ARCHIVES of
Computational Materials Science and Surface Engineering

International Scientific Journal published quarterly by the Association of Computational Materials Science and Surface Engineering

2010 • Volume 2 • Issue 3 • 125-132

Microstructure evolution in CRCS processed strips of CuCr0,6 alloy

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Received in a revised form 09.01.2010

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 the microstructure evolution and its influence on grain size refinement.

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 circles number of deformation were investigated. The microstructure was investigated using optical and electron microscopy (TEM and SEM equipped with EBSD).

Findings: 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. Generally, the mechanism of grain refinement and microstructural evolution during CRCS of CuCr0,6 alloy strips is similar to that observed in other high/medium stacking fault energy materials deformed by SPD, i.e. via dislocation manipulation and accumulation. Any effects connected with mechanical twinning were not observable.

Research limitations/implications: Investigation results are limited to the initial material in annealed state. Further investigation should focus on the description of influence of deformation-supersaturation-ageing sequence on strengthening effect.

Practical implications: A growing trend to use new copper-based functional materials is recently observed 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 show higher mechanical properties than microcrystalline copper.

Originality/value: The paper contributes to 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.P. Stobrawa, Z.M. Rdzawski, W. Głuchowski, W. Malec, Microstructure evolution in CRCS processed strips of CuCr0,6 alloy, Archives of Computational Materials Science and Surface Engineering 2/3 (2010) 125-132.

MANUFACTURING AND PROCESSING OF ENGINEERING MATERIALS

1. Introduction

Several different methods have been proposed to produce ultra fine grained (UFG) or nano-structured (NS) materials by imposing severe plastic deformation (SPD). Semi products produced by SPD are of great importance because they have low porosity, good mechanical properties such as high strength and toughness and their dimensions are suitable for testing of mechanical and physical properties. There are several SPD techniques that have been used to produce ultra fine grained or nano-structured materials, such as: equal channel angular pressing (ECAP), cyclic extrusion-compression (CEC), high-pressure torsion (HPT), cyclic closed-die forging (CCDF), accumulative roll-bonding (ARB), differential speed rolling (DSR), constrained groove pressing (CGP), constrained groove rolling (CGR), repetitive corrugation and straightening (RCS) and so on. Among them, the last two processes are especially applicable for continuous production of sheet or strip type products. These methods are based in principle on CGP method (large shear deformation in workpiece is realized by its repetitive grooving and flattening between plate shaped dies) in which flat dies are replaced with grooved and flat rolls.

The possibility to produce massive UFG or NS semi products from copper and copper alloys via SPD makes them attractive for engineering applications and creates new opportunities to explore their specific properties in comparison with coarse grain materials. Special attention is paid to optimisation of multi-functional thermal, electrical and mechanical properties. It results from growing interest in fine grained copper matrix materials or copper alloys.

The obtained grain size and character of UFG or NS forming depends on the SPD methods applied, processing regimes, phase composition and initial microstructure of the material.

Former investigations into copper alloys with UFG and NS structure focused mainly on semi-products prepared by powder metallurgy methods [1-6]. Classically prepared ingot materials were investigated in limited range. Deformation mechanism investigations were concentrated mainly on polycrystalline copper using ECAP [7-11] HPT [12-14], combined HE and ECAP [15] method or groove pressing technique [16-19]. Limited investigations were carried out on Cu-Cr-Zr alloy refined by ECAP method [20] and CuNi2Si alloy refined by RCS method [21].

In this work microstructure evolution of Cu-Cr alloy strip (in annealed state) have been investigated until UFG structure was obtained by RCS method.

2. Experimental procedure

In the investigations precipitation hardened copper alloy with addition of 0.6% Cr was used, prepared by melting and alloying in an open-air induction furnace, followed by casting into 130x170 mm mould. Ingots were hot rolled to strip thickness 3 mm. After surface brush cleaning the strips were cold rolled down to thickness 0.8 mm. Strip samples 1,000 mm (length) x 20 mm (width), annealed at 650°C per 1 hour or quenched in water from 900 °C were prepared for tests. Continuous repetitive corrugation and straightening process has been conducted by drawing of the strip through toothed rolls (corrugation) and plain rolls (straightening) set. All rolls were assembled in a die set which provided possibility for clearance control of rolling gap. This set has been installed in tensile testing machine INSTRON, which was used for strip deformation. During the test the investigated strips were firmly fixed while a set of rollers was shifted with controlled movement of tensile test machine cross-bar. Process has been conducted reversibly. A system of strip tension has been also installed because of strip elongation during the process. Process scheme and RCS rolling set were presented in a former work [21].

RCS process has been simulated to determine plastic deformation value and heterogeneity on longitudinal and cross section of strip. Continuous corrugation and straightening is a dynamic process, therefore its simulation is difficult. That is why for simplification of calculations a static simplified model of copper strip processing analogous to the corrugation and straightening process is commonly used. That procedure has been applied also in this work. The main objective was to draw up a deformation map on the strip cross sections after corrugation for existing tool geometry. Copper strip corrugation process has been simulated with finite elements method using bilinear elastic – plastic material model. Investigation results have been used for application of controlled position changing after every cycle of corrugation and straightening.

Before the main tests, deformability defined as a number of deformation cycles before breaking was measured. The determined deformability of investigated CuCr0.6 strip was about 35 cycles in annealed samples.

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. Microstructure investigations were carried out on the samples of initial material and after 6, 12 and 34 cycles, respectively, with optical and electron (SEM, TEM) microscopy. Crystallographic orientation analysis was done by electron backscattered diffraction (EDAX) system installed in Philips SEM. Transmission electron microscope investigations were done by JEOL (JEM 2000 FX). Observations were made on a thin foil, parallel and perpendicular to the strip surface. Mechanical properties of the strips after RCS cycles were investigated in tension tests performed with tensile testing machine INSTRON.

3. Results and discussion

The equivalent plastic deformation map produced during RCS process simulation (after first corrugation) has been presented in Fig. 1.

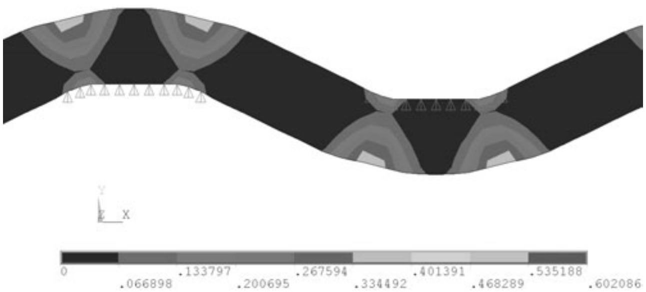


Fig. 1. Equivalent plastic deformation map

Plastic deformation was not uniform on longitudinal and cross sections of the strip after first corrugation. It changed periodically, maximum values area (bright colour) were close to the surface and presented value of 0.4. In the middle of the strip thickness this value was about 0.2. There was undeformed or slightly deformed area (dark colour) also.

Next stages of corrugation and straightening (with controlled strip movement between teeth) have led to uniformity of deformation. Uniform deformation with the average value $\varepsilon = 0.3$ can be expected after two or three cycles of corrugation and straightening on the cross section of strip sample. Every subsequent two or three cycles of deformation should increase the average by next 0.3 value.

Microstructure example of longitudinal section of initial sample (annealed at 650°C) has been presented in Fig. 2a. The microstructure of this sample after 35 cycles of deformation has been presented in Fig 2b. Fig. 2b shows that maximal stress was observed in area of maximum deformation on sample surface. The crackings occurred in the areas where stress values were greater than the critical one. Similar results were obtained for samples which were cooled in water from 900 °C. The crackings occurred after 25 cycles of corrugation and straightening in the similar areas of the surface.

During sample observation before and after RCS process by optical microscopy no significant changes in microstructure were recorded (Fig. 2). Using electron scanning microscope equipped with electron back scattered diffraction detector for precise identification of crystallographic orientation (Orientation Imaging Metallography – OIM) microstructure refinement after RCS process has been observed.

In order to characterize the uniformity of the microstructure, its evolution and grain-refinement mechanisms, deformation structure at an intermediate (6 RCS passes) and high deformation strains (12 and 34 RCS passes) were investigated using EBSD and TEM.

Fig. 3 shows development of several characteristics during RCS. The yield strength, ultimate tensile strength and elongation versus RCS passes are plotted. The yield strength and ultimate tensile strength simultaneously increase with the increase of RCS cycles (RCS passes between 10 and 24). Moreover, after 34 passes the yield strength and ultimate tensile strength slightly decrease.

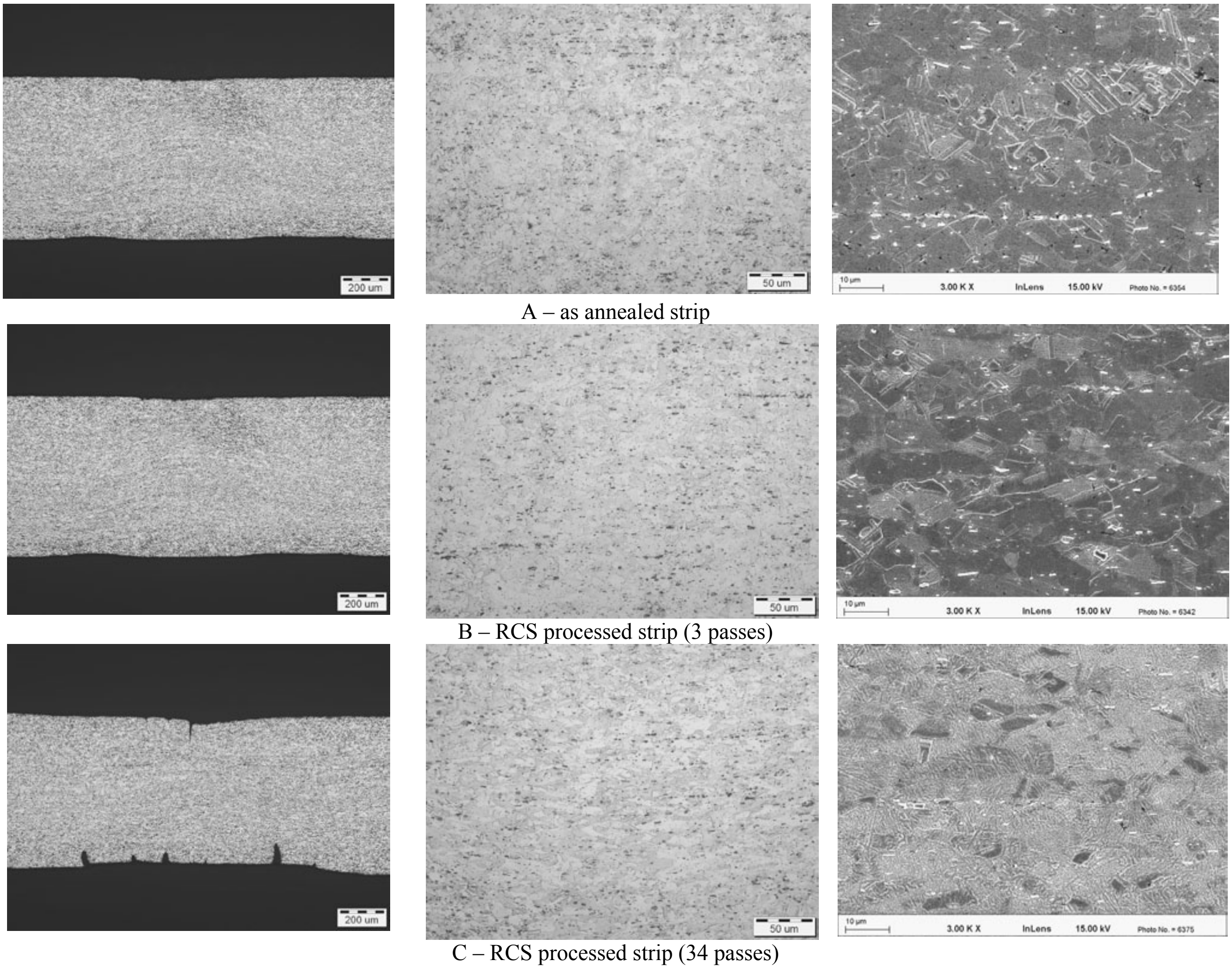


Fig. 2. Optical and SEM micrographs of as annealed and RCS processed CuCr0,6 alloy strips

Hardness is stable after 12 RCS passes. The deformation curve shows that six cycles of deformation did not result in maximal strengthening. At this stage the strip has a plasticity reserve (elongation decreased from 34 to 18%). Further deformation up to 12 RCS cycles caused decrease of elongation to about 8%. Then, it was stabilized on this level.

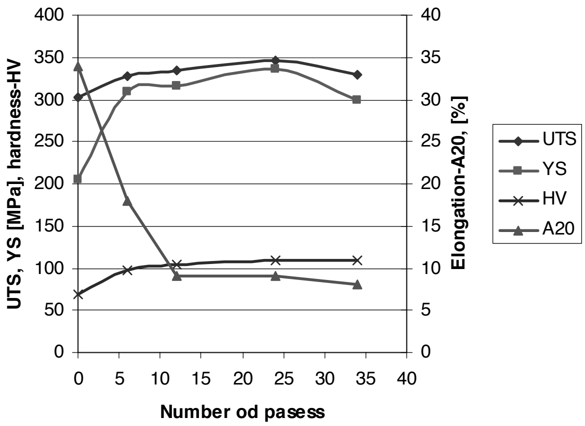


Fig. 3. Changes in mechanical properties of CuCr strips in RCS process

Microstructure of polycrystalline material is composed of an ensemble of grains separated by grain boundaries. In general, the grains differ in size, shape, crystallographic orientation and are surrounded by different types of grain boundaries. Occasionally, groups of grains are separated from their neighbours by grain boundaries of the same type, running along several grains

The investigation results of microstructure evolution using EBSD technique has been presented in Figs. 4-5 and using TEM in Figs. 6-8. The general tendency towards homogeneity of microstructures and grain size is consistent in these cases. Nevertheless, the average grain size obtained by the EBSD technique is clearly larger than that detected by TEM. This discrepancy can be due to the different intrinsic resolution ability of the two characterization methods. The scanning parameters were set in such a way that a grain boundary was defined when the misorientation between adjacent measurement points was higher than 5^o. Microstructure of initial strip is shown in Fig. 4. Fig. 4a presents quality image map (IQ map). Bright areas of the IQ map show the diffracted bands. Any area that produces poor patterns such as grain boundaries, poorly prepared phases, and surface damage will show up dark. In our case the bright grains in this image are the grains of copper while some dark areas are inclusions of large chromium particles. Very small chromium precipitates which existed in annealed or aged alloys [21-23] are not visible. Neighbouring pixels which presented maximum misorientation of 5° are grouped together as grains and the twin lamellae are identified as intragranular features. Grains size distribution according to the grain area fraction for such a case is shown in Fig. 4b. Average grains size, determined by their diameter and grain frequency (including annealing twins) was about 2.1 μm.

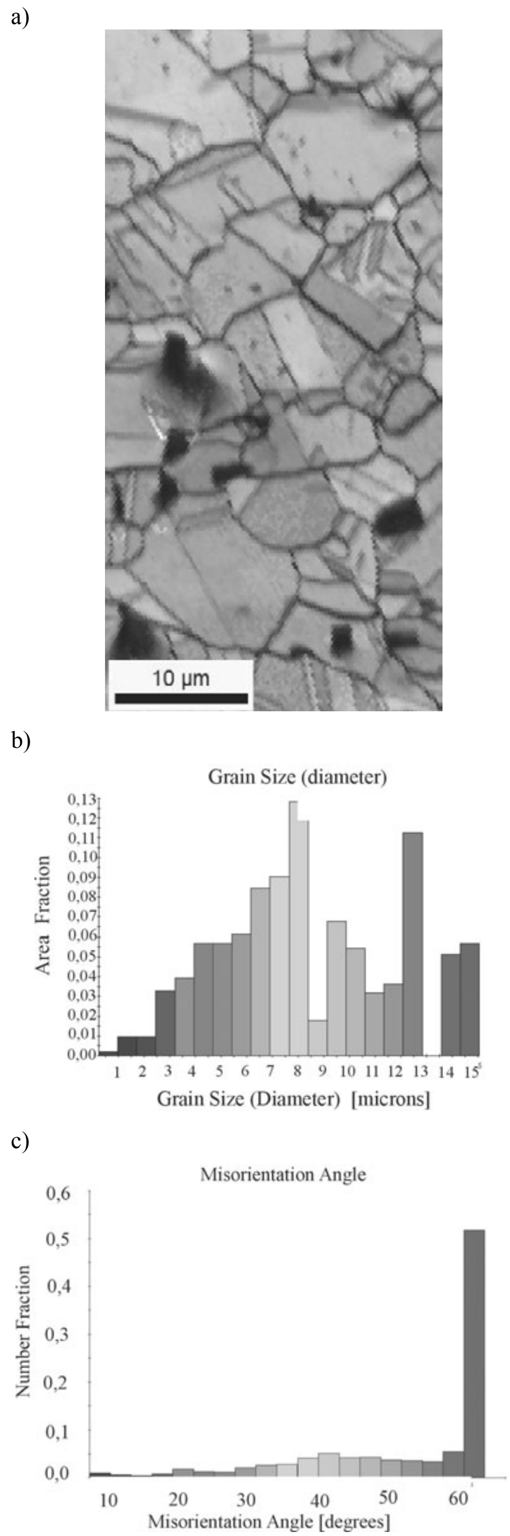


Fig. 4. Microstructure of as-annealed CuCr0,6 alloy strip (EBSD): a- IQ map, b- grain size distribution, c- misorientation angle distribution

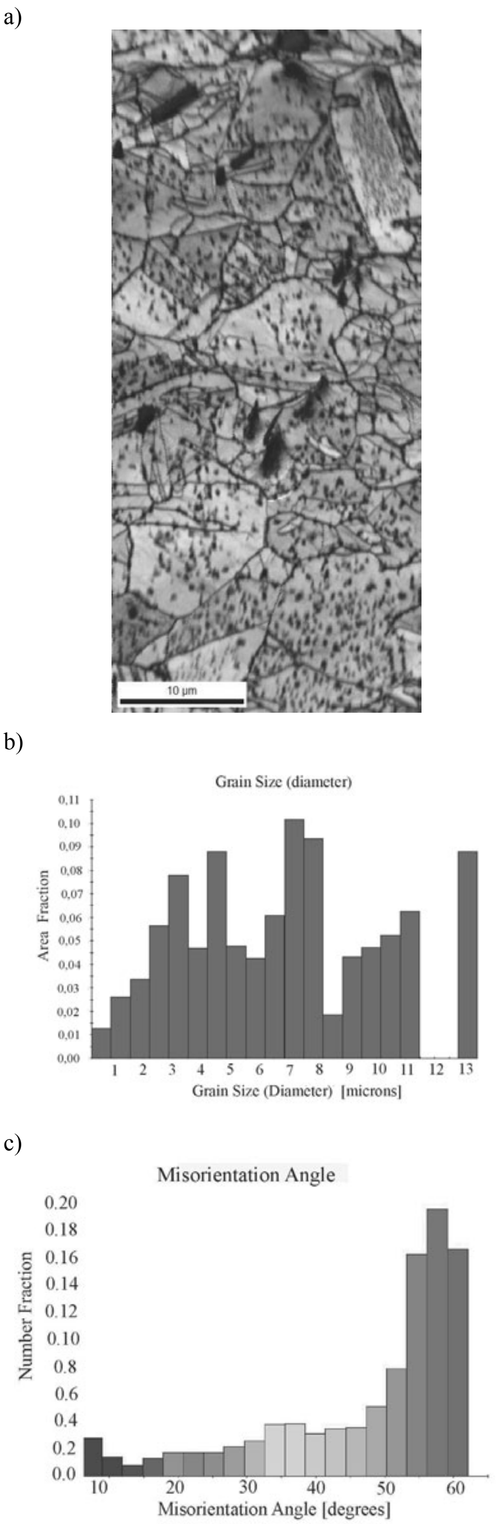


Fig. 5. Microstructure of CRCS processed (34 passes) strips of CuCr0,6 alloy (EBSD); a- IQ, b- grain size distributions, c- misorientation angle distribution

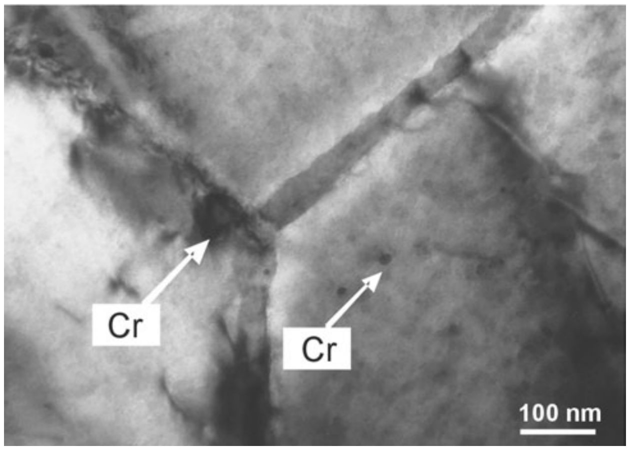


Fig. 6. Microstructure of as annealed (650 ^{o}C/1hr) CuCr0,6 alloy strip –TEM

After deformation the average grain diameter was about 1.2 µm, and at the same time, grains of lower misorientation angle were established. Distribution of misorientation angle of grain boundaries has been presented in Fig. 4c and 5c. It can be observed that before application of the process vast majority of the grain boundaries were of high misorientation angle type above 60°. After RCS process the number of grain boundaries of low misorientation angle (even below 10°) increased. Inside the grains there was a substructure consisting of subgrains and dislocation cells (Fig. 7b). Figures of electrolytically etched dislocations could be also visible.

Fig. 6 shows microstructure of as-annealed strip. Fig.7 demonstrates TEM observations of microstructural evolution of CuCr0,6 alloy strips during RCS process. The alloy matrix is completely recrystallized. The arrows show precipitates of chromium distributed in the matrix as well as at the grain boundaries. In the initial stages of deformation (6 passes) the microstructure consist of mixture of elongated (with roughly parallel boundaries) and circular subgrains or cells. Increasing the deformation (12 passes) degree the fraction of the elongated subgrains or cells decreases. Meanwhile, the activation of different slip systems truncated previous lamellar boundaries into approximately equiaxed subgrains. As a result, the deformation structure is more uniform. Further increase in the strain up to 34 passes results in stabilization of cell size and increase in cell misorientation. This can lead to immediate intensification of rotation mode of deformation within the whole sample volume and to the observed slight decrease of yield strength. At low strain value the dislocations are concentrated mainly in cell walls. Zones of tangled dislocations may also develop inside cell-blocks (e.g. circled area A, B and C in Figs. 7b, d, f). These zones may transform into dislocation cells (Fig. 8).

According to these results the grain refinement and microstructural evolution during RCS of CuCr0,6 alloy strips is similar to that observed in other the high/medium stacking fault energy materials [24-27]. In those materials the plastic strain induced grain refinement results from dislocation manipulation and accumulation. During the early stage of deformation, strong dislocation activities result in the formation of dense dislocation walls or cells. These dislocation configurations subdivide the

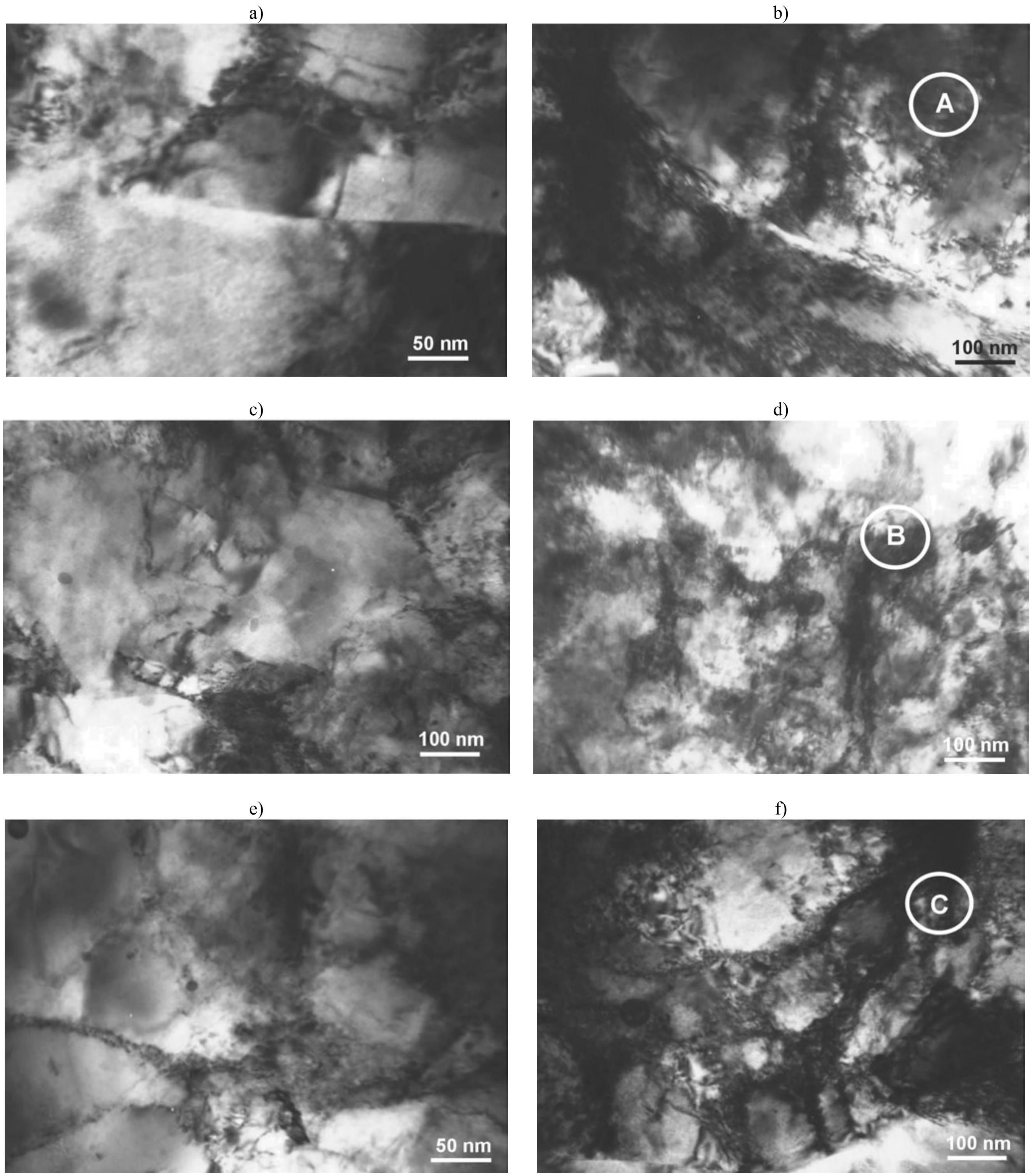
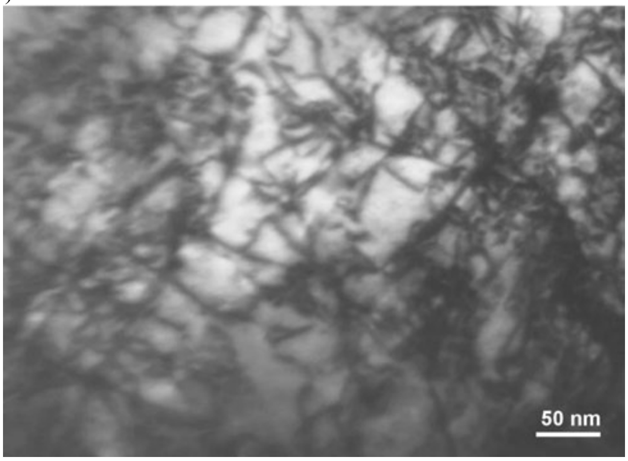


Fig. 7. TEM microstructure of CuCr0,6 alloy; a, b- RCS processed (6 passes), c, d- RCS processed (12 passes), e, f- RCS processed (34 passes). Column A of RCS processed samples represents microstructures of thin foils parallel to the strip surface and column B of RCS processed samples show microstructures of thin foils perpendicular to the strip surface and along the strip

a)



b)

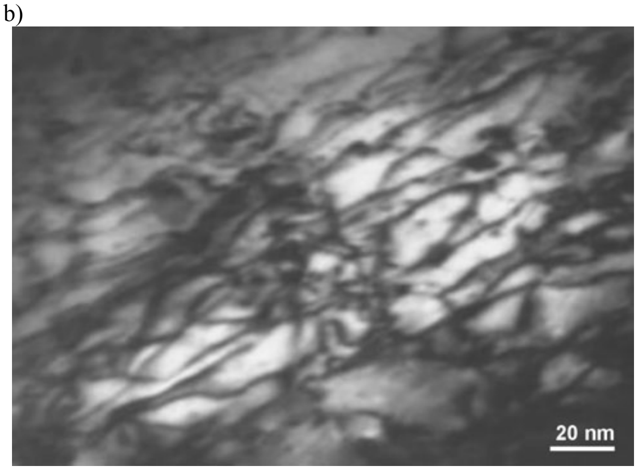


Fig. 8. Dislocation cells in CRS processed CuCr0,6 alloy strips: a- thin foil parallel to the strip surface, b- thin foil perpendicular to the strip surface

original large grains into cell-blocks, which contain dislocation cells. Increasing RCS strains, cell-blocs 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 misorientations across their boundaries are so large that they develop their own unique slip systems. The misorientation across subgrain boundaries increases with further RCS strain. Increasing the strain, the walls are transformed into subboundaries by depositing and recombining more dislocations. The energy and misorientations of subboundaries are raised progressively by generation and annihilation of dislocations in the sub-boundaries with further deformation. Eventually they become large enough to transform the subgrain boundaries into low-angle grain boundaries or high-angle grain boundaries.

The presented results are in conformity with many previous studies showing that the reduction in grain and subgrain size is faster during the early stage of SPD and slows down as the strain increases [28]. It should be also noted that the grain size reduction reached in this study is lower than the produced by other SPD methods, especially ECAP and HPT. The small reductions in both grain and subgrains are attributed to its simple deformation field and unique loading conditions.

4. Conclusions

In this work the investigation results of mechanical properties and microstructure changes in CuCr0,6 alloy strips processed by continuous repetitive corrugation and straightening (CRCS) are presented. Several findings obtained from the investigation results can be summarized as follows:

- 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. In general, grain refinement by CRCS method is lower than in others SPD methods at a given strain. This is attributed to its simple deformation field and unique loading conditions.
- The OIM analysis of microstructure of CuCr0,6 strips after CRCS (34 passes) revealed a refinement of the average grain size (determined by cross-section of the grains) including annealing of twin boundaries from 2.1 μm to about 1.2 μm. The size of structure elements revealed by OIM analysis was larger than the size determined by TEM. TEM micrographs of deformed microstructure showed individual grains or subgrains of sizes ranging from about 100 nm to a few hundred nanometres produced inside the primary grains. Also many dislocation cells and arrays of dislocations were observed.
- Generally the mechanism of grain refinement and micro-structural evolution during CRCS of CuCr0,6 alloy strips is similar to that observed in other high/medium stacking fault energy materials deformed by SPD, i.e. by dislocation manipulation and accumulation. No effects connected with mechanical twinning were observed. Specific differences result from many other external and internal factors, i.e. loading modes, strain magnitude, strain rate, temperature, melting point, activation energy and bulk modulus.
- After CRCS the strength parameters of CuCr0,6 strips, such as yield strength and ultimate tensile strength, increase by a factor of 1.2 and 1.7, respectively, when compared to the initial state and remain virtually constant in the deformation range N=12-24 cycles. Further increase in deformation resulted in the decrease of the factors to the level of 1.1 and 1.5, respectively.

Acknowledgements

This work was supported by the State Committee for Scientific Research under the projects No PBZ-MNiSW-3/3/2006 task 4.1 and MNiSW – N N507 2718 36. and N507 013 32/0542.

Additional information

Selected issues related to this paper were presented at the 18th International Scientific Conference on Achievements in Mechanical and Materials Engineering AMME`2010.

The paper is published also in the Journal of Achievements in Materials and Manufacturing Engineering.

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