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SUBJECT: Authorization for Release of Technical Information, Control Number: **AFRL-PR-ED-TP-2002-023**
Drs. Oleg Senkov & Daniel Miracle (AFRL/MLLMD), *et al.*, "Low Temperature Mechanical Properties of Scandium-Modified Al-Zn-Mg-Cu Alloys"

International Conference on Aluminum Alloys (ICAA-8) (Cambridge, UK, July 2002) **Deadline: 15 FEB 2002)** **(Statement A)**

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Low Temperature Mechanical Properties of Scandium-Modified Al-Zn-Mg-Cu Alloys

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__Keywords__: Al-Zn-Mg-Cu alloys, precipitation strengthening, tensile properties, microstructure, cryogenic temperatures, effect of scandium alloying.

Abstract. Tensile properties of three wrought alloys, (1) Al-10Zn-3Mg-1.2Cu-0.15Zr, (2) Al-10Zn-3Mg-1.2Cu-0.15Zr-0.39Mn-0.49Sc, and (3) Al-12Zn-3Mg-1.2Cu-0.15Zr-0.39Mn-0.49Sc were studied in T6 and T7 conditions at 298K and 77K. The properties depended on the alloy compositions and heat treatment conditions. An increase in the concentration of Zn increased strength and decreased ductility. An addition of Sc increased both strength and ductility. The alloys showed very high strength both at room and cryogenic temperatures. In the T6 condition, the following properties were obtained at room temperature: YS = 760 to 805 MPa, UTS = 770 to 810 MPa, El = 3 to 9%. When the temperature was decreased to 77K, strength increased while elongation decreased, and their values were YS = 1005 to 1065 MPa, UTS = 1010 to 1065 MPa, El = 0.3 to 0.7%. Over-aging allowed an increase in elongation to a value as high as 8.5% at the values of YS = 805 MPa and UTS = 835 MPa at the cryogenic temperature. The deformed alloys showed mixed type of fracture. The fraction of the brittle component increased when the temperature decreased. Although they showed high elongation, the alloys in T7 condition fractured predominantly by intergranular mode at the cryogenic temperature.

Introduction

Improved performance of aerospace and ground vehicles requires advanced metallic materials with enhanced mechanical property combinations at reduced overall weight [1,2]. The latter can most efficiently be realized by the use of low-density alloys such as aluminum and titanium. If only high specific strength and ductility were important, titanium alloys would probably be ideal materials for many applications in a wide temperature range [3,4]. Unfortunately, there are at least two negative features associated with titanium alloys, which motivate their replacement by new materials. The first limitation is the high cost of titanium alloys associated with the cost of raw material, processing, machining and maintenance. Another shortcoming is the high affinity of titanium for hydrogen and associated hydrogen embrittlement [5,6]. Aluminum alloys are of particular interest to replace titanium alloys, especially in ambient and low temperature applications, because they are much less expensive, their density is about 1.5-1.7 times lower than the density of titanium alloys, and they have very low susceptibility to hydrogen embrittlement [5]. However, for an Al alloy to achieve specific strength equivalent to that for a Ti alloy at room (298K) and cryogenic (77K) temperatures, tensile strengths of 660 MPa and 850 MPa, respectively, are required. Unfortunately, none of the currently available commercial aluminum alloys can provide such high strength [4,7].
The highest strengths attained in aluminum alloy products at room and cryogenic temperatures are developed by Al-Zn-Mg-Cu alloys of 7XXX series. These are 700-750 MPa at 20K and 570-630

MPa at 298K [4,7]. Further increase in strength can be achieved by an increased concentration of Zn leading to a higher volume fraction of Zn-rich precipitates. However, processing becomes difficult and many fracture-related properties degrade because large internal stresses are generated during casting and heat treatment [7].

It has recently been shown that an addition of Sc may improve both strength and ductility of Al alloys [8-11]. Scandium is known to be a precipitation-strengthener, grain refiner, and recrystallization inhibitor for Al alloys. The hardening effect from the addition of scandium (~ 97 MPa per 0.1 at.%Sc) is much higher than from any other alloying elements [11]. A small addition of 0.1% Sc can increase the recrystallization temperature of aluminum alloys to above 823K [9-11], which is well above the solution heat treatment temperatures of most heat-treatable aluminum alloys. Combined alloying with Sc and Zr is even more effective. Tensile strength of 780 MPa has been achieved in a 7XXX series alloy additionally alloyed with 0.1%Zr and 0.15%Sc [11]. An increase in stress corrosion resistance in Al-Zn-Mg alloys by an addition of Sc has been reported [9]. Cryogenic properties of these alloys have not been studied yet; however, improved properties are expected.

In the present work, three Al-Zn-Mg-Cu-Zr based alloys with a concentration of Zn as high as 10 and 12 % were produced by casting. Two of these alloys were additionally alloyed with Sc. The alloys were thermomechanically processed to homogenize and refine microstructure and heat treated to achieve maximum strength. Tensile properties of these alloys in T6 and T7 conditions were studied at room and cryogenic temperatures. Microstructure of the alloys after casting, thermomechanical treatment, heat treatment and deformation was also studied. Outstanding mechanical properties were reported.

Experimental Procedures

Three alloys, the chemical compositions of which are shown in Table 1, were produced in the Institute for Problems of Materials Science, National Academy of Science of Ukraine, Kiev, Ukraine. The alloys were melted in a graphite crucible at a temperature of about 1073K using an induction furnace and then cast in a water-cooled copper mold of 55 mm diameter and 130 mm height with the cooling rate of ~50 K/s. The cast ingots were machined to a diameter of 54 mm and extruded at 673K to a diameter of 25 mm and further to a diameter of 6 mm, at a total extrusion ratio of 81.

Table 1. Composition of alloys produced by IPMS.

Alloy #	Element [wt.%]					
	Zn	Mg	Cu	Mn	Zr	Sc
1	10.3	2.85	1.2	-	0.15	-
2	10.3	2.7	1.3	0.39	0.15	0.49
3	12.0	3.3	1.2	0.38	0.13	0.49

The extruded rods were cut into blanks of about 45 mm long and heat treated to a peak-aged condition (T6 temper) or over-aged conditions (T7). Tensile specimens with a gauge diameter of 3 mm and gauge length of 15 mm were machined from the heat-treated blanks. Tensile tests were conducted at 293K and 77K and a constant ram speed of 0.015 mm/s using a servo-hydraulic MTS machine. An optical microscope Neophot 23, scanning electron microscope LEICA 360FE, and transmission electron microscope Phillips CM200 were used for microstructural analysis.

Results and discussion

Microstructure of as-cast alloys. In as-cast condition, the alloy #1 (without Sc) had a non-homogeneous, columnar structure, with grains of several millimeters in size growing from the surface of the billet to the center. The alloys #2 and #3, which contained scandium, had a fine and homogeneous microstructure throughout the cross-section, with a grain size of about 15-25 μm. Dendrites were present in all three alloys; however, in the alloy without Sc, they were coarse and produced a columnar structure. With addition of Sc, the dendritic structure tended to be finer and more homogeneous, the columnar structure disappeared, and primary Al_3Sc particles were detected inside some grains.

Microstructure of the alloys after extrusion and heat treatment. Deformation by extrusion at a temperature of 673K, with an extrusion ratio of 81:1, homogenized the alloys by breaking up dendrites and extensive plastic flow of material. After extrusion, a homogeneous equiaxed microstructure was observed in the transverse cross-section, and very elongated thin grains (from 2 to 10 μm thick) were seen in the longitudinal cross-section. After solution treatment at 733K, the refined deformed (fiber-like) microstructure was retained in the Sc-containing alloys # 2 and #3, while large recrystallized elongated grains were detected in the Sc-free alloy #1, Figure 1. TEM analysis showed a sub-grain structure, with the sub-grain size of about 10-20 μm in the alloy # 1 and 1-5 μm in the alloys #2 and #3 after heat treatment. A very high number density of spherical particles of η' phase, from 2 to 15 nm in size, was detected in the heat-treated specimens. In addition, secondary $Al_3(Sc,Zr)$ particles were present in the alloys #2 and #3; their size was from 5 to 30 nm.

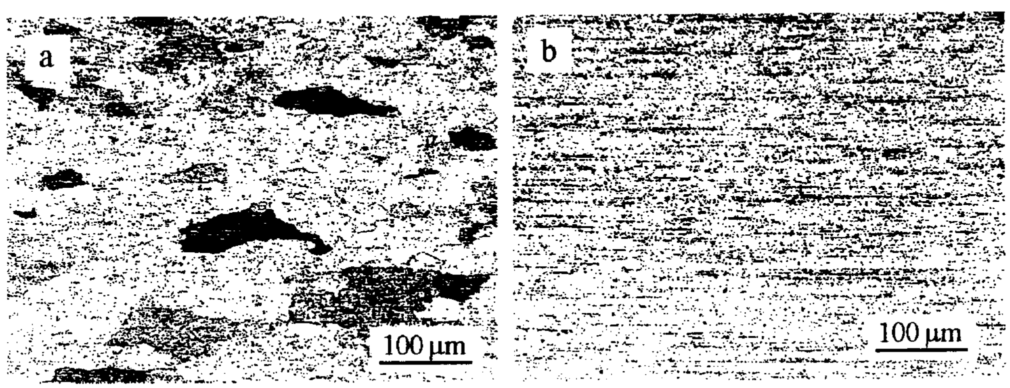

Figure 1. Microstructures of longitudinal cross-sections of the alloys (a) #1 and (b) #2 after extrusion and heat treatment.

Mechanical properties. Room temperature tensile curves are given in Figure 2a. In T6 condition, all three alloys showed very high strength, above 700 MPa. Weak strain hardening was observed in the alloys containing 10%Zn, and strain softening occurred in the alloy #3, containing 12%Zn, after yielding. An addition of Sc and an increased concentration of Zn increased strength. Plastic elongation (El) also increased from 6.5% to 7.5% when 0.49% Sc was added; however, it decreased to 2.5% when the concentration of Zn increased from 10 to 12 %. Overaging of the alloy #3 by holding at 200°C for a short time of 30 min (T7 condition) led to a decrease in UTS to 575 MPa and an increase in elongation to 8%, Figure 2a.

Figure 2b shows strength/strain curves of the alloys at a cryogenic temperature (T=77K). In the peak aged condition, the alloy #1 showed YS=995 MPa, UTS=1008 MPa, and El=0.2%. An addition of Sc in the alloy #2 led to an increase in strength to 1017 MPa and elongation to 0.7%. The alloy #3, with 12% Zn, showed a very high strength of 1068 MPA, however, elongation was only 0.6%. Overaging of the alloy #3 by holding at 473K for a short time of 30 min (T7 condition) led to an increase in ductility to about 8%; however, strength decreased to 835 MPa. Strain softening was observed in alloys # 2 and #3 in T6 condition, and a very weak strain hardening was observed in the alloy #3 in the overaged condition.

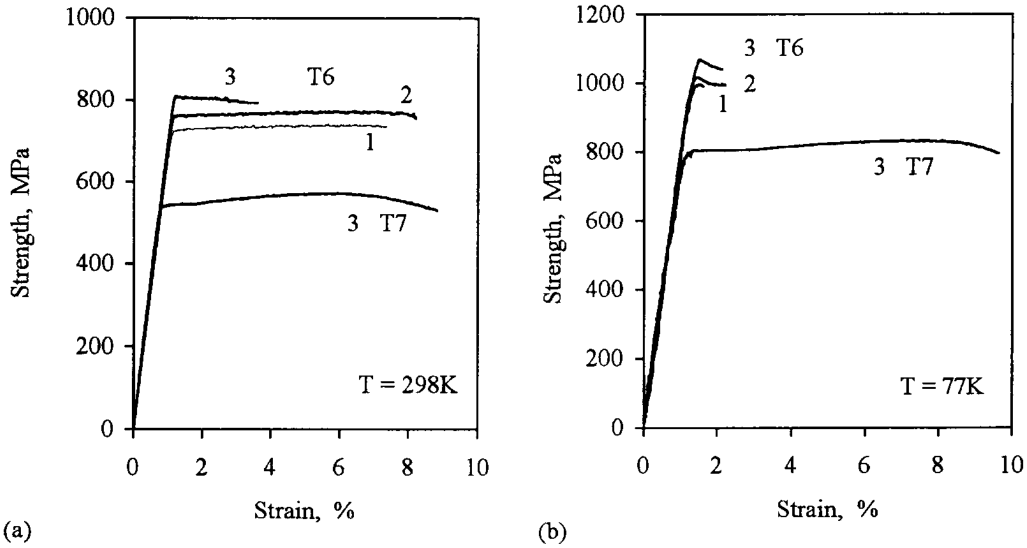

Figure 2. Tensile strength/strain curves obtained at (a) room temperature and (b) cryogenic temperature for the alloys #1 to #3 in the peak aged (T6) and over-aged (T7) conditions.

Figure 3 shows the effect of over-aging time at 423K on strength and elongation of the alloy #3 at 77K. The alloy was initially heat treated to a maximum strength. An increase in the over-aging time led to a continuous increase in elongation and a decrease in strength. The decrease in strength occurs more rapidly at shorter over-aging times while a more rapid increase in elongation takes place after longer over-aging. After 24-hour over-aging at this temperature the alloy showed properties which are typical of the properties of 7XXX series alloys: UTS=655 MPa and El=10%.

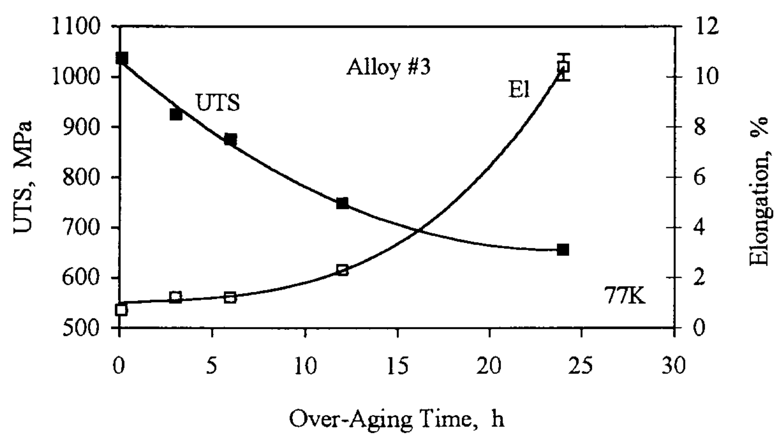

Figure 3. Effect of the over-aging time at 423K on cryogenic properties of alloy #3 that was initially heat treated to a maximum hardness.

Fractography analysis

Room temperature deformation. At room temperature, the specimens fractured by ductile, cup-and-cone, mode of fracture. Photomicrographs of fracture surfaces of the alloy specimens #3 are shown in Figures 4. Very developed dimples on the fracture surface as an evidence of ductile fracture can be seen. A detailed analysis of the fracture surfaces also showed many secondary cracks and evidence of the presence of a brittle mode of fracture by cleavage. Primary $Al_3(Sc,Zr)$ particles of about 5 μm in size were also detected, Figure 4b.

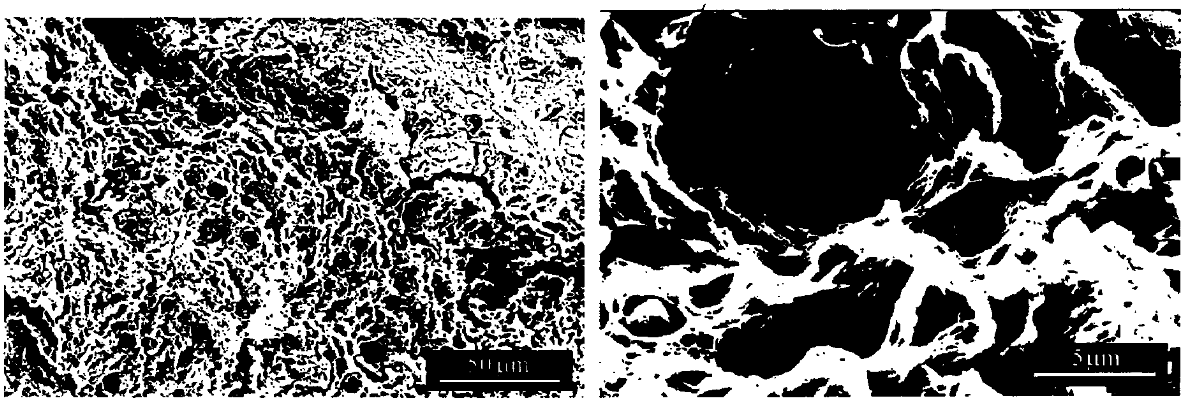

Figure 4. SEM photomicrographs of fracture surfaces of a specimen of alloy #3 after deformation at room temperature.

Cryogenic temperaure deformation. In spite of a very low elongation in T6 condition, the specimens showed very developed dimples on the fracture surface, indicating the presence of ductile mode of fracture. The fracture surfaces also showed a transgranular mode of fracture by cleavage in specimens of the alloy #1 and very fine sub-grains, about 1-3 μm, and the presence of intergranular fracture along the sub-grain boundaries in specimens of the alloy #2 and #3, Figures 5a and 5b. In the alloy #3 additionally over-aged at 200°C for 30 min, intergranular mode of fracture dominated, Figures 5c and 5d.

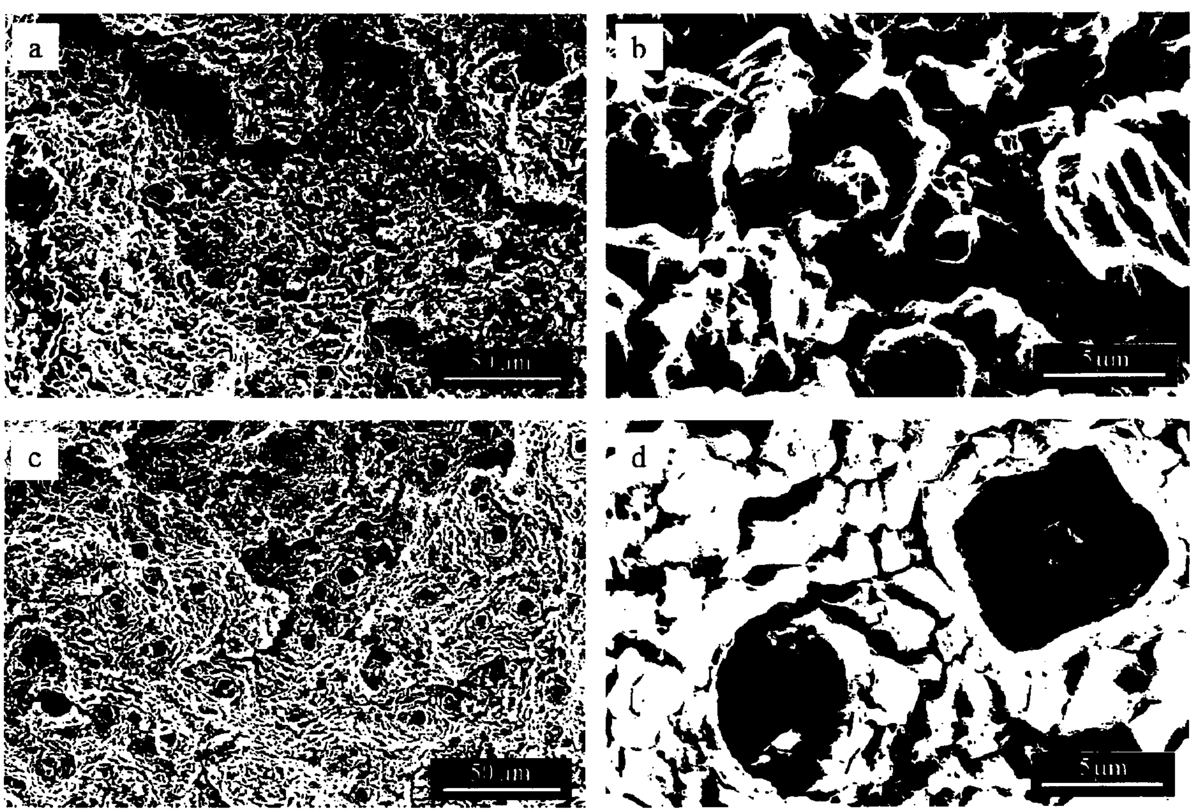

Figure 5. SEM photomicrographs of fracture surfaces of specimens of the alloy #3 in (a,b) T6 and (c,d) T7 conditions after deformation at 77K.

Conclusions

1. Three Al-Zn-Mg-Cu alloys, with high zinc concentration of 10 or 12%, about 3% Mg and ~1.2%Cu, were produced by casting. The alloy # 1 additionally contained 0.15%Zr, and the alloys #2 and #3 contained 0.38%Mn, 0.15%Zr and 0.49%Sc. After casting, the alloy #1 had a non-homogeneous, columnar structure, with grains of several millimeters in size growing from the surface of the billet to the center. An addition of Mn and Sc to the alloys #2 and #3 led to formation of a finer and more homogeneous microstructure, with a grain size of about 15-25 μm. Primary $Al_3(Sc,Zr)$ particles of about 5 μm in size were present in Sc-containing alloys.
2. Hot extrusion followed by solution treatment led to formation of large recrystallized grains, elongated in the direction of extrusion, in the alloy without Sc and a non-recrystallized, fiber-like, deformed fine microstructure in the alloys with Sc. Very fine η' particles precipitated in the alloys during aging. In addition, fine secondary $Al_3(Sc,Zr)$ particles were formed in Sc-containing alloys.
3. The alloys showed a very high strength in a peak-aging condition (T6). Yield strength and UTS as high as 800MPa at 298K and 1050 MPa at 77K were obtained. Elongation was about 5-8% at 298K, however it was very low (~0.5%) at 77K. An increase in the amount of Zn increased strength but reduced elongation. An addition of Sc led to an increase in both strength and elongation. Over-aging improved ductility of the alloys, however, at reduced levels of strength. An outstanding combination of cryogenic strength and ductility (UTS = 830 MPa and El = 8.5%) was achieved after heat treatment of a Sc-containing alloy to a peak-aging condition followed by an over-aging at 473K for 30 min.
4. The alloys showed mixed type of fracture, both at room and cryogenic temperatures. The ductile dimples prevailed in specimens in T6 condition. After over-aging, intergranular fracture along subgrain boundaries dominated in the Sc-containing specimens, in spite of an increased elongation.

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