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# Mechanical and electrical properties of mechanically alloyed nanocrystalline Cu–Nb alloys

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**Abstract**

Nanocrystalline Cu and Cu–Nb alloys were prepared by the consolidation of mechanically alloyed powder. The alloys show a microstructure with a grain size below 50 nm. The microstructure of the Cu matrix remains stable even at elevated temperatures of up to 900 °C, whereas the Nb precipitates coarsen during annealing. The mechanical strength as well as the electrical conductivity depend on the grain size of the Cu matrix, which can be influenced by the temperature of the heat treatment, i.e., a mechanical strength of about 1.6 GPa is measured for a Cu–10 at.% Nb alloy which shows an electrical conductivity of about 10% IACS (international annealing copper standard) at room temperature. The main contribution to the mechanical strength of the alloys is attributed to the grain boundary strengthening in Cu referring to the Hall–Petch relation, which is quantified. The grain boundaries are also found to influence considerably the electrical resistivity.


*Keywords:* Copper alloys; Mechanical alloying; Mechanical properties; Electrical properties; Nanocrystalline materials

## 1. Introduction

Nanostructured materials with a grain size below 100–200 nm have been much studied in recent years due to their unusual mechanical, electrical and magnetic properties [1–4]. These properties are regarded as unusual insofar as they differ from what is expected for polycrystalline materials that have a larger grain size but the same chemical composition. Thus, the properties of nanostructured materials always have to be associated with the grain size, which, at the same time, has a great influence on the volume fraction of the grain boundaries. With respect to the large number of grain boundaries, one can estimate that the mechanical strength of nanocrystalline metallic materials is enhanced as the dislocation motion is also hindered by grain boundaries. Compared to metals with coarse grains, nanocrystalline metals exhibit a much higher strength [5] than observed in, for example, pure copper. Polycrystalline copper can be hardened by cold working up to about 400 MPa [6], whereas pure copper with a grain size of 16 nm shows a mechanical strength of 800 MPa [7]. However, for some nanocrystalline materials, the opposite behaviour has been observed; the high number of grain boundaries actually softens the material [8,9]. This is referred to as the 'inverse Hall–Petch relation'. The origin of the inverse Hall–Petch relation is not yet well understood in complete detail [9]. Although much work has been performed on nanoscaled materials, the relation between their microstructure and their physical properties still has to be understood and modelled. This especially holds for electrical properties. Just a few publications analyse the influence of grain boundaries on the electrical resistivity of nanocrystalline materials [3,10,11] and their results are not very detailed at this point.

In our previous studies [12–14], we have shown that an extremely fine nanocrystalline microstructure can be achieved in Cu–Nb alloys by a combination of mechanical alloying and heat treatment. About 10 at.% Nb can be

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dissolved homogeneously within a Cu matrix by mechanical alloying. This is significant because there is only a negligible solubility of Nb in Cu at thermodynamic equilibrium conditions [15]. A subsequent heat treatment procedure applied to the milling leads to the precipitation of small and homogeneously distributed Nb particles from the solid solution. The alteration of the duration and temperature of the heat treatment controls the size of the Nb precipitates. Compacted samples of Cu–10 at.% Nb alloys show a very high mechanical strength of about 1.6 GPa [16]. The high electrical conductivity of Cu–Nb alloys can be calculated from a linear rule of mixture, as Nb shows a negligible low solubility in Cu. Therefore, after the heat treatment, Cu and Nb are present within the microstructure in parallel with respect to the electrical behaviour. Furthermore, since the elastic properties of Nb and Cu are quite similar, a large ductility of the alloys can be expected.

The properties mentioned make Cu–Nb alloys potential candidates for application in pulsed high-field magnets. The main requirements for materials suitable as wire for these magnets are a combination of high strength, high conductivity, high ductility and high fatigue life [17,18]. However, two aspects have to be considered before application of this material as a conductor for pulsed high-field magnets. First, the process of wire preparation [19] has to be scaled up in order to obtain a wire with a suitable length and cross-section. Second, the properties of the material, especially when deformed to a wire, have to be investigated at 77 K, since pulsed high-field magnets are operated at these low temperatures.

In the present study, the mechanical and electrical properties of nanocrystalline Cu–Nb alloys are investigated. The samples have a nominal composition of 0, 5, 7 and 10 at.% Nb and were prepared by mechanical alloying, hot pressing and a subsequently applied heat treatment. This study clarifies the influence of the temperature during consolidation or heat treatment on the microstructure of the alloys. The effects of the nanocrystalline microstructure on the mechanical and electrical properties at room temperature are discussed.

## 2. Synthesis and characterisation

Cu–Nb alloys with a nominal Nb content of 0, 5, 7 and 10 at.% were prepared by mechanical alloying. As starting materials, high-purity powders of Cu and Nb with a particle size smaller than 35 μm were used. The mechanical alloying was performed in a planetary ball mill under argon atmosphere at low temperatures until a complete and homogeneous dissolution of Nb within the Cu matrix was achieved, i.e. about 35 h. The procedure and parameters of mechanical alloying are detailed in our previous publications [12,13]. The mechanically alloyed powder was consolidated by uniaxial hot pressing with a load of 750 MPa for 20 min at different temperatures. The consolidated samples with a diameter of 10 mm and a length of 10–20 mm had a density of more than 95% related to the theoretical density of the alloy. The pure Cu powder was pressed at 400 °C, whereas the powders of the Cu–Nb alloys were consolidated at different temperatures, namely 600, 700 and 800 °C, respectively. Some compacted samples were additionally heat treated at different temperatures from 600 up to 1000 °C for 1 or 10 h under argon atmosphere subsequent to the consolidation process.

X-ray diffraction patterns were measured in Bragg–Brentano geometry utilising a Philips diffractometer (PW 1830) using Co Kα radiation ($\lambda = 0.178897$ nm). The lattice parameters were determined from the measured diffraction data using the DBWS Rietveld program [20]. The grain size and the internal strain were obtained from the measured data using Williamson–Hall plots [12,21]. The grain size of some compacted samples was additionally confirmed by transmission electron microscopy (TEM) from dark-field images. In order to investigate the samples by electron microscopy the samples were embedded in conductive resin and prepared by standard metallographic techniques. A high-resolution scanning electron microscopy (SEM) instrument (LEO 1530) was used for this purpose. TEM investigations of compacted samples were carried out on ion-beam-thinned foils utilising a JEOL JEM 2000 FX operating at 200 kV. The hardness of compacted samples was determined from Vickers hardness measurements using a load of 10 N and a loading time of 10 s. The mechanical properties were investigated by compression tests. The compression tests were carried out on cylindrical samples with a height of 5 mm and a diameter of 3 mm utilising an electromechanical Instron 8562 universal testing machine with a crosshead speed of $10^{-3}$ mm/s. The electrical resistivity was measured on samples with a cross-section of ~1 mm × 1 mm and a length of about 7–10 mm using the standard four-probe technique at room temperature.

## 3. Results

### 3.1. Microstructure

The X-ray patterns of Cu–10 at.% Nb samples (Fig. 1), which were taken after consolidation at different temperatures (600, 700 and 800 °C), show that Nb precipitates from the solid solution, which was achieved during mechanical alloying. This also holds for the comparatively low consolidation temperature of about 600 °C. With increasing temperature, the intensity of reflections related to Nb increases. At the same time, the fraction of phase impurities such as $Cu_2O$, NbO and $Fe_7Nb_6$ grows. Although milling was performed under argon atmosphere, the powder oxidises to some degree during mechanical alloying. The oxygen arises from the atmosphere as the sealing of the mill vessels is not sufficient for cryomilling. Impurities of Fe arise from abrasions from the milling vessels and balls. However, the total amount of impurities is very low, and therefore is not relevant here.

Fig. 2 shows SEM and TEM images of the microstructure of a Cu–10 at.% Nb alloy mechanically alloyed for