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Effect of Sc+Zr Addition on Structures and Mechanical Properties of Rapidly Solidified 7090 Based Alloys

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Abstract

Effect of minor addition of Sc+Zr has been studied on the structures and mechanical properties of rapidly solidified Al-9%Zn-2.5%Mg-1%Cu based alloys containing transition metals (Co, Fe, Fe+Ni, Mn). The amount of Sc and Zr addition was 0.2mass% each. Rapid solidification was performed by gas atomizing and splat quenching on a water-cooled Cu single roll under an argon atmosphere. The flakes were consolidated to the P/M materials by cold pressing, vacuum degassing and hot extrusion. Uniform distribution of Al_3(Sc,Zr) particles was observed by TEM in the as-extruded P/M materials with Sc+Zr addition. Hardness became higher for the as-extruded P/M materials with Sc+Zr addition than those without addition even after annealing at higher temperatures. Uniform distribution of Al_3(Sc,Zr) and η’ particles were observed in the T6 treated P/M materials. The size of η’ particle is smaller than Al_3(Sc,Zr) particle. The T6 treated P/M materials with Sc+Zr addition showed higher hardness than the as extruded materials. However, T6 treated alloys with Sc+Zr addition did not always show higher tensile strength and fatigue strength than those without addition due to low ductility.

1. Introduction

The effect of minor addition of Sc on the improvement in grain refinement and mechanical properties of 7000 series aluminum alloys has been reported. Grain refinement [1] and decrease of width of precipitation free zone [2] in 7050 alloy sheets, improvement of stress corrosion cracking in Al-8.6Zn-2.6Mg-2.4Cu alloy [3], increase of tensile strength and suppression of weld cracking in 7010 alloy [4], were confirmed experimentally. In addition, slight improvement in tensile strength of rapidly solidified Al-Zn-Mg system alloys by addition of Sc was reported [5].

Rapid solidification is one of the promising techniques to develop metallic materials of higher mechanical properties. Recent work demonstrated that minor addition of Sc+Zr improved mechanical strength of rapidly solidified powder metallurgy (P/M) materials of Al-transition metal alloys due to precipitation of Al_3(Sc,Zr) [6].

This paper deal with mechanical properties and microstructures of the rapidly solidified flakes and extruded P/M materials of 7090 based alloys with minor Sc+Zr addition. Rapidly solidified of P/M materials of 7090 based alloys without Sc+Zr were used as the reference materials [7].

2. Experimental Procedure

Table 1: Designation and chemical composition of tested alloys.

Designation	Analyzed Composition (mass%)									
	Zn	Mg	Cu	Co	Mn	Fe	Ni	Sc	Zr	Al
7090-SZ	8.9	2.6	1.04	1.46	−	−	−	0.20	0.24	Bal
79F6-SZ	9.3	2.3	0.97	−	−	5.16	−	0.22	0.22	Bal
79FN3-SZ	9.0	2.6	1.02	−	−	2.97	2.97	0.22	0.21	Bal
79M6-SZ	8.9	2.5	0.97	−	5.90	−	−	0.24	0.22	Bal

Designation and chemical composition of P/M materials of the tested alloys are listed in Table 1. Sc and Zr were added to 7090 type alloy containing transition metals such as Co, Fe, Fe+Ni and Mn. Alloy ingots were prepared by using the master alloys of Al-1.89%Sc, Al-5%Zr, Al- 50%Fe and Al-50%Cr and the pure metal of Zn, Mg, Mn and Ni. Rapidly solidified (RS) flakes were produced by remelting the alloy ingots in a graphite crucible, atomizing the alloy melt and subsequent splat quenching of the atomized droplets on a water-cooled copper single roll under argon atmosphere. RS flakes were consolidated in the following procedure : (a) cold pressing under a pressure of 540MPa to about 70% packing density, (b) degassing cold pressed flakes in 1.33×10^{-3} Pa at 623K, (c) hot extrusion at 673K at a reduction ratio of 25:1. Finally, extruded rods of 7mm in diameter were obtained as the P/M materials.

Hardness of isochronally annealed RS flakes and P/M materials were measured using Vickers testing machine. Age hardening curves of P/M materials were obtained at aging temperature of 393K after solutionizing at 733K for 3.6ks and water-quenching and holding at room temperature for 86.4ks.

Tensile test for P/M materials was carried out at room temperature, 473K and 573K at an initial strain rate of $2.0\times10^{-3}s^{-1}$ using specimens machined to 4mm in diameter and 30mm in gage length. Tensile tests at elevated temperature were started after holding a specimen for 300s at testing temperature. Torsion fatigue test were carried out on T6 treated P/M materials at a frequency of 25Hz at room temperature. Microstructures were observed for RS flakes and P/M materials by TEM operating 200kV. The TEM specimens were thinned by twin-jet electro-polishing in a solution of 33%HNO_3-67%CH_3OH at 253K. X-ray diffraction patterns were obtained with CuKα radiation in order to identify constituent phases of RS flakes and P/M materials.

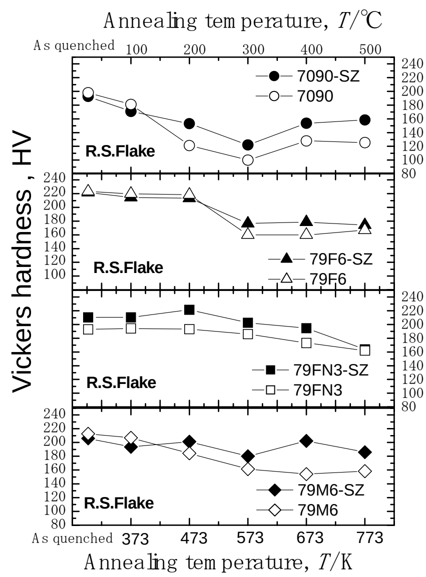

Figure 1: Hardness of RS flakes after annealing at various temperatures for 7.2ks.

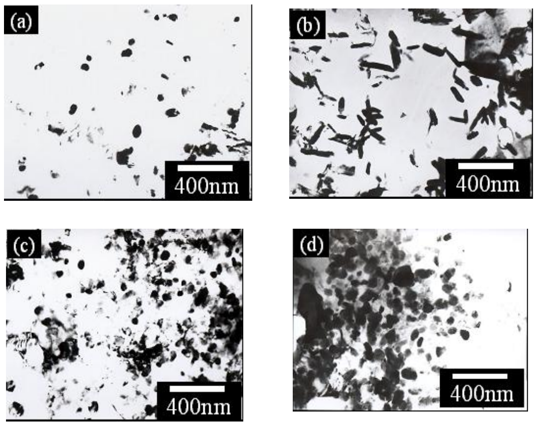

Figure 2: TEM micrographs of as extruded P/M materials. (a) 7090-SZ, (b)79F6-SZ, (c)79FN3-SZ, (d) 79M6-SZ

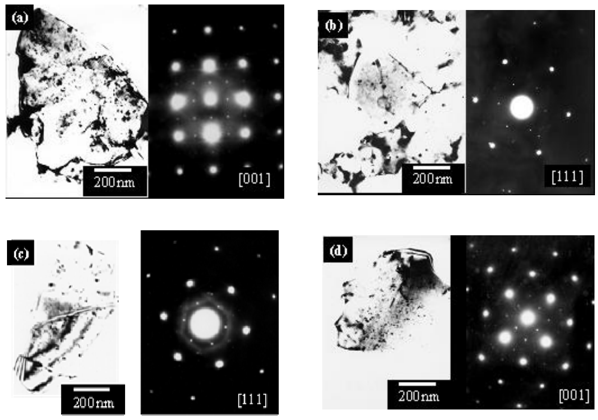

Figure 3: TEM micrographs of as extruded P/M materials. [B.F.image and SADP] (a) 7090-SZ, (b) 79F6-SZ, (c)79FN3-SZ, (d)79M6-SZ

3. Experimental Results and Discussion

3.1 Hardness and Microstructures of RS Flakes and P/M Materials

The changes of hardness of RS flakes on isochronal annealing at various temperatures for 7.2ks are shown in Figure 1. In as-RS flakes, the increase of hardness is small by Sc+Zr addition due to obtained solid solution of Sc and Zr. However, the hardness of the Sc+Zr added alloys tends to rise after heating at above 573K because of precipitation hardening of $Al_3(Sc,Zr)$.

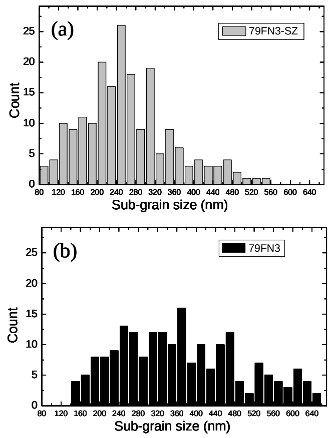

Figure 4: Histograms of sub-grain size of as extruded P/M materials. (a) 79FN3-SZ, (b)79FN3.

TEM micrographs of as-extruded materials of tested alloys are shown in Figure 2. Fine dispersion of transition metal aluminides was observed in the all tested alloys. Volume fraction of dispersoids in 7090-SZ alloy was lower than the other alloys. Microstructures of as-extruded materials observed under higher magnification and corresponding selected area electron diffraction pattern (SADP) are shown in Figure 3. Very fine spherical particles of a diameter less than 10nm were observed in bright field image of each alloy. SADP exhibited $L1_2$ type pattern and Al, Sc and Zr elements were detected by ESD analysis. Thus, those particles can be identified as $Al_3(Sc,Zr)$ of which crystallinity was coherent with the Al matrix.

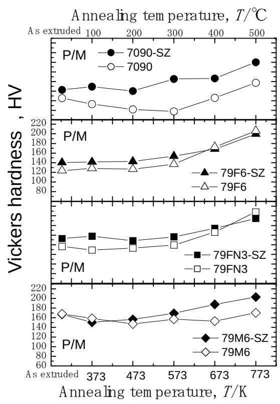

Figure 5: Hardness of as extruded P/M materials after annealing at various temperatures for 7.2ks.

Constituent phases were determined by X-ray diffraction for the RS flakes and as-extruded P/M materials. The RS flakes and P/M materials were composed of essentially the same phases in each alloy. Constituent phases of Al_9Co_2, $Al_{13}Fe_4$, $FeNiAl_9$ and Al_6Mn are equilibrium phases formed in the Al rich side of Al-transition metal alloy diagrams. Formation of the compounds between Mg and Zn were also observed in the P/M materials of each alloy. Diffraction lines from Al_3Sc phase in all Sc+Zr added alloys were not identified because its lattice parameter was similar to the Al matrix.

Figure 4 shows histograms of subgrain sizes obtained in TEM photographs of as-extruded 79FN3 alloys with or without Sc+Zr addition. Average size of subgrains for 79FN3 alloy (376nm) were slightly reduced by minor addition of Sc+Zr (270nm). In the both P/M materials, uniform distribution of transition metal aluminides cause pinning effect of subgrain growth during consolidation process.

The changes of hardness of the as-extruded P/M materials on isochronal annealing at various temperatures for 7.2ks are shown in Figure 5. At the as-extruded stage, hardness of 7090-SZ, 79F6- SZ and 79FN3-SZ alloys is higher than that without Sc+Zr by about 20HV due to dispersion of fine $Al_3(Sc,Zr)$ particles. However, no increase of hardness by Sc+Zr addition was observed on 79M6-SZ alloy. The increase of hardness in 79M6-SZ alloy seemed to be observed by hardening due to Al_6Mn compounds dispersed in high density. Hardness of all tested alloys increased after heating at above 673K. It was thought that precipitation hardening by natural aging in the P/M materials occurred at room temperature after heating at near solutionizing temperatures.

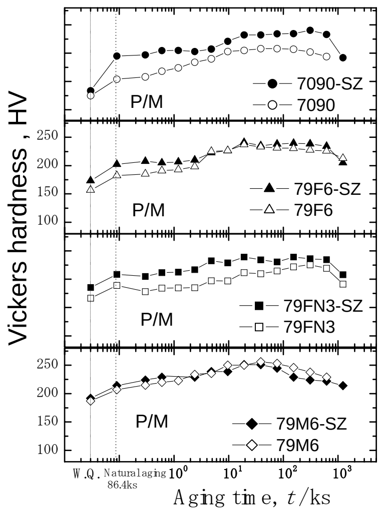

Figure 6: Age hardening curves of as-extruded PM materials.

3.2 Age Hardening of P/M Materials

Figure 6 shows age hardening curves of P/M materials at 393K after solutionizing at 753K. Peak hardness of 7090-SZ and 79FN3-SZ alloys were higher than those without Sc+Zr. However, age hardening curves of 79F6-SZ and 79M6-SZ alloys were similar to those without Sc+Zr addition.

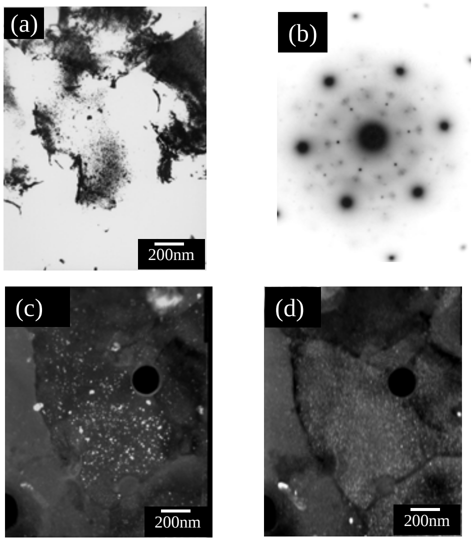

Figure 7: TEM micrographs of T6 treated P/M 79F6-SZ.
(a)B.F. image, (b)SADP, (c)D.F.image of Al3(Sc,Zr), (d)D.F.image of η'

TEM micrographs of T6 treated 79F6-SZ alloy are shown in Figure 7. Diffraction spot from $Al_3(Sc,Zr)$ and η' phase [8] were observed in the SADP (b) obtained on aluminum matrix in the bright field image (a). The similar electron diffraction pattern was obtained in all the alloys with Sc+Zr addition. Uniform distribution of fine $Al_3(Sc,Zr)$ particles of 5-10nm in size was observed in the dark field image (c). The η' phase precipitates are finer than $Al_3(Sc,Zr)$ particles (d).

3.3 Tensile Properties of P/M Materials

Tensile strength, 0.2% proof stress, elongation and reduction of area of as-extruded P/M materials at room and elevated temperatures are listed in Table 2. Tensile strength and proof stress of 7090-SZ alloy were remarkably higher than those without Sc+Zr addition at each testing temperature. The slight improvements of strength with Sc+Zr additions were obtained in 79F6-SZ, 79FN3-SZ and 79M6-SZ alloys. In the alloys with no improvements of strength by the addition of Sc+Zr, dispersion hardening strongly occurred by transition metal aluminides distributed in high density. So, increase of strength of materials with Sc+Zr addition seems to be obscured. Elongation and ductility increased with the rise of testing temperature in all tested alloys.

Table 2: Tensile properties of as-extruded P/M materials.

Alloy	σ_B (MPa)			$\sigma_{0.2}$ (MPa)			δ (%)			ψ (%)		
	Test temp. (K)			Test temp. (K)			Test temp. (K)			Test temp. (K)		
	R.T.	473	573	R.T.	473	573	R.T.	473	573	R.T.	473	573
7090-SZ	465	263	90	377	229	85	6.7	30.3	43.0	5.6	69.5	85.2
7090	395	171	75	280	165	70	19.9	26.4	34.6	19.9	74.5	86.2
79F6-SZ	535	291	122	460	287	111	4.3	17.7	29.1	9.8	54.4	71.0
79F6	522	289	154	405	263	135	3.5	11.9	20.8	8.9	25.7	63.3
79FN3-SZ	536	286	126	405	263	110	1.5	24.9	32.6	6.5	68.8	85.4
79FN3	545	267	113	399	230	93	4.9	23.4	37.3	9.9	68.9	80.1
79M6-SZ	597	307	149	491	271	140	3.5	16.6	20.3	8.6	54.2	73.2
79M6	543	315	143	386	275	140	1.2	21.0	30.3	4.5	63.5	77.6

Table 3: Tensile properties of T6 treated P/M materials.

Alloy	σ_B (MPa)			$\sigma_{0.2}$ (MPa)			δ (%)			ψ (%)		
	Test temp. (K)			Test temp. (K)			Test temp. (K)			Test temp. (K)		
	R.T.	473	573	R.T.	473	573	R.T.	473	573	R.T.	473	573
7090-SZ	735	415	137	731	388	135	1.9	10.6	13.1	5.5	47.6	74.8
7090	664	375	103	600	347	100	11.8	12.3	18.9	24.3	43.8	76.9
79F6-SZ	785	436	143	681	335	126	0.9	7.2	14.9	3.3	29.0	61.8
79F6	676	424	160	665	388	144	0.3	7.5	11.8	4.6	33.0	58.5
79FN3-SZ	705	402	146	690	369	136	1.3	14.3	20.3	4.6	47.2	72.4
79FN3	770	365	112	770	358	104	0.8	14.2	23.9	6.2	54.7	82.9
79M6-SZ	735	442	133	712	405	120	0.5	8.1	21.8	4.7	32.3	68.8
79M6	817	378	105	794	340	95	1.0	19.5	33.5	2.9	49.2	81.6

Tensile properties of T6 treated P/M materials at room and elevated temperature are listed in Table 3. 7090-SZ alloy exhibited improvements of strength with Sc+Zr addition at room and elevated temperatures. However, the improvements of tensile strength and proof stress of other alloys were not observed at room temperature. Elongation and ductility of T6 treated materials decreased compared to those of as-extruded materials as shown in Table 3.

3.4 Fatigue Strength of P/M Materials

S-N curves obtained by torsion fatigue test at room temperature for T6 treated 7090 alloys are shown in Figure 8. Fatigue strength of the P/M materials of 7090 alloy was increased by additions of minor Sc+Zr. The cyclic torsion numbers to failure in applying torsional stress of 100MPa and 130MPa are listed in Table 4 for all tested alloys. The improvements in the fatigue strength were observed in the 7090 alloys containing Fe, Fe+Ni and Mn instead of Co. However, increases of fatigue strength by Sc+Zr addition did were not clearly observed.

4. Conclusions

Rapidly solidified flakes of Al-9Zn-2.5Mg-1Cu based alloys containing transition metals (Co, Fe, Fe+Ni and Mn) with 0.2Sc+0.2Zr addition were consolidated by the powder metallurgy process. Mechanical properties and micro-structures of both RS flakes and P/M materials were studied in comparison with those without Sc+Zr addition.

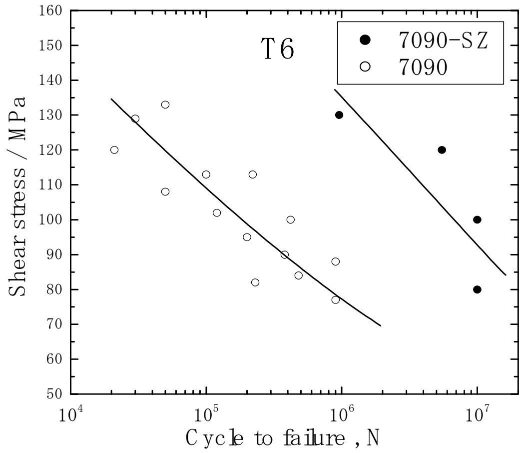

Figure 8: S-N curves of T6 treated P/M materials of 7090 and 7090-SZ alloys.

The following results were obtained.

(1) Uniform and fine dispersion of transition metals aluminide compouns and $Al_3(Sc,Zr)$ particles was observed in the P/M materials containing Sc+Zr. As-extruded P/M materials with Sc+Zr addition exhibited higher hardness than those without addition except for 79M6-SZ alloy. Improvement of hardness by additions of Sc+Zr appeared in the P/M materials after heating at high temperature above 673K.

Table 4 Cycle to failure of T6 treated P/M materials at shear stress of 100MPa and 130MPa.

Alloy	Cycle to failure (N)	
	100MPa	130MPa
7090-SZ	10^7	9.5×10^5
7090	4.0×10^5	3.0×10^4
79F6-SZ	8.9×10^6	2.9×10^6
79F6	2.0×10^6	3.7×10^5
79FN3-SZ	10^7	1.4×10^6
79FN3	10^7	7.5×10^6
79M6-SZ	10^7	6.0×10^5
79M6	10^7	2.0×10^5

(2) Hardness of T6 treated P/M materials of 7090-SZ and 79FN3-SZ alloy was higher than those without Sc+Zr addition. Distribution of very fine η' phase and $Al_3(Sc,Zr)$ particles of 5-10nm in size was observed in the T6 treated P/M materials of all tested alloys.

(3) Tensile strength and proof stress of 7090 with Sc+Zr addition were higher than those of 7090 alloy on both as-extruded and T6 treated at all testing temperatures.

(4) Fatigue strength of the P/M materials of 7090 alloy was increased by additions of minor Sc+Zr.

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Reference

[1] Y. W Riddle, and T.H. Sanders,Jr.: Proc. of ICAA7, (2000),799.
[2] F.A. Costello, J.D. Robson and P.B.Prangnell : Proc. of ICAA8, (2002), 757.
[3] Y.L. Wu, F.H.Froes, C.Li and A.Alvarez : Metall. Mater. Trans., 30A (1999),1017.
[4] A.K. Mukhopadhyay, G.M. Reddy, K.S. Prasad, S.V. Kamat, A.Dutta and C. Mondal : Proc. of Materials Solutions Conference, (2001),63.
[5] O.D.Neikov, D.V.Lotsko, A.I.Sirko, A.V.Sameljuk, G.E.Thompson, N.P.Zakharova and N.A.Yefimov : Proc. of ICAA8, (2002),1223.
[6] H.Fujii, M.Sugamata, J. Kaneko and M.Kubota : J.Japan Inst. Light Metals, 52(2002), 598.
[7] S.Lim, M.Sugamata and J.Kaneko : J.Japan Inst. Light Metals, 41(1991), 440.
[8] X.Z.Li, V.Hansen, J.Gjonnes and L.R.Wallenberg : Acta Mater., 47(1999), 2651.