PRESSURE-LESS INFILTRATION OF METAL MATRIX NANOCOMPOSITES

Schultz, B.F., Rohatgi, P.K.
Materials Department, University of Wisconsin-Milwaukee, 3200 N. Cramer St., Milwaukee, WI 53211

Introduction

Metal matrix nanocomposites, loosely defined as multi-phase metallic materials where the governing material characteristics are imparted by imbedded nanoscale phases or structure (such as grain size), are a subject of active research due to their unique properties such as high hardness, strength, electrical conductivity, and wear resistance. These materials have been synthesized by a variety of methods including powder metallurgy, liquid metallurgy, chemical vapor deposition, and deformation processing followed by heat treatment [1].

Unfortunately, the benefit of the high strength of a nanocomposite is often offset by poor ductility. This is due to material imperfections in nanoscale grains, and defects from processing, as well as plastic instability and low capacity for work hardening as described by Ma [2]. For this reason, careful strategies must be adopted for the processing and structural design of nanocomposites to impart ductility. One such strategy is to disperse nanosize ceramics in nano- or micro-grain sized metals, which improves the strength by orowan strengthening mechanisms. The pressureless infiltration method is a cross between powder metallurgy and solidification processing which can be tailored to result in a microstructure of micro and nanoscale grains with nanoscale reinforcements.

Pressureless infiltration synthesis of metal matrix nanocomposites

Pressureless infiltration has been used to prepare composites containing multiwall carbon nanotubes. Noguchi et al [3] synthesized an 99.8% pure aluminum-MWCNT composite by a modified pressureless infiltration technique. I this study, the composite was synthesized by infiltrating a preform containing MWCNTs, magnesium and aluminum powders embedded in natural rubber with an aluminum alloy placed in contact with the perform at 800 ^{o}C for 1 hr in a nitrogen atmosphere. The authors claim that the gases formed and removed by the decomposition of the natural rubber resulted in increased wetting between the aluminum and the MWCNT, as well as the breaking up of oxide films on the aluminum. The addition of only 1.6 v% of MWCNT resulted in a sevenfold increase in the yield strength of the composite relative to that of the matrix metal.

In a more recent work, an Al-3.8-4.9%Cu-1.2-1.8%Mg-0.3-0.9%Mn (LY12 alloy) –CNT composite was synthesized using pressureless infiltration [4]. The CNTs, 100 µm aluminum powder and 70 µm magnesium powders were mixed together by ball milling under an argon atmosphere and 300 rpm milling speed for 7 hours. The ball-milled mixture was then pressed into preforms that were subsequently infiltrated by melts of the LY12 alloy matrix at 800° C in a nitrogen atmosphere. The CNTs were observed to be well dispersed and embedded in the matrix near the grain boundaries. Further experiments showed that up to 20 volume percent of nanotubes could be incorporated in the matrix of aluminum alloys using this process. They found that both nitrogen and magnesium/aluminum powders (ratio of 2:7 by weight) were required in order to initiate spontaneous infiltration of the preforms.

The aim of this work is to examine the pressureless infiltration synthesis of and Al-Cu-Mg alloy reinforced with nanoscale alumina to produce a bimodal microstructure.

Experimental

In this work, the factors of processing time, temperature and volume % reinforcement were examined for the Al-4.2-5%Cu-.2-.35%Mg-.2-.5%Mn (A206 alloy) matrix systems. Pure metal powders of spherical Al (<74 µm) and Mg (<44 µm) were mixed in a ratio of 7:2 with spherical Nanotek 70:30 Delta Gamma Aluminum oxide particles (~47 nm). The powders were dry mixed using an attritor mill for 5 hours under argon cover, followed by pressing into cylindrical cans of A206 aluminum. These compacts were fired

at various temperatures and for set lengths of time. The experimental conditions are summarized in Table 1 below.

Table 1 Experimental Conditions

Factor	Coded Level		
	-1	0	1
Time (/h)	1	3	5
Reinforcement % (/wt%)	0	10	20
Temperature (/^{o}C)	850	900	950

A Box-Behnken experimental design was utilized to determine the effects of processing conditions of time, temperature and reinforcement content on the percentage porosity of the composites synthesized in this study. Samples were characterized by optical, high resolution scanning electron microscopy, EDS and TEM. Compression testing of sub-size cubic specimens (. 5 x 5 x 5 mm) was used to test the mechanical properties of the pressureless infiltrated composites due to the brittle nature of the as infiltrated material.

Solidification Microstructure of Metal Matrix Nanocomposites

The pressureless infiltrated Al-Cu-Mg-oxide nanopowder samples appear in all cases to be multi-modal; that is, their characteristic features can be represented in multiple length scales down to the nanosize. HRSEM and TEM of such a composite is shown in Figure 1. In this case they are composed of "large" infiltrated Al-Cu grains (d ~100-300 microns) surrounded by finer (cellular) Al-Cu-Mg grains (d ~ 10-50 microns) that are themselves surrounded by a second phase containing Al, Cu, Mg and Oxygen as confirmed by EDS and selected area diffraction. TEM of the samples identified the presence of nanosize Al_2MgO_4 and Cu_4O_3 rather than Al_2O_3 indicating that the aluminum oxide has converted to Copper oxides and spinel.

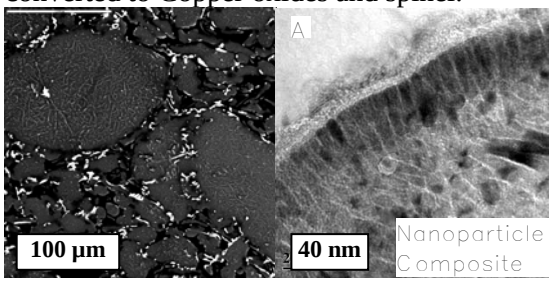

Figure 1 Backscattered high resolution SEM image (a) and TEM image (b) of Al-Cu-Mg- 20 wt% nanoparticle composite.

Each sample contained a finite percentage of porosity leading to a reduction in the mechanical properties of the pressureless infiltrated materials and the porosity content depended upon processing temperature, infiltration time/holding time, and percentage reinforcement. Response surfaces were generated that can give insight into the optimal processing conditions to result in a lowered percentage porosity. The predicted minimum % porosity for 10 and 20 weight percentage composites occur near the midpoint of time, indicating that a 3 hour process time may be sufficient for infiltration.

Compressive testing of the sub-size cubic specimens produced in the study exhibit a marked increase in compressive strength due to the presence of nano-size spinel and copper oxides. Table 2 summarizes the results of compression testing of these materials processed under the same conditions.

Table 2 Compressive strength of pressureless infiltrated materials. (n.p. indicates nanosize particles of spinel or copper oxide)

Material	σ Y.S. /psi	%increase
Al–Cu–Mg Base	12975	–
Al–Cu–Mg–10 wt% n.p.	17386	34%
Al–Cu–Mg–20 wt% n.p.	27072	109%

Conclusions

Pressureless infiltration is a viable method for the synthesis of metal matrix nanocomposites with bi-modal microstructures of coarse Aluminum grains, and fine nanoparticles reinforced regions. In this study, a 5 hour processing time resulted in the conversion of Al_2O_3 nanoparticles to nanometric Al_2MgO_4 spinel, as well as Cu_4O_3. The compressive strength of the composites synthesized in this work was increased by up to twice the strength of the base alloy synthesized under the same processing conditions, showing the potential of strengthening aluminum alloys and creating master alloys by formation of nanosize spinel and oxides by pressureless infiltration of alumina nanoparticles.

References

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