| ESRF | **Experiment title:** Mechanically-induced martensitic transformation of individual grains in low-alloyed TRIP steels | **Experiment number**: MA-354 |
|---|---|---|
| **Beamline**: ID11 | **Date of experiment**: from: 21/02/2008 to: 26/02/2008 | **Date of report**: 23-02-2009 |
| **Shifts:** 15 | **Local contact(s)**: J.P. Wright | *Received at ESRF:* |

**Names and affiliations of applicants** (* indicates experimentalists):

Enrique JIMENEZ-MELERO*, TU Delft, Applied Sciences, The Netherlands

Niels VAN DIJK*, TU Delft, Applied Sciences, The Netherlands
Lie ZHAO*, TU Delft, Materials Science & Engineering, The Netherlands
Jilt SIETSMA, TU Delft, Materials Science & Engineering, The Netherlands
Sybrand VAN DER ZWAAG*, TU Delft, Aerospace Engineering, The Netherlands
Lawrence MARGULIES*, Risø DTU, Materials Research Division, Denmark

## Report:

Low-alloyed transformation-induced plasticity (TRIP) steels are attracting an increasing attention due to their outstanding combination of strength and formability. Their room-temperature microstructure comprises three phases: ferrite, bainite and austenite. The austenite phase is retained in the TRIP microstructure in a metastable condition. The TRIP effect stems from the transformation of the metastable soft austenite into the stable hard martensite phase, induced by the presence of external stresses.

In this experiment, we have performed in-situ X-ray micro-diffraction experiments at room temperature, in order to assess the stability of the retained austenite as a function of the applied stress. For this purpose, a dedicated 2 kN tensile tester to perform synchrotorn diffraction experiments in transmission geometry was installed at the ID11 beamline. The aim was to correlate the observed austenite stability to its local microstructural characteristics before the transformation.

Two TRIP steels with the basis chemical composition of 0.2 wt.% C, 1.5 wt.% Mn, 0.25 wt.% Si, and differing only in the amount of aluminium (namely, 0.4 and 1.8 wt.% Al) were used for this experiment. The samples were heated in a salt bath to a temperature 1073 and 1173 K respectively, in order to obtain a ferrite/austenite microstructure. The intercritical holding time was 30 min in both cases. The samples were subsequently transferred to a second salt bath at a temperature of 673 K, where part of the intercritical austenite tranformed into austenite. The bainitic holding time was fixed to 60 s. The samples were then quenched in water to room temperature.

The in-situ X-ray diffraction measurements were performed using the three-dimensional X-ray diffraction microscope (3DXRD) available at the ID11 beam line. A monochromatic X-ray beam with an energy of 68.93 keV illuminated the TRIP sample placed in the clamps of the 2kN tensile tester located at the translation table of the beam line. Both cylindrical and flat samples were used in this experiment. Several microbeam sizes between 17×17 and 100×100 $\mu m^2$ were used in order to: (a) get a proper powder diffraction signal to assess the average characteristics of the austenite behaviour as a function of the applied stress, (b) to

illuminate only a few individual austenite and ferrite grains, so that the deformation/transformation mechanism of the individual austenite grains and the deformation of the surrounding ferrite grains could be analysed after each elongation step, (c) to be able to validate that the complete volume of the individual grains of interest was illuminated by the X-ray beam. A two-dimensional detector was placed behind the sample at a distance of 183 mm. The diffraction patterns were recorded continuously during a rotation of 0.5º along an axis parallel to the tester axis and perpendicular to the incoming beam. Equivalent rotation steps were repeated to cover an total angular rotation range of 90º, corresponding to two rotation scans separated by 180º. A tungsten wire inside a glass capillar was used to align the center of rotation of the tensile tester with respect to the beam. After each elongation step, both an intense ferrite reflection and a $Gd_2O_3$:S marker at the sample surface were used as reference to trace back the same illuminated sample volume. A $LaB_6$ standard was placed on the surface of the TRIP steel sample and its diffraction pattern was measured after each elongation step, aiming to assess the possible variations of the sample-to-detector distance during the tensile experiment.

A Rietveld refinement procedure has been adopted to analyse the room-temperature synchrotron diffraction data as a function of applied stress, after transforming the recorded two-dimensional diffraction data into one-dimensional data. This has allowed us to analyse simultaneously seven ferrite reflections and eight austenite reflections, some of which do overlap significantly with other reflections. Figure 1 shows the macroscopic response of the TRIP steel with 0.4 wt.% Al, together with the evolution of the austenite phase fraction and lattice parameter with strain. The former has been derived from the integrated intensities of the diffraction peaks, while the latter is determined via the refined peak positions. The experimental results indicate that for this TRIP material the metastable austenite starts to transform in the first steps of the mechanical tests, even in the elastic region. The increase of the austenite lattice parameter stems from both the transformation of the low-carbon grains into martensite and the effect of the applied stress on the interplanar lattice distances.

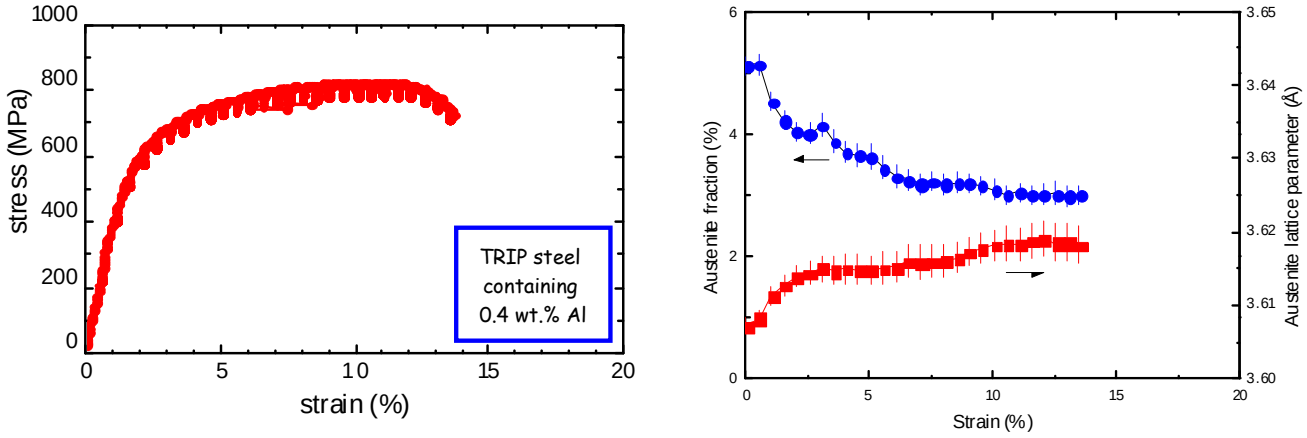


**Fig.1:** Macroscopic mechanical response of a TRIP steel containing 0.4 wt.% Al, together with the dependence of the austenite phase fraction and lattice parameter with strain.

The analysis of the mechanical stability of the individual austenite grains is currently in progress.