THE ROLE OF THE BOUNDARY PLANE IN GRAIN BOUNDARY ENGINEERING

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ABSTRACT- This paper reports new data on the distribution of grain boundary planes in commercially available GBE copper. The results show that nearly 60% of grain boundary length was $\Sigma 3$, i.e. annealing twin related. There was a slightly enhanced proportion of {111} boundary planes, even after $\Sigma 3$ boundary segments were excluded from the data set. There was a high proportion of boundary plane length on the 110 zone, which corresponds to asymmetrical tilt boundaries misoriented on the <110> axis. Some of these were geometrically necessary $\Sigma 9$ and $\Sigma 27$ boundaries. The findings support the view that grain boundary *plane* engineering is a viable way forward.

INTRODUCTION: 'Grain boundary engineering' (GBE) refers to the manipulation of microstructure, usually via iterative thermomechanical processing, in order to improve material properties. These properties can be interface transport related phenomena such as intergranular corrosion or cracking, or overall properties such as ductility. A recently published set of papers has overviewed the current status of grain boundary engineering (e.g. Homer et al [2006]). Almost always GBE relies on prolific multiple annealing twinning. In coincidence site lattice (CSL) nomenclature a twin is a Σ 3 boundary. A Σ 9 boundary (so-called 'second order twin') arises from the conjoining of two Σ 3s at a triple junction. Many GBE investigations rely on a CSL approach to categorise and explain the observations. However there is growing opinion that the CSL approach is inadequate because it is a misorientation-based characterisation, and the properties of high angle grain boundaries relate instead to the crystallography of the grain boundary plane (Randle [2006]). Recently, populations of grain boundary planes can be measured via an extension to the electron backscatter diffraction (EBSD) orientation mapping technique in a scanning electron microscope (SEM). This is known as the 'five parameter analysis' and is described in detail elsewhere (Saylor et al [2004]). The new analysis has allowed the emphasis when measuring grain boundary crystallography to be focussed on the distribution of interface planes rather than on the misorientation alone. Other recent methods also exist to extract the boundary plane (Homer et al [2006]). In this paper, we report new data on the distribution of grain boundary planes in GBE copper. The findings support the view that grain boundary *plane* engineering is a viable way forward.

PROCEDURES, RESULTS AND DISCUSSION: Samples of GBE copper were obtained from Integran Technologies Inc., Canada. The GBE processing route used is commercially protected. Twenty-seven high resolution electron back-scatter diffraction (EBSD) orientation maps of metallographically prepared specimens were obtained using HKL Channel 5 software interfaced to a Philips XL30 scanning electron microscope. A step size of 2µm was used for mapping. The sample population was very large; it comprised nearly 122,000 grain boundary trace segments, which were extracted from the orientation maps. The orientation data and boundary trace position data were used to obtain the distribution of all five independent grain boundary geometrical parameters, expressed in terms of the grain misorientation and the boundary plane normal distribution.



Fig. 1. Stereographic projections of distributions of grain boundary plane segments in (a) GBE copper, entire sample population and (b) GBE copper, sample population excluding Σ 3 boundaries. MRD is Multiples of a Random Distribution.

The length fractions of Σ 3-related boundaries recorded were as follows: Σ 3 – 58.5%, Σ 9 – 8.1%, Σ 27 – 3.3. This high proportion of Σ 3s is typical of a GBE metal. The distribution of planes from the five-parameter analysis is typically shown on a stereographic projection. The relative areas of planes are expressed in multiples of a random distribution (MRD). Figure 1a shows the density distribution for all the plane segments in the copper sample population. There is a pronounced peak at {111} at an MRD value of 4.16. On the other hand regions within approximately 20° of {100} contain a density of only 0.56 MRD. Given that over half the grain boundary length is Σ 3, and that many of these Σ 3s are coherent twins on {111}, the peak at {111} on Figure 1a is not surprising. It is therefore instructive to view the grain boundary distribution with Σ 3 misorientations excluded, as shown on Figure 1b. With Σ 3s excluded, the peak at {111} has an MRD value of 1.3, and the distribution about {111} is spread over a larger range. This shows that there is a tendency for grain boundary planes to terminate on {111} orientations, similar to what has been reported for aluminium (Saylor [2004]).

The planes distribution can be viewed according to misorientation axis, using similar figures to those shown on figure 1. There is a higher than random distribution for asymmetrical tilt boundaries on the <110> misorientation axis, particularly in the angle ranges that correspond to $\Sigma 9$ and $\Sigma 27a$. The peak MRD value on the 110 zone for these misorientations is 32 in both cases. Although $\Sigma 9s$ and $\Sigma 27s$ are geometrically necessary boundaries, this work has also shown that they have non-random planes in GBE copper.

Figure 2 show the microstructure of the GBE copper. Random high angle boundaries are black, $\Sigma 3$, $\Sigma 9 \Sigma 27$ and low angle boundaries (3°-15° misorientation) are grey. It is clear that the $\Sigma 3$, $\Sigma 9$ and $\Sigma 27$ boundaries dominate the microstructure and that they have broken up the random boundary network.



Fig. 2. EBSD Orientation map of GBE copper. Random high angle boundaries are black, Σ 3, Σ 9 Σ 27 and low angle boundaries (3°-15° misorientation) are grey.

CONCLUSIONS: The distribution of all five grain boundary parameters is reported in commercially grain boundary engineered copper. There is a higher than random distribution of {111} boundary planes in the microstructure, which is in part attributed to the high proportion of annealing twins present. There is also a high proportion of asymmetrical tilt boundaries on <110>, some of which are geometrically necessary $\Sigma 9$ and $\Sigma 27$ boundaries. The interface network has a very convoluted morphology.

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