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ScienceDirect

Scripta Materialia 58 (2008) 183–186

Scripta MATERIALIA

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Five-parameter grain boundary analysis of a titanium alloy before and after low-temperature annealing

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Received 16 July 2007; revised 17 August 2007; accepted 25 September 2007
Available online 25 October 2007

The five-parameter grain boundary analysis has been applied for the first time to a titanium alloy. The boundary distribution was related to the β-to-α phase transformation and to deformation twinning. There was a very strong planes texture, only partially related to twinning, which did not diminish during long-duration, low-temperature annealing.

Keywords: Electron back-scatter diffraction; Titanium; Grain boundary plane; Five-parameter analysis

Although there have been numerous investigations of the distribution of grain boundary types in polycrystals of cubic metals and alloys, relatively few parallel studies have been carried out on hexagonal metals. By use of standard electron back-scatter diffraction (EBSD) software, it is a straightforward task to characterize the boundary misorientation population in a hexagonal metal such as commercially pure titanium. This has recently been carried out and reported, as a precursor to the present work [1].

In the last decade or so EBSD measurement technology has evolved considerably. A particularly exciting development is the measurement of the statistics of all five grain boundary parameters. This procedure, known as the "five-parameter analysis", results in the grain boundary plane orientation distribution in addition to the misorientation distribution [2]. This is a landmark advance because the planes distribution reveals important information about grain boundary structure that is unavailable from misorientation alone. For example, in brass there is a preponderance of {1 1 1} planes, even after twins have been excluded from the data set [3].

The only five-parameter analysis published so far on a material with a hexagonal crystal structure has been the measurement of the distribution of WC/WC grain boundary types in a WC–Co composite [4,5]. It was found that 90° twist $[10\bar{1}0]$ grain boundaries, 30° twist [0 0 0 1] grain boundaries and 90° asymmetric $[\bar{2}110]$ grain boundaries had populations of 150, 18 and 8 multiples of a random distribution (MRD), respectively. Data input for the five-parameter analysis requires high-resolution orientation data from at least 80,000 grains, which is one reason why so far there has been no published work on hexagonal alloy systems, despite interest in how boundary planes are distributed in these technologically important materials. The objective of the present work is therefore to validate the five-parameter boundary analysis for titanium, and furthermore to explore, for the first time, which boundary planes occur after a low-temperature anneal performed in order to stabilize the grain boundary structure.

The alloy Ti–6%Al–4%V was selected for analysis. It was in sheet form, 10% cold rolled, and processed to be cph (referred to as Ti64). In order to encourage the equilibration of boundary planes, a low-temperature (480 °C, $0.38T_m$) anneal was carried out in air for 72 h on some of the material. Hence two specimens were examined: a reference specimen and an annealed specimen. The specimens were cut, mounted and metallographically prepared using standard techniques. An EBSD system (Channel5) from HKL Technology was used to obtain a large number of orientation maps, comprising approximately 80,000 grains from each specimen. Microtexture and misorientation (according to the length fraction of the boundary) were extracted from the maps. The microtexture and misorientation statistics were derived from the Channel5 Mambo and Tango software suites. The Brandon criterion was used

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1359-6462/$ - see front matter
doi:10.1016/j.scriptamat.2007.09.044

to categorize coincidence site lattice (CSL) boundaries in the hexagonal crystal system.

The five-parameter grain boundary distribution was characterized using software developed at Carnegie Mellon University, Pittsburgh, USA. A stereological procedure coupled with automated trace analysis software was used to extract the five-parameter distribution from EBSD maps. The procedure is described in detail elsewhere [6]. The assumptions used in this procedure are not affected by crystallographic symmetry and, although it has been applied mostly to cubic systems, results from hexagonal [4] and tetragonal [7] systems have been reported. In each case, the most commonly occurring twin planes were accurately identified. The five-parameter area distributions are expressed as "multiples of a random distribution" (MRD).

The misorientation angle distributions for the reference and annealed specimens are shown in Figure 1, expressed as relative frequency, i.e., fraction of total grain boundary line length. The random distribution, i.e., the misorientation distribution for randomly textured orientation pairs, is also included on the plot [8,9]. The experimental distributions are clearly non-random. The misorientation angle distribution for the reference specimen in Figure 1 shows a very large peak in the range 58–65°. Its breadth indicates that it could comprise multiple, overlapping peaks. A slightly smaller peak is present at 90° and a smaller one at 10°. However, these peaks should be referred to the random distribution, whereupon the maximum at 10° is relatively the greatest peak. In the misorientation axis distribution (not shown here) there is a large density concentration close to $\langle 2\bar{1}\bar{1}0\rangle$ in the 55–65° section and close to $\langle 2\bar{1}\bar{1}0\rangle$ in the 85–90° section. The very large misorientation peak at 55–65° could be associated partly with deformation twinning (because the material was deformed 10%) where the misorientation $57.4°/[2\bar{1}\bar{1}0]$ corresponds to a deformation twin (see below), although mechanical twinning in Ti64 is not commonly observed at such low strains [10]. After annealing, the misorientation distribution has changed slightly in that the peak in the range 58–65° is reduced slightly, the peak at 90° has reduced by more than half and there is no longer a peak at 10°.

In addition, the misorientation distribution has been influenced by the β-to-α phase transformation in titanium, where β is body-centred cubic (bcc) and α is hexagonal close packed (hcp). This phase transformation in titanium frequently obeys the Burgers orientation relationship, i.e., $(110)\beta \| (002)\alpha$ and $[\bar{1}1\bar{1}]\beta \| [2\bar{1}0]\alpha$. There are 12 equivalent crystallographic variants of the transformation. If there is no variant selection during the phase transformation, several different variants may nucleate within the same prior β grain. Boundaries between the variants would therefore produce misorientations of $60°/\langle 11\bar{2}0\rangle$, $60.83°/\langle 1.377, -1, 2.377, 0.359\rangle$, $63.26°/\langle 10, 5, 5, \bar{3}\rangle$, $90°/\langle 1, -2.38, 1.38, 0\rangle$, $10°/\langle 0001\rangle$ (e.g., [11]). For random variant selection, the expected ratio for occurrence of the five variants listed above is 2:3:2:2:1 [12].

The list of misorientation variants corresponds exactly to the peaks observed here in the misorientation angle and axis distributions of the reference specimen. In the experimental data the first three variants in the list are difficult to distinguish and have merged to give a rather broad peak in the region of 60° misorientation angle and $\langle 2\bar{1}\bar{1}0\rangle$ misorientation axis. It is clear that evidence of the β-to-α phase transformation has persisted in the reference structure even after 10% deformation. There is little evidence of variant selection because the ratio of occurrence of the variants is close to that for a random distribution. After annealing the presence of phase transformation variants is reduced.

The CSL model can also be used as a categorization tool for the misorientation distribution. The Σ13b boundary, which is a deformation twin, occupies 20.8% of the boundary length. Two other twins, Σ11b and Σ19c, were observed at much lower concentrations, 1.1% and 4.0%, respectively. The Σ19b CSL, which is not a twin, was also observed. This distribution is in good agreement with recent work on commercially pure titanium, deformed 40% [13]. After annealing the main change is that the proportion of Σ13b reduced from 20.8% to 13.3%. However, the total length of boundary misoriented on $\langle 2\bar{1}\bar{1}0\rangle$ increased very slightly after annealing from 25.8% to 26.8%. The misorientation statistics for both the reference specimen and the annealed specimen, including twin systems where appropriate, are summarized in Table 1.

Figure 2a and b shows the microtexture of the reference and annealed specimens, respectively, expressed as inverse pole figures normal to the rolling plane of the specimens. The texture is weak in both cases. However, it has strengthened slightly after annealing, where the maximum has shifted towards $\langle 01\bar{1}0\rangle$.

Turning now to the five-parameter distribution, examination of the five-parameter grain boundary space is carried out by selection of various two-dimensional sections through the space. The misorientation statistics are conveniently used as a guide to selection of appropriate sections. A misorientation is chosen and the plane area distribution for that misorientation is displayed in stereographic projection. Plane area distributions for misorientations Σ13b, Σ11b and Σ19b are presented here. These three misorientations had the greatest peaks in the distribution of grain boundary planes.

Figure 3a shows the planes distribution in the reference specimen for Σ13b, $57.4°/[2\bar{1}\bar{1}0]$. There is a single, very pronounced maximum of 120 MRD located at the

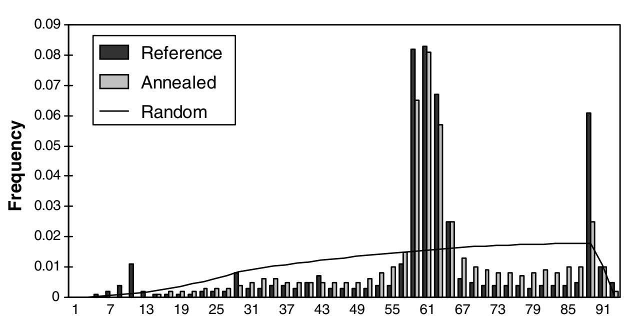

Figure 1. Misorientation angle relative frequency distributions for the reference and the annealed specimens. The misorientation distribution for randomly textured orientation pairs is also included.

Table 1. Misorientation statistics for reference and annealed specimen

Misorientation axis, $\langle uvtw \rangle$ ($\pm 5°$)	Angle range (°)	Σ	Twin system	Length fraction (%) reference specimen	Length fraction (%) annealed specimen
$1\bar{2}10$	57.42	13b	$\{10\bar{1}1\}\langle\bar{1}012\rangle$	20.8	13.3
$10\bar{5}\bar{5}1$	65.1	19b		1.5	<1
$1\bar{2}10$	84.78	11b	$\{10\bar{1}2\}\langle\bar{1}011\rangle$	1.1	1.5
$01\bar{1}0$	86.98	19c	$\{11\bar{2}3\}\langle\bar{1}\bar{1}22\rangle$	4.0	<1

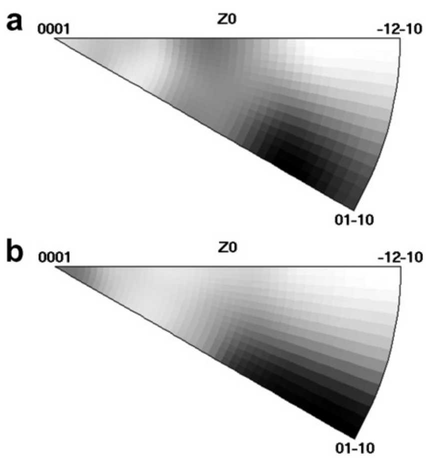

Figure 2. Microtexture of (a) the reference specimen and (b) the annealed specimen, expressed as inverse pole figures normal to the rolling plane of the specimens. The maximum densities in (a) and (b) are 2.07 and 2.78 times random, respectively.

$(10\bar{1}1)$ plane that corresponds to the $\{10\bar{1}1\}\langle\bar{1}012\rangle$ twin system of the Σ13b CSL. This result validates the five-parameter experimental technique for hexagonal metals. Figure 4a shows the planes distribution in the reference specimen for Σ19b, $65.1°/\langle 10\bar{5}\bar{5}1\rangle$. There is a pronounced maximum of 140 MRD near $(4\bar{1}\bar{3}0)$. The planes distribution for Σ11b,$84.8°/[2\bar{1}\bar{1}0]$ is shown in Figure 5a for the reference specimen. Here the maximum MRD is 20 and the boundary plane has a mixed tilt/twist character, spread around the twinning plane, $\{10\bar{1}2\}$. Although the planes distributions are labelled Σ13b, Σ19b and Σ11b, respectively, there is a 10° resolution spread and so the distributions would also encompass neighbouring satellite peaks.

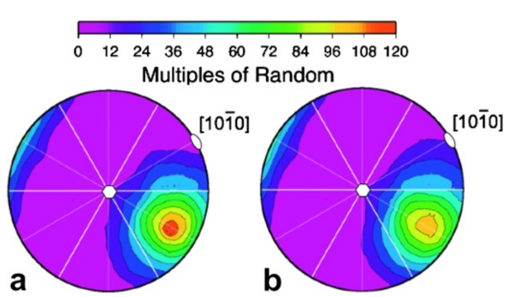

Figure 3. Grain boundary plane distributions for the Σ13b, $57.4°/[2\bar{1}\bar{1}0]$, grain boundaries in Ti6Al4V specimens (a) before and (b) after annealing. In this and the following plane distributions, the $[0001]$ direction is indicated by the hexagon and the $[10\bar{1}0]$ direction is indicated by an oval.

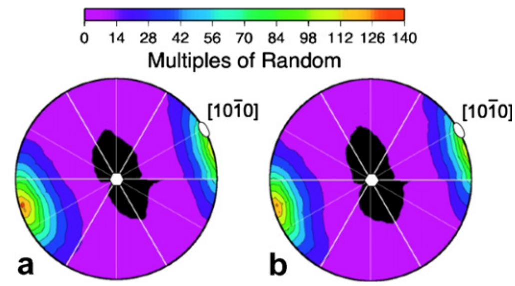

Figure 4. Grain boundary plane distributions for the Σ19b, $65.1°/[10\bar{5}\bar{5}1]$, grain boundaries in Ti6Al4V specimens (a) before and (b) after annealing.

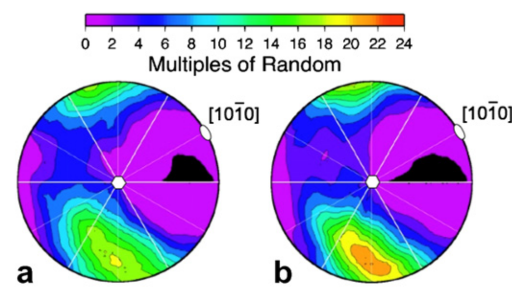

Figure 5. Grain boundary plane distributions for the Σ11b, $84.8°/[2\bar{1}\bar{1}0]$, grain boundaries in Ti6Al4V specimens (a) before and (b) after annealing.

There are subtle changes in the plane distribution as a consequence of the long anneal. The maximum in the distribution for Σ13b decreased from 120 to 100 (Fig. 3b). This is a direct outcome of the decrease in the proportion of Σ13b from 20.8% to 13.3% during the anneal. The distribution of Σ19b remained the same (Fig. 4b). The distribution for Σ11b remained similar, except that the maximum decreased from 23 MRD to 19 MRD (Fig. 5b). There was slight texture strengthening during the long anneal (Fig. 2) which implies grain rotation. This inevitably affects the misorientation distribution, and has also resulted in slight modifications to the intensity of the plane distribution, although the same peaks remain.

To summarize the five-parameter data, there is clearly a very strong "planes texture" that is only partly associated with twinning. The Σ19b is not a twin, and yet there is a particularly strong propensity for a near-prism plane here. Although the misorientation is labelled Σ19b, it encompasses approximately $60–65°/\langle 2\bar{1}\bar{1}0\rangle$ misorientation. The presence of this misorientation is attributed to the β-to-α phase transformation. The planes peak in this misorientation does not diminish during annealing, which supports the assertion that this plane and misorientation combination is energetically favourable.

The work at Carnegie Mellon was supported primarily by the MRSEC program of the National Science Foundation under Award Number DMR-0520425.

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