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In-situ investigation of quenching and partitioning by High Energy X-Ray Diffraction experiments

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ABSTRACT

We report the first ultra-fast time-resolved quantitative information on the quenching and partitioning process of conventional high strength steel by in-situ High Energy X-Ray Diffraction experiment. The time and temperature evolutions of phase fractions, their carbon content and internal stresses were determined. The austenite to martensite transformation below Ms is followed by a stagnant stage during which microstructural state remained unchanged. Afterwards, a fast kinetics of carbon enrichment of austenite during the partitioning step at 400 °C is highlighted. The analysis proposed supports the carbon diffusion from martensite to austenite as the main mechanisms responsible for this enrichment.

The need to improve fuel efficiency and safety has led to a high and growing demand for high-strength steels in the automotive industry. The potential of weight reduction directly depends on mechanical properties improvement, which are in turn controlled by the microstructural features [1]. Recently, a novel steel heat treatment termed "quenching and partitioning" (Q&P) has been proposed as an alternative way to obtain attractive properties [2,3]. The process involves quenching below the martensite start temperature (Ms) followed by a rapid heating and ageing above the initial quench temperature. The ageing step, usually performed between 300 °C and 500 °C, is also termed "partitioning step" since the carbon enrichment in austenite is expected to occur during this stage. The benefits of such a treatment in terms of improved mechanical properties have been clearly demonstrated [3,4]. However, despite the large amount of knowledge acquired in this decade, the Q&P is still a matter of debate. Indeed, the Q&P microstructures are extremely difficult to characterize by traditional metallographic methods since their constituents are on the submicron scale and respond in a similar way to etching [5]. In addition, the mechanism of carbon enrichment in retained austenite during the partitioning step is still controversial. Indeed, strong experimental evidences of carbon partitioning from martensite to austenite exist [6,7], but the possible carbon partitioning from supersaturated bainitic ferrite to austenite is still possible [8]. Moreover, from a kinetics point of view, it has been suggested that the temperature is too low for carbon diffusion and that carbon supersaturation in martensite can be eliminated by carbides precipitation during partitioning step [3,9]. Furthermore, the formation of bainite during partitioning cannot be completely ruled out and could explain the measured of enrichment of carbon in retained austenite, as the temperatures are consistent with those for bainite formation [6].

It was recently demonstrated that in-situ High Energy X-Ray Diffraction (HEXRD) is a powerful method to obtain time-resolved precise quantitative information about phase transformation during quenching of low carbon steels [10]. In the present work and for the first time, the microstructure evolution during the Q&P process of a conventional TRIP steel was investigated by in-situ HEXRD experiments. The results clarify both the time and temperature evolutions of microstructure and the mechanisms of carbon austenite enrichment during the Q&P process.

A Fe-0.3 wt% C-2.5 wt% Mn-1.5 wt% Si-0.8 wt% Cr alloy was cast in a vacuum induction melting furnace. It was then hot rolled and cold rolled to a final gauge thickness of 1.4 mm. The samples investigated in the present study were machined from cold-rolled strip. They are 30 mm long in the rolling direction and their sections are $1.4 \times 4.0\ mm^2$. All the details about the sample preparation can be found in [11].

The experiments were performed at the European Synchrotron Radiation Facility (ESRF) in Grenoble, France, on beam line ID15B under powder diffraction configuration [11,12]. The high energy monochromatic beam ($E = 87$ keV, $\lambda = 0.14$ nm) permits to work in transmission and the association with a fast 2D detector enables high acquisition rates (10 Hz) suitable to study "real time" process on bulk samples. In

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order to optimize the size of the datasets (about 2Go per experiments), two different acquisition rates were used. A slow one at 0.3 Hz during austenite soaking and for the end of the partitioning step, and a fast one at 10 Hz in between as fast processing sequences and associated metallurgical evolutions have to be followed precisely. Special attention was paid when analysing data to assure a perfect continuity in between the two recording regimes without any artefact. The beam size is of 0.7×0.4 mm^2 and the corresponding total volume analysed is of 1.1 mm^3. This corresponds to more than 50,000 austenite grains and is considered as statistically relevant. The 2D diffraction patterns were recorded with a Perkin-Elmer 2D detector positioned about 1 m behind the sample. Consequently, the full Debye-Scherrer rings were observed with a maximum 2θ angle of 12°. The electronic noise background was numerically subtracted from each dataset.

The Q&P heat treatment was reproduced using a commercial heating devices (Instron). The samples were heated up by Joule effect and cooled down by heat extraction through the device jaws. The specimen temperature was controlled by mean of a thermocouple, welded at the centre of the sample in the aplomb of the analysed zone. The samples were then first heated up to 900 °C at 10 °C/s and held for 200 s at this temperature in order to achieve a full austenitic state (cf. Fig. 1b). The samples were successively, cooled down rapidly at 50 °C/s to the quenching temperature (QT) of 230 °C, reheated at about 30 °C/s to a partitioning temperature (PT) of 400 °C, held at PT for 250 s and finally quenched down to room temperature at 20 °C/s. These particular process parameters were chosen since they were shown to correspond to an optimum in terms of austenite retained in the final microstructure (see [3]). The thermal path obtained is schematically shown given in Fig. 1a.

The 2D diffraction patterns were integrated circularly using Fit2D software [13]. The deduced 1D diffractograms (intensity vs. 2θ) were analysed with a full Rietveld refinement procedure [14,15]. The diffraction peaks were modelled from pseudo-Voigt function. It was led using Fullprof software [14,15] using 16 degrees of freedom for each record (background, phase fraction, lattice parameter, shapes of peaks, temperature effects). It is worth noting that the 2D diffraction pattern reveals the presence of a possible rolling texture in ferritic and austenitic phases. Its presence could affect a Rietveld adjustment procedure as it induces systematic over and under representations of certain diffraction peaks for certain azimuthal directions. Nevertheless, in the studied case, the integration of the intensities along complete diffraction rings permit to minimize the errors made when assuming a random texture. The errors on the phase fraction measurements have been calculated for each diffraction patterns using the Fullprof software and are not higher than 0.5% (absolute phase fraction value). Accounting for all the other possible inaccuracies in the procedure (constant chemical composition of the phases, possible peak asymmetries, …), the maximum error made on phase fraction measurements is estimated around 1%, which is the usual value retained in similar works.

Examples of diffraction patterns obtained along the thermal cycle are given in Fig. 1b. It should be emphasised that only a FCC-phase and a BCC-Phase were identified on the different diffraction patterns (Fig. 1b). The FCC-phase corresponds to austenite and the BCC-phase could be martensite and/or bainite (in view of conditions). It is also worth noting that (i) no visible peak splitting proving any tetragonality of martensite was observed (ii) the high acquisition rates make difficult carbide(s) identification.

At first, as shown Fig. 2, the evolution of the austenite lattice parameter with temperature was investigated. Three contributions can account for the observed evolution: thermal expansion, mechanical and chemical effects. The thermal expansion contribution can be evaluated from the linear regime observed on Fig. 2, corresponding to the cooling step from 900 to 300 °C. Indeed, in the temperature range (between tags a and b in Fig. 1), the alloy is fully austenitic, homogenous in terms of compositions and can be considered as stress free. Therefore, the decrease of austenite lattice parameter with temperature can be assumed to be only due to the thermal contraction of austenite. The mean thermal expansion parameter, measured between 400 °C and 800 °C, is 2.526×10^{-5} K^{-1} and is in good agreement with the one reported by Lu et al. [16].

The mechanical and chemical contributions to the evolution of the lattice parameter is given by Δa_γ, defined as the austenite lattice parameter evolution with temperature minus the previously calculated thermal expansion contribution. By construction, Δa_γ is equal to zero during initial cooling down to Ms temperature.

In the following, we will focus on the most interesting part of the cycle corresponding to phenomena occurring during cooling, rapid heating and soaking at partitioning temperature. For the sake of clarity, the reference time $t = 0$ s corresponds at the very beginning of the first cooling from 900 °C (tag a in Fig. 1a). The Fig. 3 shows the measured time-evolution of thermal path, volume fraction of FCC and BCC phases, and Δa_γ. It can be decomposed into 4 steps (i) a temperature decrease from 900 °C to 295 °C during which no phase transformation occurs from austenite and obviously Δa_γ remains equal to zero (see step 1 in Fig. 3) (ii) a temperature decrease from 295 °C to QT = 230 °C during which a significant increase in BCC-phase at the expense of austenite occurs (see step 2 in Fig. 3). The rate of this transformation is very fast in the first stages and becomes more and more sluggish during cooling. This behaviour was already been observed by [10] and was attributed to martensite transformation. The martensite start temperature (Ms), evaluated from Fig. 3b, is about 295 °C and corresponds perfectly to Ms temperature, measured by dilatometry for the same alloy and in the same conditions of cooling [11] and, calculated from empirical equations proposed by [17]. The formation of martensite was attended by a

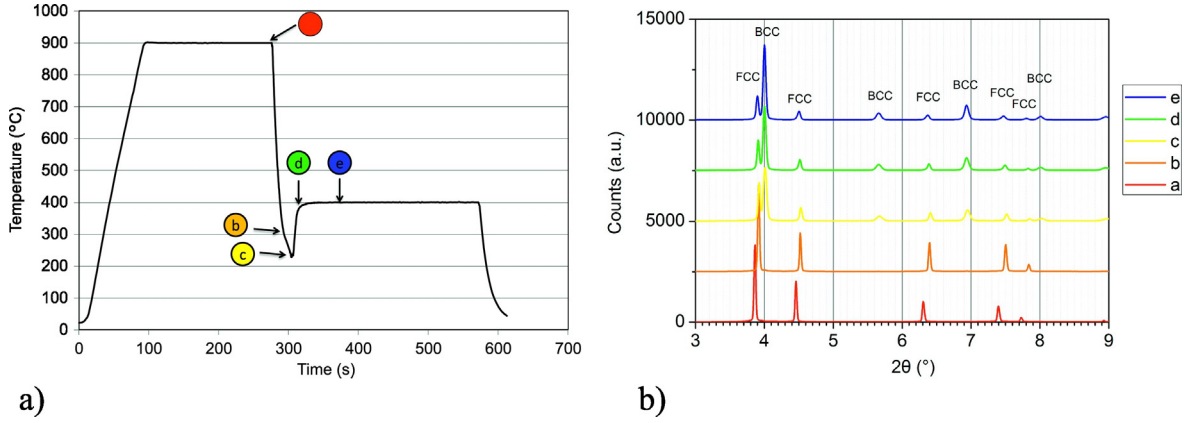

Fig. 1. Time-evolution of a) experimental thermal path b) and 1D diffractograms obtained at the selected times (a: before cooling, b: above Ms, c: QT, d: Begin of partitioning step, e: after 20 s of partitioning respectively).