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Tensile deformation and fracture behavior of a ductile phase reinforced dispersion strengthened copper composite

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Niobium particle reinforced aluminum oxide (Al_2O_3) dispersion strengthened copper composite is an attractive and emerging engineered material for applications requiring high strength, high thermal and electrical conductivities and resistance to softening at elevated temperatures. In this paper, the microstructure, tensile deformation and fracture behavior of the composite is examined. The strength of the material decreases with an increase in temperature with a concomitant improvement in ductility. The composite microstructure maintains a high value of yield strength/ultimate tensile strength ratio. The factors contributing to increased strength and the intrinsic mechanisms governing fracture characteristics of the composite are examined in light of intrinsic microstructural effects, nature of loading and deformation characteristics of the matrix. *© 1999 Kluwer Academic Publishers*

1. Introduction

An important ingredient for a spectrum of current and future engineering applications is the need for achieving active cooling. This would facilitate enhancing, with relative ease, the operating temperature of materials [1–4]. Active cooling during operational service necessitates the need for the structural material to possess high thermal conductivity. A natural choice to satisfy this requirement is copper. It is essential that the material while offering adequate strength also be resistant to softening and degradation at elevated temperatures. Copper can be effectively strengthened by solid solution strengthening achieved through judicious alloying with other elements but with a concurrent loss in thermal conductivity. An attractive and viable approach or mechanism for improving the strength of copper is to introduce a small volume fraction of a second-phase, preferably inert particles, into the matrix material, resulting in a dispersion strengthened (DS) alloy [5–8]. Oxides are the most common second-phase particles used to strengthen the copper matrix, but nitrides, borides and carbides have also been used as candidates. In fact, for optimum strengthening, the inert oxide particles must be: (a) extremely small, (b) discontinuous and randomly distributed in a continuum matrix, and have small interparticle spacing [7]. Copper matrices containing a fine dispersion of inert particles are particularly attractive for their excellent combinations of thermal and electrical conductivities, strength retention at elevated temperatures, and overall microstructural stability [6–8]. The dispersoid particles, the oxides, are sufficiently inert so that their size and spacing are relatively unaffected by heat treatment.

Since the early 1970s a patented family of dispersion strengthened copper emerged and became commercially available under the trade name GlidCop^R [7, 8]. An attractive feature of this material lies in its ability to resist softening, while concurrently retaining strength and conductivity properties even after sustained exposure to elevated temperatures approaching the melting point of the matrix.

The much superior properties of the oxide dispersion strengthened copper (DSC) material arises from the competing influences of size, distribution, and interparticle spacing of the insoluble submicroscopic aluminum oxide (Al_2O_3) particles in a high-purity copper matrix. These particles range in size from 3 to 12 nm with an interparticle spacing of 30 to 100 nanometers and a particle density of the order of 10^{16}–10^{17}/cm^3 [7, 8]. The Al_2O_3 particles are hard, and chemically and thermally stable at elevated temperatures. On account of their chemically inert nature they exert little influence on the electrical conductivity of the matrix material. Besides, the insoluble Al_2O_3 particles retain their original size, distribution and spacing even after prolonged exposure to elevated temperatures. The overall thermal stability of the dispersed particles is largely responsible for their excellent resistance to softening. Furthermore, since the volumetric percentage of the dispersed Al_2O_3 particles is small, the physical properties, namely: (a) melting point, (b) density, and (c) coefficient of thermal expansion of the oxide dispersion strengthened copper matrix, are very similar to those of pure copper, while the modulus of elasticity and strength are substantially higher [7, 8]. Additional improvements in the properties of dispersion strengthened copper matrix were achieved through the controlled addition of elemental niobium [9, 10]. The niobium reinforcements are distributed uniformly in an internally oxidized, dispersion strengthened copper matrix. The unique properties of GlidCop^R oxide dispersion strengthened copper coupled with the refractory metal characteristics of niobium results in a composite material that is a potentially viable candidate for use as resistance-welding electrodes and tooling. The composite powder mixture is consolidated in the same manner as conventional dispersion strengthened copper powder to produce a variety of mill shapes.

The objective of this study was to evaluate the microstructural characteristics, tensile deformation and fracture behavior of a dispersion strengthened copper-niobium composite. The factors contributing to increased strength of the composite and the kinetics governing fracture behavior are discussed in light of intrinsic microstructural effects, deformation characteristics of the matrix, and test temperature.

2. Material and experimental procedures

The material used in this study, dispersion strengthened copper (DSC) + niobium composite, designated as grade AL-15 + NB1000, was provided by OGM Americas (formerly SCM Metal Products Inc., at Research Triangle Park, North Carolina, USA). The conventional oxide dispersion strengthened copper material was produced by a proprietory powder metallurgy internal oxidation process in which dilute copper/aluminum alloy powder is exposed to an oxidizing environment, and the aluminum is allowed to preferentially oxidize within the copper matrix. The internal oxidation process provides a microstructure containing a very uniform distribution of fine, aluminum oxide (Al_2O_3) particles. Details of the process to make the DS copper-niobium composite are considered proprietory by the manufacturer. The composite powder is consolidated into a fully dense product. The consolidated product is finish worked to desired form using conventional metal working methods. The composite contains 10% niobium by weight, in the form of uniformly dispersed particles. The composition of the composite material is given in Table I.

Metallographic samples were cut from the as-extruded stock of the composite. The samples were mounted in bakelite and wet ground on 320, 400, 600 grit silicon carbide (SiC) paper using water as lubricant and then mechanically polished with one-micron alumina-based polishing compound. Grain morphology and other intrinsic microstructural features were revealed using an etchant mixture of 25 ml ammonium hydroxide, 20 ml hydrogen peroxide (3% concentration) and 25 ml distilled water. The polished and etched specimens were examined in an optical microscope and photographed using standard bright field technique.

Cylindrical test specimens, conforming to specifications in ASTM:E8-93 [11], were machined from the as-extruded composite. The test specimens measured 25 mm in length and 6.25 mm in diameter. To minimize the effects of surface irregularities, the gage sections were mechanically ground and then finish polished to remove any and all circumferential scratches and surface machining marks.

Uniaxial tensile tests were performed up to failure on a fully automated, closed-loop, servohydraulic mechanical test machine (INSTRON) equipped with a 22 kip (100 kN) load cell. The specimens were deformed at a constant strain rate of 10^{-4} s^{-1}. The tests were performed in controlled laboratory air environment (Relative Humidity = 55%) at ambient (27 °C) and elevated (175 °C) temperatures. The elevated temperature tests were conducted using an INSTRON Environmental Chamber system (Model: 3111). The temperature was controlled with the aid of a temperature controller fixed on the specimen's surface. Maximum temperature variation during the test was within 2 °C of the set-point temperature. Ambient temperature varied from 27 to 29 °C during any given test. Before each elevated temperature test, the specimen was soaked at the test temperature for 30 min so as to achieve stability with the environment. The axial strain was measured using a 12.7 mm clip-on extensometer fixed, with rubber bands for tests at the ambient temperature and steel springs for the elevated temperature tests, to the specimen gage section. The stress and strain measurements, parallel to the load line, were recorded on a PC-based data acquisition system.

Fracture surfaces of the fully deformed and failed tensile samples were examined in a scanning electron microscope (SEM) to: (a) determine the macroscopic fracture mode, and (b) characterize the fine-scale topography and microscopic mechanisms governing tensile fracture. The distinction between the macroscopic mode and microscopic fracture mechanism is based on the magnification level at which the observations are made. The macroscopic mode refers to the nature of failure (plane strain, plane stress or a combination of both), while the microscopic mechanism relates to the local failure process (microvoid formation, coalescence and nature of cracking). Samples for SEM observation were obtained from the fully deformed tensile specimens by sectioning parallel to the fracture surface.

TABLE I Nominal chemical composition of dispersion strengthened copper-niobium composite (GlidCop AL-15+NB1000)

	Al_2O_3 content		Niobium content	
GlidCop grade	wt %	vol %	wt %	vol %
AL-15	0.3	0.7	—	—
AL-15 + NB1000	0.3	0.7	10.0	10.6

3. Results and discussion

3.1. Microstructure

Typical microstructures of the extruded AL-15 + NB1000^{TM} composite for both the longitudinal and transverse orientations are shown in Figs 1 and 2. The longitudinal microstructure reveals fine ribbon-like filaments of the reinforcing niobium phase. The

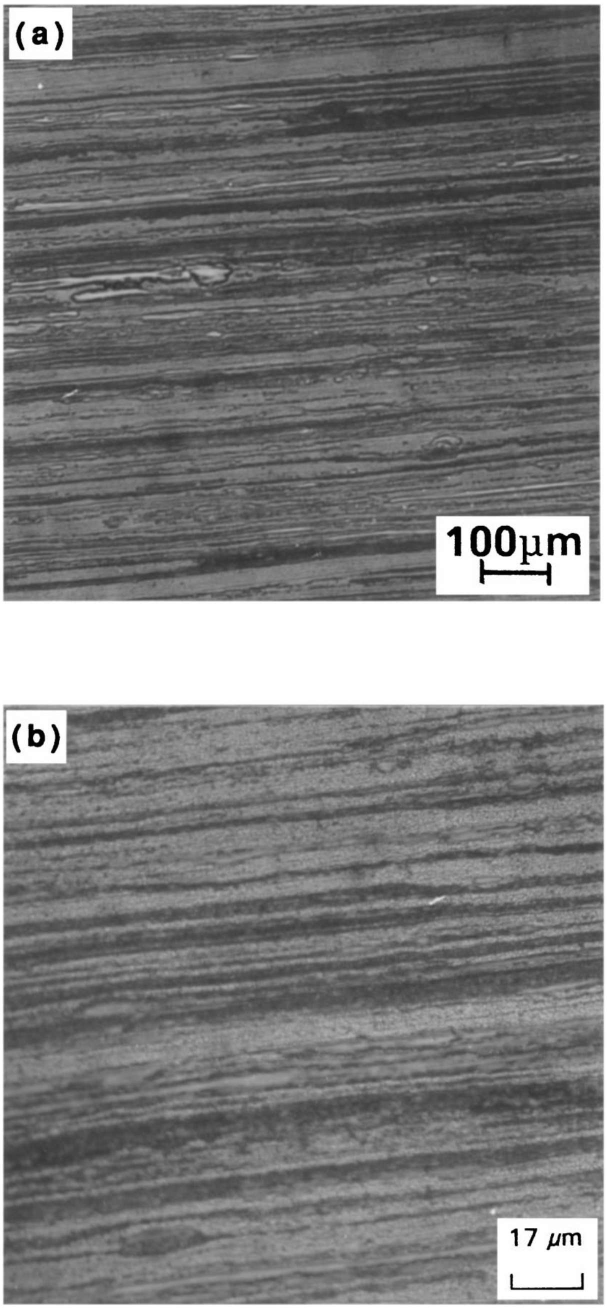

Figure 1 Optical micrographs illustrating microstructure of the dispersion strengthened copper-niobium composite in the longitudinal orientation.

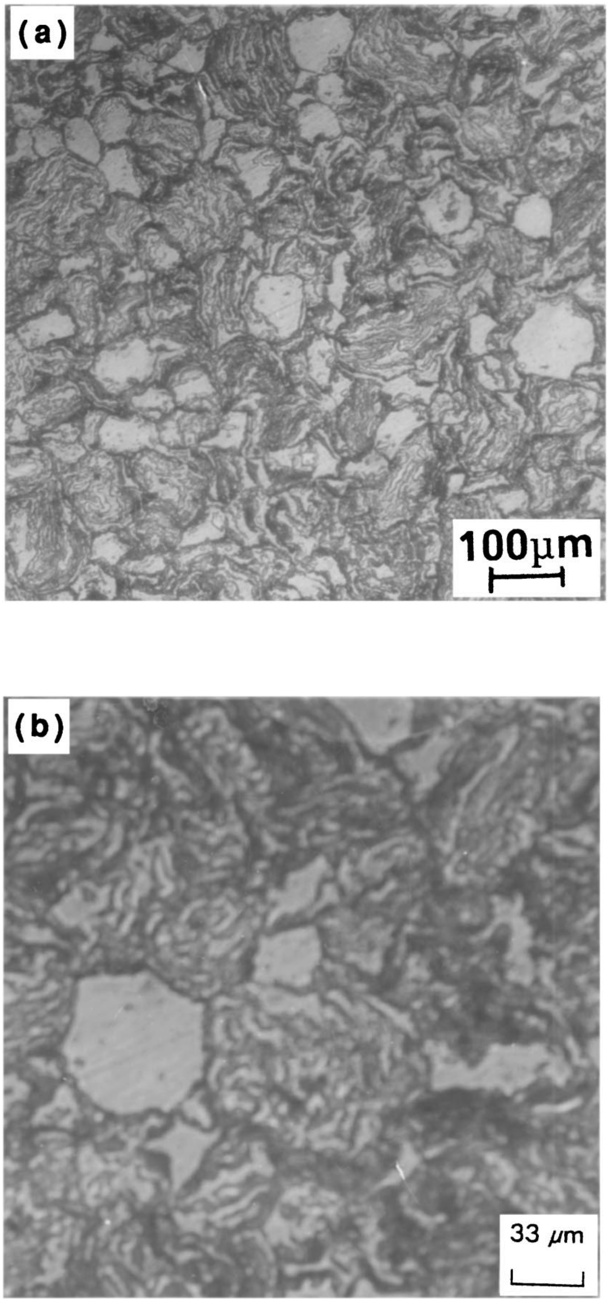

Figure 2 Optical micrographs illustrating microstructure of the dispersion strengthened copper-niobium composite in the transverse orientation.

transverse microstructure reveals a near-uniform distribution of the niobium particles in the oxide dispersion strengthened copper-matrix. The composite microstructure revealed no evidence of recrystallization. The absence of recrystallization even after high temperature deformation of the composite is typical of many oxide dispersion strengthened materials. This is ascribed to be the end result of a fine dispersion of the oxide particles, which also helps retain the elongated deformed structure.

3.2. Tensile response

The ambient temperature tensile properties of the composite are summarized in Table II. Results reveal the elastic modulus of the as-extruded composite (85 GPa) to be lower than that of the unreinforced material (107 GPa) in the as-extruded condition. The ambient temperature yield strength, defined as the stress required at a plastic strain of 0.2%, of the extruded composite material (393 MPa) is 15% greater than that of the unreinforced counterpart (341 MPa). However, the ultimate tensile strength of the AL-15 + NB1000$^{\text{TM}}$ composite (539 MPa) is 13% higher than that of the unreinforced oxide dispersion strengthened counterpart (402 MPa). The enhanced strength (yield and ultimate tensile) of the composite microstructure, much higher than predicted by the simple rule-of-mixtures theory, is attributed to the development and presence of ribbon-like filaments of the reinforcing phase (niobium) during deformation processing which act as barriers to the motion of mobile dislocations. The concomitant increase in dislocation density is partially responsible for the increased strength of the composite [12–15].

TABLE II Tensile properties of dispersion strengthened copper-niobium composite (GlidCop^{R}AL-15 + NB1000^{TM})

Condition	Reinforcement (wt %)	Temperature (°C)	Elastic modulus (GPa)	Yield strength (MPa)	(Ksi)	Ultimate tensile (MPa)	(Ksi)	YS/UTS (%)	Elongation (%)	Reduction in area (%)
Unreinforced (AL-15)	0	27	107	341	49	402	58	85	35	64
Unreinforced (AL-15)	0	175	89	263	38	284	41	93	33	42
Reinforced (AL-15 + NB1000^{TM})	10	27	85	393	(57)	539	(78)	73	14	27
Reinforced (AL-15 + NB1000^{TM})	10	175	87	383	(56)	436	(63)	88	16	27

[a] Results are the mean values based on duplicate tests.

At the elevated temperature there is no influence on elastic modulus (E) and yield strength (σ_{ys}) of the dispersion strengthened copper-niobium composite. However, elevated temperature exposure was observed to degrade the ultimate tensile strength from 540 MPa at ambient temperature to 436 MPa, that is by 19%. A plausible mechanism responsible for the decrease in tensile strength is attributed to the partial relief of any residual stresses in the composite matrix, compressive in nature, induced by the cold-working (extrusion) operation. This results in a reduced number of mobile dislocations, initially available, to interact with the intrinsic microstructural features, that is, the fine oxide particles and the reinforcing niobium filaments.

Yielding in this dispersion strengthened material occurs when the mobile dislocations either shear the dispersed oxide particles or interact with the reinforcing niobium filaments, or a combination of both [13]. The yield strength of the niobium particle-reinforced dispersion strengthened copper matrix is expressed by the relationship:

$$\text{Yield strength} = [Gb\nu/2\lambda C]^{1/2} \tag{1}$$

where G is the shear modulus of the copper matrix, ν is the shear modulus of the dispersed oxide particles (Al_2O_3), b is the Burgers vector for the dislocation, λ is the spacing between the dispersed oxide particles, and C is a constant. The partial relief of any residual compressive stresses coupled with intrinsic softening of the copper matrix are the two complimentary factors contributing to the observed decrease in ultimate tensile strength of the composite microstructure at the elevated test temperature.

The ambient temperature yield strength-to-ultimate tensile strength ratio (σ_{ys}/σ_{UTS}) of the composite microstructure (73%) is lower than that of the unreinforced extruded counterpart (85%). However, the fairly high value of this ratio (88%) at the elevated test temperature (175 °C) is indicative of the intrinsic ability of the dispersion strengthened copper-niobium composite matrix to resist appreciable softening and/or degradation.

Earlier research work performed on wire drawn Cu-20% Nb material [12, 16, 17] provided convincing evidence that the ultimate tensile strength (σ_{UTS}) correlated well with filament spacing (λ), according to the relation:

$$\sigma = \sigma_0 + K(\lambda)^{0.5} \tag{2}$$

This equation is of the Hall-Petch type [18, 19]. Furthermore, the copper-niobium interfaces are assumed to act as potential sources of dislocations which control plastic flow across the reinforcements. In fact, the number of dislocations generated during deformation is proportional to the interfacial surface area per unit volume (say S_v). For an aligned structure similar to the extruded copper (fcc)-niobium (bcc) composite:

$$S_v = [2/(\lambda + t)] \tag{3}$$

where λ and t are the spacing and thickness of the niobium reinforcements [20]. The dislocation density can be expressed as:

$$\rho_B = m(S_v) \tag{4}$$

where m is the density of dislocation sources at the interfaces of the matrix and the reinforcement and is assumed to be independent of the reinforcement spacing [21, 22]. Assuming a linear relation between flow stress and the square root of dislocation density as given by:

$$\sigma = \sigma_0 + \alpha MGb(\rho)^{0.5} \tag{5}$$

where α is a constant, M is the Taylor factor, G is the shear modulus, and b is the Burgers vector. Substituting Equations 3 and 4 into Equation 5 gives:

$$\sigma = \sigma_0 + \alpha MGb[2m/(\lambda + t)]^{0.5} \tag{6}$$

A similar relationship can be obtained from an analysis of the nonhomogeneous deformation in this plastically deforming two phase Cu (fcc)-Nb (bcc) structure, wherein one component deforms more (say plastically) than the other (say elastic) creating gradients of deformation. As a direct result of the nonhomogeneous deformation geometrically necessary dislocations are required which contribute to hardening of the composite. In this model the flow stress of the composite is given by Equation 5

$$\sigma = \sigma_0 + \alpha MGb(\rho)^{0.5}$$

The geometrically necessary dislocation density (ρ_G) needed for compatibility between the soft and plastically deforming and the hard and elastically deforming phases is given by [23]:

$$\rho_G = 4(\theta)/b\lambda \tag{7}$$

where λ is the spacing of the harder phase and θ is the angle of rotation between the less or nondeforming phase in a predominantly plastically deforming matrix.

This equation assumes that no plastic deformation occurs in the plane of the interface between the niobium reinforcements and the oxide dispersion strengthened copper matrix. The occurrence of localized bending between the dispersion strengthened copper matrix and niobium reinforcements leads to inhomogeneous plastic deformation with a concurrent increase in dislocation density during continued uniaxial tensile deformation. Thus, the increased strength of the composite can be attributed to the conjoint influence of difficulty arising in propagating plastic flow across the reinforcements coupled with a non-homogeneous deformation model where the high density of dislocations generated to maintain compatibility between the dispersion strengthened copper matrix and niobium reinforcements controls strengthening of the composite.

The other dominant mechanism responsible for providing strength to the fine Al_2O_3 particle dispersed copper matrix is dispersion strengthening. This arises as a direct result of an interaction between the mobile dislocations and the dispersed Al_2O_3 particles, which either limits or restricts their mobility. Since the Al_2O_3 particles are essentially non-shearable, there exists at least two possible mechanisms for the interaction:

(A) The first mechanism can be related to repulsive dislocation-particle interactions, according to the mechanism put forth by Orowan [24], that results in strain hardening as progressive pile-up of dislocations occur. The microstructural characteristic for this mechanism is the occurrence of dislocation loops around the Al_2O_3 particles.

(B) The second mechanism is based on the assumption of the occurrence of local climb of dislocations over the particles [25] coupled with an attractive interaction between the dislocations and the particles [26–30].

Thus strengthening of the dispersion strengthened copper composite arises from the synergistic influences of dislocation interaction with the Al_2O_3 dispersion (Orowan Mechanism) and dislocation density increase due to the presence of the niobium reinforcements.

Ductility measurements, obtained by elongation over a 12.5 mm gage length and 6.25 mm gage diameter of the test specimen, revealed the composite material to have inferior elongation over the unreinforced counterpart at both ambient and elevated temperatures. The degradation is of the order of 60% at ambient temperature and 50% at the higher test temperature. This is rationalized as being due to the early debonding at the niobium reinforcement/copper interfaces. Such debonding results in the formation of fine microscopic and macroscopic voids, and a concomitant lower elongation-to-failure than the unreinforced counterpart. Increase in test temperature from 27 to 175 °C caused elongation of the composite microstructure to increase by 14%, and is consistent with the degradation in strength with temperature. A comparison of the engineering stress-engineering strain curve of the reinforced (composite) and unreinforced material, at the two temperatures, is made in Fig. 3. The monotonic parameters are summarized in Table III.

TABLE III Monotonic parameters of dispersion strengthened copper (GlidCop^{R}AL-15+NB1000^{TM})

Condition	Reinforcement (wt %)	Temperature (°C)	n	K MPa (Ksi)
Unreinforced (AL-15)	0	27	0.07	376 (54.5)
Unreinforced (AL-15)	0	175	0.04	279 (40.4)
Reinforced (AL-15 + NB1000^{TM})	10	27	0.13	504 (73.1)
Reinforced (AL-15 + NB1000^{TM})	10	175	0.08	442 (64.1)

n = monotonic strain hardening exponent.
K = monotonic strength coefficient.

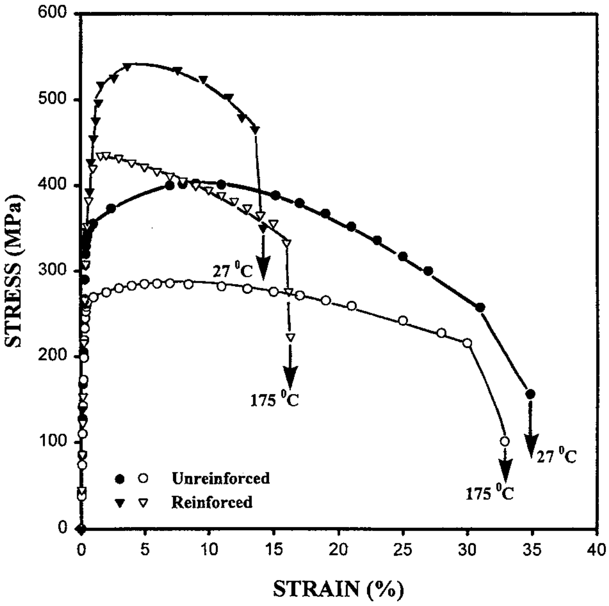

Figure 3 Influence of temperature on engineering stress-engineering strain response of the copper-niobium composite.

3.3. Tensile deformation and fracture

The fracture surfaces of the fully deformed tensile specimens were examined in a JEOL scanning electron microscope at low magnification to identify the macroscopic morphology, and at higher magnifications to identify the fine-scale fracture features. The tensile fracture surfaces provide useful information pertaining to microstructural effects on fracture.

3.3.1. Ambient temperature (27 °C)

On a macroscopic scale the tensile fracture revealed a predominantly cup and cone morphology. At the microscopic level the fracture surface revealed numerous macroscopic voids spread randomly through the fracture surface, tear ridges and microscopic cracking (Fig. 4a), features reminiscent of locally ductile and brittle failure. The macroscopic voids (Fig. 4b) were caused by the pull-out of the reinforcing niobium particulates. Observations of the transgranular fracture regions revealed a population of microscopic voids, of a wide range of sizes, and shallow dimples, features

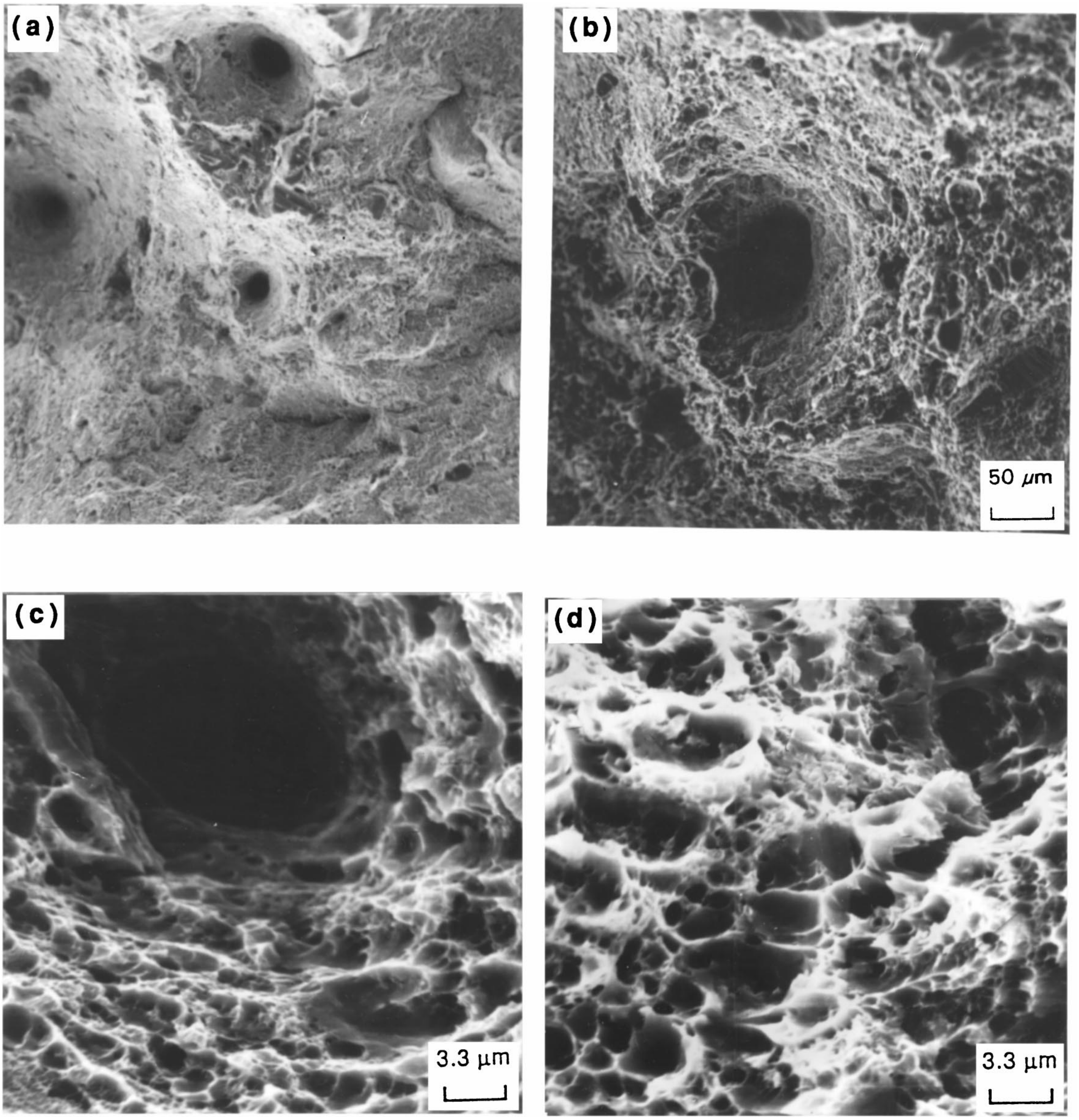

Figure 4 Scanning electron micrographs of the tensile fracture surface of the composite deformed at ambient temperature (27 °C): (a) overall morphology, (b) high magnification of (a), (c) voids and dimples on the fracture surface, and (d) shallow dimples on fracture surface.

indicative of the ductile nature of failure of the composite matrix (Fig. 4c). The fracture behavior conforms well with the ductility of the copper-niobium composite microstructure. Many of the microscopic voids coalesce and the halves of these voids are the shallow dimples visible on the tensile fracture surface (Fig. 4d). Fracture at the microscopic level revealed features reminiscent of ductile failure, that is distribution of macroscopic and fine microscopic voids and shallow dimples.

3.3.2. Elevated temperature (175 °C)

On the macroscopic scale tensile fracture revealed a cup and cone morphology. High magnification observations revealed the presence of both macroscopic and fine microscopic cracks (Fig. 5a), a population of microscopic voids of a wide range of sizes (Fig. 5b) and a large population of dimples, features reminiscent of extensive local microplasticity and ductile failure. The microscopic voids were homogeneously distributed throughout the tensile fracture surface and their near-equiaxed shape suggests that they nucleated around the second-phase inclusions, the niobium particle reinforcements, during the later stages of deformation. Under the influence of the far-field tensile load, triaxial stresses are generated but appeared to have little to no influence on void growth. Since crack extension under quasi-static loading occurs at high stress intensities comparable to the fracture toughness of the material, the presence of both microscopic and macroscopic voids degrades the actual strain to failure associated with ductile failure. The very fine microscopic voids coalesce and the halves

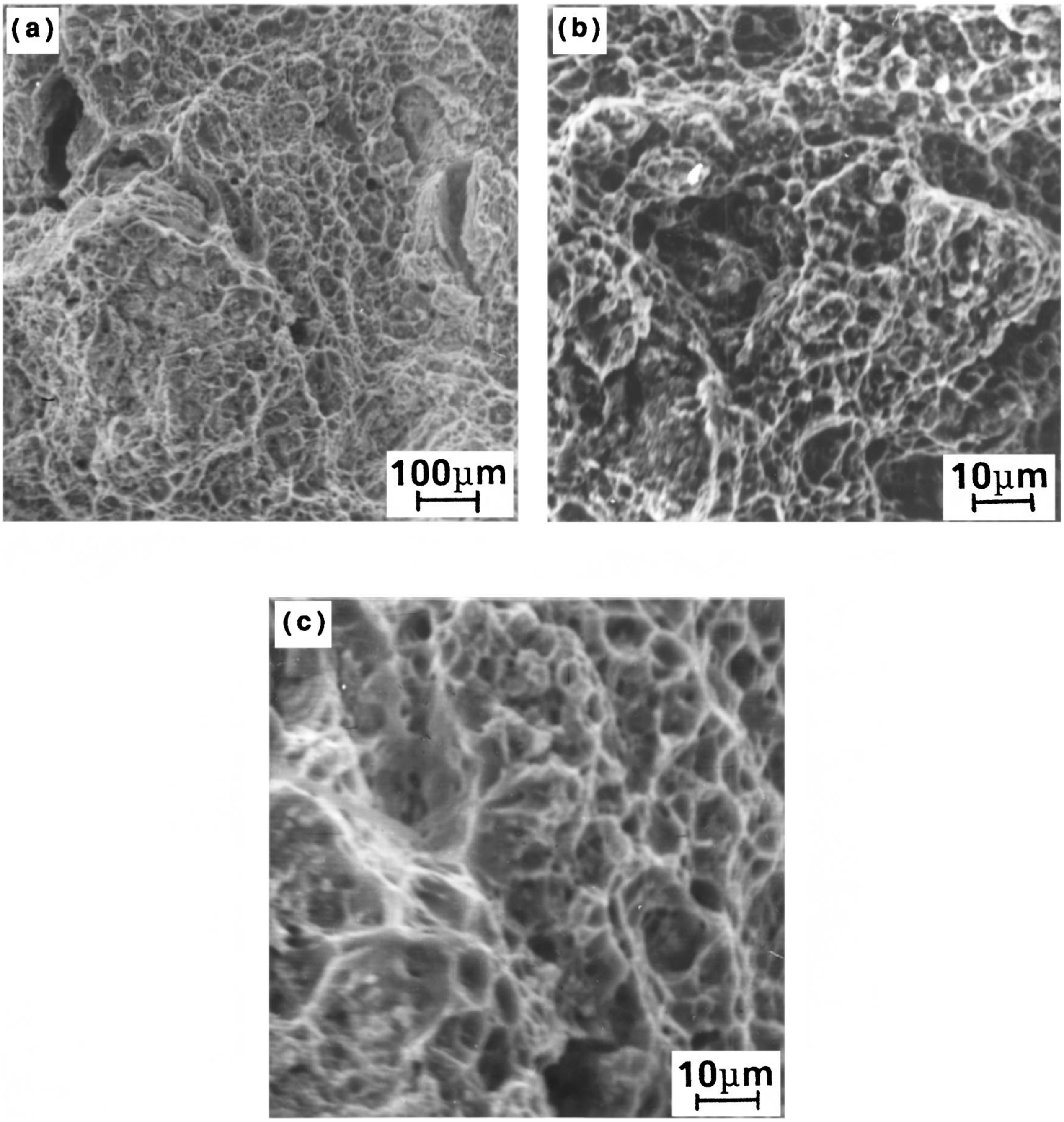

Figure 5 Scanning electron micrographs of the tensile fracture surface of the composite deformed at the elevated temperature (175 °C): (a) overall morphology, (b) high magnification of (a), and (c) shallow dimples on fracture surface.

of these voids are the shallow dimples on the fracture surface (Fig. 5c). The macroscopic fracture morphology coupled with an observation of fewer macroscopic and microscopic cracks and an enhanced population of voids and shallow dimples on the fracture surface conform well with the enhanced ductility of the composite microstructure at this test temperature. The dimples result from the coalescence of the microscopic voids.

4. Conclusions

Based on a study of the tensile deformation and fracture behavior of a ductile phase reinforced dispersion strengthened copper composite the following are the key observations:

1. The longitudinal microstructure of the AL-15+ NB1000 composite revealed ribbon-like filaments of the reinforcing phase. The transverse microstructure revealed a near-uniform distribution of the niobium particulates in the oxide dispersion strengthened copper matrix.

2. Strengthening in this dispersion strengthened copper + niobium composite arises from the mutually interactive influences of the difficulty in propagating plastic flow across the niobium particle reinforcements and nonhomogeneous deformation which necessitates a high density of dislocations be generated to maintain compatibility between the DSC matrix and the reinforcement. The Al_2O_3 particle reinforced copper matrix derives its strength from a combination of dispersion strengthening, dislocation-particle interaction and the occurrence of local climb of the mobile dislocations over the particles.

3. Increase in test temperature was found to have no influence on elastic modulus and yield strength of the composite. The degradation in ultimate tensile strength was marginal with a concomitant improvement in ductility.

4. The oxide dispersion strengthened copper-niobium composite maintained a high ratio of yield strength-to-ultimate tensile strength indicative of its intrinsic ability to resist softening with exposure to elevated temperatures.

5. Fracture morphology was identical at the two temperatures with macroscopic and microscopic features reminiscent of locally ductile failure with extensive microplasticity.

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