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Microstructure-Strength Relationship of a Deformation-Processed
Aluminum-Magnesium Composite

by

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1. DEFORMATION-PROCESSED METAL MATRIX COMPOSITES

1.1 Introduction

Composites formed by mechanical working (i.e., rolling, swaging, or wire drawing) of two-phase ductile metal mixtures have received substantial consideration in recent years because of the unique properties they show compared to conventional materials. During mechanical deformation, the two phases deform together, causing the second minor phase to elongate, which results in a fibrous structure within the matrix. Therefore, these composites are referred to as *in situ* composites or Deformation-Processed Metal Matrix Composites (DMMC's).

This type of composite can be fabricated in various ways. The first step to fabricate the deformation-processed composites is the production of a two-phase billet. This can be achieved by casting techniques or powder metallurgy processes. Casting methods can be used for composites if the phase equilibria of the alloy possess a two-phase equilibrium field. Powder metallurgy methods can be used for composites of elements with disparate melting points or unfavorable equilibrium phase diagrams.

Deformation-processed metal matrix composites encompass a fascinating group of materials with extraordinary properties. Previous studies of DMMC's have shown anomalous strengthening in face-centered cubic (FCC) matrix materials such as Cu and Al containing body-centered cubic (BCC) second phase metals such as Nb, Fe, Cr or Ta [1-12]. Some recent studies of Ti-Y [13-16], Mg-Li-Ti [17-19], Al-Ti [20] have shown hexagonal close packed (HCP) metals' potential application in DMMC's. The HCP metals will develop a $\langle 10\bar{1}0 \rangle$ wire texture when axisymmetrically deformed. This texture restricts the HCP phase to plain strain, which means that a FCC matrix containing HCP second phase material will deform in a similar way to that of FCC/BCC composites. Therefore, anomalous strengthening will also be observed. In this study, a FCC matrix Al containing HCP Mg will be studied to explore the relationship between properties and microstructures.

1.2 Cu-X Composites

During the last several years, a new class of copper-refractory metal composites (Cu-X composites) has been developed with extraordinary electrical and mechanical properties [1-10]. These composites are comprised of FCC copper with 10-30% of Nb, V, Ta, Cr, or Fe. Nanometer-scale filaments (when drawn) or lamellae (when rolled) of the secondary phase embedded in the matrix were produced after heavy deformation. The Cu-20%Nb composite is the most thoroughly studied of these composites. The initial emphasis for this material was development of Cu-Nb₃Sn superconducting wires [21-23]. More recent research focuses on their unique mechanical properties which develop with the deformation processing. An ultimate tensile strength (UTS) of 2200 MPa at a true strain of $\eta=12$ was reported [6], where $\eta=\ln(A_0/A)$ and A_0 and A are the initial and final cross-sectional areas respectively. For sheet materials, the effective deformation strain can be written as $\eta_e=(2/\sqrt{3}) \eta = (2/\sqrt{3})\ln(t_0/t)$, where t_0 and t are the initial and final thickness of the sheet respectively [24].

The Cu-X composites have unique properties. The ductility of the composites is so good that a starting billet can be drawn to a deformation ratio as high as $\eta=13.4$ without breaking, which means a 99.99985% reduction in area [8]. It is an 800-fold reduction in diameter, which is accompanied by a corresponding reduction in the size and spacing of the second phase. The extraordinary strength of these composites is one of the unique properties. In conventional composites, a property can often be calculated according to the properties of the individual components through the rule of mixtures (ROM). For a binary structure with component A and B with volume fractions V_A and V_B and properties F_A and F_B , its property F is given by: $F= V_A F_A+ V_B F_B$. But metal matrix composites in which the second phase is finely dispersed can have properties that deviate strongly from the ROM. A deformation-processed metal matrix composite can have a tensile strength much higher than the strengths predicted from ROM. Figure 1.1 shows the ultimate tensile strength for Cu, Nb and Cu-20Nb, as well as the ROM predictions for the Cu-20Nb composite at $\eta=12$ [25]. It can be seen that the Cu-20Nb wire is much stronger than the ROM prediction for this composite. Many studies were done to find the

reason for this substantial strength, most of the research centered on the strengthening mechanism. The nanoscale filament structure was thought to impede the propagation of dislocations in both the matrix and the second phase, which leads to the substantial strength of the composites [26-28].

Figure 1.1. The influence of deformation ratio on the ultimate tensile strength [25].

The typical microstructure of heavily deformed DMMC rod and sheet materials is shown in Figure 1.2. Ribbon-shaped filaments that are parallel to the rolling plane in sheet materials or convoluted filaments in rod materials can be obtained by deformation. These filaments are characterized by high aspect ratios. Figure 1.3 shows a TEM micrograph of a DMMC Cu-20Nb wire with the folded ribbon morphology. Because the Nb is a body centered cubic metal, it will develop a $\langle 110 \rangle$ texture, after which plane strain conditions are produced [29, 30], and the second phase elongates into ribbon shaped filaments aligned along the deformation axis.

Figure 1.2. Filament morphology typically seen in transverse sections of DMMC's. The ribbon-shaped filaments (A) are most common in heavily deformed rod materials and the longitudinal filaments (B) in sheet materials. [19]

Figure 1.3. TEM conical scanning dark-field image of a transverse section of wire made from Cu-20vol.%Nb alloy drawn to $\eta = 9.8$. The bright filaments are Nb and the dark matrix is Cu [3].

1.3 Ti-Y Composites

In order to extend use of the deformation processing to non-cubic systems, researchers have studied hexagonal close packed (HCP) metals [13-20]. The candidate metals should possess good ductility, mutual immiscibility and roughly similar mechanical properties. One suitable combination for a HCP/HCP composite is Ti-Y composite. A Ti-20Y composite was extruded and wire drawn to a deformation ratio of $\eta=12.8$ with 700°C annealing to relieve stress after each 60% reduction in area. The as-cast microstructure is shown in Figure 1.4, from which two immiscible metal phases can be seen. With increasing deformation, the composite showed a gradually finer microstructure. The mechanical deformation changed the dendrite structure into a filamentary structure, which was similar to that observed in the Cu-X DMMC composites.

Figure 1.4. Scanning electron microscope (SEM) back-scattered electron micrograph of as-cast Ti-20Y. The dark phase is Ti; the light phase is Y (pitted in places by the polishing agents).

The size and spacing of the ribbon-shaped filaments decreased with increasing deformation, which results in a nanofilamentary microstructure, as shown in Figure 1.5. The average thickness of Y phase is 41nm at $\eta=7.27$. Decreasing phase sizes with increasing deformation is also shown in Table 1.1 [13].

Figure 1.5. Conical scan dynamic dark field TEM micrograph of Ti-20Y deformation processed by extrusion, swaging, and drawing to $\eta = 7.27$ and sectioned transversely.

Table 1.1. Average phase sizes measured by optical microscopy (OM), scanning electron microscopy (SEM), and transmission electron microscopy (TEM) for Ti-20Y.

True Strain (η)	Ti-20Y	
	Average Ti Phase Thickness (μm)	Average Y Phase Thickness (μm)
0	9.60 (SEM)	2.40 (SEM)
2.25	3.80 (SEM)	0.96 (SEM)
5.03	0.69 (TEM)	0.17 (TEM)
7.27	0.16 (TEM)	0.041 (TEM)

When $\eta > 9$, the Ti and Y phase developed an equiaxed structure resulting from recrystallization during the stress relief annealing. The annealing was used to prevent breakage during the cold working. The large surface area between the matrix and filament phases provides a large driving force for the recrystallization process. Filamentary structure similar to that previously observed was produced when the $\eta = 9$ material was further deformed, but there was not a significant increase in the material's strength.

The ultimate tensile strength of the Ti-20Y composite wire was more than tripled by deformation processing to $\eta = 7.27$, at which point the ribbon-shaped filamentary structure was still observed. At a higher deformation ratio, the tensile strength decreased because the phases recrystallized into an equiaxed structure. Ductilities of Ti-20Y composite ranged from 36% to 60% reduction in area at the fracture surface. Compared to pure Ti, the machinability of the Ti-20Y was also improved. It was qualitatively similar to that of mild steel.

1.4 Microstructure and texture development in deformation

The tensile strength resulting from cold axisymmetric deformation, such as rod rolling, swaging and wire drawing, depends on the crystal structures of the two metal phases, being greater for FCC/BCC composites (Cu/Fe, Cu/Cr, Cu/Nb, Ag/Fe, etc) than for FCC/FCC composites (Ag/Ni, Ag/Cu) [31]. The greater strengthening in the FCC/BCC composites is attributed to the BCC metal developing a ribbon-shaped cross section as a result of the $\langle 110 \rangle$ fiber texture that develops during cold axisymmetric deformation. Because the FCC metals have a large number of slip systems, the FCC matrix can deform in an axisymmetric manner during cold working. But the BCC second phase is contained in the matrix and forced to fold and twist around the wire axis to maintain compatibility with the matrix, resulting in the irregular ribbon-shaped cross section. In FCC/FCC composites, the second phase undergoes axisymmetric deformation just like the matrix, resulting in the cross section shape remaining nearly circular during processing, which is shown in Figure 1.6.

Figure 1.6. Micrograph of a transverse section of Au-Ag composite wire ($h=5.6$). The darker regions are silver filaments in the (lighter) gold matrix in this back-scattered electron image[32].

Figure 1.7 shows a three-dimensional graphic of the Nb filaments in a Cu-20Nb composite rod-rolled and wire drawn to a deformation ratio of $\eta=5.3$. The ribbon-shaped cross section and Nb filaments along the deformation axis can be clearly seen.

With increasing deformation, the spacing and size of the second phase filaments continuously decrease. In drawn Cu-X (where X is a BCC second metal phase immiscible in Cu) deformation-processed composites, the second phase usually forms a $\langle 110 \rangle$ fiber texture while the Cu matrix forms a $\langle 111 \rangle$ and/or $\langle 001 \rangle$ fiber texture. In a BCC crystal with a $\langle 110 \rangle$ texture, two of the four $\langle 111 \rangle$ directions (the $[1\bar{1}1]$ and $[\bar{1}11]$ directions) are perpendicular to the fiber axis and thus cannot slip. Only the remaining two $\langle 111 \rangle$ directions ($[111]$ and $[11\bar{1}]$) which lie opposite one another across the specimen center line can slip. Therefore, the BCC second phase is limited to plain strain [2, 29],

Figure 1.7. Cu-20Nb wire drawn to $\eta=5.3$.

as shown in Figure 1.8. However, the FCC matrix can readily deform with either the $\langle 111 \rangle$ or $\langle 001 \rangle$ fiber texture because these orientations possess three or four active slip directions. Therefore, the Cu-X composites are characterized by plane-strained second BCC filaments embedded in the Cu matrix whose multiple slip system can accommodate the changing filament aspect ratios of the second phase. This is the reason that the BCC filaments are curled and folded in the Cu matrix. We can also use this reason to explain the greater strengthening effect in FCC/BCC composites than in FCC/FCC composites. For instance, the Ag-Cu wire was comprised of all FCC metals with a much lower ultimate tensile strength (1250 to 1400 MPa) even at filament thicknesses as small as 12 to 25 nm [31]. Bevk, et al [2, 22] suggested that the presence of the plane strain phase is one of the possible reasons for the extraordinary strength of the Cu-X composites.

In the Ti-20Y deformation processed composite, the $\langle 10\bar{1}0 \rangle$ fiber texture in both the Ti and Y was observed, which limited both the Ti matrix and Y second phase to plane strain. The available slip systems in a HCP crystal with a $\langle 10\bar{1}0 \rangle$ fiber texture are similar to that in a BCC crystal with a $\langle 110 \rangle$ fiber texture. Compared to a FCC lattice with three or four slip directions, both HCP and BCC are limited to two slip directions which are opposite to each other across the rod center line. Both will develop a plain strain phase within the composite. Therefore, the HCP DMMC will undergo the same filament shaping processes that have been observed in the BCC filaments of the Cu-X composites. Ribbon-shaped phases will be seen in both the Ti and Y since both the Ti and Y are limited to plain strain in the $\langle 10\bar{1}0 \rangle$ fiber texture.

As mentioned in section 1.3, the Ti-20Y composite developed an equiaxed structure resulting from recrystallization during the stress relief annealing at strains larger than $\eta=7.27$. The Ti-20Y composite either recrystallized at high annealing temperature or broke during deformation if low annealing temperature was used, making it difficult to compare the properties of Ti-Y with Cu-X composites at high η values. The possible reason for Ti-20Y's inability to undergo large strains is the geometric difficulty of fitting

a composite containing 100% plain straining phases into a cylinder whose radius diminishes as deformation progresses. Assuming no volume change in deformation, a phase deformed by plane strain to a total strain of $\eta=5$ would elongate to 150 times its original length and reduce its thickness by more than 99%. Fitting such plane straining ribbons into an cylinder specimen is probably impossible even with the folding possible by basal slip [11, 18].

1.5 Electrical properties of deformation processed composites

Previous studies [2] showed that deformation processed Cu-X (X is a BCC metal) alloy possessed a better combination of strength and electrical conductivity than that of commercially available Cu alloys. If there is significant solubility of the X metal in Cu, the electrical conductivity will decrease sharply. Kim *et al* [33] showed the conductivity of Cu-15 vol%Cr decreased monotonically with temperature upon heating, except over a 400-500°C temperature. The (increased conductivity was believed to result from precipitation of Cr. The electrical properties of Al-20 vol%Ti were also studied [20]. The Al-20Ti composite did not lose strength even after 24 hours heat treating at 473K. This DMMC possessed a much higher UTS: ρ ratio than any other commercial Al alloy. The electrical resistivity of Al-20Ti was measured for a $\eta=12.1$ specimen as a function of heating and cooling. As the temperature increased to around 340°C, the Al reacted with Ti, which resulted in the change in electrical resistivity (Figure 1.9)

1.6 Purpose of the research

DMMC materials have several advantages such as high strength, good conductivity, and good formability over conventional metal matrix composites. Therefore, deformation processing provides us an alternate way to develop lightweight commercial materials.

One purpose of this research is to find a high-strength, lightweight, low-corrosion composite which can be used in structural applications such as aerospace or automotive industries. According to the given criteria, Al-20 vol%Mg and Al-13 vol%Mg were chosen. Aluminum was selected because of its good ductility, low weight and low corrosion. Magnesium has a very low density. Commercially available magnesium

Figure 1.9 A plot of resistivity of a pure Al-20Ti specimen at $\eta = 12.1$ during a heating cycle of 0.055°C per second followed by cooling to room temperature. The inflection point at 340°C in the heating curve is thought to correspond to the reaction to form Al_3Ti from the pure metallic elements [20].

alloys usually utilize precipitation hardening and are limited to low temperature applications. The possibility of producing a lightweight, strong composite has provided an impetus for the research presented here.

Deformation processing may provide a method to make nano-scale composites with unique mechanical and physical properties. Al and Mg will react at high temperature and form Al_3Mg_2 intermetallic compound. When Al_3Mg_2 is formed in an Al-13vol%Mg composite, it represents 80vol%Al and 20vol% Al_3Mg_2 . That is why Al-13Mg was used. Comparison between the 80vol%Al-20vol%Mg and 80vol%Al-20vol% Al_3Mg_2 will be made to obtain optimum properties of the composites. Because Al is a FCC metal and

Mg is a HCP metal, the microstructure of Al-Mg DMMC is expected to be similar to that of the previously studied Al-Ti composites [20] due to plain strain second phase. The strength, microstructure and texture will be studied extensively in this research, and a comparison of the microstructure-texture-strength relationship of Al-Mg and previously studied DMMCs is the second purpose of this study.

2. EXPERIMENTAL PROCEDURES

2.1. Sample preparation

High-purity Al powder was produced by the gas atomized rapid solidification (GARS) method by Ames Laboratory. The purity of Al powder is 99.99% with a particle size of 45-75 μ m (Figure 2.1). Mg powder was bought from Alfa Aesar company. The purity of Mg powder is 99.80%, with a particle size smaller than 45 μ m (-325 sieve size) (Figure 2.2). These two powders were mixed and cold isostatically pressed (CIP'ed) at 132MPa. There were two compositions for the Al-Mg composites. One was 80vol.% Al-20vol.% Mg, which is referred to as Al-20Mg; the other one was 87vol.% Al-13vol.% Mg, which was referred to as Al-13Mg. Then, the 37mm diameter green compacts were loaded into a Ta-foil-lined, OFHC copper can which was soldered shut and evacuated. The copper cans were extruded at room temperature through a 25.4mm diameter die followed by water quenching (Figure 2.3).

The true strain of the specimen was calculated with: $\eta=2\ln(d_0/d)$, where d_0 and d are the initial and final specimen diameter respectively. The extruded Al-20Mg rod with Cu sleeve was swaged at room temperature to a diameter of 12.7mm ($\eta=2.77$). At this point the Cu and Ta were removed by machining. The bare Al-20Mg was swaged to a diameter of 4.1mm ($\eta=4.37$), and 1.6mm ($\eta=6.29$). Metallography coupons and two tensile specimens were cut from the specimen at $\eta=2.77$, $\eta=4.37$ and $\eta=6.29$.

The 4.1mm diameter Al-20Mg rods were cleaned with acetone, cut to 34.3mm length, and bundled inside another Ta-foil-lined, OFHC Cu can. This Cu can was soldered and evacuated. The 51mm outside diameter, 182mm long Cu can was extruded at room temperature through a 25.4mm die followed by water quenching. During the second extrusion, the separate rods bonded together into a single piece with an deformation ratio of 5.47. The extruded rod was then swaged to a diameter of 3.2mm, at which point the Cu and Ta were removed by machining. The bare Al-20Mg rod was swaged at 300K to diameters of 1.8mm ($\eta=10.13$), and 1mm ($\eta=11.01$). A small metallography coupon and two tensile specimens were cut from the specimen at $\eta=10.13$, $\eta=11.01$. The specimen

Figure 2.1 Aluminum powder produced by Ames Laboratory with gas atomized rapid solidification (GARS) process.

Figure 2.2 Magnesium powder used in the CIP'ed Al-Mg compact.

Figure 2.3 Schematic of preparation and extrusion of Cu can.

Figure 2.4 Phase diagram of Al and Mg binary system [34].

was then wire-drawing to a diameter of 0.64mm ($\eta=11.95$). A small metallography coupon and two tensile specimen were cut from this specimen.

The extruded Al-13Mg rod with Cu sleeve was swaged at room temperature to a diameter of 12.7mm ($\eta=2.77$) and 5.8mm ($\eta=4.33$). At $\eta=4.33$, the Cu and Ta were removed by machining. The bare specimen was swaged to a diameter of 0.8mm ($\eta=6.24$). Metallography coupons and two tensile specimens were cut from the specimen at $\eta=2.77$, $\eta=4.37$, $\eta=6.24$.

After the Al-13Mg ($\eta=4.33$) was machined, most of it was set aside for the second extrusion. The bare Al-13Mg rods were cleaned with acetone, cut into 51mm length, and bundled inside another Ta-foil-lined, OFHC Cu can. This Cu can was soldered and evacuated. The 51mm outside diameter, 182mm long Cu can was extruded at room temperature through a 25.4mm die followed by water quenching. During the second extrusion, the separate rods bonded together with a deformation ratio of 5.43. The extruded rod was then swaged to a diameter of 3.2mm ($\eta=9.59$), at which point the Cu and Ta were removed by machining. The bare Al-20Mg rod was swaged at 300K to a diameter of 1.8mm ($\eta=10.10$) and 1.0mm ($\eta=11.28$). A small metallography coupon and two tensile specimens were cut from the specimen at $\eta=10.10$ and $\eta=11.28$.

2.2 Mechanical testing

Tensile tests were performed for specimens with $\eta=2.77$ to $\eta=11.25$ for Al-20Mg and with $\eta=2.77$ to $\eta=11.28$ for Al-13Mg following ASTM standard E8 [35], which requires a 4:1 ratio between the gauge length and specimen diameter. None-standard tensile specimens were used for some high deformation ratio samples with a diameter less than 1.0mm. Specimen gauge lengths were polished with a 600-mesh abrasive paper to eliminate lathe tool marks on the gauge length surface. All the tensile tests were performed on an Instron 4505 tensile test machine at a strain rate of 1.0mm/min (when $\eta<6$), and 0.15mm/min (when $\eta>6$) at room temperature. At least two tensile measurements were made for each specimen whenever possible. A digital micrometer

was used to measure gauge sizes for specimen diameters larger than 1.5mm and a travelling microscope was used to measure gauge sizes for specimen diameters smaller than 1.5mm. An optical travelling microscope was used to measure the area of the fracture surface and corresponding ductility was calculated by comparing the area of the original cross sectional of the specimen to the area of the fracture surface. For all the specimens, their ultimate tensile strength (UTS) was calculated from the applied maximum load and the initial cross sectional area of the specimen ($UTS = \text{load} / \text{area}$).

2.3 Microstructure analysis

Scanning electron microscopy (SEM) was used to measure the phase spacing and inspect the surface morphology for samples at various levels of deformation. SEM specimens were prepared at the deformation ratio of 2.77, 4.33, 6.24, 10.10, 11.28 for Al-13Mg and at the deformation ratio of 2.77, 4.37, 6.49, 10.13, 11.01 and 11.95 for Al-20Mg. SEM specimens were prepared by standard metallographic methods including mounting, mechanical polishing. Both transverse and longitudinal sections were made for all the specimens. A JOEL6100 SEM unit and a Cambridge S-200 unit at accelerating voltage of 15-20KV was used to image the samples.

Quantitative stereology described by Underwood [36] was used to determine the size and spacing of Mg filaments at different η values. The filament spacing was calculated from SEM pictures using a circle of known diameter, and then counting the number of filaments that intercepted the circle (N_L).

In this study the mean true spacing was measured using:

$$\sigma_t = \sigma_r / 2 = 1 / 2 N_L$$

where σ_t = mean true spacing, σ_r = mean random spacing (or mean center-to-center spacing) and N_L = number of filaments intercepted per unit length of test line. The mean edge-to-edge spacing, called λ (or mean free path) between the filaments is given by:

$$\lambda = (1 - V_f) / N_L = V_m / N_L$$

where V_f is the volume fraction of filaments, V_m is the volume fraction of the matrix and N_L is the number of interceptions per unit length of test lines. The average filament thickness (t) of the Mg fibers was estimated from the following equation:

$$t = (1 - V_m)/N_L = V_f / N_L$$

where V_m is the volume fraction of the matrix, V_f is the volume fraction of fibers, and N_L is the number of interceptions with the fibers per unit length of test lines.

2.4 Differential thermal analysis procedures

Differential thermal analysis (DTA) is a commonly used thermal materials characterization method. The method can show phase transitions like melting, crystallization and chemical reactions easily. A Perkin Elmer DTA 7 apparatus was used. DTA was performed on samples of Al-13Mg ($\eta=11.28$) and Al-20Mg ($\eta=11.25$) to determine the reaction temperature between Al and Mg. The temperature range was from room temperature to 400°C, and heating rate was 10°C/min.

2.5 X-ray diffraction analysis

X-ray diffraction analysis was done on the $\eta=2.77$ and $\eta=10.10$ specimens for Al-13Mg and on the $\eta=2.77$ and $\eta=10.13$ specimens for Al-20Mg to determine if the intermetallic compound is formed during deformation processing. After a Al-13Mg ($\eta=11.28$) was heat treated, X-ray diffraction analysis was also done on it to determine if the new phase formed. All X-ray diffraction was performed on a Philips 1700 series diffractometer. Two theta scans from 20° to 100° were used with a scanning step size of .05 degree and an interval of 3 seconds per step.

2.6 Orientation Image Microscopy procedures

The transverse section of Al-13Mg at deformation levels of 11.28 was used for Orientation Image Microscopy (OIM) analysis. The sample was prepared by

conventional metallographic method followed by ion milling for about 3 hours at 3.5 KV and with 0.5 mA ion current.

An Amray 1845 field emission gun SEM and equipment developed by TexSEM Laboratories (TSL) were used to obtain OIM pole figures from electron back scatter diffraction patterns (EBSP's). The EBSP's were analyzed using TSL's software running on a Silicon Graphics Inc. Indy workstation.

2.7 Resistivity measurement procedures

The specimen Al-13Mg($\eta=11.28$) was annealed at 538K in vacuum for 3600s (1 hr). The electrical resistivity, ρ , of the specimen both before and after the annealing was measured at room temperature. The ultimate tensile strength of this sample was also measured both before and after the 1hr annealing at 538K in vacuum.

A Keithley 580 micro-ohmmeter was used to measure the sample's resistivity. The micro-ohmmeter was operated in the pulse mode to avoid possible thermal EMF's. The drawn wire samples have uniform cross section area and small grooves were machined into each sample for voltage probes. Both ends of the sample were clipped with source probes. Four points resistance measurement was performed. The measured resistance R was converted into resistivity with following equation:

$$\rho = \frac{RA}{d}$$

where ρ is the resistivity of sample, R is the resistance, A is the cross sectional area of the wire, and d is the distance between voltage probes.

3. RESULTS AND DISCUSSIONS

3.1 Microstructure analysis

The microstructure of a material directly determines the properties of the material. Only when microstructure is understood well can people know the relationship between microstructure and properties. And only after this can people know how to change and control microstructures to achieve better properties.

SEM samples at various deformation ratios were prepared by traditional metallographic method. No etching agent was needed because Al and Mg show good contrast in back-scattered electron imaging mode. Both longitudinal and transverse section pictures were taken by SEM for all specimens.

SEM photographs gave information on the phase morphology of the specimens at various levels of deformation processing. SEM photographs were also used to measure the phase spacing by quantitative stereology method. The results are presented in Table 3.1 and Table 3.2.

After the Al-Mg composite was extruded and swaged, an elongated filament structure of Al and Mg was developed (Figure 3.1-3.2). Subsequent deformation processing further reduced the filament thickness and spacing, as shown in Figure 3.3-3.5. Many of the Mg filaments in the Al matrix finally displayed a convoluted ribbon shape at transverse section predicted by the plain strain mode described in the previous literature review. The “convoluted ribbon shaped” filaments in Figure 3.3-3.5 presumably result from the $\langle 10\bar{1}0 \rangle$ fiber texture of HCP Mg. All of the sample displayed a closely spaced, elongated filament structure after heavy deformation, which was thought to be the possible reason for the increase in strength with deformation.

Based on the definition of deformation strain, the filament size in rod material at a given level of deformation can be described by:

$$d = d_0 \exp(-\eta/2)$$

where d_0 is the initial fiber size and d is the fiber size at a given deformation ratio(η). The

a)

b)

Figure 3.1 SEM micrographs of Al-13Mg deformation-processed to a true strain (η) of 2.77. a) is longitudinal section, b) is transverse section. The Al matrix is light gray, and the Mg is dark gray in these back-scattered electron images.

a)

b)

Figure 3.2 SEM micrographs of Al-13Mg deformation-processed to a true strain (η) of 4.33. a) is longitudinal section, b) is transverse section. The Al matrix is light gray, and the Mg is dark gray in these back-scattered electron images.

a)

b)

Figure 3.3 SEM micrographs of Al-13Mg deformation-processed to a true strain (η) of 6.24. a) is longitudinal section, b) is transverse section. The Al matrix is light gray, and the Mg is dark gray in these back-scattered electron images.

Figure 3.4 SEM micrographs of Al-13Mg deformation-processed to a true strain (η) of 10.10 and mounted in transverse section. The Al matrix is light gray, and the Mg is dark gray in these back-scattered electron images.

a)

b)

Figure 3.5 SEM micrographs of Al-13Mg deformation-processed to a true strain (η) of 11.28. a) is longitudinal section, b) is transverse section. The Al matrix is light gray, and the Mg is dark gray in these back-scattered electron images.

Figure 3.6 SEM micrographs of transverse section of Al-13Mg. a) $\eta=2.77$, b) $\eta=4.33$, c) $\eta=6.24$, d) $\eta=10.10$, e) $\eta=11.28$. The Al matrix is light gray, and the Mg is dark gray in these back-scattered electron images.

Figure 3.7 SEM micrographs of transverse section of Al-20Mg. a) $\eta=2.77$, b) $\eta=4.37$, c) $\eta=6.49$, d) $\eta=10.13$, e) $\eta=11.01$, f) $\eta=11.95$. The Al matrix is light gray, and the Mg is dark gray in these back-scattered electron images.

Table 3.1. Mean true spacing, mean free distance, and Mg filaments thickness of Al-20Mg at various levels of deformation strain.

Deformation strain (η)	Mean true spacing (σ_t)	Mean free distance (λ)	Filament thickness (t)
2.77	9.72 μm	15.56 μm	3.89 μm
4.37	8.29 μm	13.38 μm	3.34 μm
6.49	5.60 μm	8.95 μm	2.24 μm
10.13	3.36 μm	5.37 μm	1.34 μm
11.01	1.88 μm	3.00 μm	0.75 μm
11.95	1.65 μm	2.67 μm	0.64 μm

Table 3.2. Mean true spacing, mean free distance, and Mg filaments thickness of Al-13Mg at various levels of deformation strain determined by quantitative stereology.

Deformation strain (η)	Mean true spacing (σ_t)	Mean free distance (λ)	Filament thickness (t)
2.77	13.84 μm	24.09 μm	3.60 μm
4.33	11.15 μm	19.41 μm	2.90 μm
6.24	5.08 μm	8.85 μm	1.33 μm
10.10	2.96 μm	5.15 μm	0.77 μm
11.28	2.71 μm	4.72 μm	0.71 μm

calculation results were shown in Table 3.3. This equation assumes that the fiber and matrix deform in proportion to the amount of deformation applied to the bulk material. The theory prediction was compared to the quantitative stereology measurements of filament size, as shown in Figure 3.8.

According to Figure 3.8, there is difference between the stereology measurement results and calculation results. The Mg filaments size from stereology measurement are

Table 3.3 The Mg filaments thickness at various levels of deformation strain determined by calculation.

Al-13Mg		Al-20Mg	
Deformation strain (η)	Filament thickness of Al-13Mg (t)	Deformation strain (η)	Filament thickness of Al-20Mg (t)
2.77	3.60 μm	2.77	3.89 μm
4.33	1.65 μm	4.37	1.75 μm
6.24	0.63 μm	6.49	0.61 μm
10.10	0.09 μm	10.13	0.098 μm
11.28	0.05 μm	11.01	0.063 μm

Figure 3.8 The dependence of filament thickness on deformation ratio from quantitative stereology measurement and from calculation for Al-13Mg and Al-20Mg.

larger than the filaments size from calculation. The theory calculation assumes that both the Al matrix and Mg filaments deform in proportion to bulk material. However, because Al is FCC metal with lots of slip systems, Mg has fewer slip systems since it is a HCP metal, Mg does not deform at the same extent as Al matrix. This is the possible reason why Mg filaments size is larger than that of theory prediction.

3.2 Tensile test results

To investigate the influence of deformation on the strength of composites, tensile tests were performed for both compositions at the previously described deformation ratios. According to previous study on Cu-X [2-10] and other DMMCs [11-16], the larger the deformation ratio, the higher the ultimate tensile strength. Tensile tests were done for the Al-Mg composites to see if a similar phenomenon exists here.

Table 3.4 and Table 3.5 show the tensile test data and ductility of Al-20Mg and Al-13Mg respectively.

According to Table 3.4 and Table 3.5, the tensile strength of Al-Mg composites increased as deformation processing proceeded, which is shown in Figure 3.9. The tensile strength of Al-20Mg increased 223% from $\eta=2.77$ to $\eta=11.95$, and the tensile strength of

Table 3.4. Ultimate tensile strength (UTS) and ductility of Al-20Mg measured at 300K for various levels of deformation strain.

Deformation strain (η)	Ultimate Tensile Strength (MPa)	Ductility as percent reduction in area
2.77	130	21%
4.37	170	24%
6.49	210	14%
10.13	320	7%
11.02	370	7%
11.95	420	8%

Table 3.5. Ultimate tensile strength (UTS) and ductility of Al-13Mg measured at 300K for various levels of deformation strain.

Deformation strain (η)	Ultimate Tensile Strength (MPa)	Ductility as percent reduction in area
2.77	120	24%
4.33	134	30%
6.24	220	20%
10.10	250	14%
11.28	310	9%

Figure 3.9 The dependence of ultimate tensile strength on deformation ratio for Al-13Mg and Al-20Mg.

Al-13Mg increased 160% from $\eta=2.77$ to $\eta=11.28$ (Figure 3.9). However, the ductility decreased with the extent of deformation. One significant feature of Figure 3.9 was the relatively low increase in ultimate tensile strength when the specimen was deformed from $\eta=6.24$ to $\eta=10.10$ for Al-13Mg. When the Al-13Mg specimen was deformed, they all underwent a second extrusion as described in section 2 of this thesis to achieve a higher deformation value η . During the a second extrusion, the separate rods may not bond well, which was harmful to the strength of the composite. This is the possible reason for the relatively low increase in ultimate tensile strength. Because those second phase filaments act as the barrier of dislocation movement, the increase of ultimate tensile strength presumably results from the decreasing of filaments spacing.

The strengthening mechanisms of the deformation processed composites is less well understood and has remained to be the subject of extensive debate and study. The Al-Mg data obtained in this study were used to compare the predicted data of some of the models to the experimental data. The strengthening models used here were:

1. Hall-Petch model[1];
2. The Embury modified Hall-Petch model[37].

The flow stress of a metal is almost universally observed to increase as the grain size decreases. There exists an empirically observed correlation between the strength and grain size:

$$\sigma = \sigma_0 + kd^{-1/2}$$

where σ_0 and k are constants, d is grain size. This equation is known as the Hall-Petch relationship. Similarly, the Hall-Petch relationship can be applied to deformation-processed composites:

$$\sigma = \sigma_0 + k\lambda^{-1/2}$$

where λ is the filament spacing.

Utilizing the data of Al-20Mg and Al-13Mg, it can be calculated that (Figure 3.10):

$$\text{For Al-20Mg, } \sigma = -35.4 + 738\lambda^{-1/2}$$

$$\text{For Al-13Mg, } \sigma = -13.2 + 660\lambda^{-1/2}$$

This strengthening mechanism is called microstructure hardening. The strengthening produced by the microstructure results from blockage of dislocation motion by the interfaces in the microstructure, i.e., the interface between Mg filaments and Al matrix. If a source within a grain is feeding dislocations into a grain boundary, the dislocation motion will be finally blocked at the boundary. Further motion requires generation of new dislocations in the neighboring grain by the local stress field generated by the blocked dislocations in the parent grain. The large number of interfaces between Mg filaments and Al matrix inhibit the motion of dislocations, which gives rise to the high strength of the deformation processed composites. Moreover, the more the composites were deformed, the larger the total area of interfaces between the filaments and matrix, leading to higher strength of the composites.

Figure 3.10 Hall-Petch relationship for Al-13Mg and Al-20Mg composite.

Embury modified Hall-Petch model assumed that the barrier spacing d_e controlling flow changed from initial value d_0 in proportion to the reduction of the wire diameter:

$$d_0/d_e = D_0/D_e$$

where D_0 and D_e are specimen diameter before and after strain. Since $\eta = 2\ln(D_0/D_e) = 2\ln(d_0/d_e)$, then $d_e = d_0 \exp(-\eta/2)$. It can be derived by combining with the Hall-Petch equation ($\sigma = \sigma_0 + kd^{-1/2}$) to obtain:

$$\sigma_f = \sigma_0 + kd_0^{-1/2} \exp(\eta/4)$$

The above model is simplistic, but it provides a useful way of assessing the influence of the microstructure on the strength.

According to the Embury modified Hall-Petch model, there exists an exponential relationship between strength and deformation ratio. To apply this model to Al-Mg data, the data in Table 3.4 and Table 3.5 were used to simulate exponential curves. It was found from the curve fitting that:

$$UTS = 95.0 \exp(0.123\eta) \text{ for Al-20Mg}$$

$$UTS = 92.4 \exp(0.107\eta) \text{ for Al-13Mg.}$$

The predicted values of σ_f were tabulated in Table 3.6 and shown in Figure 3.11.

Table 3.6 Ultimate tensile strength predicted by curve fitting for Al-13Mg and Al-20Mg.

Al-13Mg		Al-20Mg	
Deformation strain (η)	Calculated ultimate tensile strength (MPa)	Deformation strain (η)	Calculated ultimate tensile strength (MPa)
2.77	124	2.77	134
4.33	147	4.37	163
6.24	180	6.49	211
10.10	272	10.13	330
11.28	310	11.01	368
N/A	N/A	11.95	413

Previous studies on various DMMCs have quantified the relationship between filament spacing and ultimate tensile strength using a relation similar to the Hall-Petch type power function for yield strength. In Cu-20Nb axisymmetrically deformed DMMC's [3,6], UTS change was proportional to (Nb filament spacing)^x where x varied between

-0.5 at low strains to -0.38 at high strains, while in axisymmetrically deformed Ti-20Y DMMC [13], UTS change was proportional to (Y filament spacing)^{-0.37}. In the Al-20Ti DMMC, UTS change was proportional to (Ti filament spacing)^{-0.25}[20]. In Al-Mg DMMCs of this study, the relationships between ultimate tensile strength and mean true spacing of Mg filaments are as follows (Figure 3.12):

$$\text{UTS}=498 \sigma_t^{-0.54} \text{ for Al-13Mg}$$

$$\text{UTS}=585 \sigma_t^{-0.61} \text{ for Al-20Mg.}$$

where UTS= Ultimate tensile strength (MPa) and σ_t = mean true spacing of Mg filaments (nm). The strengthening effect in Al-Mg system is less than those in Cu-Nb, Ti-Y and Al-Ti systems. According to Verhoven et al[3], these relationships are quite sensitive to the resolution of the microscopy used to photograph the microstructure for stereology and the equipment used to obtain these micrographs (TEM vs. SEM) .

3.3 Differential thermal analysis results

DTA was performed on Al-13Mg ($\eta=11.28$) and Al-20Mg ($\eta=11.01$) to determine the reaction temperature between Al and Mg. Because the reaction between Al and Mg is an exothermic reaction, there will be a upward peak in DTA diagram. Therefore, the reaction temperature can be shown in DTA diagram. (Figure 3.13 and 3.14)

The Al-Mg compact was initially extruded at the temperature of 538K. But Al and Mg reacted at this temperature and intermetallic compound was formed according to DTA results (Figure 3.13). This intermetallic compound does not deform during swaging and wire drawing. Therefore, extrusion was performed at room temperature to avoid the formation of intermetallic compound.

Figure 3.11. The dependence of ultimate tensile strength on deformation ratio from quantitative stereology measurement and from calculation for Al-13Mg and Al-20Mg.

Figure 3.12 The dependence of ultimate tensile strength on mean true spacing for Al-13Mg and Al-20Mg.

Figure 3.13 DTA diagram of Al-13Mg ($\eta=1.28$). The reaction starts at 534K.

Figure 3.14 DTA diagram of Al-20Mg ($\eta=11.01$). The reaction starts at 535K.

3.4 X-ray diffraction analysis

According to the Al-Mg phase diagram as shown in Figure 2.4, Al and Mg will form Al_3Mg_2 intermetallic compound at appropriate temperature and composition. In order to determine if Al_3Mg_2 is formed, X-ray diffraction (XRD) is performed because it is an easy and fast way to identify phases present in the specimen.

For Al-13Mg, XRD was performed on the as-extruded ($\eta=2.77$), the second extruded ($\eta=10.10$) sample and heat treated sample ($\eta=11.28$) to see if reaction happened (Figure 3.15-3.17). For Al-20Mg, XRD was performed on the as-extruded ($\eta=2.77$), and the second extruded ($\eta=9.63$) sample to see if reaction happened (Figure 3.18-3.19).

According to Figure 3.15-3.16 the first extruded ($\eta=2.77$) and the second extruded ($\eta=10.10$) Al-13Mg samples showed a mixture of pure Al and Mg patterns without intermetallic phases, which suggested that Al and Mg did not react during the first and the second extrusion. The Cu peak in Figure 3.16 and Figure 3.17 resulted from the Cu sleeve surrounding the composite.

For the heat-treated (3600 seconds at 538K) Al-13Mg sample ($\eta=11.28$), there was not an obvious Al_3Mg_2 peak in XRD pattern despite DTA showed there was a reaction. Some Mg atoms may diffuse into Al matrix to form solid solution. Other Mg atoms may react with Al and form small amount Al_3Mg_2 or $\text{Al}_{3-x}\text{Mg}_{2+x}$. The resulted intermetallic compounds covered Mg filaments and separated Al and Mg, which prevented further reaction between Al and Mg. Therefore, the signal from Al_3Mg_2 in XRD is very faint. This may be a possible reason why there is not Al_3Mg_2 peak in XRD pattern.

The XRD patterns of Al-20Mg were similar to those of Al-13Mg as shown in Figure 3.18-3.19, which suggested no intermetallic compound was formed during the first and the second extrusion of Al-20Mg.

Figure 3.15 XRD of Al-13Mg ($\eta=2.77$). This pattern consists of a mixture of Al and Mg without intermetallic compound.

Figure 3.16. XRD of Al-13Mg ($\eta=10.10$). This pattern consists of a mixture of Al and Mg without intermetallic compound. The Cu peak results from the outside Cu sleeve.

Figure 3.17. XRD of Al-13Mg ($\eta=11.28$, heat treated for 3600 seconds at 538K). This pattern consists of ~~a~~ Al. The Cu peak results from the outside Cu sleeve.

Figure 3.18. XRD of Al-20Mg ($\eta=2.77$). This pattern consists of a mixture of Al and Mg without intermetallic compound.

Figure 3.19. XRD of Al-20Mg ($\eta=9.63$, the second extrusion). This pattern consists of a mixture of Al and Mg without intermetallic compound. The Cu peak results from outside Cu sleeve.

3.5 Orientation image microscopy

The effect of texture on the mechanical properties of materials is significant. When a polycrystalline metal is plastically deformed, all the individual grains will rotate. But the rotation is restricted due to the requirement of coherency at the grain boundaries. For FCC wire drawn or extruded sample, the texture is $\langle 111 \rangle$ and/or $\langle 001 \rangle$ fiber texture; For BCC wire drawn or extruded sample, the BCC metal has a $\langle 110 \rangle$ fiber texture; For HCP wire drawn or extruded sample, the texture is $\langle 10\bar{1}0 \rangle$ fiber texture[1]. The limited slip systems of BCC and HCP metals result in a plane strain mode.

Orientation image microscopy (OIM) pole figures for the Al-13Mg wire sample ($\eta=10.10$) confirmed that there was a $\langle 100 \rangle$ fiber texture in Al matrix, which was shown in Figure 3.20. Figure 3.21 showed there was not strong texture in Mg. One possible reason may be there is dynamic recovery and/or recrystallization, which needs further investigation.

Figure 3.20 OIM pole figure of Al for Al-13Mg ($\eta=10.10$) composite.

Figure 3.21 OIM pole figure of Mg for Al-13Mg ($\eta=10.10$) composite.

3.6 Resistivity measurement results

One of advantages of DMMCs is the combination of high strength and high conductivity (or low resistivity). Previous studies on Cu-Cr system and other Cu-X systems [33, 38] showed that the conductivity decreased with deformation ratio, as shown in Figure 3.22. The purpose of this study is to obtain a high strength wire which can be used for high conductivity applications. Since Al and Mg will react and forms intermetallic compound upon heating, the effect of the formation of this compound on conductivity needs to be studied.

Figure 3.22 - Strength versus conductivity of Cu-7 vol.% Cr data compared to literature data on Cu-15 vol.% X (where X = niobium, tantalum, and chromium). [36]

The highest η (11.28) value sample of Al-13Mg was studied since it has the highest tensile strength. Its electrical resistivity, ρ , was measured at room temperature both before and after 1hr annealing at 538K in vacuum. The ultimate tensile strength of this specimen was also measured both before and after the heat treating. The results were shown in Table 3.7.

Table 3.7 Electrical resistivity and ultimate tensile strength of Al-13Mg at $\eta=11.28$ before and after annealing 1hr at 538K

	Electrical resistivity ($n\Omega\cdot m$)	Ultimate tensile strength (MPa)
As-drawn (no annealing)	32.7	310.4
After annealing	55.2	287.2

According to Table 3.7, the Al-13Mg (as drawn) composite has a low electrical resistivity. The resistivity of pure Al is $28.2 n\Omega\cdot m$ at 293K[39]. The electrical resistivity of Al-13Mg composite is only 16% higher than that of pure Al. It is not surprising that Al-13Mg composite has a low electrical resistivity. The matrix of the DMMC is 99.99% purity Al without solid solution, GP zones. The electrical resistivity of pure Mg is $46 n\Omega\cdot m$ at 293K [39], which is moderately higher than that of pure Al ($27.3^{28.2} n\Omega\cdot m$ at 293K). Moreover, although the electrical resistivity of Mg is larger than that of Al, about 80% of the cross sectional area of the DMMC is long strands of high purity Al, which allows the electrons pass through easily.

After heat treating, some Mg diffused into Al matrix and formed solid solution. Other Mg reacted with Al and formed intermetallic compound. These led to the decrease of electrical resistivity.

The UTS: ρ ratios of some alloys are shown in Table 3.8. The reason that Al-13Mg has a lower UTS: ρ ratio than Al-20Ti is that Al-13Mg has a lower ultimate tensile strength than Al-20Ti. As mentioned in section 3.2 of this thesis, when Al-13Mg was deformed from $\eta=6.24$ to $\eta=10.10$, there was only moderate increase in tensile strength due to poor bonding between Al-13Mg rods. Further deformation processing may increase the tensile strength and thus increase the UTS: ρ ratio.

Table 3.8 Comparisons of UTS: ρ ratio of the different Al matrix composite, commercial Al alloy and pure Al.

Alloy	UTS: ρ ratio (MPa/n Ω ·m)
Al-13Mg DMMCs as drawn ($\eta=11.28$)	9.49
Al-13Mg DMMCs with 1 hour, 538K anneal ($\eta=11.28$)	5.20
Al _{GARS} -20Ti DMMC as drawn ($\eta=12.1$)	13.86
Al _{GARS} -20Ti DMMC with 24 hours, 200°C anneal ($\eta=12.1$)	13.81
Commercial alloy 7049, T73 condition	12.56
Pure Al, alloy 1100, O condition	3.10

4. CONCLUSIONS

1. Deformation processing can be used to produce a high-strength, high-conductivity Al-Mg composite. The highest deformation true strain is 11.28 for Al-13Mg and 11.95 for Al-20Mg.
2. The filamentary microstructure of Al-Mg composite is typical of FCC/HCP systems with a convoluted, ribbon-shape second phase. The Mg phase changes its shape from the initially flake powder particles to elongated filaments. The composite has a plane straining Mg phase in a very ductile Al matrix. The strength of the Al-Mg DMMC increases as the Mg phase spacing and filaments size decrease.
3. The ultimate tensile strength of the Al-13Mg DMMC increases 160% as the deformation processing proceeds from $\eta=2.77$ to $\eta=11.28$. The ultimate tensile strength of the Al-20Mg DMMC increases 220% as the deformation processing proceeds from $\eta=2.77$ to $\eta=11$.
5. The relationship between ultimate tensile strength and deformation ratio is:

$$UTS=92.4 \exp(0.107\eta) \text{ for Al-13Mg.}$$

$$UTS=95.0 \exp(0.123\eta) \text{ for Al-20Mg;}$$

where UTS= Ultimate tensile strength (MPa) and η is deformation ratio.

4. The Hall-Petch deformation processing model and Embury modified Hall-Petch model can be used to predict the strengthening of the Al-Mg composite over the range $0 \leq \eta \leq 12$.
5. The electrical resistivity of Al-13Mg is only slightly higher than that of pure Al. This is because that 80% of the cross sectional area of the DMMCs is high purity Al which forms a parallel conduction path with the Mg filaments.
6. An exposure of the Al-13Mg DMMC to high temperature would react the two pure metals to form intermetallic compound in the Al matrix. The Al-13Mg retains its tensile strength even after being annealed at high temperature. While most precipitation-hardening Al alloys will lose their strength substantially because of the reversion to equilibrium solid solution at high temperature.

7. It remains for future work to study the influence of higher deformation ratio and different composition on the properties of the composites.

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