ESRF	**Experiment title:** *In situ* investigation of ferrite tetragonality during carbon partitioning.	**Experiment number**: MA-2893
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Report:

The aim of the experiment was to determine the kinetics of carbon redistribtuion throughout the various phase transformation occurring in steels, where the objectives were:

- *In situ* determination of ferrite's tetragonality during tempering.
- *In situ* quatification of the ferrite and austenite phase fractions throughout the thermal cycle.
- Investigation of austenite's stability at the end of the heat treatment on cooling to room temperature.

The conditions used were 80 keV beam energy and samples were measured in transmission mode.

To gain resolution, a double detector configuration was requested to vary the sample-to-detector distance to ~30 cm, ~60 cm and ~3.4 m for the near-field (NF), mid-field (MF) and far-field (FF) detector positions, respectively. Furthermore, additional to the initial objectives, in taking full advantage of the electro-thermal mechanical testing (ETMT) facility, the original experimental scope was broadened to the understanding the kinetics of phase trasnformation with respect to load conditions. Currently, several nanuscripts resulting from this beamtime are being prepared for publication.

Martensite tetragonality (B. Kim & A. Navarro)

Martensite is super-saturated in carbon, and this gives rise to tetragonality within the ferrite unit cell. In order to account for the tetragonaltiy of ferrite, Rietveld refinement was carried out using the tetragonal form (I4/mmm). An example of the thermal cycle is shown in Figure 1.

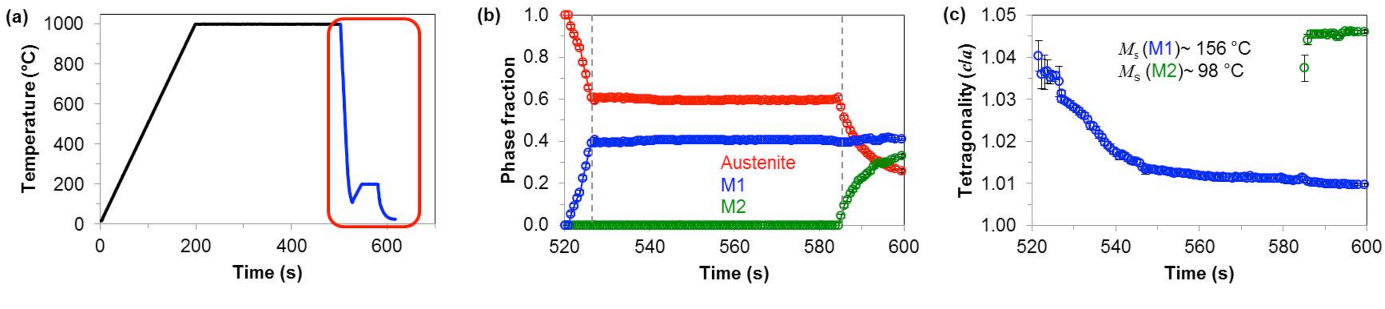

Figure 1- (a) An example of heat treatment recorded by the thermocouple, showing the section of the thermal cycle that focuses on martensite. (b) Phase fraction evolution involving the formation of two ferrite products (M1 and M2). (c) Tetragonality evolution within the ferrite product phases (M_s = martensitic start temperature).

The changes in the lattice parameters throughout the thermal cycle gives information on the carbon redistribution process. For the particular example shown in Figure 1, the study was focused on the carbon competition reactions in complex multiphase steels, in the context of carbide precipitation *vs.* austenite stabilisation via carbon enrichment.

The use of different sample-to-detector distances (B. Kim & A. Navarro)

In order to gain resolution some experiments were repeated by incresing the sample-to-detector (d) configuration, as outlined in Figure 2.

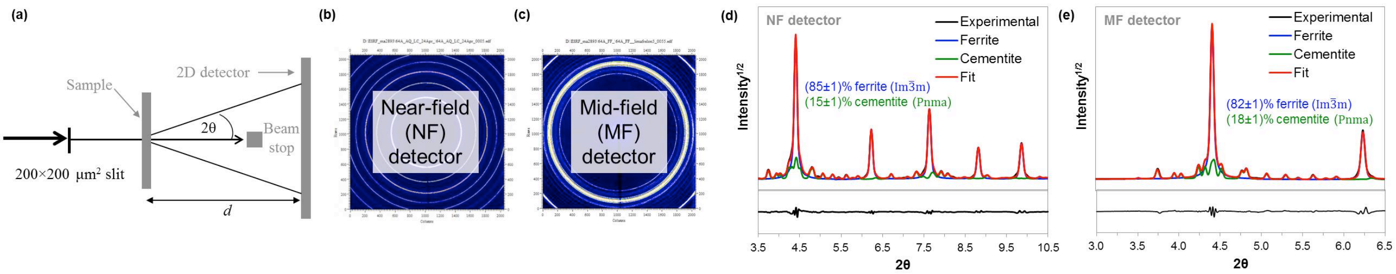

Figure 2 - (a) Sketch of sample set-up. Example of 2D diffraction image acquired using the (b) near-field and (c) mid-field detector positions. (d) and (e) show the respective 1D pattern analysed by Rietveld refinement.

There is some discrepancy in the phase fractions calculated using the two spectra, owing to the reduced number of ferrite peaks in the MF spectrum. Nevertheless, the cementite peaks were better resolved by using the MF detector position. The lattice parameter evolution will be cross-checked.

The effect of load the FCC to BCC phase transformation (P. Efteftekharimilani & R. Huizenga)

The effects of mechanical loading on the FCC to BCC phase transformations of an advanced high strength steel during cooling using *in situ* synchrotron diffraction was studied. Time-temperature-load synchrotron X-ray diffraction patterns were gathered. The volume fractions of the phases during cooling and simultaneous loading were calculated. In addition, volume fractions of retained austenite at room temperature under different loading conditions were obtained. The results show that applying a load during cooling of the FCC phase significantly increases the volume fraction of a BCC phase before the start of the martensitic transformation, Figure 3. The volume fraction of retained austenite at room temperature varies in different samples.

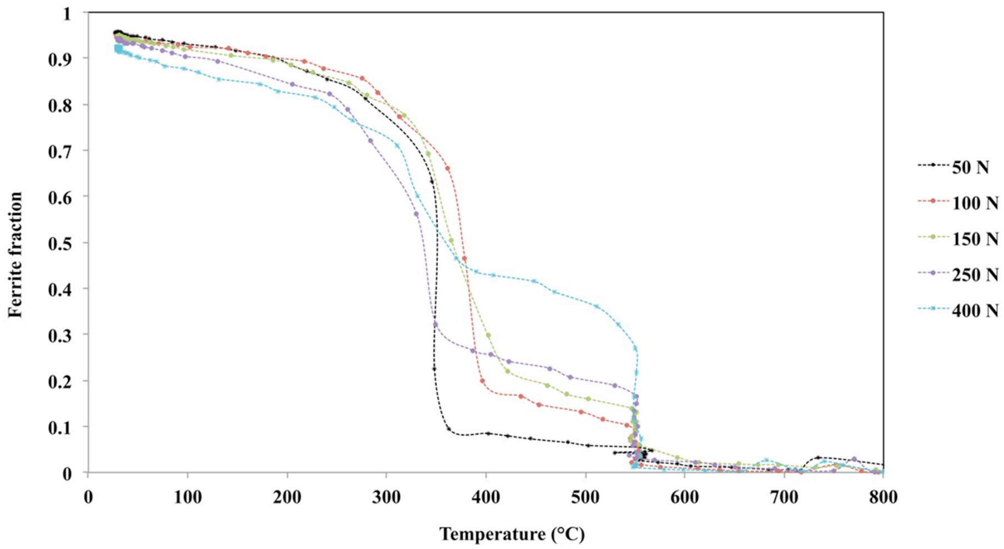

Figure 3- BCC phase volume fraction as a function of temperature during cooling for different heat treatment cycles.

Effect of tempering on the strain partitioning of martensite/austenite microstructures by *in situ* synchrotron (J. Hidalgo & R. Huizenga)

Quenching and Partitioning (Q&P) steels form part of the third generation of advanced high strength steels. Conventionally, Q&P steels focuses on designing austenite stability for strength and ductility. In a previous study it was revealed that although the work hardening at high-strains is mainly influenced by austenite stability, the work hardening behaviour at low-strains is mostly controlled by the martensite phase in Q&P microstructures and has an impact in the overall mechanical response of the steel. It was also evidenced that the work hardening at low-strains can be engineered by martensite tempering. These conclusions were based on the mechanical response of the composite material and discussed assuming differences in the martensite strength due to changes in the measured dislocation densities and carbon contents.
The behaviour of individual phases during mechanical testing needs to be studied for a better understanding of how differences in martensite strength (controlled by tempering conditions) influence in the work hardening of material. This was done by (i) the strain partitioning of different phases due to differences in the individual work hardening, and (ii) the effect of austenite induced mechanical transformation. Moreover, it was relevant to know how the strength of surrounding martensite affect the mechanical stability of retained austenite and implicitly the work hardening at different strain levels. This motivates the use of synchrotron to study individual phases contribution to the material mechanical response at different strain levels.
Three different materials with microstructures of specific characteristics were created and their mechanical response was resolved *in situ* in the ETMT in combination with synchrotron X-ray diffraction (see Figure 4).

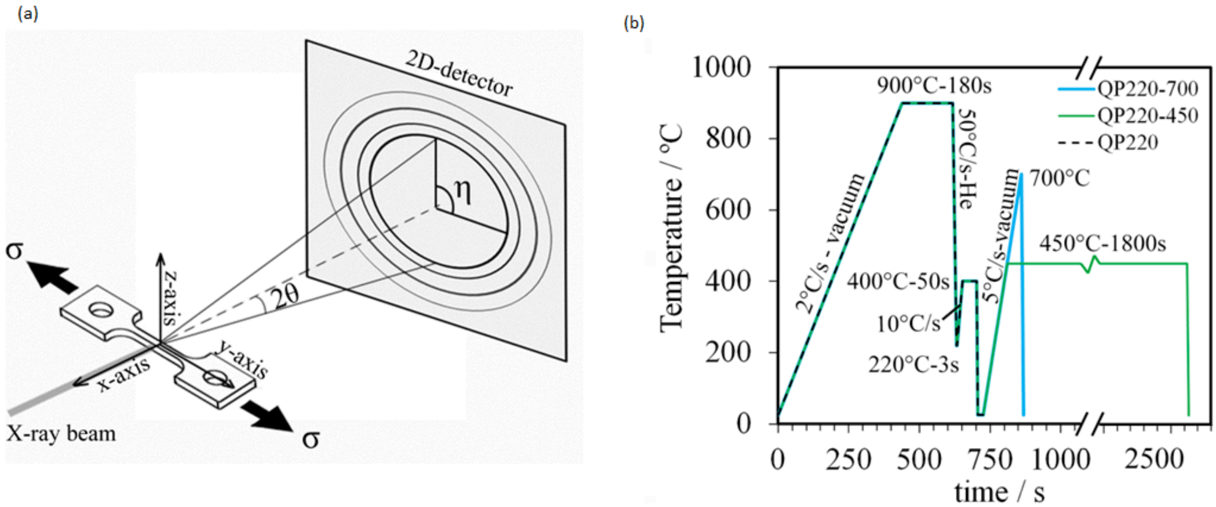

Figure 4 – (a) Set-up used in the ETMT. (b) Three heat-treatments of the different QP materials studied.

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