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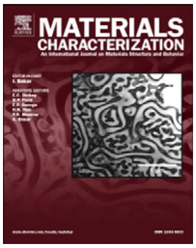

Microstructural analysis of martensite constituents in quenching and partitioning steels

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A methodology to distinguish martensite formed in the first quench (M1) from martensite formed in the second quench (M2) of the Quenching and Partitioning process is presented, enabling the study of the structural characteristics of both microstructural constituents. Investigations show that M1 displays larger block size and less lattice imperfections than M2, differences that can be related to their respective carbon contents.

1. Introduction

The Quenching and Partitioning (Q&P) process is a heat treatment for the development of Advanced High Strength Steels (AHSS) [1]. It starts with a full austenitization [2], followed by rapid cooling (quenching step) in order to transform part of the austenite into martensite. The heat treatment continues with an isothermal stage (partitioning step) aiming to partition carbon from the carbon-supersaturated martensite to neighboring austenite [3]. As a consequence, this martensite becomes carbon depleted (in the following, this martensite is called M1). The process ends with a second quench to room temperature in which austenite that is not sufficiently enriched in carbon transforms into fresh martensite (in the following, M2), whereas the rest of the austenite is retained at room temperature [4]. Assuming that the formation of ferrite, pearlite, bainite and carbides is inhibited during the entire Q&P-process, the resulting microstructure consists of M1, M2 and retained austenite (RA) [5]. Since volume fractions and morphologies of these constituents affect the mechanical properties, the knowledge of their microstructural characteristics is essential. The mechanical properties of the Q&P steels are directly controlled by the strength of the martensite phase and the transformation stability of the retained austenite. The martensite which originates from the first quench is tempered during partitioning and eventually has a yield stress of approximately 400 MPa. On the other hand, the yield stress of the "new" (i.e. untempered) martensite is reported to be more than 1200 MPa [6] but it is brittle due to its higher carbon content. Although there is no experimental evidence it is suggested that the fresh, high carbon martensite has a significant constraining effect on the surrounding austenite. By this it does not only influence the

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austenite transformation stability but could contribute negatively to the ductility of the steel.

The analysis of RA in Q&P steels is relatively simple combining electron backscatter diffraction (EBSD) for the analysis of morphology and dimensions, and X-ray diffraction for the quantification of volume fractions and carbon content [5]. However, the characterization and distinction of M1 and M2 are more complex. Techniques such as dilatometry and scanning electron microscopy provide quantitative measurements of volume fractions of M1 and M2, but do not provide information in terms of morphology, crystallography or internal structure of these constituents. The present study investigates these microstructural aspects of martensite in two different Q&P microstructures with EBSD.

2. Material and Methods

The chemical composition of the studied steel is 0.204C–2.5Mn–1.47Ni–1.01Cr–1.50Si (wt.%). Microstructural analyses of this steel after different Q&P heat treatments have been presented elsewhere [5]. They show variable fractions of retained austenite and 2 types of martensite, namely M1 and M2 in different proportions depending on the type of heat treatment. In the present work, two of these microstructures were selected for further investigations. They were obtained after austenitization at 1173 K (900 °C) for 600 s, quenching to 503 K (230 °C for material Q230C) and to 523 K (250 °C for material Q250C), partitioning at 673 K (400 °C) for 100 s in both cases and quenching to room temperature. Heat treatments were performed in a Bähr DIL 805 dilatometer.

The retained austenite volume fraction was measured by X-ray diffraction experiments using a Bruker type D8-Advance diffractometer equipped with a Bruker Vantec Position Sensitive Detector (PSD). Co-Ka radiation was used and a 2θ range from 30° to 135°, containing the (111), (200), (220) and (311) austenite reflections was scanned with a step size of 0.05°/step. Error bars in calculations of the volume fraction of retained austenite were estimated to account for possible deviations caused by crystallographic texture.

Specimens for EBSD analysis were metallographically prepared with a final polishing step of 1 μm diamond paste. The last preparation step was electrolytic polishing for 10 s at 40 V with an electrolyte consisting of 78 ml perchloric acid, 90 ml distilled water, 730 ml ethanol and 100 ml 2-butoxyethanol. EBSD patterns were acquired on a FEI Nova 600 Nanolab dual-beam (focused ion beam) scanning electron microscope equipped with a Field Emission Gun (FEG) column by means of the Flamenco Oxford-HKL system. The analysis was performed with an acceleration voltage of 20 kV, working distance of 7 mm, tilt angle of 70° and step size of 70 nm (material Q230C) and 40 nm (material Q250C) in a square scan grid. The orientation data were post-processed with TSL software.

3. Results and Discussion

Results of the retained austenite volume fraction, quantified through X-ray diffraction, and the M1 and M2 fractions, quantified through scanning electron micrographs, are presented in Table 1. Dilatometry data did not show evidence for the formation of ferrite, pearlite or bainite during the heat treatments [5].

Fig. 1a and b display combined image quality (IQ) and color-coded phase maps of materials Q230C and Q250C, respectively, in which green corresponds to bcc lattice (martensite) and red to fcc lattice (austenite). Darker zones represent areas of lower IQ, which correspond to grains with a higher density of lattice imperfections, such as dislocations and point defects [7]. It is important to note that M1 undergoes an isothermal heat treatment during the partitioning step, during which the martensitic structure decreases its carbon content and reduces its lattice distortion. In contrast, M2 martensite is formed in the final quench to room temperature from austenite with carbon content higher than the nominal content of the steel (and higher than M1). Consequently, M1 grains contain less lattice imperfections than M2 and, accordingly, display higher IQ than M2.

It is therefore interesting to explore if this correspondence between IQ and martensitic constituents can be used to quantify M1 and M2. Crosses in Fig. 1c and d show the distributions of the number of pixels versus normalized-IQ for the martensitic microstructures of Fig. 1a and b. Both distributions display an asymmetric shape. In principle, it is tempting to associate the peak corresponding to low IQ to pixels belonging to M2, and the peak corresponding to high IQ to pixels belonging to M1. However, as was indicated by Wu et al. [7], neighboring grains may produce the interference of diffraction patterns in regions close to grain boundaries, leading to low IQ at these points. As a result, grain boundaries are affecting the IQ within adjacent pixels. To exclude the effect of the grain-boundary regions (GBR), further data post-processing was performed to exclude pixels that were contiguous to interfaces with a misorientation larger than 15° [7], resulting in the values represented as open symbols in Fig. 1c and d.

The method of Wu et al. [7] for the quantification of bcc constituents in steel microstructures was applied. The open symbols in Fig. 1c and d represent the fraction of pixels belonging to martensite in Fig. 1a and b, respectively, excluding grain-boundary regions, versus normalized IQ. Both distributions were fitted with a Gaussian distribution and the volume fractions of M1 and M2 were derived from the area below the high-IQ and the low-IQ distributions, respectively. Results of these estimations are shown in Fig. 1e and f and compared with the values in Table 1. EBSD measurements of volume fractions of retained austenite in Fig. 1a and b are also included. Local measurements of M2 in Fig. 1a and b on the basis of IQ in EBSD lead to overestimations of the volume fraction of M2. The reason is that the applied

Table 1 – Volume fractions of retained austenite (V_{RA}), martensitic constituent M1 (V_{M1}) and M2 (V_{M2}) in materials Q230C and Q250C.
[5]

Materials	V_{RA}	V_{M1}	V_{M2}
Q230C	0.12 ± 0.01	0.88 ± 0.04	<0.01
Q250C	0.09 ± 0.01	0.81 ± 0.03	0.10 ± 0.05