ESRF	**Experiment title:** 3D-XRD Study of the Role and Mechanism of Sympathetic Nucleation in the Austenite-to-Ferrite Transformation in Steel.	**Experiment number:** ME-1009
Beamline: ID11	**Date of experiment:** from: 14 April 2005 to: 19 April 2005	**Date of report:** 01 Sept 2005
Shifts: 15	**Local contact(s):** Jonathan Wright	*Received at ESRF:*

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Report:

The primary goal of this project was to use *in-situ* three-dimensional X-ray diffraction microscopy (3D-XRD) to reveal the role of sympathetic nucleation in the ferrite sideplate precipitation reaction in steels. Sympathetic Nucleation (SN) is a heterogeneous nucleation process in which a crystal of the precipitate phase forms at the interface between an earlier-formed precipitate crystal and the matrix. Although SN is often presumed to be a near-ubiquitous mechanism, there has never been a direct observation of the SN process during solid state phase transformations.

A secondary aim of this project was to develop procedures to correlate the data between three different analytical techniques. These techniques are complimentary, with the 3D-XRD box-beam scanning approach forming the required link between the ferrite crystallite orientation data from the *in-situ* 3D-XRD experiment and the ferrite spatial distribution data as determined by serial sectioning and 3D reconstruction.

Results

The alloy selected for this study, Fe-0.4%C-7%Ni, produces Widmanstatten ferrite precipitates with the appropriate morphology at temperatures between 500 and 550 °C. The formation of these precipitates is initially rapid, yet decreases dramatically within the first couple hours. Thus, the initial stages of the transformation were followed *in situ* with the fast readout Frelon detector and then, after the reaction rate had slowed sufficiently, box beam scans were conducted to spatially locate the diffracting volumes and graindex scans were performed to identify the crystallographic orientation of those precipitates. The samples were heated under vacuum to approximately 1000 °C for at least 1 hour, then cooled to just above

the transformation temperature. Because the sample temperature deviated in a non-systematic manner from the furnace temperature, the transformation temperature was identified in each specimen by gradually cooling the sample until the low-temperature precipitates started to form. The sample was then cooled further to a specific undercooling and the precipitation reaction was followed *in situ*. After initial beam alignments and calibration of the furnace, we successfully tracked the Widmanstatten ferrite precipitation reaction, *in-situ*, in three samples at approximate undercoolings of 15 °C, 22 °C, and 30 °C below the 545 °C transformation temperature.

The results demonstrated that distinct crystallites, which exhibit sharp diffraction reflections, always form anew as would be expected through the SN mechanism (see Fig. 1). If the new crystallites had formed by the most likely competing mechanism, polygonization (in which the small crystallites form through recovery of a large, highly dislocated precipitate), the sharp diffraction reflections would develop from within pre-existing diffuse reflections. Thus this experiment satisfied the primary goal by indicating, through direct *in-situ* observation, that the ferrite precipitates did not form by polyganization but instead formed via a sympathetic nucleation mechanism.

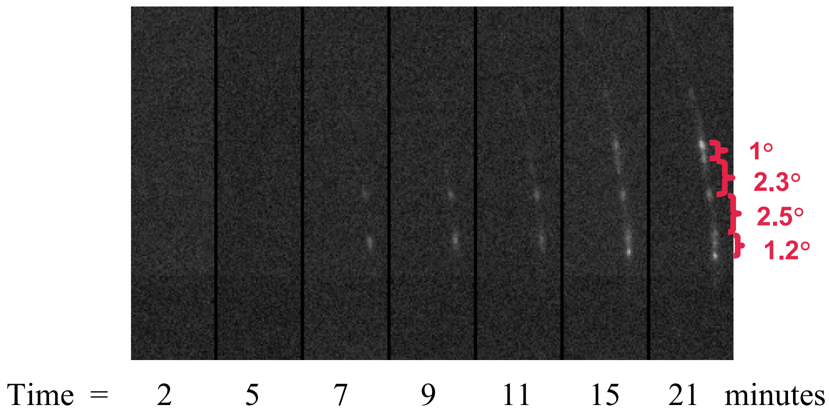

Fig. 1. A time series of diffraction patterns obtained at the largest undercooling (at approximately 510 °C) showing the appearance of sharp, new diffraction spots, indicating sympathetic nucleation. The alternative mechanism, polygonization, predicts the evolution of these spots from pre-existing diffuse reflections.

These experiments yielded some additional new and interesting results that are still being analyzed. One of these observations is that individual diffraction reflections became elongated with time in both theta and eta, indicating the development of deformation or elastic strain within the diffracting volumes. This phenomenon was displayed by both the decomposing matrix and the developing precipitates and appears to be an indication of the large transformation strain present in this reaction. Also, the precipitate reflections appear on two closely-spaced yet separate diffraction rings, indicating a discrete change in lattice parameter. Further analyses of this phenomenon is underway and could provide important new insight into the austenite-to-ferrite transformation in steels.

About 700 Gb of images were acquired during this 5 day beam time, and analysis of this vast data set is still in progress. Thus, the secondary aim of linking 3D-XRD kinetics data - though the box-beam scanning approach - to serial sectioning data, awaits further analysis of the data.

The results of this research will be written up for publication in the peer-reviewed scientific literature upon completion of the data analysis.