STRUCTURAL CHARACTERISTICS AND MECHANICAL PROPERTIES OF DISPERSION STRENGTHENED Cu-Cr-Nb ALLOY

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Abstract

The strength and hardness in some metal or alloys may be enhanced by the presence of extremely small and uniformly dispersed particles within the original phase matrix. The aim of this paper was to prepare and characterize the Cu-0.4Cr-0.1Nb (wt.%) alloy strengthened by the high melting point intermetallic compound Cr_2Nb. This phase is stable in solid and liquid Cu up to temperatures above 1600 °C and it contributes to the microstructural stability of the alloy during its application at elevated temperatures. Both chromium and niobium have a minimal solubility in solid Cu, which limits the decrease in conductivity. This alloy was produced by a combination of plasma melting and vacuum induction melting and it was formed into wires of diameters 0.3 and 0.5 mm. The structural properties and chemical composition were evaluated using scanning electron microscope JSM-7600F equipped with a WDS spectrometer INCA and the high-resolution field emission gun-scanning electron microscope QUANTA 450 FEG equipped with an energy dispersive X-ray analyzer APOLLO X. The samples were subjected to the tensile testing. The Cr_2Nb precipitates were fairly uniformly distributed in Cu-matrix and their mean size was approx. 1-3 um. The ultimate tensile strength of wires was substantially increased in comparison to an unalloyed copper wire.

Keywords: Cu-Cr-Nb alloy**,** Cr_2Nb precipitates, induction melting, wire drawing, mechanical properties

1. INTRODUCTION

The Cu-Cr-Nb alloy provides the combination of high strength, high thermal conductivity, and good long term stability at elevated temperatures and it can be considered as a candidate material for uses in the thrust chamber liners in rocket engines and in nuclear reactors, such as fusion reactor divertor and first wall components [1-7]. The Cu-Cr-Nb alloy with the Cr/Nb ratio (at. %) of 2:1 is strengthened by the high melting point Cr_2Nb intermetallic phase, which is formed in-situ in the copper matrix (**Figure 1**). These very fine sized precipitates with uniform distribution are stable in solid and liquid copper to temperatures above 1600 °C. The primary precipitates formed directly from the melt have approx. 0.5-2 μm in size [1-3]. Secondary Cr_2Nb intermetallic particles (<100 nm) precipitate from the solid solution. The solubility of Cr and Nb is very low in solid Cu, limiting the decrease in conductivity. This phase also contributes to the microstructural stability of the alloy during subsequent elevated temperature application. These alloys can be produced by different methods, such as self-propagating high temperature (SHS) process, chill block melt spinning (CBMS), gas atomization, cryomilling, extrusion, hot isostatic pressing (HIP), casting, warm rolling, mechanical milling and vacuum plasma spraying (VPS) [1-7].

This paper deals with the preparation of Cu-0.4Cr-0.1Nb (wt.%) alloy with the optimized properties using an innovative metallurgical route and its processing into a form of wires of 0.3 and 0.5 mm in diameter. The improved mechanical properties were achieved by introducing a small volume fraction of dispersed Cr_2Nb precipitates (f < 0.5 %) in a microstructure. The structural characteristics and the phase composition of as-cast and as-drawn samples were investigated and the effect of processing on the distribution of precipitations was evaluated. The ultimate tensile strength, elongation, and micro-hardness of wires at room temperature were tested.

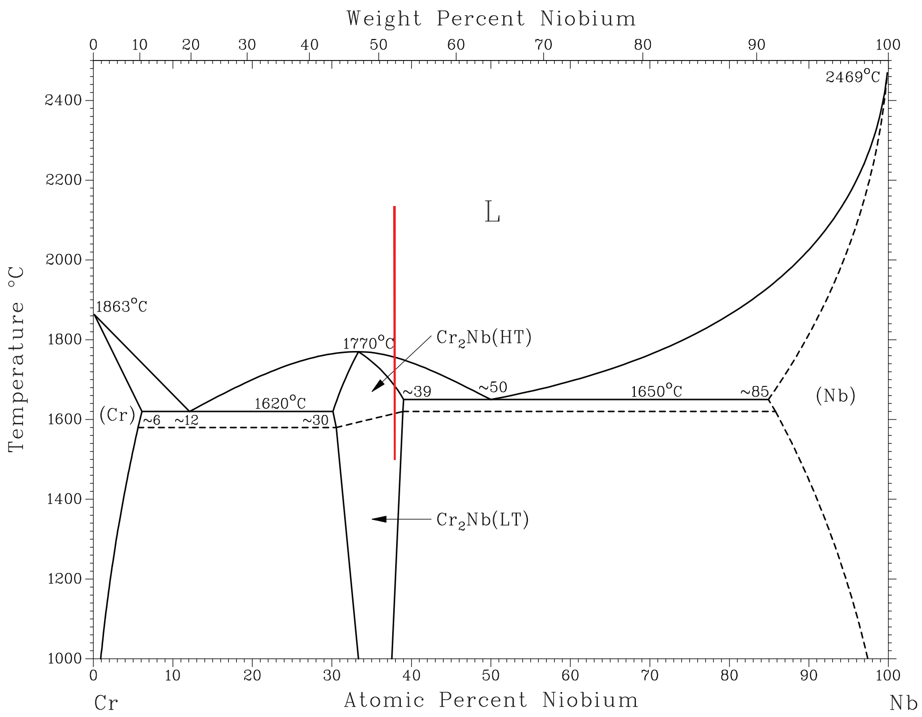

Figure 1 The phase diagram for binary Cr-Nb system [8]

2. EXPERIMENTAL

The Cu-0.4Cr-0.1Nb (wt.%) alloy was prepared using the induction furnace SecoWarwick S/A under the protective atmosphere of argon and it was cast into the graphite mold of a diameter of 50 mm and length of 300 mm. As the input materials, copper blocks (99.99 wt.%) and chromium chips (99.5 wt.%) were used. The niobium was used in the form of 85Nb-15Cu (wt.%) master alloy that was produced by a plasma metallurgy with using similar conditions as described in the article [9,10]. The melting range of this alloy was determined by means of a direct thermal analysis technique, which is described in more detail in [11]. The sample was completely melted in the temperature interval of 1790-1890 °C, which was substantially lower in comparison with a melting point of pure niobium. This master alloy was machined and the chips were used as an input material.

The ingot of Cu-Cr-Nb alloy was surface machined in order to remove the oxidation layer and surface defects. This ingot was then formed into a cylindrical rod of 30 mm in diameter by a cold rotary swaging. The wires of 0.5 and 0.3 mm in diameter were obtained by cold drawing through the hard metal drawing bench dies. The conditions of the drawing process are the know-how of the company CAMEX, s.r.o. and cannot be published.

The structural properties and phase chemical composition of the samples were evaluated using scanning electron microscope JSM-7600F equipped with a WDS spectrometer INCA and the high-resolution field emission gun-scanning electron microscope QUANTA 450 FEG equipped with an energy dispersive X-ray analyzer APOLLO X. The structure was also observed by the GX51 inverted metallographic microscope equipped with digital camera DP12. The oxygen content in as-cast and as-drawn samples was determined by the inert gas fusion method using ELTRA ONH 2000 analyzer. The results of the chemical analysis are given as the mean values from three measurements. The micro-hardness of samples at a loading of 200 g for 5 s was measured by means of the FM-100 hardness tester. The multifunction electro-mechanical equipment Zwick/Roell Z150 was used for the realization of tensile testing of wires. The strain rate was 2 mm/min.

3. RESULTS AND DISCUSSION

3.1. Microstructural characteristics and phase chemical analysis

The two-phase microstructure of Nb-Cu master alloy is documented in **Figure 2**. These elements have a minimal mutual solubility in solid state and their structure consists of (Nb)-dendrites with an interdendritic (Cu) phase - see **Table 1**. The structure of Cu-Cr-Nb alloy in as-cast, as-swaged and as-drawn state are documented in **Figure 3**. The microstructure of as-cast alloy consisted of the coarse grains of Cu-matrix in a cross section. In the as-swaged sample, the deformation bands were visible inside the elongated grains. The microstructure of wires in longitudinal section exhibited small grains elongated in the drawing direction when the final recovery process was not realized.

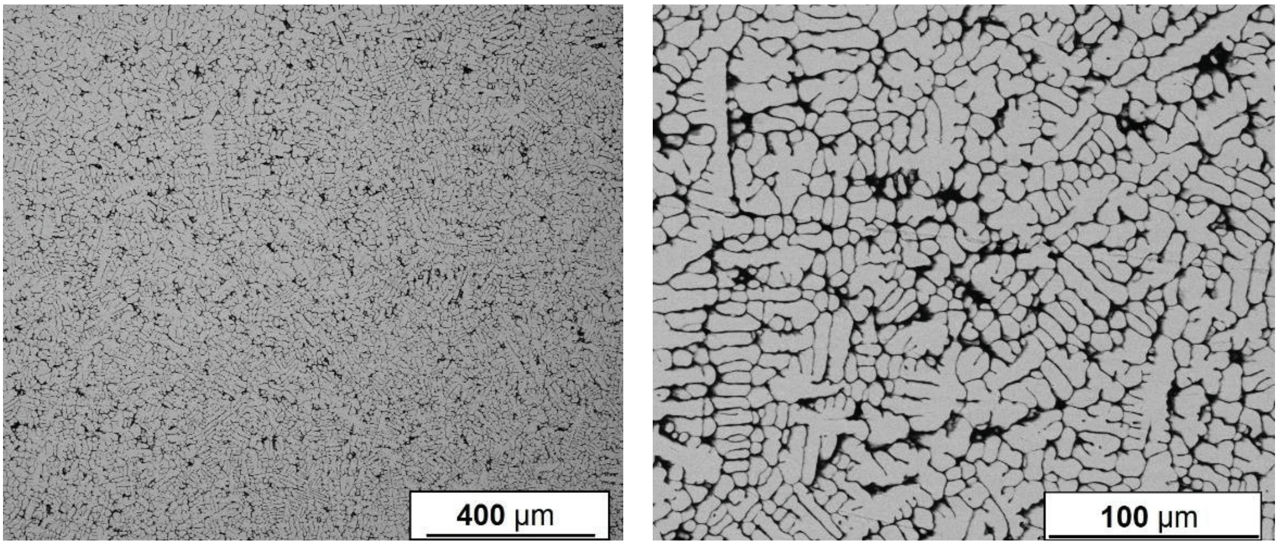

Figure 2 The two-phase microstructure of Nb-Cu master alloy

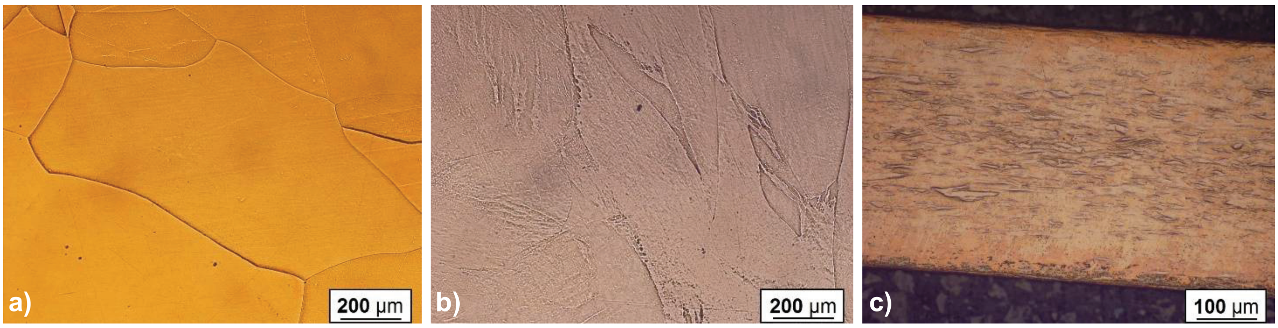

Figure 3 The structure of Cu-Cr-Nb alloy in as-cast (a), as-swaged (b) and as-drawn state (c)

The chemical composition of the Nb-Cu master alloy and the cast Cu-Cr-Nb alloy is summarized in **Table 1,** which correspond with the required composition of these alloys. The oxygen content in this alloy was 16 ppm. The SEM micrographs in **Figure 4** revealed the presence of very fine (1-3 μm) and quite uniformly dispersed Cr_2Nb precipitates (as dark phase) with nearly stoichiometric composition (see **Table 1**) in the copper matrix. Because of the very small size of these precipitates, it was difficult to accurately ascertain their chemical composition by SEM-EDX.

Table 1 Chemical analysis of the Nb-Cu master alloy, as-cast Cu-Cr-Nb alloy and Cr_2Nb precipitates

Sample	Cr		Nb		Cu		Note
	(wt.%)	(at.%)	(wt.%)	(at.%)	(wt.%)	(at.%)	
Nb-Cu master alloy (area)			85.0	79.5	15.0	20.5	EDX
(Nb)-dendrites			97.5	96.3	2.5	3.7	
Inter-dendritic (Cu)-phase			1.5	1.0	98.5	99.0	
As-cast Cu-Cr-Nb alloy (area)	0.39	0.48	0.09	0.06	99.52	99.47	WDX
Cr-Nb precipitate	23.9	29.9	23.3	16.3	52.8	53.9	EDX
	Cr:Nb = 1.8 (at.%)						

The fine precipitates were also investigated using X-ray maps, where chromium and niobium content was confirmed - see **Figure 5**. It can be seen in **Figure 6** that the forming processes caused an uneven distribution of precipitates in wires and some were ordered to a line. Their size was different from the initial state when the average value decreased below 1 μm. The content of oxygen in the wires increased to 220 ppm.

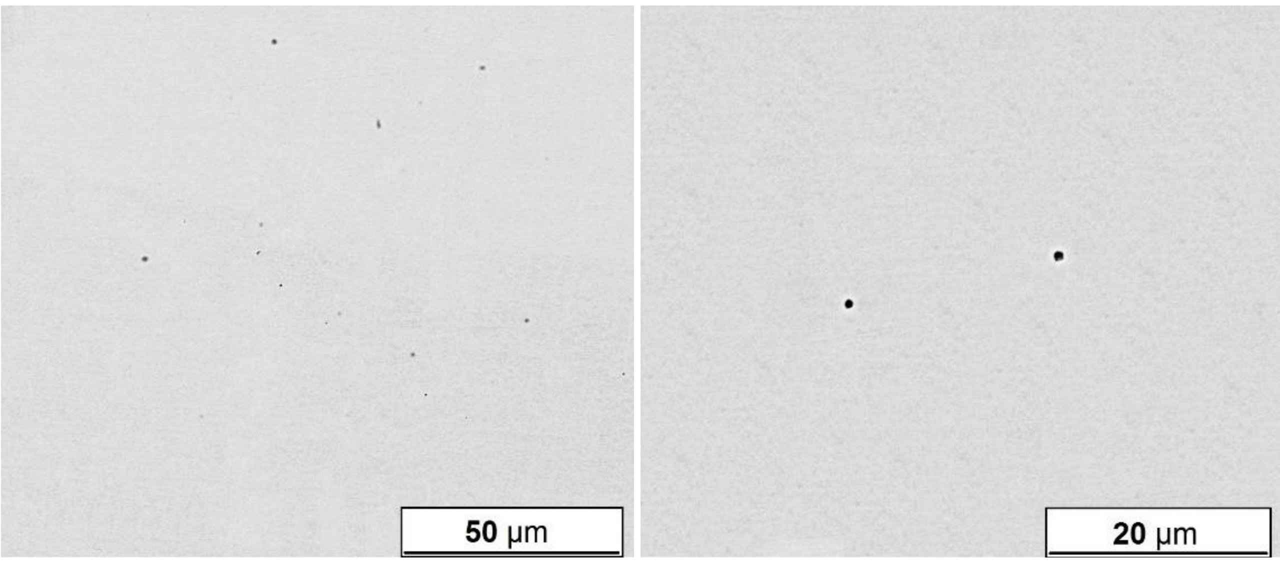

Figure 4 Fine Cr_2Nb precipitates (as dark phase) in the copper matrix

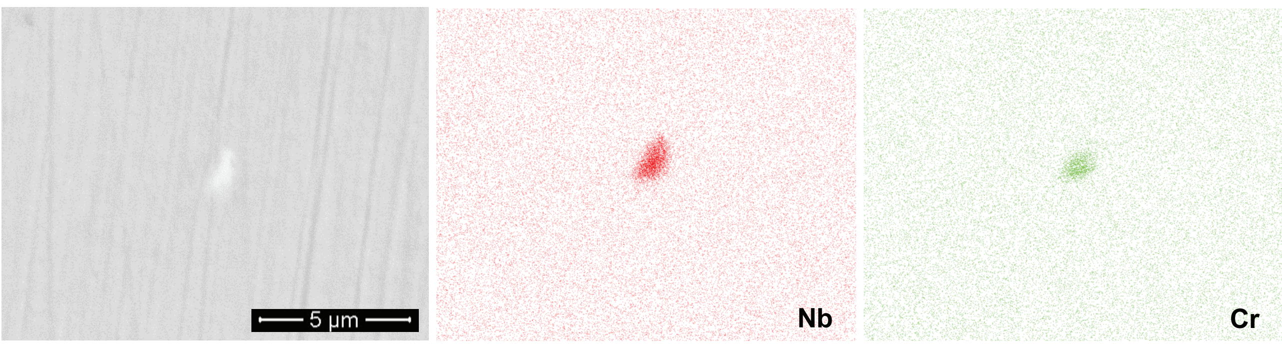

Figure 5 X-ray maps of chromium and niobium in Cr_2Nb precipitate and Cu-matrix

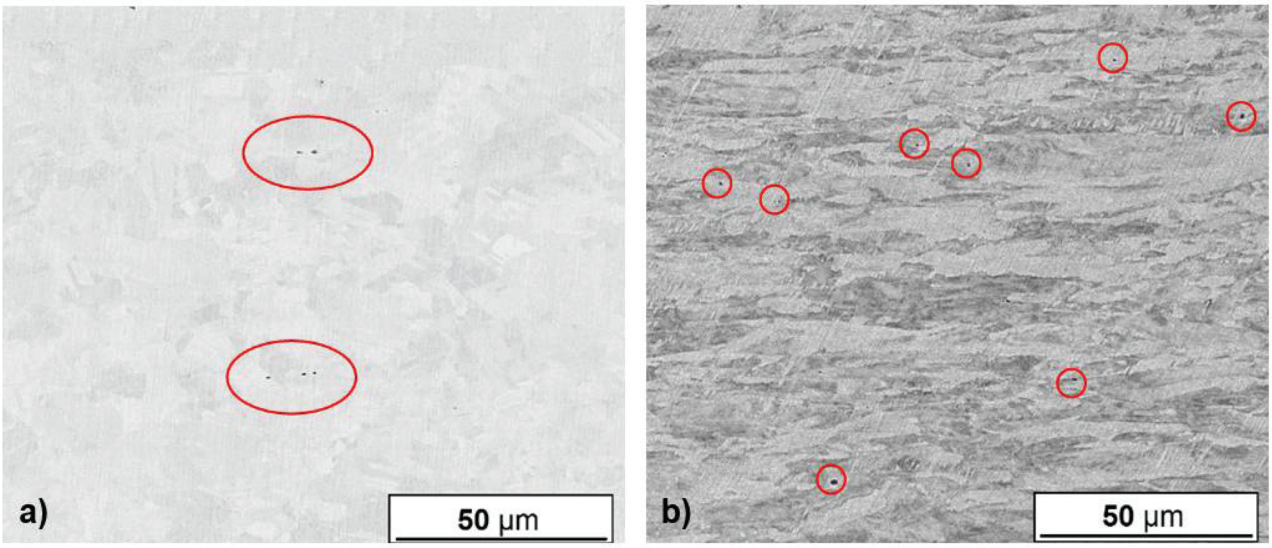

Figure 6 Ordering of precipitates in the wire with a diameter of 0.5 mm (a) and an uneven distribution of precipitates in the wire with a diameter of 0.3 mm (b)

3.2. Mechanical properties

Tensile tests were conducted to determine the ultimate tensile strength (UTS), yield strength (YS) and total elongation (A) of wires with the diameter of 0.5 and 0.3 mm, respectively. Generally, the greater cross-sectional area reduction formed during the cold drawing led to the narrowing of the spread between UTS and YS. The stress-strain curves of wires containing the small Cr_2Nb precipitates obtained for the given strain rate are documented as examples in **Figure 7**. As shown, the curves did not exhibit the yield strength point. The wires mostly did not evince the plastic deformation up to the rupture corresponding to the tensile strength. It means that the yield strength of wires is coincident with their ultimate tensile strength.

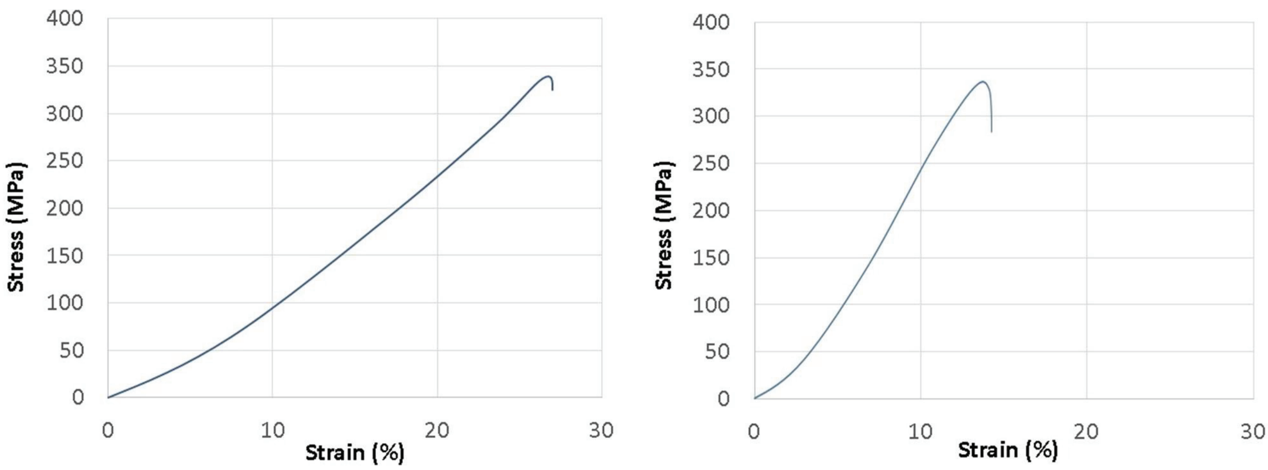

Figure 7 Stress-strain curves of wire with the diameter of 0.5 mm (a) and 0.3 mm;
strain rate = 2 mm/min

Caused a slight increase in UTS, while the total elongation was decreased by approx. 30 %. These values are consistent with the specified minimum requirements for the tensile mechanical properties of this alloy. It can be also seen from **Table 2** that the micro-hardness of wires without the final recovery process was about two times higher in comparison of the as-cast alloy.

Table 2 Ultimate tensile strength, total elongation, and micro-hardness of Cu-Cr-Nb alloy in as-cast, as-swaged and as-drawn state

Sample	UTS	A	$HV_{m0.2}$
	(MPa)	(%)	(-)
As-cast alloy	-	-	54.7 ± 2.4
Wire - 0.5 mm	293	27	90.3 ± 1.5
Wire - 0.3 mm	329	18	118.6 ± 3.4

4. CONCLUSION

The Cu-0.4Cr-0.1Nb alloy was prepared by means of the innovative liquid metallurgical route. This included the preparation of 85Nb-15Cu master alloy by plasma melting, which has a substantially lower melting range than the pure niobium and was used as the alloying component. The Cu-Cr-Nb alloy was then cold rotary swaged and drawn into a form of wires with a diameter of 0.5 and 0.3 mm, respectively. The small-sized Cr_2Nb precipitates were observed in the microstructure of as-cast alloy. Their distribution was changed after the cold rotary swaging and drawing when the precipitates were dispersed unevenly and some were arranged in a line. The results of a tensile test of wires demonstrated that their yield strength was coincident with the ultimate tensile strength. The wires mostly did not evince the plastic deformation to the rupture corresponding to the ultimate tensile strength. The values of mechanical properties are, however, consistent with the specified minimum requirements for the tensile mechanical properties of this alloy used in a nuclear technology.

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