	Experiment title: Time-resolved austenite decomposition kinetics in high-carbon bearing steels	**Experiment number:** MA-850
Beamline: ID 15	**Date of experiment:** from: 11/03/2010 to: 16/03/2010	**Date of report:** 28/01/2014
Shifts: 15	**Local contact(s):** V. Honkimäki	*Received at ESRF:*

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Report:

Through-hardened steel SAE 52100 (DIN 100Cr6) is widely used in bearing applications due to its high strength and resistance to rolling contact fatigue. After the manufacturing process, this material presents a complex multiphase microstructure containing either bainitic-ferrite or martensite, finely-dispersed ε/η transition carbides, cementite and retained austenite. Under service conditions, significant microstructural changes take place in the bearing: the decay of martensite into ferrite, the decomposition of the metastable austenite and changes in texture and in the residual carbides. The austenite evolution with time is considered as a useful local probe of the microstructural changes occurring during the service life of the bearing.

The aim of this experiment was to assess the combined effect of temperature and applied stress on the austenite decomposition kinetics in SAE 52100 steels. For this purpose, we performed an in-situ X-ray diffraction experiment at the ID15 beam line of the ESRF [1]. We used an intense monochromatic X-ray beam with an energy of 69.95 keV (λ = 0.1772 Å). The diffracted intensity was collected on a 2D Pixium 4700 detector placed behind the sample. The chemical composition of the studied material was: 1.44Cr-0.96C-0.34Mn-0.22Cu-0.20Si-0.05Mo-0.011Al-0.007P-0.002S (wt.%). We used cylindrical dog-bone-shaped tensile specimens with a gauge length of 10 mm and a diameter of 1 mm. The tensile specimens were mounted on a 2-kN micro-tensile rig placed on the Huber translation table of ID15. The cartridge heaters mounted in the clamps of the rig allowed us to heat the samples above room temperature.

The experiment was designed to provide us with: (1) average information about the behaviour of the constituent phases from the powder analysis of the diffraction data, and (2) information about the structural characteristics and decomposition kinetics of the individual metastable austenite grains. To achieve both goals, the X-ray beam was focused down to $5 \times 25 \ \mu m^2$ using parabolic focusing lenses. Before heating, each sample was continuously rotated along its cylindrical axis in steps of $\Delta\omega$ = 0.5° while recording the diffraction patterns. We covered a total angular rotation range of 90° on both sides of the tensile rig. The diffraction patterns were collected at five sample heights. The summation of the 2D patterns recorded at five different heights for a given step in ω yielded an effective beam size of $25 \times 25 \ \mu m^2$. The samples were then heated under vacuum to a selected temperature (T_{exp}) in the range of 200-235°C, and kept at a constant temperature for 2-6 h to complete the austenite decomposition. During this process we collected the diffraction patterns for the five sample heights continuously, covering a total angular range of 10° in steps of

Δω = 0.5° on only one side of the tensile rig. Two equivalent samples were measured at each of the final temperature: one at zero applied stress and one under an applied stress of σ = 295 MPa.

The powder analysis was carried out by summing the 2D patterns in bunches of hundred (20 patterns for each ω-scan × 5 sample heights). We then integrated each of the summed 2D pattern and performed a Rietveld analysis of the resultant 1D patterns. This analysis yielded the values of the volume fraction and lattice parameter of the austenite phase and the surrounding ferritic matrix. At about 30 min the austenite fraction starts to decrease, and its decomposition progresses over time until it has disappeared completely (see Fig.1). An increase in the transformation temperature or the presence of an applied stress causes a significant acceleration of the decomposition kinetics.
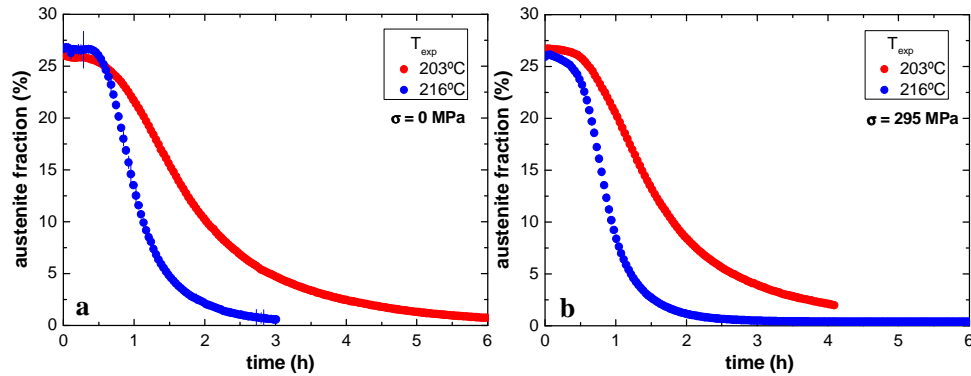

Fig. 1. *Time evolution of the austenite fraction at two temperatures for (a) σ = 0 MPa and (b) σ = 295 MPa.*

At temperatures lower than 160 °C, the temperature dependence of the lattice parameter is governed by the coefficient of linear expansion for each phase (Fig. 2a and b), as no phase transformation takes place in this temperature range. Above 160°C the deviations from the lattice thermal expansion are attributed to a change in the carbon content of each phase (Fig. 2c and d). The occurrence of carbon enrichment in the metastable austenite takes place when its decomposition has not yet started. The additional carbon enrichment can be attributed to the partition of carbon from martensite into austenite. This carbon partitioning takes place simultaneously with the precipitation of transition carbides inside the martensite phase.
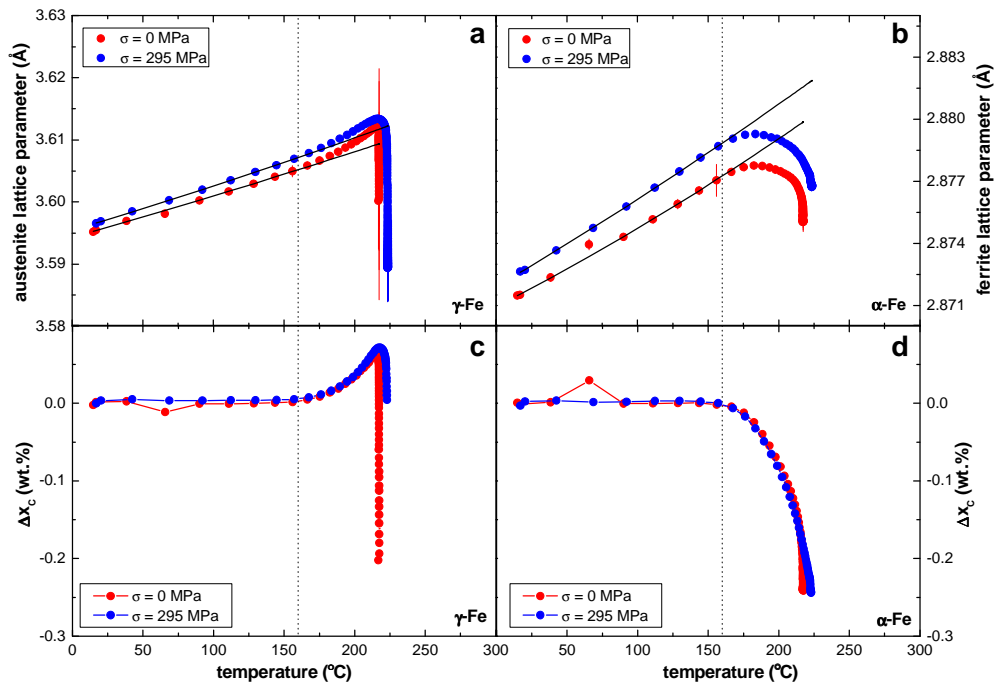

Fig.2. *Temperature dependence of the lattice parameter of (a) austenite and (b) ferrite during heating, at σ = 0 MPa and σ = 295 MPa. (c) and (d) correspond to the change in carbon content of both phases after correcting for the lattice thermal expansion (solid line in (a) and (b)).*

The analysis of the austenite decomposition at the level of individual grains is currently in progress.

Reference:

[1] E. Jimenez-Melero, R. Blondé, M. Sherif, V. Honkimäki, N.H. van Dijk, Acta Materialia 61 (2013) 1154-1166.