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# Carbon partitioning during quenching and partitioning heat treatment accompanied by carbide precipitation

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Abstract—Carbon partitioning from martensite into austenite in the quenching and partitioning (Q&P) process has been suggested to be controlled by the constrained carbon equilibrium (CCE) criterion. It defines an approach for predicting the carbon concentration in austenite under the condition that competing reactions such as carbide formation and bainite transformation are suppressed. Carbide precipitation in martensite is, however, often observed during the partitioning step, even in low-carbon steels as well as in high-carbon steels, even when containing a high amount of Si. Therefore, carbon partitioning from martensite into austenite is studied here, considering carbide precipitation in martensite. Carbon partitioning was investigated by means of a field-emission electron probe micro analysis (FE-EPMA) and atom probe tomography (APT), using 1.07 wt.% and 0.59 wt.% carbon steels with various martensite volume fractions. Carbon partitioning from martensite to austenite was clearly observed all specimens, even though a considerable amount of carbide precipitated inside the martensite. The austenite carbon concentration after the partitioning step was not influenced by either the martensite volume fraction or the bulk carbon content. A modified model for predicting the austenite carbon concentration after the partitioning step was proposed to explain the experimental results by assuming carbon equilibria between austenite, ferrite and cementite under a constrained condition.

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### 1. Introduction

Quenching and partitioning (Q&P) steels yield an excellent balance of high tensile strength and good elongation, with chemical compositions similar to conventional TRIP steels [1–4]. They are produced via the Q&P process, which consists of a quenching and a following partitioning step. During the quenching step, fully austenitized or intercritically annealed steels are quenched to temperatures (hereafter referred to as "quench temperature") below the martensite start  $(M_s)$  temperature, but above the martensite finish  $(M_{\rm f})$  temperature in order to form a controlled volume fraction of martensite. The quenched steels are then held at temperatures the same as or higher than the quench temperature during the subsequent partitioning step. Austenite that prevails after quenching is considered to be stabilized through carbon partitioning from martensite into austenite during the partitioning treatment.

It has been suggested that the carbon partitioning from martensite into austenite is controlled by the constrained carbon equilibrium (CCE) criterion [5]. This criterion aims to predict the carbon concentration in austenite under the condition where: (i) competing reactions, such as cementite or transition carbide formation or bainite transformation, are suppressed; (ii) an identical carbon chemical potential exists in both ferrite (or martensite) and austenite; and (iii) the carbon partitioning proceeds under the assumption that the interface between ferrite and austenite does not migrate. However, carbide precipitation in martensite is often observed during the partitioning step, even in lowcarbon steels [6] as well as in high-carbon steels [7,8], even if they contain a high amount of Si [9]. If carbide precipitates, some of the carbon is consumed to form the carbide, reducing the remaining amount of carbon in martensite that can be enriched in austenite during partitioning. Hence, the austenite carbon concentration after the partitioning step in this case is presumed to be lower than that predicted under the CCE conditions excluding carbide precipitation. As Speer et al. [1] pointed out, it is important to choose appropriate chemical compositions in order to avoid carbide precipitation in realizing an ideal Q&P condition. However, as some carbide formation may always occur, adequate models are required that describe such a case, thus providing a more precise estimate of the carbon concentration in austenite after the O&P heat treatment. There is, however, currently no model dealing with the

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carbon partitioning behavior from martensite into austenite under conditions in which carbide precipitation occurs in martensite during the partitioning step.

Therefore, this study conducts an experimental analysis of the carbon partitioning behavior from martensite into austenite accompanied by carbide precipitation inside the martensite during a partitioning step. A modified CCE model is introduced to explain the experimental results.

#### 2. Experimental procedure

The chemical compositions of the steels used in this study are listed in Table 1. The carbon contents in these model alloys were higher than that in typical alloys used for Q&P processing [1,10] in order to lower the  $M_{\rm f}$  temperature below room temperature. For these alloys, room temperature or even lower temperatures can be used as the quench temperature, which enables direct observation of the initial microstructure and atomic distribution before the partitioning step. The high amount of Si ( $\sim 2 \text{ wt.}\%$ ) was added to suppress carbide formation [9]. The steels were prepared by vacuum induction melting. The ingots were homogenized at 1240 °C for 48 h and then air cooled to room temperature. The homogenized ingots were reheated and held at 1200 °C for 30 min, followed by hot rolling to sheets with thickness 3.6 mm, and finally air cooled to room temperature. Specimens with dimensions  $15 \times 50$  mm, cut from the hot-rolled sheets, were then heat-treated according to Fig. 1. They were austenitized at 900 °C for 3 min, then quenched in water at a temperature of 30-17 °C, followed by a partitioning step at 400 °C for 300 s in a salt bath furnace. Some specimens cut from steel A (see Table 1) quenched in 17 °C water were further quenched to -20 °C or -63 °C prior to the partitioning step to vary the martensite volume fraction. The specimens before and after the partitioning step are hereafter referred to as "as-quenched specimen" and "partitioned specimen", respectively. Although slight decarburization

Table 1. Chemical compositions of steels used (wt.%).

Steel	С	Si	Mn	Al	Fe
А	1.07	2.2	2.9	0.048	Bal.
В	0.59	2.0	2.9	0.038	Bal.



Fig. 1. Schematic diagram of heat treatment:  $M_s$ , martensite start temperature;  $M_f$ , martensite finish temperature; WQ, water-quenching.

occurred near the surface during homogenization and reheating, it was confirmed that the carbon concentration at least at one-quarter of the thickness of the heat-treated sheets was identical to the bulk carbon concentration. Therefore, all the following characterization steps were performed at one-quarter of the thickness of the heat-treated sheets to avoid the decarburized layer.

Microstructures in the cross section perpendicular to the transverse direction (TD cross section) etched with 0.1–0.3% Nital were observed by optical microscopy and scanning electron microscopy (SEM). The austenite volume fraction change during the partitioning step was investigated by X-ray diffraction (XRD) with Co  $K_{\alpha}$  radiation, using the intensities of the  $(200)\alpha$ ,  $(211)\alpha$ ,  $(200)\gamma$ ,  $(220)\gamma$  and  $(311)\gamma$  reflections. The samples for XRD analysis were ground from the surface to one-quarter of the thickness of the heat-treated steels and, subsequently, another 100 µm was removed from the ground surface by means of electrolytic polishing to exclude any influence of strains that might have been introduced by the grinding step.

Carbon partitioning between martensite and austenite was investigated mainly using a field-emission electron probe micro analysis (FE-EPMA) [11]. The use of a FEtype electron emitter can achieve a narrower emission area compared with the conventional W or LaB<sub>6</sub>-type electron emitter. Also, a relatively low voltage of 6 kV was used to minimize the excitation volume. A probe current of 70 nA was used. The carbon concentration was determined using a standard calibration curve, which was obtained using seven standard specimens in the range 0.0083–1.07 wt.% C (Fig. 2). Standard deviations for each plot in Fig. 2 are expressed as error bars, which are <0.021 wt.%. In the FE-EPMA measurements, the line-analysis mode is used to obtain carbon profiles across regions of interest. The detection time for each point was 2 s in single-scan mode.

Atom probe tomography (APT) [12–21] was used for the atomic scale quantitative investigation of elemental partitioning during the partitioning step. Samples for APT measurements were prepared using focused ion beam milling and the lift-out procedure described in Ref. [22]. APT analyses were performed using a local electrode atom probe (LEAP 3000X HR, Cameca Instruments) in voltage mode at a specimen temperature of ~65 K. The pulse fraction and the pulse rate were 15% and 200 kHz, respectively.



Fig. 2. Standard calibration line for FE-EPMA measurements. The error bars represent standard deviation.

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