Materials Science and Engineering A 496 (2008) 256–261

Contents lists available at ScienceDirect

Materials Science and Engineering A

journal homepage: www.elsevier.com/locate/msea

Carbide precipitation and grain boundary plane selection in overaged type 316 austenitic stainless steel

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ARTICLE INFO

Article history:
Received 15 February 2008
Received in revised form 8 May 2008
Accepted 9 May 2008

Keywords:
Grain boundary plane
Five-parameter stereology
Sensitisation
Aging
Precipitation
Twin boundary

ABSTRACT

The misorientation and the grain boundary plane distribution in an overaged type 316 specimen have been measured, coupled with observations of $M_{23}C_6$ precipitation. There were many grain boundary planes on {1 1 1} in the random boundary population, notably {1 1 1} twist boundaries and ⟨1 1 0⟩ asymmetric tilt boundaries having one {1 1 1} plane. Precipitation was selective with respect to boundary plane type. Only coherent twins were immune to precipitation. For both random boundaries and incoherent Σ3s, boundaries which were not on {1 1 1} had more precipitation than did {1 1 1} twist boundaries. The enhanced proportion of {1 1 1} twist boundaries promoted plate-like carbide coarsening, which is associated with reduced cavity nucleation. A microstructure having a high proportion of {1 1 1} boundary planes, especially {1 1 1} twists, is therefore beneficial. It is suggested that the long age allowed a 'fine tuning' effect at interfaces whereby they approached energy minima.

1. Introduction

$M_{23}C_6$ type carbides precipitate intergranularly after solution treatment and aging of many austentic stainless steels causing sensitisation, i.e. depletion of the local concentration of chromium in the matrix. This is detrimental, both in terms of increased susceptibility to intergranular stress-corrosion cracking by removal of the protective chromium oxide and because the intergranular carbides form preferential sites for cavity nucleation and subsequent failure under creep-fatigue conditions. Hence, the phenomenon of $M_{23}C_6$ precipitation in austenitic stainless steels is an important area of study.

A few previous studies have been carried out which address the crystallography of carbide precipitation and the relationship to grain boundary category in type 316 austenitic stainless steel and other similar steels such as type 304 [1,2]. An example in type 304 showed that only low angle boundaries and coherent twins were not sensitised after a treatment of 3 h annealing at 650 °C. For all other boundaries, the majority of facets were sensitised. It was suggested that grain boundary planes should be measured in addition to misorientation to get more in-depth information [3]. In another study on 304 it was concluded that only coherent twins deterred precipitation and sensitisation [4]. More recently, the crystallographic features of $M_{23}C_6$ carbides in type 304 steel have been studied by TEM and linked to SEM-based observations. It was shown that carbide morphology could be plate-like or triangular. Triangular carbides precipitated predominantly at random boundaries whereas plate-like carbides precipitated at coincidence site lattice (CSL) boundaries. Boundaries with triangular carbides were less resistant to subsequent cavitation than were boundaries with plate-like carbides. Carbides were preferentially coherent to the grain for which one of the {1 1 1} planes made the smallest angle with the grain boundary plane [5]. A similar investigation was also carried out in type 316 steel. Here it was found that grain boundary serration can occur at an early stage of aging treatment, and this influences the carbide characteristics. Planar carbides were observed at serrated grain boundaries whereas triangular carbides were observed at flat boundaries. The interfacial plane of carbides at serrated boundaries was {1 1 1} [6]. It has also been observed that grain boundary engineered type 304 steel displayed higher resistance to intergranular carbide precipitation than conventionally processed alloy 304 [7].

The majority of these studies highlights that carbide precipitation during aging is influenced by the grain boundary plane, both in terms of the potential for precipitation on certain facets and more subtle effects such as the morphology and subsequent growth characteristics of carbides. Clearly there is a need for investigations which give more emphasis to measurement of the boundary plane crystallography during aging. The purpose of the present study therefore is to measure the misorientation and grain boundary plane distribution in an overaged type 316 steel specimen and to couple these data both with observations of pre-

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doi:10.1016/j.msea.2008.05.028

cipitate morphology and density, supplemented by single-section trace analysis on a subset of the sample population. The grain boundary plane distribution measurements are made using the new ‘five-parameter’ stereological analysis method in conjunction with orientation mapping by electron back-scatter diffraction (EBSD).

2. Experimental details and methodology

Specimens of 316 steel (0.07 wt% C, 17% Cr, 11% Ni, 2% Mo, 0.6% Si, 0.04 P, 0.02% S) were solution treated at 1200 °C for 30 min followed by water quenching. They then underwent a prolonged aging treatment of annealing at 750 °C for one week in air in order to coarsen $M_{23}C_6$ precipitates and hence to facilitate the study. Specimens were metallographically prepared using standard routes, with a colloidal silica polish as a final step. Backscattered imaging in a scanning electron microscope was used to obtain images of intergranular precipitates. An electron backscatter diffraction system from HKL Technology was used both to obtain and to analyse microstructure and grain boundary misorientation data. A step size of 2 μm was used for the orientation mapping. The total dataset comprised approximately 40,000 grains. The Brandon criterion was used to classify $\Sigma 3^n$ boundaries, with $n \leq 3$, i.e. Σ3, Σ9 and Σ27, both as a length fraction and as a number fraction.

All interface positions in the overaged specimen were reconstructed and displayed using an algorithm in the TSL (TexSEM Laboratories Ltd.) suite of EBSD software. The grain boundary plane distribution was then obtained by a stereological method known as the ‘five-parameter analysis’ which was designed in the Department of Materials Science and Engineering at Carnegie Mellon University. The methodology is described in detail elsewhere [8,9]. The distributions were calculated as ‘Multiples of a Random Distribution’, MRD, and displayed as density contours on stereographic projections with a resolution of 10°.

Data from the five-parameter analysis was supplemented by a single-surface trace analysis procedure. This is a rapid method which provides information on whether or not it is possible for an individual boundary plane or facet to be a particular crystallographic type, {1 1 1} in this case [10]. The procedure is as follows. The crystallographic boundary trace direction plus the orientation of each neighbouring grain provides four out of the five boundary parameters. A crystallographic vector *T*, the ‘trace vector’, can be calculated in the coordinate systems of both interfacing grains. This requires knowledge of the angle between the boundary trace and a reference direction in the orientation map, and the orientation matrix of both grains as measured by EBSD. Since *T* lies in the grain boundary plane it is orthogonal to the boundary plane normal vector *N* and the condition $N{\cdot}T = 0$ applies. This condition can be used to check if the boundary plane normal could be ⟨1 1 1⟩ in one or both grains by substituting ⟨1 1 1⟩ for *N*. If the condition $N{\cdot}T \neq 0$ applies in one or both interfacing grains when *N* is ⟨1 1 1⟩, then it is confirmed that the boundary cannot be on {1 1 1} since the plane must contain *T*. On the other hand if $N{\cdot}T = 0$ when *N* is ⟨1 1 1⟩, then the boundary plane might be on {1 1 1}. If the condition $N{\cdot}T = 0$ applies in *both* interfacing grains, then it is likely that the boundary is either a coherent twin (if the boundary is a Σ3) or a {1 1 1} twist boundary (if the boundary is not a Σ3). This calculation therefore supplies valuable information about the boundary plane crystallography: what the plane *cannot* be and what it is *likely* to be. In previous work where the method was rigorously tested and validated, for Σ3 boundaries analysed in brass, only 10% of cases were ambiguous in terms of recognising the type of Σ3 (coherent or incoherent) by the single-surface trace analysis compared to a full serial sectioning analysis [11].

In the present study ninety-seven grain boundaries having a straight trace were selected for the single-surface trace analysis. Those interfaces that were recognised from their morphology as being coherent twins were avoided in the sample population, because it is known that their interface plane is {1 1 1}. Boundaries could then be unambiguously identified if they were ‘not {1 1 1}’ as having traces in both interfacing grains which were <80° from *T*, therefore it was impossible for *N* to be ⟨1 1 1⟩. The ‘{1 1 1} or vicinal to {1 1 1}’ category had planes which were >80° from *T* in both grains and therefore it is possible for *N* to be ⟨1 1 1⟩.

3. Results and discussion

3.1. Grain size and misorientation analysis

The grain structure after solution treatment consisted of equiaxed grains containing many annealing twins. Neither the grain structure nor the grain size changed as a consequence of the long aging treatment. The grain size stability can be attributed to pinning of the grain boundaries by the precipitates, which had coarsened during long aging treatment, according to classical laws of grain growth control [12]. The proportion of Σ3 boundaries (which includes annealing twins) after aging was 47.6% as a percentage fraction of total boundary length and 28.8% as a percentage fraction of total numbers of boundaries. The fraction of Σ3 length or number did not increase during the aging treatment.

The difference in the length and number statistics indicates that the Σ3s are mostly present as long, straight, interfaces across grains, sometimes several per grain—i.e. classic annealing twinning. Fig. 1a shows this distinctive interface morphology on an orientation map. In Fig. 1a Σ3 interfaces which are within 2° of the reference misorientation (60° rotation about a ⟨1 1 1⟩ axis) are white whereas Σ3 interfaces which are 2–8.7° from the reference misorientation are coloured red. It can be seen that the straight type of Σ3s all have misorientations <2° from the reference whereas Σ3s with larger misorientation deviations tend to have more complex morphologies. The former Σ3s correspond usually to coherent annealing twins on {1 1 1} planes whereas the latter are incoherent Σ3s. Ten percent of Σ3s (by number) fall into the higher deviation category.

In Fig. 1a, $\Sigma 3^n$ boundaries Σ9 and Σ27, which mainly arise via Σ3 interactions, are featured as blue and yellow lines respectively. There are 1.4% Σ9 by length fraction, 3.0% by number fraction, and Σ27 fractions are <1%. The map background is shaded grey according to a diffraction pattern quality parameter, therefore high angle boundaries appear darker grey than the background. The black pixels in the map correspond to unsolved pixels. These are sited mainly at random boundaries and never on coherent twins, as discussed further below.

3.2. Five-parameter interface analyses

Data from many orientation maps of the type shown in Fig. 1a have been compiled to provide input for the ‘five-parameter’ analysis of the interface population. The distributions of boundary planes (for all the interface length in the sample population) are shown in Fig. 2a. The area distributions of grain boundary planes as a function of boundary normal are shown in stereographic projection in units of Multiples of a Random Distribution (MRD). There are pronounced peaks at {1 1 1} having an MRD value of 7.28. The high MRD peak overall reflects the high proportion of coherent twins, nearly half by length. Boundaries can be selectively removed from the five-parameter distribution if they meet the Brandon criterion for a Σ3 and if the grain boundary trace on the planar section is within 10° of the orientation expected for a coherent twin. When the data were