NANOCRYSTALLINE Cu - 5 VOL.% \(\gamma\)-Al\(_2\)O\(_3\) MATERIAL

J. Řurišin, M. Orolínová, K. Řurišinová

Abstract
The first part of the paper is aimed toward the preparing of nanocrystalline Cu – \(\gamma\)-Al\(_2\)O\(_3\) powder mixture with 5 vol.% of the secondary phase (dispersoid) by a method based on a combination of mechanical milling and the phase transformations of the precursors. The \(\gamma\)-Al\(_2\)O\(_3\) secondary particles were prepared by phase transformation in situ within CuO powder. The nanometric Cu matrix was obtained by chemical reduction of CuO precursor. The influence of various technological steps on Cu powder matrix microstructure was estimated. The second part of the study maps the microstructural characteristics of Cu – 5 vol.% \(\gamma\)-% Al\(_2\)O\(_3\) compact composite. The effect of secondary phase on the preservation of the initial powder nanostructure after consolidation into macroscopic compact and thermal structural stability, as well as tensile properties, is analyzed. The results indicate that alumina dispersoid is suitable for nanostructure stabilization of the Cu matrix.

Keywords: powder technology, nanomaterials, copper composite, dispersoid, microstructure stability, mechanical properties

INTRODUCTION
Nanostructural materials are progressive materials defined as polycrystals with a grain size \(\leq\) 100 nm and they have been the subject of intensive investigation in recent years. They are noted for different and often considerably better properties compared with conventional coarse-grained polycrystalline materials. A change of properties of nanocrystalline materials is caused as a consequence of a significant grain refinement and new characteristics of grain boundaries [1-4].

The development of nanocrystalline microstructure creation principles has emerged as an important step in synthesizing the new materials [5]. In principle, any method capable of producing very fine grain-sized polycrystalline materials can be utilized to produce nanocrystalline materials. However, nanostructure crystallites have generally been prepared by modified gas-condensation methods, vapour deposition or by electrodeposition. Other potential methods for preparing these materials include mechanical milling, mechanical alloying, molecular beam epitaxy, rapid solidification from the liquid state, ion beam, reactive sputtering, sol-gel, and chemical vapour deposition processes [6]. High-energy milling is increasingly studied as a method of preparing nanophase materials due to its potential for industrial scale production. The grain size, morphology and texture can be varied by suitably modifying/controlling the process variables in each of these methods.

Due to a great volume portion of grain boundaries, a concentration of free energy takes place in nanocrystalline materials. These materials are characterised by high

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thermodynamic instability. For example, significant grain growth was even observed at the ambient temperature and at lower temperatures in single phase nanocrystalline materials whose melting points ($T_m$) were below 600°C. Grain growth was retarded for higher $T_m$ metals, e.g. for Cu up to 100°C, for Pd up to 250°C. Just as in conventional polycrystals, grain growth in nanocrystalline materials may be inhibited by second-phase particles. The presence of secondary phases in the matrix inhibits the grain boundary migration and it thus stabilizes the structure up to temperatures close to the melting point of the matrix. Chemical stability in relation to the matrix, fineness and distribution homogeneity in the matrix are the most important demands on the secondary phases [7-9].

Intensive industrial development places high demands on structural parts. The working parameters of a lot of them are so high that classic materials are not suitable. The objective of our work was by the PM way to develop nanocrystalline Cu base material for electrotechnical applications with high strength characteristics at room and elevated temperatures, as well as good electrical conductivity. The nature of a suitable system shows the Cu – 5 vol.% Al$_2$O$_3$ material [9].

RESULTS AND DISCUSSION

The Cu - 5 vol.% $\gamma$-Al$_2$O$_3$ powder mixture preparation

The properties of powders determine the properties of final compact materials. Hence prediction and regulation of powder microstructures evolution is important. Our partial goal was to prepare the nanocrystalline powder mixtures with high temperature stability of the matrix microstructure. The crystallite size of the CuO, eventually Cu matrix was evaluated on the basis of X-ray measurements. X-ray diffraction analysis of the powder mixtures was carried out on the diffractometer with Bragg-Brentan parafocusing arrangement with a 300 mm diameter focusing circle, using Cu K$\alpha$ radiation, voltage of 30 kV, current of 20 mA, Ni filter and the data gathering rate of 0.5°/20 min.

The nanocrystalline dispersion strengthened Cu - 5 vol.% $\gamma$-Al$_2$O$_3$ powder was prepared by modification of the original mechanical-chemical preparation of nanocrystalline Cu powder [10], which is based on the combination of high-energy milling of powders (attritor Netzsch, 400 rev./min., steel balls with the diameter of 2 mm, milled material/balls weight ratio of 1:5) and phase transformations during preparation process. This method has the advantage of producing large quantities of materials. In addition, it does not require extensive processing equipment and the cost of production can be relatively low.

The initial raw material was prehomogenized CuO powder with the corresponding amount of aqueous solution Al(NO$_3$)$_3$ $\cdot$ 9 H$_2$O and by the addition of ammonia. Amorphous aluminium hydroxide gel precipitates were formed by the action of ammonia on the aqueous solution of aluminium salt. CuO powder was obtained by annealing of electrolytic Cu powder with a particle size below 40 µm in air at the temperature of 800°C and subsequent dry milling in the attritor for 2 h. This initial mixture was used with an aim of attaining Cu matrix particles and the dispersoid particles as fine as possible, and at the same time, the homogeneous distribution of dispersoids. The role of the aqueous solution of Al(NO$_3$)$_3$ $\cdot$ 9 H$_2$O was to cover the surfaces of the CuO particles and to penetrate into all the cracks in the brittle CuO particles which were formed during the preceding CuO milling process.

The gel changed into $\gamma$-Al$_2$O$_3$ phase during the heating at 235°C and transformed into $\alpha$-Al$_2$O$_3$ at temperatures over 1000°C [11]. The initial mixture was annealed at the temperature of 400°C for 1 h. The ramp up of temperature to 400°C was 2 h to allow a gradual elimination of free and bound water. In the annealing process, the secondary phase, $\gamma$-Al$_2$O$_3$, was formed from aluminium nitrate directly in the CuO matrix, Fig.1.
Fig. 1. The infrared spectra of CuO+NH₃ - curve a, CuO+Al(NO₃)₃·9H₂O+NH₃ - curve b, CuO+Al(NO₃)₃·9H₂O+NH₃ after annealing at 400°C/air – curve c, γ-Al₂O₃ - curve d.

The crystallite size of the CuO matrix was evaluated on the basis of X-ray measurements in the range of 2θ ~ 20°- 80°, Fig.2, using the Scherrer method [12], from reflections of (110) and (202) planes, because the intensiver reflections of (111), plane (100% intensity) coincides with the reflection of (002) plane (49% intensity) and the other intensiver reflection of (111) plane (96% intensity) coincides with the reflection of (200) plane (30% intensity) and the separation is usually complicated. After annealing at the temperature of 400°C for 1 h, the CuO crystallites are nanometric, Tab.1.

The formed CuO - 5 vol.% Al₂O₃ mixture was then dry-milled in the attritor for 3 h with the aim further to refine the particles and the microstructure of the matrix, and to eliminate possible agglomerates of Al₂O₃ particles. This process also improves the distribution homogeneity of the Al₂O₃ secondary phase. CuO crystallites were refined by milling, which is also evidenced by a broadening of the diffraction lines compared with the as-annealed state, Fig.2. The average CuO crystallite size after milling is 23 nm, Tab.1.

Fig.2. The X-ray diffraction patterns of CuO - γ-Al₂O₃ (5 vol.% calculated on Cu) powder mixtures after annealing and after milling.
Tab.1. The linear dimensions of CuO crystallites D in the direction perpendicular to individual crystallographic planes.

<table>
<thead>
<tr>
<th>POWDER MIXTURE CuO–γ-Al₂O₃ (5vol.% calculated on Cu)</th>
<th>hkl</th>
<th>(110)</th>
<th>(020)</th>
</tr>
</thead>
<tbody>
<tr>
<td>After annealing</td>
<td>D [nm]</td>
<td>63</td>
<td>34</td>
</tr>
<tr>
<td>After milling</td>
<td>D [nm]</td>
<td>22</td>
<td>22</td>
</tr>
</tbody>
</table>

Subsequent chemical reduction of CuO into Cu took place. CuO → Cu transformation was performed by chemical reduction of CuO powder in static conditions in a furnace at 150°C during 2 h - static reduction. The condition for the fine Cu matrix formation is the chemical reduction of nanometric CuO particles. X-ray measurements of the Cu - 5 vol.% Al₂O₃ powder mixtures were carried out in the ranges of 2θ ~ 42°-76°. The crystallite size of the as-reduced Cu was evaluated on the first three reflection angles from (111), (200) and (220) planes, Fig.3.

Fig.3. The X-ray diffraction patterns of Cu - 5 vol.% γ-Al₂O₃ powder mixtures after reduction and after milling.

Cu particles have the nanometric structure and linear dimensions of the crystallites in the individual crystallographic direction range from 37 to 55 nm, Tab.2. Since Cu particles retained their nanocrystalline structure even at relatively high temperatures during reduction (the reduction temperature is increased due to the heat release from the strongly exothermic reaction CuO + H₂ → Cu + H₂O, ΔHR = - 86.6 kJmol⁻¹), it can be stated that fine and uniformly distributed Al₂O₃ particles inhibited the interface migration.

Tab.2. The linear dimensions of Cu crystallites D in the direction perpendicular to individual crystallographic planes.

<table>
<thead>
<tr>
<th>POWDER MIXTURE Cu - 5vol.% γ-Al₂O₃</th>
<th>hkl</th>
<th>(111)</th>
<th>(200)</th>
<th>(220)</th>
</tr>
</thead>
<tbody>
<tr>
<td>After reduction</td>
<td>D [nm]</td>
<td>37</td>
<td>38</td>
<td>55</td>
</tr>
<tr>
<td>After milling 4.5 h</td>
<td>D [nm]</td>
<td>20</td>
<td>16</td>
<td>18</td>
</tr>
</tbody>
</table>

The final step in preparation of powder mixture by static reduction is wet milling in an attritor after reduction. The purpose of this process was to achieve uniform dispersion.
of the secondary phase, because $\gamma$-$\text{Al}_2\text{O}_3$ particles prepared by several methods have a high tendency to have a formation of clusters, which is also confirmed in literature [13]. The high energy milling of Cu – $\gamma$-$\text{Al}_2\text{O}_3$ powder mixtures is important, in terms of an additional refinement of the Cu matrix powder by repeated particle welding and fragmentation [14] and mainly by the generation of dislocations. During milling, the powder particles are cold-worked resulting in a multiplication of dislocations. Eventually the dislocations interact, form dislocation tangles, and congeal into cell walls and produce nano-sized grains [15,16]. With grain size decreasing, the dislocations migrate or are forced to the grain boundary leaving the nano-grain interior essentially defects free. The resulting milled grains are highly strained, the boundaries have a disordered structure and the resulting particles contain numerous defects [17].

The crystallite size cannot be reduced without limits by mechanical milling. This is explained by considering the Hall-Petch relation. For a small grain size, a very high stress would by required to maintain plastic deformation via dislocation motion. Therefore, it must be assumed that further deformation occurs by a grain boundary slip mechanism which does not refine the microstructure any more. The pileups cannot form in materials with a grain size $d < l_c$ (where $l_c$ is the dislocation spacing in the pileup). For Cu it is $l_c = 19.3$ nm [7]. The crystallite size after 4.5 h milling is about 18 nm. It is clear, that in our case the final nanocrystalline structure of Cu particles was achieved under the given conditions after 4.5 h of milling.

An unpleasant fact, which has to be expected during mechanical milling, is usually contamination of the powder mixture from the milling tools (vial and balls) [18]. E.g. after 4.5 h of wet milling the powder mixture in an attritor, the presence of 0.056 wt.% of Fe was confirmed by chemical analysis. This fouling negatively manifests itself at the following compacting of powder, and negatively influences electric conductivity of resultant Cu compact material.

THE Cu - 5 vol.% $\gamma$-$\text{Al}_2\text{O}_3$ COMPACT MATERIAL

Compaction

One of the main problems in ultrafine powders processing is consolidation with full densification without sacrificing their nanocrystalline structure. The metastable nature of the nanostructure dictates that the use of high temperature densification techniques is limited. For this reason, improvement of the thermal stability of milled powders against grain growth is of paramount importance in the development of an economical production of bulk nanocrystalline materials [19].

Ultra-fine powders show quick oxidation due to their great specific surface, which results in reduced compressibility [20]. Before compacting, the powder mixture was therefore pre-reduced at 200°C for 10 min. To suppress the adsorption of oxygen and humidity from the atmosphere, the powder mixture was cold pressed in hydrogen. In materials with nanometric grain sizes, compacting processes based on diffusion run more quickly and at lower temperatures than in conventional powder [21]. The compact was therefore sintered only for a short time (10-min.) at 800°C in H$_2$ stream. It was then densified by extrusion at 800°C with 95% deformation recalculated to the section.
The microstructure

Light microscopy

The microstructure observation of ultrafine materials by light microscopy is limited by equipment resolution. The microstructure of the compacted material was observed on etched specimens via light microscopy. The microstructure of material in perpendicular direction to the compacting pressure, as well as in the compacting pressure direction, is characterized by equiaxed formations with a mean size of 500 nm, Fig.4a,b, which corresponds with the size of the original powder particles, Fig.5.

Fig.4. Light microscopy micrograph of the as-extruded Cu - 5 vol.% Al₂O₃ material: a) in cross machine direction; b) in machine direction; c) after annealing 800°C/1 h, in cross machine direction

Fig.5. SEM micrograph of the Cu - 5 vol.% Al₂O₃ powder.
In the microstructure of material in the extrusion direction, there are isolated formations of copper without dispersoid, which are elongated in the compacting direction. Despite extrusion with 95% deformation, the expected row microstructure was significantly suppressed by the presence of very fine and uniformly distributed secondary phase particles. The material was subjected to the temperature structure stability test by annealing in H$_2$ at 800°C for 1h. The microstructure of as-annealed material was unchanged when compared with as-extruded material, Fig.4 c.

The resolution of light microscopy is insufficient for ultra-fine materials, therefore the material microstructure was also observed via scanning and transmission electron microscopy, using the thin foil, replica and electron diffraction methods.

**Scanning electron microscopy (SEM)**

By observing polished and etched specimens using the scanning electron microscope, a high porosity of the samples was observed, Fig.6.

![SEM micrograph of the as-extruded Cu - 5 vol.% Al$_2$O$_3$ material.](image)

The pores are very fine, their size ranges up to 100 nm and they occur at the boundaries of the original powder particles. Pores with a size up to 500 nm occur in the microstructure only sporadically. The high porosity of the samples can be due to the fact that insufficient contacts between the powder particles were formed during cold pressing and the pores were not closed during sintering. This can be due to several factors. Conventional consolidation of attritor milled powders to form dense nanocrystalline solids has been found to be difficult, however primarily due to the hardness of the matrix powder particles [22]. Another reason is the above-mentioned quick adsorption of gases from the atmosphere due to the extremely great specific surface of fine powders. As a result, copper oxides can form, and in the oxide-oxide contact points (cold joints) no sintering takes place, or gases from the atmosphere are closed in closed pores.

**Transmission electron microscopy (TEM)**

The TEM photograph of thin foil, Fig.7a, documents the microstructure characterized by equiaxed, polygonal grains with the mean size of 150 nm and the uniform distribution of the dispersoid particles in the matrix. Inside the grains, dislocations are anchored on the secondary phase particles and create a dislocation network. The points on
the electron diffraction pattern of a selected foil area, Fig.7b where connected to circles that suggest the microstructure fineness. The electron diffraction pattern confirms the presence of $\gamma$-$\text{Al}_2\text{O}_3$ particles in the Cu matrix.

Fig.7. TEM micrograph of the as-extruded Cu - 5 vol.% Al$_2$O$_3$ material (a) and selected area diffraction pattern (b).

Tab.3. The measured values of electron diffraction circle diameters (D), the calculated interplanar spacing values (d), the interplanar spacing values for Cu and $\gamma$-$\text{Al}_2\text{O}_3$ (d) and their intensities (I) given in JCPDS Catalogue.

<table>
<thead>
<tr>
<th>Measured values</th>
<th>Cu (PDF 04-0836)</th>
<th>$\gamma$Al$_2$O$_3$ (PDF 47-1308)</th>
</tr>
</thead>
<tbody>
<tr>
<td>D [nm]</td>
<td>d [nm]</td>
<td>I [%]</td>
</tr>
<tr>
<td>12.8</td>
<td>0.239</td>
<td>-</td>
</tr>
<tr>
<td>14.5</td>
<td>0.208</td>
<td>0.209</td>
</tr>
<tr>
<td>15.3</td>
<td>0.200</td>
<td>-</td>
</tr>
<tr>
<td>17.1</td>
<td>0.179</td>
<td>0.181</td>
</tr>
<tr>
<td>22.2</td>
<td>0.138</td>
<td>-</td>
</tr>
<tr>
<td>24.3</td>
<td>0.127</td>
<td>0.128</td>
</tr>
<tr>
<td>26.3</td>
<td>0.116</td>
<td>-</td>
</tr>
<tr>
<td>28.4</td>
<td>0.108</td>
<td>0.109</td>
</tr>
<tr>
<td></td>
<td></td>
<td>0.104</td>
</tr>
</tbody>
</table>

Table 3 gives the measured values of circle diameters of individual electron diffraction patterns (D), the interplanar spacing values calculated from the circles (d) and compared with the interplanar spacing values for Cu and $\gamma$-$\text{Al}_2\text{O}_3$ given in the JCPDS (Joint Committee on Powder Diffraction Standards) Catalogue. The mean size of $\gamma$-$\text{Al}_2\text{O}_3$ particles determined using direct statistical measurements on photographs of two-stage carbon replicas is 15 nm, Fig.8a. The electron diffraction pattern of a replica, Fig.8b,
confirmed the presence of $\gamma$-Al$_2$O$_3$ particles only. The diffusion circles of the electron diffraction pattern confirm the presence of the very fine secondary particles.

![TEM micrograph of the Al$_2$O$_3$ nanoparticles obtained from the as-extruded Cu - 5 vol.% Al$_2$O$_3$ material (a) and selected area diffraction pattern (b).](image)

**X-ray diffraction**

Figure 9 documents the comparison of the course of the diffraction lines of the powder coming up to compaction and the compacted material in the extrusion direction. It is evident from Fig.9 and Tab.4 that the intensities of the individual reflections of the compact material do not correspond with the intensities given in the JCPDS Catalogue for Cu. The reflection from the slip (111) plane was suppressed and the reflections from the (200) plane and the (220) plane were significantly increased. Cu has the face centered cubic (fcc) lattice K12, and in metals with fcc lattice the texture [110] create during extrusion. The deformation texture can result in the anisotropy of properties, which is undesirable in our case.

![The comparison of the X - ray diffraction pattern of powder and as-extruded Cu - 5 vol.% Al$_2$O$_3$ material (in maschine direction).](image)
Tab.4. The comparison of the Cu relative intensities given in JCPDS Catalogue with the relative intensities of the experimental Cu- 5 vol.% Al₂O₃ compact material in the extrusion direction.

<table>
<thead>
<tr>
<th></th>
<th>(111)</th>
<th>(200)</th>
<th>(220)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cu (PDF 04-0836)</td>
<td>100</td>
<td>46</td>
<td>20</td>
</tr>
<tr>
<td>Cu - 5 vol.% Al₂O₃</td>
<td>77</td>
<td>100</td>
<td>100</td>
</tr>
</tbody>
</table>

From the X-ray diffraction records, the size of crystallites was evaluated on the first three (111), (200) and (220) reflection planes using the Scherrer method [12]. By comparing the diffraction line profiles in Fig.9, a narrowing of the compact reflections is evident when compared with the reflections of the powder, which gives evidence of the growth of the crystallites of the compact material. However, the structure of Cu - 5 vol.% Al₂O₃ material kept its nanocrystalline character even during the compacting process, where the material was exposed to a high temperature for a certain period, because the mean Cu crystallite size grew from the original 18 nm in the powder to 45 nm in the compact. It can be assumed that the fine and uniformly distributed γ-Al₂O₃ secondary phase particles efficiently suppressed the interface migration in the individual stages of the compacting process.

The mean Cu crystallite size in the experimental material after annealing at 800°C for 1 h in hydrogen remained unchanged when compared with the as-extruded state (45 nm), which is also confirmed by a comparison of the course of the diffraction lines from (111) plane, Fig.10. We assume that during the compacting process the Cu crystallite size increased from the original 18 nm (in the initial powder mixture) to 46 nm within a very short time. Further elongation of the annealing period at the given temperature has no influence on the coarsening of the structure.

The tensile properties

The mechanical properties of polycrystalline metals and alloys are very sensitive to their grain size. Refining its grain size, as draw from the commonly known Hall-Petch relationship between yield strength/hardness and grain size, can effectively increase the
material strength. In nanocrystalline materials the Hall-Petch relation holds up under the condition that grain size $d_c$ is over a critical value for dislocations to form pileups. When the grain size is below $l_c$ ($l_c$ is the dislocation spacing in the pileup), weakening mechanisms, e. g., viscous type flow, set in and lead to a decrease in hardness with decreasing grain size. For nanocrystalline copper $d_c$ is the value in the 8 to 16 nm range ($l_c = 19.3$ nm) [7,23]. When grain sizes are below these values, the dislocation pileup mechanism breaks down.

Tab.5. Comparison of the ultrafine copper composite and coarse-grained pure copper properties.

<table>
<thead>
<tr>
<th>Material</th>
<th>Yield strength [MPa]</th>
<th>Ultimate tensile strength [MPa]</th>
<th>Ductility [%]</th>
<th>Brinnel hardness HB</th>
<th>Electrical conductivity [% IACS]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cu - 5 vol.% Al$_2$O$_3$</td>
<td>517</td>
<td>574</td>
<td>3.5</td>
<td>158</td>
<td>52</td>
</tr>
<tr>
<td>Cu</td>
<td>100</td>
<td>135</td>
<td>22</td>
<td>62</td>
<td>100</td>
</tr>
</tbody>
</table>

Table 5 gives an overview of the room temperature tensile test results, hardness and electric conductivity of Cu - Al$_2$O$_3$ in contrast to the pure, coarse-grained Cu compacted by the same method as the composite sample. The 0.2% offset yield strength for Cu - Al$_2$O$_3$ is about 5.17 times, the tensile strength about 4.2 times and hardness about 2.5 times higher than pure Cu. As expected from an extrapolation of the Hall-Petch relationship, the Cu - Al$_2$O$_3$ nanocomposite shows appreciable strength properties due to stabilization of the very fine copper grains by Al$_2$O$_3$ during densification. The strength characteristics of the compact material after annealing 800°C/1h/H$_2$ are practically unchanged (yield strength = 513 MPa, tensile strength = 560 MPa).

The elongation of Cu - Al$_2$O$_3$ is low, only 3.5% what is the usual value for nanomaterials with homogeneous structure. The dislocation-based deformation mechanisms, which are likely to be active above 100 nm, may not operate at smaller grain sizes where the failure seemed to be flaw dominated [24]. The presence of small processing flaws of a critical dimension (micropore or crack) could lead to brittle failure with a little deformation.

The electric conductivity of the material is reduced; however, this value is still satisfactory for electrotechnical applications, Tab.5.

Tab.7. Strength properties of the ultrafine copper composite and coarse-grained copper at different temperatures.

<table>
<thead>
<tr>
<th>Material</th>
<th>Yield stress [MPa]</th>
<th>Ultimate tensile strength [MPa]</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Temperature (°C)</td>
<td></td>
</tr>
<tr>
<td>Cu - 5 vol.% Al$_2$O$_3$</td>
<td>517</td>
<td>286</td>
</tr>
<tr>
<td>Cu</td>
<td>100</td>
<td>-</td>
</tr>
</tbody>
</table>

Table 6 documents yield stress and tensile strength of the copper composite and pure copper at different temperatures. At 400°C the ultrafine Cu – Al$_2$O$_3$ yield stress and strength values are still higher than the values of coarse-grained Cu at room temperature, which is in accordance with the suppression of recrystallization and grain growth in this material at elevated temperatures.
CONCLUSIONS

By modifying the mechanical–chemical preparation method of copper nanocrystalline powder, a nanocrystalline dispersion-strengthened mixture of Cu-Al₂O₃ can be prepared from the CuO and Al(NO₃)₃·9H₂O precursors by applying phase transformations in combination with mechanical milling. The particles of the secondary phase, formed by the in situ reaction directly in the matrix, efficiently inhibit the grain boundary migration and maintain the nanocrystalline structure of the matrix particles in the individual stages of preparation.

By compacting (cold pressing, short-term sintering at 800°C and subsequent extrusion) the nanocrystalline Cu – 5 vol.% Al₂O₃ powder a compact body can be obtained with the mean grain size of 150 nm, a mean crystallite size of 45 nm and the thermal structural stability up to 0.74 Tₘₐ₇. The γ-Al₂O₃ secondary phase particles with the mean size of 15 nm uniformly distributed at the grain boundaries and inside the grains efficiently hinder the interface migration, and make it possible to maintain the nanocrystalline structure during the compacting process, they provide an excellent temperature stability of the compact microstructure as well.

We assume that the high-density Cu – Al₂O₃ composite with very good strength properties and hardness, good plasticity and electric conductivity could be prepared by the process improvements. Improvements to the synthesis and compaction process have led to reductions in both the porosity and impurity levels in the resultant ultrafine composite, which should facilitate the measurement of the intrinsic mechanical behaviour in this material.

Acknowledgement

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