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# EFFECT OF ECAP PROCESSING ON THE PROPERTIES OF COLD ROLLED COPPER

# WPŁYW PROCESU ECAP NA WŁASNOŚCI WALCOWANEJ NA ZIMNO MIEDZI

The study of the mechanical properties comprised fracture surface examination, texture and microstructure observations of copper samples, which were cold rolled, beginning from the recrystallized state or initially deformed by Equal-Channel Angular Pressing (ECAP) up to equivalent deformation $\varepsilon = 2.7$. In cold rolled recrystallized copper, intensive shear band formation on the sample scale as well as on micro-scale was observed; shear bands in cold rolled copper, initially ECAP processed, were difficult to observe. Higher ultimate strength and greater elongation were measured in tensiled samples, cut out in RD direction of rolled to $z = 96\%$ copper sheet and preliminarily ECAP processed samples; strain marks on the lateral surface of such a sample in the neck region demonstrated the grain size diameter lower than 1 μm. Fracture surface observation of ruptured sheet samples of recrystallized copper has shown that it is possible to explain the lower elongation as a result of localised deformation in shear bands. In both cold rolled sheets, texture with {112}<11-1> ideal orientation was observed, although in preliminarily ECAP processed copper the dispersion of crystal orientation around the ideal orientation was smaller.

Przeprowadzono badania własności mechanicznych, połączone z obserwacjami powierzchni przełomu, tekstury i mikrostruktury walcowanych próbek miedzi, albo od stanu zrekrystalizowanego albo od wstępnie odkształconego przez wyciskanie w kanale kątowym aż do odkształcenia zastępczego $\varepsilon = 2.7$. W zrekrystalizowanych próbkach walcowanych obserwowano intensywne pasma ścinania, zarówno w skali próbki jak i pod mikroskopem elektronowym; pasma ścinania w walcowanych próbkach uprzednio wyciskanych były rzadko obserwowane. Stwierdzono wyższe wartości wytrzymałości na rozciąganie i większe wydłużenie w rozciąganych próbkach wyciętych w kierunku walcowania z walcowanej do $z = 96\%$ miedzi, uprzednio wyciskanej w kanale kątowym; ślady odkształcenia na powierzchni bocznej szyjki rozciągniętej próbki wykazywały wielkość ziarna mniejszą od 1 μm. Obserwacje przełomu pokazały, że obniżenie wydłużenia próbek rozciąganych, wyciętych z walcowanej miedzi zrekrystalizowanej, może być wynikiem zlokalizowania odkształcenia w pasmach ścinania. W obu walcowanych blachach występowała tekstura z główną składową o idealnej orientacji {112}<11-1>; w próbkach wstępnie wyciskanych obserwowano mniejsze rozmycie orientacji wokół położeń idealnych.

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## 1. Introduction

Improved mechanical properties of materials with ultra-fine grained microstructure were reported [1]. They are manifested by higher strength, hardness and ductility in comparison with conventional coarse-grained materials; in these materials the super-plastic flow at lower temperatures in comparison with the conventionally processed materials is also observed. Severe plastic deformation (SPD), also known as hyper-deformation processing up to extremely high deformation degree, is commonly proposed as a method to obtain ultra-fine grained microstructure. One of such methods is processing by means of Equal-Channel Angular Pressing (ECAP) [2]. That type of processing has been elaborated and a corresponding equipment was constructed [3]. Its attractiveness and its advantage is obtaining of bulk samples of relatively great dimensions and free from porosity in opposition to the methods based on powder metallurgy, rapid cooling or crystallization from gaseous phases.

In the course of cold rolling of FCC structure metals (A1 structure), the strain localizations in the form of macroscopic shear bands SB are noted, when initial cold working z, depending on the stacking fault energy (SFE), is equal to $z = 40\%$ in metals with low SFE as in CuZn30 brass, and is equal to $z = 70\%$ in metals with moderate SFE such as copper. The shear bands appear as plate-like elements lying in the plane parallel to transverse direction and inclined to rolling direction at an angle of the order 35°. Such non-homogeneities were thoroughly investigated in the author's studies of copper and aluminium alloys under cold rolling [4] and under the shear plane test where the shear deformation is acting in the plane of sheet [5,6].

The main objective of the paper is to investigate the mechanical properties, related to the texture and microstructure changes, under cold rolling of copper, initially recrystallized or pre-deformed by Equal-Channel Angular Pressing.

## 2. Material and experimental technique

Experiments were carried out on copper Cu 99.97% B recrystallized at 773 K, with the initial grain size diameter $d = 30 - 40$ μm. ECAP processing, using a specially constructed equipment [3] was performed by means of INSTRON 6025 universal testing machine. Multiple procedure of pressing a sample of $10 \times 10$ mm$^2$ cross section was performed according to route $A$ [1,2,7], i.e. without any rotation around the sample axis or changes of the sample position. Samples of copper were then cold rolled on a laboratory two-high mill with rolls of 150 mm diameter at a velocity of 2.8 m/min.

Strain marks induced by deformation on the lateral surface of samples cold rolled up to $z = 70\%$ were observed by means of optical microscope. At that deformation step also the microstructure observations were carried out using Philips CM 20 electron microscope; thin foils, perpendicular to transverse direction TD of a rolled sample, were prepared by electrolytic thinning.

Tensile tests specimens were cut out in rolling direction RD of sheets cold rolled up to $z = 96\%$. At that deformation step also the changes of crystallographic orientations were studied by the method of pole figures. The measurements of pole figures, with the normal direction ND perpendicular to the sheet plane and rolling direction RD along the extrusion direction ED, were performed using Philips $X$-ray diffractometer and $CoK_\alpha$ characteristic radiation.

Table 1 presents the characteristics of all investigated copper samples.

TABLE 1

Characteristics of the investigated copper samples

| Equivalent deformation by ECAP $\varepsilon$ | Reduction of area by cold rolling* $z$ [%] | Total equivalent deformation $\varepsilon$ | Samples |
|---|---|---|---|
| 0 | 80 | 1.4 | CWR |
| 2.7 | 80 | 4.1 | CWE |
| 0 | 96 | 3.7 | CWRS |
| 2.7 | 96 | 6.4 | CWES |
| * equivalent deformation $\varepsilon = -(2/\sqrt{3})\ln(1\text{-}z)$ | | | |

## 3. Results and discussion

The properties of samples processed by different methods were studied by means of three experimental techniques as enumerated before: optical observations, crystallographic texture measurements and microstructure observations.

### 3.1. Sample scale shear banding

Shear banding under cold rolling is usually characterised by strain marks which are observed on the lateral surface of a cold rolled piece and in an extreme case, at high reduction of area, they cross the whole thickness of the deformed sample, sample scale or macroscopic shear bands [4]. Both copper samples were cold rolled to 70 % reduction of area with the aim to make the shear banding appear. Figure 1 presents photographs of observations in an optical microscope of strain marks on the lateral surface of rolled samples. Small and very rare shear bands traces are observed on the lateral surface of samples cold rolled to 70 % reduction of area, pre-deformed to equivalent deformation $\varepsilon = 2.7$ by ECAP processing (CWE in Fig. 1, surface additionally etched in water solution of a mixture: $CrO_3$, $NH_4Cl$, $HNO_3$ and $H_2SO_4$). It is worthy of note that the vanishing of macroscopic shear banding was registered by other researchers when reduction of area under cold rolling was increased above 96 % in case of brass Cu-30Zn [8] or when the cold rolled sample was characterized by initial, fine-grained microstructure (case of copper and copper alloys [9]). In the case of recrystallized copper, optical examination of strain markings (CWR in Fig. 1) has revealed the formation of regular shear bands crossing the whole thickness of the rolled sample (sample scale shear bands).

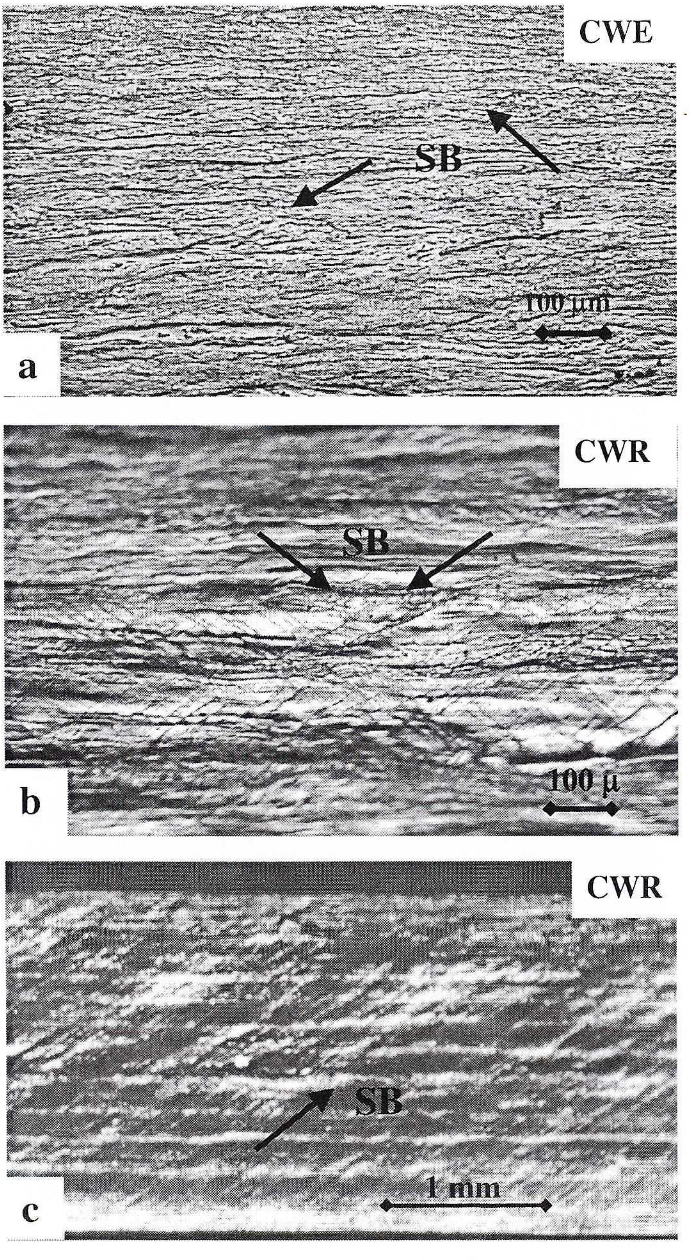



Fig. 1. Strain marks on the lateral surface of 70% cold rolled copper samples: a) CWE sample, pre-deformed by ECAP processing and additionally etched, b) and c) photographs of CWR sample after polishing and additionally cold rolled about 10%. Arrows indicate shear bands (SB), magnification mark indicates RD direction

## 3.2. Microstructure at shear banding

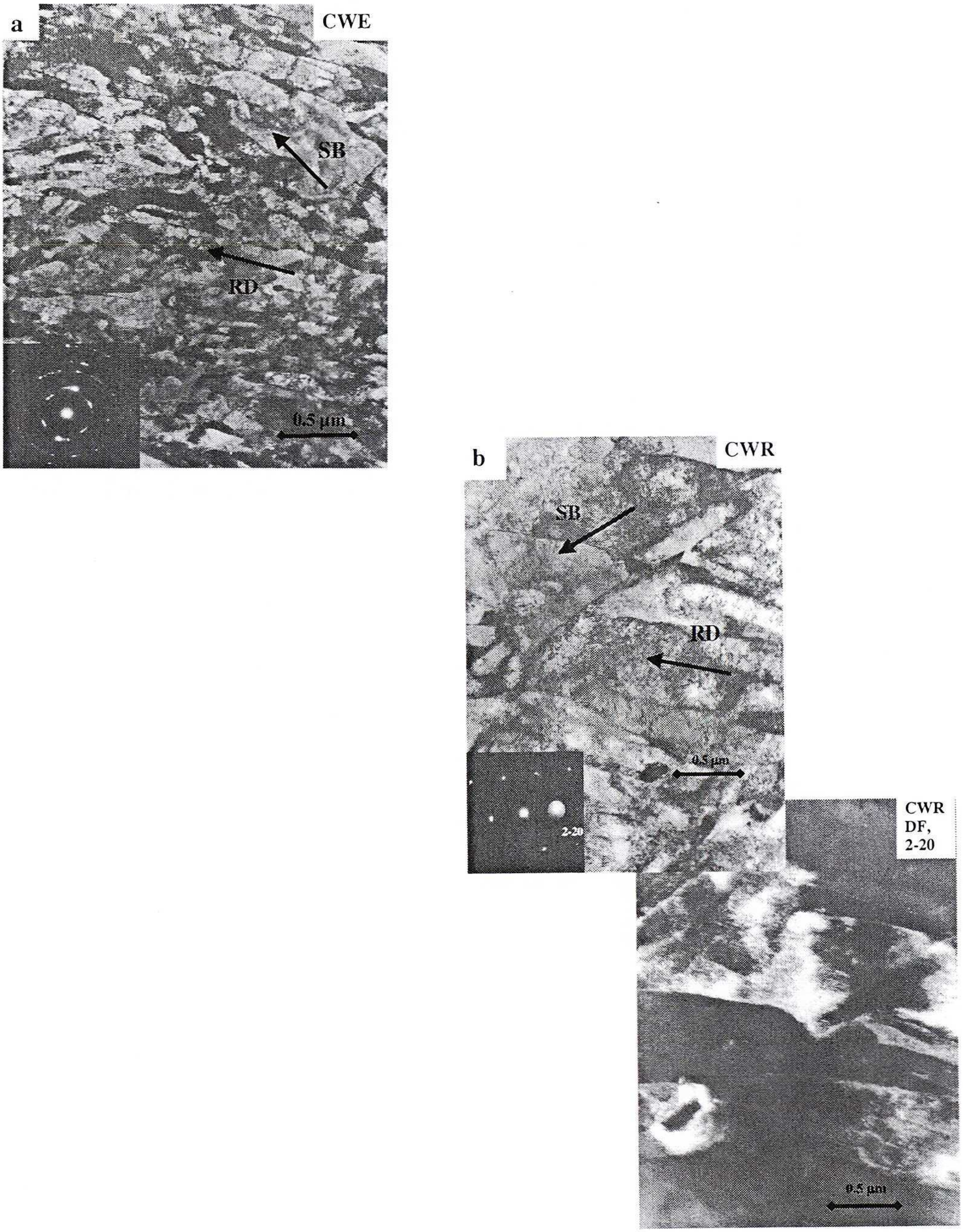



Fig. 2. Photographs of microstructures of samples observed in transmission electron microscope: a) CWE sample, preliminarily deformed by ECAP processing, b) CWR sample, recrystallized copper after cold rolling of 70%. Arrows indicate RD and SB directions, in Fig. 2b the dark field image DF in reflex 2-20 is additionally presented

Figure 2 presents the microstructure of samples, which were cold rolled up to z = 70%. In the case of a sample, preliminarily processed by ECAP up to equivalent deformation $\varepsilon$ =2.7 and subsequently cold rolled (CWE), the grain size was considerably below 1 μm (Fig. 2a) and shear banding was difficult to recognize. In the sample which was cold rolled to $z$ = 70%, directly after recrystallization treatment (CWR), as it can be observed in Fig. 2b, intensive shear banding on micro-scale is registered.

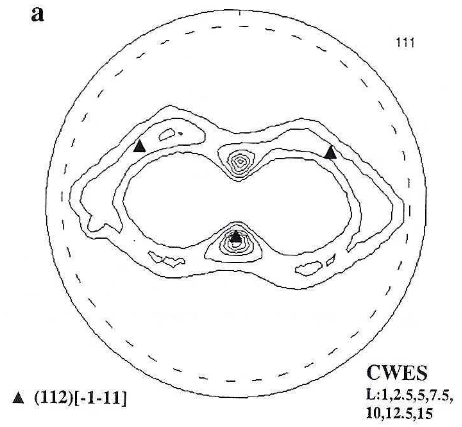
### 3.3. Crystallographic texture of rolled sheets

The evolution of crystallographic texture was traced analyzing the $X$-ray measured {111} pole figures (Fig. 3). We observed a decreasing dispersion of crystallite orientations for ECAP processed and subsequently rolled copper (CWES in Fig. 3a, maximum pole density attains 15). For comparison, in the recrystallized copper which was cold rolled up to the same cold reduction (CWRS in Fig. 3b), we observed the maximum pole density of 10 random units; both had the same main ideal component {112}<11-1>. The Orientation Distribution Functions [10] demonstrated consequently also the decreasing dispersion around the ideal orientations, both for the main and for the rest of the main ideal components (Table 2) for the sample CWES, pre-deformed by ECAP processing.

TABLE 2

Main ideal components of crystallographic texture

| Sample | Idal orientations $(hkl)[uvw]$ | Orientation Distribution Function $f$ [random units] | |
|---|---|---|---|
| | | $f_{hkl)[uvw]}$ | Maximum |
| CWRS | (123) [11-1] | 49 | 67.4 |
| | (112) [11-1] | 55 | |
| | (011) [3-11] | 48 | |
| CWES | (123) [11-1] | 82 | 95.4 |
| | (112) [11-1] | 59 | |
| | (011) [3-11] | 22 | |



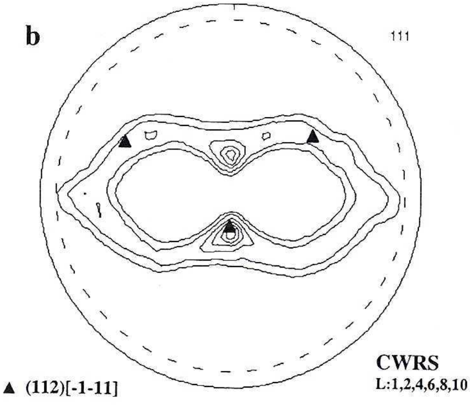




Fig. 3. $X$-ray measured {111} pole figures of CWES samples (ECAP processed and cold rolled 96%) and CWRS (cold rolled 96%)

### 3.4. Mechanical properties of sheet samples

Hyper-deformation leads to refinement of the grain size and improves also the mechanical properties of processed materials. ECAP processing [3] of copper up to equivalent deformation $\varepsilon = 8.2$ has increased Vickers hardness to value HV = 1165.6 MPa in comparison to the HV = 1093.6 MPa of copper deformed by cold rolling to $z = 62$ In our case, two copper samples were cold rolled up to $z$ = 96% of cold reduction and sheet samples, destined to be examined under tensile tests: a) ECAP processed by route A up to equivalent deformation $\varepsilon = 2.7$ (CWES) and b) initially recrystallized (CWRS), were prepared. Samples with gauge dimensions: $L_o = 20$ mm, thickness $a_o = 0.4$ mm and width $b_o = 4.05$ mm were then stretched in the INSTRON testing machine at room temperature at a strain rate $\varepsilon = 0.001$ 1/s.The tension stress $\boldsymbol{S} = \boldsymbol{F}/(\boldsymbol{a}_o{*}\boldsymbol{b}_o)$ depending on elongation $\boldsymbol{e} = (\boldsymbol{L} - \boldsymbol{L}_o)/\boldsymbol{L}_o$ for the representatives of both sheet samples is presented in Fig. 4. The difference in ductility can be noted when comparing the total tensile elongation; elongation of CWES specimens (ECAP processing before rolling) is equal to 5.2%, whereas for CWRS samples (rolling of recrystallized copper) it is equal to 4.0% (Table 3). The yield point as well as the ultimate strength of CWES samples, pre-deformed by ECAP processing before rolling, are higher than those of cold rolled samples after recrystallization. The CWES samples were characterized by smaller grain size (compare the next paragraph) which improved their mechanical properties [1].

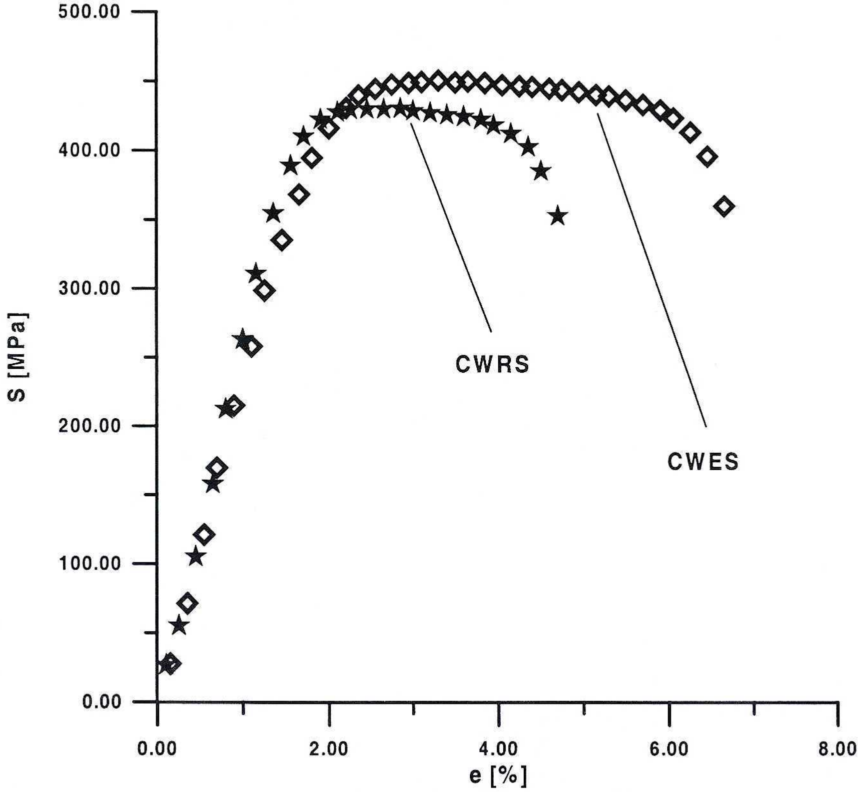



Fig. 4. Tension stress $S$ dependence on elongation $e$ for representative samples of 96% cold rolled sheets: CWRS and CWES

TABLE 3

Tensile properties of sheet samples

| Sheet samples | Equivalent deformation by ECAP $\varepsilon$ | Reduction of area by cold rolling $z$ [%] | Yield Point $R_{0.2}$ [MPa] | Ultimate Strength $R_m$ [MPa] | Total Elongation $e$ [%] |
|---|---|---|---|---|---|
| CWRS | 0 | 96 | 402.4 | 424.0 | 4.0 |
| CWES | 2.7 | 96 | 416.7 | 450.3 | 5.2 |

### 3.5. Fractography of sheet samples

Observation of the final stage of a plane sample stretching has shown that fracture is accomplished as a result of shearing in a band running along localized necking, inclined to the tension axis, as it was already observed in the author's earlier examinations [4]. Fracture was accomplished in both cases by a process of dimple and cone formation (Fig. 5). Regular striations, which form the band-like structure were observed on the lateral surface of both ruptured samples, Fig. 5b and 5d. In case of CWES sample (Fig. 5a,b) the furrows illustrate also grain size of the order of 1 $\mu$ and below (Fig. 5b). In case of ruptured CWRS samples (Fig. 5c,d) the lateral surface demonstrates furrows (Fig. 5d), as it was observed in ruptured sheet samples of rolled copper [4,11]. Intensive shear banding, observed during cold rolling of copper, directly after recrystallisation treatment, may account for the lower elongation of tensiled samples. In the author's earlier studies [12], even pre-matured fracture along shear bands, especially in rolled material characterized by a lower, in comparison to copper, stacking fault energy was observed.

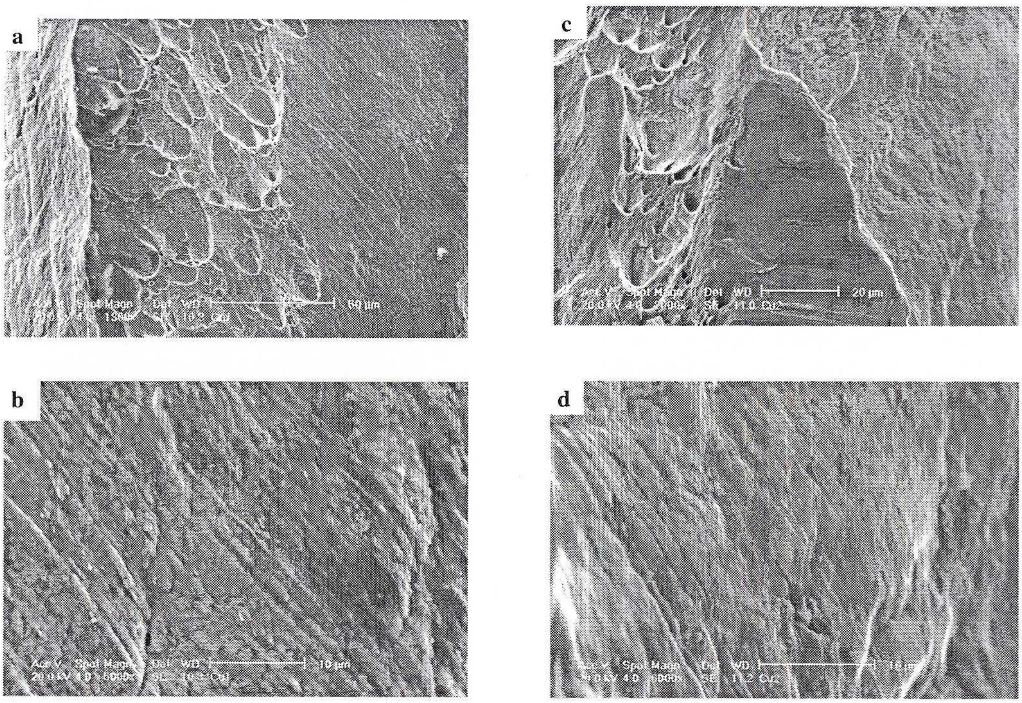



Fig. 5. Microphotographs of the fracture surface of ruptured sheet samples, obtained in scanning electron microscope: a) CWES fracture surface, b) lateral surface of CWES sample, c) CWRS fracture surface and d) lateral surface of CWRS sample. Magnification mark is perpendicular to the fracture surface

## 4. Conclusions

Shear banding in preliminarily ECAP processed Cu is difficult to observe, which is in agreement with the rolling of fine-grained materials.

Cold rolling of coarse-grained copper results in intense shear banding, which reduces the mechanical properties of cold rolled sheets:

a) The elongation in RD direction of cold rolled up to 96% copper sheet, preliminarily deformed by ECAP processing, is about 30% greater than that of cold rolled sheet after recrystallization,

b) The yield point as well as the ultimate tensile strength in RD direction of sheet, pre-deformed by ECAP processing before rolling, are higher than those of cold rolled sheet after recrystallization.

Crystallographic texture in 96% cold rolled copper is more pronounced in copper pre-deformed by Equal-Channel Angular Pressing and a small dispersion of orientation is registered around the ideal position, although in both the main component {112}<11-1> is observed.

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