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A TEM study of the crystallography of austenite precipitates in a duplex stainless steel

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Abstract

The ferrite → austenite transformation in a duplex stainless steel serves as a good model alloy for studying body-centered cubic (bcc) → face-centered cubic (fcc) transformations in metals and alloys. However, the morphology and crystallography of austenite precipitates have not been well understood. A detailed TEM study was made on the crystallography of austenite precipitates to improve the understanding of this transformation. The orientation relationship (OR) was reassessed by an OR matrix to characterize the three-dimensional crystallography between austenite and ferrite. Meanwhile, interfacial dislocation structures were investigated for all of the three prominent facets by using the $\Delta\mathbf{g}$ method and the dislocation contrast extinction method. Each facet was found to contain a set of dislocations parallel to the long axis of precipitates. Their Burgers vectors have been characterized; one of them, the Burgers vector $[0\,0\,1]_f|[0\,0\,1]_b$, is reported for the first time in an fcc/bcc system. The O-line model was applied to explain the observations, and produced consistent results. However, some features cannot be explained by the O-line model. These will be further rationalized in the next paper.

Keywords: Phase transformations; Crystallography; Interface; Dislocation structure; TEM

1. Introduction

Many engineering alloys acquire comparably high-strength and toughness by solid–solid phase transformations, such as precipitation reactions. Their multiphase microstructures usually have reproducible crystallographic features. Lath- or plate-shaped precipitates are most commonly observed in engineering alloys. In many cases, they hold irrational orientation relationships (OR) with their matrix and a high-indexed habit plane in terms of both phases. Transformations between the face-centered cubic (fcc) and body-centered cubic (bcc) forms in metals and alloys have typically been studied by a number of investigators [1–9]. While most of transformations start from an fcc matrix and produce bcc precipitates, the ferrite → austenite transformation in duplex stainless steels is a case of the inverse transformation (bcc → fcc). Since both high-temperature ferrite and austenite can still exist at room temperature following water quenching, it is worthwhile investigating the crystallographic features of the transformation in a duplex stainless steel and analyzing how the matrix structure may affect the behavior of precipitation crystallography in fcc/bcc systems.

A number of studies of morphological crystallography have been made on the ferrite → austenite transformation in duplex stainless steels. Lath-shaped austenite precipitates holding the Kurdjumov–Sachs (K–S) OR with ferrite matrix were reported over 20 years ago [10]. Further investigations [11–14] indicated that the OR of the austenite precipitates is not exactly the ideal K–S OR, but deviates slightly from it. The austenite precipitates' long axis is close to the nearly parallel close-packed directions as observed in the Ni–Cr [6] and Cu–Cr systems [8], which have a similar

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lattice parameter ratio to that in a duplex stainless steel. However, neither of the two major facets of the austenite precipitates has an orientation near $\{121\}_f$ [6] or near $\{335\}_f$ [8]. Recently, Jiao et al. reported a high-resolution transmission electron microscopic (HRTEM) study of the interfacial defects between austenite precipitates and their matrix in a stainless steel [15]. A single set of regular dislocations were observed from each of the two major facets. However, the dislocation structure has not been completely characterized in their study because the Burgers vector for dislocations could not be directly identified just from the HRTEM results. Furthermore, another minor facet apparently shown in their micrograph (Fig. 3a in Ref. [15]) was not addressed at all. In the same paper, Jiao et al. applied the topological model of interface defects [16,17] to interpret their observations. Two different rational ORs were taken to explain the orientations of two major facets of one precipitate. While one major facet and the angle between nearly parallel close-packed planes are consistent with their calculation results, the angular discrepancy of nearly parallel close-packed directions and the long axis of precipitates could not be rationalized in the framework of the topological model.

This paper will present a systematic TEM analysis to the crystallographic features of austenite precipitates, including the OR, the orientation of facets, the direction of the long axis and a quantitative description of the interfacial defects in each prominent facet. The experimental results will be compared with those for both duplex stainless steels and other fcc/bcc systems in the literature, and the calculated results from the O-line model [18]. It should be noted that the majority of investigators have usually focused only on the orientation and the structure of the habit plane. In contrast, we aim to provide more complete descriptions of all facets enclosing the precipitates and interpret all the features of one precipitate within the framework of one model.

2. Experimental procedure

The alloy used in the present analysis is a commercial duplex stainless steel with the composition of Fe–24.9 wt.%Cr–7.0 wt.%Ni–3.1 wt.%Mo, which is similar to that used in the previous studies [11–15]. The alloy was prepared by arc melting in an argon atmosphere and forged into a bar of 10 mm × 10 mm. The bar was encapsulated by a large silica tube filled with high-purity argon (99.99%) and homogenized at 1300 °C for 3 days followed by water quenching. Each bar was further divided into a dozen blocks. These blocks were encapsulated in a smaller argon-filled silica tube and held at 1300 °C for 30 min. The sample was then transferred directly to a nearby furnace for the precipitation reaction at 960 °C for 10 min, followed by water quenching. A volume fraction of austenite of around 30–40% can be obtained after the above heat treatment. Slices 0.5 mm thick were electric discharge machined from the treated samples for TEM analysis. The slices were thinned to 50 μm for perforation by twin-jet electropolishing using a solution of 8% perchloric acid in ethanol with 50 V at −30 °C. A conventional JEOL 200CX TEM was used to determine the crystallographic features of these austenite precipitates.

3. Experimental observations

3.1. Precipitate morphology

Using a selective electrochemical etching method adopted from Ref. [19], we can reveal the three-dimensional morphology of austenite precipitates to some extent. Fig. 1 is an SEM image showing the morphology of austenite precipitates taken after selective etching for 30 s. The austenite precipitates under investigation always present a preferred growth direction, growing about 100–200 μm in length and 2–5 μm in the other two dimensions. These parallel precipitates all hold a similar rod-shaped morphology. The detailed morphological and crystallographic features will be described below.

3.2. Orientation relationship

Selected area diffraction patterns taken from more than 30 individual austenite precipitates confirmed a near-K–S OR between precipitates and their matrix. Fig. 2 shows a systematic deviation from the exact K–S OR. Fig. 2a and b shows two diffraction patterns taken at the same tilt condition but from a precipitate and its adjacent matrix near the interface, respectively. It was confirmed that the misalignment was not caused by local bending. The beam direction for the diffraction pattern in Fig. 2a is exactly parallel to $[0\bar{1}1]_f$, while the diffraction pattern in Fig. 2b shows a small deviation from $[1\bar{1}1]_b$. Fig. 2c is a superimposed diffraction pattern showing the small angle between a pair of nearly parallel conjugate planes. In this figure, only one pair of diffraction spots related to the conjugate planes are discernible because the specimen was tilted to ensure both $(111)_f$ and $(011)_b$ planes were in an edge-on

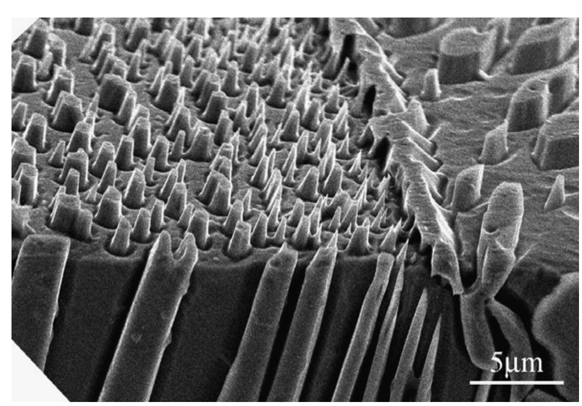

Fig. 1. SEM image showing the morphology of austenite precipitates after selective etching.