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PRESSING OF PARTIALLY OXIDE-DISPERSION-STRENGTHENED COPPER USING THE ECAP PROCESS

IZTISKANJE DISPERZIJSKO UTRJENEGA BAKRA PO POSTOPKU ECAP

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A combination of internal oxidation (IO) and equal channel angular pressing (ECAP) was used to explore the possibility of uniting the mechanisms of dispersion and deformation strengthening to improve the properties of a Cu-Al alloy with 0.4 % Al. The IO of Cu-Al billets served in the first step of the experiment as a means for dispersion, strengthening the mantle of the billets with a fine dispersion of nanosized oxide particles. The experimental procedure continued with deformation strengthening performed by ECAP, which allowed an intense plastic strain through simple shear.

Material flow in a partly internally oxidized Cu-0.4 % Al billet and in a homogenous reference sample made of modelling mass was also studied to analyse, on the macroscale, the influence of the internal oxidation zone (IOZ) on the material flow behaviour during the ECAP process. The analysis was performed with the aim of revealing the uniformity of the strain distribution and to obtain information about the deformation strengthening across the volume of the billet.

We found that the oxide particles have a minor influence on the material flow on the macroscopic scale during the ECAP process. However, the degree of deformation strengthening in the IOZ was much lower than in the unoxidized core region. The combination of IO and ECAP allows us to produce a Cu composite composed of a hardened oxidized mantle region with good electrical and thermal conductivity and a high- hardened core region. This combination represents a new technological route for the production of high-hardness Cu composites, which could also be used at higher temperatures.

Keywords: ECAP process, internal oxidation, Cu-Al alloy, strengthening mechanism

Kombinacija notranje oksidacije (NO) in postopka ECAP je bila uporabljena za združitev disperzijskega in deformacijskega utrjanja za izboljšanje lastnosti zlitine Cu-Al z 0,4 % Al. Notranja oksidacija je v prvem koraku eksperimenta zagotovila disperzijsko utrjanje plašča preizkušanca s fino disperzijo nano velikih oksidnih delcev. Eksperiment se je nadaljeval z deformacijskim utrjanjem, izvedenim z ECAP-postopkom, ki je omogočil ekstremno plastično deformacijo s čistim strigom.

Preučeno je bilo tudi tečenje materiala na makronivoju v delno notranje oksidiranem Cu-0,4 % Al preizkušancu in homogenem referenčnem vzorcu iz modelirne mase z namenom analize vpliva cone notranje oksidacije na vedenje tečenja materiala med ECAP-postopkom. Analiza je bila izvedena z namenom odkriti enakomernost porazdelitve deformacije in pridobitve informacij o deformacijskem utrjanju po volumnu preizkušanca.

Ugotovili smo, da imajo oksidni delci na makronivoju zelo majhen vpliv na tečenje materiala med ECAP-postopkom, vendar je bila stopnja deformacijskega utrjanja v coni notranje oksidacije veliko nižja kot pa v neoksidiranem jedru. Kombinacija notranje oksidacije in ECAP-postopka nam omogoča izdelavo Cu-kompozita, sestavljenega iz utrjenega oksidiranega plašča z dobro električno in toplotno prevodnostjo ter visoko utrjenega jedra. Ta kombinacija je nova tehnološka pot za izdelavo visokotrdnostnega Cu-kompozita, ki bi se lahko uporabljal tudi pri višjih temperaturah.

Ključne besede: ECAP-postopek, notranja oksidacija, zlitina Cu-Al, mehanizmi utrjanja

1 INTRODUCTION

Considerable interest has been developed in making use of materials with high electrical and thermal conductivity, with microstructural stability and with high-temperature strength. These materials can be used in different segments of the electric and machine-building industry. One of the most promising materials that correspond to the above-mentioned condition is copper, because it has the highest thermal and electrical conductivity among structural materials. Copper has good physical properties but, on the other hand, requires a considerable improvement in strength to be applicable at high temperatures.

To improve its mechanical properties a different strengthening mechanism can be used, such as solid-solution hardening, work hardening, precipitation hardening and dispersion hardening.[1,2] These strengthening mechanisms give engineers the ability to tailor the mechanical properties of materials to suit a variety of different applications. In all these cases the strengthening effect is a consequence of the interactions of gliding dislocations with the barriers (solute atoms, precipitate, dispersoids), and with other dislocations.

An attractive and viable approach for improving the strength of copper is to introduce fine Al_2O_3 particles into the Cu matrix, resulting in an oxide-dispersion strengthening of alloys (ODS alloy). A copper matrix containing fine, nanosized particles is particularly attractive because of its excellent combinations of thermal and electrical conductivity and overall microstructural stability. In order to achieve the fine Al_2O_3 particles in the Cu matrix, powder metallurgy and the IO process are mostly used. However, for non-porous large billets the IO is more suitable. IO is a diffusion-controlled process involving the selective reactions of a less noble solute or second-phase particles with oxygen (also nitrogen or carbon) diffusing in from the surface.[3,4] The problem that occurs during dispersion strengthening with the IO process is to achieve a sufficient depth of the IOZ in a technologically acceptable time.[4]

In engineering practise work hardening is one of the most frequently used methods to improve the strength of metallic products. To achieve a high degree of plastic deformation different techniques have been developed, such as high-pressure torsion (HPT) and equal channel angular pressing (ECAP)[5]. Of these, ECAP is an especially attractive process because the overall billet dimensions do not change during the processing and high shear strains can be accumulated by repeated extrusion through the die.[6–8] The deformation mechanism in the ECAP process depends strongly on many processing parameters, such as the processing route, the geometry of the channel, the presence of friction and the properties of the billet material. These parameters affect the material's flow behaviour, strain distribution and, consequently, the mechanical properties of the pressed material.[9–11]

Significant progress has been made in understanding the fundamental properties and microstructures of ECAPed materials using theoretical analyses and experimental methods. These investigations are based mainly on one-phase materials such as Cu and Al.[12–14] The influence of the second phase particles, such as oxides or precipitates, on the material flow behaviour and on the mechanical properties during ECAP was rarely investigated. In fact the combination of IO and the ECAP process has not yet been studied and no information is available.

This paper describes the possibility of combining the mechanisms of dispersion and deformation strengthening to achieve better mechanical properties of the Cu matrix. The results of material flow in partially internally oxidized Cu-0.4 % Al billet were compared with the flow of a homogenous modelling mass billet to analyse, on the macroscale, the influence of IOZ on the material flow behaviour during the ECAP process.

2 EXPERIMENTAL DETAILS

For the research activities we used a partially internally oxidized Cu-0.4 % Al billet and billets made of modelling mass. Cu-0.4 % Al billets with a cross-section of 10 mm × 10 mm and 50 mm in length were made by vacuum melting, mould casting and calibration rolling. The IO procedure was performed in a mixture of equal parts of copper oxide, copper metal and Al_2O_3 powders enclosed in a glass ampoule and held at 1173 K for 72 h in a tube furnace. This procedure allowed a partial pressure of oxygen equal to the decomposition pressure of copper oxide and maintained the saturation concentration of oxygen in the surface of the billet for longer annealing times. As a result we obtained a billet with thick IOZ 1000 μm.

A simplified, yet representative material system, consisting of different collared modelling mass rings was chosen to simplify the analysis of the macroscopic material flow with homogeneous properties throughout the volume. Two types of modelling billets (MB1, MB2) were used. MB1 was made out of black and white rings in the form of cubes with the dimensions 10 mm × 10 mm × 10 mm to investigate the material flow of homogenous material during the ECAP process (**Figure 1a**). The rings were connected to each other under very low pressure to produce a 10 mm² cross-section billet. To simulate the flow of the IOZ billet MB2 was made from horizontal bands that were connected to each other to form a 10 mm² cross-section billet (**Figure 1b**). The billets were photographed before extrusion through the die and after each required number of passes.

For the deformation experiments the ECAP toll was designed. Many parameters were taken into consideration regarding the billet shape, the size and the maximum work load. The die with 90° and a square section of 10 mm × 10 mm cross-section and a billet length of 50 mm was used. The outer corner angle was 30°. The die that consisted of two blocks, which were bolted together to give a single internal channel, and the piston were made from UTOP M01 tool steel, hardened to approximately 61 HRc. A simple standard press with 60 t capacity was used. The ECAP pressings were carried out at room

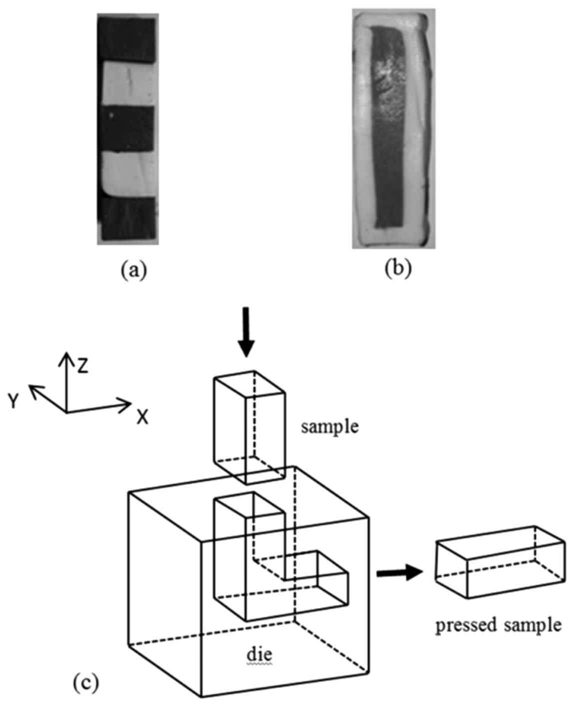

Figure 1: a) Modelling mass billets MB1 and b) MB2, c) schematic illustration of the ECAP process

Slika 1: a) Preizkušanec iz modelirne mase MB1 in b) MB2, c) shematski prikaz ECAP-postopka temperature using route A where the billet is not rotated in any direction[7]. The billet was lubricated with motor oil (OLMALINE SF/CD 20W50). A schematic illustration of the ECAP process is shown in **Figure 1c**.

The samples for the material flow investigation were taken on the *X* – *Z* plane according to the coordinate system in **Figure 1c**. The material flow was observed with light microscopy (LM). The samples for LM were prepared with standard metallographic methods and etched with $FeCl_3$. The morphology of the oxide particles (size, shape and distribution) in the IOZ after IO was examined with a scanning electron microscope (SEM) Sirion 400 NC. The metallographic preparations consisted of mechanical wet grinding down to 5 µm with SiC and polishing with an aluminium oxide suspension (0.05 µm). After the ECAP process the IOZ was examined with transmission electron microscopy (TEM) on a Jeol JEM-2100 operated at 200 kV. The samples for TEM were thinned with a Jeol EM-09100IS Ion slicer.

To determine the mechanical properties during the ECAP process we measured the microhardness in a partly internally oxidized Cu-0.4 % Al billet. Microhardness measurements were made before and after the first, second, third, and fourth ECAP passes on the *X* – *Z* plane in the IOZ and in the core region.

3 RESULTS AND DISCUSSION

3.1 Internal oxidation

After partial internal oxidation of the sample we obtained IOZ with fine dispersed oxide particles (**Figure 2a**, Area 1). The core of the sample (**Figure 2a**, Area 2) remained a one-phase Cu-0.4 % Al solid solution with slightly increased grains as a consequence of grain coarsening at higher temperatures. In contrast to this, in the IOZ precipitated oxide particles hindered the process of grain growth. The internal oxidation front (IOF) is

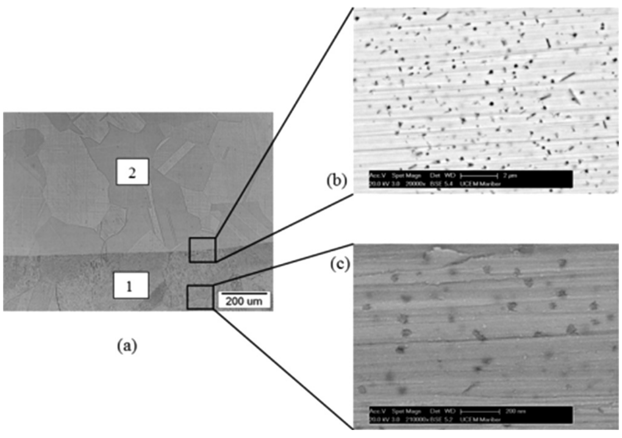

Figure 2: a) LM image of partially internally oxidized Cu-0.4 % Al sample; area 1 is IOZ, area 2 is Cu-0.4 % Al alloy, b) SEM image of the IOF, c) SEM image of the IOZ near the surface area of the billet
Slika 2: a) SM-posnetek delno notranje oksidiranega Cu-0,4 % Al-vzorca; področje 1 je cona notranje oksidacije, področje 2 je Cu-0,4 % Al-zlitina, b) SEM-posnetek fronte notranje oksidacije, c) SEM-posnetek cone notranje oksidacije blizu površine preizkušanca

straight because of the diffusion of oxygen through the volume of the grains, which prevailed against diffusion along the grain boundaries. The mean width of the IOZ was 1000 µm. It is very difficult to achieve such a depth of the IOZ because of the conglomeration of the Cu and Cu_2O powders, which hindered the supply of the oxygen to the surface of the billet. With the addition of Al_2O_3 powders we prevented the conglomeration of powders and maintained the saturation concentration of oxygen in the surface of the billet for longer annealing times.

During the process, oxygen atoms penetrate into the Cu matrix where they react with aluminium. The critical concentration of oxygen for this chemical reaction is very low because of the very high negative free energy of Al oxide formation. Afterwards the solubility product for the oxide ($K_{sp}^{Al_2O_3} = C_{Al}^{2} \cdot C_{O}^{3}$) is exceeded by the fine oxide particles precipitated from the solid solution.[15]

Figures 2b and **2c** show Al_2O_3 particles at the IOF and in the IOZ near the free surface of the billet. The particles at the IOF have different shapes, from spherical to rod-like, while the particles in the IOZ near the free surface of the billet have a spherical shape. The mean size of the particles increases with the depth of the IOZ, from 50 nm near the free surface (**Figure 2c**) to 300 nm at the IOF (**Figure 2b**). The increase in the mean size of the Al_2O_3 particles with increasing depth of the IOZ is a consequence of the hindered diffusion of oxygen to the IOF that stimulates contra diffusion of the alloying element Al down the concentration gradient towards the IOF. At lower depths of the IOZ the influence of oxide particles on the diffusion of oxygen is negligible. Moreover, with increasing depth of the IOZ the velocity of oxygen diffusion decreases because of the numerous obstacles, i.e., oxide particles. Consequently, there is more time at the IOF for growing the precipitated oxide particles.[4]

3.2 Material flow in modelling mass billets during the ECAP process

Figure 3 shows the flow of the MB1 billet during the first ECAP pass in the *X* – *Z* plane. Passing through the deformation zone (dashed lines in **Figure 3**), the horizontal straight bands evolve to inclined bands, which are at an angle with respect to the pressing direction. The angle decreases with the increasing number of passes (**Figure 4**). After the first pass the angle α was 26°, after the second 13°, after the third 10° and after the fourth 6°, which is quite comparable to the angle of inclination of the structural elements mentioned by Segal.[11]

The direction of material flow during the ECAP process was analysed with the changing position of the grey and black dots. The direction of material flow in the outer channel (OC) surface is the opposite of the pressing direction, as shown with a black dot in **Figure 4**. The black dot changes its position in the reverse direction of pressing. In contrast to this, the material flow in the internal channel (IC) surface is in the pressing direction

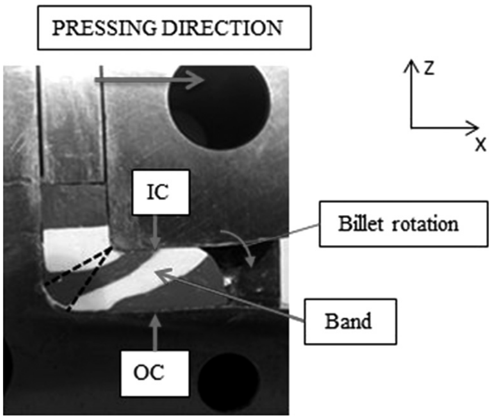

Figure 3: The MB1 billet during the ECAP process
Slika 3: Preizkušanec MB1 med ECAP-postopkom

(grey dot in **Figure 4**). These differences in the material flow from the IC to the OC surface leads to the rotation of the end mass of the billets to the OC surface, as shown in **Figure 3**.

Figure 5 shows the material flow of the MB2 billet during the first ECAP pass. The white layer simulates the flow of the mantle region of the homogenous material. In the OC surface the mean width of the white layer has narrowed and in the IC surface the mean width has increased. As the billet enters the main deformation zone (dash lines in **Figure 5**) the IC layer is subjected to compression in the pressing direction, causing an extension of the white to the centre of the billet. At the same time, the OC surface of the billet is subjected to the tension in the pressing direction, causing narrowing of the white layer with respect to its initial thickness. As a

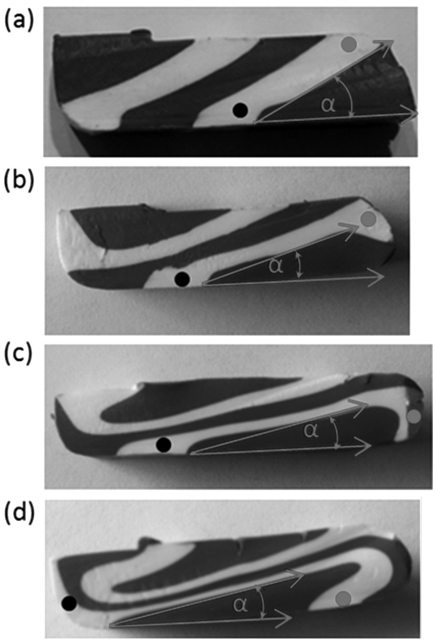

Figure 4: The MB1 billet in the *X* – *Z* plane after: a) the first, b) second, c) third and d) fourth ECAP pass
Slika 4: Preizkušanec MB1 v *X* – *Z*-ravnini po: a) prvem, b) drugem, c) tretjem in d) četrtem ECAP-hodu

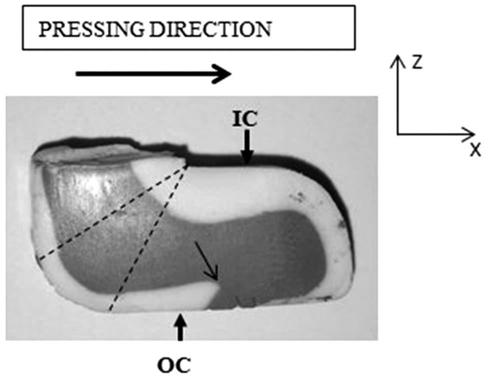

Figure 5: The MB2 billet during the ECAP process
Slika 5: Preizkušanec MB2 med ECAP-postopkom

consequence of billets end mass rotation to the OC surface, the grey layer reaches the OC surface. While the white layer was displaced by the grey layer in the OC surface, the white layer's thickness increases slightly (black arrow in **Figure 5**).

3.3 Material flow in a partly internally oxidized Cu-0.4%Al billet during the ECAP process

Figure 6 shows a partially internally oxidized Cu-0.4 % Al billet in the *X* – *Z* plane during one ECAP pass. The bright area on the edges of the billet is IOZ, which has a similar distribution in the *X* – *Z* plane as the white layer in the MB2 billet (**Figure 5**). In the OC surface the mean width of the IOZ has narrowed to 500 µm and in the IC surface the mean width has increased up to 2000 µm. The slight increase in the width of the IOZ in the OC surface (grey arrow in **Figure 6**) is a consequence of the billet's end mass rotation to the OC surface as also shown in billets MB1 and MB2. The rotation of the billets end mass causes high tension in the IC surface, which leads to the formation of cracks (black arrows in **Figure 6**).

The microstructures in **Figure 7** correspond to Areas 1 and 2 indicated in **Figure 6**. In Area 1 (**Figure 7a**) the

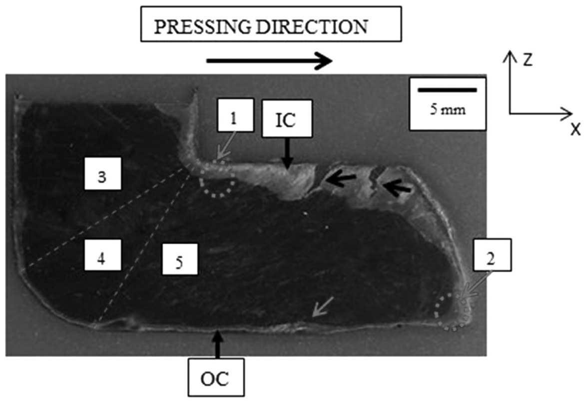

Figure 6: Partially internally oxidized Cu-0.4 % Al billet in the *X* – *Z*-plane after one ECAP pass. The black arrows indicate cracks.
Slika 6: Delno notranje oksidiran Cu-0,4 % Al preizkušanec v *X* – *Z*-ravnini po prvem ECAP-hodu. Črne puščice kažejo razpoke.

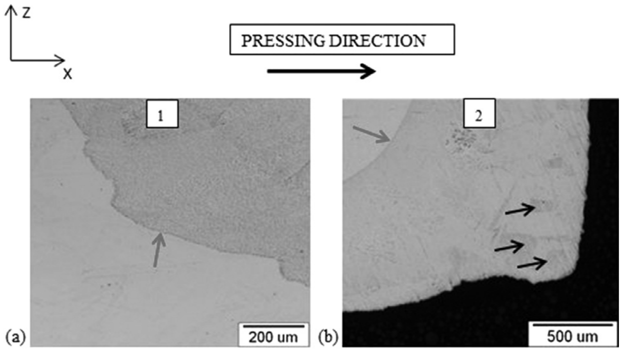

Figure 7: LM image of IOZ in: a) area 1 and b) area 2. The grey arrow indicates the IOF. Black arrows indicate undeformed grains.
Slika 7: SM-posnetek CNO: a) področje 1 in b) področje 2. Sive puščice kažejo fronto notranje oksidacije. Črne puščice kažejo nedeformirana zrna.

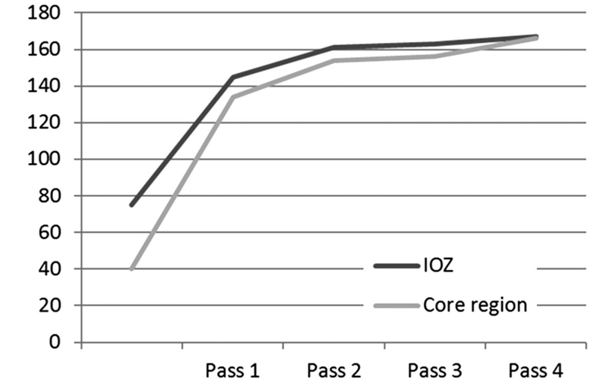

Figure 9: Microhardness of the internal oxidation zone and core region (HV5)
Slika 9: Mikrotrdota cone notranje oksidacije in jedra (HV5)

IOF is wavy, which indicates that the flow of IOZ is different than the flow of the core region. In Area 2 (**Figure 7b**) the IOF is straight and the un-deformed grains are still visible, indicating that the billet's end part was not subjected to the plastic deformation. The presence of the un-deformed end part of the billet cannot be avoided during the ECAP process because when the billet is inserted into the inlet channel the end part is not in the deformation zone (**Figure 8**).

Variations of material flow from the IC to OC surface are a consequence of a non-uniform strain distribution throughout the cross-section of the billet[11]. The strain distribution in the billet depends on the friction between the channel walls and the billet, die geometry, and properties of the billet material.[9] According to Balasundar et al.[10] the strain distribution in the billet depends on the corner angle in the ECAP die. At lower corner angles (< 40°) the differences in material flow from the IC to OC surfaces leads to the rotation of the end mass of the billet to the OC surface. This rotation was observed in billets MB1, MB2 and in the partially internally oxidized billet.

Prangnell et al.[16] have reported that inhomogeneous deformation increases with friction. As the friction between the billet and the channel walls increases the billet has a tendency to stick to the OC surface. As a consequence the strain at the OC increases and the strain distribution becomes highly inhomogeneous.

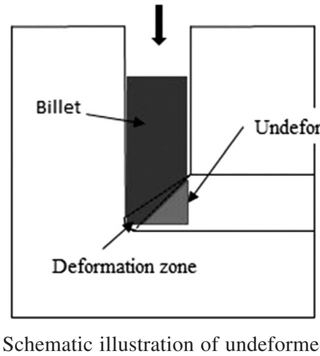

Figure 8: Schematic illustration of undeformed end part of the billet
Slika 8: Shematski prikaz nedeformiranega konca preizkušanca

Numerical analyses have shown that the stress-strain response of the billet material has an influence on the strain distribution during the ECAP process.[10] The modelling mass billets have a flow-softening response, while partially internally oxidized billets have a strain hardening response. The materials with the strain hardening type are subjected to greater strain at the IC than in the OC surface.[17] This can be attributed to the formation of cracks in the partly internally oxidized billet.

On the macroscopic scale the flow of the internally oxidized coat region and the core with the solid solution is similar to the material flow in the billets MB1 and MB2, where the material properties were the same in the mantle and in the core region. The results indicate that oxide particles have a minor influence on the material flow in the IOZ. The differences could be attributed to the different strain distribution in the IC surface as a consequence of the strain-hardening rate of the IOZ.

3.4 Microhardness measurements

The microhardness has been measured in the IOZ and in the core region. Measurements were taken in the centre of the billet where the deformation is the most homogeneous in the IC, OC surface and in the core region.

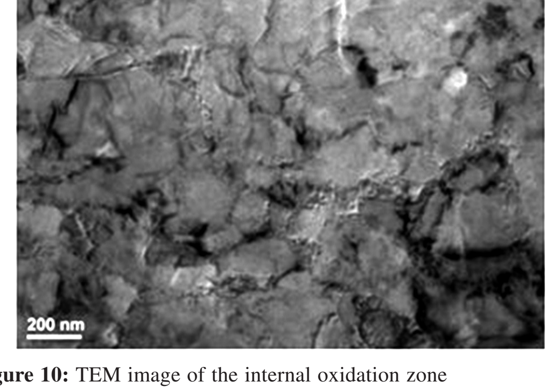

Figure 10: TEM image of the internal oxidation zone
Slika 10: TEM-posnetek cone notranje oksidacije

The results are presented in **Figure 9**. It would be noted that because of the interaction between the Al_2O_3 particles and the sliding dislocations after four passes through the die the ΔHV would be larger in the IOZ. Due to an inhomogeneous strain distribution in the ECAP die the IOZ was subjected to higher strain in the IC surface, which could also contribute to a higher ΔHV in the IOZ. After four passes through the ECAP die the hardness of the IOZ and the core region are the same, which could be attributed to different deformation mechanisms.

During the ECAP process, strong dislocation activities subdivide the original coarse grain into smaller grains[17]. The strength and hardness of the material increases with the decrease of the grain size, as determined by the Hall-Petch equation:

$$\sigma_y = \sigma_0 + \frac{k_y}{\sqrt{d}}$$

where d is the average size of the grain, σ_y is the yield stress, σ_0 is the starting stress and k_y is the strengthening coefficient.

Meyers[18] has analysed the deformation mechanisms in nanostructured materials where grain-boundary sliding has been proposed to be the dominant deformation mechanism at grain sizes < 50 nm. The grain-boundary sliding is a mechanism where the layer of grains slides with respect to the other. This mechanism can lead to a decrease in strength and hardness of the material. Although we have in the IOZ (**Figure 10**) a grain size of approximately 150 nm this effect is present because the Al_2O_3 particles have blocked the dislocation motion. Consequently, from an energy point of view, the sliding was preferable along the grain boundaries. Due to this, deformation strengthening was no longer present in the IOZ.

4 CONCLUSIONS

In our research a combination of IO and ECAP was used to explore the possibility of uniting the mechanisms of dispersion and deformation strengthening to improve the properties of a Cu-Al alloy with 0.4 % Al. Material flow in a partly internally oxidized Cu-0.4 % Al billet and in a homogenous reference sample made of modelling mass was studied to analyse on the macroscale the influence of IOZ on the material-flow behaviour during the ECAP process.

From the obtained results and their analysis several conclusions have been made:

- On the macroscopic scale the material flow in the partly internally oxidized Cu-0.4 % Al billet, which has different material properties in the core and in the mantle region, is the same or similar to that in the homogenous material, where the properties of the mantle region are the same as in the core region. This indicates that the oxide particles have a minor influence on the material flow during the ECAP process.
- Non-uniform strain distribution during the ECAP process leads to an uneven thickness of the internal oxidation zone throughout the mantle region of the billet.
- The combination of IO and ECAP allows us to produce a Cu composite composed of a hardened oxidized mantle region with good electrical and thermal conductivity and a high-hardened core region.
- The combination of dispersion and a deformation strengthening mechanism represents a new technological route for the production of a high-hardness composite that could also be used at higher temperatures, but it is necessary to investigate the solution to achieve an even thickness of the oxidation zone throughout the mantle region of the billet. A possible solution would be the application of back pressure during the ECAP process.
- In an upcoming publication a detailed analysis will be made of the deformation mechanisms in the Cu composite.

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