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The Effect of Severe Plastic Deformation and Heat Treatment on CuCrZr Alloys

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CuCrZr alloy was subjected to equal channel angular pressing method, belonging to the severe plastic deformation group, followed by heat treatment under different ageing conditions to optimize mechanical properties of the alloy. Before equal channel angular pressing, CuCrZr alloy was treated by solution annealing at temperature 1020 °C for 1 h. Afterwards, samples were pressed through an equal channel angular pressing die once at room temperature and subjected to artificial ageing under different conditions (200, 400, 450, 480 °C for 30, 60, 90, 120, 150 min). Optimization of the CuCrZr alloy was done through the study of mechanical properties and microhardness as a function of ageing temperature and time considering the progress in microstructural/substructural features.

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1. Introduction

A lot of current engineering applications require a sophisticated combination of physical and mechanical properties that can be quite controversial [1]. Severe plastic deformation (SPD) methods applied to metals enable specific microstructural and substructural features to be formed during the processing resulting in an unexpected combination of mechanical properties [2, 3]. Formation of such structures in materials causes microstructural refinement to the ultrafine-grained (UFG) or even nanocrystalline (NC) area [4–6].

There are a number of SPD techniques such as high pressure torsion (HPT) [7], accumulative roll bonding (ARB) [8], equal channel angular rolling (ECAR) [9], but one of the most popular is equal channel angular pressing (ECAP) due to the equipment configuration simplicity. ECAP has been successfully used over the last decade to insure the ultrafine-grained structures in a wide range of ductile metals including pure Cu and Cu alloys [4, 5, 10–13]. Microstructure is refined to equiaxed subgrains/grains below 1 µm under high strains through simple shear, accompanied with a significant increase of strength. During ECAP processing, boundaries with relatively low misorientation are formed by dislocations rearrangement at lower strain (dislocation cells substructure), which are progressively transformed to higher angle grain boundaries at higher strains [5, 14, 15]. At the same time ductility remains good for such heavily deformed Cu alloys [16].

Cu and Cu alloys are widely used because of their excellent electrical and thermal conductivities, outstanding resistance to corrosion, good strength and fatigue resistance, suitable ductility [17–20]. As the mechanical strength of pure Cu is limited for current engineering applications, Cu alloys as an example CuCrZr are attractive for a number of industrial fields. CuCrZr alloys are classified as precipitation-hardened (PH) materials processed by heat treatment which are traditionally used in applications where a combination of high mechanical strength, heat resistance and electrical (or thermal) conductivity is demanded (electrodes for point welding, heat exchangers, etc.) [21–23]. It has to be mentioned that CuCrZr alloys are considered as a prime material for fusion reactors (international thermonuclear experimental reactor — ITER) [24, 25].

The aim of the study was to examine the mechanical behaviour of CuCrZr alloys processed by ECAP followed by ageing treatment focused on the study of microstructure.

2. Experimental conditions

The CuCrZr alloy supplied as a round bar of diameter 10 mm and length 100 mm (98.54 wt% Cu, 1.1 wt% Cr, 0.043 wt% Zr) was used. Before ECAP processing, samples were given a solution annealing of 1020 °C for 1 h and water quenched to form the single coarse-grained phase (CG) with mechanical properties: yield strength (YS) = 144 MPa, ultimate tensile strength (UTS) = 277 MPa, reduction of area (RA) = 81%. Consequently, the samples were processed once through an

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ECAP die (channel angle $\Phi = 90°$, $\Psi = 32°$) at ambient temperature. Heat treatment after ECAP processing was carried out at 200–480 °C for 30–150 min followed by water quenching.

Mechanical properties were evaluated by uniaxial tensile testing on samples with a cylindrical gauge carried out at ambient temperature according to the EN ISO 6892-1. Two samples were tested for each material condition, confirming great reproducibility of both strength and reduction of area. Microhardness was measured in terms of HV0.1 using the Struers apparatus.

The microstructure was characterized by FEI transmission electron microscope Tecnai G2 (TEM) operating at 200 kV equipped with high-angle annular dark field scanning transmission electron microscopy detector (HAADF-STEM) combined with energy dispersive X-ray (EDX) EDAX microanalysis. A Tenupol-5 double jet electropolisher was used for the thin foil preparation in electrolyte containing nitric acid and methanol (1:3), at the temperature of −28 °C and voltage of 20 V.

3. Results

3.1. Mechanical behaviour

Results from engineering stress–strain measurements were presented in the graphical forms. Figure 1 shows the progress in YS and UTS on ageing time at different temperatures, as is seen the change in properties reproduces each other. According to Fig. 1, ageing at 200 °C for 30, 60, 120, and 150 min provides no significant changes in the strength properties compared to the initial state without ageing (YS = 390 MPa, UTS = 392 MPa, RA = 66%). Although it seems that at the temperature of 200 °C and 90 min ageing time, samples provide enhanced strength properties. Considering ageing

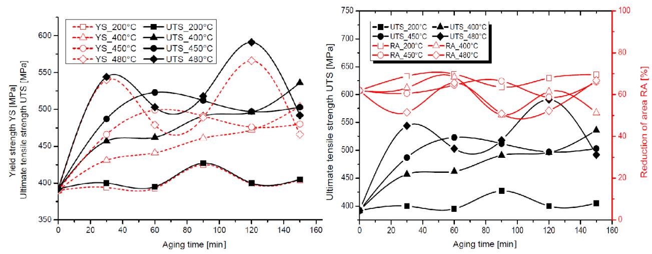

Fig. 1. Strength properties as a function of ageing time at different temperatures for CuCrZr alloy (compared to RA).

at 400 °C, strength properties gradually increased with longer ageing time, even progress in the properties was constant within the whole range of ageing time. On the other hand, ageing at 450 °C provides the growth in the strength compared to previous thermal processing but only up to 60 min. Then longer annealing time brings the reduction of the strength in CuCrZr alloys. Considering ageing at 480 °C for 30 min, the strength increased significantly even annealing for 120 min brings most visible growth in the strength. On the other hand, ageing at 480 °C also provides rapid changes in the strength progress, heat treatment under such conditions could be quite unstable having a negative impact on the stability of material properties.

The RA changed from 81% to 62% after first ECAP pass (without ageing). Within the ageing, the values of RA were in the range of ≈ 50−70% depending on processing conditions (Fig. 1, right, compared to UTS).

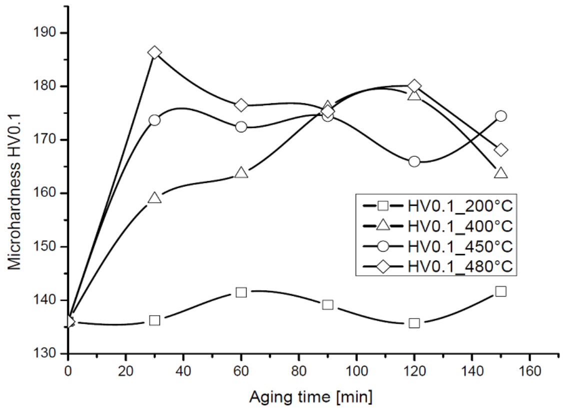

Fig. 2. Microhardness as a function of ageing temperature and time for CuCrZr alloys.

The results of microhardness measurements are summarized in Fig. 2. The initial microhardness of 50 HV0.1 has been measured for CuCrZr samples after solution annealing. After first ECAP pass (before ageing), the microhardness increased at 137 HV0.1. Ageing at 200 °C after ECAP processing does not provide any significant changes in the microhardness compared to a non-aged state. More visible increase in the microhardness was revealed at higher temperatures. At 400 °C, the microhardness increased with ageing time up to 120 min but for 150 min there was seen decrease in the values. Maximum microhardness of 186 HV0.1 was obtained under processing conditions at 480 °C for 30 min however further ageing provides the lower values. Ageing at 400 °C is seen as a most stable in the progress of microhardness. According to Fig. 2, most significant increase of the microhardness within ageing is recognized in the first 30 min. Similar progress was observed in the strength properties (Fig. 1). Moreover the next microhardness development (60, 90, 120, 150 min.) responds to the progress in mechanical properties (YS, UTS). Strong peaks in Fig. 1 corresponding to the strength properties under ageing at 480 °C for 120 min were not seen in the microhardness that could mean the mistake in measuring.

3.2. Microstructure observations

The microstructure of CuCrZr alloys after solution annealing and water quenching provides coarse grains with an average equivalent diameter of 50 µm. There were also visible numbers of twin boundaries [26].

In general, SPD methods insure significant microstructures refinement and already after the first pass of processing, re-arrangement in internal substructural features. Typical microstructure of CuCrZr alloys after solution annealing and 1 ECAP pass is shown in Fig. 3.

Dislocation cells of different shapes with curved boundaries and low misorientation angles have been recognized (Fig. 3a). The first ECAP pass also provides huge increase in the dislocation density as is seen in Fig. 3b.

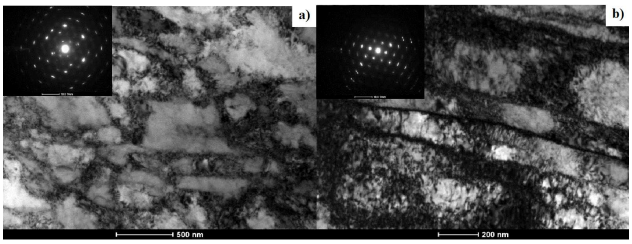

Fig. 3. Typical microstructure of CuCrZr alloys after 1 ECAP pass.

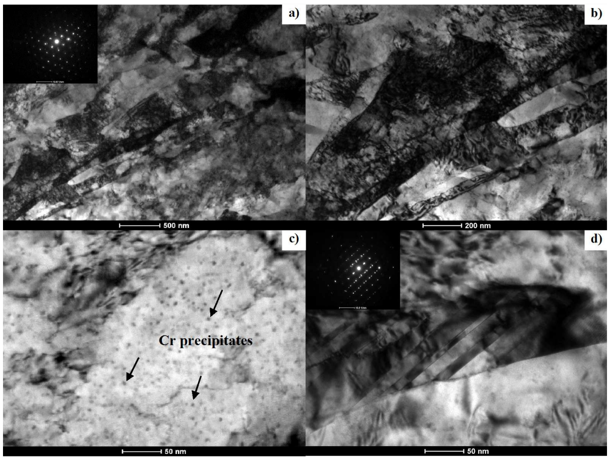

Fig. 4. Typical microstructure of CuCrZr alloys after 1 ECAP pass and ageing at 480 °C for 120 min.

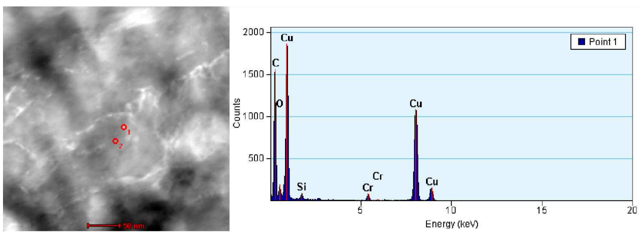

Fig. 5. HRTEM micrograph of CuCrZr alloys after 1 ECAP pass and ageing at 480 °C for 120 min (a) and the typical EDXS qualitative analysis spectrum of phases (b).

The structure with very high density of dislocations is typical for SPD copper based alloys that exhibit rather low stacking fault energy (SFE) [27]. As a result, dislocations are separating during plastic deformation which induces more dislocation tangles. They baffle dislocations to move and with further deformation they behave like a dislocation sources which leads to accrue the dislocations. As a result the morphology with high density of dislocations is obtained [27].

After ageing at 480 °C for 120 min the structure of CuCrZr alloys remains without any significant grain coarsening (Fig. 4). As is seen in Fig. 4a, grain boundaries become more sharp, meaning recovery occurring in the grains boundaries and their vicinity. Besides, the dislocation density is still high (Fig. 4b), indicating that dislocations are strongly pinned. A lot of Cr-rich precipitations in the grain interior and narrow annealing twins were also recognized in the structure of CuCrZr alloys after the ageing (Fig. 4c,d). Typical EDXS qualitative analysis spectrum of the chromium-rich phase from HRTEM micrograph is shown in Fig. 5 (there are visible copper spectrum peaks and chromium peaks).

4. Discussion

In the early stage of ECAP processing, the microstructure typically consists of initial grains with a high density of dislocations in their interior, which are arranged to tangled dislocations (cells) and to dense dislocation walls (DDWs). DDWs with low misorientation are classified as geometrically necessary boundaries since they participate in accommodation of lattice rotations in the adjoining volume [28]. Moreover, DDWs are in a nonequilibrium state as they contain a high density of extrinsic dislocations. Such specific micro/substructural features which are being formed in the early stage of ECAP processing can also assist in further age hardening and controlling how the precipitates would decorate the microstructure. Besides, alloying of pure metals results in significant increase of their mechanical strength controlling by microstructure, of which grain size, morphology of second phases, and their distribution as well as dislocation structure, are the most important parameters.

In this study, the ageing process at 400, 450, 480 °C for 30 min applied to the supersaturated CuCrZr alloy after one ECAP pass increased its strength and microhardness considerably. This is mainly due to the formation of the fine and coherent nanosized Cr-rich particles, which precipitate from supersaturated copper matrix [29]. The microhardness reached the peak value of 185 HV0.1 at 480 °C after 30 min, which can be attributed to the precipitation of Cr that are fully coherent with the Cu matrix and have the same fcc lattice parameter, as reported by Liu et al. [21] who suggest fully coherent Cr-rich precipitates at 480 °C after 60 min. Though, this behavior appears to be due to the enhanced kinetics of precipitation as a result of ECAP processing. During ageing, the previously formed dislocations within ECAP process interact with the stress fields around these coherent particles and thus the strength and hardness increased considerably and moreover faster as such dislocations are in a nonequilibrium state [29].

Precipitation process during ageing treatment at 400 °C in CuCrZr alloys after one ECAP pass provides the constant increase in the strength in a whole range of ageing time. Considering ageing at 450 and 480 °C, there are obvious significant differences in properties depending on ageing time. According to the [29], ageing after SPD processing has no significant effect on the grain morphology and size, and no recrystallization is evident in the UFG microstructure. However, this process leads to formation of precipitates inside the UFG microstructure, and decreases the dislocation density and elastic stresses especially at the grain/subgrain boundaries of the UFG microstructure due to the recovery processes activated during ageing. Also according to the [29], the softening (recovery) process during ageing coming from re-arrangement of dislocations in UFG CuCrZr alloy aged at 450 °C for 1 h. Gao et al. [30] claimed in binary Cu–Cr alloy that if precipitation takes place earlier than recrystallization at relatively low temperatures or prestrains, the precipitates have enough time to grow significantly and retard the rearrangement of dislocations, which in turn delays the onset of recrystallization. Also, the recrystallization would be inhibited to a great extent because of the precipitation in the deformed UFG microstructure.

5. Conclusions

The effect of ECAP followed by further ageing on the mechanical properties, microhardness and microstructure of CuCrZr alloy was investigated. The main findings can be summarized as below:

1. High strength properties and microhardness can be obtained in the alloy processed through an ECAP method followed by ageing. The most significant increase in the strength and microhardness is recognized in the first 30 min of heat treatment. Maximum microhardness of 186 HV0.1 was obtained under processing conditions at 480 °C for 30 min.

2. Microstructure of the alloy after solution annealing and ECAP processing is dramatically changed already in the first pass. Dislocation cells of different shapes with curved boundaries and low misorientation angles as well as a huge increase in the dislocation density were observed in the microstructure of CuCrZr alloys. Ageing at 480 °C for 120 min provides any significant grain coarsening. Grain boundaries become more sharp, a lot of precipitations in the grain interior and narrow annealing twins were also recognized in the structure of CuCrZr alloys.

Acknowledgments

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