



# Segregation of boron at prior austenite grain boundaries in a quenched martensitic steel studied by atom probe tomography

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The distribution of B and other alloying elements (C, Cr, Mo) at prior austenite grain boundaries (PAGBs) and in the matrix was quantified by atom probe tomography in a quenched martensitic steel. B and Mo were observed to be segregated only at PAGBs and to be absent at martensite–martensite boundaries. C is segregated both at PAGBs and at martensite–martensite boundaries, whereas Cr is homogeneously distributed in the probed volume. Our results indicate that B undergoes a non-equilibrium segregation.

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Addition of a small amount of B (typically 3–30 wt ppm) to low-alloy steels can significantly enhance their hardenability [1]. The reason for such an improvement has been frequently attributed to segregation of B at (prior) austenite grain boundaries (PAGBs), which may reduce the grain boundary energy, thus preventing the austenite to ferrite transformation and rendering bainite or martensite formation more favorable [2,3]. Depending on heat treatment conditions, segregation of B at grain boundaries (GBs) can be of equilibrium and/or non-equilibrium type. Equilibrium segregation [4] is driven by the difference in free enthalpy of solute atoms in the matrix and at interfaces such as grain boundaries. Solute segregation at grain boundaries hence thermodynamically minimizes the overall free energy of the system. Its magnitude decreases with increasing temperature. Non-equilibrium segregation [5], on the other hand, mainly occurs upon cooling from high temperatures, during which supersaturated vacancies are induced in the matrix. These vacancies diffuse towards vacancy sinks such as grain boundaries. They may drag solute atoms with them, leading to the non-equilibrium segregation of solutes at GBs. Therefore, in such cases the magnitude of segregation depends on the austenitization temperature and the cooling rate [6–9]. It is widely accepted that B segregation at PAGBs during cooling is controlled by a non-equilibrium mechanism [2,6–18].

Segregation of B at PAGBs in martensitic steels has been studied by particle tracking autoradiography (PTA) [2,9,13,16,18], secondary ion mass spectrometry (SIMS) [9,18,19] and electron energy loss spectroscopy (EELS)

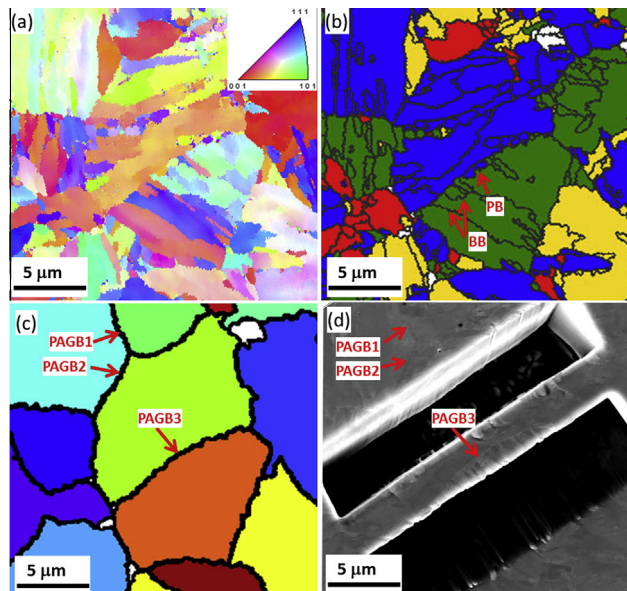
[17]. These characterization techniques, except EELS, are advantageous for large-scale (up to several hundred micrometers) investigations, providing overviews of distributions of B at GBs and B-containing precipitates. However, they are not the most appropriate approaches for quantitative analyses. As a complementary technique atom probe tomography (APT) traces individual atoms at the atomic scale, and thus delivers precise information on solute distribution at GBs, in precipitates and in the matrix. An APT study of B distribution in a tempered martensitic steel is reported in Ref. [19]. While segregation of B at PAGBs in tempered steels was observed [19], it is still not clear whether B segregation at PAGBs occurs during cooling (non-equilibrium segregation) or during subsequent tempering (equilibrium segregation). Also it is unclear whether B segregates to martensite–martensite (M–M) boundaries and how other alloying elements may influence B segregation. To answer these questions, one needs to investigate as-quenched steel. Since PAGBs are hard to reveal by chemical etching and hard to distinguish from other types of GBs such as block, packet boundaries or lath boundaries in martensite when preparing APT samples by focused-ion-beam (FIB) milling, investigations of B segregation at PAGBs become more difficult in a quenched steel.

In the present work we apply APT to study segregation of B, C, Mo and Cr at PAGBs and other types of boundary in an as-quenched martensitic steel. The quenched microstructure contains various boundaries, such as PAGBs and the newly formed packet and block boundaries as well as the martensite lath boundaries. For simplicity the newly formed boundaries during cooling are referred to as M–M boundaries. Prior austenite grains (PAGs) were reconstructed from electron backscatter diffraction (EBSD) orientation maps. Subsequently, FIB milling was applied

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for site-specific preparation of APT samples containing PAGBs. We found that B and Mo are segregated only at PAGBs and B is not detected in the matrix. Based on the APT results, the mechanism of B segregation is discussed.

The material studied in this work is a low-carbon steel (Fe–0.19C–0.35Si–1.20Mn–0.20Cr–0.50Mo–0.06Al–0.03Nb–0.03V–0.00124B in wt.% or Fe–0.88C–0.70Si–1.21Mn–0.21Cr–0.29Mo–0.12Al–0.02Nb–0.03V–0.012B in at.%). The steel was austenitized at 930 °C for 15 min followed by water quenching. The cooling rate at the APT sampling location was approximately 30 °C s<sup>-1</sup> within the first 20 s of quenching from 930 °C. A software, developed by CEA-Grenoble, Laboratory of Innovation for New Energy Technologies and Nanomaterials, France, was applied for reconstructing the PAGs from EBSD data according to the orientation relationship (Kurdjumov–Sachs) between the martensite variants and their parent austenite grain (see details in Ref. [20]). Figure 1a shows the EBSD orientation map of the as-quenched steel containing various unidentified boundaries of PAGs, blocks, packets and martensite laths. The reconstructed results are shown in Figure 1b and c. Three PAGBs marked by the red arrows in Figure 1c are correlated with those shown in the SEM image in Figure 1d. The PAGB to be investigated by APT is marked as PAGB3. These results were used as references to locate the corresponding PAGBs in both SEM (Fig. 1d) and FIB secondary electron image. Next, site-specific preparation [21] of APT samples containing PAGBs was performed using a dual-beam FIB (FEI Helios NanoLab 600TM). A local electrode atom probe (LEAP) (LEAP 3000X HR™,

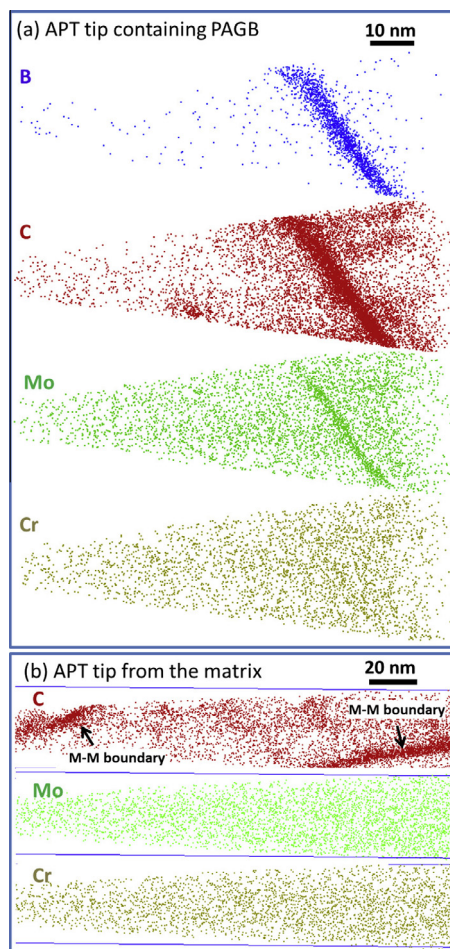


**Fig. 1.** Example of revealing PAGBs in an as-quenched martensitic steel. (a) Crystal orientation map measured using EBSD in SEM. (b) Reconstructed images displaying packet boundaries (PBs) and block boundaries (BBs). Colors represent packets with different orientations. The lines within a packet (a uniform color) are identified as block boundaries. Unindexed regions are marked by white color. (c) Reconstructed images showing only PAGs. The colors represent PAGs with different orientations. (d) SEM image marking three correlate PAGBs with those in (c) by red arrows. Two mill cuts were done to extract PAGB3. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

Cameca Instruments) was employed to analyze the element distributions. The measurements were performed in voltage mode at 70 K. B ions were detected at 3.6 Da (B<sup>3+</sup>), 5 Da and 5.5 Da (B<sup>2+</sup>), and 10 Da and 11 Da (B<sup>+</sup>) in the sample containing a PAGB. No molecular MoB ions were detected.

Figure 2 shows the atom maps of several selected solute species (B, Mo, C and Cr) in the as-quenched martensite. Results on other elements such as Nb and P (similar to Mo) and Si, Mn, Al and V (similar to Cr) will be presented elsewhere. Figure 2a reveals that B, C and Mo are strongly segregated to the PAGBs. The substitutional solute Cr is homogeneously distributed throughout the entire detected volume. Figure 2b shows the distribution of these atoms in the martensite matrix. The volume probed does not contain any PAGBs but two M–M boundaries. While no B is detected in the entire probed volume, C is observed to be segregated at the two M–M boundaries, which are almost parallel to each other and located at a distance of about 50 nm from each other. Interestingly, Mo and Cr are not observed to segregate at the M–M boundaries in this sample.

1-D concentration profiles of these elements within ROI1 (oval cross-sectional cylinder, Fig. 3a) along the arrow direction show a peak value of 2 at.% for B at the



**Fig. 2.** 3-D atom maps of the as-quenched martensitic steel. (a) Distributions of atoms in the probed volume containing a PAGB. Significant segregation of B, C and Mo and no segregation of Cr at the PAGB. (b) Distributions of atoms in the martensite matrix containing two M–M boundaries with C segregation. Mo and Cr are homogeneously distributed.

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