ESRF	**Experiment title:** Combinatorial study of the influence of chemical composition on phasetransformations in steels using compositionally graded materials and in-situ HEXRD	**Experiment number:** MA-4199
Beamline: ID11	**Date of experiment:** from: 09/11/2018 to: 13/11/2018	**Date of report:** 13/09/2021
Shifts: 12	**Local contact(s):** Pavel SEDMAK	*Received at ESRF:*

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Report:

This experiment aimed at mapping the transformation kinetics of the austenite-to-ferrite (γ→α) phase transformation in the compositional space of steels. It relies on a combinatorial methodology combining compositionally graded samples and in situ high energy X-ray diffraction HEXRD. The samples were submitted to a specific heat treatment designed to induce the transformation, as shown in Fig. 1a.

The experiment was carried out at ID11 with an 87 keV monochromatic beam. The thermal schedule was achieved using the ETMT stress rig and the temperature was monitored using S-type thermocouples spot-welded onto the samples. The composition gradient was positioned perpendicularly to the electric current used for heating the sample in an attempt at minimizing the thermal gradient in the region probed by the X-ray beam as shown in Fig. 1b. The ETMT was moved up and down during the heat treatment in order to scan the sample in the beam. 10 compositionnally graded samples were investigated at up to 4 different temperatures.

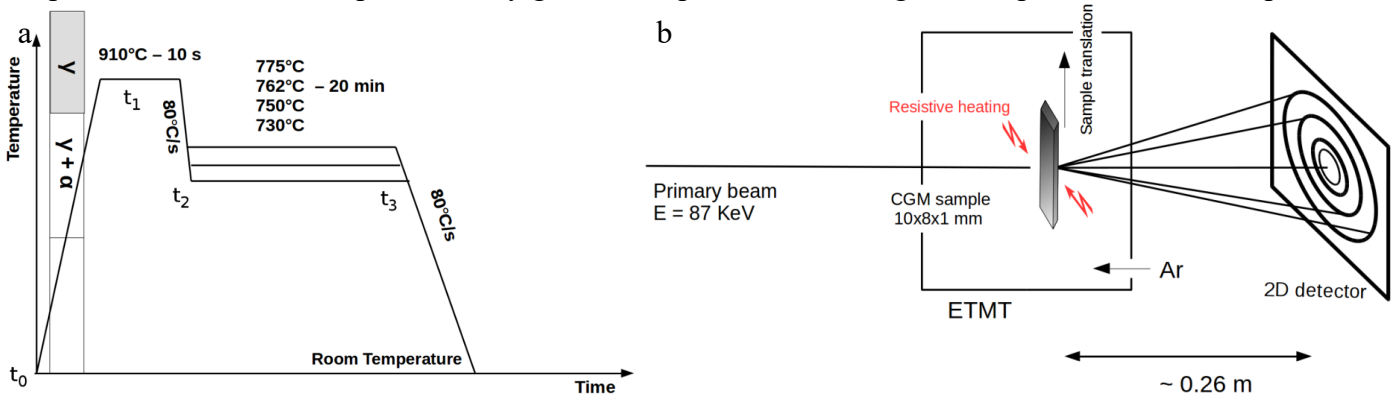

Fig. 1 : a) Thermal schedule applied to the compositionnally graded samples during the in situ experiment. b) Schematic representation of the experimental setup.

The experiment suffered a number of problems as detailed below but it still led to the publication of one article [1]. It was also decisive in finalizing the methodology, which later led to a highly successful experiment [2]. The PhD project it was part of has been succesfully defended [3].
As mentioned above, several problems were encountered during the experiment. Below is a summary of some observations. More details can be found in [1].

- Grain size

The experimental conditions led to grain growth to the extent that the patterns no longer showed continuous rings but dotted circles as shown in Fig. 2a. The spots were high intensity and caused detetector saturation. This led to significant shouldering on diffraction peaks after integration as seen in Fig. 2b.

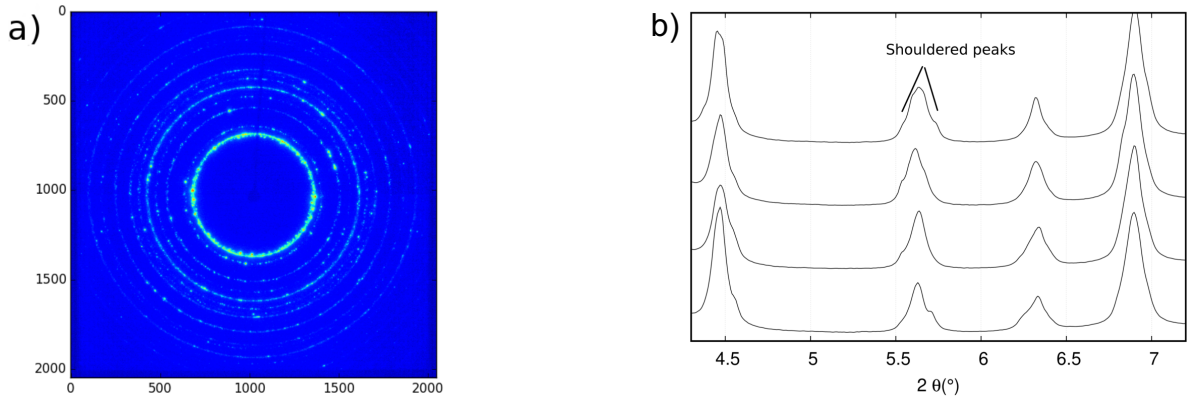

Fig. 2: a) Typical diffraction patterns obtained during the transformation. b) Examples of patterns after circular integration exhibting significant shouldering.

- Decarburization

Samples were found to exhibit significant decarburization after even just one heat treatment, as shown in Fig. 3. This decarburization is thought to be related to the Ar flushing in the ETMT, which was insufficient to properly protect the sample during the experiment. Decarburization changes the thermodynamics of the alloy and causes nucleation and growth of additionnal ferrite at the surface, which otherwise would not occur, seriously complicating data interpretation.

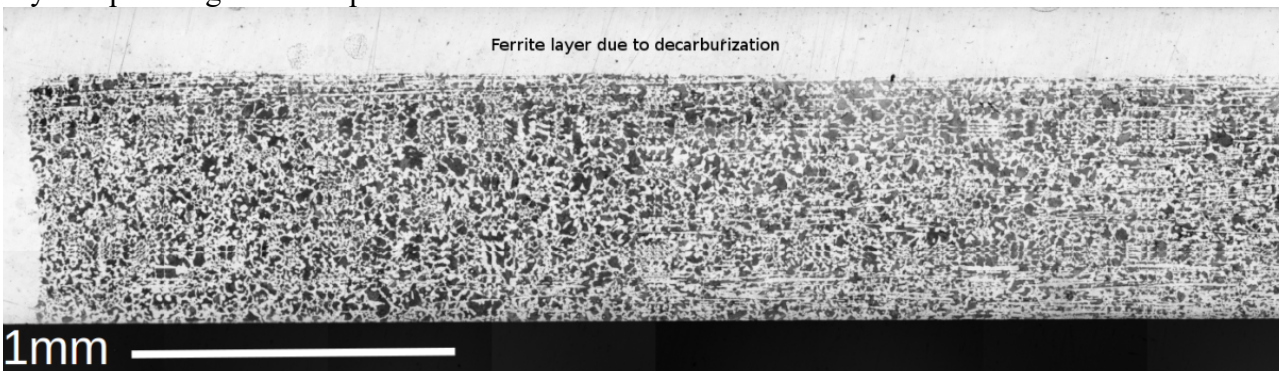

Fig. 3: Cross-section of a Fe-C-Mn/Fe-C-Mo compositionnally graded sample exhibiting a large ferrite at its surface due to decarburization after a heat treatment in the ETMT.

This experiment was instrumental in identifying and correcting these issues. They were solved mostly by using a special lamp furnace designed at Institut Jean Lamour [4] with a rotary sample holder. The investigation that had been started with this experiment was later sucessfully completed at DESY PETRA P21.2 [2].

References:

[1] I.-E. Benrabah, H.P. Van Landeghem, F. Bonnet, F. Robaut, A. Deschamps, Quantum Beam Sci. 4 (2019) 1–17.
[2] I.-E. Benrabah, F. Bonnet, B. Denand, A. Deschamps, G. Geandier, H.P. Van Landeghem, Appl. Mater. Today 23 (2021) 100997.
[3] I.-E. Benrabah, Développement d'alliages métalliques à gradient de composition pour l'exploration combinatoire des microstructures, PhD thesis, Université Grenoble Alpes, 2021.
[4] B. Denand, M. Dehmas, E. Gautier, C. Bonnet, G. Geandier, J.-P. Sarteaux, Four d'analyse Portable Pour Ligne de Rayonnement, WO2019081266A1, 2019.