CONTROLLING THE FORMABILITY AND STRENGTH OF Al-Mg-Si ALLOYS

S J Harris, B. Noble, D G McCartney, X J Jiang and Y Xie
Department of Materials Engineering and Materials Design
University of Nottingham
UK

ABSTRACT

In the processing of 6000 series Al-Mg-Si sheet alloys for automobile applications a processing sequence of solution treatment, quench, hold at ambient, cold forming and paint baking at ~175°C applies. It has been demonstrated that raising the silicon content of a 1%Mg alloy in the as quenched condition improves the strength without a significant change in uniform elongation. Holding for 14 days at ambient increases the strength irrespective of silicon content and maintains the ductility but reduces the work hardening coefficient. On ageing at 175°C for 30 mins (typical of a paint bake) after the 14 day hold, a reduction in strength can occur in high silicon alloys. This reduction has been interpreted as being caused by the dissolution of clusters of solute atoms.

Keywords: Ageing Kinetics, Excess Silicon, 'Paint Bake' ageing, Cluster Dissolution

INTRODUCTION

The development of 6000 series alloys for panel applications in automobiles has received much attention as the ageing pattern has to relate to specific processing conditions. These conditions involve: solution treatment → cold water quenching → store at ambient → cold form → paint → bake for 30 mins at a temperature in the range 170-180°C. It is important that sheet has sufficient ductility for cold forming and that during paint baking sufficient strength is developed to resist local deformation in service[1]. A number of commercial alloys based upon the Al-Mg-Si and Al-Mg-Si-Cu systems, eg. 6009, 6010, 6016 and 6111 have been investigated and treated in a number of different ways to meet the processing conditions specified above[2-6].

Saga[7] has demonstrated that if a simple ternary Al-0.81wt% Si-0.64wt% Mg alloy is held at 25°C for periods up to 27h then the strength of the subsequent bake-hardened alloy (30 min at 175°C) is not sufficient for automobile applications. Furthermore the hold at 25°C prior to cold forming may reduce the formability of the alloy.

To increase the strength of the simple –Al-Mg-Si alloy, additions of copper can be made but this may have adverse effects on the corrosion resistance of the alloy. Alternatively, the silicon concentration can be increased to a level above that corresponding to the quasi-binary Al-Mg_2Si system. The excess silicon approach is the one adopted in the present paper. The data described are the preliminary results of a programme which is investigating the influence of various additions and treatments on the ageing characteristics of Al-Mg-Si alloys at ambient and elevated temperatures. The overall aim of this programme is to maximise cold formability and strength after paint baking 6000 series aluminium sheet alloys.

The results are discussed in terms of the ageing sequence that has evolved from work reported over many years (6) (8) (9).

SUPERSATURATED → SOLUTE → $\beta'' \rightarrow \beta' \rightarrow \beta$ (Mg_2Si)
SOLID SOLUTION ATOM CLUSTERS

EXPERIMENTAL

The compositions of the alloys used in the investigation, together with the levels of excess silicon, are shown in Table I. They were prepared form high purity materials using an Al-Ti-B grain refiner. After casting, the alloys were heated at controlled rates (50 °C/h) to the homogenisation temperature and held for 2h. The alloys were subsequently hot and cold rolled to 1.6mm strip. Samples for tensile testing were cut from the sheet, solution treated and tested in the L-direction as soon as possible after water quenching (WQ condition). Other samples were held at ambient for periods up to 14 days prior to testing (T4 condition). To simulate the paint bake, ageing was carried out on the T4 materials at 175°C for various times. Tensile testing, hardness measurement, differential scanning calorimetry (DSC) and transmission electron microscopy (TEM) were carried out on small samples taken after various stages of heat treatment.

Table I Alloy Compositions

Weight %						
Magnesium	**Silicon**	**Zirconium**	**Iron**	**Aluminium**	**Mg_2Si**	**Excess Silicon**
1.06	0.49	0.08	0.04	Balance	1.34	-
0.99	0.82	0.13	0.04	Balance	1.39	0.31
0.95	1.45	0.10	0.04	Balance	1.26	0.66

AGEING BEHAVIOUR OF THE ALLOYS

Fig. 1 shows the natural ageing behaviour of the three alloys. All showed approximately the same increment in hardness after ageing for 14 days, i.e. 30HV, but it should be noted that the initial hardness levels, immediately after quenching, were significantly different. With increasing excess silicon the as-quenched hardness increased and this is probably the result of enhanced solute clustering taking place during the quench or immediately after quenching before the first hardness determination could be made.

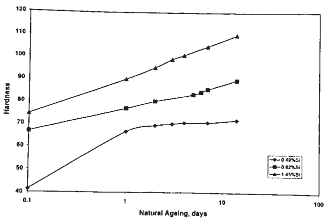

Fig. 1. Natural ageing plots for the three Al-Mg-Si alloys containing 0.49%, 0.82% and 1.45% silicon.

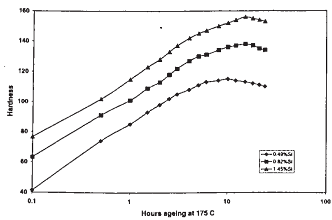

Fig. 2. Ageing plots at 175°C immediately after quenching for the three Al-Mg-Si alloys containing 0.49%, 0.82% and 1.45% silicon.

Fig. 2 illustrates the ageing behaviour at 175°C for alloys aged immediately after water quenching. Again, the trend is for the hardness increment (~75HV) to be unchanged by increasing the excess silicon, but when the difference in as-quenched hardness is taken into account, the strength developed is significantly higher in the alloy with 0.6% excess silicon.

Holding the alloys 14 days at ambient before ageing at 175°C produced significant differences in the balanced alloy compared to those with excess silicon (Fig. 3). In the balanced alloy the hardness increment at 175°C is reduced to 25HV with the hardness increasing continuously from 0-15h ageing. In the alloy with 0.6% excess silicon the hardness increment is again 25HV but in this case the initial stages of ageing at 175°C are characterised by a decrease in hardness. This indicates that some of the smaller solute clusters, developed during the room temperature hold of the 0.6% excess silicon alloy, become unstable at 175°C. They therefore dissolve causing the hardness decrease; the larger sized clusters are more stable and these develop into β'' precipitates thus causing the sharp increase in hardness beyond 1h at 175°c (Fig. 3).

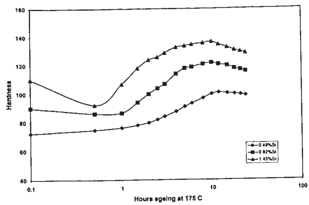

Fig. 3. Ageing plots at 175°C after 14 day hold at ambient for the three Al-Mg-Si alloys containing 0.49%, 0.82% and 1.45% silicon.

TENSILE PROPERTIES OF THE ALLOYS

Having established the ageing behaviour of the alloys, tensile tests were carried out after various ageing conditions to measure the strength, ductility and work hardening behaviour of the alloys. These heat treatment conditions include that which corresponds to a commercial paint bake cycle, i.e. 30 min at 175°C (Table II).

Ageing all the alloys for 30 mins at 175°C immediately after water quenching produced significant increases in proof stress of between 100 and 135 MPa and this was accompanied by a decrease in ductility from ~30% to ~25%. Interposing a 14 day hold between the water quench and the 30 mins 175°C age (i.e. the simulated paint bake cycle) produced large changes in the strength characteristics. The 30 mins age at 175°C for the balanced alloy produced a proof stress increment of 6 MPa (relative to the 14 day age at ambient). For the 0.3% excess silicon alloy this increment increased to 19 MPa, but for the 0.6% excess silicon the increment decreased by 18 MPa. This is brought about because the paint bake heat treatment cycle is ageing the alloy to the minimum in the hardness plot of Fig. 3. In this 0.6% excess silicon alloy the 30 mins heat treatment at 175°C is dissolving the majority of the solute clusters that have developed during the 14 day hold at ambient, but β'' precipitation is not yet sufficiently developed to cause a significant increase in strength.

Table II Tensile Test Data on Heat Treated Alloys

%Si	Alloy condition	0.2%PS MPa	UTS MPa	Total Elongation %	Uniform Elongation %	Work Hard Coefficient
0.49	WQ	83	199	34	33	0.36
0.49	WQ + 14 days at RT	152	263	29	29	0.28
0.49	WQ+30 min at 175°C	186	271	24	21	0.22
0.49	WQ+14 days at RT and 30mins at 175°C	158	261	28	26	0.27
0.49	WQ+15h at175°C	265	305	17	13	0.13
0.82	WQ	115	235	30	29	0.32
0.82	WQ + 14 days at RT	176	288	31	28	0.27
0.82	WQ+30mins at 175°C	245	331	26	23	0.19
0.82	WQ + 14 days at RT, 30mins at 175°C	186	218	30	29	0.26
0.82	WQ+15h at 175°C	350	362	9	7	0.04
1.45	WQ	126	268	30	29	0.33
1.45	WQ + 14 days at RT	199	313	34	32	0.28
1.45	WQ + 30mins at 175°C	261	352	23	21	0.18
1.45	178WQ + 14 days at RT + 30mins at 175°C	178	291	32	30	0.27
1.45	WQ+15h at 175°C	354	372	12	7	0.05

TRANSMISSION ELECTRON MICROSCOPY

Transmission electron microscopy (TEM) was undertaken on the balanced alloy and that containing 0.6% excess silicon after ageing 14 days at ambient and 30 min at175°C. No evidence could be found for the presence of any precipitation, i.e. β'' precipitation had not yet commenced and the solute atom clusters could not be resolved using conventional TEM. After ageing 2h at 175°C clear β'' precipitates could be resolved. Fig. 4 shows the typical microstructures for the balanced alloy aged 5h at 175°C with and without a prior hold of 14 days at ambient. The 14 day hold results in a reduction in precipitate number density and an increase in the size of the β'' precipitate which suggests that excess silicon stimulates the formation of solute atom clusters, which are then able to nucleate β'' precipitation during the age at 175°C.

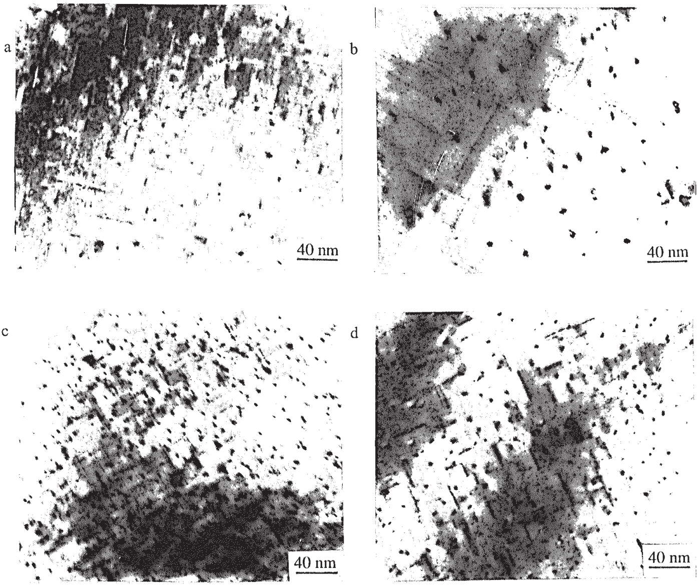

Fig. 4 TEM micrographs taken with a[001] zone axis
a) 0.49% silicon alloy after 5h ageing at 175°C directly after quenching
b) As a) but the sample was held for 14 days at ambient prior to ageing at 175°C
c) 1.45% silicon alloy after 5h ageing at 175°C directly after quenching
d) As c) but sample was held for 14 days at ambient prior to ageing at 175°C

DIFFERENTIAL SCANNING CALORIMETRY

Since there was no TEM evidence of precipitation taking place after ageing the alloys for 30 min at 175°C, differential scanning calorimetry (DSC) was carried out in an attempt to detect the formation and dissolution of solute clusters during the heat treatment cycles. Fig. 5 shows DSC traces for the balanced alloy. After 14 days at ambient there is a strong endotherm at 210°C which represents the dissolution of solute clusters that have formed during the hold at room temperature. A further heat treatment of 30 min at 175°C increases the size of this endotherm indicating that further solute clusters have developed during the hold at 175°C. This is consistent with the change in mechanical properties for the balanced alloy shown in Fig. 3 and Table II, i.e. 30 min at 175°C after a 14 day hold at ambient produces a small increase in strength.

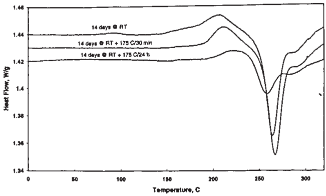

Fig. 5. DSC plots for an Al-Mg-Si alloy containing 0.49% silicon after holding for a) 14 days at ambient and then b) 30 min at 175°C and c) 24h at 175°C

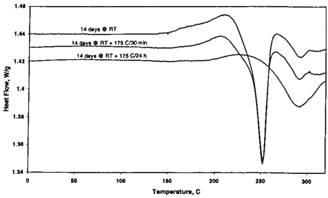

Fig. 6. DSC plots for an Al-Mg-Si alloy containing 1.45% silicon after holding for a) 14 days at ambient and then b) 30 min at 175°C and c) 24h at 175°C

Fig. 6. shows equivalent DSC traces for the 0.6% excess silicon alloy. Again, there is a cluster dissolution endotherm at 210°C after ageing 14 days at ambient. However, after a further heat treatment of 30 min at 175°C the size of this endotherm decreases, indicating that the population of solute clusters at 175°C in the excess silicon alloy is decreasing rather than increasing. This suggests that the solute clusters in the excess silicon alloy are of a smaller size then those in the balanced alloy. These observations are consistent with the change in mechanical properties for the 0.6% excess silicon alloy shown in Fig. 3 and Table II, i.e. 30 min at 175°C after a 14 day hold at ambient produces a small decrease in strength.

CONCLUSIONS

1. Increasing the silicon content of a 1.0wt% Mg alloy above its balanced composition promotes an increase in strength and hardness in the as-quenched condition.
2. Naturally ageing the alloy for up to 14 days increases the strength of balanced and excess silicon alloys by ~70MPa whilst uniform elongation remains constant and the work hardening coefficient reduces.
3. Naturally ageing for 14 days prior to ageing 30 mins at 175°C (simulated paint bake) results in a softening process taking place in alloys with 0.6% excess silicon.
4. The softening process in high excess silicon alloys arises because of the dissolution of solute clusters formed during the hold for 14 days at ambient.
5. The softening process in high excess silicon alloys is complete after ageing for 1h at 175°C. Thereafter rapid strengthening occurs due to the formation of β'' precipitates.

REFERENCE

1. J. Hirsch, Materials Science Forum, 242, (1997), 33.
2. F. Ostermann, in 'Aluminium Technology for Automobile construction', ed. W. J. Bartz, Mech.Eng Publications, London, (1993), p.1.
3. A. K. Gupta, G. B. Burger, P. W. Jeffrey and D. J. Lloyd, ICCA 4, T. H. Sanders and E. A. Starke, vol. 3, Atlanta, USA (1994), p. 177.
4. A. K. Gupta, P. H.. Marois and D. J. Lloyd, Materials Science Forum, 217-222, (1996), 801.
5. P. E. Fortin, M. J. Bull and D. M. Moore, SAE Int. Congress, (1983), SAE paper No. 830096.
6. G. Huppert, and E. Hornbogen, ICAA 4, vol. 1, Atlanta, USA (1994), p. 628.
7. M. Saga, Y. Sasaki, M. Kikuchi, Z. Yan and M. Matsuo, Materials Science Forum, 217-222, (1996), 821.
8. G.W. Lorimer, in 'Precipitation Processes in Solids', ed. K.C. Russell, H.I Aaronson, AIME Conf. Proc, Warrendale, PA (1978), p. 107.
9. G.A.Edwards, G.L. Dunlop and M.J. Couper, ICAA4, vol 1, Atlanta, USA (1994), p. 620.