

## Ultrafine grained strips of CuCr0.6 alloy prepared by CRCS method

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Received 27.02.2009; published in revised form 01.04.2009

### Methodology of research

#### ABSTRACT

**Purpose:** The aim of this work was to evaluate the ability of a continuous repetitive corrugation and straightening (CRCS) technique in creating ultra fine grained copper-chromium strips as well as to determine their deformability, mechanical properties, deformation behaviour and microstructure evolution.

**Design/methodology/approach:** Tests were performed with the 0.8 mm thick CuCr0.6 strips using original die set construction. The changes of mechanical properties as well as microstructure evolution versus number of deformation cycles were investigated. The microstructure was investigated using optical and electron microscopy (TEM and SEM equipped with EBSD).

**Findings:** The obtained strengthening effects and observed microstructure changes have been discussed basing on the existing theories related to strengthening of ultra fine grained copper based materials. The CRCS process effectively reduced the grain size of a CuCr0.6 alloy strips, demonstrating the CRCS as a promising new method for producing ultra fine grained metallic strips.

**Research limitations/implications:** Research results are limited to the initial material after annealing only. Further investigations should be aimed towards determination of CRCS sequence including deformation-precipitation-ageing influence on strengthening effect.

**Practical implications:** A growing trend to use new copper-based functional materials is observed recently world-wide. Within this group of materials particular attention is drawn to those with ultra fine or nanometric grain size of a copper matrix, which exhibit higher mechanical properties than microcrystalline copper.

**Originality/value:** The paper describes the mechanical properties of precipitates strengthened ultra fine grained copper - chromium alloy strips obtained by original RCS method and to the microstructure evolution.

**Keywords:** Severe plastic deformation; Ultra fine grained material; Mechanical properties; Electron microscopy

#### Reference to this paper should be given in the following way:

J. Stobrawa, Z. Rdzawski, W. Głuchowski, W. Malec, Ultrafine grained strips of CuCr0.6 alloy prepared by CRCS method, Journal of Achievements in Materials and Manufacturing Engineering 33/2 (2009) 166-172.

### 1. Introduction

Precipitation strengthened copper is a group of functional and structural materials used where combination of high electrical

and/or thermal conductivity with high strength at room and elevated temperatures is required.

A growing trend to use such functional materials is recently observed world-wide. Within this group of materials particular attention is drawn to those with ultrafine grain size (UFG) of a

copper matrix, which exhibit higher mechanical properties than microcrystalline copper alloys.

Many methods have been used to produce bulk copper based semi products of ultrafine grain sizes. The most common methods are powder metallurgy technique (with high-energy ball milling, compacting, low temperature sintering and controlled extrusion) [1-6] and severe plastic deformation methods, mainly equal – channel angular pressing (ECAP) [7-11], high-pressure torsion (HPT) [12-14] and hydrostatic extrusion (HE) [15]. These methods are not conducted continuously, requiring labor intensive handling of the work– piece between process steps and they seem to be impractical for manufacturing plate – shaped ultrafine grained materials. Recently a new techniques, groove pressing [16, 17] and repetitive corrugation and straightening (RCS) [18-20] were developed for production of UFG microstructures in plate– shaped products. In the RCS process, a work-piece is repetively corrugated and straightened without significantly changing the cross-section geometry - and what is very important - this process can be easily adapted to large – scale production. In this work, using original construction of laboratory–scale die set for continuous RCS process, the efficiency of refinement of microstructure in Cu-Cr alloy was investigated. The main objective of this work was to study the changes of mechanical properties as well as microstructure evolution versus circles number of deformation.

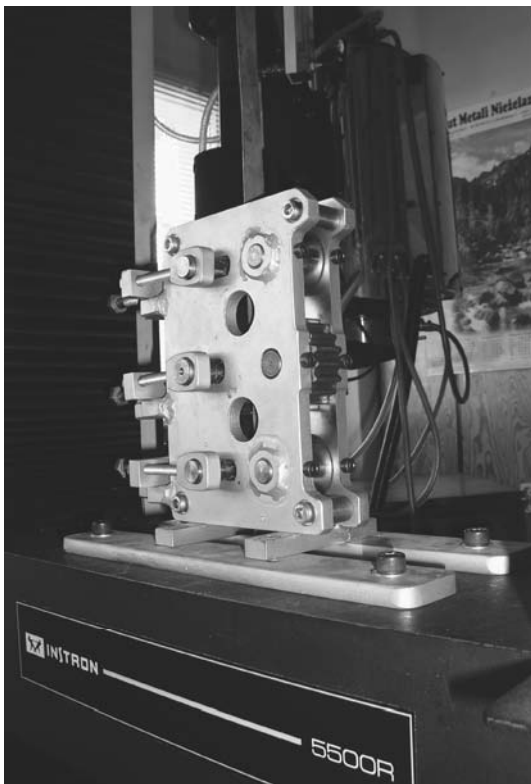


Fig. 1. Die set up for continuous RCS process installed on tensile testing machine INSTRON

## 2. Experimental procedure

Investigated material was precipitation hardened copper alloy with addition of 0.6% Cr, prepared by melting and alloying in an open-air induction furnace, followed by casting into 130x170 mm mould. Ingots were hot rolled up to strip thickness 3 mm. After surface brush cleaning the strips were cold rolled down to thickness 0.8 mm. Strip samples 1000 mm (length) x 20 mm (width), annealed at 650°C per 1 hour, were prepared for tests.

Continuous repetitive corrugation and straightening process have been conducted by strip drawing through toothed rolls (corrugation) and plain rolls (straightening) set. All rolls were assembled in a die set giving possibility to clearance control of rolling gap. This set have been installed on tensile testing machine INSTRON (Fig. 1), giving possibility to strip deformation (true strain about 0.8 in one pass). The investigated strips, during test, were immovable fixed and set of rollers was shifted with movement of tensile test machine cross-bar. Process has been conducted reversibly. System of strip tension has been also installed because of strip elongation during the process.

Before main tests, deformability defined as a number of deformation cycles to breaking was measured. Determined deformability of investigated CuCr0.6 strip was about 35 cycles which corresponds to the maximum true strain of about 30.

6, 12, 24 and 34 cycles of continuous repetitive corrugation and straightening were carried out. Thickness of strips after the process was reduced to 0.7mm. Also initial material was classically rolled to the thickness 0.7 mm, also. Microstructure investigations were carried out on samples of initial material and after 34 cycles, using optical and electron (SEM, TEM) microscopy. Transmission electron microscope investigations were done using JEOL (JEM 2000 FX). Observations were made on thin foil parallel to the strip surface. Crystallographic orientation analysis was done using electron backscattered diffraction (EDAX) set up on Philips SEM. The scanning parameters were set in such way that a grain boundary was defined when the disorientation between adjacent measurement points was higher than 6°. In the results the grain size determined by EBSD was smaller than the actual value. To ensure precision, these results were used in combination with TEM to determine the correct value. Mechanical properties of strips after 6, 12, 24 and 35 cycles were investigated in tension tests on tensile testing machine INSTRON.

## 3. Results and discussion

### 3.1. Microstructure

Microstructure of initial CuCr0.6 strip is shown on Fig. 2a. There were precipitated chromium particles in a copper matrix. Microstructure of these precipitations was investigated in former works [21, 22]. It could be said that after last annealing, without any mid-process precipitation microstructure of these particles correspond to their equilibrium BCC structure.

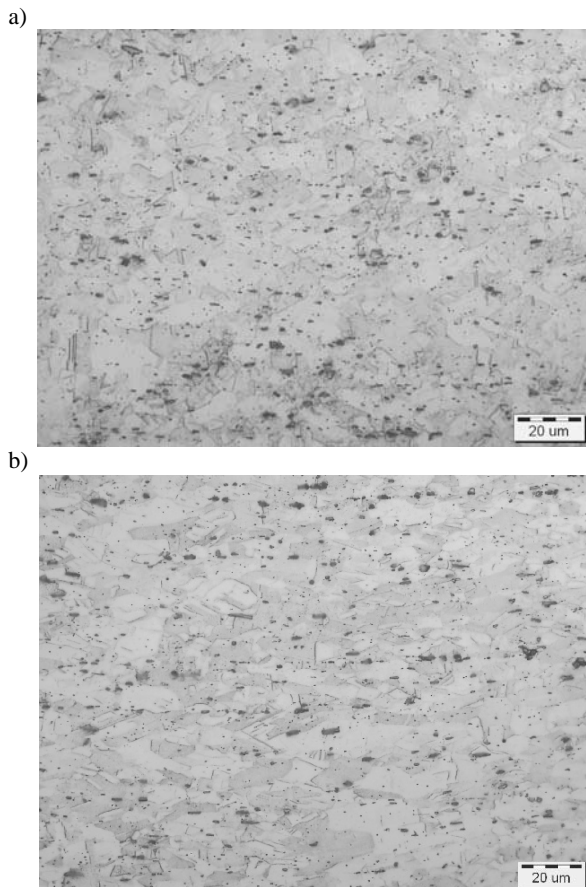


Fig. 2. Microstructure of CuCr0.6 alloy strip: as annealed (a), after 34 passes of deformation (b); Light microscopy

There were no significant changes in microstructure observed with optical microscopy before and after CRCS process (Fig. 2). Investigations with SEM equipped in electron back scattered diffraction system provides possibility for precise identification of crystal structure of investigated materials (Orientation Imaging Metallography – OIM). Microstructure refinement has been observed after CRCS process.

The microstructure observed in SEM is shown in Fig. 2a and Fig. 3a. Average grains size, determined by their cross section (including annealing twin boundaries) was about  $6.2 \mu\text{m}^2$ . After deformation substructure consisted of subgrains and dislocation cells were visible (Fig. 3b). The initial texture is not sharp. Crystal orientation of grains was from [00] to [111]. There were few large grains with orientation between [001] and [101].

After deformation, average cross section area of grain boundaries was about  $3 \mu\text{m}^2$ . Microstructure refinement was accompanied by forming of grains with lower disorientation degree. Grain boundaries disorientation distribution has been presented on Figs. 4a and 5a. On the histograms we can see that before deformation most of the grain boundaries were of high angle type (over  $60^\circ$ ). After CRCS process number of lower angle grain boundaries increased, also with disorientation angle under  $10^\circ$ . Grain size after deformation also decreased (Fig. 4b and Fig. 5b)

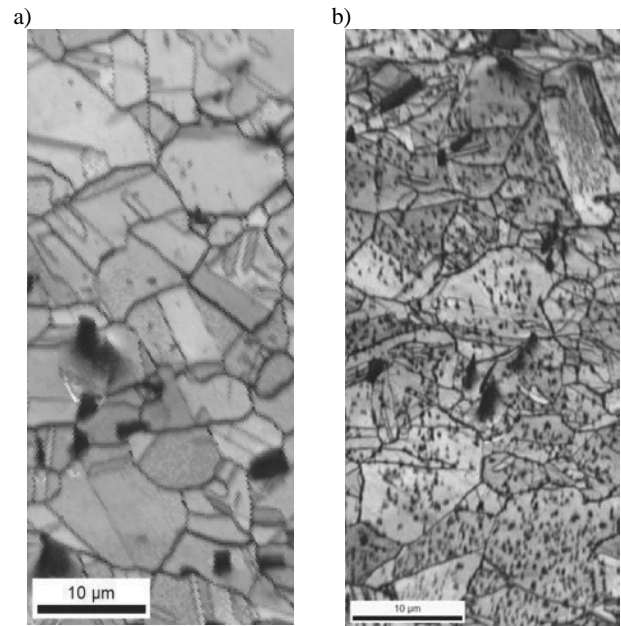


Fig. 3. Microstructure of CuCr0.6 alloy strip: as annealed (a), after 34 passes of deformation (b); SEM with EBSD

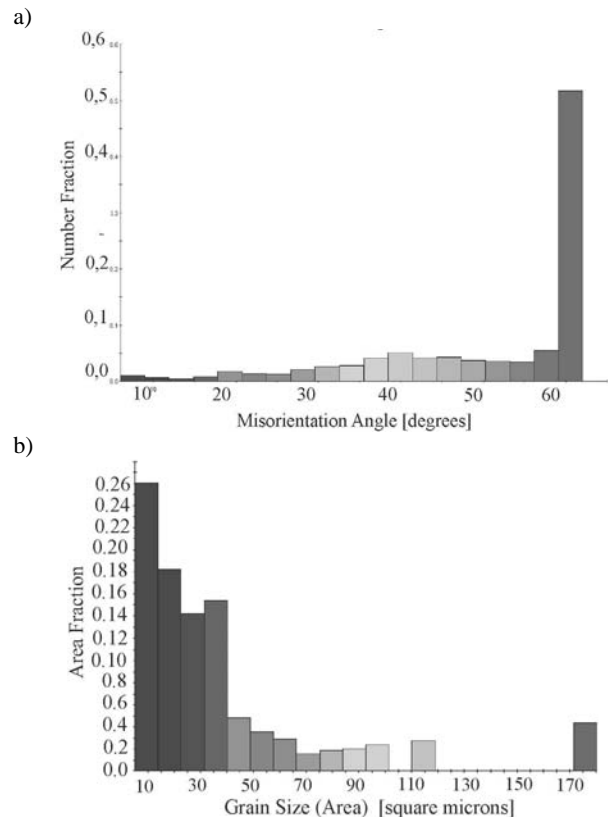


Fig. 4. Disorientation (a) and grain size distributions (b) of annealed sample (from EBSD)

During processing there were no significant changes of material texture. However some differences could be seen. There was a tendency to change a texture towards crystallographic orientation  $\langle 101 \rangle$ . It is indicated by more green grains and weakening of red and blue colour primary grains (Fig. 6).

All these changes of grain size and texture are also clearly seen in the pole figures presented in Fig. 7.

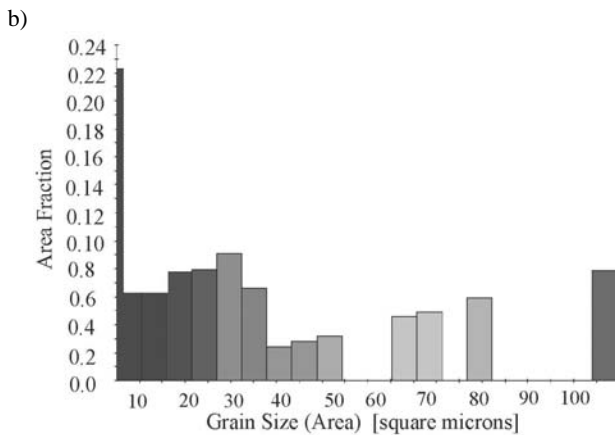
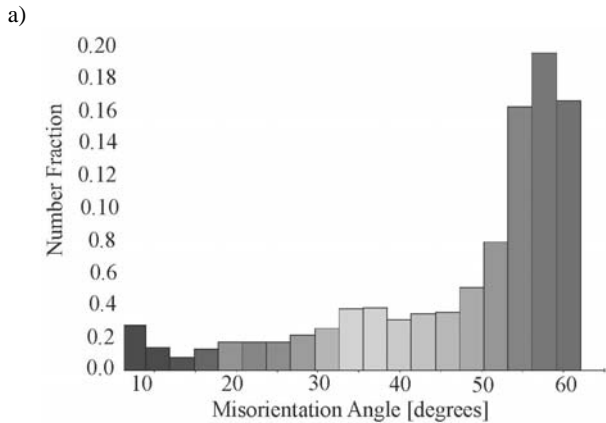


Fig. 5. Disorientation (a) and size of grains (b) distributions for 34 passes of deformation (from EBSD)

As it was mentioned the grain size, as predicted by EBSD data seems to be smaller than the actual value. To ensure precision, these results were used in combination with TEM to determine the correct value. Figs. 8a and 8b show a TEM micrographs of initial and deformed (34 passes) microstructure. Inside the primary grains individual grains or subgrains were produced of sizes ranging from about 100 nm to a few hundred nanometers. Also many dislocation cells and arrays of dislocations were observed.

Microstructural evolution in copper processed by severe plastic deformation was investigated mainly in processes using equal channel angular pressing.

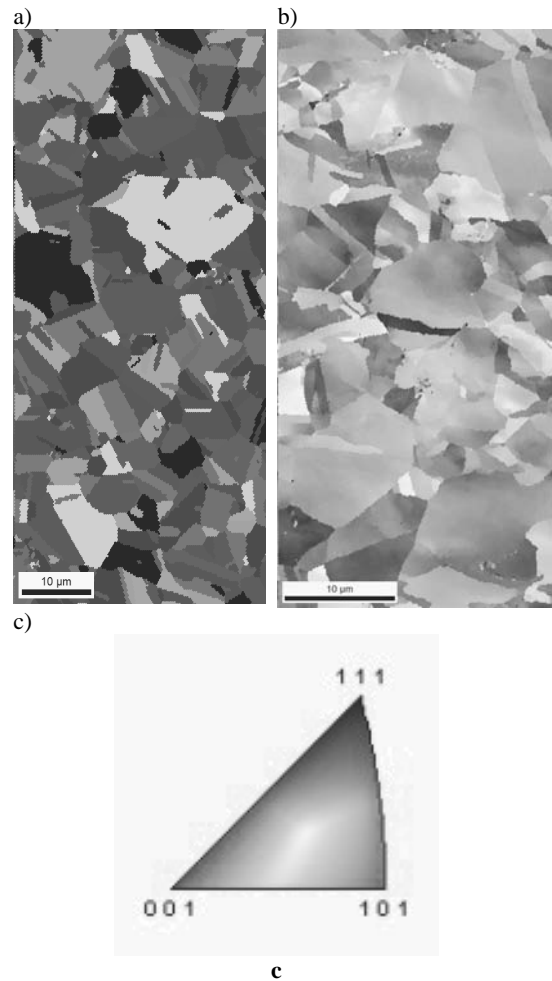


Fig. 6. Orientation maps for the annealed (a) and deformed (b) CuCr0.6 alloy strips, representation of the color code used to identify the crystallographic orientation: [001] red, [111] blue, [101] green

A preliminary model for microstructural evolution leading to ultrafine grain formation in copper by severe plastic deformation is discussed by A. Mishra at al. [7, 8] (ECAP) and by Huang at al. [20] (RCS). According to those models the grain refinement and microstructural evolution during RCS is as follows: at low strains, grains are first divided into cell-blocks, which contain dislocation cells. Dislocation angle zones may also develop inside cell-blocks. With increasing RCS strains, cell-blocks may further subdivide into smaller cell-blocks and dislocation-tangle zones may transform into dislocation cells. Subgrains will develop from both cell-blocks and dislocation cells. The later become subgrains when the disorientations across their boundaries are so large that they develop their own unique slip systems. The disorientation across subgrain boundaries increases with further RCS strain, and eventually becomes large enough to transform the subgrain boundaries into low-angle grain boundaries or high-angle grain boundaries.

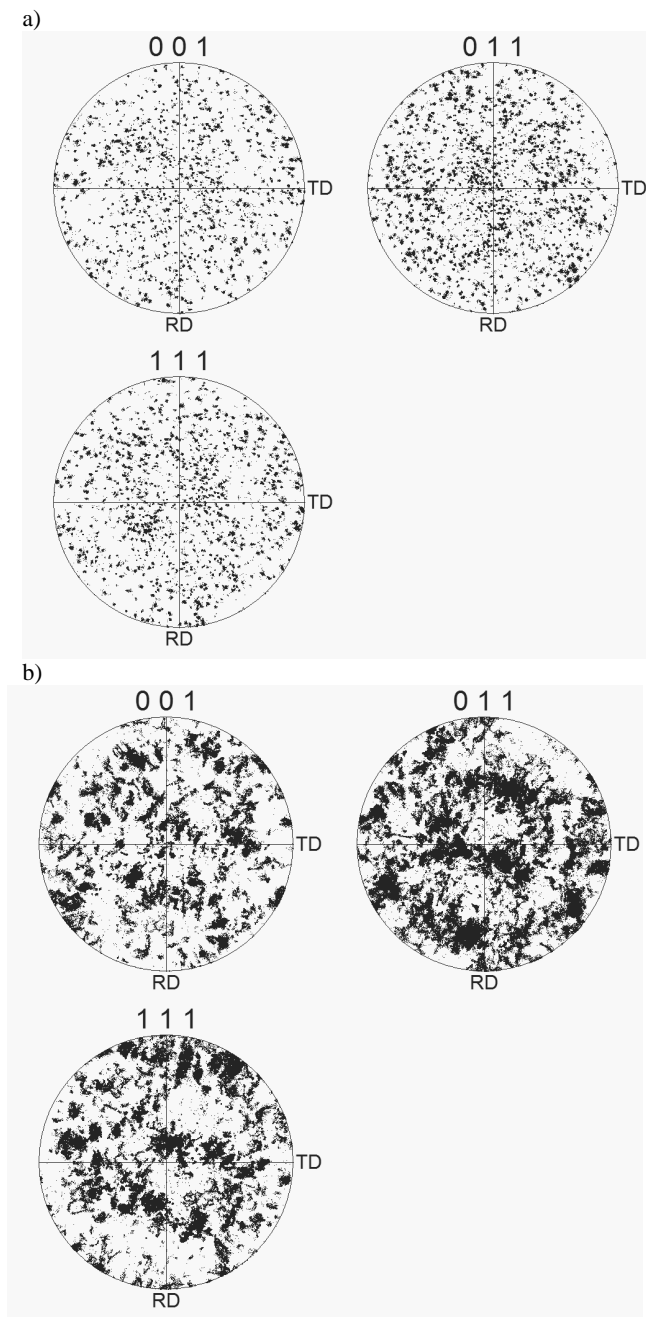


Fig. 7. Pole figures for (a) annealed and (b) deformed CuCr 0.6 strips

### 3.2. Mechanical properties

The study of the mechanical properties (tensile and hardness tests) were conducted on RCS samples that were annealed at 650°C for 1 h before corrugation and strengthening. The results of these versus number of deformation passes are shown in Fig. 9.

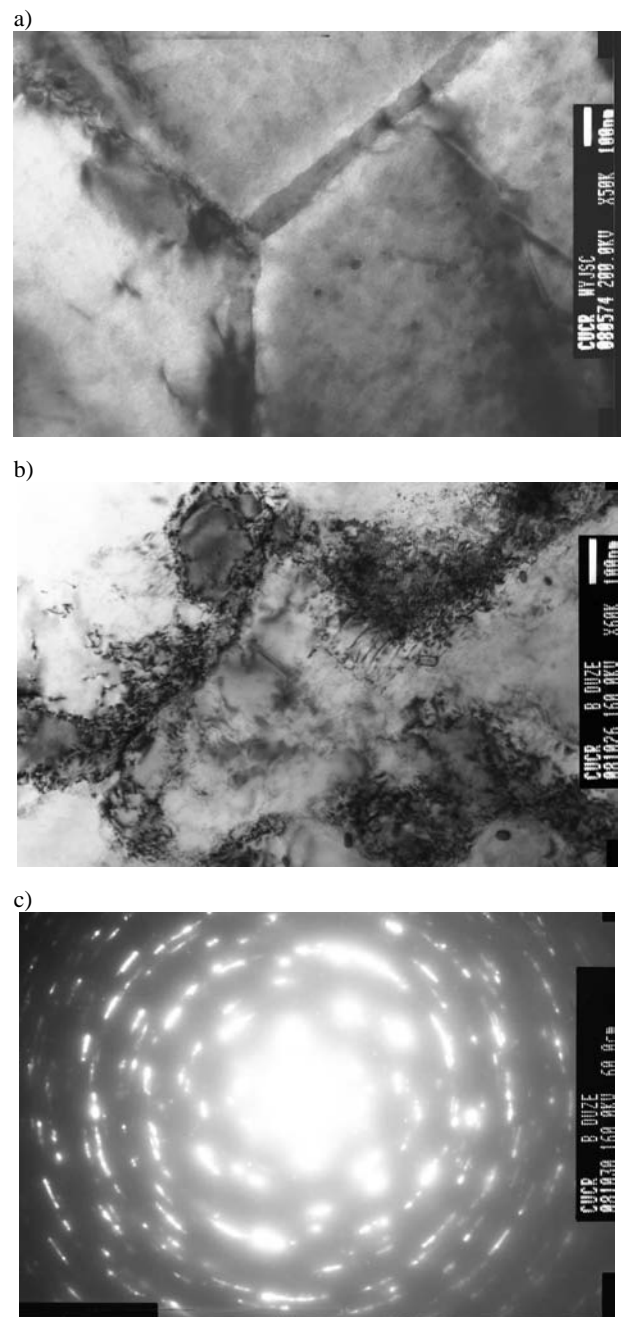


Fig. 8. Microstructure of CuCr0.6 alloy strips (z plane) TEM: as annealed (a), after 34 passes of deformation (b), corresponding SAED pattern (c); TEM

The initial sample showed large ductility. Work hardening in the RCS process caused a large decrease in ductility of the sample and increase of strength. The strength characteristics such as yield strength and ultimate tensile strength increases by a factor 1.7 and 1.2 respectively. Maximal strengthening effect has been achieved after 24 cycles of CRCS process. Further deformation caused decreasing of strengthening factors to the values of about 1.5 and 1.1.

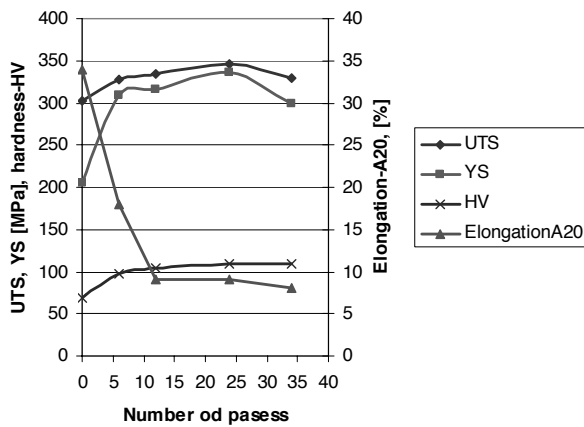


Fig. 9. Changes of mechanical properties of CuCr0.6 strip during CRCS processing, versus the classically rolled samples

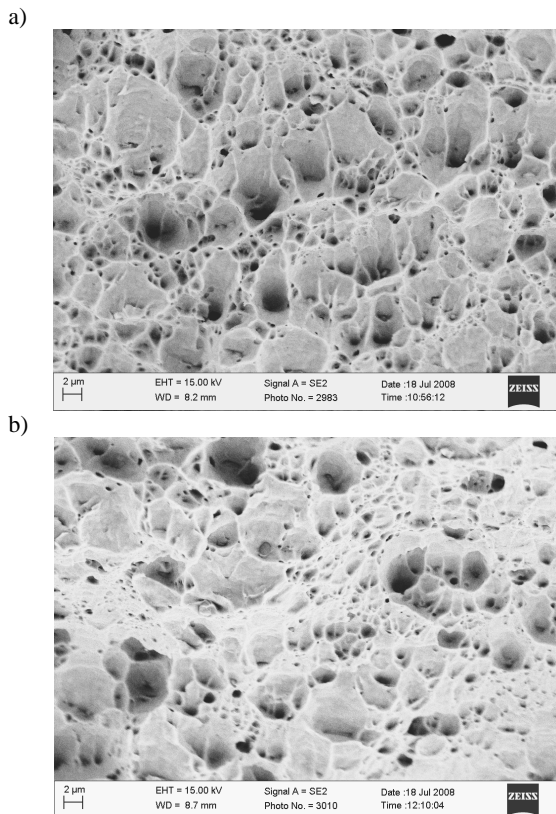


Fig. 10. SEM micrographs of fracture surface of CuCr0.6 samples: (a) initial annealed and (b) ultrafine-grained fracture

The ductility of the samples expressed by relative elongation decrease from 34% to about 8% after 12 passes then it becomes stable or slowly decreases.

Microhardness of the strip increased from about 70 to about 110 HV.

These results indicated complex deformation pattern and possibility to start new slipping systems, thus lowering of deformation resistance during CRCS deformation.

### 3.3. Fracture

Fig. 10 shows the fracture surface of the initial and 34 – pass RCS samples after tensile test. The fractographs show, at the bottom of the dimples, holes which are nucleation sites for fracture

There is no significant difference in the dimple size between the two fractographs, in spite of the significant difference in grain size.

The results obtained are comparable with those obtained by Mishra at al. [7,8] for copper. The interpretation for the similarity in the fracture morphology is that the nucleation sites (chromium particles), have similar distribution for both conditions. The same distributions of these particles results in formation of dimples, regardless the grain size. Nevertheless, the dimples seem to be shallower in the ultrafine-grained material which is a direct consequence of the decreased ductility.

## 4. Conclusions

This study was aimed to investigate mechanical properties and microstructure in CuCr0.6 alloy strips processed by continuous repetitive corrugation and straightening (CRCS). Based on the obtained results, the following conclusions can be drawn:

- The CRCS process effectively reduced the grain size of CuCr0.6 alloy strips, demonstrating the CRCS as a promising new method for producing ultra fine grained metallic strips.
- The OIM analysis of microstructure of CuCr0.6 strips after CRCS (34 passes) revealed refinement of average grain size (determined by their cross section) including annealing twin boundaries from  $6.2 \mu\text{m}^2$  to about  $3 \mu\text{m}^2$ .
- The sizes of structure elements revealed by OIM analysis were larger than those determined by TEM. TEM micrographs of deformed microstructure showed that individual grains or subgrains were, produced inside of primary grains with sizes ranging from about 100 nm to a few hundred nanometers. Also many dislocation cells and arrays of dislocations were observed.
- The strength characteristics of CuCr0.6 strips such as yield strength and ultimate tensile strength increase after CRCS by a factor of 1.2 and 1.7 respectively relative to initial state and remain virtually constant in the deformation range  $N=12-24$  cycles. Further increase of deformation caused decreasing of strengthening factor to value 1.5 and 1.2 respectively.

## Acknowledgements

This work was supported by the State Committee for Scientific Research under the projects No PBZ-MNiSW-3/3/2006 zad.4.1

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