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A direct evidence of solute interactions with a moving ferrite/austenite interface in a model Fe-C-Mn alloy



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ABSTRACT

The coupled-solute drag during ferrite growth in steels is widely discussed in the literature and remains still controversial since little direct evidences were adduced to support this effect. In this paper, from a correlative microscopy approach, a selected migrating ferrite/austenite interface in a model FeMnC alloy is quantitatively analysed by atom probe tomography and Energy Dispersive X-Ray Spectroscopy at the nanoscale. They show unambiguously a strong co-segregation of both carbon and manganese at the interface during ferrite growth at 680 °C. The obtained results support the coupled-solute drag effect as an operating mechanism during ferrite growth.

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The development of modern steels such as Dual-Phase, TRIP and Medium Mn steels and the control of their properties require a better understanding of the role of alloying elements during the transformation of austenite. In this context, the interactions of alloying elements with migrating α -ferrite/ γ -austenite interface during ferrite growth remains a topic of interest in physical metallurgy. The theory and the models for ferrite growth in low alloyed steels have been extensively investigated and documented over the last decades [1,2,3]. From a conceptual point of view, following the seminal work of Gibbs [4], the simplest model reduces the α/γ interface into a mathematical surface, and a local equilibrium is applied at the interface. In this case, this interface has no width (sharp interface), no specific properties, the energy dissipated by the interface is considered as negligible and the transformation rate is mainly controlled by atom diffusion. In the particular case of the Fe-C binary system, this type of approach has proved its worth [5]. However, most steels of practical importance contain substitutional elements, in particular Mn. The situation then becomes more complicated, because both interstitial (C) and substitutional (Mn) can diffuse, and their diffusivities differ substantially. The ParaEquilibrium (PE) and Local Equilibrium with Non-Partitioning (LENP) conditions represent the thermodynamic limits of non-partitioned growth. PE was introduced by Hultgren and is based on a constrained equilibrium for C, Mn is supposed to be unaffected by interface motion [6]. LENP assumes that a full local equilibrium is achieved, and a very thin Mn spike is supposed to build at the α/γ . However, this raises legitimate questions. First, substitutional atoms (Mn) must undergo a reconstructive rearrangement form fcc to bcc at the α/γ migrating interface and it is then difficult to imagine they are immobile. Second, the physical meaning of Mn spike is questioned since its width was calculated to be of the order, or even smaller, than the interatomic distance [3,7,8]. Lastly, it was recognized that a deviation from local equilibrium may occur and PE and LENP would only represent limits that may, or may not be reached. To deal with this issue, many physical based models were developed, which are explicitly or implicitly based on the fact that the total chemical driving force can be dissipated by diffusion within the interface and/or by interface friction [3,9,10,11,12,13]. Some of them are directly based on the solute drag-like effect (or the coupled-solute drag effect) where segregation of solutes can take place at the moving interface. This approach was initially developed by Cahn, Lucke and Stuwe for the migration of grain boundaries [14, 15] and was extended to phase transformations in steels [13,16,17]. The segregated atoms consume part of the driving force for ferrite growth during migration, affecting the α/γ interface motion. This coupled-solute drag effect would result from local interactions at the nano-scale in a few atomic plane thick layer covering the interface. It is certainly one of the main reasons that makes it difficult to experimentally prove such an effect during the α/γ interface motion. Some attempts to analyse Mn, and to a lower extent C, concentration profiles through the α/γ interface during ferrite growth were conducted [17,



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Fig. 1. a) Isothermal section at 680 °C in the Fe-C-Mn phase diagram. The nominal composition, plotted as a red point, is located in a region where a competition between the LENP and the PE modes is expected. b) Comparison between the measured kinetics and the calculated ones under PE and LENP conditions at 680 °C.

18,19,20,21], but none of these succeeded in showing any convincing evidence for Mn and C segregation over a few nanometers within the transformation interface.

In this paper, we propose to investigate such transformation interfaces down to the nanometer scale with state of the art analytical microscopes, in the aim of quantifying their substitutional and interstitial element local enrichments. Both scanning transmission electron microscope (STEM-EDX) and atom probe tomography (APT) were used, and unambiguously evidence Mn and C co-segregation at the migrating interface. This result is discussed, and is shown to support the couplesolute drag effect as the operating mechanism during ferrite growth.

A ternary Fe-0.12 wt%C-2 wt%Mn was prepared by vacuum induction melting. The ingot was hot- and then cold-rolled. The samples were then reaustenitized at 1250 °C for 48 h under Ar atmosphere in order to remove any Mn microsegregation and prevent any decarburization, and then finally cold-rolled to 1 mm. The samples were heated in a dilatometer at 10 °C/s to 1100 °C for 1 min, cooled down rapidly to 680 °C, and held at this temperature for various durations. The kinetics for ferrite growth was measured by both image analysis and dilatometry. For the latter, hollow silica rods were used, and nickel was deposited on samples in order to limit thermal heterogeneities and decarburization. For image analysis, the samples were polished and etched using 3% Nital. The microstructure was observed by optical microscopy and the volume fraction estimated from the area fractions.

The isothermal section of the Fe-C-Mn phase diagram at 680 $^{\circ}$ C is shown Fig. 1a). The alloy considered in this study (indicated by a



Fig. 2. Description of the original combined selective approach used: a) Optical microscopy view of the selected region – b) identification of 680 °C ferrite (grey) – austenite (color) microstructure of the selected region, each color represents one specific prior austenite orientation – c) initial ferrite – austenite interfaces orientation at 680 °C. Colors code the orientation difference with respect to K-S orientation from 0 (red) to 40° (blue) – d) final TEM and APT specimens with their initial location along the selected ferrite – austenite interface.

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