THE 4TH INTERNATIONAL CONFERENCE ON ALUMINUM ALLOYS

MICROSTRUCTURAL STABILITY AND FRACTURE BEHAVIOR IN AI-Si-Ge ALLOYS

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<u>Abstract</u>

The present study investigates the coarsening behavior of SiGe diamond structure precipitates in ternary Al-Si-Ge and quaternary Al-Si-Ge-Cu alloys aged at 120° C and 160° C and compares the experimental results with predictions by the Lifshitz-Wagner coarsening theory. Furthermore, tensile and fracture properties of these alloys are studied.

Introduction

A recently developed age-hardenable Al-Si-Ge alloy utilizes a fine and uniform distribution of SiGe particles which have an estimated critical size for the transition from shearing to looping by dislocations at the yield stress of about 2nm. These features result in a high degree of hardening for a small volume fraction of particles. SiGe particles have a diamond structure and can be expected to be more thermally stable than precipitates of other age-hardenable aluminum alloys due to the low solubility of both Si and Ge in Al and the incoherent precipitate interface. The low strength of ternary Al-Si-Ge alloys can be increased by the addition of Cu since quaternary Al-Si-Ge-Cu alloys have a higher number of precursory clusters during quenching which act as nucleation centers for diamond SiGe precipitates during aging [1-4].

As compared to binary Al-Si and Al-Ge alloys, the precipitates in ternary Al-Si-Ge alloys are found to nucleate on a much finer scale and with a lower tendency to overage under comparable conditions. The aging characteristics of ternary Al-Si-Ge alloys are not affected by prior low temperature aging or preaging cold-work. The addition of less than 2.5wt.% Cu does not influence the distribution of SiGe particles and leads to an increase of the hardness level by about 60%. However, the addition of more than 2.5wt.% Cu results in the nucleation of θ' (Al₂Cu) precipitates in addition to the SiGe precipitates; the rapid coarsening of θ' precipitates leads to rapid overaging of θ' containing alloys [3,4].

Experimental Procedure

The alloys used in this study were provided by ALCOA; their chemical composition is given in Table I. Alloys 1 and 2 have a nearly identical composition except for Cu so that the influence of Cu, independent of other factors, could be studied. Since Si has a smaller and Ge has a larger atomic diameter than Al, the composition of alloy 3 was chosen in such a way that the atomic size misfit is compensated after the formation of pairs of Si and Ge. Alloys 1, 2 and 3 contain nearly the same total atomic fraction of Si and Ge. Alloy 2014-T4 was selected for comparison of the mechanical properties but contains a much higher level of solute than alloys 1, 2 and 3.

		Composition (at.%)					
No.	Alloy	Si	Ge	Cu	Mg	Mn	Al
1	Al-1.02Si-0.95Ge	0.99	0.36				Balance
2	Al-1.00Si-0.99Ge-2.57Cu	0.98	0.38	1.12			Balance
3	Al-0.55Si-2.02Ge	0.54	0.76				Balance
4	2014-T4	0.84		1.95	0.57	0.40	Balance

Table I.	Chemical	Composition	of Allo	ys
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Optimum homogenization temperatures of 500°C for the ternary alloys (1 and 3) and of 490°C for the quaternary alloy (2) were selected based on the thermal analysis using a Perkin Elmer Differential Scanning Calorimeter (DSC). These alloys were homogenized within 30hrs, hot rolled, solution heat treated at 490°C (ternary alloys) or 480°C (quaternary alloy), and cold water quenched. Alloys 1, 2 and 3 were then aged in air furnaces at 120°C for up to 60 days or at 160°C for up to 16 days.

Vickers microhardness measurements were performed at room temperature using a Kentron Microhardness Tester AK. Samples for Transmission Electron Microscopy (TEM) were prepared using standard techniques; all TEM examinations were conducted using a Philips EM 400T. The average radii of the diamond structure precipitates were determined by quantitative stereological methods [5] and corrected for truncation and overlap [6]; foil thicknesses were determined under two-beam conditions from oscillations in intensity of Convergent Beam Diffraction Patterns (CBED) [7].

Samples of all alloys were peak-aged at 160°C. Tensile tests were performed at a strain rate of 10-3s-1 with peak-aged alloys 1, 2 and 3 in the longitudinal direction [8]. Kahn tear tests were performed with peak-aged alloys 1 and 2 in both longitudinal and transverse directions [9]. In addition to this, tensile and Kahn tear tests were conducted with alloy 2014-T4 for comparison. All mechanical tests were performed using an 810 Material Test System (MTS). The fracture surfaces were investigated using a JSM-35 Scanning Electron Microscope (SEM).

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Results and Discussion

Figures 1a and 2a show the average diameters of the SiGe precipitates in alloys 1 and 2 after different aging times for aging temperatures of 120°C and 160°C, respectively (note the different time scales used for the two temperatures). The magnitude of the values is in correspondence with the estimated particle size for the transition from shearing to bypassing [2] which is related to the size at which such particles form from a cluster. Figures 1b and 2b depict the relationship between the cube of the average radii and the aging time at 120°C and 160°C, respectively. The relationship is in both cases linear within the errors of measurement. This is in agreement with the prediction of the coarsening theory by Lifshitz [10] and Wagner [11]. The coarsening rate for both the ternary and the quaternary alloys is significantly greater at 160°C than at 120°C as can be expected from the exponential temperature dependence of the diffusion coefficients. The average radii for the SiGe precipitates are slightly greater in the quaternary alloy than in the ternary alloy at both aging temperatures although both alloys contain nearly identical amounts of Si and Ge. However, the difference between the average diameters of the SiGe precipitates at a given aging time and temperature is well within the error bars of the measurement suggesting that the presence of Cu has no significant effect on the nucleation and coarsening of the SiGe precipitates.

The coarsening theory by Lifshitz and Wagner predicts that the variation of the mean radius, r, with time, t, is given by the following equation [12]:

$$r^{3}-r_{o}^{3} = 8\gamma Dc_{o}V_{m}^{2}(t-t_{o})/9RT$$
 (1),

where r_o is the mean particle radius when coarsening commences at the time t_o , γ is the specific precipitate-matrix interfacial free energy, D and c_o are the diffusivity and the equilibrium molar concentration, respectively, of the solute species in the matrix phase at the temperature T, V_m is the molar volume of the precipitate, and R has its usual meaning. The interfacial energy can be estimated from the slope of the graphs in Figures 1b and 2b using equation (1). The diffusivity data of Si in Al and Ge in Al used in the calculation are those published in [13]. The ratio between Si and Ge is taken into account for both alloys, but the influence of Cu is neglected. A total equilibrium concentration of Si and Ge of 0.2wt.% is estimated from [14]. The molar volume is calculated based on the values published in [2] for Si and Ge diamond cubic structures. The interfacial energy values shown in Figures 1b and 2b are in the expected range; differences are due to significant errors in low temperature diffusivity data [12].

Table II shows the results of the tensile tests performed with peak-aged alloys 1 through 3 in longitudinal direction and compares them with alloy 2014-T4. Note that the first three alloys contain nearly the same total atomic fraction of Si and Ge. The experimentally determined yield strength is for all alloys in good agreement with the theoretical upper limit (also shown in Table II) described by the modified Orowan equation [1]:

$$\Delta \sigma_{\rm p} \approx {\rm Gb} \sqrt{f/d} \tag{2},$$

where $\Delta \sigma_p$ is the contribution of the SiGe particles to the yield strength, G is the



Figure 1. Coarsening behavior of SiGe diamond precipitates in alloys 1 and 2 aged at 120°C. (a) Average diameters. (b) Relationship between cube of average radii and aging time, interfacial energy values (Y).



Figure 2. Coarsening behavior of diamond SiGe precipitates in alloys 1 and 2 aged at 160°C. (a) Average diameters. (b) Relationship between cube of average radii and aging time, interfacial energy values (Y).

shear modulus of the matrix (26GPa [1]), b is the Burgers vector (0.284nm [1]), f is the volume fraction, and d is the diameter of the particles (both determined by TEM). Note that this calculation does not consider the contribution of θ' in case of the quaternary alloy. The significant increase of the yield strength of the second alloy as compared to the first alloy results from the contribution of θ' . The increase of the yield strength of the third alloy by a factor of about 1.4 as compared to the first alloy is due to a decrease of the average precipitate diameter from 8.0nm to 5.0nm. The decrease of the precipitate diameter is achieved by choosing the composition of the third alloy in such a way that the atomic size misfit cancels. The tensile properties of the first three alloys are significantly lower than those of alloy 2014-T4.

Δσ _p * (MPa)	σ _y * (MPa)	σ _{TS} * (MPa)	ε _f * (%)
77	73	137	24
76	168	280	10
112	105	164	17
	344	486	21
	Δσ _p * (MPa) 77 76 112 	$\begin{array}{ccc} \Delta \sigma_{p}^{*} & \sigma_{y}^{*} \\ (MPa) & (MPa) \end{array}$ 77 73 76 168 112 105 344	$\begin{array}{c ccccccccccccccccccccccccccccccccccc$

Table II. Comparison of Tensile Properties

* $\Delta \sigma_p$ - theoretical contribution of SiGe particles to the yield strength, σ_y - yield strength, σ_{TS} - tensile strength, ε_f - strain after fracture

The fracture surfaces of the first three alloys investigated by SEM reveal that in all cases fracture occurred as dimpled rupture in a transgranular mode.

Table III shows the results of the Kahn tear tests performed with peak-aged alloys 1 and 2 in both longitudinal and transverse directions.

The unit propagation energy which can be used as a relative index of fracture toughness is significantly higher for alloys 1 and 2 than for alloy 2014-T4. Furthermore, this value is higher for the quaternary alloy than for the ternary alloy if samples tested either in longitudinal or transverse direction are compared.

In the near future the volume fraction of voids as a function of strain and aging time will be determined in alloy 3 using a pycnometer [15]. The fracture behavior of this alloy will be related to the SiGe precipitates based on the dislocation model for void nucleation developed by Goods and Brown [16].

Alloy	Tear Strength (MPa)	TYR*	Unit Propagation Energy (J/mm ²)	
Al-1.02Si-0.95Ge (L)*	179	2.45	0.248	
Al-1.02Si-0.95Ge (T)*	170		0.190	
Al-1.00Si-0.99Ge-2.57Cu (L)*	299	1.78	0.304	
Al-1.00Si-0.99Ge-2.57Cu (T)*	274		0.216	
2014-T4	545	1.58	0.113	

Table III. Comparison of Fracture Properties

* L - longitudinal orientation, T - transverse orientation, TYR - ratio between tear strength and tensile yield strength

Conclusions

The relationship between the cube of the average radii of the SiGe diamond precipitates in alloys 1 and 2 and the aging time at 120° C and 160° C is linear as predicted by the coarsening theory by Lifshitz [10] and Wagner [11]. The difference between the average diameters of the SiGe precipitates in the ternary and quaternary alloys at a given aging time and temperature is well within the errors of measurement suggesting that the presence of Cu has no significant effect on the nucleation and coarsening of the SiGe precipitates. The estimated precipitate-matrix interfacial free energy of 500 to 800mJ/m² is in the expected range.

The experimentally determined yield strength of all alloys agrees well with the theoretical upper limit described by the modified Orowan equation [1]. An increase of the yield strength of alloy 3 by a factor of 1.4 as compared to alloy 1 is achieved by decreasing the average precipitate diameter from 8.0nm to 5.0nm as a result of choosing the composition in such a way that the atomic size misfit is compensated after the formation of pairs of Si and Ge.

Acknowledgement

The authors wish to thank NASA-Langley Research Center for providing funding under Grant No. NAG-1-745, Dennis Dicus, program monitor.

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