EFFECT OF OXIDE DISPERSION ON ELECTRIC CONDUCTIVITY OF ROTARY SWAGED POWDER-BASED COPPER COMPOSITES

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Abstract

Copper is a very popular electro-conductive material, however, the mechanical properties of pure Cu are low. They can be typically improved by (micro)alloying, or via structure modifications introduced by optimized deformation and thermomechanical treatments. Designing a Cu-based composite, possibly strengthened by a dispersion of fine oxides, is another way how to favourably improve the strength properties of Cu. In this study, we performed mechanical alloying of a Cu powder with a powder of Al_2O_3 oxide, which is known to have strengthening effects on metallic materials. After mixing, we sealed the powder mixture into evacuated tubular Cu containers (i.e. cans). As for the consolidation procedure, we applied direct consolidation of the canned powders via the intensive plastic deformation method of rotary swaging, performed under warm conditions. Subsequently, we subjected the swaged conductors to measurements of electric conductivity and detailed structure observations. The results revealed that the applied swaging ratio was sufficient to fully consolidate the canned powders as the final conductor was unrecognizable from a cast alloy from the viewpoints of visual and structure assessment. In other words, the structure did not exhibit any voids or remnants of unconsolidated powder particles. The observed fine grains with homogeneous dispersion of Al_2O_3 oxide particles provided improvement of the mechanical properties, as proven by microhardness measurements. Moreover, the electric properties remained favourable.

Keywords: Copper, oxide dispersion, electric conductivity, rotary swaging, microstructure

1. INTRODUCTION

The majority of contemporary produced metallic materials is prepared by bulk casting, or by powder metallurgy techniques (e.g. mechanical alloying, sintering, additive manufacturing, etc.). Besides providing the material with enhanced properties due to the possibility to achieve fine grain size, the primary advantage of powder metallurgy is that powders of materials with (way) different properties, e.g. different melting temperatures can be mixed together without the need to adapt the production technology [1]. To (further) enhance the structures of the prepared workpieces, powder-based techniques can be combined with deformation processing [2].

Both conventional and unconventional methods of plastic deformation can be used to improve the properties of the processed material, but also to (if required) modify the geometry of the workpiece. The conventional processes of plastic deformation involve forging, rolling, drawing, or extrusion (e.g. [3,4]), whereas the unconventional methods involve the severe plastic deformation (SPD) methods (e.g. [5–9]), or rotary swaging [10]. Rotary swaging is an intensive plastic deformation method, which can favourably be used for mutual enhancement of the properties of the processed material, and imparting a desired geometry to the product. Swaging can be carried out under various temperature conditions, from hot to cryogenic, and can be also used in combination with another method (e.g. SPD). Due to its incremental character and favourable stress state, swaging is suitable also for processing of challenging materials, including composites, gradient materials, materials with low plasticity, pseudo-alloys, and oxide-dispersion-strengthened (ODS) materials (e.g. [11–14]). It can also be applied to (post-)process powder-based materials to improve the density, reduce/eliminate porosity, and increase the properties [2].

Cu features excellent electric conductivity, which is, however, purity-dependent [15]. Cu prepared by electrolytic refining typically features the highest possible purity, and also the highest achievable electric conductivity. On the other hand, (commercially) pure Cu features very low strength. The mechanical properties of Cu can be improved by alloying, which can typically provide hardening/strengthening via precipitates and interstitial atoms (e.g. [16,17]). On the other hand, the fact that alloying elements typically also deteriorate the electric conductivity is known. Nevertheless, the action of a dispersion of very fine oxides within consolidated powder-based Cu material has not been widely studied yet, and is definitely worth investigating.

The aim of the presented study was to characterize the structure of a directly consolidated ODS Cu-based material. The composite was prepared by direct consolidation of a mixture of Cu and Al_2O_3 powders via rotary swaging. The final effect of the oxidic dispersion on mechanical, as well as electric properties, was also studied.

2. EXPERIMENT

As for the original material, cans with the initial diameter of 25 mm were prepared as follows: Cu powder (**Figure 1a**) was mechanically alloyed (MA) for two hours with the addition of 5 wt.% of Al_2O_3 powder particles (**Figure 1b**). After MA, the powders were vacuum-sealed into Cu tubes, by which powder-containers, i.e. cans, for subsequent deformation processing were created. The prepared cans were pre-heated to the temperature of 600 °C and directly consolidated via rotary swaging, which was performed in three subsequent passes to the final diameter of 10 mm. The final reduction ratio, calculated using formula (1), the S_0 and S_n in which were cross-sectional areas of the consolidated composite at input and output of dies, respectively, was 1.4. Afterwards, samples cut cross-sectionally from the consolidated bar were subjected to analyses of microstructure, as well as measurements of electric conductivity and microhardness.

$$\varphi = \frac{S_0}{S_n} \tag{1}$$

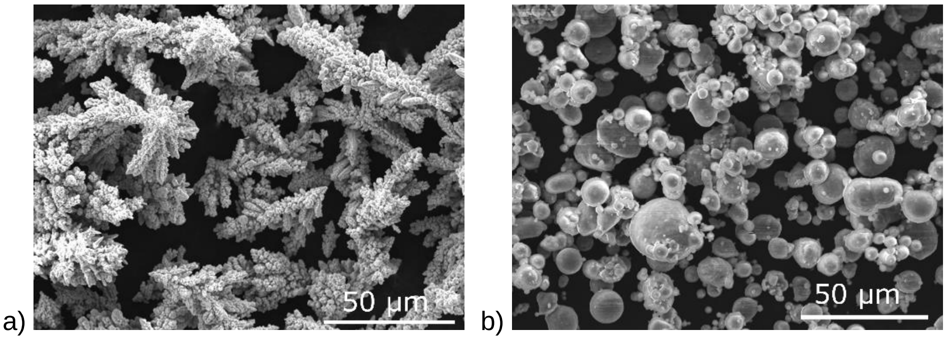

Figure 1 SEM-BSE scan of used: Cu powder (a), Al_2O_3 powder particles (b)

The analyses of the swaged composite were performed by scanning electron microscopy (Tescan Lyra 3 XMU FEG/SEMxFIB microscope), the electron backscatter diffraction (EBSD) and EDX methods were used to examine the structure and chemical composition, respectively, of the composite. As for preparation of the samples, this step involved transversal cutting of the consolidated bar, manual grinding and polishing of the samples, and final electrolytic polishing. As the microstructure was (expected to be) very fine, the EBSD scans were acquired with the step of 0.05 µm. The results of the analyses were evaluated using the AZtecCrystal software. For the analyses, the limits for low and high angle grain boundaries, i.e. LAGBs and HAGBs, were 5° and 15°, respectively. The grains orientations, i.e. textures, were evaluated with the maximum deviation from the ideal orientation of 15°. The electric conductivity of the consolidated Cu bar was evaluated using the SIGMATEST 2.070 device by FOERSTER TECOM s.r.o, Prague, Czech Republic. This eddy current based portable device enables to measure the electric conductivity using a high-tech measuring probe, which is favourable especially for determining the electric conductivity of small specimens, or of challenging materials, such as those featuring fine grains, or fine particles (e.g. oxide dispersions). The advantage of the device is, among others, that the measured value of the electric conductivity can be seen directly on the screen (both in *% IACS* and $MS \cdot m^{-1}$). Last but not least, the HV0.2 Vickers microhardness (i.e. load of 200 g) along a perpendicular line across a cross sectionally cut sample was measured using a Zwick Roell DuraScan 70 G5 device (Zwick Roell CZ s.r.o., Brno, Czech Republic). The load time for each indent was 10 s.

3. RESULTS AND DISCUSSION

3.1 Oxide dispersion

The performed analyses involved characterization of the chemical composition, i.e. evaluation of the dispersion of the oxides. **Figure 2a** shows the data summarizing the chemical composition based on the EDX analysis, while **Figure 2b** shows detailed maps of the distribution of the characteristic elements for the scanned area of 30x30 μm^2. As can be seen in **Figure 2a**, the structure contained about 6 wt.% of the Al_2O_3 oxide, which is in accordance with the expected (desired) chemical composition. **Figure 2b** then confirms that the distribution of the oxides throughout the structure was more or less homogeneous (note that the data acquisition was performed on a 70° tilted sample, due to which the shapes of the oxide particles seem to be slightly deformed to the direction of the tilt). The oxides primarily concentrated at the boundaries of the fine grains of Cu which formed during the direct consolidation (as documented by the structure analyses, see section 3.2).

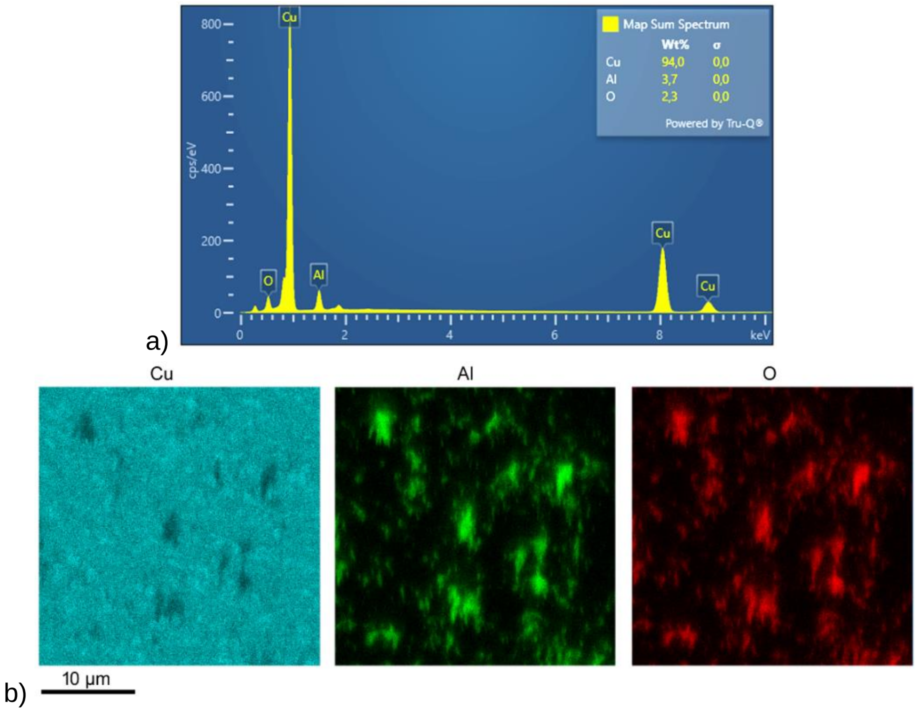

Figure 2 Results of EDX analysis: summary (a), maps of elements across scanned area (b)

3.2 Microstructure

The orientation image map (OIM) showing the individual grains with depicted grain boundaries (LAGBs in grey and HAGBs in black) is depicted in **Figure 3a** (the black non-indexed regions correspond to the locations in which the Al_2O_3 oxides were present, compare to **Figure 2b**). Evidently, the consolidated structure contained very fine grains, which was confirmed by the analysis of grain size distribution, the results of which are depicted in **Figure 3b** (the mean grain size, measured as grain area in μm^2, was determined to be 1.5 μm^2). Such grain size is way smaller than achievable for conventionally cast commercially pure Cu, swaged under cold conditions with a comparable swaging ratio [18]. As regards the grain boundaries, the structure involved comparable portions of both the characteristic boundary types (LAGB and HAGB), as evident not only from **Figure 3a**, but also from the disorientation angle distribution plot shown in **Figure 3c**. Last but not least, the structure analyses involved more detailed characterization of grains orientations via texture assessment; the results of the analyses are depicted in **Figure 3d** showing both the texture orientations and intensity in the inverse pole figure (IPF) images. The data correspond to the OIM data shown in **Figure 3a**. In other words, the structure exhibited a slight dominance of the (001)||SD (swaging direction) texture fibre, however, the maximum texture intensity was as low as 2.6. This phenomenon points to the fact that, even at the relatively low consolidation temperature, the swaging process ensured sufficient consolidation of the powders (also due to the favourable stress/strain character). Moreover, the mutual effect of decreased processing temperature and addition of oxides ensured the presence of fine grains within the consolidated composite bar, and also contributed to random grains´ orientations (both given by the pinning effect aggravating movement of grain boundaries and undesirable grain growth [19]).

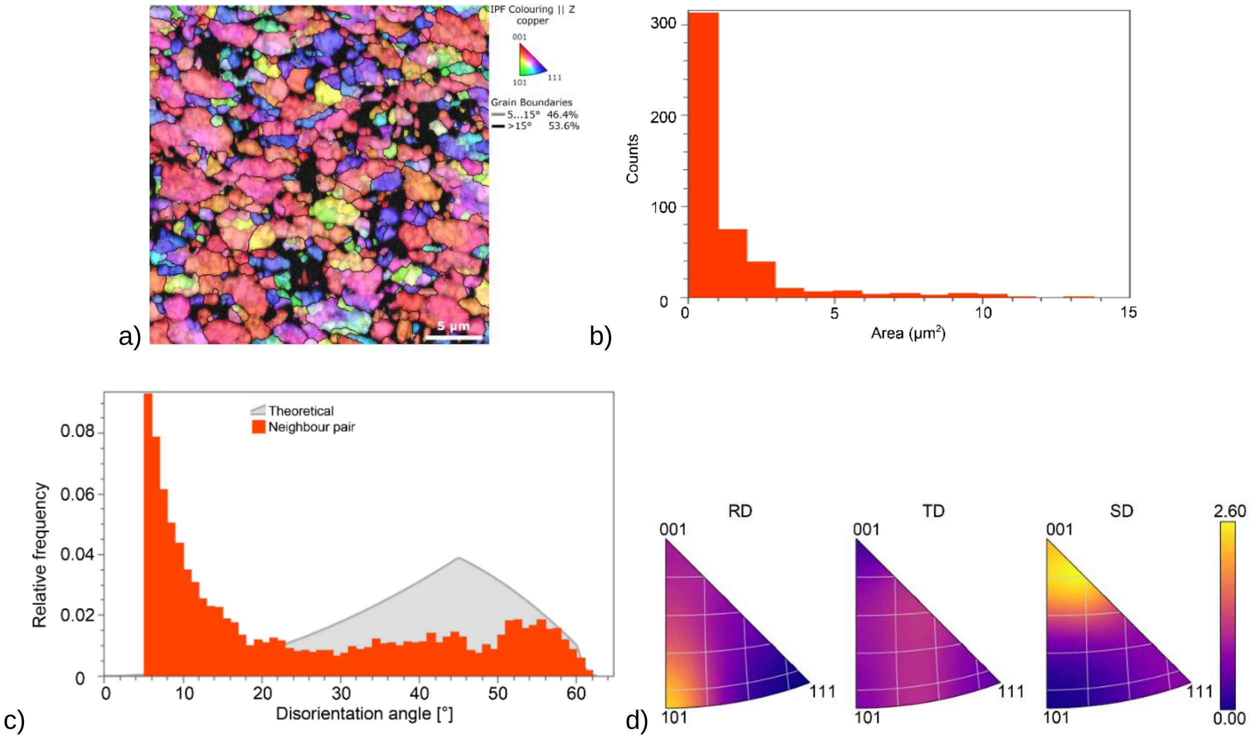

Figure 3 OIM map from scanned area (a), corresponding grain size distribution (b), corresponding disorientation angle distribution (c), IPFs for the consolidated swaged bar (d)

3.3 Microhardness

Figure 4a summarizes the values of Vickers microhardness HV02 measured across the cross-section of the sample cut from the consolidated composite bar. The values varied between 95.3 HV02 and 115.1 HV02, with the average value of 103.3 HV02 and standard deviation of 6.2. The highest values were measured in locations in which the probe hit a location with a higher concentration of the oxide particles, whereas the “lowest” values were acquired in the locations in which the concentration of the particles was scarce (see **Figure 4b**). Nevertheless, the values of the microhardness of the consolidated bar were, overall, higher than values of conventional CP Cu (for the comparison, see the line depicting the value of 50 HV02 for CP Cu in completely recrystallized state in **Figure 4a**, based on [20]).

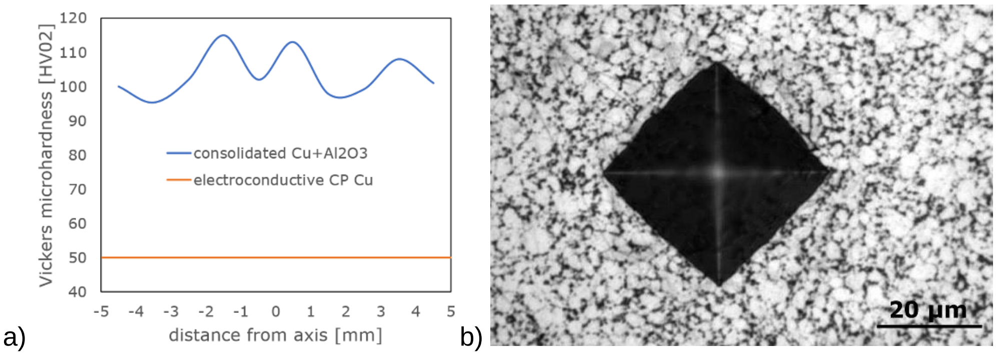

Figure 4 Microhardness measured along a line (i.e. diameter) across cross-section of consolidated sample (comparison with average value measured for electroconductive CP Cu) (a), measured indent (b)

3.4 Electric conductivity

The results of the experimental measurements of the electric conductivity revealed that the conductivity decreased in comparison with the 100 % IACS conductivity of the commercially available CP Cu; the value for the consolidated ODS Cu bar was 79.3 % IACS. This means that, compared to the cast electroconductive CP Cu, the electric conductivity decreased by almost 21%. On the other hand, the average Vickers microhardness increased by approximately 60 %. Moreover, as stated above, the grains within the consolidated structure were very fine and featured more or less random orientations. Optimized texture can be advantageous for the electric conductivity of long conductors [18]. Nevertheless, the presence of texture typically increases anisotropy of the (electric) properties, and thus application of textured conductors for small electroconductive components, such as for various components in the electrotechnics, can be unfavourable. Therefore, the presence of fine grains with (more or less) random orientations within a material can contribute to favourable electric behaviour when used for small conductive components. Moreover, as such small material volumes can be subject to temperature increase as a side effect of transfer of the electric current, the presence of small oxide particles acting as barriers for movement of grain boundaries can provide increased temperature stability of the structure (which is highly advantageous despite the lower electric conductivity). Nevertheless, further refinement of the oxide particles and increase in the homogeneity of their distribution can contribute to improvement of the mutual ratio of electric vs. mechanical properties.

4. CONCLUSIONS

The presented study focused on the assessment of the effects of direct consolidation of Cu + Al_2O_3 powder-based composite via warm rotary swaging (600 °C) on structure, microhardness, and electric conductivity of the final conductors. The selected combination of the swaging degree and temperature was sufficient for consolidation of the mechanically alloyed powders, i.e. the deformation method proved to be advantageous for direct consolidation of such composites. The oxide particles affected the structure favourably, as they not only provided fine grain size, but also more or less random grains orientations. The structure consisted of fine Cu grains with the average areas of ~1.5 μm^2, with homogeneous dispersion of Al_2O_3. The microhardness measurements showed that the mechanical properties more than doubled compared to conventional cast and annealed electroconductive Cu. However, the electric conductivity decreased to 79.3 % IACS. Nevertheless, it is expected that the ratio of mechanical vs. electric properties can be improved by further optimizing the deformation processing, or by involving a post-process heat treatment. By this reason, optimizing the processing conditions in order to enhance the electric conductivity is the main topic of our further research.

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