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Grain Boundary Characterization of Nanocrystalline Cu from the Stereological Analysis of Transmission Electron Microscope Orientation Maps

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Our recent studies have demonstrated the dominant role of grain boundary scattering in the observed resistivity increase of nanocrystalline Cu films [1]. Our aim now is to quantify the impact of different boundary types on this resistivity increase. To this end, acquisition and analysis of nanoscale crystal orientation maps are essential. In this work, we report results from the stereological analysis of transmission electron microscope (TEM) orientation maps for nanocrystalline Cu thin films. In particular, for a given boundary, we can determine the misorientation between the crystals forming the grain boundary and the trace of the grain boundary plane from a two dimensional orientation map. As a result, four of the five parameters for characterizing a grain boundary are fixed. The unknown parameter is the inclination of the grain boundary plane, for which we can make a statistical estimate using a stereological analysis [2]. In this fashion, TEM orientation maps were obtained from the analysis of diffraction patterns using the ASTAR system developed by NanoMegas [3], combined with the D-STEM lens configuration to achieve a quasi-parallel nano size probe [4]. Precession microscopy was also employed to reduce the dynamical diffraction effects [5].

The sample studied is a 46.4 nm thick sputter deposited Cu thin film forming a SiO_2 (sputtered)/Cu/SiO_2(sputtered)/SiO_2(thermal)/Si structure. The sample was annealed at 600°C for 0.5 hr in Ar + 4% H_2 [1]. Electron microscopy was performed on a 200 KV JEOL 2010F TEM/STEM instrument. The probe size for the acquisition is 5 nm approximately. The grain boundary line segments from the orientation map are extracted by joining the triple junctions using the TSL OIM software. Since grain boundaries are curved, multiple line segments are used to fit the grain boundary when the distance between the grain boundary and the line segment joining the triple junction is more than 4 pixels [5]. Comparison of Figs. 1a and b shows a good match between the image quality and the orientation map. The misorientation distribution (Fig. 1c) shows a peak about the $\Sigma3$ misorientation. The distribution of boundary normals for the $\Sigma3$ misorientation from stereological analysis (Fig. 1d) shows the presence of a high density of coherent twin boundaries. We observe that coherent twin boundaries make up 20% of the total length of all boundaries.

References
[1] T. Sun et al., *Phys. Rev. B* 81 (2010), 155454.
[2] D. M. Saylor et al., *Metallurgical and Materials Transactions A* 35 (2004) 1981-89.
[3] E. F. Rauch et al., *Microsc. Microanal.* 22(6) (2008) S5-S8.
[4] K. J. Ganesh et al., *Microsc. Microanal.* 16(5) (2010) 614.
[5] P. Oleynikov, S. Hovmöllera and X.D. Zou *Ultramicroscopy* 107 (2007) 523,

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[6] S.I. Wright, R.J. Larsen, *J. Micro.* 205(3) (2002) 245.
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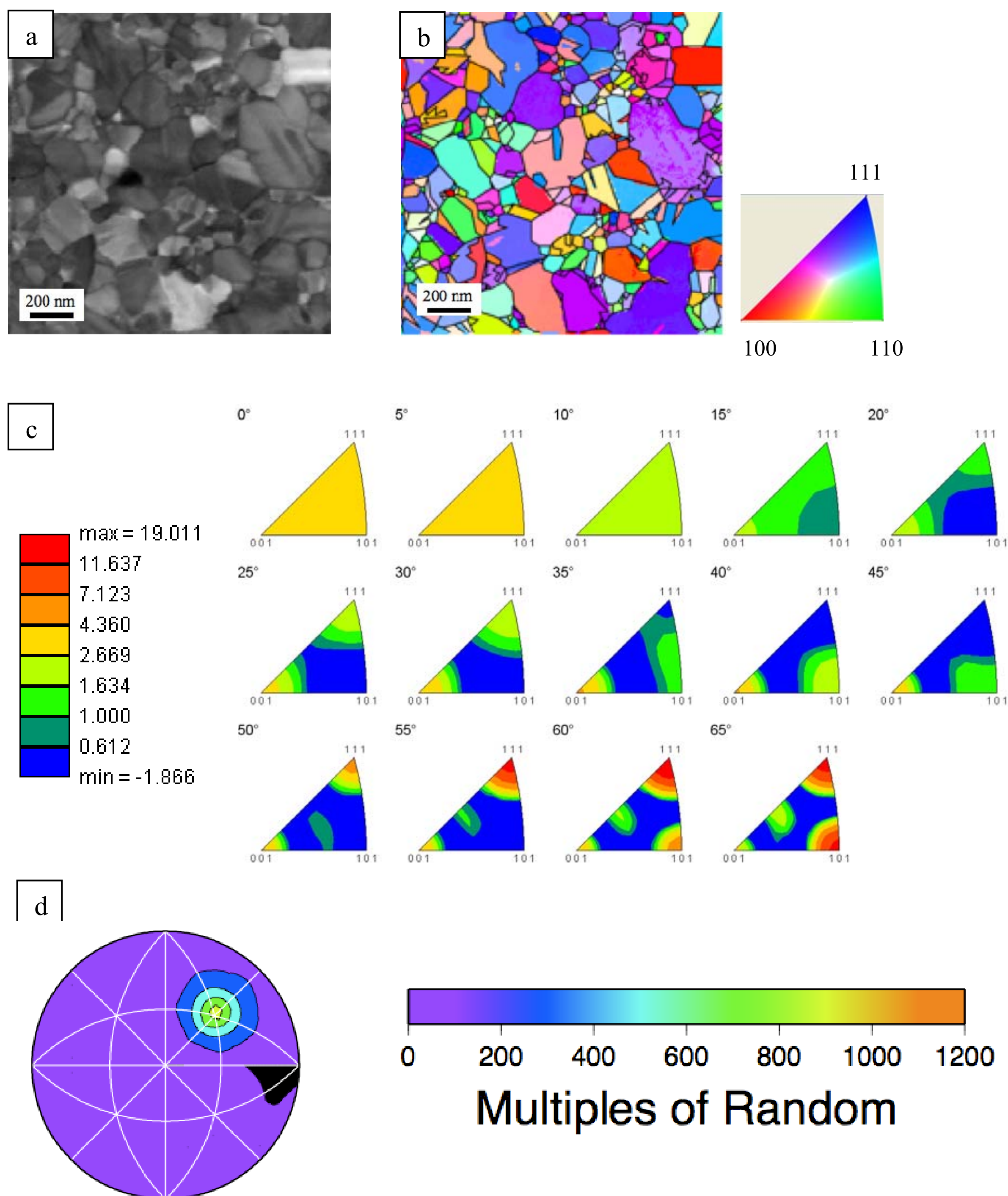

Fig. 1: (a) Image quality map (b) Color coded inverse pole figure map (c) Misorientation distribution function expressed as multiples of random (d) Distribution of grain boundary plane normals for the Σ3 misorientation plotted in the crystal frame of reference. Peak about the <111> boundary normal shows a high coherent twin density.