X-ray line profile analysis of equal channel angular pressing processed Cu

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Abstract. The effect of equal channel angular pressing on the microstructure of copper samples was studied by X-ray line profile analysis. Pure Cu samples were processed by equal channel angular pressing with 3 passes in route A. Samples were taken from the vicinity of the channel intersection, and along a profile across the deformation zone, microhardness and XRD measurements were performed. For the high resolution line profile analysis of the diffraction spectra, convolutional-multiple-whole-profile CMWP method was applied, dislocation density and grain size were calculated, furthermore the density of twin boundaries were determined. Results show a rearrangement in the dislocations in the third pass leading to a rise in the density of twin boundaries.

1. Introduction

Equal channel angular pressing (ECAP) is one of the most common severe plastic deformation (SPD) methods [1] to create bulk ultrafine grained materials. During SPD, dislocation density is highly increased through large amount of cold deformation applied to the material. Dense dislocation walls develop, and will later be transformed to high angle grain boundaries [2]. In ECAP, the material is pressed through a die consisting of two intersecting channels [3]. As channel cross sections are equal, specimen cross sections remain unchanged, therefore it can be pressed through again in multiple passes. The real deformation zone is located in the close vicinity of the plane of channel intersection (main shear plane), where large amount of shear deformation occurs, that causes the strengthening of the material. While strength is easy to determine even in the vicinity of the main shear plane by microhardness measurements [4], the background microstructural mechanisms which are based on dislocation dynamics are more difficult to explore. By X-ray diffraction (XRD) with line profile analysis, it is possible to extract qualitative and quantitative information about dislocations in the ECAP processed materials [5]. In a recent study, effectiveness of different ECAP processing routes was analyzed by XRD line profile analysis [6].

In our work, one and three times ECAP processed (by route A) pure Cu samples were analyzed, and emphasis was put on the mechanical and microstructural transformations across the deformation zone. Samples were taken from the vicinity of the channel intersection, and along a profile across the deformation zone, microhardness and XRD measurements were performed. For the high resolution line profile analysis of the diffraction spectra, convolutional-multiple-whole-profile (CMWP) method
was applied, dislocation density and grain size were calculated, furthermore the density of twin boundaries were determined.

2. Experimental procedures
The starting material was commercially available pure copper in the form of forged rods. Specimens were machined with a diameter of 10 mm and a typical length of 60 mm. Before ECAP, specimens were annealed at 350°C for 1h. ECAP was carried out in a screw driven testing machine at a traverse speed of 1 mm/s at room temperature. A lithium based grease was used for lubrication. The channel angle was 110°, with 0° corner angle (figure 1), in a specially designed openable die. In multiple passes, route A was applied. For the further investigations, the specimens were taken out from the die, cut along the length by a cooled diamond saw, and the surface was polished. Seven measurement locations were defined in the vicinity of the main shear plane as shown in figure 1. To define measurement locations, finite element simulation were carried out to estimate the homogeneity of the strain distribution in the pressed specimens, and to estimate the size of the deformation zone.

Further specimens were pressed up to three ECAP passes. Samples were prepared for X-ray diffraction analysis. A 0.2x1.5 mm² X-Rays spot size was used for X-ray analysis. Line profiles for the X-ray diffraction analysis were evaluated by using the extended convolutional multiple whole profile (eCMWP) procedure [7], which yield dislocation density, grain size and the density of twin boundaries. The effect of twinning on the dislocation pattern was calculated as described in [8]. Finally, microhardness was measured at each measurement locations by a Zwick 3212 tester with Vickers head.

3. Results and discussion

3.1. Strain distribution and hardness in the 1st ECAP pass
A finite element simulation of the first ECAP pass was carried out in order to analyze the deformation of the specimen. Several studies were conducted in this field [9-13]. In the present case, we focussed on the strain homogeneity and size of the deformation zone. Plane strain assumption was applied for 2-D modeling. Mesh was built by quadratic elements with an initial grid size of 0.25x0.25 mm². The material for the specimen was linear elastic and plastic hardening without rate sensitivity. Non-linearity due to large deformations was considered. The die was modeled as rigid and the contact was frictionless.

The simulation results are shown for the distribution of the total equivalent plastic strain and equivalent plastic strain rate in figure 2. The magnitude of the equivalent strain in the main homogeneous part of the specimen is equal to value of 0.8, i.e. within 1% of the calculated value by the simple shear approach [2]. Although deviations in the strain can be found in the vicinity of the contact region, the strain distribution in the bulk of the processed specimen is homogeneous. The homogeneity of the strain is affected by other factors besides the channel and corner angle, such as the strain hardening rate and friction [9]. Increasing channel angle is beneficial for strain homogeneity [9].
An homogeneous strain is expected to result in a constant microstructure and hardening throughout the bulk of the specimen.

The deformation zone is located where the strain rate is non-zero, as shown in figure 2 (b). The zone is about 2 mm wide. Its axis slightly deviates from the projection line of the main shear plane, due to the fact that the pressed material cannot fill the corner of the die, as also observed on the specimens. The size of corner gap is smaller for high hardening materials, and with increasing friction [10].

The microhardness measurement results are shown for the first ECAP pass in figure 3. The microhardness increases from about 60 HV to max. 120 HV across the main shear plane, on a 2 mm distance as shown in 3 (a). In the processed part, the hardness is homogeneous as shown in figure 3 (b), as predicted by the simulations. The location of the measurement points for the XRD are defined and shown in figure 1.

Figure 2. Finite element simulation of the first ECAP pass, results for (a) equivalent plastic strain and (b) equivalent plastic strain rate.

Figure 3. Microhardness results after the first ECAP pass of a Cu sample (a) along the channel direction in the vicinity of the main shear plane, and (b) perpendicular to channel direction in the processed side.
3.2. Hardness and XRD line profile analysis results for the 3rd ECAP pass

The microhardness results along the measurement locations are shown in figure 4 for the multiple ECAP processed Cu sample. Each point is an average of 5 measurements within an area corresponding to the spot size of XRD. Hardness only slightly increases with increasing number of passes, from the second to the third pass, about 5 HV increase is measured. The lowest strength is measured at position 4 (corresponding to the deformation zone).

XRD measurements were carried out at each measurement locations. From the measurement data, dislocation density, grain size (size of coherently diffracting domains), and the density of the twin boundaries were extracted.

The local variations in the dislocation density in the range of $10^{15}$ m$^{-2}$ in the vicinity of the main shear plane for multiple ECAP passes are plotted in figure 5. These results show a similar trend as found in the microhardness data. The increase from the second to the third pass was about 20%, but a large drop is measured at location 7.

![Figure 4. Microhardness results along the measurement locations of multiple ECAP processed Cu sample.](image)

![Figure 5. Dislocation density in the vicinity of the main shear plane along the measurement locations of multiple ECAP processed Cu sample.](image)

![Figure 6. Grain size variations along the measurement locations of multiple ECAP processed Cu sample.](image)
The measured grain size is below 200 nm, the trend along the measurement locations are plotted in figure 6. An actual increase is measured from the second to the third pass. As the measured grain size (or size of coherently diffracting domains) is also affected by changes at the grain boundaries, it is expected that such effect is captured in this plot.

The density of the twin boundaries is plotted at the same points in figure 7. No twins are detected before shearing, and a 0.1% afterwards.

![Density of twin boundaries along the measurement locations of multiple ECAP processed Cu sample.](image)

Figure 7. Density of twin boundaries along the measurement locations of multiple ECAP processed Cu sample.

![Modified Williamson-Hall plot for measurement location 5 and 7 of multiple ECAP processed Cu sample: (a) uncorrected measurement data, (b) corrections considering the effect of twin boundary frequency.](image)

Figure 8. Modified Williamson-Hall plot for measurement location 5 and 7 of multiple ECAP processed Cu sample: (a) uncorrected measurement data, (b) corrections considering the effect of twin boundary frequency.

Modified Williamson-Hall plots are shown in figure 8 for measurement locations 5 and 7. In these plots the full width at half maximum of the peak broadening ($\Delta K$) is plotted as a function of reciprocal spatial coordinate ($K$) multiplied by the dislocation contrast factor ($C$). The slope of the trend line varies as the dislocation density, and the intersection with the vertical axis varies as the inverse of the grain size. The data points in figure 8 (a), data points deviate from the trend line, as lattice distortions are not only caused by dislocations. Taking into account a possible contribution of twinning to the
peak broadening improves the quality of the fit and lowers the intersection of the line with the vertical axis (figure 8 (b)).

4. Conclusions
Based on the finite element simulations, microhardness measurements, and XRD line profile analysis of single and multiple ECAP processed pure copper, the following conclusions are drawn:

1. The width of the deformation zone is 2 mm, furthermore deformation is homogeneous in the ECAP processed specimen, as predicted from the simulations of the first pass, and verified by hardness measurements.
2. The dislocation densities determined by XRD are in good correlation with the hardness measurements.
3. In the third ECAP pass, the density of twin boundaries increases to 0.1%. This results in a decrease of the dislocation density.

References
[1] Valiev R Z, Estrin Y, Horita Z, Langdon T G, Zechetbauer M J, Zhu Y T 2006 JOM 58(4) 33
[2] Segal V 1995 Mater. Sci. Eng. A 197 157-164
[3] Valiev R Z, Islamgaliev R K, Alexandrov I V 2000 Prog. Mat. Sci. 45 103-189
[4] Gonda V, Varga P, Réger M and Verő B 2012 Hardness evolution in Cu processed by ECAP Proc. Conference on Metal Forming, Miskolc, Hungary.
[5] Ungár T, Gubicza J, Ribárik G and Borbély A 2001 J. Appl. Cryst. 34 298
[6] Sarkar A, Suwas S, Goran D, Fundenberger J-J, Toth L and Grosdidier T 2012 Powder Diffr. 27(3) 194
[7] Ribárik G, Gubicza J and Ungár T 2004 Mat. Sci. Eng. A 387-389 343
[8] Balogh L, Tichy G and Ungár T 2009 J. Appl. Cryst. 42 580
[9] Dumoulin S, Roven H J, Werenskiold J C and Valberg H S 2005 Mat. Sci. Eng. A 410–411 248–251
[10] Kim H S, Seo M H and Hong S I 2000 Mat. Sci. Eng. A 291 86–90
[11] Arruffat-Massion R, Toth L S and Mathieu J-P 2006 Scripta Mater. 54 1667–1672
[12] Kim H S and Estrin Y 2005 Mat. Sci. Eng. A 410–411 285–289
[13] Nagasekhar A V, Yoon S C, Tick-Hon Y and Kim H S 2009 Comp. Mat. Sci. 46 347–351