Experimental quantification of carbon gradients in martensite and its multi-scale effects in a DP steel

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ABSTRACT

In multi-phase steel grades such as ferrite-martensite dual phase (DP) steels, it is of the utmost importance to understand the underlying effects of solid phase transformations with respect to their consequences on the final mechanical properties. In the present research, by employing dilatometry experiments two equivalent DP microstructures have been designed which are identical in most primary aspects such as constituents' volume fractions, the corresponding grain sizes and their morphologies. The difference, however, is the fashion in which carbon atoms in the martensite constituents are distributed. Electron probe microanalysis (EPMA) shows that excess carbon atoms which are rejected as a consequence of ferrite formation are accumulated in the adjacent austenite grains close to the boundaries yielding sharp carbon gradients. During final quenching, the characteristics of martensite transformation are therefore severely influenced. Through electron backscatter diffraction (EBSD) analysis many coarse substructures were detected in the interior regions of the inhomogeneous martensite associated with low dislocation densities. However, the areas close to the ferrite/martensite boundaries demonstrate finer substructures. Moreover, by means of nanoindentation tests it was found that the interior areas with lower carbon contents and coarser substructures show lower local strength as compared to the marginal regions. Macroscopic stress-strain behavior revealed that a higher ultimate tensile strength and enhanced post-uniform elongation are achieved in the specimen with sharp carbon gradients in its martensite constituents.

1. Introduction

Dual phase (DP) ferrite-martensite steels are one of the popular multi-phase grades being widely employed in industry and have been investigated by numerous researchers of the field for decades [1–5]. The many years of research focused on functionality of DP steels has led to gain valuable achievements in understanding, design, control and optimization of microstructural constituents in regard to the final macroscopic behavior [6–11]. It is well documented that the overall mechanical properties of low carbon low alloy DP steels are mainly governed by factors such as constituents' volume fractions, microstructural morphology, grain size and their corresponding average chemical compositions, especially carbon content [4,12–17]. Furthermore, each individual constituent's property itself can also alter the macroscopic behavior, even if the other above-mentioned parameters are kept unchanged.

During austenite decomposition into ferrite, due to the negligible carbon solubility of ferrite, austenite gets enriched in carbon. The average level of carbon enrichment in austenite, which during quenching to room temperature transforms to martensite, is calculated according to the well accepted lever rule (mass balance) [18]. Martensite as a stress-carrying phase [19,20] plays a significant role on the performance of DP steels and therefore its associated properties are at the immense importance. Martensite is usually assumed as a single homogeneous phase; hence, possible heterogeneity in its micromechanical strength and importantly the following consequences on final mechanical properties has not been considered. Whereas depending on applied heat treatment process parameters and chemical compositions, carbon atoms inside untransformed austenite, which is partly consumed by prior ferrite formation, can be distributed inhomogeneously [21,22]. Since martensite characteristics are primarily determined by its carbon content [23], thus, inhomogeneous carbon distribution inside prior austenite can influence martensite transformation kinetics, microstructural features of product phases, their associated local strength and eventually the global mechanical properties. In the work of

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Zhu et al. [24], the effect of carbon segregation near ferrite/austenite interfaces on the following bainite and martensite transformation kinetics have been researched. It was further discussed that the interfaces could act as a source of carbon heterogeneity and accelerate the following martensite transformation. Besides, the inhomogeneous carbon distribution in untransformed austenite, developed during austenite to ferrite phase transformation, has also been reported in the work published by Ghasemi Banadkouki and Fereiduni [25]. This observation was further confirmed by considerable hardness variations in martensite constituents of the samples intercritically annealed for longer soaking times.

Some researchers have investigated the correlation between individual constituents' properties and overall mechanical response in DP steels. Through a set of systematic nanoindentation tests on eight commercially produced DP980 steels, Taylor et al. [26] showed that for a similar martensite volume fraction and microstructural morphology, yielding is governed by the ferrite strength. Kadkhodapour et al. [27] have investigated the effect of localized ferrite plasticity generated by austenite to martensite transformation on the overall flow behavior of a commercial DP steel via an experimentally-calibrated finite element modeling. They have indicated that the geometrically necessary dislocations (GNDs) associated with the martensite expansion, pile-up at the ferrite/martensite interfaces and lead to heterogeneous hardening response in ferrite grains, thereby affecting the macroscopic tensile properties. In a research detailed in [28], it has however been stated that the heterogeneous ferrite hardening response cannot be entirely explained by GNDs and in addition to that, the carbon content gradient within ferrite grains can be another source of the observed behavior. Ghassemi-Armaki et al. [29] conducted a set of experiments by using uniaxial micro-pillar compression tests and nanoindentation to reveal the deformation response of ferrite and martensite micro constituents in a DP steel. Through their work, it was shown that due to the presence of unpinned GNDs in ferrite grains close to the ferrite/martensite interface, micro-pillars fabricated from different positions of a single ferrite grain respond differently under same compression test conditions. They concluded that the strain partitioning between ferrite and martensite as well as the yielding initiation in martensite are accordingly influenced and therefore, impacts the overall flow behavior of the steel.

In spite of all the dedicated efforts on the correlation between microstructural constituents' properties and overall mechanical response, the exclusive effect of inhomogeneous carbon distribution within martensite and its further consequences has not yet been investigated systematically. Therefore, the objective of this paper is to solely unravel the effect of carbon gradients in prior austenite on the subsequent microstructural development of martensite, local constituents' strength and eventually on macroscopic stress-strain behavior in a low carbon low alloy DP steel by means of a multiscale-interconnected experimental approach. This work contributes to understanding the role of constituents' characteristics, their origins and subsequent consequences towards a more accurate control of the overall performance of multi-phase steels.

2. Material and experimental procedures

2.1. Material

The chemical composition of the investigated steel is given in Table 1. The material has been melted in a vacuum inductive furnace and casted in an 80 kg ingot with a dimension of $140 \times 140 \times 500$ mm^3. The ingot was further homogenized at 1200 °C for 3 h and underwent multi-step hot forging process producing two smaller blocks each with a final dimension of $70 \times 70 \times 1000$ mm^3. The hot-forged blocks were thereafter exposed to

Table 1
Chemical composition of the investigated steel (wt%).

C	Si	Mn	P	S	Cr	Mo	Al	N	Fe
0.18	0.39	0.75	0.01	0.022	0.35	0.50	0.033	0.007	Bal.

normal atmosphere and air cooled to room temperature. It was then followed by normalizing process at 900 °C to obtain a homogeneous microstructure.

2.2. Designing equivalent microstructures using dilatometer

The objective here is to design two dual phase (DP) ferritic-martensitic microstructures with an identical fraction of constituents, microstructural morphology and average grain sizes while having two distinct carbon distribution fashions in their respective martensite (α'). This is in order to exclusively quantify the effect of carbon gradients in austenite on following microstructural development, local and global mechanical properties. The aimed microstructures are named "Dual-Phase microstructure with inhomogeneous and homogeneous carbon distribution in martensite", hereinafter abbreviated as: (i) DP-ic and (ii) DP-hc, respectively.

Cylindrical dilatometry samples of 10 mm length and 5 mm diameter were machined from the normalized blocks perpendicular to the hot-forged direction and have been employed for the following dilatometry tests. The fabricated dilatometry samples have been heat treated under vacuum in a dilatometer Bähr 805 A/D. During the applied heat treatments, Fig. 1, dimensional changes (dilatation) of the samples in both longitudinal and radial directions have been measured via contact and laser-measurement systems, respectively. According to cycle A, the samples are heated at the rate of 3.3 °C/s up to 950 °C and austenitized at this temperature for 10 min. It was then followed by step quenching to 675 °C where austenite (γ) starts to decompose into ferrite (α) and soaked at this temperature isothermally for 1 min (cycle A-stage I). The samples were subsequently quenched to room temperature. Based on the metallography investigations, 40% ± 2 ferrite transformed out of austenite during stage I of cycle A. Therefore, taking the fast kinetic of ferrite transformation at this stage into account, it is plausible to assume that the carbon atoms are inhomogeneously distributed in austenite. Hence, these samples are labeled as DP-ic. The second set of samples were annealed according to cycle B which is partly similar to cycle A; i.e., the samples were identically heat treated up to the endpoint of the isothermal section at 675 °C (cycle B-stage I). Following this, they were further heated at the rate of 50 °C/s up to different higher temperatures in the range of 685–800 °C and soaked isothermally for 5 min (cycle B-stage II) before quenching to room temperature. The objective of using different temperatures in stage II of cycle B was to detect the right temperature at which carbon atoms can homogeneously rearrange themselves without any further phase transformation. This sample will therefore be labeled as DP-hc. Due to the higher employed temperatures of stage II in cycle B and its corresponding longer soaking time (5 min), it is expected that the carbon atoms of austenite are dispersed in a homogenized manner.

2.3. Electron probe microanalysis (EPMA)

The partitioning of alloying elements between ferrite and austenite occurred during the applied heat treatments has been inspected by means of a Schottky field-emission gun electron microprobe, JEOL JXA-8530F (JEOL Ltd., Tokyo, Japan) with an accelerating voltage of 15 kV, a beam current of 100 nA and a step size of 100 nm and 300 nm for map and line scan measurements, respectively. In order to minimize the effect of contamination, produced by cracking of hydrocarbon molecules adsorbed on the specimen surface, on quantitative carbon analysis an acquisition procedure was employed which has been detailed in [30].

2.4. Electron backscatter diffraction (EBSD)

The microstructural-crystallographic characterizations of the selected samples were analyzed by means of Electron backscatter diffraction (EBSD). The samples were slightly mechanically polished to remove the contamination layer leftover after EPMA measurements and also to prepare a flat and damage-free area. The EBSD measurements were performed by employing a Schottky field-emission gun SEM, JEOL