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# The interface character distribution of cold-rolled and annealed duplex stainless steel



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#### ABSTRACT

The interface character distributions (ICDs) of cold-rolled and annealed duplex stainless steel specimens, previously solid-solution-heated (SHT) at 1573 K and 1323 K, are investigated using electron backscatter diffraction (EBSD) and five-parameter analysis (FPA). For the  $\delta$ -ferrite phase, high concentrations of low angle grain boundaries (LAGBs) are developed, and the boundary planes are predominantly oriented on {111}. High angle grain boundaries (HAGBs) with misorientations ranging from 50° to 60° are mostly pure tilt boundaries, and the boundary planes are primarily located on {112}. For the austenite phase, very high density of coherent twin boundaries are introduced, implying that the boundary planes are exactly oriented on {111}. The phase boundary character distribution (PBPD) appears to be connected with the K-S and N-W orientation relationships (ORs) terminating on {110}  $_{\rm F}$ ||{111}  $_{\rm A}$  and {110} $_{\rm F}$ || $_{\rm A}$ |, respectively.

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#### 1. Introduction

Duplex stainless steels (DSSs) have been used for a variety of applications in the marine construction and chemical industries because of their excellent corrosion resistance, high strength, and high toughness [1–3], which are mainly controlled by their microstructures resulting from recrystallization and solid phase transformation. The combined process of recrystallization and solid phase transformation typically allows for refinement of the microstructure by means of appropriate thermal mechanical processing (TMP) in DSSs. In this case, an extremely high density of boundaries, such as interphase boundaries and grain boundaries, is introduced. In the past few decades, grain boundary and interface engineering have drawn increasing attention of material scientists and engineers [4–7]. It is now well established that grain boundary properties strongly depend on the grain boundary structure and character defined at least by the misorientation relationship between adjacent grains and the boundary inclination. Misorientation, i.e., the crystallographic orientation difference between adjacent grains, has been an important parameter for characterizing the grain boundary character distribution of polycrystalline materials. An important example is that of coincident site lattice (CSL) boundaries, which are

generally characterized by the parameter  $\sum$ , suggesting that low- $\sum$  CSL boundaries have special properties such as low boundary energy compared to the general high angle boundaries. Numerous studies [8–11] have been conducted to examine the grain boundary character distribution(GBCD)and its effect on boundary-related bulk properties, especially in FCC metals, in terms of CSL models. Moreover, the effect of boundary inclination on boundary-related properties has been given significant attention. Early studies focused on building up the relationship between boundary inclination and boundary energy using bicrystals. Recently, the distribution of grain boundary inclinations in polycrystalline materials has been studied by Rohrer et al. for metallic as well as ceramic materials using five-parameter analysis (FPA) [12–16].

To date, most of the studies have been dedicated to single phase metallic materials. Interface engineering involving interphase interface is still immature. Lee et al. [17] characterized the full five parameter heterophase interface distribution in a Cu-Nb alloy using two-dimensional electron back-scatter diffraction (EBSD) images for both physical vapor-deposited (PVD) Cu-Nb film and Cu-Nb multilayer composites alloyed by accumulative roll-bonded (ARB). It was found that the predominant Cu-Nb interface that developed in ARB was different from that developed in PVD. Additionally, the texture in the vicinity of the interface and the selection of heterophase interfaces in ARB Cu-Nb multilayers were mutually correlated. A similar examination of interface

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character distribution (ICD) was done for WC-Co composites [18–19]. The interphase boundary character in the above-mentioned cases was investigated in composites, in which the recrystallization or annealing behavior may be considered in explaining the evolution of ICD. However, the effect of solid-solid phase transformation and especially the combined effect with recrystallization has not yet been examined. In the current study, in order to understand the combined effect of recrystallization and phase transformation on the interface character distribution, two DSS specimens having different initial microstructures are subjected to severe cold rolling and subsequent annealing. The ICDs are then examined by means of FPA based on EBSD measurements.

#### 2. Experimental

#### 2.1. Sample preparation

Type UNS S31803 duplex stainless steel (DSS) was used in this study. The chemical composition is 22.16 wt.% Cr, 5.28 wt.% Ni, 3.09 wt.% Mo, 1.11 wt.% Mn, 0.017 wt.% C, 0.0016 wt.% S, 0.026 wt.% P, 0.151 wt.% N, and 0.52 wt.% Si. Two DSS hot-rolled plates with dimensions of 60 mm  $\times$  20 mm  $\times$  15 mm (length  $\times$  width  $\times$  thickness) were first solution-heat-treated (SHT) for 30 min at 1573 K (specimen A) and 1323 K(specimen B), respectively, and water quenched. Then, these two SHT plates were subjected to identical cold rolling with a true strain of  $\epsilon=3$  and subsequent annealing for 2 h at 1323 K, which are hereinafter referred to as specimens CRAnn-A and CRAnn-B.

#### 2.2. EBSD mapping

EBSD mapping was conducted on the rolling plane (RD  $\times$  TD) in the as processed specimens. The EBSD system was a HKL-Channel 5 attached to a Sirion-200 field emission scanning electron microscope (FESEM). The step size of EBSD mapping was 2 µm, and each mapping covered an area of 200  $\mu m \times 180 \mu m$ . There were typically two types of interfaces: Grain boundaries (GBs) and phase boundaries (PBs). Here, an individual grain was defined as a region which was completely bound by phase boundaries and/or grain boundaries having a misorientation angle larger than the critical value of 2°. The fractions of the two boundary (GB and PB) types were determined on the basis of the length fraction. To ensure statistically significant results, no <30 EBSD mappings were obtained for each specimen, and at least 10 EBSD maps were combined using Map Stitcher provided by Channel-5 and used for the measurements of texture and orientation difference distributions between adjacent grains. Misorientation in the form of angle/ axis pairs  $(\theta/\langle uvw \rangle)$  for GBs and orientation relationship (OR) in the form of parallelism conditions applied to the crystallographic planes and directions  $(\{hkl\}_A \|\{hkl\}_F, \{uvw\}_A \|\{uvw\}_F)$  for PBs were employed to evaluate the orientation difference between adjacent grains.

For the EBSD procedure, the specimens were mechanically ground on standard emery papers down to No. 2000 followed by electropolishing in a solution of  $HClO_4:CH_3COOH=20:80$  (volume fraction) under 30 V for 20 s.

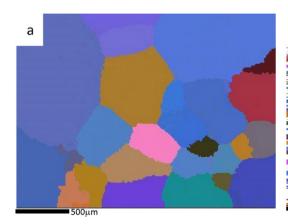
## 2.3. Interface character distribution (ICD) measurement (segmentation of interface boundary)

Initial EBSD orientation, expressed as 3 Euler angles, along with the phase ID and coordination (x, y) from each pixel, were exported and input to our own custom-designed programs for extracting all the interface boundary (including both GBs and PBs) data. If the orientation difference between two neighboring grains is larger than the critical value of 2°, GBs with identical phase IDs or PBs with different phase IDs were determined and reconstructed. Each interface boundary (IB) connecting some triple junctions and quadruple nodes can be broken up into several line segments, which are referred to as boundary traces, using the Douglas-Peucker compression algorithm [20]. The direction of the trace and the lattice orientation across each trace specify four of the five parameters necessary for determining the ICD,  $\lambda(\Delta g, n)$ , which is defined as the relative areas of distinguishable IBs characterized by their lattice misorientation ( $\Delta g$ ) and boundary plane orientation (n), using the five parameter analysis developed by Rohrer et al. [21–22]. The ICD is measured in multiples of a random distribution (MRD) and values greater than one indicates that planes are observed more frequently than expected in a random distribution. In the current paper, >50.000 GB traces for each phase, i.e., austenite and ferrite, and >80.000 PB traces for each specimen were analyzed.

#### 3. Results and discussion

#### 3.1. Initial microstructure

EBSD-reconstructed microstructural maps of specimens subjected to SHT at 1573 K (specimen A) and 1323 K (specimen B) for 30 min are shown in Fig. 1a and b, respectively. A fully ferritic structure was produced in specimen A, and the mean grain size of the ferrite was 550  $\mu m$ . However, in specimen B, a typical banded duplex structure of alternating austenite and ferrite grains elongated in the rolling direction was observed. The mean grain sizes were 20  $\mu m$  and 38  $\mu m$  for the austenite and ferrite, respectively.



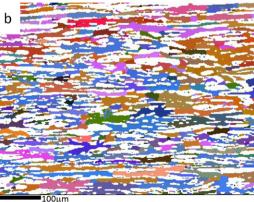


Fig. 1. EBSD-reconstructed microstructure of initial specimens subjected to solid solution treatment at 1573 K (a) and 1323 K (b) for 30 min, respectively. The colored and white areas represent ferritic and austenitic phases in (b), respectively.

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