

Effect of Sc and Heat Treatment on Microstructure and Properties of a 7XXX DC Cast Alloy

O.N. Senkov, R.B. Bhat, S.V. Senkova, and J. Tatalovich
UES, Inc., 4401 Dayton-Xenia Rd., Dayton, Ohio 45432-1894, USA

Keywords: Al-Zn-Mg-Cu alloy; casting; effect of Sc; microstructure; heat treatment; tensile properties

Abstract

Four developmental 7XXX series alloys modified with Sc, Zr and other dispersoid-forming elements were produced by continuous direct chill (DC) casting. Microstructure and mechanical properties were evaluated in both the longitudinal and transverse directions at room and cryogenic temperatures. The homogenization, solution treatment and aging schedules were optimized to obtain superior mechanical properties. In particular, the Sc-containing cast alloys showed tensile properties similar or even superior to the properties of a wrought 7075-T6 alloy. The strengthening mechanisms responsible for this improvement, optimal Sc content and the processing parameters are discussed.

1. Introduction

Scandium additions to advanced aluminum alloys have been studied extensively and synergistic advantages are known to accrue when scandium is added together with zirconium [1-4]. Starting from refining the grain size of the cast Al alloys, these additions also increase the resistance to recrystallization during hot working and introduce additional strengthening by fine dispersoid formation [5]. Uniform distribution of fine dispersoids promotes formation of a stable refined subgrain structure during deformation processing, which adds an additional increase in strength [6]. Accordingly, the maximum benefit of these additions is obtained by producing a supersaturated solid solution of these elements in aluminum which is then decomposed to produce a high number density of fine $Al_3(Sc,Zr)$ dispersoids during controlled heat treatment [7]. In the present paper, the microstructure and tensile properties of four developmental 7XXX alloys containing different amounts of Sc are reported. The room and cryogenic temperature properties were determined in as-cast and cast-and-heat-treated conditions, and the effect of Sc on the properties was identified.

2. Experimental

Four Sc-modified 7XXX alloys having the composition shown in Table 1 were produced by continuous direct chill (DC) casting at Wagstaff, Inc., Spokane, WA. The cast billets were 76 mm in diameter and had the same nominal composition, but a different Sc concentration of 0%, 0.18%, 0.38% and 0.48% Sc. Tensile specimens were machined from the cast billets at both longitudinal and transverse orientations, about one inch away from the center of the billet. The longitudinal direction is parallel to the cast billet axis, and the transverse direction is perpendicular to the radial direction in the transverse cross-section of the billet. The specimens'

Report Documentation Page

Form Approved
OMB No. 0704-0188

Public reporting burden for the collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden, to Washington Headquarters Services, Directorate for Information Operations and Reports, 1215 Jefferson Davis Highway, Suite 1204, Arlington VA 22202-4302. Respondents should be aware that notwithstanding any other provision of law, no person shall be subject to a penalty for failing to comply with a collection of information if it does not display a currently valid OMB control number.

1. REPORT DATE 01 MAR 2004		2. REPORT TYPE		3. DATES COVERED -	
4. TITLE AND SUBTITLE Effect of Sc and Heat Treatment on Microstructure and Properties of a 7XXX DC Cast Alloy				5a. CONTRACT NUMBER F04611-02-C-0014	
				5b. GRANT NUMBER	
				5c. PROGRAM ELEMENT NUMBER	
6. AUTHOR(S) O Senkov; R Bhat; S Senkova; J tatalovich				5d. PROJECT NUMBER 3005	
				5e. TASK NUMBER 02AF	
				5f. WORK UNIT NUMBER 300502AF	
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) UES Inc,4401 Dayton-Xenia Road,Dayton,OH,45432-1894				8. PERFORMING ORGANIZATION REPORT NUMBER	
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)				10. SPONSOR/MONITOR'S ACRONYM(S)	
				11. SPONSOR/MONITOR'S REPORT NUMBER(S)	
12. DISTRIBUTION/AVAILABILITY STATEMENT Approved for public release; distribution unlimited					
13. SUPPLEMENTARY NOTES					
14. ABSTRACT Four developmental 7XXX series alloys modified with Sc, Zr and other dispersoid-forming elements were produced by continuous direct chill (DC) casting. Microstructure and mechanical properties were evaluated in both the longitudinal and transverse directions at room and cryogenic temperatures. The homogenization, solution treatment and aging schedules were optimized to obtain superior mechanical properties. In particular, the Sccontaining cast alloys showed tensile properties similar or even superior to the properties of a wrought 7075-T6 alloy. The strengthening mechanisms responsible for this improvement, optimal Sc content and the processing parameters are discussed.					
15. SUBJECT TERMS					
16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT	18. NUMBER OF PAGES 6	19a. NAME OF RESPONSIBLE PERSON
a. REPORT unclassified	b. ABSTRACT unclassified	c. THIS PAGE unclassified			

gauge had a rectangular cross section of 3.6 x 2.5 mm and a length of 20 mm. The microstructure and tensile properties were studied in as-cast and heat-treated conditions. The tensile tests were conducted at room temperature (RT, 25°C) and a cryogenic temperature (CT, -196°C) using a servo-hydraulic MTS testing machine and a constant ram speed of 0.02 mm/s (initial strain rate is 10^{-4} s^{-1}). Three types of heat treatment were used in this study. One consisted of homogenization annealing at 460°C for 48 hours, water quenching and T6 temper (120°C for 19 hours); this heat treatment is further referred as H+T6. Another set included heating at 20°C/h or 60°C/h from room temperature to 460°C or 480°C, holding 20 or 48 hours at these temperatures, water quenching and T6 temper. The third set of heat treatment involved isothermal annealing at 270°C, 350°C or 460°C for different period of time, homogenization at 480°C, water quenching, and T6 tempering. While the H+T6 treatment was studied in all the alloys, only the alloy SSA018 containing 0.18% Sc was subjected to the latter heat treatments. Optical and scanning electron microscopes were used for microstructural analysis.

Table 1. Composition (wt.%) of alloys used in this work.

Alloy ID	Zn	Mg	Cu	Zr	Sc	Others	Al
SSA000	7.14	2.30	1.61	0.17	0	0.8	Bal.
SSA018	7.17	2.20	1.58	0.18	0.18	0.8	Bal.
SSA038	7.11	2.14	1.56	0.17	0.38	0.8	Bal.
SSA048	7.05	2.35	1.55	0.14	0.49	0.8	Bal.

3. Results and Discussion

As-Cast Condition. Macro-etching of transverse cross-sections of the DC cast billets revealed very large columnar grains and a crack in the SSA000 alloy (Figure 1, 0% Sc). These grains were several millimeters in size, elongated from the surface to the center of the billet. In the other three alloys, which additionally contained Sc, the macro-etching revealed a very homogeneous equiaxed microstructure, and no cracks or macro-porosity were detected (Figure 1).

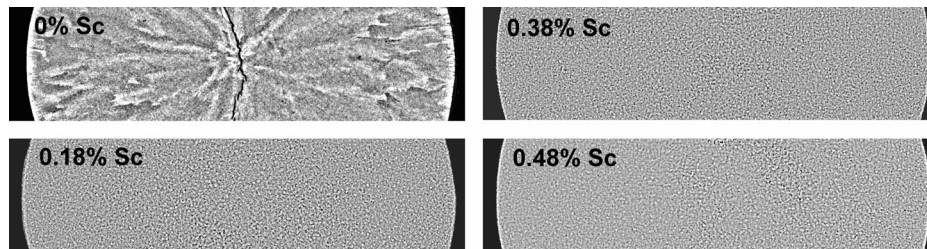


Figure 1: Photographs of etched transverse cross-sections of 76-mm diameter DC cast billets.

On the microscopic level, all the alloys had a dendritic microstructure of grains with the eutectic phases between the dendritic arms. Figure 2 shows the SEM micrographs of the cast alloys in the back-scattered electron (BSE) mode, where the dendrite arms have a gray contrast and the eutectic has a bright contrast. Faceted bright particles visible in SSA038 and SSA048 are the primary $\text{Al}_3(\text{Sc,Zr})$ particles. The grain size decreased when the concentration of Sc increased. For example, the grain size in the SSA018, SSA038, and SSA048 alloys was determined to be $\sim 150 \mu\text{m}$, $100 \mu\text{m}$ and $70 \mu\text{m}$, respectively. An additional feature typical only to the SSA038 and SSA048 alloys, is the presence of primary $\text{Al}_3(\text{Sc,Zr})$ particles inside almost every dendrite colony (figures 2c & 2d), which indicates that these particles controlled crystallization in these alloys. On the other hand, no primary $\text{Al}_3(\text{Sc,Zr})$ particles were detected in the SSA018 alloy, which had a low concentration of Sc (0.18%) and also exhibited considerable grain refinement,

relative to the Sc-free SSA000 alloy. The mechanism of the grain refinement for this alloy has not yet been clearly understood.

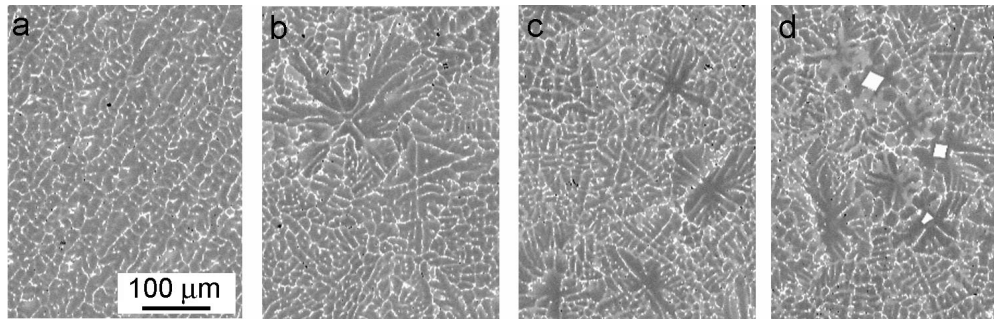


Figure 2: SEM micrographs of the developmental cast alloys (a) SSA000 (b) SSA018 (c) SSA038 and (d) SSA048.

Tensile properties of the as-cast alloys after about one year holding at room temperature are given in Table 2. The alloy SSA000, without Sc, showed very anisotropic properties. This alloy had almost zero transverse ductility, although ductility in the longitudinal direction was very good, about 15% at RT and 9% at CT. It is evident that the coarse columnar grain structure of this alloy is responsible for this anisotropic behavior. An addition of Sc improved isotropy of the cast alloys remarkably, mainly due to an increase in the transverse ductility. Ductility in the longitudinal direction however decreased, as compared to the SSA000 alloy. The increase in the Sc concentration also led to an increase in the yield strength (YS) and ultimate tensile strength (UTS). For example, the alloy SSA048 showed YS=317-324 MPa, UTS=425-433 MPa and EI=3-4% at room temperature. None of the currently available commercial cast Al alloys has such superior combination of the tensile properties [8].

Table 2. Room and cryogenic tensile properties of four as-cast developmental alloys in longitudinal/transverse directions.

Temperature	25°C			-196°C		
	YS (MPa)	UTS (MPa)	EI (%)	YS (MPa)	UTS (MPa)	EI (%)
SSA000	257/318	378/318	15/0.01	357/377	439/377	9/0.01
SSA018	292/284	402/379	5/3	395/368	462/415	2/1
SSA038	313/305	445/409	8/3	406/411	495/481	4/2
SSA048	324/317	433/425	4/3	422/421	504/478	2/1

Cast and Heat Treated Condition. Homogenization of the cast billets is normally carried out to mitigate the effects of micro- and macro-segregation, by allowing time at high temperatures to dissolve the Zn-, Mg-, and Cu-rich intermetallic phases and to achieve a uniform composition throughout the ingot. In the Sc-containing alloys, however, homogenization may also result in the precipitation and subsequent growth of secondary $Al_3(Sc,Zr)$ dispersoids from the supersaturated solid solution of Sc and Zr [1]; the latter is formed during rapid DC casting. Strengthening of the cast alloys from the Sc addition is a combined effect of different mechanisms, such as solid solution and dispersoid strengthening, as well as the grain size effect (Hall-Petch strengthening). The presence of Sc in these alloys may also affect the kinetics of precipitation of $MgZn_2$ particles during aging. As far as the $Al_3(Sc,Zr)$ particles precipitate, the concentrations of Sc and Zr in the solid solution decrease and the effect from the solid-solution strengthening decreases. There should be therefore an optimum combination of solid solution and dispersoid strengthening, which provides maximum increase in strength. The dispersoid strengthening is a function of the size, number density and distribution of secondary $Al_3(Sc,Zr)$ particles [9], and the heat treatment schedule should have an important influence on these

parameters. A model for the Al_3Zr dispersoid precipitation in commercial aluminum alloys has recently been developed and compared with experimental results [10,11]. It was found that in addition to the temperature and time of homogenization, the rate of heating to the homogenization temperature is also important in reducing the dispersoid free zones normally seen adjacent to the grain boundaries in these alloys. The slow rate of heating is mainly important to achieve a high number density of dispersoids and these are expected to form at relatively lower temperatures as compared to the homogenization temperatures. In the current work, three different heat treatment schedules described in the experimental procedures were used to improve tensile properties of the cast alloys.

The H+T6 heat treatment consisted of homogenization by holding at 460°C for 48 hours with subsequent water quenching and artificial aging to a T6 temper. This heat treatment led to almost complete dissolution of the non-equilibrium eutectic and Mg and Zn rich phases and re-precipitation of $MgZn_2$ particles. The tensile properties of the alloys after H+T6 treatment are given in Table 3. The H+T6 treatment of the alloy SSA000 increased the longitudinal strength and did not improve the transverse properties, so that the anisotropy of the alloy increased. The heat-treated alloy was brittle in the transverse direction and, consequently, no improvements in the transverse strengths were detected. On the other hand, all Sc-containing alloys benefited from the heat treatment, and both strengths and ductility values increased. In fact, the tensile properties of the Sc-containing cast alloys after H+T6 treatment were similar to the properties of hot-worked 7X75-T6 [8].

The increase in strengths of the heat-treated alloys is mainly due to precipitation of $MgZn_2$ particles during aging. This conclusion is made based on the fact that the strength increase almost does not depend on the amount of Sc. For example, YS increased by ~180 MPa and UTS increased by ~150 MPa for all four alloys at room temperature in the longitudinal direction. On the other hand, the increase in plastic strain is totally related to the presence of Sc; i.e., formation of fine $Al_3(Sc,Zr)$ dispersoids from the supersaturated solid solution. An additional study is required to understand the mechanism leading to a remarkable increase in ductility in Sc-containing alloys after the heat treatment.

The results presented in Tables 2 and 3 also show that an increase in the Sc content beyond 0.38% has almost no advantage on tensile properties and even leads to a decrease in ductility. This can be explained by the presence of brittle coarse $Al_3(Sc,Zr)$ particles in the alloys with high Sc content. These particles add not much to strength; however, their presence can limit the volume fraction of fine secondary $Al_3(Sc,Zr)$ dispersoids responsible for additional strengthening of these alloys and initiate fracture.

Table 3. RT and CT tensile properties of cast and H+T6 heat-treated developmental alloys in longitudinal/transverse directions.

Temperature	25°C			-196°C		
	YS (MPa)	UTS (MPa)	EI (%)	YS (MPa)	UTS (MPa)	EI (%)
SSA000	439/304	520/304	15/0.01	528/384	607/384	9/0
SSA018	457/452	552/559	14/11	584/570	674/679	10/7
SSA038	494/497	593/595	15/13	610/603	719/700	10/6
SSA048	505/508	600/600	12/9	613/624	717/709	8/7

The effect of the heating rate to a homogenizing temperature, as well as the homogenizing time and temperature, on tensile properties was studied using the alloy SSA018 (0.18%Sc). This set of heat treatments was conducted in order to obtain a better contribution to strengthening from the $Al_3(Sc,Zr)$ precipitates. The results are given in Table 4. Slow heating to a homogenizing temperature indeed increases strength of the Sc-containing alloy. For example, the alloy homogenized at 460°C for 48 hours had about 30-40 MPa strength increase when it was slow heated ($V=60^\circ C/h$) to this temperature instead of direct placing in the pre-heated furnace (no ramp). A decrease in the heating rate from 60°C/h to 20°C/h led to an additional

slight increase in strength. These additions to the strengths are definitely due to different precipitation kinetics of the $Al_3(Sc,Zr)$ particles from the super-saturated solid solution at different heating rates, so that a slower heating rate provides more homogeneous distribution and finer size of these particles [10,11]. The results collected in Table 4 also show that the properties of the heat-treated alloy additionally depend on the annealing temperature and time. An increase in the homogenizing time from 20 h to 48 h at 460°C led to an increase in the tensile strength, while at 480°C this led to a strength decrease. Such behavior resulted from operation of two concurrent processes during the homogenizing annealing; i.e., dissolution of Zn-, Mg-, and Cu-rich phases and coarsening of the $Al_3(Sc,Zr)$ particles. The first process should lead to higher concentrations of these elements in the solid solution and, correspondingly, to higher strengthening effect after aging, while the second process should lead to a decrease in the strengthening effect from the $Al_3(Sc,Zr)$ particles. At a lower annealing temperature of 460°C the first process dominates, while at a higher temperature (480°C) dissolution of the Zn, Mg, and Cu-rich phases requires a shorter period of time, and coarsening of the Sc-rich particles leads to a decrease in the tensile strength. The heating rate and the annealing temperature and holding time almost do not have an effect on the ductility of the Sc-containing alloy.

Table 4. Room temperature tensile properties of the cast alloy SSA018 after heat treatment that includes heating at a constant rate V to a temperature T holding at this temperature for a period of time t water quenching and aging to T6 temper.

T (°C)	460			480			
V (°C/h)	60		No ramp	60		20	
t (hrs)	20	48	48	20	48	20	48
Properties							
YS (MPa)	480	500	457	508	495	513	498
UTS (MPa)	554	582	552	590	586	600	589
EI (%)	14	12	14	11	15	12	12

Although it is beneficial for strengthening of the Sc-containing cast alloys, the homogenization process involving slow heating is a time consuming and economically unwanted process. Therefore, it is worth looking for other (faster) ways to accomplish the rational precipitation of the $Al_3(Sc,Zr)$ particles during or prior to the homogenization. The kinetics of precipitation of these particles, as mentioned above, are very critical for the final properties, because once these particles precipitate, they cannot be dissolved and they can only coarsen during holding at high temperatures. It was found in this work that a **multi-step heat treatment** (generally two or three steps) can provide the properties of the Sc-containing cast alloy, which are similar or higher than the properties after the heat treatment with slow heating (Table 5). The multi-step heat treatment consists of pre-annealing the alloy in the temperature range of 250-400°C and subsequent homogenization. The pre-annealing is aimed at precipitating the fine $Al_3(Sc,Zr)$ particles from the supersaturated solid solution of as-cast material. The tensile properties similar to the properties of the best wrought commercial 7XXX-T6 alloys were achieved in the developmental SSA018 cast alloy after this heat treatment (see Table 5).

Table 5. Room temperature tensile properties of the cast alloy SSA018 after isothermal multi-step heat treatment.

Heat treatment	YS (MPa)	UTS (MPa)	EI (%)
270°C/23h+460°C/1h+480°C/1h+WQ+T6 aging	535	601	6
270°C/72h+460°C/1h+480°C/1h+WQ+T6 aging	556	602	10
270°C/72h+460°C/1h+480°C/4h+WQ+T6 aging	504	592	11
350°C/05h+460°C/1h+480°C/1h+WQ+T6 aging	533	599	5
350°C/15h+460°C/1h+480°C/1h+WQ+T6 aging	529	592	7
460°C/1h+480°C/2h+WQ+T6 aging	524	586	8

4. Conclusions

Scandium-containing 7XXX-series Al alloys have been developed with superior tensile properties in as-cast and cast plus heat treated conditions. The presence of Sc in the amounts of 0.18 to 0.48% refines the microstructure, improves tensile properties and makes the tensile properties of the cast alloys much more isotropic, as they are compared to the alloy with the same composition but without Sc. The strengthening of the Sc-containing cast alloys is due to a combination of several mechanisms, such as precipitation strengthening, solid solution strengthening, dispersoid strengthening, and grain boundary (Hall-Petch) strengthening. As Sc, together with Zr, forms fine insoluble dispersoids of the $Al_3(Sc,Zr)$ phase by precipitation from a supersaturated solid solution during the temperature exposure after casting, special heat treatment is required to achieve the maximum strengthening effect from Sc in these alloys. The analysis of different heat treatment processes applied to these alloys shows that the multi-step isothermal treatment consisting of a low temperature annealing in the temperature range of 250-400°C followed by the homogenization annealing, water quenching and aging provides the tensile properties of the developmental cast alloys that are similar to the properties of the best wrought commercial 7XXX-T6 alloys.

Acknowledgements

The authors would like to thank Dr. D.B. Miracle, AFRL, WPAFB and Dr. M.G. Mendiratta, UES Inc., for useful technical discussions. The work was supported through US Air Force SBIR contract No. F04611-02-C-0014 (Dr. J. Shelley, Program Manager).

References

- [1] L.S.Toropova, D.G.Eskin, M.L.Kharakterova and T.V.Dobatkina, *Advanced Aluminum Alloys Containing Scandium*, (Gordon & Breach, Amsterdam, The Netherlands, 1998).
- [2] T.D.Rostova, V.G.Davydov, V.I.Yelagin and V.V.Zakharov, *Materials Science Forum*, 331-337, pp. 793-798, (2000)
- [3] Y.W.Riddle and T.H.Sanders Jr., *Materials Science Forum*, 331-337, pp.799-804, (2000)
- [4] Y.V. Milman, A.I. Sirko, D.V. Lotsko, D.B. Miracle, O.N. Senkov, *Mater. Sci. Forum*, 396-402, pp. 1217-1222, (2002)
- [5] Y.W.Riddle and T.H.Sanders Jr., *Materials Science Forum*, 331-337, pp. 939-944, (2000)
- [6] O.N. Senkov, D.B. Miracle, Y.V. Milman, J.M. Scott, D.V. Lotsko, A. Sirko, *Mater. Sci. Forum*, 396-402, pp. 1127-1132 (2002).
- [7] A.K.Mukhopadhyay, G.M.Reddy, K.S.Prasad, S.V.Kamat, A. Dutta and C.Mondal, *J.T.Staley Honorary Symposium on Aluminum Alloys, Advances in the Metallurgy of Al Alloys*, (ASM International, Materials Park, OH, USA, 2001) pp. 63-71
- [8] J.G. Kaufman, *Properties of Aluminum Alloys: Tensile, Creep, and Fatigue Data at High and Low Temperatures* (ASM International, Materials Park, OH, USA, 1999).
- [9] K.L.Kendig, D.B.Miracle, *Acta Mater.*, 50, pp. 4165-4175, (2002)
- [10] J.D.Robson and P.B.Prangnell, *Acta Mater.*, 49, pp. 599-613, (2001).
- [11] J.D.Robson and P.B.Prangnell, *Mater. Sci. Eng.*, A352, pp. 240-250, (2003).