

## Improved Atom Probe Methodology for Studying Carbon Redistribution in Low-Carbon High- $M_s$ Lath Martensitic Steels

L. Morsdorf<sup>1</sup>, B. Gault<sup>1</sup>, D. Ponge<sup>1</sup>, C.C. Tasan<sup>2</sup> and D. Raabe<sup>1</sup>

<sup>1</sup> Max-Planck-Institut für Eisenforschung, Department of Microstructure Physics and Alloy Design, Düsseldorf, Germany.

<sup>2</sup> Massachusetts Institute of Technology, Department of Materials Science and Engineering, Cambridge MA, USA.

Nano-scale carbon redistribution from super-saturated interstitial solutions in bcc/bct iron-based matrices right after martensitic transformation is a crucial process that determines later macroscopic mechanical properties of high strength steels. Thus, numerous studies were dedicated to unravel the carbon relaxation path in lath martensite upon quenching and tempering, as e.g. reviewed by Speich and Leslie in 1972 [1]. Later, model Fe-Ni-C alloys with sub-zero martensite-start ( $M_s$ ) temperatures were intensely studied, in order to avoid any ill-defined autotempering during the quenching process. In these alloys room temperature ageing results in spinodal decomposition of the Zener-ordered iron-carbon solid solution, resulting in carbon-rich and carbon-poor alternating layers [2]. Yet, it was recently demonstrated that comparative studies on low-carbon low-alloyed steels with high  $M_s$  temperature not only face the autotempering dilemma, but also the heterogeneous distribution of autotempering states within those microstructures [3]. To overcome the latter aspect, the authors introduced a step-wise lift-out procedure for atom probe measurements from a single coarse lath (and thus single autotempering condition), but at different tempering conditions through in-between heat treatments (Figure 5 in [4]). The experimental details are given in [4].

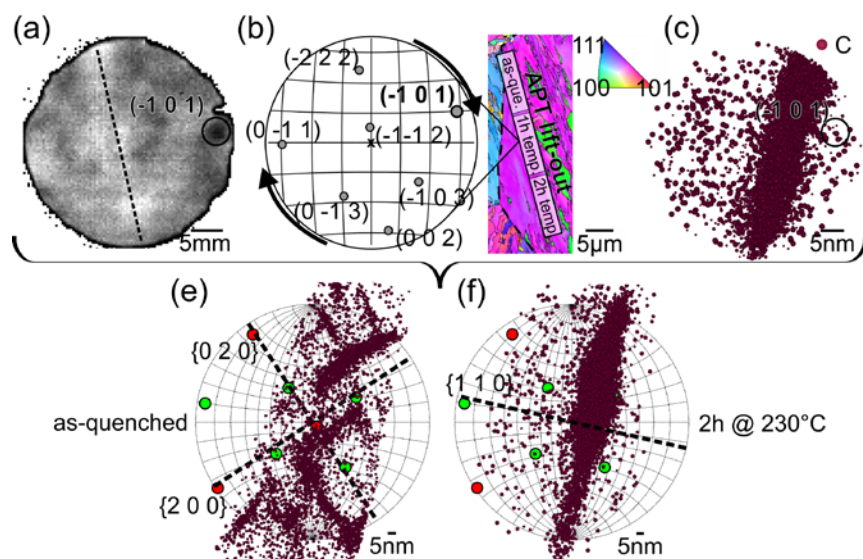
Now, we revisit this atom probe data set obtained on a Fe-2.51Mn-0.19Si-0.20Cr-0.225C (wt.%) alloy and conduct a correlative crystallographic investigation by using the electron back-scatter diffraction (EBSD) orientation information retrieved from the sample surface. Figure 1a shows a detector histogram map of the first ~ 2 million collected ions in an atom probe measurement taken after 2 h tempering at 230°C. Though the available crystallographic pole or zone axis information seems to provide rather limited information, still a spot with locally lower intensity than the surrounding region is clearly visible. This spot is interpreted as a {110} pole, which relates to the widest lattice spacing in bcc crystal structures and therefore enables to resolve lattice planes in atom probe data. Figure 1b shows how the stereographic projection of the average coarse lath orientation from the lift-out area is, first, limited to the atom probe detector field-of-view and then rotated to match the low intensity spot position with the (-101) pole. This is a unique rotation solution, as the orientation of the zone axis connecting the (-222) and (002) poles is also reflected in the detector map. This example shows how complementary orientation information from EBSD data enables to crystallographically inform atom probe data, even when the crystallographic information that can be derived from the detector information itself is insufficient. Figure 1c shows the carbon elemental map that is rotated (and flipped, which is necessary due to the reflectron in the atom probe instrument) into the same view as the detector map and stereogram. The reconstruction is optimized in comparison to [4] by fitting the periodic arrangement of iron atoms below the pole to the {110} bcc-Fe lattice spacing of 0.203 nm. The same reconstruction procedure is applied to all measurements and tempering conditions in this lift-out area owing to the homogeneous lattice orientation throughout the coarse lath.

The capability of this approach is demonstrated in Figures 1e and f, where the carbon distribution is analyzed for the as-quenched and for the 2 h tempered conditions, respectively. Both elemental maps are rotated in the same way to achieve perpendicular orientations of plate-shaped carbon-enriched zones, i.e. normal vectors lay perfectly in-plane. Respective habit planes with the martensite matrix are determined from the correspondingly rotated stereograms, yielding  $\{200\}$  matrix planes for carbon clusters after quenching (max. carbon concentration about 10 at.%) and a  $\{110\}$  plane for the precipitated carbide after tempering (max. carbon concentration close to 25 at.%). The first habit plane was observed for transition carbides in low-carbon steels [1] - however a large discrepancy in carbon content exists - while the latter plane is frequently observed as the matrix habit plane of  $\text{Fe}_3\text{C}$  cementite [1].

In conclusion, the step-wise lift-out procedure from a single martensite lath coupled with crystallographic atom probe analysis through EBSD correlation allows a systematic analysis of tempering reactions in high- $M_s$  steels. The crystallographic analysis is validated by well-known cementite precipitation along  $\{110\}$  martensite habit planes. Carbon distribution after quenching resembles the structure obtained after room temperature ageing in low- $M_s$  Fe-Ni-C alloys with a network of carbon clusters, yet parallel to  $\{200\}$  matrix planes.

#### References:

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**Figure 1.** Detector histogram map (a) and stereographic projection from electron backscatter diffraction analysis (b) showing  $(-101)$  pole position coincidence after  $260^\circ$  in-plane rotation; (c) carbon elemental map in reconstructed tip volume rotated (and flipped) to the same view as in (a) and (b). Crystallographic atom probe data, informed by EBSD analysis, reveals carbon clustering parallel to  $\{200\}$  matrix planes after quenching (e) and carbide precipitation with  $\{110\}$  matrix habit plane after 2 h tempering at  $230^\circ\text{C}$  (f).