ESRF	**Experiment title:** A Synchrotron Look at the thermally-induced Martensitic transformation in TRIP steels	**Experiment number**: ME-771
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Report:

Recently, low-alloyed transformation induced plasticity (TRIP) steels have attracted a growing interest for their high strength and good formability. TRIP steels possess a multiphase microstructure containing ferrite (bcc α-Fe), bainite, and (metastable) austenite (fcc γ-Fe). The so-called TRIP effect in these steels arises from a martensitic transformation of the metastable retained austenite phase induced by external stress.

In the current study we performed in-situ X-ray diffraction measurements at ID11 to monitor the stability of retained austenite in TRIP steels during cooling. Part of the retained austenite phase is unstable when the material is cooled from room temperature to 100 K and transforms into martensite. In these measurements the austenite fraction and the average interstitial carbon concentration was monitored as a function of temperature for TRIP steels with different compositions and bainitic holding times.

Three TRIP steels (0.2 wt.% C, 1.5 wt.% Mn, 0.25 wt.% Si) with different Al and P concentrations were studied: $Al_{0.4}$ (0.4 wt.% Al), $Al_{0.4}P_{0.1}$ (0.4 wt.% Al and 0.1 wt.% P), and $Al_{1.8}$ (1.8 wt.% Al). The samples were heated in a salt bath to the intercritical holding temperature (1000-1200 K) to form a two-phase material containing austenite and ferrite. The intercritical holding temperature was selected to obtain a maximum fraction of retained austenite in the final material. After the intercritical annealing the samples were transferred to a second salt bath, held at the bainitic holding temperature of 673 K. After bainitic holding times of 20, 60, 240, and 1000 s the samples were quenched in water.

X-ray diffraction measurements were performed using the three-dimensional X-ray diffraction microscope (3DXRD) at the beam line ID11. A monochromatic X-ray beam with an energy of 80 keV (wavelength of 0.155 Å) and a beam size of 39×39 μm^2 illuminated the 0.50 mm diameter of the cylindrical sample. The sample was cooled by a nitrogen gas cryostream cooler (Oxford Cryosystems) from 300 K down to 100 K in steps of 20 K. After each temperature step the displacement of the sample with respect to the beam was monitored by scans in the horizontal and vertical sample positions, and the sample rotation angle. The intensity of a selected reflection from a characteristic individual ferrite grain in these scans was used to retrace exactly the same illuminated sample volume during the entire experiment.

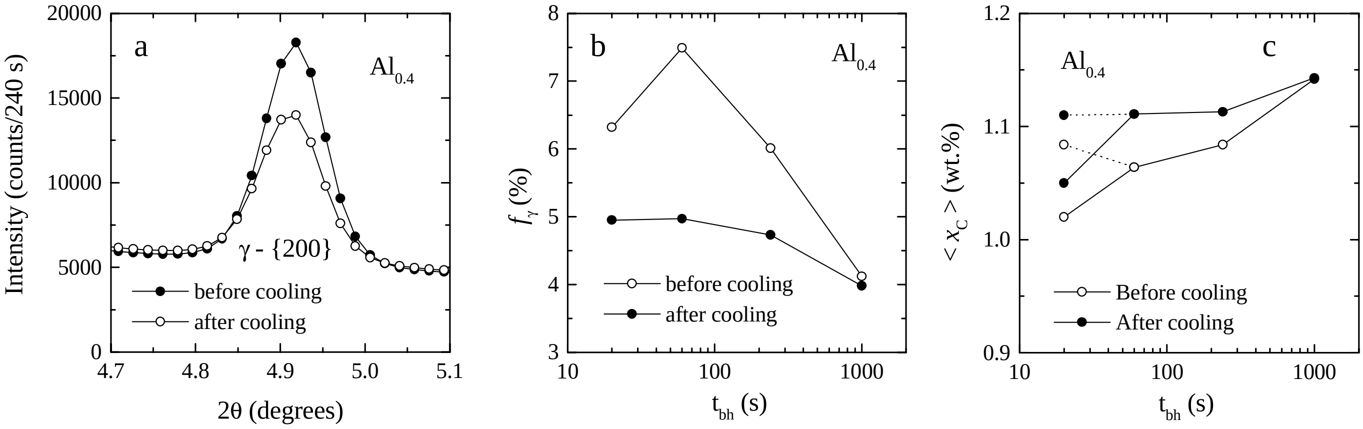

Figure: (a) The {200} austenite powder peak before and after cooling to 100 K. Part of the austenite has transformed into martensite and the average scattering angle 2θ has decreased. (b) The fraction retained austenite as a function of the bainitic holding time t_{bh} before and after cooling to 100 K. (c) The average interstitial carbon concentration in the austenite phase before and after cooling to 100 K. The dashed line assumes the presence of a compressive strain caused by the presence of martensite in the sample with t_{bh} = 20 s before cooling to 100 K.

A powder analysis of the diffraction data reveals a martensitic transformation of part of the retained austenite during cooling. The scattered intensity at room temperature is compared before and after cooling the sample to T = 100 K. A clear reduction in intensity is observed for all austenite reflections, indicating that part of the retained austenite has indeed transformed during cooling. Furthermore, it is interesting to note that the position of the austenite peaks is shifted to lower scattering angles after cooling to 100 K, and subsequent reheating to room temperature. This indicates that the average austenite lattice parameter has increased due to a martensitic transformation of the retained austenite grains with the lowest interstitial carbon concentration.

The prime results of the powder analysis are:

1. Part of the initial retained austenite formed in the TRIP steels was found to transform into martensite during cooling, while another part remained stable down to the lowest temperature reached. The fraction of retained austenite that transforms during cooling strongly depends on the bainitic holding time and the composition. For the longest bainitic holding time virtually no retained austenite transformed during cooling. Apparently, only the most stable austenite grains remain after the long bainitic holding time of t_{bh} =1000 s.
2. Austenite grains with a lower carbon concentration have a lower stability during cooling for all studied samples. The average carbon concentration in the untransformed retained austenite after cooling was higher than the average carbon concentration before cooling.
3. For the $Al_{0.4}$ steel with the shortest bainitic holding time of t_{bh} = 20 s the temperature stability of the austenite phase can only be understood when a compressive strain of $\Delta\varepsilon_\gamma$ = 0.08% in the austenite phase is assumed.

The analysis of the thermal stability of individual austenite grains is currently in progress. First results indicate that the unstable austenite grains show one or several jumps in grain volume due to a martensitic transformation during cooling. Again the data suggest that grains with a lower carbon concentration (larger lattice parameter) have a lower stability.

Publication resulting from the experiment:

"Thermal stability of retained austenite in TRIP steels studied by synchrotron X-ray diffraction during cooling", N.H. van Dijk, A.M. Butt, L. Zhao, J. Sietsma, S.E. Offerman, J.P. Wright, and S. van der Zwaag,
Acta Materialia 53 (2005) 5439-5447.