Mechanical Properties of Cu-Al₂O₃ System at Elevated Temperatures

Michal Besterci¹, Oksana Velgosova*², Ivan Kohutek³

¹ Institute of Materials Research, Slovak Academy of Sciences, 04353 Kosice, Watsonova 47, Slovak Republic, E-mail: mbesterci@imr.saske.sk
² Dept. of Non-ferrous Metals and Waste Treatment, Faculty of Metallurgy, Technical University, Letna 9/A, 04200 Kosice, Slovak Republic, E-mail: oksana.velgosova@tuke.sk
³ US Steel Kosice s.r.o., 04000 Kosice, Slovak Republic, E-mail: ikohutek@sk.uss.com

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ABSTRACT

A review starting from material science through production technology to testing properties of the Cu-Al₂O₃ system, based on several years of research and development in materials research, is presented. Microstructural parameters and strength and plastic properties at higher temperatures are presented and the failure mechanism described. Influence of dispersed Al₂O₃ particles on recrystallization is characterized, too. Microstructural parameters were investigated by light microscopy and TEM, was found that the Al₂O₃ particles size 85 nm are textured in the bands and the grains mean size is less than 1.0 μm. The high thermal stability of microstructure was confirmed. The fracture mechanism up to 673 K is transcrystalline and from 673 K is intercrystalline. Plastic deformation up to 673 K is carried out by dislocation sleep in the grains and at higher temperature the deformation is controlled by the restoring processes, dynamic polygonization and sliding along the grain boundaries.

Key words: Cu-Al₂O₃ system, microstructure, mechanical properties, fracture, recrystallization.

1. INTRODUCTION

Copper has the leading role in industrial applications. A variety of Cu alloys has been developed, but they show a large increase of resistance in both electric and heat conductivity, and low long time stability at elevated temperatures. They are hardened by ageing, and they lose their strength at elevated temperatures. The alloys show great susceptibility to grain growth by precipitation, or by the solution of precipitates. Powder metallurgy (PM) technologies can give a solution, if the dispersed particles in the prepared material are small enough, less than 50 nm, and proper distribution, with the mean free path also small, can be obtained. Dispersed particles like Al₂O₃ are thermally stable with no susceptibility to grain growth or to solution at high temperatures, so they offer a possibility to produce dispersion strengthened systems with good mechanical properties at high temperatures, too. This way high strength material can be produced, even at relatively low dispersed particle content. This is very important, because the lower dispersed particle content in the Cu matrix the higher conductivity of the material.

The resistance to recrystallization and recovery is an
important property of dispersion strengthened systems. A large number of models were introduced for the description of the influence of dispersed particles on recrystallization. Large number of works tested this influence experimentally /3-8/. The dispersed particles in the matrix change the nucleation and growth of grains by recrystallization, so recrystallization depends on the spatial distribution of the particles. In general, it is valid that the presence of large particles with large interparticle distances (local cell distortion) increases the recrystallization rate, and small particles with short interparticle distances slow down or cut the recrystallization at all. Other influences are so large that any more precise definition of “the transition parameter” is extremely difficult.

Cotterill /9/ in his work introduced a critical interparticle distance for recrystallization initiation and critical interparticle distance for grain growth, though not considering particle size. The process is limited by the assumptions that particles are not deformable with the matrix, local cell distortions are present due to strain, and both the final grain size and kinetics of recrystallization depend on interparticle distance.

For powder metallurgy materials also the influence of residual porosity must be considered. The pores can migrate at higher temperatures due to diffusion. The recrystallization can be depressed by the interaction of pores with grain boundaries.

The aim of this work is to analyze the mechanical properties at elevated temperature and recrystallization of Cu-Al₂O₃ system.

2. MATERIAL AND EXPERIMENTAL METHODS

Dispersion strengthened materials, Cu-Al₂O₃, were prepared by the method of reaction milling. The first mixture was CuAl alloy containing 1.25 mass % Al. Homogenization milling was applied in an oxidizing atmosphere. During this milling copper oxide is produced evenly distributed in granulate. By the next heat treatment the oxygen is bound to Al producing this way the Al₂O₃ content. Different materials were prepared, the Al₂O₃ content ranging from 5 vol.% in the Cu matrix. Then the excess oxygen was reduced from the Cu matrix by annealing in a reducing atmosphere of H₂+H₂O. The powder mixture was then pressed, sintered and extruded at 1073 K. The majority of tested materials were produced at the Technical University of Vienna. The flow chart of the production is in Fig. 1.

![Fig.1: Scheme of reaction milling.](image)

The aim was to analyze the mechanical properties of the Cu-Al₂O₃ system with 5 vol. % of Al₂O₃, at test temperatures ranging from 293 to 973 K. Test pieces for mechanical properties with threaded ends, 3.5 mm in diameter, with 30 mm working lengths were tested in tension at a static strain rate $4.5 \times 10^{-4}$ s⁻¹ calculated from the cross head speed 0.75 mm/min, at temperatures 293, 673, 773, 873, 973 K. The strain was measured by a strain gauge fixed on the outer ends of pulling rods outside of the furnace. A large 200 kN and stiff test machine was used. For the large diameters of the pulling rods and low loading forces (2700 N at 293 K, and about 800 N at high temperatures) the uncertainty introduced by the measurement on the pulling rods was small, less than 5%. Values of the yield point $R_{y0.2}$, ultimate tensile strength $R_m$, and ductility $A_5$ were determined.

3. RESULTS AND DISCUSSION

Microstructure investigations by light microscopy have shown that the second phase particles Al₂O₃ were textured into bands in the direction of hot extrusion. The grain size was not made visible. Transmission electron microscopy using thin foils revealed the composition of
the matrix. It showed large angled disoriented aggregates, supposed to be the grains with a mean size less than 1.0 μm. The majority of the dispersed particles of Al₂O₃ are globular. This has been double checked by carbon replicas. The mean size of dispersed particles is around 85 nm. They are distributed single, or in clusters on grain boundaries as well as inside in the grains. There have been positive interactions shown between the Al₂O₃ particles of this kind and dislocations. Strain at temperatures up to 473 K has induced the increase of the dislocation density in the microstructure. At the implicated temperatures from 773 to 973 K no significant changes were observed when compared to the original microstructure. This provides evidence of the high thermal stability of the microstructure.

It is possible to sort the stress-strain curves obtained into three groups according to the layout in Fig. 2. The first group labeled (a) is the fracture at 293 K. The second group (b) is for temperatures up to 673 K, and the third group (c) for temperatures from 773 to 973 K. The stress-strain curve at 293 K consists only from the part with uniform strain 0-02 and the part with work hardening 02-1. The point 1 represented the maximum load and the fracture point, too. The stress-strain curve for the temperature 673 K, besides the previous parts had also the part 1-2 from the maximum load to the loss of plastic stability. The reduction of area at fracture was very small near to measureless. For temperatures from 773 to 973 K, besides the previous, there was also part 2-3, a marked local strain and the loss of plastic stability.

The obtained temperature dependencies of the mechanical properties are plotted in the following figures. In Fig. 3 the dependence of the yield stress \( R_{p0.2} \) on temperature is shown. The straight part from 293 K to 773 K can be described by the following relation:

\[
R_{p0.2} = 370 - 0.40.T
\]  

(1)

where \( T \) is the absolute temperature in Kelvin.

In Fig. 4 the temperature dependence of the ultimate tensile strength is shown; the dependence is near parallel to the yield stress as can be seen from the coefficients of the equation for temperatures from 293 to 773 K, as:

\[
R_m=381 - 0.41.T
\]  

(2)

The dependence of ductility \( A_5 \) on temperature is shown in Fig. 5. There is a significant growth of plasticity at about 600 K. According to /10/ it can be related to a change of activation energy on the grain boundaries.

The mechanical properties obtained are in good agreement with the values released by the manufacturer for this trade mark Glidcop. In Fig. 6 the dependencies of the strain contributions are plotted. The labels are: the uniform strain up to the maximal load \( (\varepsilon_u = \varepsilon_1) \) the strain by decreasing load and near constant true stress \( (\varepsilon_2 = \varepsilon_2) \), and the localized strain \( (\varepsilon_3 = \varepsilon_3 - \varepsilon_2) \).
Considering the obtained stress-strain curves and microstructural analysis, it has been settled that at temperatures up to 673 K the dominant mechanism controlling the plastic deformation is the dislocation slip in the grains. At higher temperatures, about 873 K, there is a significant strain after the point of the maximal load showing the presence of the restoring processes at near constant true stress, the so-called “plateau”. There is also a final local strain or neck forming and loss of plastic stability. We suppose, the deformation of plateau is controlled by the dynamic polygonization, leading to the balance in work hardening and softening, with slides along the grain boundaries, or in their vicinity. This growth of the ductility is a superplastic like behavior, and is important for the technology of hot forming. The comparison of the results with other work, e.g., /2/, suggests that the higher increase of plasticity is probably due at higher strain rates. It comes from dynamic reactions of dislocations in the pattern of very fine size grain boundaries.

We have defined the influence of the test temperature on the micromechanism of fracture by microscopic analyses of the fractured surfaces on samples tested at temperatures 293, 673, 773, and 873 K. On samples tested at 293 K the fracture is ductile, transcrystalline, with dimples and with ridges. The ridges are formed by local strain into protruding peak points or peak lines formed by near to 100% reduction of micro cross sections, before the failure. The dimensions of dimples are around 1 µm with a low scatter. As described previously, the distribution and dimensions of dimples are in close relation with the distribution of the dispersed particles. The microscopic images of the surfaces fractured at higher temperatures are positively temperature dependent. At low magnification the morphology of the surface fractured at 673 K resembled the one fractured at 293 K. But at high magnification the dimples are not visible, though the fractured surface is ductile in majority. There are some areas with intercrystalline fracturing, too. The microscopic image of the surface fractured at 873 K revealed a decrease of microplasticity and it can be described as intercrystalline. As a result of testing in air the fracture surface is covered with an oxide layer.

The introduction of the dispersed particles into the matrix caused strengthening but also the high
probability of the initiation of cavities on the interface of the particle with the matrix. The coalescence of cavities leads to a transcrystalline ductile fracture. The equi-cohesive temperature (about 633 K) of the matrix is increased. At the test temperature 673 K, there were also intercrystalline ductile areas on the fractured surface. It agrees with the assumptions made and proves a significant increase of the equi-cohesive temperature.

Curves showing the dependence of recrystallization on temperature after 50% cold deformation are shown in Fig. 7. For comparison also the curve for pure Cu is included. The volume content of the phase Al₂O₃ the higher recrystallization temperature is needed. For the material with 5 vol.% of Al₂O₃ the \( T_R = 768 \) K. That is twice the value for pure Cu.

![Fig. 7: Recrystallization curves after 50% deformation.](image)

We calculated the interparticle distances in electron microscopy images made by carbon replicas (mean free path \( \lambda \)) according to the formula:

\[
\lambda = (2d_0 / 3f)(1 - f)
\]

where \( d_0 \) is the mean diameter of Al₂O₃ particle, \( f \) is the volume content.

The dependence of the recrystallization temperature on the mean interparticle distance is shown in Fig. 8 and it is also in good agreement with the results in /9/. After annealing at 600 K the recrystallized microstructure is visible with twins.

![Fig. 8: Dependence of recrystallization temperature on the interparticle distance.](image)

4. CONCLUSION

The mean grain size of such prepared materials is less than 1\( \mu \)m. The dispersion phase is globular with the mean size 85 nm, Al₂O₃ particles are distributed on grains boundaries and inside the grains.

The dominant mechanism controlling the plastic deformation up to 673 K is the dislocation slip in the grains and at 873 K there occur the processes of restoring. The deformation in the restoring area is controlled by the dynamic polygonization with slide along the grain boundaries. The fracture at 293 K is ductile and transcrystalline, the intercrystalline ductile areas have occurred at 673 K, the surface fractured at 873 K revealed a decrease of microplasticity with intercrystalline fracture.

According to the results described in this article it was shown that Cu-Al₂O₃ material can be prepared by powder metallurgy technology with microstructure, material properties at room or elevated temperatures, and resistance to grain growth by recrystallization, which are more suitable than the properties of Cu.

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