Contents lists available at ScienceDirect

Materials & Design

The role of silicon in carbon partitioning processes in martensite/austenite microstructures

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A B S T R A C T

Understanding carbon redistribution in steels is crucial in developing advanced high strength steels. For instance, Quenching & Partitioning (Q & P) processes rely on the partitioning of carbon from martensite into austenite, where at the end of the heat treatment the carbon-enriched austenite shows higher stability at room temperature. Recent literature gives increasing evidence of carbide precipitation occurring during partitioning despite the addition of silicon, conventionally thought to suppress carbide precipitation. The aim of the present study is to gain insight into carbon-competing processes by applying a series of Q & P heat treatments, with particular focus on the partitioning stage, where the role of silicon in the stability of austenite is evaluated. Various characterisation techniques are combined in order to unveil the microstructural changes. While carbide precipitation does appear to occur in the presence of silicon, it is found that silicon plays an active role in the stabilisation of the austenite during the partitioning reaction.

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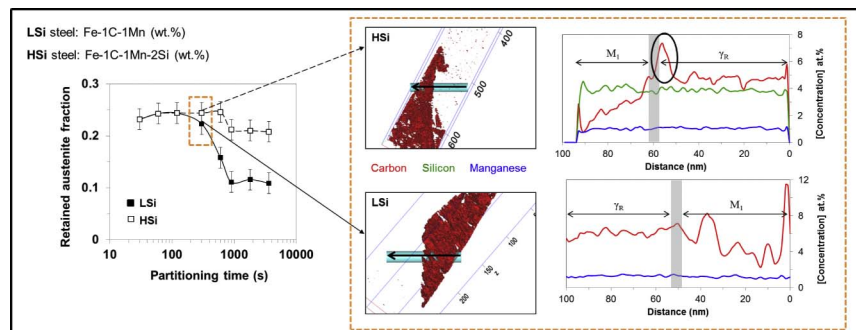
1. Introduction

Advanced high strength steels typically consist of complex microstructures that are optimised by both heat treatment and alloying composition. Understanding the redistribution of carbon within the microstructure during processing is fundamental in designing new steels, but still remains a challenge due to the various carbon-competing processes that occur throughout the heat treatment. In the case of Quenching & Partitioning (Q & P) steels, heat treatments are designed such that carbon partitions from martensite into austenite, where at the end of the heat treatment the carbon-enriched austenite shows higher stability at room temperature. The Q & P heat treatment typically consists of austenitising and quenching to a temperature between the martensitic start temperature and room temperature, in order to obtain a desired fraction of martensite in the microstructure. The steel is then reheated to a higher temperature, where carbon partitioning from martensite into austenite occurs. At the end of the partitioning stage, the sample is quenched to room temperature. During this second

quench, the austenite that is not sufficiently enriched in carbon transforms into martensite.

The typical microstructure developed during a Q & P heat treatment consists of

- M_1– this is the martensite that forms during the first quench, where the fraction of M_1 depends on the quenching temperature (QT) and becomes carbon-depleted and tempered during the partitioning stage;
- γ_R– this is the austenite that is retained at the end of the heat treatment after the sample is cooled to room temperature (RT); and
- M_2– this is the fresh martensite that forms during the second quench, having a higher carbon content than M_1 since it forms from carbon-enriched (although insufficiently) austenite.

These are the notations that will be used throughout the present study to denominate the different products forming throughout the heat treatment.

In the Q & P design, carbide precipitation must be suppressed in order to allow maximum carbon partitioning from martensite into austenite. In this aspect, Q & P steels are typically alloyed with silicon [1,2]. Nevertheless, recent literature gives evidence of more complex interactions involving carbon: (i) there is increasing evidence of carbide precipitation occurring during partitioning [3–5], and (ii) recent literature also indicates that carbon solubility in ferrite when in equilibrium in austenite is higher than predicted using conventional thermodynamic databases [6,7]. Both phenomena are relevant to the Q & P process as they reduce the amount of carbon available to partition into austenite, hence the stability of the austenite at the end of the thermal cycle becomes affected. The present study focuses on point (i) carbide precipitation, where the role of silicon will be investigated.

Carbide precipitation during martensite tempering is not surprising, especially if considering the paraequilibrium conditions in which carbides form by inheriting the matrix composition including the silicon content [8–10]. Hence the precise role of silicon in Q & P steels is questioned. Furthermore, during the partitioning stage there is also the possibility of austenite decomposing into bainite [5]. In a different context, in recent work on bearing steels technology by Sourmail and Millot-Méheux [11] it was shown that the addition of silicon enhances the thermal stability of γ_R against decomposition into ferritic products during tempering. Therefore, it is the aim of the current study to gain insight into the role of silicon in carbon-partitioning processes and its influence on the stability of austenite by applying a series of Q & P style heat treatments, with particular focus on the partitioning stage. Quantitative characterisation techniques such as dilatometry, X-ray diffraction (XRD) and atom probe tomography (APT) are combined in order to unveil the role of silicon in carbon partitioning processes.

From the studies it is observed that carbide precipitation occurs even in the presence of silicon. On the other hand, silicon is seen to enhance austenite stability and inhibit the decomposition of austenite during partitioning. Furthermore, it is also observed that martensite formation is accompanied by transformation strain in austenite, affecting austenite's lattice parameter, which may give an unreliable carbon content estimation in austenite.

Table 1
Measured chemical composition of steels in wt% (in at.%), where Fe is to balance, and the measured M_s determined by dilatometry.

	C	Mn	Si	Al	M_s (°C)
Alloy LSi	1.00 (4.49)	1.08 (1.06)	0.01 (0.01)	0.01 (0.02)	151 ± 1
Alloy HSi	0.98 (4.40)	1.04 (1.02)	1.98 (3.80)	0.01 (0.02)	140 ± 6

2. Experimental procedure

Two model alloys have been selected for the purpose of this study, where the chemical compositions are shown in Table 1. A relatively high carbon content (1 wt% C) was chosen for the following reasons: (i) owing to the high carbon content a relatively low M_s is expected, thus minimising the effect of autotempering during the quenching process which would affect the carbon content in M_1 before the partitioning stage is reached, and (ii) the supersaturation of the carbon in ferrite solid solution is likely to give rise to tetragonality of the martensite forming upon the final quench, hence M_1 and M_2 can be distinguished by X-ray diffraction (XRD). The as-received material was in the form of hot-rolled plate of 6 mm thickness.

The heat treatments applied were Q & P style heat treatments, shown in Fig. 1. In the first set of heat treatments, Fig. 1 (a), a series of different quenching temperatures were applied for fixed austenitisation and partitioning conditions. In the second set, Fig. 1 (b), the effect of partitioning time was studied for two quenching temperatures. The heat treatments were carried out using a Bähr DIL 805 A/D dilatometer, where cylindrical samples of 3.5 mm diameter and 10 mm length were obtained from the middle of the rolled plate, parallel to the rolling direction. The heating rate used was 5 °C/s and all quenching processes were done using helium gas. The measured cooling rate from the thermocouples was ~ 150 °C/s between 1000 °C and M_s. First, experiments were carried out in order to determine the martensitic start temperature, M_s. Using the offset method [12], M_s of alloys LSi and HSi were determined to be 151 ± 1 °C and 140 ± 6 °C, respectively.

In order to determine the volume fraction of γ_R, M_1 and M_2, X-ray diffraction experiments were performed using a Bruker type D8-Advance diffractometer equipped with a Bruker Vantec Position Sensitive Detector (PSD). Co Kα radiation was used, for scans in the 35–140° 2θ range. The obtained diffractograms were analysed by Rietveld refinement using the MAUD software [13].

In order to study local variations in carbon concentration and other alloying elements, one particular condition from alloys LSi and HSi was examined by atom probe tomography (APT) using a local electrode atom probe (LEAP 4000X HR) at Eindhoven University of Technology. The microtips were prepared by FIB (dual beam FEI Nova600i NanoLab), where prior EBSD measurements were made in order to specify the regions of interest (ZEISS Ultra 55 equipped with HIKARI EBSD camera with TSL software). APT analyses were performed using laser pulse mode, where measurements were taken at 20 K, 30 pJ laser energy and 200 kHz pulse rate. The reconstructions were performed using the IVAS 3.6.8 software, with settings of delocalisation 2.5 nm and voxel size 0.7 nm. Peak decomposition was carried out using the algorithm provided in the IVAS software.

3. Results and discussion

3.1. Dilatometry

The dilatometry results obtained during the final quench from the set of heat treatments listed in Fig. 1 (a) are summarised in Fig. 2. As seen in Fig. 2 (a), a full quench in the dilatometer from 1000 °C to room temperature leads to an incomplete martensitic transformation in both alloys. In Fig. 2 (b) it is observed that the formation of M_2 in the final cooling is dependent on QT. No M_2 formation is detected in dilatometry for the QT50. The trend is clearly seen in Fig. 2 (c), where M_s of M_2 is seen to decrease linearly with a decrease in QT. Following the Q & P line of thought, a lower QT produces higher fractions of M_1, increasing the amount of carbon available to partition from the super-saturated ferrite into austenite. Hence, under the same partitioning conditions, a higher M_1 fraction will likely result in higher carbon-enrichment in austenite at the end of the partitioning stage. Since the M_s decreases with increasing carbon contents [14], a decrease in M_s upon final cooling is an indication of higher carbon content in austenite prior quenching.