Microstructure-strength relationships of heavily deformed Cu-based composites

Carole Lynne Trybus
Iowa State University

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Iowa State University, 1988
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Microstructure-strength relationships of heavily deformed Cu-based composites

by

Carole Lynne Trybus

A Dissertation Submitted to the Graduate Faculty in Partial Fulfillment of the Requirements for the Degree of DOCTOR OF PHILOSOPHY

Department: Materials Science and Engineering Major: Metallurgy

Approved:

Signature was redacted for privacy.

In Charge of Major Work

Signature was redacted for privacy.

For the Major Department

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For the Graduate College

Iowa State University
Ames, Iowa
1988
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Heavily deformed or in situ composites are made from a ductile two phase mixture which is mechanically worked to relatively high deformation levels to form the composite. For example, in the Cu-Nb system, the volume fraction of Nb is kept low (less than 20 volume percent) so that the Nb is essentially isolated within the Cu matrix. Upon mechanical deformation the Nb attains a filamentary morphology parallel to the direction of working [1]. Swaging, rolling, and wire drawing can each be used to produce in situ composites. These composites combine ductility with high strength and in the case of some Cu-based composites, high electrical and thermal conductivities. Some potential applications of these materials are as magnets, spot welding electrodes, aerosurfaces for hypersonic vehicles, and as mold lining for continuous casting units.

A wide variety of in situ composites have been investigated including Ag-Cu [2,3], Cu-Fe [4,5], Cu-Cr [6,7], Cu-Mo [6], Ag-Ni [7], Ni-W [6,8], Cu-Ta [9], pearlite [10,11] as well as Cu-Nb [1,12-16]. Many methods have been used to fabricate the initial composite. Ag-Cu, Ni-W, and pearlite are all formed from the aligned products of eutectic or eutectoid reactions. Other processes include casting techniques [17,18] where the initial composite consists of an array of dendrites surrounded by a metal matrix. The 'bundle and draw' technique described by Levi [4] consists of preparing an initial compact of wires within a matrix, drawing this down and assembling this into another compact then drawing down the composite again and so forth. Powder metallurgy (P/M)
approaches have also been utilized to produce heavily deformed composites [6]. P/M techniques are comprised of mixing powders followed by a consolidation step such as press and sinter, hot isostatic pressing (HIP) or hot extrusion. Unlike solidification and phase transformation methods, the realm of P/M composites is not restricted by the phase diagram. Through proper selection of powders and consolidation method virtually any initial composite billet can be made.

In all of the systems discussed above (with the exception of Cu-\(\text{Mo}\)) tensile strengths greater than those predicted by the rule of mixtures (ROM) have been reported. A simple ROM equation based on the ultimate strengths of the fiber \(\sigma_f\) and the matrix, \(\sigma_m\), can be written as,

\[
\sigma_c = \sigma_f V_f + \sigma_m (1 - V_f)
\]

where \(V_f\) is the volume fraction of the fibrous phase and \(\sigma_c\) is the composite strength. The increases in tensile strength over the ROM prediction for heavily cold worked composites are a function of the crystal structures of the two phases. Composites with a BCC phase in a FCC matrix tend to have higher incremental increases in strength over FCC-FCC combinations [7,16]. These larger deviations from ROM by FCC-BCC combinations have been attributed to the ribbon-like cross sections developed by the BCC filaments [12]. During wire drawing of a pure BCC material the \(<110>\) direction aligns itself parallel to the wire axis leaving only 2 active slip systems which results in a plane-strain mode of deformation [19]. In the composite, this causes the BCC filaments to kink and fold about the wire axis in order to maintain compatibility between the filaments and the axisymmetrically deforming matrix.
The physical basis of the large deviations from ROM observed in in situ composites is not clear. It has been proposed [12] that the strengthening in these materials is due to opposing dislocation populations in the Cu versus the Nb. The Cu was believed to possess dislocation densities on the order of $10^{13}$/cm$^2$. This is in contrast to the Nb which was reported to be essentially free of dislocations [12]. Thus the Nb can be thought of as a thin film within a heavily dislocated Cu matrix. Another model for the large strengthening is based upon large populations of dislocations ($10^{12} - 10^{13}$/cm$^2$) in both the filamentary Nb and the matrix Cu [7,20]. The large number of dislocations is a combination of 'statistical' and 'geometrically necessary' dislocations. Statistical dislocations are the dislocations which would normally occur in a heavily worked pure metal. Additional, geometrically necessary dislocations are the dislocations which arise due to the inherent strain incompatibility between two deforming phases and are necessary in order for the two phases to deform equally in the composite.

Recently, Spitzig et al. [16] showed that the strength of heavily cold worked wires of Cu-Nb increased as spacing between the Nb filaments decreased. It was found that the composite obeyed a Hall-Petch type relationship indicating that the Nb filaments act as planar barriers to dislocation motion between the Cu and Nb. Furthermore, transmission electron microscopy (TEM) analyses [16,21] indicate that the Cu matrix undergoes a cycle of deformation-dynamic recovery-recrystallization during cold working and its dislocation density was estimated to be on the order of $10^{10}$/cm$^2$. The Nb on the other hand possessed multiple
subgrain boundaries aligned parallel to the working direction oriented about a common Nb [110] direction [21]. No evidence of Nb recovery or recrystallization was observed and the maximum dislocation density in the Nb was estimated to be 10^{10}/cm^2 [21]. Clearly, the unique microstructures which develop in BCC-FCC composites during cold working are the source of the strength increases. A full understanding of the strengthening mechanisms present in these composites is desired. The goal of this research is to characterize the microstructure-strength relationships present in heavily deformed Cu-based composites. Towards this goal, two major experimental efforts are undertaken. First of all, a consumable arc melted Cu-20 vol.% Nb casting is cold rolled into sheet. Specimens for mechanical and microstructural evaluations are obtained at various levels of deformation. These materials will be subjected to intensive microstructural characterization employing optical microscopy, scanning electron microscopy (SEM), and TEM. Secondly, the potential of P/M processed materials will be explored. Elemental Cu and Nb powders and pre-alloyed Cu-20 vol.% Nb powders are hot extruded then swaged. These P/M composites are subjected to mechanical and microstructural evaluation.

This dissertation is presented in an alternate format. It consists of five major sections each in a form suitable for publication in the scientific literature. In addition, a General Introduction and Summary and Conclusion sections are included which pertain to the whole work.
CHARACTERISTICS OF Cu-20 VOL.% Nb IN SITU COMPOSITE SHEET
PART I : STRUCTURE

C. L. Trybus, W. A. Spitzig, and L. S. Chumbley

Ames Laboratory USDOE, Iowa State University
SECTION I. CHARACTERISTICS OF Cu-20 VOL.% Nb IN SITU COMPOSITE SHEET
PART I : STRUCTURE

Abstract

The structure of a Cu-20 vol.% Nb in situ composite sheet is characterized by SEM and TEM analyses. The unusually high tensile strengths observed in this material are dependent upon the microstructures which develop during cold rolling to a true effective strain of 7.9. Nb elongates and becomes ribbon-like during deformation while the Cu matrix undergoes a cycle of deformation-dynamic recovery-recrystalization which allows for the further reduction of the Nb. The dislocation density of the Cu matrix is found to be between 1-2 x 10^{10}/cm^2 throughout the rolling process. The influence of ion-thinning Cu specimens for TEM analyses was examined and found to increase dislocation densities of annealed Cu by more than 400% but had a relatively small effect on the dislocation population of heavily worked Cu (+46%).

Introduction

Heavily deformed Cu-Nb composites are atypical composites. They are formed by mechanically working a 2 phase mixture of Cu and Nb. Rolling, swaging, and wire drawing can each be used to form the composite. The Nb elongates parallel to the wire axis or rolling direction during the deformation processing. These composites are termed in situ composites. Unlike other composites, which obey a simple "rule of mixtures" (ROM)
where the composite strength is a linear function of the volume fraction of each of its components, the strength of an in situ composite can actually exceed the strength of its strongest constituent. Figure 1 illustrates this point for Cu-20 vol.% Nb wire composite at a draw ratio or true strain of $\eta = 10.3$ ($\eta = \ln (A_0/A)$ and $A_0$ and $A$ are the initial and final cross sectional areas respectively). As the total strain increases, inordinately large deviations from ROM occur (Fig. 2) [1]. The ultimate tensile strength of the composite shows no sign of peaking out even at the highest draw ratios investigated [1]. The unusual mechanical behavior exhibited by in situ composites has been subjected to extensive research and several models have been proposed to explain this phenomenon [2-9]. These models all correlate some microstructural feature (i.e., dislocation densities or filament spacings) to the observed mechanical properties. From these studies, it can be concluded that the microstructure of heavily deformed composites plays a key role in the observed strengthening.

A recent comprehensive microstructural analysis on Cu-20 vol.% Nb wire documented that the Cu matrix underwent a cycle of deformation-dynamic recovery-recrystallization [10]. Furthermore it was found that filament spacing controlled the strength according to a Hall-Petch type mechanism [9]. Dislocation densities were estimated to be on the order of $10^{10}/cm^2$, much lower than the $10^{13}/cm^2$ predicted by other models [2]. In the present investigation, Cu-20 vol.% Nb cold rolled sheet is subjected to microstructural analysis. Cu-Nb sheet composites display unusual mechanical properties similar to wire composites. The mechanical behavior and strengthening mechanisms of these composites
Figure 1. Strength of a rod rolled and wire drawn in situ Cu-20 vol.% Nb composite and its components at a total strain of 10.3.
Figure 2. Tensile strength of heavily worked Cu, Nb, and Cu-20 vol.% Nb wires with ROM strength prediction [1]
are reported in a companion paper [11]. Here, the primary concern is structural and this analysis consists of 3 major areas. First, the gross microstructural changes in Nb morphology are established by employing scanning electron microscopy (SEM). Second, transmission electron microscopy (TEM) is utilized to evaluate Cu matrix substructure, Cu dislocation density, orientation of the Cu and Nb phases, and the internal structure of Nb filaments extracted from the composite. TEM sample preparation for Cu-Nb in situ composites is a complex process and the potential for artifacts to occlude the true structural features exists. Hence, the last part of this study will attempt to evaluate the effects of TEM sample preparation steps.

Experimental

The initial Cu-20 vol.% Nb billet was prepared by consumable arc melting of a Cu-Nb electrode as described previously [12,13]. The billet was about 7.6 cm in diameter initially. The diameter was machined to 7.4 cm and diametrically opposite flats were machined in the ingot to give a nearly rectangular cross section of 6.1 cm x 7.4 cm. The ingot was successively rolled at room temperature from 6.1 cm to a minimum thickness of 0.064 mm using reductions of about 10% per pass.

Specimens for microstructural evaluation were obtained after different amounts of reduction. Throughout this paper the amount of mechanical deformation will be given as an effective true strain, \( \varepsilon \). For sheet material this is,
\[ n_e = \frac{2}{\sqrt{3}} \ln \left( \frac{h_0}{h} \right) \]

where \( h_0 \) and \( h \) are the initial and final sheet thicknesses respectively.

The thinnest material investigated, 0.064 mm, corresponds to an \( n_e = 7.9 \) and an areal reduction of 99.9%.

Metallographic sheet sections are designated in Figure 3, A is referred to as a face section, B a longitudinal section, and C a transverse section. Both longitudinal and transverse sheet sections were prepared for SEM analysis using methods described in [10]. This procedure produced surfaces with very little topographic difference. Thus, unless otherwise noted all SEM micrographs were imaged with 100% backscattered electrons which causes the Nb to appear light and the Cu dark. SEM analysis was carried out on a Cambridge S200 SEM operated at 20 KV.

TEM sample preparation of the composite longitudinal and transverse sections was an involved process. Details are given elsewhere [14]. In brief, sheet material is cut and glued into a stack which is plated with Cu. Slices from the plated stack are cut and mechanically thinned prior to final thinning by ion milling using Ar⁺ on a liquid nitrogen cooled stage. Face sections were prepared directly from the sheet by mechanical thinning followed by ion milling.

In order to evaluate if these sample preparation steps affected the composite microstructure, separate sets of pure Cu samples were prepared for TEM examination by 2 different methods. TEM foils were prepared from pure Cu sheet which was annealed for 2 hours at 450°C in vacuum and from Cu cold rolled to \( n_e = 8.2 \). One set of Cu foils was made by
Figure 3. Scanning electron micrographs of Cu-20 vol.% Nb sheet composite at $\eta_E \sim 7$; (a) face section with the Cu etched away imaged with backscattered and secondary electrons, (b) longitudinal section, (c) transverse section, (d) schematic illustrating sheet sections
"mechanical" means, the same mechanical thinning and ion milling steps used to make the composite foils. Another set was made by a "chemical" means; jet thinning the Cu in a solution of 20 vol.% HNO₃-80% CH₃OH cooled to -25°C. A twin jet polishing device operating at 20 V and a current of about 60 mA was used. The dislocation character and densities in Cu prepared by both ways, chemical and mechanical, was evaluated and compared.

Dislocation densities were evaluated by measuring the total dislocation line length per unit volume [15]. This method requires the foil thickness to be measured. Intensity oscillations in the reflected beam of convergent beam diffraction patterns (CBDP) were utilized to measure the foil thickness [16,17]. Details and problems evaluating dislocation densities in heavily deformed composite materials by TEM are described elsewhere [18]. Dislocation densities were evaluated using brightfield images and CBDDs from a Philips 430 operating at 300 KV. All other microstructural characterizations were carried out on a JEOL 100CX operating at 120 KV. Microdiffraction patterns were obtained in TEM mode with a ~30 nm probe and a convergence half angle of 1.0 mrad.

Results

The ultimate tensile strength of the sheet composite as it varies with deformation strain is presented in Figure 4 [11]. Since mechanical properties of these composites are discussed elsewhere [11], only the pertinent mechanical characteristics will be considered here. Like wire composites [2,3,9], the sheet shows large increases in strength with
Figure 4. Tensile strength of sheet composites; arrowed points correspond to microstructures investigated.
increasing deformation. The microstructure of the as-cast material is reported in [10]. Thus, the microstructural analysis here is focused on the arrowed points in Figure 4.

**SEM: Nb morphology**

In the as-cast condition, the Nb exists as a dendritic array within a Cu matrix [9,10]. During rolling, the Nb flattens and becomes ribbon-like as revealed in Figure 3. Nb morphological changes are summarized in Figure 5. At low deformations in the transverse section, Nb appears as irregular flattened shapes while in the longitudinal sections as elongated teardrops. As the deformation intensifies, the Nb becomes flatter and can be described as undulating ribbons of irregular cross section. But even at high deformations (Figs. 5g and h) some very large Nb particles persist.

**TEM: substructure**

**Low deformation: \( \eta_r = 4.2 \)** Figures 6 and 7 typify the microstructure of the sheet rolled to \( \eta_r = 4.2 \) in the transverse section. Some areas are predominantly elongated Cu grains (Fig. 6) while others are chiefly small grains and cells with heavily dislocated walls (Fig. 7). Dark field imaging techniques were used to identify the Cu and Nb. At this low deformation, most of the Nb is still very thick (\(~ 0.4 \mu m\)) and difficult to thin uniformly with the Cu by ion-thinning,
Figure 5. Summary of SEM results; transverse sections (a,c,e,g); corresponding longitudinal sections (b,d,f,h); deformation levels, $\varepsilon_0$, 4.2 (a,b), 5.7 (c,d), 6.9 (e,f), and 7.9 (g,h)
Figure 6. Microstructure of $\eta = 4.2$ sheet, transverse section, arrows indicate a Nb filament
Figure 7. Transverse section of $\eta_e = 4.2$ sheet, compare with Figure 6, only Cu is seen here
thus Nb is not seen in all areas. In Figure 7 a large equiaxed Cu grain (~ 0.5 μm, right-hand side) along with smaller (~ 0.1-0.2 μm) equiaxed cells and grains together are indicative of dynamic recovery and recrystallization processes. Not many matrix dislocations are present and typical areas were found to contain a dislocation density of 1.0 x 10^{10}/cm^2.

Large aperture selected area diffraction patterns, SADP, (corner of Fig. 6) reveal a diffuse inner ring due to (110)_Nb and a spotty (111)Cu ring (next ring). Diffraction information from a small aperture, Figure 8, yields a Cu-Nb orientation of <110>_{Nb}||<112>_{Cu}||rolling direction (RD)||beam (B). Microdiffraction of individual Cu grains resulted in a mixture of <112> and <100> orientations within ~ 7° of B. Some grains had no particular orientation with the RD. Overall, two Cu rolling textures were identified, (110)<112> and (100)<001>.

The longitudinal section (Fig. 9) looks very similar to the transverse section in bright field, showing elongated Cu grains with high angle grain boundaries and heavily dislocated boundaries. In addition some deformation twinning was observed. Its corresponding SADP shows a much greater texturing on the longitudinal section than on the transverse one, <111>_{Cu} reflections are parallel with <110>_{Nb}. Microdiffraction of individual Cu grains yielded several grains with either <111>||B or <110>||B.

Intermediate deformation: \( \eta_b = 6.9 \) As the total rolling strain increases the Cu and Nb become lamellar in nature. Figures 10 and 11 are transverse and longitudinal sections of the Cu-Nb composite sheet
Figure 8. Illustration of Cu-Nb orientation relationship at $\eta_e = 4.2$
(a) SADP, transverse section, (b) sketched SADP
Figure 9. Longitudinal section of $n_e = 4.2$ sheet
Figure 10. Brightfield and SADP of $h_0 = 6.9$, transverse section
Figure 11. Brightfield and SADP of η = 6.9, longitudinal section
rolled to \( \eta_e = 6.9 \). At this degree of deformation Nb has a very large aspect ratio being very long and thin. Adjacent Nb filaments are separated by single and multiple blocks of Cu. Cells are not common in the Cu but small equiaxed strain-free grains are observed like those in the lower left hand corner of Figure 10. The dislocation density of the Cu is about twice as great as after rolling to \( \eta_e = 4.2 \) i.e., \( 2.0 \times 10^{10}/\text{cm}^2 \). In the longitudinal section, Nb is slightly curved unlike its straight appearance in the transverse section. Another difference between the two views is that small twins (c.f. top of Fig. 11) are not observed on the transverse sections. Comparison of the two SADPs denote textural differences. In the transverse view (Fig. 10) both the Cu and Nb rings are spotty with several spots grouped near the \(<111>_{\text{Cu}}\) and the \(<110>_{\text{Nb}}\). All of the following Cu orientations were found in microdiffraction; \(<110>||\text{RD}, <112>||\text{RD}, \) and \(<100>||\text{RD}. Many other rational and irrational orientations were found and no obvious Cu orientation trend was detected. On the other hand, the SADP of the longitudinal section (Fig. 11) is very regular and similar to the longitudinal sections of the \( \eta_e = 4.2 \) material, \(<110>_{\text{Nb}}||<111>_{\text{Cu}}\). Microdiffraction evaluation of the Cu yielded identical results as the \( \eta_e = 4.2 \) sample. Examination of the face section, Figure 12, disclosed the Cu rolling texture \(<112><110>\). Other textures found were \(<100><001>\) and \(<110><112>\).

**High deformation: \( \eta_e = 7.9 \) TEM observations of sheet rolled to \( \eta_e = 7.9 \) can be summarized by Figures 13 and 14. As at the previous deformation level, the Cu and Nb appear lamellar. However, the Cu
Figure 12. Face section of sheet rolled to $n_e = 6.9$ illustrating $(112)(110)$ Cu rolling texture, arrow indicates the rolling direction (RD) and Cu $<220>$
Figure 13. Typical transverse section of the $\eta_{e} = 7.9$ sheet
Figure 14. TEM image of a transverse section at $H_e = 7.9$ showing twinned Cu
between the Nb is predominantly single grains and neither cells nor equiaxed Cu grains are observed. Twinned Cu (Fig. 14) was plentiful in the transverse sections at this deformation only. The SADP is of \( B = [110]_{\text{Cu}} \) and contains the extra reflections indicative of twinning. The measured dislocation density for this material was \( 1.6 \times 10^{10}/\text{cm}^2 \).

Wide aperture SADPs of this sample (Fig. 13 corner) indicate the strong texturing of \( \langle 110 \rangle_{\text{Nb}} || \langle 111 \rangle_{\text{Cu}} \). Consistently the \( \langle 110 \rangle_{\text{Cu}} || B \) was found in microdiffraction of single Cu grains on the transverse section. Figure 15 is a microdiffraction pattern of Cu and Nb as viewed on a transverse section which illustrates \( \langle 110 \rangle_{\text{Nb}} || \langle 111 \rangle_{\text{Cu}} \) with \( \langle 110 \rangle_{\text{Cu}} || \text{RD} \).

Extracted Nb filaments Nb filaments were extracted from the composite by dissolving the Cu away [14]. Filaments extracted from sheet rolled to \( \eta_e = 6.9 \) and \( \eta_e = 7.9 \) are shown in Figures 16 and 17. At intermediate deformation levels dislocations are randomly arranged (Fig. 16). As the deformation is increased, the dislocations rearrange into low angle dislocation boundaries parallel to the \( \langle 110 \rangle_{\text{Nb}} \). Previous studies of Nb filaments extracted from wire and sheet [10,19] also reported boundaries aligned along \( \langle 110 \rangle_{\text{Nb}} \) with misorientations about a common \( \langle 110 \rangle_{\text{Nb}} \) of \( 2^\circ \) to \( 35^\circ \).

Many filaments have a \( \langle 100 \rangle <011> \) rolling texture as the SADP in Figure 16 shows. The ideal Nb rolling texture, \( \langle 113 \rangle \langle 110 \rangle \) [20] was also observed and it is displayed by the microdiffraction pattern in Figure 17.
Figure 15. Cu–Nb orientation relationship at $\eta = 7.9$ (a) Microdiffraction of Cu and Nb from the transverse section, (b) sketched SADP
Figure 16. Nb filament extracted from $n_e = 6.9$ composite
Figure 17. Nb filament extracted from $n_e = 7.9$ composite
Results from TEM analysis of pure Cu are presented in Figures 18-21 and summarized together with composite data in Table 1. Included in Table 1 are $\bar{T}_f$, the average foil thickness used to evaluate dislocation densities and $\bar{s}$, the average barrier spacing of the composite which counts both high angle Cu grain boundaries and Cu-Nb boundaries. Comparison of the annealed Cu samples, jet thinned (Fig. 18) versus ion milled (Fig. 19), shows that an increase in the number of dislocations in the ion milled sample is apparent. In the worked Cu (Fig. 20 vs. 21), the difference is not obvious to the eye and measurements reveal that the dislocation content of the two types of specimens are close (Table 1).

Discussion

Microstructural characterization

The microstructure of Cu-Nb cold rolled to high deformation strains possesses a myriad of features each of which contributes to the macroscopic properties. Throughout the deformation the microstructure is changing and these changes are quantified in Table 2. As the deformation level increases, Cu grain and cell size decrease as does the Nb thickness and spacing. The structural features change too, from a mixture of cells and grains to an absence of cellular structures at high deformation. This can be explained in terms of a cycle of deformation-dynamic recovery-recrystallization. Previous results on cold drawn Cu wire established that dynamic recovery starts to occur at total strains
Figure 18. Annealed Cu, jet thinned
Figure 19. Annealed Cu, ion-thinned
Figure 20. Cu rolled to $\eta_e = 7.0$, jet thinned
Figure 21. Cu rolled to $\eta = 8.2$, ion-thinned
Table 1. Summary of dislocation densities

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Dislocation Density ( \text{cm}^{-2} )</th>
<th>( \overline{\gamma} ) ( \text{A} )</th>
<th>( \overline{\delta} ) ( \text{A} )</th>
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<td>Annealed Cu jet thinned</td>
<td>( 4.5 \times 10^9 )</td>
<td>1342</td>
<td></td>
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<tr>
<td>Annealed Cu ion milled</td>
<td>( 2.6 \times 10^{10} )</td>
<td>1481</td>
<td></td>
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<tr>
<td>Cu ( \eta_e = 8.2 ) jet thinned</td>
<td>( 1.3 \times 10^{10} )</td>
<td>2031</td>
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<td>Cu ( \eta_e = 8.2 ) ion milled</td>
<td>( 1.9 \times 10^{10} )</td>
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<td>Cu-Nb sheet ( \eta_e = 4.2 )</td>
<td>( 1.0 \times 10^{10} )</td>
<td>1953(^a)</td>
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<td>Cu-Nb sheet ( \eta_e = 6.9 )</td>
<td>( 2.0 \times 10^{10} )</td>
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<tr>
<td>Cu-Nb sheet ( \eta_e = 7.9 )</td>
<td>( 1.6 \times 10^{10} )</td>
<td>1838</td>
<td>520</td>
</tr>
</tbody>
</table>

\(^a\)Minimum \( \overline{\gamma} \) was 1880 \( \text{A} \).
Table 2. Quantitative substructure characterization of Cu and Nb from TEM analyses

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Total Strain, ( n_e )</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>0</td>
</tr>
<tr>
<td>Cu cell size, ( \mu m )</td>
<td>--</td>
</tr>
<tr>
<td>Cu grain size, ( \mu m )</td>
<td>50(^b)</td>
</tr>
<tr>
<td>Nb spacing, ( \mu m )</td>
<td>24.8(^b)</td>
</tr>
<tr>
<td>Nb thickness, ( \mu m )</td>
<td>6.2(^b)</td>
</tr>
</tbody>
</table>

\(^a\)Not observed.

\(^b\)Optical analysis.

\(^c\)SEM.
of ~1.2 and recrystallization at 3.0 [21]. Thus at a total strain of 4.2 in the sheet, recovery and recrystallization mechanisms are well underway (Figs. 6 and 7). Upon raising the deformation level, the recrystallized material starts the cycle again by deforming then dynamically recovering and finally recrystallizing again. Driving this process is the reduction of energy. With deformation comes a large build-up of dislocations which subsequently tangle and begin grouping into cellular walls. Eventually within these walls, annihilation of redundant dislocations occurs [22] and the walls become sharper. The continuation of the process leads to further microstructural refinements in the Cu matrix and Nb spacing and thickness (Table 2). The appearance of nearly dislocation free equiaxed subgrains in Cu is typical of the low energy dislocation structures observed in stage IV deformation of Cu [23]. Therefore, since Cu in Cu-Nb composites shows similar structures to that in pure Cu [10], the presence of the Nb filaments does not appear to prevent dynamic recovery and recrystallization in the Cu matrix which was postulated as the reason for the exponential strengthening in Cu-Nb [3,5].

Measurements of Cu grain size from pure Cu and from Cu-Nb wires [9] yielded higher values than those observed in the sheet. At a total strain of 6.9 in the wire, the average Cu grain size was 0.23 μm [9] versus 0.072 μm in the sheet. Accompanying the decreased grain size in the sheet is a smaller Nb filament spacing. Thus the total area of Cu-Nb boundaries are increased in the sheet as compared to the wire for a given deformation. The lack of cells in the sheet Cu matrix at high deformations could be due to the increased proximity of the Cu-Nb
boundaries which act as effective sinks for dislocations because the shear modulus of Cu, $G_{\text{Cu}} = 48.3 \text{ GPa}$, is higher than that of Nb, $G_{\text{Nb}} = 37.5 \text{ GPa}$ [24]. Analysis of image forces on a screw dislocation in a two-phase lamellar structure [25] indicates that for Cu-Nb where $G_{\text{fiber}} < G_{\text{matrix}}$ dislocations are attracted to the fiber-matrix interfaces. Because Cu-Nb interfaces are more potent attracters of dislocations than the Cu-Cu boundaries (due to the difference in $G$) as the Cu-Nb interfacial area grows with deformation, Cu-Cu cellular walls are not needed: dislocations prefer to move to Cu-Nb interfaces. Thus, the amount of Cu-Cu cellular structure is drastically reduced at higher deformations.

Throughout the deformation, measured dislocation densities were fairly constant, $1-2 \times 10^{10}/\text{cm}^2$ in contrast to $10^{12}$-$10^{13}/\text{cm}^2$ predicted by other workers [2,4,5]. Dislocation populations are not uniformly distributed throughout the Cu matrix. Cell walls contain possibly up to an order of magnitude greater number of dislocations $\sim 10^{11}/\text{cm}^2$. In comparison, the dislocation density of new strain-free grains is much lower $\sim 10^8$-$10^9/\text{cm}^2$. The areas measured are typical of heavily dislocated areas in Cu: the maximum dislocation densities in the Cu exclusive of cell walls.

High dislocation densities increase the total energy of a material and when dislocations get closely spaced annihilation mechanisms will occur in an effort to lower the total system energy [26]. Theoretical estimates of the maximum dislocation densities possible range from $4 \times 10^{10}/\text{cm}^2$ for screw dislocations to $4 \times 10^{13}/\text{cm}^2$ for edge dislocations [26]. Experimental observations support much lower values of $6 \times$
$10^{11}/\text{cm}^2$ for edge dislocations in cell walls and up to $4 \times 10^8/\text{cm}^2$ for screw dislocations inside cells [26]. These values are in reasonable agreement with our observations for Cu-Nb in situ sheet composites. Heavily worked materials exhibit low energy dislocation structures as typified by equiaxed subgrains with low interior dislocation densities [23]. In other words in highly deformed materials, dislocations move into cellular structures to reduce the long-range stresses and thus the overall system energy [23,26,27]. As previously stated, these cellular structures give way to new strain-free grains.

Twinning was observed in the Cu at all deformation levels examined in the Cu-Nb sheet and was most prevalent at the highest total deformation, $\eta_e = 7.9$. Deformation twinning is not common in pure FCC metals at room temperature but it does occur at low temperatures where high stresses are required for plastic deformation and in some FCC solid solutions twinning occurs readily at room temperatures or higher [28]. The Cu in Cu-Nb contains about 1 atomic % Nb [29] which is polyvalent and thus very effective at lowering the SFE (stacking fault energy) of Cu and enabling deformation twinning to occur at lower twin stresses. Deformation twinning has been observed in other low SFE materials subjected to high deformation strains as well [30]. Therefore, the observed twinning in the heavily deformed Cu is not unusual.

Ideal rolling textures of pure Cu and Nb are $\{110\}<112>$ and $\{113\}<110>$ respectively [31,20]. Both these rolling textures were observed in Cu and Nb via TEM. Textures identified by TEM do not yield the overall texture of the material but they are guidelines to the trends exhibited in the composite. At higher deformations, a secondary
texture $(112)<111>$, starts to form in Cu [31]. In the Cu-Nb sheet at $\eta_e = 4.2$, two Cu rolling textures were identified $(110)<112>$ and $(100)<001>$, the latter texture being the recrystallization texture of Cu [31]. This is further evidence that dynamic recovery and recrystallization mechanisms are occurring in the Cu during rolling. At intermediate deformation, $\eta_e = 6.9$, Cu exhibits many textures: $(110)<112>$, $(112)<110>$, and $(100)<001>$ as well as several others. No particular texture dominates the Cu. But at the highest strain, $\eta_e = 7.9$, the $<110>_{Cu}||RD$ is consistent, i.e., within $5^\circ$ and the rolling textures are $(112)<110>$ and $(100)<001>$. The $(112)<110>$ rolling texture found in Cu at $\eta_e = 6.9$ is not the expected secondary Cu texture. This difference could be due to Cu-Nb orientations in the composite. Longitudinal sections reveal a $<110>_{Nb}||<111>_{Cu}$ at all strain levels while transverse sections show $<110>_{Nb}||<112>_{Cu}$ at $\eta_e = 4.2$ which gradually becomes $<110>_{Nb}||<110>_{Cu}$ at $\eta_e = 7.9$. The textural changes are summarized in Figure 22.

Nb textures remain fairly constant throughout the deformation processing, $<110>_{Nb}||RD$. Extracted filaments yielded both $(113)<110>$ and $(001)<110>$ which is another common texture in rolled Nb.

**Effect of TEM sample preparation**

Results from dislocation density analysis indicate that ion milling does not reduce the dislocation population in Cu-Nb composites. In the annealed Cu, a significant increase in the dislocation density in the ion milled sample was recorded but for the worked Cu the difference between the two was small. The upper left-hand area of Figure 21 shows
Figure 22. Schematic stereographic projections illustrating orientation relationships between Cu and Nb.
several dislocation loops which are not present in jet thinned materials. These loops are most likely due to ion milling damage. Duker and Schlette [32] report that dislocation loops and dense tangles result from ion milling Al and Cu foils respectively. Ion mill damage to several other metallic alloys has been documented [33]. The effect of ion milling on the TEM foils of Cu-Nb composites may increase the dislocation population slightly but does not reduce it.

Other potential problems of evaluating dislocation density are accurate foil thickness measurement and dislocation rearrangement and relief in thin foils. Foil thicknesses measured using CBDP have accuracies of ±2-5% [16,17]. Dislocations in thin films may shorten their lengths by rotating so they lie normal to the foil surface [34]. Hirsch et al. [15] suggest this error can be reduced by employing circles instead of straight lines for point counting, which was done in our evaluations. The error could be further reduced by counting surface intersections instead of dislocation line length but this method is not applicable to dislocation densities greater than $3 \times 10^9$/cm$^2$ [35]. Finally, dislocation relief due to image force attraction to the foil surface is minimal in this analysis because bulk internal barrier spacing was smaller than foil thickness in all areas of the composite analyzed. Thus, most dislocations are closer to bulk internal interfaces than to the foil surface and therefore more likely to migrate to these interfaces in the bulk prior to thinning. Accounting for all these errors yields a total error of about 25% [34].

Clearly our observations of dislocation densities in Cu-Nb in situ composites are not in accord with previous estimations of $10^{12}-10^{14}$/cm$^2$
These calculated densities are significantly larger than estimated maximum dislocation densities in Cu of about $5 \times 10^{11}/\text{cm}^2$ even after very large strains [36]. Furthermore, care was taken to eliminate large errors in the measurement of the dislocation densities and it was demonstrated that ion milling does not reduce dislocation populations. Both theoretical considerations and direct experimental observations [23,26] are in agreement with our data. Thus, in light of the above discussion, dislocation densities in Cu-Nb in situ composite sheet are on the order of $10^{10}/\text{cm}^2$ and the presence of the Nb filaments does not prevent dynamic recovery and recrystallization in the Cu matrix. These two observations are in direct conflict to models based on large accumulations of dislocations for strengthening in in situ composites [5,6].

Conclusions

1. During rolling of Cu-Nb alloys the Nb elongates parallel to the rolling direction and becomes ribbon-like with increasing deformation levels.

2.Spacing between the Nb filaments and Nb filament thickness are reduced with increased deformation.

3. In the course of the rolling operation Cu undergoes a cycle of deformation-dynamic recovery-recrystallization. No such process was evident in the Nb.

4. Both ideal Cu $\{110\}<112>$ and Nb $\{113\}<110>$ rolling textures were observed. In addition the textures $\{112\}<110>_{\text{Cu}}$ and $\{001\}<110>_{\text{Nb}}$
were present. Textures in the Cu and Nb phases in the composite appear to be similar to those found in heavily deformed pure Cu and Nb.

5. Ion milling of Cu was shown to produce additional defects which was very pronounced in annealed Cu but insignificant in heavily deformed Cu sheet.

6. Low energy dislocation structures were observed in Cu, similar to stage IV low energy structures found in heavily deformed pure metals.

7. Dislocation densities of the Cu matrix were found to be on the order of $10^{10}/\text{cm}^2$.

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CHARACTERISTICS OF Cu-20 VOL.% Nb IN SITU COMPOSITE SHEET
PART II: MECHANICAL BEHAVIOR

C. L. Trybus and W. A. Spitzig

Ames Laboratory USDOE, Iowa State University
SECTION II. CHARACTERISTICS OF Cu-20 vol.% Nb IN SITU COMPOSITE SHEET
PART II: MECHANICAL BEHAVIOR

Abstract

The mechanical properties have been evaluated for a heavily cold rolled Cu-20 vol.% Nb composite. The strength of the rolled composite dramatically increases with increasing rolling deformation up to a true strain of 6.9, the maximum investigated. Longitudinal and transverse specimens both possessed equivalent mechanical properties. During rolling, Nb is formed into ribbon-like filaments that are perfectly aligned parallel to the sheet surface that is, in two directions longitudinal and transverse. These results are compared to those for a wire drawn Cu-20 vol.% Nb composite. The strengthening in rolled and wire drawn Cu-Nb correlates with the amount of deformation in a similar manner even though during wire drawing the Nb filaments curl about the wire axis and, therefore, are only aligned in the longitudinal direction. As a consequence of the Nb morphological difference in the sheet versus the wire composite, strengthening in the sheet does not show a Hall-Petch relationship, as the wire does, but a much weaker dependence on Nb filament spacing. Strengthening in the rolled composite is in accord with a modified rule of mixtures model but this model cannot account for the observed strengthening in wire drawn Cu-Nb.
Introduction

In situ composites prepared by extensive cold working of ductile two phase mixtures constitute a class of interesting composites because of their high strengths [1-4]. During deformation processing both phases in the composite equally deform resulting in a microstructure of filaments with large aspect ratios in a matrix material. The high strengths result because the individual phases undergo large deformations and extensive work hardening, and because the microstructural scale and spacing of the phases is drastically reduced as the mechanical work intensifies. Other investigations [4,5] quantitatively characterized the mechanical properties along with the filament sizes and spacings and with the filament and matrix substructures for Cu-Nb composites rod rolled then wire drawn to true strains up to about \( \eta = 11.9 \) (\( \eta = \ln \left( \frac{A_0}{A} \right) \), where \( A_0 \) and \( A \) are the initial and final cross sectional areas). Strengthening in these wire drawn composites appears to arise from the Nb filaments acting as planar barriers to dislocation motion and is governed by the interfilamentary spacing in accord with a Hall-Petch relationship [6,7]. These wire drawn Cu-Nb composites exhibited work hardening behavior and strength-filament correlations akin to those observed in cold worked pearlite [8,9].

Results from experiments on cold worked pearlite indicated that the mode of deformation, wire drawing (axisymmetric deformation) or strip drawing (plane strain deformation), produced similar work hardening behavior and strength dependency on the reciprocal square root of the
pearlite spacing [8,9]. These analogous properties occurred in spite of
the fact that the average cementite spacing varied as the square root of
the plate thickness in strip drawing but in direct proportion to the
diameter in wire drawing.

It is the intent of this paper to evaluate the mechanical properties
and to quantitatively characterize the filament sizes and spacings for
an in situ prepared Cu-20% Nb composite cold rolled up to 99.9%
reduction in thickness. The matrix and filament substructures developed
in these composites during cold rolling are reported in an accompanying
paper and only some of the highlights will be discussed here [10]. The
results are compared with those obtained previously [4,5] on Cu-Nb
composites deformed by wire drawing.

Materials and Procedures

An ingot of Cu containing 20 vol.% Nb was prepared by consumable arc
melting of an electrode containing Nb strips in a Cu cylinder, as
described in [11,12]. The fabrication steps employed to form sheet
material from the cast billet are given in [10].

In order to compare the mechanical properties of specimens obtained
from rolled and wire drawn in situ composites it is necessary to compare
them at equivalent $\eta$ values. Because rolling involves plane strain
deformation while wire drawing involves axisymmetric deformation it is
appropriate to evaluate an effective total strain, $\eta_e$ for the rolled
material which is comparable to the $\eta$ obtained for axisymmetric
deformation when comparisons between rolled and wire drawn Cu-Nb are
made [13]. Therefore, \( \eta \) for the sheet material is taken as \( 2/\sqrt{3} \eta \) where \( \eta = \ln (h_0/h) \) and \( h_0 \) and \( h \) are the initial and final thicknesses of the sheet. However, the comparison is not exact because the mode of deformation is changed in using tension tests on previously rolled material as compared to wire drawn material. This can have an influence in the resulting strengthening behavior in the tensile deformation of rolled sheet [14,15]. The thinness of the heavily deformed sheet precluded performing plane strain compression tests on the composite.

Tensile specimens were machined from as cast and rolled material at various levels of deformation. Their gage section was about 0.5 cm wide by the sheet thickness and the gage length was about 2.8 cm. All tensile tests were done at 295 K using a nominal strain rate of \( 1.7 \times 10^{-4} \) sec\(^{-1}\). A 2.5 cm strain gage extensometer with a strain resolution of \( 5 \times 10^{-5} \) was used for elongation measurements on specimens with thicknesses down to 0.28 mm (\( \eta = 5.4 \)). Machine stiffness corrections were made for the thinner sheet specimens to get more accurate elongation data. Both longitudinal (parallel to rolling direction) and transverse (perpendicular to the rolling direction) tensile specimens were analyzed down to a sheet thickness of 0.15 mm. Figure 1 identifies the sheet planes A, B, and C which are referred to as the longitudinal, transverse, and face planes respectively. At smaller sheet thicknesses only transverse specimens were tested because of a limited amount of material.

Filament sizes and spacings were measured on both transverse and longitudinal sections using standard stereographic intercept procedures [16] on micrographs taken on a metallograph or a SEM for the thinner
\[ \eta_e = 5.0 \]

Figure 1. Cu-20\% Nb rolled to an \( \eta = 4.3 \) (\( \eta_e = 5.0 \))
rolled specimens. The specimen preparation procedures and experimental conditions have been previously reported [10,17].

Experimental Results

**Mechanical properties**

Figures 2 and 3 show the stress-strain curves of the rolled specimens receiving reductions up to $\eta = 6.9$ prior to tensile testing. These results illustrate that the longitudinal and transverse specimens have similar strengths although the initial work hardening behavior is greater for the longitudinal specimens. The uniform elongation appears to be slightly greater for the transverse specimens. The elastic line has a slope of $115 \pm 5$ GPa which is about the same as that measured for Cu. There was no evidence that the elastic modulus increased with increasing mechanical working to an $\eta = 5.4$, which was the thinnest sheet specimen where a strain gage extensometer could be used. Comparable results were obtained on Cu-20% Nb composite wire drawn to $\eta = 10.3$ [4].

Because of the pronounced work hardening that occurs during initial straining in the rolled and wire drawn specimens, the ultimate tensile stress rather than an offset stress was taken as a measure of strength for comparison purposes. The ultimate tensile strengths of the longitudinal and transverse rolled specimens are given in Figure 4. The differences between the strengths of these specimens are small and
Figure 2. True stress-true strain curves of longitudinal specimens of Cu-20% Nb rolled to various reductions ($\eta$)
Figure 3. True stress-true strain curves of transverse specimens of Cu-20% Nb rolled to various reductions ($\eta$)
Figure 4. Effect of rolling reduction on the ultimate tensile stress of Cu-20% Nb
within experimental scatter except at the smallest \( \eta \)'s where the longitudinal specimens were consistently slightly stronger than the transverse specimens.

Figure 5 compares the ductility of the rolled and wire drawn composites. The dashed lines in Figure 5 reflect the experimental data for a wire drawn Cu-20\% Nb composite [4]. The uniform elongation is severely decreased by cold working (rolling or wire drawing) although it appears to remain relatively constant over a large range of mechanical deformation. The composites have greater uniform elongations than pure Cu at a given degree of deformation [4]. The fracture strain in both the rolled and wire drawn composites appears to decrease in a consonant linear manner with increasing mechanical deformation.

**Fracture characteristics** Fracture surfaces of longitudinal and transverse specimens of the composite rolled to \( \eta = 5.4 \) are shown in Figure 6. These fractures are typical of those observed in the rolled composite. The fracture surfaces of longitudinal and transverse specimens are alike and the dimples on the fracture surfaces do not bear any obvious relationship to the aligned filament morphologies on planes parallel to the fracture planes. However, etched fracture surfaces indicate that the filaments are concentrated in the rims of the dimples. As \( \eta \) is increased the fracture surfaces continue to exhibit dimpled fracture with the dimples becoming smaller and shallower. The decreasing dimple sizes correspond to the decreasing average spacings between the Nb filaments with higher rolling deformation.
Figure 5. Effect of rolling reduction ($\eta_e$) on the ductility of the sheet composite
Figure 6. SEM images of fracture surfaces of (a) transverse and (b) longitudinal tensile specimens of Cu-20% Nb rolled to a reduction of $\eta = 5.4$. 
Microstructure

The three dimensional nature of the Nb filaments after rolling is evident in Figure 1. The filamentary nature of the Nb is apparent in both the longitudinal and transverse planes. Figure 7 reveals the three dimensional characteristics of the Nb filaments after wire drawing to an η of 5.3. While the longitudinal plane (A) shows a fibrous structure which resembles that observed for the rolled composite in this plane (Fig. 1) the Nb filaments in the transverse section (B) have an irregular kinked shape. This difference in Nb filament morphology on transverse sections of Cu-Nb composites undergoing plane strain (Fig. 1) versus axisymmetric (Fig. 7) deformation is a consequence of the <110> wire texture developed in BCC metals during wire drawing [18]. Corresponding differences in structure morphology on transverse sections resulting from the mode of deformation are observed in pearlite [9,19]. As the deformation level rises, the Nb filaments are continuously reduced in thickness and they become more aligned with the rolling or drawing direction.

Nb filament sizes and spacings The effect of increasing mechanical deformation on decreasing the thickness, T, and spacing, X, of the Nb filaments in the rolled composites is displayed in Figure 8. Included in this figure are data for a wire drawn composite [4]. The spacing of the filaments was about 25 μm in the initial composite casting. At the larger values of η for the rolled and wire drawn composites, the average filament thicknesses are less than 0.1 μm. Nonetheless, TEM analyses show that some very fine filaments are present which are not resolved in the SEM [10]. Although neglecting these very
Figure 7. Cu-20% Nb wire drawn to an $\eta = 5.3$. 
Figure 8. Effect of rolling reduction ($\eta_b$) or draw ratio ($\eta$) on the spacing ($\lambda$) and thickness ($\bar{t}$) of Nb filaments in Cu-20% Nb.
fine filaments in evaluating the averages in Figure 8 biases them toward larger values, this should not have any significant effect in the trends shown for the filament sizes and spacings in Figure 8.

It is clear in Figure 8 that the sizes and spacings of the Nb filaments decrease much more rapidly with increasing degree of rolling as compared to wire drawing. However, at the larger rolling reductions the filaments are less uniform in thickness in the rolled composite as compared to those in the wire drawn composite [4,10]. The three dimensional morphology of the Nb filaments in the rolled composite can be described as undulating ribbons of irregular cross section [10]. Comparison of Nb filaments in rolled and wire drawn composites shows that individual filaments in the rolled material have greater variability in their thicknesses [10].

Discussion of the Results

Mechanical properties

The synonymity in tensile behavior with deformation for the rolled or the wire drawn composite indicates that the mode of large strain deformation, plane strain or axisymmetric had no appreciable effect on the substructures developed during mechanical processing. TEM analyses of rolled [10] and wire drawn [5] Cu-20% Nb confirm this conclusion. These direct comparisons of the tensile properties of rolled composites with those of wire drawn composites have to be viewed with some caution because the deformation process is reversed for the rolled composites during tensile deformation [14,15]. However, for heavily rolled Cu,
subsequent plane strain compression tests are in good agreement with tensile tests when compared on an equivalent strain basis [20]. Tests on rolled Cu showed that the strengths were the same as those attained on wire drawn Cu [4] at equivalent $\varepsilon_e$ values. Therefore, comparisons between tensile tests of longitudinal specimens of rolled or wire drawn Cu-20\% Nb composites should be meaningful.

The more pronounced initial work hardening rate in the longitudinal tensile specimens as compared to the transverse tensile specimens of the rolled material is probably due to texture development in the composite. Table 1 summarizes the textural differences between the longitudinal and transverse tensile specimens as identified in TEM [10]. The Cu-Nb orientation with respect to the tensile axis of the longitudinal specimens changes during the deformation while that of the transverse specimens remain fairly constant. The equivalence of strength for the longitudinal and transverse specimens is highly desirable and unusual for rolled material and in particular for composites. The closeness of strength for both longitudinal and transverse directions is most likely a result of the Nb filament structure being similar on both transverse and longitudinal planes. It seems that the aspect ratios of the filaments on the transverse sections are large enough to produce optimum strengthening [21].

A comparison between the strengths of the rolled and wire drawn tensile specimens at comparable reductions is given in Figure 9. Averages of the longitudinal and transverse values were used for the rolled specimens. At effective true strains up to 6.3 tensile specimens of rolled and wire drawn composites have identical strengths. At
Table 1. Textural differences between longitudinal and transverse tensile specimens as observed in TEM [10]

<table>
<thead>
<tr>
<th>Cu and Nb orientations with respect to the tensile axis, T</th>
<th>( \eta )</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>3.6</td>
</tr>
<tr>
<td>Longitudinal tensile specimens</td>
<td>(&lt;112\text{Cu}</td>
</tr>
<tr>
<td>Transverse tensile specimens</td>
<td>(&lt;111\text{Cu}</td>
</tr>
</tbody>
</table>

^aNo predominant texture found.

^bRecrystallization texture of Cu.

^cSame texture as \( \eta = 3.6 \) transverse tensile specimen.
Figure 9. Ultimate tensile strength of Cu-20% Nb as a function of deformation, $\eta_e$ for rolled material and $\eta$ for wire drawn material.
greater effective true strains the rolled composite appears to be slightly stronger than the wire drawn composite. Further reductions of the sheet were possible but impractical because of the difficulty in performing reliable tensile tests on sheet less than 0.064 mm thick, the minimum thickness tested ($\eta = 6.9$). Figure 9 shows that both the rolled and wire drawn tensile specimens exhibited exponential work hardening behavior akin to that observed in strip drawn and wire drawn pearlite [8,9].

**Microstructure characterization**

The effect of cold working on the Nb filament spacings in rolled and wire drawn Cu-20% Nb composites is replotted in Figure 10. Data for pearlitic steels are included in this figure [9]. For the rolled composite the spacings are plotted for both $\eta$ and $\eta_s$. The greater effect of rolling as compared to wire drawing on decreasing the filament spacing is clearly evident. The data for pearlite [9] are much less extensive and appear to scatter in between those for the rolled and wire drawn composites. The equations of the lines through the Cu-20% Nb composite data in Figure 10 are

$$\ln \left( \frac{\lambda}{\lambda_0} \right) = -0.64 \eta_s$$

(1)

for the rolled composite and

$$\ln \left( \frac{\lambda}{\lambda_0} \right) = -0.36 \eta$$

(2)

for the wire drawn composite.

If the decrease in filament spacing was in direct proportion to the sheet thickness ratio or the wire diameter ratio the coefficients in
Figure 10. Effect of rolling reduction or wire draw ratio on the spacing of Nb filaments in Cu-20% Nb, effective strain ($\eta_e$) is also plotted for the rolled specimens for direct comparison with the results for wire drawn material.
equations (1) and (2) would be 0.87 and 0.5, respectively. Therefore, the internal shape changes do not exactly coincide with the external shape changes, that is, the average Nb filament spacing is reduced more slowly than the sheet thickness in rolling or wire diameter in wire drawing. In pearlite [9], the internal and external shape changes were similar during wire drawing but exhibited a greater discrepancy than observed for the Cu-20\% Nb composite during strip drawing. It was proposed that strain inhomogeneity was the cause of the reduced internal shape change during strip drawing of pearlite [9]. However, there was no evidence of localized shear bands in the rolled Cu-20\% Nb composite [10]. A plausible explanation for the discrepancy between external and internal shape changes in Cu-Nb composites is that the Nb filament population is continuously evolving during mechanical processing. The coefficients 0.87 and 0.5 for rolled and wire drawn processing assume no new barriers are created or old ones destroyed [8].

Substituting the rolled and wire drawn composite dimensions for \( \eta_c \) and \( \eta_t \), respectively, in equations (1) and (2) yield

\[
\frac{X}{X_0} = (h/h_0)^{0.74} \tag{3}
\]

for the rolled composite and

\[
\frac{X}{X_0} = (d/d_0)^{0.72} \tag{4}
\]

for the wire drawn composite, where \( h_0 \) and \( h \) are the initial and final thicknesses of the rolled composite and \( d_0 \) and \( d \) are the initial and final diameters of the wire drawn composite. For both modes of deformation the exponent is about \( 1/\sqrt{2} \) and Figure 11 demonstrates the correlation between filament spacing and sheet and wire dimensions for
Figure 11. Correlation between Nb filament spacing and the thickness of rolled sheet or the diameter of wire drawn Cu-20% Nb
the Cu-20% Nb composites. Included in Figure 11 are the data for pearlitic steels [9] which show appreciable scatter about the Cu-20% Nb correlation. The results for the Cu-20% Nb composite indicate that the internal shape changes correlate with the external shape changes in a similar manner in both rolling and wire drawing but they are less than the imposed external shape change.

Microstructure-strength correlations

Figure 12 illustrates the dependence of the ultimate tensile strength on average values of filament thicknesses (T) and spacings (λ) for rolled and wire drawn Cu-20% Nb. For the large range of Nb filament spacings obtained by rolling and wire drawing the data clearly show a drastic difference of strength dependence on filament spacing for these two modes of deformation. The slopes of the lines in Figure 12 are about -1/4 and -1/2 for the sheet and wire composites respectively. As reported previously, the strength of wire drawn Cu-Nb in situ composites satisfies a Hall-Petch [6,7] relationship with filament spacing, that is, mean free path in Cu. The ultimate tensile strength correlates well with (λ)^-1/4 and (λ)^-1/2 for the rolled and wire drawn composites, respectively (Fig. 13).

The difference in strength dependence on filament spacing for rolled and wire drawn Cu-Nb is in conflict with previously reported results on pearlite where both strip drawn and wire drawn pearlite were proposed to exhibit a Hall-Petch [6,7] relationship with cementite spacing [9]. However, the data for strip drawn pearlite are not in nearly as good agreement with such a relationship as the data for wire drawn pearlite
Figure 12. Ultimate tensile stress dependence on the spacing ($\bar{\lambda}$) and thickness ($\bar{t}$) of Nb filaments in rolled or wire drawn Cu-20% Nb composites.
Figure 13. Ultimate tensile strength of Cu-20% Nb composites as a function of Nb filament spacing ($\lambda$)
In addition, the range of levels of deformation and cementite spacings in pearlite were much less than those for Nb filament spacings in Cu-Nb in situ composites (Fig. 10).

The weaker strengthening effect of the Nb filaments in the rolled composite as compared to the wire drawn composite could imply that some other substructural feature of the Cu matrix may be controlling the strength. For example, dislocation cell walls, deformation twins or Cu grains between the Nb filaments. The strengthening in wire Cu-Nb composites could not be correlated with any of these structural features [4].

**TEM analysis of rolled Cu-Nb**  
Substructural features i.e., Cu cell and grain sizes have been previously given [10]. The amount of cellular structure in the Cu decreases as the deformation rises and was not observed at the highest reduction, η ~ 7.0. Therefore, only the Cu grain size seemed to change in a consistent manner with increasing mechanical working. From the previous data, the Cu grain size is determined by Nb filament spacing [10]. Therefore, the grain size is expected to lie on a line connecting the initial Cu grain size (50 μm) to the smallest Nb filament spacing (0.14 μm). This line would have a slope slightly smaller than that observed for the filament spacings (Fig. 12). Deformation twins in the Cu matrix of the rolled composite were only prevalent at the highest deformation level (η = 6.9) [10] thus no correlation between strength and twin dimensions could be made.


**Strengthening mechanisms**

Various relationships besides the Hall-Petch [6,7] relationship have been proposed between strength and grain size or other substructural features where the exponent can have values between $-1/3$ and $-1$ [22] and even as low as $-1/4$ [23]. In most of these cases, different exponents can be used for correlations with the same data because of scatter in the data and/or a very limited grain size or substructural range. For wire drawn Cu-Nb composites analyses of the data in terms of the Hall-Petch [6,7] equation indicated that strengthening resulted from the difficulty in transmitting slip between the Cu and Nb phases [4]. In wire drawn Cu-Nb the filaments curl about the wire axis during deformation and thereby essentially form tubes of Nb parallel to the wire axis with Cu inside them, while in rolled material Nb exists as a planar array. Figure 14 schematically illustrates how the morphological differences of the Nb in the wire and sheet composites affects dislocation motion. Dislocations cannot travel far in any direction without encountering Nb barriers in the wire composites. But in sheet composites the filaments are not very effective barriers to dislocation motion because the dislocations can move relatively unimpeded in directions parallel to the filament surfaces (c.f. Fig. 14). This could be the reason for the weaker strength dependence on filament spacing in rolled versus wire drawn Cu-20% Nb.

Simple rule of mixtures (ROM) models, where the composite strength is predicted from a linear function of the sum of the product of the volume fraction and strength of the individual components, greatly underestimate the strength of Cu-20% Nb composites at high deformation.
Figure 14. Schematic of slip planes in (a) wire drawn and (b) rolled Cu-Nb in situ composite
strains [24]. Modifications have been made to simple rule of mixture models to account for plasticity effects when both the filament and the matrix undergoes extensive plastic deformation [25,26]. These mixture laws for two ductile phase components are based on a condition of plastic instability of the individual composite components. In these analyses, it is assumed that the necking of the individual components is impossible without necking of the composite as a whole. Both models yield similar predictions and the present results will be compared to only one of them [25] because of its simpler analytic evaluations.

According to this model the composite tensile stress, $\sigma_{x}^C$, is given by

$$\sigma_{x}^C = V_f \lambda^f \sigma_{x}^f + (1 - V_f) \lambda^m \sigma_{x}^m$$

(5)

where $\sigma_{x}^f$ and $\sigma_{x}^m$ are the ultimate tensile stresses of the individual filaments and the matrix, respectively. In the above equation, $V_f$ is the volume fraction of the filaments and the $\lambda$'s account for the constraint between the matrix and filament. More complex equations are also given for evaluating the uniform plastic strain of the composite, $\epsilon_{x}^C$, from the uniform plastic strains of the individual filaments, $\epsilon_{x}^f$ and the matrix, $\epsilon_{x}^m$.

The individual tensile stress-strain curves for Cu, Nb, and Cu-20% Nb are shown in Figure 15 for sheet rolled to $\eta = 5.3$ prior to tensile testing. Analysis of Figure 15 shows that Cu-20% Nb is not a typical composite because its uniform plastic strain exceeds that of the matrix and it is similar to that of the filament material. In general, the uniform plastic strain of two ductile phase composites exceeds that of the filament but is less than that of the matrix [25,26]. In addition,
Figure 15. True stress-true strain curves for Cu, Nb and Cu-20% Nb rolled to $\eta = 5.3$
the strength of the composite is close to that of the filament even though it contains only 20% filament material. In heavily drawn Cu-20% Nb, the composite strength can exceed that of the filamentary material [24].

Table 2 gives values of the different experimental parameters for the curves in Figure 15 that are needed to evaluate \( \sigma_{p}^C \) and \( \varepsilon_{p}^C \) from the model [25]. The results are very close to a simple rule of mixtures because the \( \lambda \) values are 1.00 for both Cu and Nb [25]. In addition, the calculated uniform plastic strain is low because it has to be somewhere between \( \varepsilon_{p}^f \) and \( \varepsilon_{p}^m \). Therefore, the rule of mixtures models proposed for two ductile phase mixtures are completely inadequate for predicting the strength and ductility for Cu-20% Nb. This is probably because of the importance of the filament size on mechanical properties. The simple rules of mixtures take no account of the scale of the microstructures and modifications have been proposed to include microstructural strengthening of the matrix using a Hall-Petch approach [27]. The difference in structure size of the Cu in the matrix as compared to pure Cu has not been evaluated for rolled Cu. However, the comparison has been made for drawn Cu and Cu-20% Nb [4]. At large reductions \( \eta \geq 6.9 \) the average grain size of pure Cu was about 0.5 \( \mu m \) whereas that of Cu in Cu-20% Nb was about 0.2 \( \mu m \). Using literature values of the Hall-Petch slope of Cu [21] indicates that the grain size refinement in the composite would add only about 75 MPa to \( \sigma_{p}^m \) and therefore about 58 MPa to the calculated \( \sigma_{p}^C \). Grain size strengthening in Nb is very small [21] and, because of the small volume fraction of Nb any additional strengthening from substructural refinement of the Nb in
Table 2. Mechanical property data for Cu, Nb, and Cu-20% Nb rolled to $\eta = 5.3$

<table>
<thead>
<tr>
<th>Cu</th>
<th>Nb</th>
<th>Cu-20% Nb</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\sigma^m$ (MPa)</td>
<td>$\varepsilon^m$</td>
<td>$\sigma^f$ (MPa)</td>
</tr>
<tr>
<td>468</td>
<td>0.015</td>
<td>746</td>
</tr>
</tbody>
</table>

$^a$Equation 5 and [25].
the composite as compared to pure Nb is expected to increase \( \sigma_k^C \) by less than 10 MPa. Therefore, even with these corrections the calculated \( \sigma_k^C \) is over 100 MPa below the observed value.

The importance of the size of the second phase on the stress-strain behavior of two ductile phase composites has been shown using the finite element method to calculate strengthening [28]. This approach could yield more meaningful predictions of the stress-strain behavior of in situ composites.

An alternate model proposed to explain strengthening in pearlite was based on the critical stress necessary to propagate dislocations in the matrix between the cementite plates [29]. According to this model, the critical stress necessary to propagate dislocations in between lamellae is given by

\[
\sigma_c = \sigma_0 + \frac{MAGb \ln (A/b)}{2M}
\]

where \( G \) is the shear modulus, \( b \) is the Burgers vector, \( A \) is the lamellae spacing, \( \sigma_0 \) is a friction stress, \( M \) is the Taylor factor (taken as 2 for BCC materials and 3 for FCC materials [30]) and \( A \) is a constant depending on the character of the dislocations (\( A \) was taken as 1.21 assuming mixed dislocations [31]). Equation (6) gives an approximate inverse correlation between strength and lamellae spacing corrected by a logarithmic factor. Cementite was not considered to be plastically deformed during testing. In Cu-Nb composites both Cu and Nb are plastically deformed during tensile testing [4] and equation (6) should be applied to both components. Substituting values of 48.3 GPa and
0.25 nm for the shear modulus and Burgers vector of Cu and 37.5 GPa and 0.29 nm for the corresponding values in Nb, equation (6) was used to calculate $\sigma_c - \sigma_o$ for Cu and Nb at various $\eta$ values where the observed $\Lambda$ and $T$ values were used for $\Lambda$ for Cu and Nb, respectively.

Because both the Cu and Nb contribute to the flow stress of the Cu-20% Nb composite, a rule of mixtures criterion was used to predict the composite strength ($\sigma_c$) as was done for pearlite [29]. The composite strength would then be

$$\sigma_c = \sigma_{Cu}V_{Cu} + \sigma_{Nb}V_{Nb}$$

where $\sigma_{Cu}$ and $\sigma_{Nb}$ are calculated from equation (6) and $V_{Cu}$ and $V_{Nb}$ are the volume fractions of Cu and Nb, taken as 0.8 and 0.2 respectively. Values used for $\sigma_o$ in equation (6) for Cu and Nb were the ultimate tensile stresses at the different $\eta$ values previously obtained for heavily cold drawn wires [24]. Substructural refinement of the Cu in Cu-20% Nb as compared to pure Cu at similar $\eta$ values was accounted for using previous data for wire drawn material [4]. The correction ranged from about 25 to 85 MPa as $\eta$ increased from 3.6 to 6.9. The similarity in strength for the rolled and wire drawn Cu and Cu-20% Nb composites make this a reasonable approximation. The use of ultimate tensile stresses in evaluating equation (7) is not exactly correct in light of equation (6) which relates to yield behavior. However, the 0.2% offset stresses would slightly lower both the calculated and observed stress values but would not change the comparisons.

Figure 16 compares the calculated and observed values of the ultimate tensile stresses. The agreement is very good and gives
Figure 16. Comparison of observed and calculated ultimate tensile stress of rolled Cu-20% Nb
Credence to the proposal that the increased difficulty in propagating dislocations in Cu and Nb with increasing structure refinement is primarily responsible for strengthening in rolled Cu-20% Nb. But, this model cannot be used to explain the strengthening observed in wire drawn Cu-Nb composites. This is mainly because the filament spacings and thicknesses in the wire drawn composite are much larger than those of the rolled composite at any given \( \eta \) value (Fig. 12). At the largest \( \eta \) analyzed (\( \eta = 11.9 \)) for wire drawn Cu-20% Nb the stress predicted from equation (7) is 950 MPa which is only about one half of the observed strength (Fig. 10). Therefore in wire drawn Cu-Nb, it appears that the difficulty in transmitting plastic deformation between the Cu and Nb phases is the major cause of strengthening, because the highly kinked morphology of the Nb filaments makes them more effective barriers to dislocation motion than the aligned planar Nb filaments in the rolled composite (c.f. Fig. 13).

Conclusions

1. Sheet specimens of a Cu-20% Nb composite containing aligned Nb filaments in a Cu matrix were formed by extensive cold rolling to true strains up to 6.9 of a casting containing Nb dendrites in a Cu matrix. During rolling, Nb is formed into planar filaments that are perfectly aligned parallel to the sheet surface, that is in both the transverse and longitudinal directions.

2. With increasing rolling reduction the strength of the composite dramatically increases while the uniform elongation remains at about
3% and the fracture strain continuously decreases to a value of 0.45 at the highest rolling reduction.

3. Longitudinal and transverse specimens have equivalent strengths and ductilities and both exhibit a dimpled rupture fracture mode.

4. Strengthening in the rolled composite correlates with $(\lambda)^{-1/4}$, where $\lambda$ is the Nb filament spacing and, therefore does not show Hall-Petch [6,7] type behavior in contrast to wire drawn Cu-20% Nb [4,24].

5. The weaker dependence of strengthening on Nb filament spacing in the rolled composite appears to be a consequence of the filaments being planar and perfectly aligned parallel to the sheet surface which prevents them from being effective barriers to dislocation motion. In a wire drawn composite the Nb filaments become curly because they are forced to bend about the wire axis during deformation thereby becoming aligned only in the longitudinal direction making them more effective barriers to dislocation motion.

6. Strengthening in rolled Cu-20% Nb is in accord with a rule of mixtures model where the strength of Cu and Nb are taken at an equivalent deformation of the composite and a component is added to account for the difficulty in inducing plastic flow in Cu and Nb. This model cannot account for the observed strengthening in wire drawn Cu-Nb composites where the difficulty in transmitting plastic flow between Cu and Nb phases appears to be the primary cause of the strengthening.
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References


PREPARATION OF IN SITU Cu-Nb COMPOSITE SHEET
AND WIRE FOR TEM ANALYSIS

C. L. Trybus, F. C. Laabs, A.R. Pelton,* and
W. A. Spitzig

Ames Laboratory USDOE, Iowa State University
*Department of Materials Science and Engineering,
University of Notre Dame
SECTION III. PREPARATION OF IN SITU Cu-Nb COMPOSITE SHEET AND WIRE FOR TEM ANALYSIS

Abstract

Cu-Nb in situ composites attain anomalous increases in strength upon mechanical deformation. The unique filamentary microstructures that evolve during processing (cold rolling and/or wire drawing) are the source of the strengthening. Results from transmission electron microscopy characterization studies have played a key role in the understanding of the relationships between structure and properties. However, the fabrication of reliable TEM samples has been extremely challenging for the following reasons: (1) traditional electrochemical techniques are not suitable for the two-phase microstructure, (2) preparation of longitudinal and transverse sections of fine (~150 μm diameter wires) and thin (~60 μm thick) sheet is tedious, and (3) it is necessary to avoid excessive heat (< 300°C) during sample preparation to preserve the metastable structural arrangements. This paper will review the procedures used to prepare TEM specimens from bulk wire and sheet samples as well as from extracted Nb filaments. Proper techniques for plating, mounting, sectioning, polishing, and ion-thinning will be discussed.

Introduction

In situ composites are made by mechanically working a mixture of two ductile phases. During the deformation processing, such as rolling or
wire drawing, the minor constituent becomes elongated, parallel to the rolling direction or wire axis [1-3].

Cu-Nb in situ composites which are comprised of low Nb volume fractions (<20 vol.%), possess tensile strengths much in excess of the "rule of mixtures" [1-2]. These high tensile strengths are a direct result of the microstructural development which occurs during the mechanical processing steps used in forming the composite. The physical basis for this observed strengthening has been modeled using high dislocation densities in both Cu and Nb [4-5]. In another proposed strengthening mechanism the Nb is essentially dislocation-free while the Cu contains high dislocation densities ~10^{13}/cm^2 [2]. Both mechanisms are based on a supposed dislocation population and arrangement.

Transmission electron microscopy (TEM), allowing for the direct observation of the substructure, would clarify the applicability of the aforementioned models as well as others. Unfortunately, these materials do not lend themselves to conventional TEM sample preparation techniques. Their two phase microstructures defy the use of electrochemical methods and the small size of the wire (~150 μm in diameter) and sheet (~60 μm thick) can present handling difficulties. In addition, metastable substructures must be retained therefore excessive heating of the sample during preparation must be avoided. The purpose of this paper is to review the sample preparation procedures for wire [6] and to describe effective methods to make sheet specimens. The metallographic sections of the composite sheet will be designated by the directions which border them [7] and are given in Figure 1. Methods for
Figure 1. Illustration of sheet directions, RD = rolling direction, L = longitudinal direction, ST = short transverse, and LT = long transverse
preparing both longitudinal-short transverse (L-ST) and long transverse-short transverse (LT-ST) sheet sections for TEM analysis will be reported. Extracted Nb filaments provide supplemental information on dislocation arrangements and methods for extraction of the Nb from the Cu matrix are also reviewed [6,8].

Experimental

Sheet and wire TEM specimens are prepared in a similar manner from Cu-20 vol.% Nb in situ composites. Transverse wire specimens are made by plating bundled composite wires with Cu [6]. Sheet L-ST and LT-ST sections were fashioned from a plated "sandwich". This sandwich was fabricated by employing a modified technique from [9]. Strips of the composite sheet 1-3 mm x~40 mm long were cut perpendicular to the rolling direction for L-ST sections or parallel to it for the LT-ST sections. These pieces were cleaned in acetone, dried, and glued into a stack. The stack was assembled by applying an extremely thin layer of glue (Permabond or Eastman 910 brands) on one side of two strips. After sitting about 10 seconds they were placed together in a vise for about 2 minutes. This process was repeated until a stack of 7 to 12 sheets was built up. A variety of adhesives were tried, including epoxy types and Superglue. Epoxy types were too weak to slice cleanly and were subject to degradation during plating. Superglue acted very fast and made handling difficult. Heat-cured glues were purposely avoided because a hot cure cycle could alter the dislocation structure. The complete sample preparation process for sheet is outlined in Figure 2.
Figure 2. Schematic of sample preparation steps for sheet composite
Wire bundles and sheet sandwiches were both plated in a copper sulfate and sulfuric acid solution [10] with an applied voltage of about 0.5 V and an initial current density of 0.5 A-cm$^{-2}$. Wires were plated to a minimum diameter of 3mm and sheet sandwiches to about 4-5mm. Transverse slices about 500 μm thick were cut from the wire and sheet with a low speed diamond saw. These slices were then hand ground on 600 grit SiC paper with water to a thickness of ~75 to 100 μm. Care was taken to avoid excessive heating during the grinding process. Wire samples were mounted directly on a Gatan Dimple Grinder and dimpled to a thickness of ~10 μm prior to ion-thinning. Sandwich slices cut from thin sheet (< 100 μm) were very fragile and could not be dimpled. Instead they were hand ground to ~25 μm then fitted with a pair of oval grids and ion milled. Slices of thicker sheet were trimmed to fit one oval grid and dimpled on one side only to ~10 μm. Edge pores (c.f. Figure 2) which form between the sheet edge and the plating weaken the specimen. Careful alignment of the sheets on the oval grid overcome this problem and strengthens the foil. Conditions for dimpling were identical for both types of samples and are given elsewhere [6].

Final thinning was completed by ion milling on a liquid nitrogen cooled stage. Both types of samples were thinned by dual beam milling with 4kV Ar$^+$ and 20-30 μA at an incident angle of 12-15° until perforation. To obtain more thin areas milling was continued at 2kV at an incident angle of 10°. These methods produced several small perforations in the composite sheet and wire as shown in Figure 3. Note numerous thin areas associated with the perforations which are suitable for examination at higher magnifications. Unlike sheet specimens,
Figure 3. Low magnification TEM images of (a) sheet and (b) transverse wire [6] sections
longitudinal wire samples were not prepared from plated wires because of difficulties in distinguishing between the plating and the sample. These samples were made from epoxy mounted wires as described in [6]. Nb filaments from both sheet and wire were extracted by dissolving the Cu with a solution of 1HNO₃:1H₂O. The filaments were cleaned in methanol and collected on TEM mesh grids. Some filaments extracted from wires (≤ 2000 μm in diameter) and from sheet (≤ 200 μm thick) were electron transparent and were examined directly. TEM analysis was performed on a JEOL 100CX operated at 120kV.

Results and Discussion

Salient results from TEM analysis of composite wire and sheet can be drawn from Figure 4. More complete TEM characterizations of these materials are given in [6] for the wire and in [11] for the sheet. Our goal here is to demonstrate the effectiveness of the sample preparation techniques described. In particular, the techniques must be able to: (1) retain the deformation-induced defect arrangements and (2) thin uniformly both Cu and Nb for crystallographic studies.

Figures 4a and b are LT-ST sections of composite sheet at intermediate and high rolling strains respectively. In both cases the Nb is lamellar in appearance. The Cu, between the Nb in Figure 4a is comprised of multiple grains some of which are elongated while others are equiaxed. At the higher rolling strains (Figure 4b), only elongated Cu is observed in addition to deformation twinning [11]. Both specimens reveal low Cu dislocation densities ~10¹¹ cm⁻² [11]. The Cu matrix in
Figure 4. Summary of TEM analyses; sections from materials undergoing intermediate deformation strains (a), (c), (e); and high strains (b), (d), (f); LT-ST sheet sections (a), (b); transverse wire sections (c), (d); and extracted Nb filaments from sheet (e), (f); see text for explanations.
the in situ composite sheet, as in the wire, undergoes a cycle of deformation followed by dynamic recovery and recrystallization [11]. The selected area diffraction patterns (SADP) illustrate textural evolution in the composite. At intermediate deformations (Figure 4a SADP) the inner ring due to the (110)\text{Nb} is still diffuse while the next ring, (111)\text{Cu}, shows some spot localization around the \langle 110 \rangle_{\text{Cu}}. At the highest strains (Figure 4b SADP) the texture \langle 110 \rangle_{\text{Nb}} || \langle 111 \rangle_{\text{Cu}} is fully established [11].

Figures 4c, and 4d typify the transverse microstructures observed in Cu-20% Nb wires that have been deformed to the same effective strains as the sheet samples in Figures 4a, and 4b, respectively. In general, at the lower deformation values (c.f. Fig. 4c) the microstructure consists of regions of Cu subcells with coarse cell walls with \langle 100 \rangle orientations and regions of recrystallized grains with \langle 111 \rangle orientations. At higher deformations the microstructure contains fine (<0.25 μm) recrystallized Cu grains interspersed with (~0.01 μm) ribbons of Nb. The predominant orientation relationship in these samples is \langle 110 \rangle_{\text{Nb}} || \langle 111 \rangle_{\text{Cu}}. The maximum dislocation content observed in these wires was \sim 10^{10} \text{cm}^{-2} [1,6].

The last two photomicrographs, Figures 4e and 4f are of extracted Nb filaments from sheet corresponding to Figures 4a and 4b, respectively. The texture of these filaments is \langle 100 \rangle <011> [11] which is the characteristic rolling texture of BCC metals [12]. Other investigations on extracted wire and sheet [6,8] found boundaries misorientated 2° to 35° about a common \langle 110 \rangle. The origin of these boundaries is revealed here as random dislocations in Figure 4d rearrange with increasing
deformation level and form the low angle dislocation boundaries in Figure 4e.

Conclusions

Consistent and effective methods for preparing samples of Cu-Nb in situ composite wire and sheet for TEM analysis has been established and some preliminary results are discussed. Foils for TEM analysis are prepared in a similar manner for both sheet and wire and consist of plating, sectioning, mechanical thinning and dimpling steps followed by final thinning by ion milling on a liquid nitrogen cooled stage. The initial observations indicate dislocation densities on the order of $10^{10}$ to $10^{11}$ cm$^{-2}$ [6,11] which are lower than previous models have predicted [4-5].

Acknowledgements

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References


CHARACTERISTICS OF P/M PROCESSED Cu-Nb COMPOSITES

C. L. Trybus, W. A. Spitzig, J. D. Verhoeven, and F. A. Schmidt

Ames Laboratory USDOE, Iowa State University
SECTION IV. CHARACTERISTICS OF P/M PROCESSED Cu-Nb COMPOSITES

Abstract

The feasibility of producing high strength in situ Cu-Nb composites via P/M processing techniques is explored because P/M offers a much wider range of compositions for the composites as compared to traditional ingot metallurgy. In this study, two Cu-20 vol.% Nb composites were produced by hot extrusion of encapsulated Cu and Nb powders. Both composites were subsequently swaged into wire and mechanical properties were evaluated as a function of the amount of deformation. One composite comprised of spherical elemental particles did not display increased strength upon deformation while another one made from irregularly shaped powders did possess increased strength. The strength-deformation strain relationships are correlated with microstructural characteristics. These results are compared to the properties exhibited by cast Cu-Nb in situ composites.

Introduction

Cu-Nb alloys can be formed into in situ composites. These alloys consist of a mixture of Cu and Nb from which the composite is formed upon mechanical deformation. The volume fraction of Nb is kept low (less than 20 volume per cent) so that the Nb is essentially isolated within the Cu matrix. Rolling, wire drawing, and swaging can each cause
the Nb to attain a filamentary morphology parallel to the direction of working.

Tensile strengths reported for in situ Cu-Nb composites are much greater than those predicted by the rule of mixtures [1,2]. The high strengths of these materials have been attributed to the ribbon-like cross sections developed by the Nb filaments [1]. Upon wire drawing of a pure BCC material, like Nb, the $<110>$ directions align themselves parallel to the wire axis which results in Nb undergoing a plane-strain mode of deformation [3]. In the axisymmetrically deforming composite, this causes the Nb filaments to kink and fold about the wire axis in order to maintain compatibility between them and the Cu matrix. Thus, the Nb filament morphology can be described as long flat ribbons with highly irregular cross sections.

Recently, Spitzig et al. [4] showed that the strength of heavily cold worked wires of Cu-Nb increased as the spacing between the Nb filaments decreased. It was found that the composite obeyed a Hall-Petch type relationship indicating that the Nb filaments act as planar barriers to dislocation motion between the Cu and Nb. Clearly, the unique microstructures developed in in situ composites are the source of the strength increases observed.

The majority of Cu-Nb composites have been fabricated by two methods, casting and powder metallurgical (P/M) techniques. Casting methods, including consumable arc melting [5] and rf levitation melting [1], have been utilized to produce high strength in situ Cu-Nb composites. P/M methods appear to be favored for the production of wire to be used in superconductor applications. Both hot [6-9] and cold
[9-13] extrusion of loose powders have been employed to produce Cu-Nb composites. These composites are then reacted with Sn to form the superconducting phase Nb$_3$Sn. Although P/M billets have been successfully used to produce superconducting wires, they have not been proven suitable for the fabrication of high strength in situ Cu-Nb composites. The goal of this research was to fabricate high strength Cu-Nb composites via P/M. The microstructural characteristics and tensile strengths were evaluated and compared to consumable arc melted composites.

Experimental Procedures

Two billets comprised of elemental Cu and Nb were hot extruded at 750°C at an areal reduction of 12:1. A third compact made from powders pre-alloyed by REP (rotating electrode process) was also hot extruded at 750°C at 14:1 areal reduction. The properties of the Cu-Nb REP powder and the resulting composite microstructure are given elsewhere [14]. Composites made from REP powder did not show increases in strength with increasing cold working. The characteristics of the elemental powders employed are given in Table 1 and Figure 1. The Nb must be ductile in order for it to become filamentary during cold work. Nb powder produced by the hydride-dehydride process has been successfully employed in P/M processing of superconducting Cu-Nb-Sn composites [13].

In all three cases, the powders were mixed and packed to tap density into an extrusion can of 101 copper 7.62 cm in diameter. Prior to mixing, the elemental Cu powders were heated in flowing H$_2$ at ~450°C.
### Table 1. Initial Powder Characteristics

<table>
<thead>
<tr>
<th>Extrusion No.</th>
<th>Powder Size Range (μm)</th>
<th>Other</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td><strong>Cu</strong></td>
<td><strong>Nb</strong></td>
</tr>
<tr>
<td>1</td>
<td>200-400</td>
<td>200-400</td>
</tr>
<tr>
<td>2</td>
<td>&lt; 44</td>
<td>88-125</td>
</tr>
</tbody>
</table>
Figure 1. Scanning electron micrograph of the Nb hydride-dehydride powder used in extrusion 2
for 4 hours to reduce surface oxides. The extrusion can top, which was shaped as a truncated cone, had a concentric series of 30 holes (1.5 mm nominal diameter) drilled parallel to the longitudinal axis of the can halfway up the conical surface. After the can was packed with powder, it was placed under vacuum in an electron beam welder and heated to approximately 800°C by a defocused electron beam. Subsequently, the holes in the cap were welded shut. This procedure was used to remove adsorbed gases from the powder surface.

Post extrusion forming processes included swaging from 2.22 cm to 1.27 cm in diameter, removing the copper clad, followed by swaging end-over-end (reversing the direction of swaging after each pass) to a diameter of 0.64 mm. Successively smaller wires were drawn to a minimum wire diameter of 0.15 mm. This minimum diameter represents the smallest diameter wire which we are able to test accurately and is not indicative of composite fracture during drawing. The level of deformation will be given in logarithmic strain by \( \eta = \ln \left( \frac{A_0}{A} \right) \), termed the draw ratio, where \( A_0 \) and \( A \) are the initial and final cross-sectional areas, respectively. In this study the largest draw ratio attained was \( \eta = 9.4 \) which corresponds to a reduction in area of 99.992%.

Tensile specimens were machined from the extruded and swaged material and from wires drawn down to 0.25 cm in diameter. Smaller wires were tested by embedding their ends into Cu sleeves using a superbond adhesive. All tensile tests were done at 22°C using a nominal strain rate of \( 1.7 \times 10^{-4} \) sec.\(^{-1} \). Multiple specimens (2 to 3) were tested at each wire size and the results were the same, within 5%. 
Samples for microstructural examination were prepared using a standard series of fine grinding steps (SiC) followed by final polishing in alumina slurries. All samples were lightly etched in a solution of $\text{H}_3\text{PO}_4:1\text{H}_2\text{O}_2:1.7\text{H}_2\text{O}$. Filament sizes and spacings were measured using a metallograph on the longitudinal sections. Standard quantitative metallographic techniques were employed in these calculations [15].

Results and Discussion

The as-extruded microstructures of extrusions 1 and 2 are shown in Figures 2 and 3. Pores at the Nb and Cu interface (Fig. 2) enlarged upon swaging and no increase in strength was observed in this material due to their persistent presence. Mechanical test results on extrusion 2 are given in Figure 4. For comparison, results are also shown for wire drawn pure Cu and for a cast composite [4]. Both the cast and extruded composite show large increases in ultimate tensile strength over pure Cu at draw ratios greater than ~6. At low draw ratios (<3) the P/M composite is stronger than the cast material due to an additional increment in strength from the refined Cu matrix grain size. The grain size of Cu in the as-extruded material (Fig. 3) is about half that of the as-cast. In addition, it is difficult to compare the as-extruded composite directly with the as-cast material. During the extrusion process the composite is mechanically worked while in casting no such deformation occurs. As the deformation level increases both of these effects are eliminated.
Figure 2. Microstructure of extrusion 1; as-extruded longitudinal section; note pores at Nb-Cu interface
Figure 3. Microstructure of extrusion 2 as-extruded (a) cross section, (b) longitudinal section
Figure 4. Effect of deformation on the ultimate tensile strength
Interstitially dissolved oxygen in Nb has a deleterious effect in its post-extrusion deformability [6]. Borman et al. [6] reported oxygen levels below 0.1 at.% in the Nb after extrusion of a Cu-Nb powder mixture. In this study, the initial Nb powder had more than twice as much oxygen (Table 2) and yielded a well developed Nb filamentary structure upon cold working. The oxygen content in extrusion 1 was lower than in 2, indicating that excessive oxygen was not the cause of its failure.

Figure 5 shows qualitatively and Figure 6 shows quantitatively how the microstructure is developing during the deformation process. Filaments get finer and closer together as deformation increases. Figure 7 is a plot of $\sigma_{UTS}$ vs. $\mathcal{X}$, filament spacing, and $\mathcal{T}$, filament thickness, for both extrusion 2 and a cast composite [4]. The slopes of the lines are each $-1/2$ indicating that in both materials the Hall-Petch relationship is being obeyed. Some points at large $\mathcal{X}$'s in extrusion 2 do not fall on the lines; this is due to the added strengthening of the refined grain size. If that incremental strength was subtracted out, the points would fall on the lines. Beyond this region of the plot, a given $\mathcal{X}$ (or $\mathcal{T}$) yields the same strength for both cast and P/M composite.

Results from a TEM analysis of a cast and wire drawn Cu-20 vol.% Nb composite [4,16] show that the Cu undergoes a deformation-recovery-recrystallization cycle which allows for further Nb deformation and refinement. Large populations of dislocations were not observed, but rather a cellular microstructure exists at lower reductions ($\eta < 5$). As intermediate deformations are reached ($5 < \eta < 9$) cells and recrystallized grains were observed while at high $\eta$'s the microstructure
Table 2. Oxygen content of initial powders and extrusions

<table>
<thead>
<tr>
<th>Extrusion 1</th>
<th>Oxygen Content (ppm, wt.)</th>
<th>Oxygen Content (at. %)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cu</td>
<td>190</td>
<td>0.075</td>
</tr>
<tr>
<td>Nb</td>
<td>120</td>
<td>0.070</td>
</tr>
<tr>
<td>Composite(^a)</td>
<td>240</td>
<td>——</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Extrusion 2</th>
<th>Oxygen Content (ppm, wt.)</th>
<th>Oxygen Content (at. %)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cu</td>
<td>790</td>
<td>0.313</td>
</tr>
<tr>
<td>Nb</td>
<td>350</td>
<td>0.202</td>
</tr>
<tr>
<td>Composite(^a)</td>
<td>980</td>
<td>——</td>
</tr>
</tbody>
</table>

\(^a\)The oxygen level was measured in the as-extruded material, which contained both Cu and Nb, thus no atomic percent is reported.
Figure 5. Microstructural evolution of extrusion 2 cross sections (a) and (c); longitudinal sections (b) and (d); (a) and (b) η = 4.5 material, optical; (c) and (d) η = 8.0 material, SEM, Nb appears light while Cu is dark.
Figure 6. Illustration of the refinement of filament spacing for the cast and extruded composites.
Figure 7. Log-log plot of strength as a function of $\lambda$ and $\bar{t}$
consisted mainly of recrystallized grains. It is believed that the same type of cyclic behavior occurs in the hot extruded P/M composite because in both cases the same microstructural property, \( \chi \) of Nb, appears to be controlling the strength. For a given \( \chi \) the P/M composite possesses the same strength as the cast one indicating further that similar substructures exist in both materials. Thus, in each case the Nb is acting as planar barriers to dislocation motion and the Cu, by undergoing a cold cycle of deformation-recovery-recrystallization allows for its continual deformation without need for intermediate heat treatments.

Conclusions

1. Both the cast and P/M processed Cu-Nb composites show dramatic increases in strength with increased deformation.

2. Strengthening in both the cast and P/M composites follows a Hall-Petch relationship which indicates that the strength arises from Nb filaments acting as planar barriers to dislocation motion.

3. Microstructures of both the cast and P/M composites appear to evolve in a like manner with deformation because both yield similar mechanical properties.

4. It has been demonstrated that the P/M route is a feasible method for the fabrication of high strength in situ composites.
Acknowledgments

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References


USE OF THE ROTATING ELECTRODE PROCESS IN THE FABRICATION OF Cu-Nb COMPOSITES

C. L. Trybus, J. D. Verhoeven, F. A. Schmidt, and W. A. Spitzig

Ames Laboratory USDOE, Iowa State University
SECTION V. USE OF THE ROTATING ELECTRODE PROCESS IN THE FABRICATION OF Cu-Nb COMPOSITES

Copper-niobium composites which are cast and then drawn exhibit ultimate tensile strengths much in excess of the rule of mixtures [1,2]. As the liquid cools from the melt, essentially pure Nb dendrites solidify within a Cu matrix. During deformation processing, such as wire drawing, the Nb dendrites become elongated and filamentary in nature. Thus, these materials are often termed in situ composites because the composite microstructures form during conventional casting and drawing operations.

A recent study of Cu-Nb in situ composites indicated that greater ultimate tensile strengths result throughout the deformation process as the dendrite spacing in the initial casting is reduced [3]. According to [3] composite ultimate tensile stress, \( \sigma_{\text{UTS}} \) in MPa, is related to the initial dendrite size, \( \bar{T}_0 \) in \( \mu m \) by

\[
\sigma_{\text{UTS}} = 82 + 459 (\bar{T}_0)^{-1/2} \exp (\eta/5.4) \quad (1)
\]

where \( \eta \) is the draw ratio for the wires, defined as \( \eta = \ln \left( \frac{A_0}{A} \right) \) and \( A_0 \) and \( A \) are the initial and final cross sectional areas respectively. Equation (1) predicts that \( \bar{T}_0 > 1.5 \text{ \( \mu m \)} \) and \( \eta = 11.9 \) would produce a composite possessing \( \sigma_{\text{UTS}} = 3477 \text{ MPa} \), which is greater than the maximum stress measured in pure Cu whiskers [4]. Such a dramatic increase in strength appears unlikely, but exemplifies the impact that the initial dendrite size has on the strength of in situ composites.

Extremely fine Nb dendrites could be attained by rapid solidifica-
tion of Cu-Nb alloys. The rotating electrode process (REP) is a powder atomization technique with cooling rates on the order of $10^3$ to $10^4 \text{ °C/s}$ [5,6]. These cooling rates are predicted to yield Nb dendrites of ~1.4 to 3.0 μm in diameter [7]. In addition, the rotating electrode process uses no crucible and the melting is done in an inert environment which makes it an attractive process for reactive metals like Nb. During REP, the meltstock is rotated inside a large round chamber and is melted by a tungsten cathode (REP) or a DC transferred arc plasma gun (PREP) [6]. As the metal melts, it forms droplets which come off the bar and solidify before hitting the chamber walls. In this letter we report the microstructural characteristics of a Cu-Nb REP powder and the strength properties of an in situ composite formed from it.

A consumable arc melted Cu-20 vol.% Nb casting [8] was machined into a rod 6.35 cm in diameter by 25.4 cm long which served as the meltstock. Its microstructure consisted of a fine uniform array of Nb dendrites, having a mean diameter approximately 8 μm, in a Cu matrix. This bar was converted into powder by PREP at Nuclear Metals Corporation, Concord, MA. Details and a schematic of the process are given in [6].

Median particle diameter (based on weight) is empirically given by Champagne and Angers [9],

$$d_{50} = K \frac{\omega^{0.12} \left( \frac{\gamma}{\rho} \right)^{0.42}}{\omega^{0.95} D^{0.61}}$$

(2)

where $K$ is a constant, $\omega$ is the angular velocity, $\gamma$ is the liquid surface tension of the melt, $\rho$ the density, $D$ is the diameter of the
rotating bar and $Q$ is the melt rate, which experimentally was found to be directly proportional to the electric arc power. The power level is selected by holding the voltage constant and varying the current. Equation (2) can be rewritten in terms of variable operating parameters, $Q$ and $\omega$,

$$d_{50} = K' \frac{0.12}{\omega^{0.95}}$$ (3)

Here $K'$, a constant for a particular meltstock, contains the fixed parameters of $\gamma$, $\rho$, and $D$. Equation (3) demonstrates that particle size depends on two processing parameters, power level and angular velocity, and therefore a variety of particle sizes and microstructures was produced by varying these parameters. The operating conditions and powder sizes are given in Table 1. Between each run the powders were collected then sieved prior to microstructural evaluation.

Particles greater than 600 $\mu$m can be characterized as "splats" and "chunks" for all runs. Pancake shaped splats were composed of primarily Cu with Nb from the original ingot, and some fine new Nb dendrites. Chunks were Nb rich pieces of the original Cu-Nb bar. Powders smaller than 212 $\mu$m were essentially pure Cu spheres. The other two powder sizes listed in Table 1 contained varying amounts of the following five different microstructure types: (1) Nb dendrites from the original ingot within a Cu matrix, (2) fine new Nb dendrites surrounded by Cu, (3) tiny incompletely formed Nb dendrites in a Cu matrix, (4) fine Nb particles outlining Cu dendrites, and (5) large solid Nb particles.
Table 1. PREP Conversion Conditions and Resulting Powder Size Distributions

<table>
<thead>
<tr>
<th>Run No.</th>
<th>Operating Conditions</th>
<th>Size distribution in % by weight</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Current (A)</td>
<td>ω (RPM)</td>
</tr>
<tr>
<td>1</td>
<td>750</td>
<td>5000</td>
</tr>
<tr>
<td>2</td>
<td>750</td>
<td>2000</td>
</tr>
<tr>
<td></td>
<td>1000</td>
<td>2000</td>
</tr>
<tr>
<td>3</td>
<td>1000</td>
<td>2940</td>
</tr>
<tr>
<td>4</td>
<td>1000</td>
<td>500</td>
</tr>
<tr>
<td></td>
<td>1000</td>
<td>5000</td>
</tr>
<tr>
<td>5</td>
<td>1000</td>
<td>2530</td>
</tr>
</tbody>
</table>
Microstructure types 1-3 are illustrated in Figure 1 and type 4 is shown in Figure 2.

The microstructural variability can be understood with reference to the Cu-Nb phase diagram (Fig. 3) in conjunction with the REP process itself. The large liquid plus solid Nb region in the phase diagram coupled with short liquid dwell time in the rotating electrode prevents complete alloying of the melt causing incomplete Nb melting and subsequent Nb dendrite formation.

The powders of the -600 µm +300 µm size fraction of runs 2 and 5 (Table 1) had typically dendritic Nb microstructures (types 2 and 3). These powders were mixed, canned in a 5 cm Cu container and hot extruded at 750°C at an areal reduction of 14:1. The composite was then swaged and tensile strengths were evaluated at various draw ratios. Details of the extrusion, swaging and mechanical testing procedures are given elsewhere [11]. Mechanical strength data for the in situ composite formed from REP powders is presented in Figure 4. The tensile strengths of the composite are approximately equal to those of similarly deformed Cu wires, showing no composite strengthening.

Interstitially dissolved oxygen in Nb has been reported [12] to decrease the post-extrusion deformability of Nb in in situ Cu-Nb composites. Results of chemical analysis for C, N, H, and O of the REP powder used in the extrusion are given in Table 2. Levels of all interstitials were very low. The effect of C, N, and O on the ductility of nonhydrogenated Nb is very slight [13]. Thus, interstitial contamination did not contribute to the low tensile strengths observed.
Figure 1. Cross section of as-extruded composite illustrating microstructure types 1 (large Nb in center), 2 (most of the photomicrograph) and 3 (upper left-hand corner)
Figure 2. Microstructure type 4
Figure 3. Cu-Nb phase diagram from [10]
Figure 4. Strength of composite made from REP powder as a function of deformation; data from a cast composite (\(t_0 = 6.2\) \(\mu\)m) and for pure Cu are given for comparison.
Table 2. Interstitial Impurity Levels in PREP powders

<table>
<thead>
<tr>
<th>Element</th>
<th>Content in ppm, wt.</th>
</tr>
</thead>
<tbody>
<tr>
<td>C\textsuperscript{a}</td>
<td>8</td>
</tr>
<tr>
<td>N</td>
<td>11</td>
</tr>
<tr>
<td>H</td>
<td>none detected</td>
</tr>
<tr>
<td>O</td>
<td>68</td>
</tr>
</tbody>
</table>

\textsuperscript{a}Impurity detected by combustion analysis. All others by vacuum fusion.
The nonuniformity of the composite microstructure, consisting of large Nb particles (microstructure type 5) along with pure Cu and fine filamentary Nb (Fig. 5) appears to be the reason for the lack of strengthening.

In conclusion, Cu-Nb in situ composites formed from powders prealloyed by REP did not exhibit increased tensile strength upon deformation due to the nonuniform microstructure of the powder. These nonuniformities are a direct result of a combination of the Cu-Nb phase diagram with the rotating electrode process itself. It is our opinion that the rotating electrode process will experience difficulty in the production of refined microstructures from most two phase alloys. Because the liquid dwell time on the electrode is very short, the complete homogenization of both phases in the liquid state will be difficult, and will depend primarily upon two parameters, the solidus/liquidus temperature gap $\Delta T$ of the alloy as well as the size of the high melting phase. In the present case with a $\Delta T$ of $\sim 600^\circ$C and a particle size of $\sim 8$ $\mu$m complete solutioning did not occur even at the lowest melt rate and lowest angular velocities. A similar result was found on a study of a steel containing carbides [14]. Vanadium carbides, VC, failed to dissolve during REP and were flung off the electrode as irregularly shaped particles. This insolubility was attributed to short liquid dwell time coupled with the high temperatures required for complete solutioning in tool steels.
Figure 5. Longitudinal section of Cu-Nb composite swaged to $\eta = 1.6$
Acknowledgements

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References


SUMMARY AND CONCLUSIONS

Microstructural features of heavily cold rolled Cu-20 vol.% Nb composites to a true strain of 6.9 have been correlated with mechanical strength. In the initial cast material, Nb exists as isolated dendrites within a Cu matrix. As the deformation level increases, the Nb evolves into long flat ribbons with irregular cross sections. SEM was utilized to analyze the gross microstructural changes and TEM to analyze substructural ones.

1. Consistent and effective methods for preparing samples of Cu-Nb in situ composite wire and sheet for TEM analysis has been established. Preparation of foils for TEM analysis are consist of plating, sectioning, mechanical thinning, and dimpling steps followed by final thinning by ion milling on a liquid nitrogen cooled stage.

2. Spacing between the Nb filaments and Nb filament thickness are reduced with increased deformation.

3. In the course of the rolling operation Cu undergoes a cycle of deformation-dynamic recovery-recrystallization. No such process was evident in the Nb.

4. Both ideal Cu (110)<112> and Nb (113)<110> rolling textures were observed. In addition the textures (112)<110>\textsubscript{Cu} and (001)<110>\textsubscript{Nb} were present. Textures in the Cu and Nb phases in the composite appear to be similar to those found in heavily deformed pure Cu and Nb.
5. Low energy dislocation structures were observed in Cu, similar to stage IV low energy structures found in heavily deformed pure metals.

6. Dislocation densities of the Cu matrix were found to be on the order of $10^{10}$/cm$^2$.

7. With increasing rolling reduction the strength of the composite dramatically increases while the uniform elongation remains at about 3% and the fracture strain continuously decreases to a value of 0.45 at the highest rolling reduction.

8. Longitudinal and transverse specimens have equivalent strengths and ductilities and both exhibit a dimpled rupture fracture mode.

9. Strengthening in the rolled composite correlates with $(X)^{-1/4}$, where $X$ is the Nb filament spacing and, therefore does not show Hall-Petch type behavior in contrast to wire drawn Cu-20% Nb [9,16].

10. The weaker dependence of strengthening on Nb filament spacing in the rolled composite appears to be a consequence of the filaments being planar and perfectly aligned parallel to the sheet surface which prevents them from being effective barriers to dislocation motion.

11. Strengthening in rolled Cu-20% Nb is in accord with a rule of mixtures model where the strength of Cu and Nb are taken at an equivalent deformation of the composite and a component is added to account for the difficulty in inducing plastic flow in Cu and Nb. This model cannot account for the observed
strengthening in wire drawn Cu-Nb composites where the difficulty in transmitting plastic flow between Cu and Nb phases appears to be the primary cause of the strengthening.

Powder metallurgy (P/M) processing for producing high strength heavily deformed composites is explored because of the flexibility the P/M route offers.

12. High strength Cu-20vol.% Nb in situ composite wire was successfully fabricated from a P/M billet produced by hot extrusion of encapsulated Cu and Nb powders.

13. Cu-20% Nb in situ composite wire fabricated from hot extrusion of powders pre-alloyed by the rotating electrode process did not show strengthening upon deformation.
REFERENCES


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