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SOLUTE ELEMENT PARTITIONING
AND AUSTENITE STABILIZATION IN STEELS

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Considerable amounts of thin film retained austenite have been found in low to medium carbon lath martensitic structural steels. The existence of this metastable phase at room temperature has been attributed to several mechanisms which involve diffusion and redistribution of carbon during the austenite to martensite transformation. In this investigation electron microscopy (TEM), convergent beam electron diffraction (CBED) and field ion-atom probe (FIAP) analysis methods have been utilized to measure the C and other solute element distributions in retained austenite, γ , and martensite laths, α' . Considerable carbon partitioning to γ and to α'/γ interfaces occurs during cooling, but there was no detectable change in substitutional alloying elements (Cr, Mn, Ni, Si) between α' and γ . Thus while C plays a vital role in stabilization, it cannot account completely for this phenomenon. It is concluded that mechanical stabilization and the size effect are also major factors.

Introduction

Thin film retained austenite has important effects in medium carbon lath martensitic steels, both from the viewpoint of transformation mechanism and also because retained austenite improves the mechanical properties, especially toughness (1,2). The presence of this parent phase austenite in a martensitic structure allows accurate measurements to be made of the crystallography (3) leading to a better understanding of the nature of the lath martensitic transformation (4,5). Figure 1 is typical of the microstructure of such steels.

The existence of this high temperature γ phase at low temperatures may be attributed to several stabilization mechanisms. Since the redistribution of solute elements, especially carbon, may be significant in these mechanisms as

was indicated by lattice imaging methods (4), the problem of microchemical analysis of the microconstituents (Fig. 1) is obviously very important. In this case the carbon measurement is the key issue. It is difficult, if not impossible, to quantitatively measure carbon content in small volumes by EDS analysis in TEM/STEM instruments, and it is not possible to measure carbon in steels by electron energy loss spectroscopy (as our own experience verifies). However, the methods of convergent beam electron diffraction (via lattice parameter measurements over a few $100\text{\AA}$ cross sections (6)) and the very powerful atom-by-atom spectroscopic method of field ion-atom probe analysis (7) (spatial resolution laterally of $\sim 20\text{\AA}$) now enable more accurate and reliable chemical analyses to be performed on such steels (8). These techniques have been used in the present research.

Experimental Procedure

Several alloys were used in this investigation with carbon contents of 0.1 to 0.3 wt.% and with a total alloying addition (mainly Cr, Mo, Si, Mn, and Ni) of nearly 5 wt.% (see, e.g., ref. 2). The steels have relatively high M_s ($>300^\circ\text{C}$) and M_f ($>200^\circ\text{C}$) temperatures. Samples for examination were initially high temperature austenitized (1100°C) to ensure chemically homogeneous structure and then quenched in oil. Steel foils for TEM were prepared using twin-jet electropolishing in chromic/acetic acid solution at room temperature, and Cu (99.999%) and Ni (99.99%) standards for CBED in 25% HNO_3 in $\text{CH}_3\text{-COOH}$ at -30°C . Steel foils for CBED were etched by "dipping" method in 15% HClO_4 - 5% $\text{C}_3\text{H}_3(\text{OH})_3$ - CH_3COOH at -25°C . FIM tips were electropolished in 25% HClO_4 followed by 2% HClO_4 in 2-Butoxyethanol at room temperature. (TEM studies were accomplished by Philips EM400 at 100kV and the Atom Probe Analyzer was a conventional time-of-flight instrument (7)).

Results

The characteristic microstructure of the alloys (in the as-quenched and 200°C temperature condition) consists of dislocated "lath martensite" matrix and thin film retained austenite at the lath boundaries, as shown in the electron micrographs in Fig. 1.

The CBED method involves the determination of the shift in the position of the HOLZ lines in the CBED patterns in relation to the change in the lattice parameter of the retained austenite due to solute elements. Fig. 2 presents a result of such an experiment. Pure Ni and Cu samples were used for reference since the lattice parameters of these standards are close to those for austenite. The austenite was found to be enriched in carbon and the average carbon concentration was found to be 4.9 ± 0.6 at.%. (From $a_{\text{ret-}\gamma} = 3.578 + 0.044 \text{ wt\%C}$ (9), Δa is isomorphic).

A more detailed study of the distribution of solute elements is possible by FIM-AP studies. The example of Fig. 3a shows that while the C content in martensite is on the average 10% or more below the nominal carbon content of the alloy, there is considerable enhancement of C within retained γ and especially at the α'/γ interface. The distribution of C in the martensite matrix is very non-uniform due to autotempering. There is no apparent partitioning of the substitutional alloying elements (Cr,Mn) in either phase (Fig. 3a). Even at higher cooling rates ($\sim 10^3$ °C/sec), a similar C profile across α'/γ interfaces and enrichment in the γ was observed (Fig. 3b). This suggests that the partitioning of C occurs during the transformation of austenite to lath martensite since the redistribution is apparently independent of quench rate and tempering condition.

Of course, the main conclusion is that the transformation is not strictly martensitic because carbon diffusion is also occurring. This point is discussed in ref. 5.

Summary and Conclusions

The main results of this work are:

1. Carbon partitioning occurs during the transformation independent of quench rate up to 10^3 °C/sec.
2. The maximum carbon level is found at α'/γ interfaces.
3. No detectable substitutional element partitioning is observed.
4. The carbon content of retained austenite may be as much as 2.5at.%, i.e., twice the nominal composition. This implies that the M_s temperature of the retained austenite is decreased about 150°C (extrapolation from Fig. 4).

The implications of results 1-3 are obvious with regard to transformation mechanism and precipitation on tempering, but these will not be discussed further here. Result 4 is important with regard to austenite stability. Clearly composition effects alone do not account for the presence of the retained austenite. Mechanical stabilization and the size effect (10-11), i.e., the retained austenite exists as thin films, 50-250Å thick, must be the dominant factors. A high magnification micrograph showing the high degree of plastic deformation of the austenite is shown in Fig. 5. This deformation is created as a result of the growth of the martensite laths bounding the austenite. Destabilization takes place only by tempering when austenite to carbide reaction occurs or under high strains when the reaction to twinned martensite occurs (the martensite is twinned because of its higher C content).

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Figure Legends

- Fig. 1. Transmission electron micrographs: a) Bright field and b) dark field, reveal thin films of retained γ surrounding dislocated laths in the as-quenched Fe/3Cr/2Mn/0.5Mo/0.3C steels ($M_s = 320^\circ\text{C}$).
- Fig. 2. a) $\langle 111 \rangle$ Convergent beam electron diffraction pattern from a Cu crystal. b), c), and d) show the CBED patterns from the forward scattered beam from Ni, retained γ and Cu, respectively. All patterns at 100kV.
- Fig. 3. a) Field ion-atom probe analysis of C, Cr, Mn of the steel shown in Fig. 1 after tempering at 200°C for 1 hour. b) Atom probe analysis of C in 0.35C alloy following quenching 0.5mm dia. sample in iced water.
- Fig. 4. The effect of C on the M_s and M_f ; extrapolation gives the data for retained γ due to C enrichment, in Fe/Cr/Mn/0.3C steels.
- Fig. 5. High magnification a) bright field and b) dark field micrographs, show heavy deformation of retained austenite (Fe/3Cr/2Ni/0.5Mo/0.3C alloy), due to the impinging laths.

Fig. 1

XBB 817-7031

Fig. 2

XBL 818-10992

Fig. 3

Fig. 4

XBB 817-7030

Fig. 5

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