Report on the experiments performed at ESRF – Beamtime MA-1090

Subject: In-situ characterization of the bainitic transformation kinetics in carbide-free bainitic steels

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The aims of this experiment were to perform in situ characterizations of the bainitic phase transformation kinetics under constant stress or not in carbide-free bainitic (CFB) steels, using high energy X-ray diffraction. We performed several experiments on four CFB steels, with different carbon content ranging from 0.1%C to 0.5%C. Tensile tests at room temperature have also been achieved in order to confirm the existence of a TRIP (TRansformation Induced Plasticity) effect in these grades.

The characterization of the phase transformation kinetics needed a specific thermomechanical cycle to be applied to the specimen, while structural information was gathered simultaneously by X-ray diffraction. Thanks to the previously gained experience from MA-861, and the ETMT device from Instron available on ID15 beamline, several heat treatments have been performed with or without applying a constant stress on the specimen during the bainitic phase transformation. The ETMT device allowed reaching high temperatures (above 1200°C) and high heating and cooling rates thanks to a resistive furnace controlled by DC current (maximum current = 400 A). The stress were applied via an electric actuator (maximum load = 3 kN). The tensile specimens were designed with the specificities of the device in mind. Moreover, since these grades exhibits large grain size at high temperatures, we needed analyzing specimens with large width of 4 mm.

During our previous experiment MA-861 on the ID11 beamline, we encountered several problems to fit the diffraction data by the Rietveld method. The grain size in the austenitic domain was indeed too large to acquire continuous diffraction rings, and some very intense spots were acquired instead. Therefore, the analysis of the diffraction data by the Rietveld method were difficult to perform because of the poor quality of the diffraction data, and it led to bad statistics and a great source of error to calculate the evolution of the cell parameters. Therefore, one of our main concerns during the experiment MA-1090 was to reduce the average grain size in the austenitic domain to acquire complete diffraction rings, and to further confirm the cell parameters evolution we observed during the experiment MA-861. We achieved this goal by reducing the dwell time of the austenitization condition as shown in the Fig. 1.

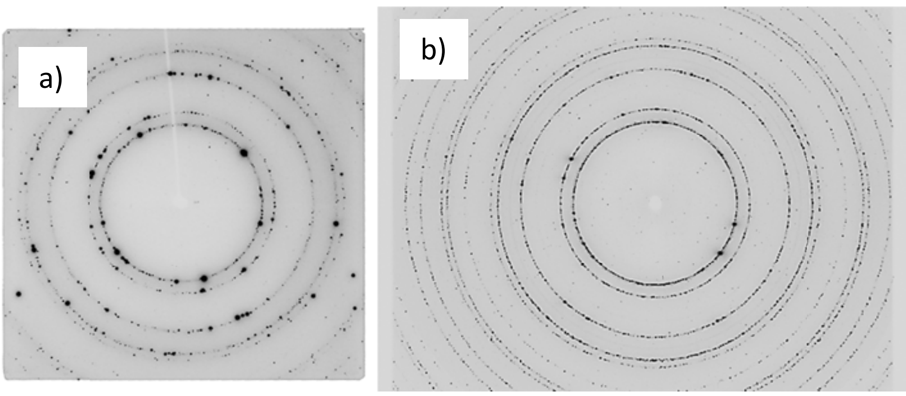

Fig. 1: Diffraction rings acquired at the end of the austenitization. a) MA-861, b) MA-1090

During the beam time, experiments were conducted in various conditions. We varied the austempering conditions (austempering temperatures ranging from Ms – 50°C to Ms + 50°C, and dwell times ranging from one hour to 15 hours in the case of the grades with high carbon content). Conclusive data were acquired, which allowed us to compare the volume fraction of the retained austenite, as well as its carbon content, at the end of the bainitic transformation for all grades at all austempering conditions. The evolution of the cell parameters of the austenitic and ferritic phases were observed and compared to the previous data acquired during the MA-861 experiment. Concerning the evolution of the austenitic phases, results are similar between the two experiments, and they showed a continuous increase of the cell parameter during the phase transformation. It is due to an enrichment of carbon in the austenite, and confirmed by applying a Vegard's law to the evolution of the cell parameter and to the evolution of the carbon content in the austenite. On the other hand, the evolutions of the ferritic cell parameter of the two experiments showed dissimilar results. In the case of MA-861, the ferritic cell parameter decreased during the phase transformation, whereas it increased in the case of MA-1090, as shown in Fig. 2.

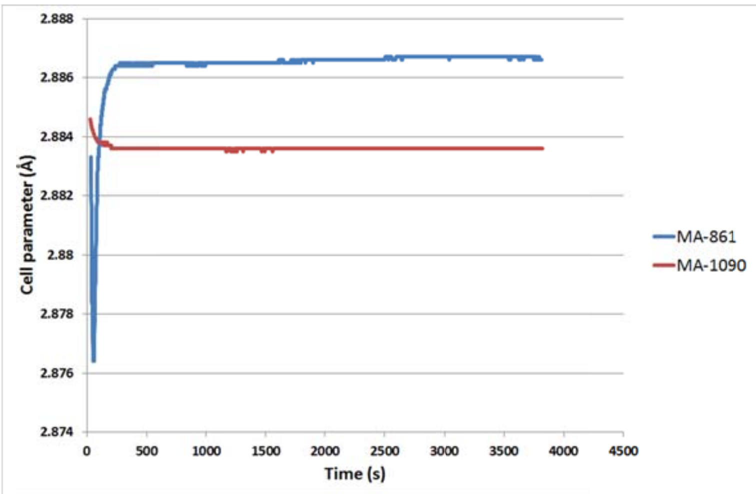

Fig. 2: Comparison of the evolution of the ferritic cell parameter between the two experiments MA-861 and MA-1090 (grade with 0.2%C austempered at Ms)

The evolution observed during the experiment MA-1090 seems to be more accurate than the one observed in the experiment MA-861. Indeed, no large fluctuations are observed at the beginning of the transformation, and this evolution is consistent with the fact that ferrite grows in compression in the γ →α phase transformation. Moreover, some authors have reported similar evolutions in the bainitic transformation[1,2]. Therefore, the evolutions of the ferritic cell parameter observed in the experiment MA-861 are considered to be wrong, and is attributed to the poor statistics resulting from the acquisition of discontinued rings at the beginning of the phase transformation.

We also managed to perform in-situ characterization of the bainitic transformation under constant stress. The applied stress ranged from 50 MPa to 150 MPa in order to not deform plastically the austenite before the phase transformation at low stress, and to deform it plastically at high stress.

[1] HJ Stone, MJ Peet, HKDH Bhadeshia, PJ Withers, SS Babu and ED Specht, Proc. of the Roy. Soc. A 464 (2008), pp. 1009

[2] X Chen and E Vuorinen, ISIJ International 49 (2009), pp. 1220

Results showed an increase of the transformation kinetics as well as a higher volume fraction of bainitic ferrite at the end of the transformation. Tensile tests have also been performed at room temperature on these grades. Data confirmed the existence of a TRIP effect which led to high uniform elongation and a depletion of volume fraction of austenite near the fracture. A load of 2 kN was sufficient to apply a stress of 150 MPa on a specimen with a section of 1 mm^2.

During the beam time we also encountered several problems with the plate detector. We have indeed observed intensity variation on the integrated diagrams made from the data acquired with the Pixium detector. This behavior has been observed on all series of tests and seems to only affect the 0.4 s exposure mode. After investigations on the beamline, the problem comes from a fast shutter with no reproducible movements. Moreover, we have encountered another issue with the detector after a crash of the server. The images displayed on the computer during the acquisition are observed with a shift of 5 images, which means that the image displayed was in fact an image acquired before.

From our tests some comments could pointed out concerning the ETMT device:

- The ETMT device is very efficient to control thermomechanical cycles, although the temperature measured by the thermocouple has to be taken with care since it measures an additional tension from the thermocouple itself. We have observed a gap of ± 30°C from the real temperature at the range of austempering temperature we worked with.
- The ETMT device is efficient to perform tensile tests, although the tensile specimens have to be design with the specificities of the ETMT device in mind (max load of 3 kN, and nonlinear response above 1.5 kN).