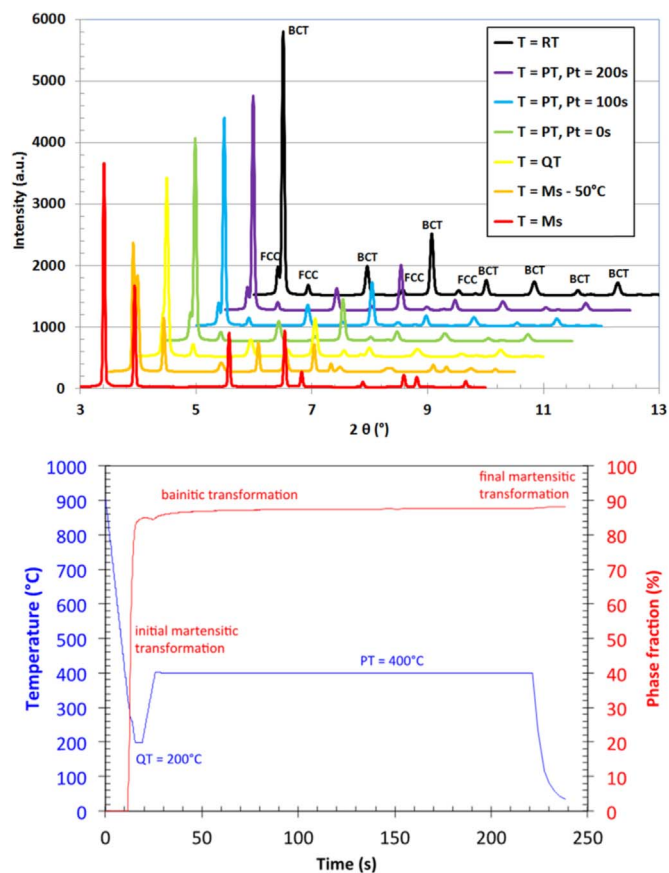


Fig. 1. (a) 1D diffractograms (intensity vs 2θ) obtained at different characteristic times of the Q & P process (at Ms, Ms-50 °C, QT, at the beginning, the middle and the end of partitioning step respectively and after final cooling at RT). The phases (FCC or BCT) explaining the main diffraction peaks have been indicated on the pattern corresponding to T = RT. The spectra have been offset from one another for readability reasons. (b) Evolution of temperature and of bct-phase fraction measured on diffractograms as a function of time measured along the cycle defined by QT = 200 °C and PT = 400 °C. The maximum error made on phase fraction measurements was about $\pm 1\%$.

our previous experiments on a Fe-0.3C-2.5Mn-1.5Si-0.8Cr [9], weak peaks corresponding to η carbides have been observed on diffraction patterns (precipitation during reheating at about 260 °C). The precipitation of such carbides in Q & P steels has already been suggested by Pierce et al. [15] on the basis of Mössbauer and Transmission Electron Microscopy experiments. This third phase was not quantified so far as the fraction remains below 1% and excluded from the refinement procedure.

Samples have been heat-treated in situ in a Bähr dilatometer. As compared to previous works, the Quench Temperature (QT) is 200 °C and the Partitioning Temperature (PT) is 400 °C. This treatment permits to reach high fraction of martensite at QT and minimize the fraction of bainitic transformation at PT. The full austenitization have been performed at 900 °C during 5 min. The cooling rate during initial quench is about 50 °C/s to avoid any ferritic or bainitic transformation. A 5 s isothermal step is followed at QT to guaranty a good thermal homogeneity in samples. The reheating rate is fixed at 30 °C/s. The partitioning time is about 200 s and the final cooling rate down to RT is about 50 °C/s. Fig. 1(a) shows examples of 1D diffractograms obtained by HEXRD for remarkable times along this thermal cycle.

3. Results

The temperature cycle followed for the experiment defined by QT = 200 °C/ PT = 400 °C is represented in Fig. 1(b), as well as the bct-phase fraction deduced from the Rietveld refinement as function of time. The

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