



Microstructure and mechanical properties of $\text{Cu}_x\text{Nb}_{1-x}$ alloys prepared by ball milling and high pressure torsion compacting



M.D. Abad ^{a,*}, S. Parker ^a, D. Kiener ^b, M.-M. Primorac ^b, P. Hosemann ^a

^a Department of Nuclear Engineering, University of California – Berkeley, Berkeley, CA, USA

^b Department of Materials Physics, Montanuniversität Leoben, Jahnstraße 12, 8700 Leoben, Austria

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ABSTRACT

New $\text{Cu}_x\text{Nb}_{1-x}$ alloys were prepared using a two-step process of ball milling and subsequent high pressure torsion compacting at ambient temperature. The alloys showed a nanocrystalline structure with grain sizes between 13–26 nm for the Cu-rich samples and 11–13 nm for the Nb-rich samples. The grain sizes decreased with the milling time and the amount of Nb in the Cu-rich and Cu in the Nb-rich samples, respectively. The distortion of the crystallographic lattice of the fcc Cu and bcc Nb suggests the formation of a solid solution. The mechanical properties studied by nanoindentation show an increase of the hardness with milling time and the amount of added element. A thermal treatment was carried out under a protective Argon atmosphere. Up to 400 °C the Cu-rich samples do not show significant signs of oxidation and in increase of hardness. The Nb-rich samples showed a continuous increase of the hardness up to 500 °C, due to the precipitation of Cu, and the initial formation of NbO_x .

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1. Introduction

Copper is considered to be used in high heat flux applications in fusion energy systems where a high thermal conductivity, high strength and radiation resistance is required. However, in spite of its high thermal conductivity, it has a rather low strength [1]. Alloying Cu with different bcc elements (e.g. Fe, Cr, Ta, W, Nb) has been used for years to increase the relatively low intrinsic strength of copper [2–15], as well as to enhance the irradiation tolerance by forming specific Cu/Nb interfaces (Kurdjumov–Sachs) [16–18]. While $\text{Cu}_x\text{Nb}_{1-x}$ shows very good mechanical properties and high strength [10], as well as good electrical properties [14,19–21], its radiation tolerant properties as nanostructured multilayers are also of interest to the nuclear materials community [22–25].

$\text{Cu}_x\text{Nb}_{1-x}$ alloys show a negligibly low mutual solid solubility (0.1% Nb) [26]. However, the formation of a supersaturated Cu–Nb solid solution through non-equilibrium processing techniques, such as vapour deposition techniques or mechanical alloying, allows a solubility of up to 15 at.% Nb in Cu and up to 25 at.% of Cu in Nb. $\text{Cu}_x\text{Nb}_{1-x}$ alloys prepared by ball milling have been studied exhaustively by Botcharova et al. [10–13], and more

recently by Mula et al. [7], Lei et al. [8,9] and Banerjee et al. [21]. All these studies created a 3D-Cu/Nb interface alloy, which is in contrast to a 2D layered material as prepared typically by PVD processing, accumulative roll bonding, or cold drawn [27–31]. In the past, using small amounts of Nb in Cu was the most common approach [7–15]. The opposite, adding Cu into Nb, is less common, and only few authors have published studies of Nb-based alloys prepared by ball milling [32–34].

While the powder synthesis via ball milling is similar in the study presented here, post powder processing to create a dense solid sample via cold compacting and additional severe shear deformation has not been demonstrated previously. In this research, we have developed a novel synthesis of $\text{Cu}_x\text{Nb}_{1-x}$ alloys by combining two high deformation methods. Our technique for preparing $\text{Cu}_x\text{Nb}_{1-x}$ alloys is based on the combination of ball milling and high pressure torsion (HPT) processes, delivering compound disc shaped specimens. This HPT process has been used previously to produce bulk nanostructured materials due to a severe plastic deformation process with great success [3,35,36]. The materials produced here have been studied utilizing scanning electron microscopy (SEM), energy-dispersive X-ray spectroscopy (EDX), transmission electron microscopy (TEM), scanning transmission electron microscopy (STEM), X-ray diffraction (XRD) and nanoindentation. Furthermore, post compacting heat treatment study was conducted in order to evaluate thermal stability and improve the mechanical properties through precipitate formation.

* Corresponding author at: Department of Nuclear Engineering, University of California – Berkeley, Etcheverry Hall, 2521 Hearst Avenue, Berkeley, CA 94720-1731, USA.

E-mail address: manuel.d.abad@berkeley.edu (M.D. Abad).

2. Material and methods

2.1. Materials fabrication

The starting powders were purchased from Alfa Aesar, Johnson Matthey Catalog Company, Inc. USA as 99.999% pure powder of Cu and Nb with a mesh size of 100 for Cu and 325 for Nb. The powder milling was performed under Argon atmosphere in the Simoloyer CM01 powder processing centre located at UC Berkeley. The powders were mixed with different compositions for different milling times as shown in Table 1. The milling media consists of type 316 stainless steel balls (1 kg), with an average mass of 43 mg and an average diameter of 4.7 mm up to a rotor speed of 1200 rpm. The powder-to-ball weight ratio was kept larger than 1:10. A simplified specimen label has been used as can be seen in Table 1.

The HPT powder consolidation process has been carried out under a pressure of 7.78 GPa. In order to avoid flow of the powder mixture out of the anvils during the compaction stage, a thin 8 mm diameter copper ring was used to stabilize the powder and to keep it in position during processing. The height of the sample (powder fill) pre-deformation was ~ 2.5 mm and after deformation ~ 0.6 mm. The applied torsional rotation speed was 0.6 rpm. The applied revolutions varied between 20 and 30 while the force was kept at 385 kN. The upper plunger was cooled with compressed air during the entire HPT process. The temperature during consolidation was room temperature (23 °C). More details about the process can be found in [36,37]. These materials typically become fully dense from what it can be seen on different alloys manufactured by HPT [3,35–37]. The samples were subsequently polished, using a 1 μm diamond suspension as the last step. Initial characterisation on the as-consolidated powders was conducted as described below. Small samples from each batch were also heat treated in a tube furnace at temperatures of 200, 300, 400 and 500 °C for one hour under Argon atmosphere, at a heating rate of 20 °C/min in Ar flow (100 ml/min). Once the set point was attained, the temperature was maintained for 60 min before cooling down to room temperature.

2.2. Microstructural characterisation

A Quanta 3D field emission gun SEM with focused ion beam (FIB) has been used to manufacture STEM foils. FIB surface milling provided a clean surface for better imaging of the samples and to prepare TEM foils. A FEI Tecnai TEM was utilized to evaluate the microstructure after milling and HPT of FIB lift out foils. XRD measurements were made using Cu $K\alpha$ (1.54 Å) radiation in a Bruker Advance D8 diffractometer working at 40 mA and 40 kV and with a step size of $0.02^\circ\theta$. The grain size was evaluated by the Scherrer equation [38]. Cu $K\alpha$ peak stripping and background subtraction were accomplished by using the DIFFRAC.EVA software from Bruker.com. The lattice parameter have been calculated by the formula: $\lambda = 2d_{hkl}\sin\theta_{hkl}$, for a fixed λ (Cu $K\alpha$) = 1.54 Å. The Miller indices for the fcc Cu and bcc Nb are known, therefore the lattice parameter for Cu (111) and Nb (110) were calculated.

2.3. Nanoindentation

Nanoindentation was performed on the samples using a Micro Materials NanoTest™ (Wrexham, UK). The nanoindenter tip was calibrated using fused silica prior to the measurements. The calibration leads to an area function correcting for geometrical inconsistencies in the used diamond tip. The nanoindentation experiment was carried out using the load control mode. The thermal drift correction data was collected post-indentation. The hardness (H) and reduced indentation modulus (E_r) were calculated from the indents utilizing the Oliver and Pharr method [39]. Cu (200 nm grain size) and Nb (prepared by HPT) have been measured under the same conditions as the prepared $\text{Cu}_x\text{Nb}_{1-x}$ alloys to serve as reference materials.

3. Results

3.1. XRD of the $\text{Cu}_x\text{Nb}_{1-x}$ alloys

The $\text{Cu}_x\text{Nb}_{1-x}$ alloys have been studied by XRD. The XRD diffractograms from the $\text{Cu}_x\text{Nb}_{1-x}$ alloys together with the Cu

Table 1
Chemical composition and milling time for the different samples prepared.

wt.% Nb	wt.% Cu	Milling time (h)	at.% Cu	at.% Nb	Label after HPT ^a
5	95	10	96.5	3.5	$\text{Cu}_{97}\text{Nb}_3$ -10
9.9	90.1	20	93.0	7.0	$\text{Cu}_{93}\text{Nb}_7$ -20
10	90	30	92.9	7.1	$\text{Cu}_{93}\text{Nb}_7$ -30
15	85	30	89.2	10.8	$\text{Cu}_{89}\text{Nb}_{11}$ -30
90	10	20	14.0	86.0	$\text{Cu}_{14}\text{Nb}_{86}$ -20
95	5	10	7.1	92.9	$\text{Cu}_7\text{Nb}_{93}$ -10

^a Note: the subsequent sample name reflects the Nb content and milling time.

and Nb powders used to produce the alloy are plotted in Fig. 1a. The main peaks identified for the samples with high amount of Cu are the peaks associated with the reflection of the fcc Cu (111), (200) and (220) (JCPDS, copper file No. 04-0836). For the Nb-rich samples, the reflection of the bcc Nb (110) is observed mainly (JCPDS, niobium file No. 34-0370). No diffractions associated with Nb have been found in the Cu-rich samples, nor have Cu diffractions been found in the Nb-rich samples. This indicates that the prepared materials are within the limits of solubility of Cu in Nb and Nb in Cu, however, the XRD technique deployed here is not able to detect the presence of small amounts of undissolved element in an alloy; thus the lack of observation of a peak does not rule out the possibility of the presence of small amounts of solute elements [7,26]. A broadening and displacement of the Cu (111) and Nb (110) peaks have been observed for the different diffractograms, and thus, the grain size and change in lattice constants have been studied. The shift to lower angles of the Cu peak indicates an increase in lattice strain [7,10].

From the broadening of the main peak, average crystallite sizes were determined using the Scherrer formula. A plot of the grain size as a function of the Nb content is shown in Fig. 1b. The grain size varies between 26–13 nm for the Cu rich samples and 11–13 nm for the Nb-rich as a function of the Nb content. A decrease of the grain size is observed for the Cu rich samples when the Nb content and milling time are increased. Comparing samples with similar content of Nb ($\text{Cu}_{93}\text{Nb}_7$ -20 and $\text{Cu}_{93}\text{Nb}_7$ -30), a slight decrease of the grain size is observed, indicating that the milling time decreases the crystal size. The crystal size of the Nb-rich samples decreased with the amount of Cu and milling time. This is rather remarkable

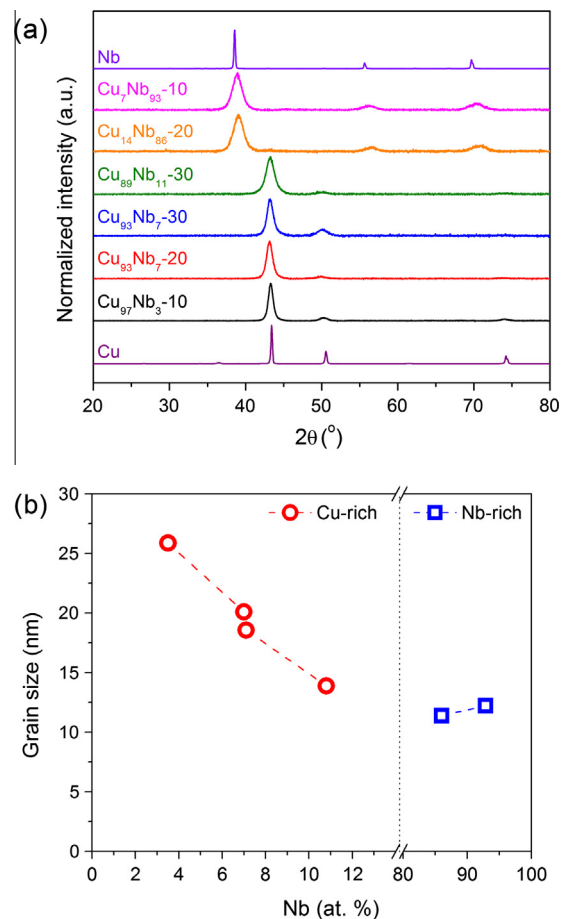


Fig. 1. (a) XRD diffractograms from the $\text{Cu}_x\text{Nb}_{1-x}$ alloys and the original powders. (b) Grain size as a function of the Nb content.

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