AIP Conference Proceedings

Recrystallization, Grain Growth in Copper Foil at High Temperature Studied By Electron Back Scatter Diffraction

Pragya Tiwari, Himanshu Srivastava, Sanjay Rai, and S. K. Deb

Citation: AIP Conf. Proc. **1349**, 705 (2011); doi: 10.1063/1.3606053
View online: http://dx.doi.org/10.1063/1.3606053
View Table of Contents: http://proceedings.aip.org/dbt/dbt.jsp?KEY=APCPCS&Volume=1349&Issue=1
Published by the AIP Publishing LLC.

Recrystallization, Grain Growth in Copper Foil at High Temperature Studied By Electron Back Scatter Diffraction

Pragya Tiwari*, Himanshu Srivastava, Sanjay Rai and S.K.Deb

ISUD, Raja Ramanna Centre for Advanced Technology, Indore – 452013
**corresponding author –e mail: pragya@rrcat.gov.in*

Abstract. As-received cold rolled and isothermally re-crystallized textures of high purity copper foil were studied using electron back-scattered diffraction patterns (EBSD) in a scanning electron microscope. The evolution of the re-crystallization texture was investigated as a function of annealing temperature. In as is sample, the Cu grains are small and equi-axed, but re-crystallization results in grains several microns in size. We observe a significant enhancement of the (311) texture by pole figure measurements till annealing temperatures of 400^{o}C and then enhancement of (200) texture at annealing temperatures of 600^{o}C. We propose that multiple twinning is the mechanism for this phenomenon.

Keywords: re-crystallization, grain growth, texture, EBSD.
PACS: 61.05.-a, 61.05.cp,61.05.J-, 61.72.Mm, 68.37.Hk

INTRODUCTION

The texture, grain size distribution, grain morphology and spatial distribution of the grain orientations are important micro-structural characteristics which influence the mechanical and electrical properties [1–2] of copper foil. Therefore, tailoring the properties of copper foil and improving their reliability and functionality require control of the grain size and texture that develop during post cold work annealing processes. Important for the nucleation and growth of new grains are regions of the deformed microstructure with high orientation and dislocation, density gradients, e.g. deformation bands, twins and grain boundaries [1]. Electron diffraction methods are most suitable for information about local orientation differences within these structures because they allow simultaneously for the recording of the microstructure and the determination of individual crystal orientations. For copper, when the strain amount is low, all grains, i.e. all orientations, have the same probability to grow, and the re-crystallization texture becomes isotropic because of the twinning mechanism. For a high amount of strain produced by cold rolling, the {100} ⟨001⟩ cube orientation dynamically recovers during deformation. Then, this component quickly develops at the expense of the deformed matrix. Consequently the re-crystallization texture is essentially composed of the cube component plus its twin orientation. In all cases, when the nucleus has reached a “critical” size, it grows at the expense of the deformed matrix by bulging. This is possibly due to the large stored energy difference between the nucleus and the matrix. A nucleus can twin during the first stages of its growth and several twin generations may be observed before complete re-crystallization [2,3].

In the present study, EBSD measurements have been performed on copper foil after vacuum annealing. The study focuses on the grain growth and texture enhancement in the re-crystallized microstructure.

EXPERIMENT

Commercially available copper foil (Merck 99.5% purity) was used for these studies. A small piece (1cm X 1cm) was cut and rinsed in dilute sulphuric acid to remove any oxide layer present on the surface. After washing it in de-ionised water, the sample was loaded in vacuum furnace and annealed at 100^{o}C, 200^{o}C, 300^{o}C, 400^{o}C, 500^{o}C, 600^{o}C and 700^{o}C for 2 hours and furnace cooled till room temperature. Then the sample was examined for microstructure on XL 30 CP Philips SEM, and EBSD patterns were taken using Bruker eFlash detector. The area scanned was 100µmX100µm.

RESULTS AND DISCUSSION

By observing the microstructure and EBSD patterns of the copper foil before and after annealing at

Solid State Physics, Proceedings of the 55th DAE Solid State Physics Symposium 2010
AIP Conf. Proc. 1349, 705-706 (2011); doi: 10.1063/1.3606053

different temperatures (fig.1), it is seen that at the annealing temperatures below 300^{o}C, the microstructure of the foil resembles fibrous crystals with a little re-crystallization. Figure2 shows the pole figure for the as is and 600^{o}C annealed sample which is used for texture analysis and fig. 3 gives the grain growth as a function of annealing temperature. After annealing at 400^{o}C, the foil is fully re-crystallized, compound twins are found and there is an evolution of (311) orientation. After annealing at 500^{o}C, the re-crystallized grains coarsened and (200) texture starts emerging. Annealing at 600^{o}C and 700^{o}C leads to further grain growth with a decrease in the defect structure and (200) as the dominant texture.

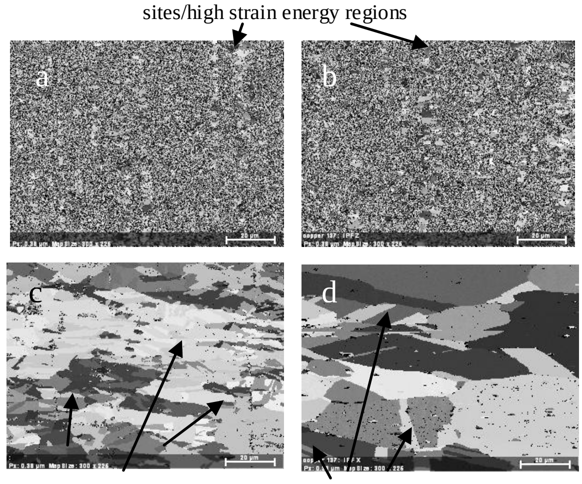

FIGURE 1. EBSD patterns for the copper foil sample (a) as is, vacuum annealed for 2 hours at (b) 200^{o}C (c) 400^{o}C (d) 600^{o}C

The re-crystallized foils at 300^{o}C contain grains with ragged edges and numerous twins within one grain that have both coherent and incoherent twin boundaries. This suggests that the driving force for re-crystallization derived from reduction in grain boundary energy and reduction of micro-strains and defects is limited. To further reduce grain boundary surface area, the input of additional energy is required, as in the 400 °C anneal, after which we observe smooth grain boundaries and a reduction in both the number of twins and the incoherent twin boundaries. Most of the twins now span the width of a grain. We have found that the as-is foil consists of small equi-axed grains that are not oriented, the re-crystallized Cu at 400^{o}C exhibits grains up to 6-7 µm (average) in size and (311) texture, and the re- crystallized Cu at 600^{o}C exhibits grain size upto 23-25 µm (average) in size &(200) texture .

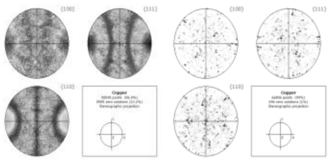

FIGURE 2. Pole Figure a) as is (b) Annealed at 600^{o}C

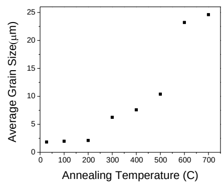

FIGURE 3. Average grain size vs annealing temp.

Recent studies on the re-crystallization mechanism of rolled sheets of Cu have addressed how the new orientations are formed within the matrix of the small-grained Cu and stated that Cu, having a low stacking fault energy, can easily undergo multiple twinning in early stages of the growth process [4-6]. It is also well established that almost any orientation can be generated by multiple twinning [6] from a parent orientation. We have also found that the twin fraction is mainly distributed in the large grains. Indeed, it appears that large grains favor twin formation. This conclusion could be used to develop isotropic textures often desired from an industrial point of view to improve physical properties.

CONCLUSIONS

In this paper, we have observed an enhancement of [311] texture during annealing till 400^{o}C, characterized using XRD and EBSD. The pathway for textural transformation is attributed to the first order twinning in the early stages of the growth process, with [110] orientation undergoing twinning to [411], followed by slow evolution to [311] to reduce the surface energy. The average grain size in the as is sample is ~1 µm and the sample annealed at 600^{o}C is ~25 µm. The driving force for the following grain growth is the minimization of total grain boundary energy in the system.

ACKNOWLEDGMENTS

The authors are thankful to Shri Rakesh Kaul and Shri P.Ganesh for valuable discussions.

REFERENCES

[1]R.D.Doherty et.al. Mat. Sci.and Eng.A238 (1997)219.
[2] J.R.Hirsch and K.Lticke, Acta Metall.,36 (1988) 2863.
[3] Pantleon,M.A.J.Somers,J.Appl.Phys.100(2006)114319
[4] A. Berger et.al. Prog.Mater. Sci. **32**, (1988) 1–95.
[5] F. J. Humphreys and M. Hatherly, Recrystallization & Related Annealing Phenomena ,Pergamon, New York, 1996.
[6] C. Lingk, et.al., J. Appl. Phys. 87 (5) (2000) 2232.