Ti-2003
Science and Technology
Proceedings of the 10th World Conference on Titanium
Held at the CCH-Congress Center Hamburg, Germany
13–18 July 2003

Volume IV

Edited by
G. Lütjering and J. Albrecht
The Grain Boundary Character Distribution in BCC and O+BCC Ti-Al-Nb Alloy Microstructures

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Abstract

The grain boundary character distribution (GBCD) of the body-centered-cubic (BCC) phase in the orthorhombic (O) + BCC class of Ti-Al-Nb alloys was investigated. The alloy compositions ranged between 12-23at.%Al and 25-38at.%Nb and the processed sheet microstructures were either fully-BCC or O+BCC. The alloys were processed by pancake forging and hot-pack rolling followed by heat treatments varying within the O, BCC, and O+BCC phase regimes. Through scanning electron microscopy (SEM) and electron backscatter diffraction (EBSD) the effects of sheet orientation, alloy composition, and grain size on the GBCD were evaluated. No strong significant correlation was observed between any of the studied microstructural parameters. The low-angle boundaries (LAB) fraction was least prevalent throughout the fully-BCC microstructures and always less than 0.1, while general high-angle boundaries (GHABs) were dominant. The fraction of special boundaries was the minority and typically ranged between 0.25-0.3. The fine-grained BCC-phase present in the subtransus O+BCC microstructures exhibited more LABs than coincident site lattice boundaries (CSLBs), though the total GHAB fractions were not significantly different than the supertransus microstructures. Using EBSD, the twin-related O-phase variant interfacial planes were identified and quantified. The variant boundaries are near {110} or {130} and represented over 33% of all O-phase boundaries. This indicates the potential to modify the GBCD in the O-phase to enhance properties in Ti-Al-Nb alloys.

1 Introduction

Intermetallics may exhibit attractive mechanical, physical, and chemical properties, which make them candidate materials for applications in severe conditions such as high temperatures and corrosive environments where intergranular failure can be prevalent. One important microstructural feature that significantly influences intergranular behavior is the grain boundary structure. Optimizing grain boundary structure is accomplished by exerting control over the types of grain boundaries through grain boundary engineering (GBE) [1], which may permit property improvements at the boundary without detrimentally affecting phase stability or thermal conductivity. This is accomplished through thermomechanical processing (TMP) treatments involving strain and recrystallization that cause special boundaries to replace random boundaries, thereby altering the GBCD, when the appropriate conditions are obtained. Intergranular fracture, corrosion, cavitation, and creep are directly influenced by boundary structure, hence, control of boundary structure is a potent tool for improving overall material
performance. GBE has shown that one can significantly improve mechanical properties and corrosion resistance for a variety of pure metals and alloys, including in-service high-temperature Ni-based superalloys as well as intermetallics [2-12]. For L12 intermetallics, it has been shown that LABs and twin boundaries (Σ3) are particularly resistant to intergranular fracture while GHABs are particularly prone to intergranular fracture [13-15]. Therefore, increasing the fraction of LABs and twin boundaries can increase the fracture strength of an intergranularly brittle intermetallic. Though significant property improvements have been demonstrated, GBE is still in its infancy and a significant window of opportunity exists for optimizing the mechanical behavior of high-temperature alloys and intermetallics. In addition to Ni-based superalloys, which are based upon the face-centered cubic (FCC) structure, the GBCD of BCC alloys can also be significantly altered through TMP. In fact, it has been shown that special boundary fractions between 0.9-0.97 can be obtained for the B2 intermetallic FeAl through shock loading and subsequent annealing [12]. However, the effect of TMP on the GBCD of Ti-Al-Nb B2 intermetallics and alloys has yet to be evaluated. Based on the dramatic improvement in properties for FCC alloys, there is potential for significantly improving the mechanical behavior of non-FCC alloys through GBE. In the present work, experimental results on the relation between GBCD, alloy composition, grain size, and sheet orientation for Ti-Al-Nb alloys containing 50 at.% Ti are presented. The ratios of Nb:Al were 3.17, 1.17, and 1. In addition, this work also examines the GBCD of the O-phase as influenced by transformation twin relationships.

2 Experimental Procedure

2.1 Materials and Processing

The alloy chemistries, nominally Ti-12Al-38Nb, Ti-23Al-27Nb, and Ti-25Al-25Nb(at.%), and details of the processing treatments are provided in references [16,17]. The starting materials of this work comprised vacuum or arc melted ingots. Hot working procedures consisted of pancake forging and pack rolling performed at 1000°C, 982°C, and 932°C for the Ti-25Al-25Nb, Ti-23Al-27Nb, and Ti-12Al-38Nb alloys, respectively. The total effective true shear strain imparted on the ingots during processing was on the order of three.

Spatially resolved EBSD orientation maps were obtained from colloidal silica-polished sections using either a Phillips XL30 field emission gun SEM or a Phillips 515 SEM with a LaB6 filament. EDAX-TSL, Inc., Draper, Utah manufactured the EBSD hardware and

![Diagram of rolling directions]

**Figure 1:** Schematic illustrating the sheet orientations of the software. The Brandon criterion [18] was used to distinguish between GHABs and CSLBs. Special boundary fractions were then defined as LABs+CSLBs. Using this criterion the population of the grain boundaries is divided into three types. LABs were those with a misorientation less than 15°. CSLBs were those characterized by 1-Σ≤27, where Σ is the reciprocal density of the coincident site lattice points between two grains at a boundary.
GHABs were those with a misorientation greater than 15° or when Σ>27. The reported fractions of GHABs, LABs, and CSLBs were the average values taken from several orientation maps, performed on the cross-sections (CS), rolling faces (F), or longitudinal (L) sections of the processed sheets (see Figure 1). For each specimen more than 500 boundaries were analyzed and a step size of between 1.25-2μm was chosen. An additional high-resolution scan with a step size of 100nm was performed to investigate details of the O-phase GBCD. The minimum boundary tolerance angle selected was five degrees.

3 Results and Discussion

The heat treatments, grain sizes, and GBCD parameters are listed in Table 1. The post-rolling heat treatments were either above or below the BCC-transus temperature, and equiaxed microstructures resulted. Larger BCC grain sizes resulted from higher solution-treatment temperatures [17,19]. The higher Al-containing alloys exhibited the ordered-BCC (B2) structure, while Ti-12Al-38Nb favored the disordered-BCC (β) structure [17,19].

Table 1. The BCC-phase grain boundary character distribution parameters of the Ti-Al-Nb alloys

<table>
<thead>
<tr>
<th>Heat Treatment</th>
<th>CSLBs</th>
<th>GHABs</th>
<th>LABs</th>
<th>CSLBs+LABs</th>
<th>Σ3</th>
<th>d (μm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ti-12Al-38Nb</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>As-melted ingot</td>
<td>0.256</td>
<td>0.657</td>
<td>0.087</td>
<td>0.343</td>
<td>0.218</td>
<td>572</td>
</tr>
<tr>
<td>900°C/5h/WQ</td>
<td>0.213</td>
<td>0.744</td>
<td>0.043</td>
<td>0.256</td>
<td>0.176</td>
<td>138</td>
</tr>
<tr>
<td>950°C/5h/WQ</td>
<td>0.223</td>
<td>0.722</td>
<td>0.050</td>
<td>0.278</td>
<td>0.168</td>
<td>198</td>
</tr>
<tr>
<td>1200°C/7h/CC</td>
<td>0.274</td>
<td>0.710</td>
<td>0.016</td>
<td>0.290</td>
<td>0.255</td>
<td>337</td>
</tr>
<tr>
<td>1200°C/7h/CC</td>
<td>0.301</td>
<td>0.694</td>
<td>0.005</td>
<td>0.306</td>
<td>0.291</td>
<td>577</td>
</tr>
<tr>
<td>Ti-23Al-27Nb</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1125°C/4h/WQ</td>
<td>0.224</td>
<td>0.729</td>
<td>0.047</td>
<td>0.271</td>
<td>0.193</td>
<td>204</td>
</tr>
<tr>
<td>900°C/5h/WQ</td>
<td>0.133</td>
<td>0.696</td>
<td>0.171</td>
<td>0.304</td>
<td>0.005</td>
<td>2.3</td>
</tr>
<tr>
<td>950°C/7h/CC/650°C/30h/WQ</td>
<td>0.113</td>
<td>0.770</td>
<td>0.117</td>
<td>0.230</td>
<td>0.039</td>
<td></td>
</tr>
<tr>
<td>Ti-23Al-25Nb</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1150°C/4h/WQ</td>
<td>0.175</td>
<td>0.752</td>
<td>0.073</td>
<td>0.248</td>
<td>0.120</td>
<td>660</td>
</tr>
<tr>
<td>1040°C/5h/WQ</td>
<td>0.199</td>
<td>0.715</td>
<td>0.086</td>
<td>0.285</td>
<td>0.101</td>
<td>100</td>
</tr>
</tbody>
</table>

d: average grain size of BCC phase; WQ: water quenched; CC: control cooled at 15°C/minute to RT.

3.1 The BCC-Phase GBCD for Super-Transus Heat-Treated Samples

Figure 2 illustrates EBSD data including an orientation map, an image quality map with highlighted boundaries, and pole figures for a Ti-25Al-25Nb fully-B2 microstructure. The pole figures indicate that the microstructure was not strongly textured, and this was the case for all the fully-BCC microstructures evaluated. The image quality map indicates that the majority of the equiaxed BCC grain boundaries were GHABs. The LAB fractions of the fully-BCC microstructures were the minority and always less than 0.1. The Σ3 and CSLBs fractions typically ranged between 0.15-0.3. Special boundary fractions ranged from approximately 0.25-0.30, suggesting that further TMP is necessary to promote large special boundary fractions for fully-BCC Ti-Al-Nb alloys. The GBCD was not dependent on spatial position within the sheet, as similar GBCD parameters were characterized on the L, T, and F sections. For Ti-12Al-38Nb, GHABs decreased slightly and CSLBs increased slightly with increasing grain size. Overall, however, the GBCD for the fully-BCC microstructures was not strongly dependent on grain size, sheet orientation, or alloy composition, and the processing treatments changed the GBCD of the as-melted ingot only slightly, see Table 1.
Comparing the current data with other BCC alloy systems, low special boundary fractions have also been noted for textureless B2 FeAl and NiAl intermetallics [12]. However, high-pressure shock-loading followed by annealing of B2 FeAl leads to subgrain formation and LAB fractions greater than 0.9, while for NiAl shock loading did not lead to subgrain formation [12]. LAB fractions less than 0.15 have also been observed in textureless B2 alloys, including Fe-39-46Al (at.%) and B2 NiAl [12], as well as disordered BCC alloys, including Fe-3.1Si (wt.%) [20].

3.2 Sub-Transus Heat-Treated Samples

An equiaxed Ti-23Al-27Nb two-phase O+BCC microstructure, containing less than 50% B2 phase by volume is illustrated in the EBSD orientation maps of Figure 3a and b. It is noted that neither of the phases were sharply textured as depicted by the pole figures. The GHABs were dominant for the B2 phase in the subtransus annealed O+B2 microstructures and the minimum GHAB fraction was 0.7, see Table I. The LAB fractions were slightly greater than the CSLB fractions for these microstructures, and the Σ3 fractions were less than 0.05. Thus, twin formation in the BCC phase is largely impeded for O+BCC microstructures.

Dependent on the aging schedule and alloy composition, the O-phase can grow from the BCC phase through several methods including martensitic transformation and cellular precipitation. For Ti-23Al-27Nb subtransus heat-treated two-phase O+B2 microstructures, upon aging below 850°C the equiaxed B2-phase undergoes a transformation characterized as cellular precipitation according to the reaction [17]:

B2 →

The results moving 1 depicted treated w regime, two-phs regime di
B2 $\rightarrow$ $\beta$+O

The resultant cellular structure replaces the equiaxed B2-phase through a discontinuous moving front initiated at the equiaxed B2-phase boundaries [17]. Such a microstructure is depicted in Figure 4a and b, which illustrates EBSD data for a Ti-23Al-27Nb sample heat treated within the O+BCC phase field followed by water quenching then aging in the O-phase regime. The GBCD parameters were similar for the B2 phase for both the aged and unaged two-phase microstructures of Figures 3a and 4a (see Table 1). Thus, aging in the O-phase regime did not significantly affect the subtransus B2 GBCD.

![Figure 3: EBSD orientation map and pole figures for a 900°C/5.5h/WQ heat-treated O+BCC Ti-23Al-27Nb microstructure (L section): (a) BCC-phase data (b) O-phase data](image)

Twin-related O-phase variant boundaries were characterized, using the twin-identification technique for EBSD previously described by Mason et al. [21] and Bingert et al. [22], in the equiaxed O-phase grains for both the aged and unaged microstructures of Figures 3b and 4b. These boundaries were most likely a result of $\alpha_2$→O transformation, from the ingot to the processed microstructure, which results in twin-related O-phase variant interfacial plane formation as described by Muralleedaran et al. [23]. The hcp/O orientation relationship results in variant boundaries very near {110} or {130}, however they were not uniformly distributed between the two planes. The {110} plane twin variants were characterized by an approximately 64.4° rotation about [001], and using a boundary tolerance of two degrees, these boundaries measured 28% of all the O-phase boundaries, see Figure 5. The {130} plane twin-related variants were characterized by an approximately 55.6° rotation about [001] and
Figure 4: EBSD orientation map and pole figures for a 950°C/45s/HQ/650°C/300s/HQ heat-treated O+BCC Ti-23Al-27Nb microstructure (F section), which exhibited equiaxed O grains and cellular precipitation within B2 grains: (a) BCC-phase data (b) O-phase data.

Represented 5% of the O-phase boundaries. The discrepancy in misorientation from 60° can be justified by the distortion between the α2-to-O transformation product reported by Mozur et al. [24] as described in reference [23], while the relative clockwise or counterclockwise rotation of variants results in the two distributions straddling 60°. The fine scale details of the misorientation distribution could have been affected by relative fractions between O1 and O2 phase designations which have not been distinguished here. Insignificant fractions of twin-related boundaries were observed at near-30° and 90° rotations about [001]. The dearth of non-60° variant interfaces, the former of which would be expected to be the majority variant boundary type [23], may result from differences in strain energy affected by composition, lattice parameter, and processing conditions. Thus, more than 33% of all the O-phase boundaries were twin related, indicating the potential for GBE the O-phase in Ti-Al-Nb microstructures. For the aged microstructure the [110] and [130] boundary plane variants measured 13% and 4%, respectively. Lower fractions would be expected in the aged microstructure as the additional O boundaries created by the cellular structure were not associated with the α2 phase transformation. For near Ti2AlNb alloys, the flexibility to

change and establish 17μm of resistance exhibits microstr...
change the subtransus microstructures through post-processing heat treatment has been established previously where the O-phase grain sizes and volume fractions ranged from 4-17μm and 40-100%, respectively [17]. The mechanical properties, and in particular creep resistance, varied considerably for such microstructures and the greatest creep resistance was exhibited by the fully-O microstructure [25]. The GBCD of the O-phase in O-dominated microstructures has yet to be fully evaluated and will be a target of this ongoing work.

![Image](image)

Figure 5: (a) An EBSD image quality map highlighting an O-phase twin-related variant interfacial plane and the corresponding (b) (001) and (110) pole figures taken from the aged microstructure represented in Figure 4b. The coincident points from the twin-related orientations are circled on the pole figures, which were used to identify the interfacial plane, (110), and the orientation, 64.4° about [001]. Using a 2° tolerance angle, all such twin-related boundaries are indicated in (c) for the unaged microstructure represented in Figure 3b.

4 Summary and Conclusions

The GBCD of single-phase BCC Ti-Al-Nb microstructures obtained through hot forging and rolling followed by super-BCC transus heat treatment was investigated using EBSD. The fully-BCC microstructures were not sharply textured, and no significant trend in GBCD parameters was recorded through changing grain size (grain size ranged between 100-660μm), sheet orientation, or alloy composition. The GHABs dominated the equiaxed BCC grain boundary network, and they always maintained more than 0.69 of the entire BCC boundaries. The LAB fraction was least prevalent and always less than 0.1. Twin boundaries, which dominated the CSLB fractions, ranged between 0.17-0.3.

Slight differences in the GBCD were observed between the BCC GBCD of the subtransus and supertransus microstructures. The subtransus B2 phase was dominated by GHABs while the CSLB and LAB fractions were almost evenly distributed. Aging in the O-phase regime did not significantly affect the subtransus B2 GBCD. Overall, the results indicate that the hot work and annealing treatments performed did not dramatically change the GBCD from the as-cast condition. It was determined that additional TMP would be required to produce large BCC-phase special boundary fractions.
Using EBSD, the twin-related O-phase variant interfacial planes, most likely formed from α3-to-O transformation, were identified and quantified. The (110) boundary plane twin variants, characterized by an approximately 64.4° rotation about [001], represented up to 28% of the O-phase boundaries, while up to 5% of the O boundaries exhibited {130} planes and were associated with an approximately 54.6° rotation about [001]. Thus, EBSD analysis proved to be a useful technique for determining the fraction of O-variant plane boundaries and distinguishing variant selection. Results suggest manipulation of the O-phase transformation variant boundaries may provide fertile ground for GBE of Ti-Al-Nb alloys.

5 Acknowledgments

This work was supported by NSF (DMR-0134789) and NYSTAR (8CO20080), and both JFB and CJB were supported by the U.S. Department of Energy under Contract W-7405-Eng-36.

6 References

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