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COMPOSITIONAL ANALYSIS OF DUAL PHASE STEELS
BY TRANSMISSION ELECTRON MICROSCOPY

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I. INTRODUCTION

Steels heat treated to produce mixed structures of ferrite and martensite, termed dual phase steels, have emerged as a new generation of HSLA steels of high formability. The interest in these steels has grown enormously in the past few years as was demonstrated in recent symposia (1,2). One of the steps in the heat treatment to produce the dual phase structures consists of annealing the steel at an appropriate temperature in the two phase ($\alpha + \gamma$) region. During this annealing treatment, partitioning of alloying elements in the steel can occur depending on the competitive solubility in the ferrite and austenite for the specific partitioning species. Subsequent quenching to room temperature achieves a composite mixture of strong, tough martensite in a ductile ferrite matrix. Investigation of the partitioning phenomenon through chemical compositional analysis is of paramount importance with respect to understanding the resulting microstructure-mechanical property relationships. Determination of compositional changes due to partitioning cannot be accurately obtained from the

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equilibrium phase diagram because the two phase annealing times used in practice (less than 20 minutes) may lead to non-equilibrium situations, and, in any case, detailed phase diagram information on each steel is not usually available. Conventional analytical measurements involving electron probe X-ray fluorescence analysis on bulk samples suffer from poor spatial resolution and cannot provide accurate analysis from the fine grained martensitic islands in the dual phase microstructure. Hence, in the present investigation, the complementary techniques of high resolution lattice imaging and scanning transmission electron microscopy with X-ray energy energy dispersive capability have been utilized to determine compositions in two dual phase steels. High resolution electron microscopy has been shown to provide localized chemical compositions in alloys through correlation of lattice fringe spacing with composition (3,4). Carbon cannot be accurately detected by X-ray fluorescence analysis but since it has a pronounced influence on the lattice parameter, measurements of local d spacings by lattice fringe imaging can be used to evaluate carbon partitioning between ferrite and martensite (5). Lattice spacings in the ferrite (which essentially has zero carbon content for plain carbon steels) provide an internal standard for measuring the lattice spacing accurately in the martensite. Hence, factors affecting lattice fringe spacings such as electron-optical parameters and instrumental errors can be minimized or eliminated. The X-ray microanalysis technique was employed to estimate the partitioning of the substitutional alloying elements which, at small levels (≤ 1 wt.%), do not significantly affect the d spacing.

Carbon Analysis:

The sample used in this study was a AISI 1010 steel annealed at 1025K

in the ($\alpha + \gamma$) range for 10 minutes followed by water quenching. The volume fraction of martensite after the treatment was approximately 20%. The tetragonality of martensite increases with its carbon content as follows (6):

$$\begin{aligned} c &= 2.8664 + 0.116 \times \text{wt.\% C} \\ a &= 2.8664 - 0.013 \times \text{wt.\% C} \end{aligned} \quad (1)$$

The d spacing of any (hkl) plane in the tetragonal structure is related to the lattice parameter in the following manner:

$$\frac{1}{d^2} = \frac{h^2 + k^2}{a^2} + \frac{l^2}{c^2} \quad (2)$$

{011} are the preferred planes for lattice fringe imaging in the present investigation because of their large d spacing and higher probability of obtaining tetragonality. Figure 1 shows a bright field image of martensite and ferrite and the (101) lattice fringe image from the area marked is shown in Figure 2. Two beam tilted illumination mode was used for the lattice imaging. Using the d_{101} spacing for the ferrite as reference, $2.03\text{\AA}$, d_{101} for martensite was calculated to be $2.047 \pm 0.003\text{\AA}$ from the measured fringe spacings. This difference in d spacing can be attributed totally to the difference in carbon contents between the phases since no preferential partitioning of Mn was observed by x-ray STEM analysis. This increase in d_{101} spacing of $0.014\text{--}0.020\text{\AA}$ in martensite corresponds to a carbon content in the range of $0.50\text{--}0.65 \text{ wt.\%}$ based on equations (1) and (2). The high carbon concentration is expected since twinned substructure of martensite was seen as shown in Figure 1. This indicates the carbon level is $> 0.4\%$. Optical diffraction taken from the areas of ferrite, martensite and ferrite/martensite interface in Figure 2 are shown in Figure 3. The optical diffraction is an alternative method of evaluating the lattice parameters, in addition to direct densitometer measurements on the fringes

themselves (3). The martensite tetragonality and slight misorientation across the interface are clearly seen from the doublet reflections in Figure 3(c).

X-ray Microanalysis:

Having estimated the carbon distribution by lattice imaging, the X-ray microanalysis using STEM can then be used for evaluating partitioning of substitutional alloying elements ($Z \geq 11$). The 1010 steel analyzed above, STEM microanalysis showed no detectable partitioning of manganese. However, another (experimental) steel of current interest is one containing 1 wt.% aluminum, the design basis for which is described elsewhere (8). The steel was annealed at 1061K in the ($\alpha + \gamma$) range for 10 minutes, water quenched and after this treatment the martensite volume was about 20%. Analysis was conducted in thin samples prepared for electron microscopy using a Philips EM400 STEM equipped with X-ray energy dispersive capability. Average aluminum concentrations in ferrite and martensite were obtained from X-ray spectra generated from about 5 μ m regions of these phases. X-ray spectra were also obtained using a 400 $\text{\AA}$ probe adjacent to the ferrite/martensite interfaces. The X-ray data were analyzed using the thin foil ratio method (7) and the absolute aluminum concentrations are plotted in Figure 4. The aluminum concentration was uniform within the ferrite and martensite but aluminum had preferentially partitioned to ferrite. On an average, the aluminum concentration in ferrite was about double that of the aluminum concentration in martensite.

SUMMARY

In summary, this investigation illustrates how the partitioning of alloying elements can be quite accurately estimated using modern methods of transmission electron microscopy, viz., combining the lattice fringe

imaging method for elements such as carbon and X-ray microanalysis technique for substitutional alloying elements. This information is important in developing a better understanding of microstructure-mechanical property relationship in dual phase steels especially with regard to fine scale precipitation in ferrite (8).

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FIGURE CAPTIONS

- Figure 1. Dual phase AISI 1010 steel, bright field image of ferrite-martensite dual phase structure. M and F refer to martensite and ferrite regions, respectively.
- Figure 2. Dual phase AISI 1010 steel, high resolution (101) lattice fringe images of ferrite and martensite from the area marked in Figure 1. The ferrite/martensite (F/M) interface is inclined to the incident beam.
- Figure 3. Dual phase AISI 1010 steel, optical diffraction pattern from the lattice fringe image in Figure 2 by placing an aperture on (a) ferrite, (b) martensite and (c) both ferrite and martensite regions (F/M interface).
- Figure 4. Dual phase steel containing 1 wt.% aluminum; aluminum concentration profile in ferrite and martensite. The inset is a micrograph of an austenite/martensite interface. Points P_1 to P_4 are average concentrations of Al in ferrite and martensite, respectively. Points P_3 and P_4 were obtained using a $400\text{\AA}$ probe on either sides of the F/M interface.

Fig. 1

XBB 795-7068

Fig. 2

XBB 795-7069

a

F

$$d_{101} = 2.03 \text{ \AA}$$

b

M

$$d_{101} = 2.05 \text{ \AA}$$

c

F/M

Interface

XBB 795-7071

Fig. 3

Fig.4

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