



In situ austenite–martensite interface mobility study during annealing

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Abstract—A 1C3Mn1.5Si steel was partially transformed into martensite by quenching to room temperature after full austenitisation. A partitioning treatment was applied in situ in a high resolution transmission electron microscope. The width of an austenite grain in between martensite constituents was followed as a function of the annealing time. Migration of the martensite–austenite interfaces was quantitatively measured and compared with results from a model for carbon partitioning from martensite to austenite involving interface motion. The kinetics of the observed movement suggests that the interface has a semi-coherent nature. This work shows that grain boundary mobility of the martensite–austenite interfaces during annealing may play an important role in the microstructure development during the process of Quenching and Partitioning in steels. © 2015 Acta Materialia Inc. Published by Elsevier Ltd. All rights reserved.

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1. Introduction

A recent concept for processing advanced high strength steel was proposed by Speer et al. [1] in 2003 as a method to produce low carbon high strength steels with a mixed microstructure of martensite and retained austenite: “Quenching and Partitioning” (Q&P). Additions of silicon and aluminium in the steel compositions were used to inhibit cementite precipitation, opening the possibility to obtain carbon-enriched austenite at room temperature (i.e. with improved stability) by carbon partitioning from supersaturated martensite [1–3]. The process involves an initial partial or full austenitisation, followed by quenching to a temperature between the martensite start (M_s) and finish (M_f) temperature to create a controlled fraction of martensite. Then, the steel is held isothermally or reheated to a higher temperature to allow carbon diffusion from martensite into the remaining austenite. During this partitioning step, it is intended to enrich the austenite with sufficient carbon to aim for its stabilization at room temperature after final quenching. The resulting microstructure consists of retained austenite, ferrite (for intercritical annealing) and possibly two types of martensite: the one formed during the first quench, “tempered” or “primary” martensite, and possibly the one formed during the final quench, “fresh” or “secondary” martensite [4].

A thermodynamic model to predict the endpoint of complete or *ideal* partitioning at a given temperature was described by the “Constrained Carbon Equilibrium” (CCE) condition [1–3,5,6]. This is defined by two conditions: an equal chemical potential of carbon in each phase, and fixed austenite–martensite interfaces. Hence, the difference with paraequilibrium [7] is that grain boundary movement is precluded by short-range iron or substitutional diffusion.

There are however experimental observations which question the immobility of the austenite–martensite grain boundary. The occurrence of interface migration was proposed by Zhong et al. [8] to explain changes in the curvature of austenite grains before and after partitioning, without nucleation of bainite at the interfaces. They stated that “the endpoint of the CCE model is the startpoint of Fe-diffusion or interface migration”. Santofimia et al. [9] and Toji et al. [10] conducted atom probe tomography experiments on Q&P steels and observed that a small amount of manganese partitioned from martensite into austenite, most probably during the partitioning step. This could be explained by two mechanisms: partitioning of Mn from martensite to austenite, which would be a process with a much slower kinetics compared to the process of carbon partitioning or solute drag due to the movement of the interface during the partitioning step. Santofimia et al. [9] observed that such Mn enrichment was not a general result observed in all martensite–austenite interfaces in the same material, which shows that the process of Mn partitioning may be related with the particular character

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of every single interface. If Mn partitioning is a process linked with migration of martensite–austenite interfaces, this means that possibly not all interfaces “migrate” during the partitioning step which is most likely related with the characteristics of each interface.

Expected motion of the interface during partitioning, due to a chemical potential difference of iron, was qualitatively examined by Speer et al. [11] and quantitatively by Santofimia et al. [12] illustrating a bidirectional movement of the interface. The latter calculations were made under the assumption of an incoherent interface irrespective of their semi-coherent nature [13]. Later on [14], different grain boundary mobilities related to the activation energy for iron migration to simulate the microstructural evolution during partitioning were assumed: (1) completely immobile with an infinite activation energy, (2) limited mobile representing a semi-coherent interface with an activation energy of 180 kJ/mol, and (3) highly mobile demonstrating an incoherent interface with an activation energy of 140 kJ/mol. The studied composition was a binary Fe–0.25C (wt.%) alloy with an alternating morphology of martensite and austenite. The input microstructure was formed by a single lath of martensite sharing the interface with a film of austenite. Mirror conditions were assumed in the calculations. The martensite lath was considered to have a fixed width of 0.2 μm [15] whereas the austenite film width changed according to varying quenching temperatures.

In general, the calculations [12,14] resulted in a sharp increase of the carbon content in the austenite near the austenite–martensite interface for short partitioning times, stabilising a small austenite fraction with a high C-content. If the partitioning time increased, this peak was progressively reduced while the overall carbon content of the austenite increased.

An immobile interface resulted in an equivalent behaviour as reported for CCE, which means that partitioning of carbon from martensite to austenite continues until the chemical potential of carbon is the same through the whole system under the restriction of a fixed martensite–austenite interface.

For an activation energy of 180 kJ/mol, which was an arbitrary chosen value for a semi-coherent interface, the carbon concentration profiles were similar to those for an immobile interface for short partitioning times. However, longer partitioning times lead to the initiation of interface migration and enrichment of carbon at the interface until full equilibrium was reached. The way in which the system reached equilibrium involved martensite–austenite interface migration at longer partitioning times, and the direction of this migration depended on the initial relative sizes of the austenite and martensite grains. For example, for a very low quenching temperature, which corresponds with a small austenite grain size at the beginning of the partitioning process, the equilibrium involved the growth of austenite, whereas the opposite result was obtained in the case of high quenching temperatures.

For an incoherent interface which was represented by an activation energy typical for reconstructive austenite to ferrite transformations, the carbon profiles and evolution of the interface were coupled during the partitioning process leading to a bidirectional movement of the interface prior to achieving equilibrium. Carbon partitioning started with an increase of the carbon content in the austenite which was compensated by an interface movement from the austenite to the martensite. Once the carbon content was

lower than the equilibrium value, the interface reversed its direction if that was required in order to reach full equilibrium in the system.

Phase field simulation was addressed by Takahama et al. [16] to show details of the carbon redistribution as well as possible interface migration. When the calculated local carbon peak in the austenite in the vicinity of a martensite grain was higher than the paraequilibrium value, a driving force for interface movement was created, causing growth of austenite.

In summary, the revised literature shows experimental evidences suggesting the migration of martensite–austenite interfaces during annealing at relatively low temperatures such as 400 °C. These migrations may not occur in all interfaces, but on specific ones having particular characteristics. These experimental observations are well supported by thermodynamical and kinetic models, showing that the coherency of the martensite–austenite interfaces may play a role in the mobility during annealing. This work reports for the first time the in situ migration of austenite–martensite grain boundaries during annealing. Experimental values were used as input for the model proposed by Santofimia et al. [12,14] to fit the activation energy characterising the mobility of the studied interfaces.

2. Experimental procedure

To conduct the annealing heat treatment in situ in TEM we have to start with an adequate microstructure consisting of austenite and martensite at room temperature. Therefore, a chemical composition to retain sufficient austenite at room temperature after full austenitisation was selected: 1C3Mn1.5Si (wt.%).

According to the literature [8,17–19], 1.5 wt.% of silicon is enough to suppress cementite formation during the partitioning step by formation of a kinetic barrier to the growth of cementite [20,21]. Manganese is an austenite stabilising element and increases the hardenability during cooling [22]. Though, high concentrations result in segregation banding [23] and thus the Mn-content was limited to 3 wt.%. Hence, the carbon content was adapted to retain sufficient austenite at room temperature. According to the Koistinen–Marburger equation, 50% retained austenite would be retained at room temperature for steel with a M_s temperature of approximately 80 °C. By employing its relationship with the chemical composition as proposed by van Bohemen et al. [24], 1 wt.% of C would result in a calculated M_s temperature of 82 °C retaining approximately 50% austenite at room temperature after cooling.

The first part of the heat treatment was carried out in a Bähr DIL 805 A/D dilatometer on hot-rolled rectangular samples with dimensions of 2.5 × 3.5 × 10 mm. The sample was fully austenitised at 950 °C for 60 s and cooled to room temperature at 20 °C/s with N₂-gas.

EBSD analysis was conducted in a FEI Quanta™ 450-FEG-SEM equipped with a Hikari detector controlled by the EDAX-TSL OIM-Data Collection software. The EBSD data were acquired on a hexagonal scan grid using an accelerating voltage of 20 kV, a working distance of 16 mm, a tilt angle of 70° and a step size of 60 nm. The orientation data were post-processed with TSL-OIM Analysis 6.2® software.

Transmission Kikuchi diffraction data were acquired in SEM (TKD-SEM) with a spot size of 5, an accelerating

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