



Full length article

Investigation of solute/interphase interaction during ferrite growth

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ABSTRACT

Knowledge of solute interaction with the interface during the transformation of austenite into ferrite is fundamental in predicting its kinetics in multicomponent steel. This interaction notably translates in segregation, or depletion, of the solutes at the transformation interface. Here, this segregation was successfully quantified by atom probe tomography (APT) in four ternary Fe-X-C systems involving substitutional solutes commonly found in modern steel grades (X = Cr, Mn, Ni, Mo). Controlled decarburization was used to grow a uniform, planar and incoherent ferrite layer at the surface of fully austenitic samples. In the case of Fe-Cr-C and Fe-Mo-C, the interfacial concentrations permitted the evaluation of the binding energy of each substitutional solute to the interface, which was found to be comparable to its respective grain boundary binding energy. In the case of Fe-Mn-C and Fe-Ni-C, undesirable motion of the interface during the quench of the samples could not be avoided, preventing a reliable estimation of their binding energy since temperature and interface velocity were unknown.

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1. Introduction

The decomposition of austenite is a key reaction in steel processing. Predicting the decomposition kinetics is particularly important in modern steel grades such as dual phase (DP) and transformation-induced plasticity (TRIP) steels; the properties of these advanced grades can vary dramatically as a function of the amount of ferrite formed [1,2]. Reliable and accurate predictions of ferrite volume fraction as a function of time and temperature are highly desirable for efficient alloy design. The steel grades in question typically contain many substitutional solutes (e.g. Mn, Cr, Mo etc.). The presence of these solutes alters the kinetics of the decomposition, which renders it difficult to predict accurately. Major aspects contributing to this complexity include the interaction of solutes with the moving interface and solute-solute interactions within the interface. A number of ferrite growth models

have been devised in order to account for this interaction in ternary Fe-X-C systems [3]. In these models, the interaction is solely attributed to the substitutional solute X through a parameter referred to as the binding energy, which captures the affinity of the element for the interface. In practical situations where a value is required for this binding energy, such as transformation kinetics modeling, it is usually kept as a fitting parameter [4] or estimated using ab-initio calculations [5]. However, the interaction of the solutes with the interface also leads to segregation at the transformation interface, with the amount of segregation being a function of the binding energy. Thus, if this segregation can be accurately quantified, then an estimate for the binding energy can also be determined. This approach has been extensively utilized to quantify solute interaction with grain boundaries [6].

Applying the same technique to solute segregation at ferrite/austenite interface boundaries has proven much more challenging. Earlier attempts [7,8] showed great variations in the segregation from one interface boundary to another, which were attributed to varying crystallography between interfaces, as well as differences in the velocity of the interfaces prior to quenching. It can therefore be expected that variations in segregation at the interface would be minimized if the velocity of the interface boundary and its crystallography were well defined.

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In traditional ferrite precipitation experiments, significant uncertainty typically exists concerning the velocity of the interface. Much effort is also necessary to determine the crystallography of the interface and to obtain good statistics on a sufficient number of interfaces of a given crystallography. As a starting point for the present study, interfaces that form under decarburization conditions were chosen for examination. Under controlled decarburization conditions [9–12], a continuous ferrite layer grows, isothermally, into the austenite. The velocity of the ferrite/austenite interface, prior to quenching, can be determined accurately from the thickness of the ferrite layer. In addition, the ferrite/austenite interface formed under these conditions quickly becomes incoherent because a single ferrite grain would consume multiple austenite grains as the ferrite layer advances into the bulk. In this way, studies can be performed on interfaces with well-characterized velocities that are almost free from crystallographic effects. Determining the solute interaction with these incoherent interfaces is an important first step towards understanding the more important case of ferrite/austenite interface with strong orientation relationships typically observed in industrial materials.

In order to assess accurately the segregation to the interface, small variations of concentration (i.e. a few atomic percent) must be measured over a distance as small as several nanometers. Analytical spectroscopy techniques based on electron microscopy, such as energy dispersive x-ray spectroscopy (EDS) or electron energy loss spectroscopy (EELS) may be suitable. However, since the collection of data is a two-dimensional projection of the volume, the thickness and orientation of the sample become important factors in establishing composition profiles that can be difficult to measure or control [13]. Alternatively, atom probe tomography (APT) may provide reliable quantification of phase boundary segregation and thus has been applied many times to the related problem of grain boundary segregation [14–19].

The aim of this work is to assess the segregation of solutes at the transformation interface between austenite and a ferrite layer growing under well-defined decarburization conditions. It should be noted that in the systems investigated here most of the austenite transforms into martensite upon quenching down to room temperature (RT) at the end of the decarburization treatment. Thus, most of the interfaces examined were interfaces between martensite and ferrite.

In the following, the relevance of APT measurement of solute segregation at ferrite/austenite interfaces obtained by controlled decarburization is first evaluated, considering possible artefacts related to either preparation or analysis, by examining the Fe–Cr–C system. The APT analysis method is then applied to three additional ternary systems involving common substitutional alloying elements in steel: manganese, molybdenum and nickel. The measured segregation values are discussed against the generally accepted behavior of these elements.

2. Experimental method

The results presented in this paper were obtained from four Fe–X–C ternary alloys ($X = \text{Cr, Mn, Ni, Mo}$). The compositions of the alloys were chosen to maximize the amount of X at the interface while avoiding the formation of secondary phases, such as carbides. These alloys were all synthesized by arc-melting high-purity stocks of the different elements. The achieved compositions were measured by inductively coupled plasma optical emission spectroscopy (ICP OES). They are reported in Table 1. The ingots were cold-rolled prior to being homogenized at 1100 °C for at least 3 days in order to break the as-cast structure and ensure a uniform distribution of the alloying elements. Thin slices, typically $3 \times 3 \times 10 \text{ mm}^3$, were cut for the decarburization experiments. The

Table 1

Composition of the four initial Fe–X–C ternary alloys used in this study as measured by ICP OES (at. %, $\pm 0.05\%$).

	Fe–Cr–C	Fe–Mn–C	Fe–Ni–C	Fe–Mo–C
X	2.10	0.94	0.91	0.29
C	2.64	2.61	2.29	2.48
Fe	Balance			

decarburization treatment involved spot welding the samples to a holder, which was then inserted through an O-ring seal into the tube-furnace, under a flow of a non-decarburizing gas (argon or dry-hydrogen). A heating time of 5 min allowed the samples to austenitize and to reach the desired decarburization temperature. Water-saturated hydrogen (at room temperature) was then used to decarburize the samples for 7680 s. At the end of the treatment time, the furnace was purged with Ar for 150 s, gradually stopping the decarburization and the motion of the interface, before the sample was quenched. Blank samples, with no flow of wet hydrogen, were used to verify that the austenitizing and purging steps resulted in no ferrite formation. Alternatively and where specified, the sample was directly quenched from the decarburizing medium. Overall, the temperature was controlled to within ± 2 °C and quenching times were generally < 5 s. Upon quenching, most of the austenite in the samples transformed into martensite. The ferrite layer formed during the decarburization experiment is shown in Fig. 1. Where indicated, the as quenched samples were aged at RT prior to the extraction of APT specimens.

Wedges containing the ferrite/martensite (formerly austenite) interface were lifted out in a Zeiss NVision 40 dual beam microscope, sectioned, and mounted onto an array of silicon posts using tungsten deposition. These sections were sharpened into needle-shaped specimens suitable for APT by annular milling in the same microscope at 30 kV and successively lower beam currents from 1.5 nA to 40 pA. The gallium-contaminated layer was minimized by completing the ion milling at a lower accelerating voltage of 10 kV and 80 pA current. The specimens were analyzed using a Cameca LEAP 4000X HR atom probe, operating in laser-pulsing mode. The stage temperature was set at 47.5 ± 5 K and laser pulsing rate was 250 kHz. The laser pulse energy and the detection rate were varied between 50 pJ and 85 pJ and in the range of 3–6 ions detected per 1000 pulses, respectively, in order to maximize yield. Initial evaporation conditions were chosen to give a $\text{Fe}^{++}/\text{Fe}^+$ charge state ratio (CSR) of at least 100, to ensure that the specimens were not

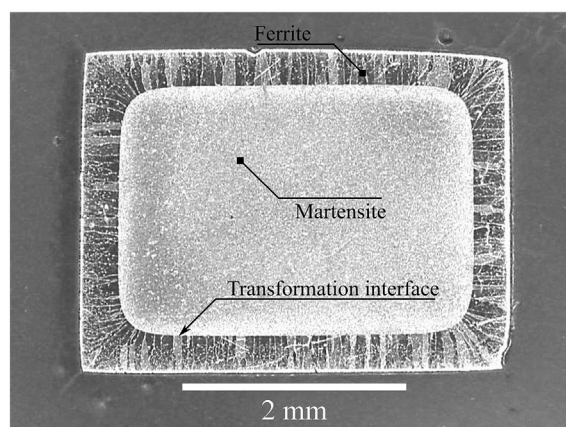


Fig. 1. Optical micrograph of a sample after decarburization. The large columnar grains visible at the surfaces of the sample are the ferrite grains grown during decarburization.

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