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# Effect of Rolling with Cyclic Movement of Rolls Method on Structure and Hardness of CuCr0.6 Alloy

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A novel severe plastic deformation method entitled rolling with cyclic movement of rolls was proposed to fabricate ultrafine grained CuCr0.6 copper alloy. The alloy in the solution treated conditions was processed by rolling with cyclic movement of rolls method by using process parameters as amplitude of rolls movement (A = 0.9 mm), frequency of rolls movement (f = 1 Hz), rolling reduction ( $\varepsilon_h = 50\%$ ) and rolling rate (v = 1, v = 2, v = 3 rpm). Light microscopy, scanning electron microscopy and a electron backscattering diffraction detector, and scanning transmission electron microscopy were used for microstructural characterization, and hardness tests for a preliminary assessment of mechanical properties. Quantitative studies of the average diameter of the subgrains d (µm) and the average diameter of the grains D (µm) were performed using the scanning electron microscopy/electron backscattering diffraction method. Misorientation angles were analyzed by the Kikuchi-line technique using TSL-OIM software. The results show that the samples underwent very high strain at the lateral areas and smaller at the central areas. As a result, the microstructure became heterogeneous and remained unchanged with change in compression rate. The transverse movement of rolls causes in the material significant effect of refining structure in peripheral areas of sample.

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### 1. Introduction

Several noble techniques have been developed for to manufacture ultrafine grained sheets and plates among which are: repetitive corrugation and straightening (RCS) [1] and accumulative roll-bonding (ARB) [2]. Rolling with cyclic movement of rolls (RCMR) is a severe plastic deformation process that allows large deformations. This original method of deformation has been patented in Silesian University of Technology [3]. The rolled strip is deformed by reducing the height and additionally is affected by movement of the material layers in a direction perpendicular to the main direction of rolling. By repeating this procedure in several passes, very high strains have been introduced into material and significant structural refinements have been achieved [4]. Rolling mill consists of two working rolls, the power unit and mechanism for cyclic movement of the rolls — transversely to the rolling direction. During the RCMR processing, the rolls rotate around an axis and, in addition, there are realized axial movements of the rolls in opposite directions. Main parameters affecting plastic flow (total effective strain value) are: rolling reduction  $\varepsilon_h$  [%], rolling rate v [rpm], amplitude of transverse movement of rolls A [mm] and frequency of transverse movement of rolls f [Hz]). The research was focused on precipitationhardenable CuCr0.6 alloy. For refinement structure of CuCr0.6 alloy, equal-channel angular pressing methods were applied [5–7]. The authors [5–7] reported more grain refinement and significant improvement in the mechanical properties.

Currently, this method for the deformation of Cu–Fe alloy is not well-known in the literature, and therefore this problem is addressed in the present article. For this reason the present paper describes some aspects of influence deformation parameters (rolling rate) on formation of ultrafine grained (UFG) structure and hardness. Especially one important element as heterogeneous in microstructure is shown.

## 2. Experimental

In this study, precipitation hardened copper alloy with addition of 0.6 wt% Cr was used. The alloy was prepared by melting and alloying in open-air induction furnace, followed by casting into diameter of 50 mm mould. Ingots were hot rolled to bar sample  $8 \times 8 \times 60 \text{ mm}^3$ . Samples were heat treated by solution at 1000 °C per 1 h with cooling in water. Figure 1 shows the microstructure of CuCr0.6 alloy obtained by light microscopy (LM) and scanning transmission electron microscopy (STEM) after solution treatment. Microstructure of CuCr0.6 alloy is characterized by the presence of undissolved equiaxial chromium precipitates in Cu matrix (Fig. 1) [1, 8].

RCMR was conducted at room temperature, with the parameters:  $\varepsilon_h = 50\%$ , A = 0.9 mm, f = 1 Hz. In our experiment variable values of rolling rate (v): v = 1, v = 2, v = 3 rpm were applied. Total effective strain  $(\varepsilon_{ft})$  accumulated in material after RCMR could be estimated by equations [4]:

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Fig. 1. The LM (a) and STEM (b) microstructure of Cu0.6Cr alloy after heat treatment by solution of 1000 °C/1 h.

$$\varepsilon_{ft} = \sum_{i=1}^{n} \sqrt{\varepsilon_{hi}^2 + \varepsilon_{ti}^2},\tag{1}$$

$$\varepsilon_{hi} = \ln \frac{h_i}{h_{i-1}},\tag{2}$$

$$\varepsilon_{ti} = \frac{2A}{\sqrt{3}(h_{i-1} + h_i)},\tag{3}$$

where  $\varepsilon_{ft}$  — total effective strain,  $\varepsilon_{hi}$  — strain included by rolling reduction,  $\varepsilon_{ti}$  — strain included by transverse movement of working rolls, n — number of passes,  $h_{i-1}$ ,  $h_i$  — height of sample before and after single pass (reduction), A — amplitude of transverse movement of rolls.

For rolling rates: v = 1, v = 2, v = 3 rpm, the total effective strains were:  $\varepsilon_{ft} = 1$ ,  $\varepsilon_{ft} = 1.2$ ,  $\varepsilon_{ft} = 1.7$ , respectively.

The microstructure was characterized in this study through electron backscattering diffraction (EBSD) analysis. EBSD analysis was conducted using FEI INSPECT F scanning electron microscope (SEM) equipped with a cold field emission gun at an accelerating voltage of 20 kV. Data acquisition and subsequent analysis were performed using a TSL orientation image microscopy system. Grain size and misorientation angles were determined with the EBSD analysis.

Orientation maps were acquired with a step size of 50 nm at 20 kV. All data points characterized by a confidence index (CI) lower than 0.05 were excluded from the analysis as dubious. During post- processing using  $TSL^{\textcircled{R}}$  software the following values have been applied: grain tolerance angle — 0.5°, minimum grain size — 2 pixels.

The specimens were polished by using an ion thinning device (PECS) manufactured by Gatan, Inc. Additionally, STEM Hitachi HD-2300A equipped with a cold field emission gun at an accelerating voltage of 200 kV was used for dislocation microstructure characterization. Figure 2a shows the scheme of RCMR method. Definition of directions are presented in Fig. 2b. LM, SEM/EBSD, and STEM observations were performed on samples taken from transverse plane section (Fig. 2c). Detailed microstructure and hardness measurements were made in the areas marked by A and B points (Fig. 2c). The Vickers hardness was measured on an electrolytically polished surface of the samples by means of a Zwick microhardness tester. A 1000 g load applied for 15 s was ensured for these measurements. The hardness values were taken as the average of a minimum of 10 measurements.



Fig. 2. Illustration of the RCMR deformation (a), definition of directions (b), and points of SEM/STEM observations (c).

### 3. Results and discussion

Figure 2c shows optical micrographs of sample after RCMR deformation on transverse section. The structure is very inhomogeneous with less deformed central areas (point B) and heavy deformed surface areas (point A). EBSD/STEM analysis was then conducted at the central areas (point B) (Fig. 3) and at the surface areas (point A) (Fig. 4). Summary of the EBSD analysis is given in Fig. 5 and Table I.



Fig. 3. Orientation/STEM images after 3 rpm (a,b), 2 rpm (c,d), 1 rpm (e,f) of RCMR at point B (central area).

EBSD analysis and TEM examination showed that low angle boundaries (LABs) are dominant in the microstructure (point B). Visible fragments of high angle boundaries (HABs) on EBSD maps (Fig. 3a,c,e) belong to the original grain boundaries and the shape of the primary grains is related to the direction of the compression deformation. In the microstructure, subgrains are elongated and look like a banded structure. The deformation introduces in material a high dislocation density (Fig. 3b,d,f). Independently of compression rate (Table I) the fraction of



Fig. 4. Orientation/STEM images after 3 rpm (a,b), 2 rpm (c,d), 1 rpm (e,f) of RCMR at point A (surface area).



Fig. 5. Area fraction of grains distribution after RCMR deformation for different values of rolling rate for A position.

low angle grain boundaries is large. This mean that influence of transverse rolling in central area of samples is insufficient. EBSD maps (Fig. 4a,c,e) clearly show what the microstructure becomes finer at the surface areas (point B).

Observations reveal that the new grains/subgrains are rather elongated in rolling direction. EBSD results well correspond to the STEM observation. After RCMR deformation with rolling rate at 3 rpm the microstructure is

Hardness data and EBSD data (HV1, HABs >  $15^{\circ}$  [%],  $D \ [\mu m]$ ,  $A_{1\mu m}$  [%]) for CuCr0.8 alloy deformed by RCMR method at points A and B. HV1 = 55 for initial state.

TABLE I

$v  [\mathrm{rpm}] / \varepsilon_{ft}$	pos.	HV1	HABs	D	$A_{1\mu m}$
3 rpm	А	$106 \pm 3$	52	0.38	68
1.0	В	$122 \pm 4$	25	0.73	8
2 rpm	А	$109 \pm 4$	65	0.26	97
1.2	В	$133 \pm 7$	33	0.81	7
1 rpm	А	$105\pm5$	58	0.27	63
1.7	В	$131 \pm 4$	30	0.76	7

characterized by the formation of a network of intersecting bands (Fig. 4b). The decrease of rolling rate (2 rpm) causes a progress in grain refinement. The newly formed grains are enclosed by HABs. Some of grains contain relatively low dislocation density in their interiors (Fig. 4c,d). Thus in the best refined areas (Fig. 4c,d), the fraction of high angle boundaries became more than 65% and the area fraction of ultrafine grains (up to 1  $\mu$ m in diameter) was 97%. As was suggested by EBSD analysis the average grain size is 0.26  $\mu m$  (Table I). The next decrease of rolling rate (1 rpm) does not cause the further refiement of grains because structure recovery occurred (Fig. 4f, Table I). The cyclic character of this process determined the course of structure changes. From the above observations, it can be concluded that the Vickers hardness well corresponds to the structure evolution. The development of submicron-sized structures during severe plastic deformation (SPD) has generally been shown to be accompanied by an increase in hardness. Average hardness of alloy in initial state was 55 HV1. In deformed samples with 2 rpm, the hardness varies from maximal values of about  $\approx 133$  HV1 in the vicinity of surface microareas to values about  $\approx 109$  HV1 in the vicinity of the central microareas.

Based on the performed investigations should be noted that in heavy processed CuCr0.6 alloy, the distribution of strain is heterogeneous which is typical for most SPD processes [9, 10]. It is evident that applied value of total effective strain  $\varepsilon_{ft} = 1.7$  in RCMR process is insufficient in generating UFG structure in the whole volume of sample. From literature it is known that the heterogeneity is reduced during next cycles of deformation [9, 10]. Not all the process variables affecting structure development have been taken into account. Therefore, further improvements in effectiveness of RCMR method by change in deformation parameters is important. The results obtained here with the RCMR method are comparable with other method of SPD deformation. For example, in CuCr0.6 alloy after continuous repetitive corrugation and straightening (CRCS) for strain of 28, the hardness was 110 HV [11]. As a consequence, the microstructure consisted of dislocation cells with individual nanograins and subgrains.

### 4. Summary

- 1. The samples underwent very high strain at the lateral areas and smaller at the central areas. As a result, the microstructure became heterogeneous and remained unchanged with change in compression rate. EBSD/STEM investigations were limited to representative areas.
- 2. In the central areas on the cross-section plane, the grains have elongated shape. The banded structure is observed in STEM technique, which is similar to that obtained in the conventional rolling.

3. The transverse movement of rolls causes in the material significant effect of refining structure in peripheral areas of sample. The best results of refinement were obtained for samples deformed at compression rate of 2 rpm because these values lead to both more rapid elimination of the elongated subgrain and increased evolution of low- to high-angle grain boundaries.

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