Lightweight Alloys for Aerospace Applications

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LIGHTWEIGHT ALLOYS FOR AEROSPACE APPLICATION

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Dr. William Frazier and Dr. Nack J. Kim

Proceedings of Symposium Sponsored by
the Non-Ferrous Metals Committee of
the Structural Materials Division (SMD) of

Held at the TMS Annual Meeting
in New Orleans, LA, USA
Partial funding for this publication was provided by the Seeley W. Mudd Fund.
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FOREWORD

This manuscript contains a collection of 27 papers presented at the Lightweight Alloys for Aerospace Applications symposium at the TMS Annual Meeting in New Orleans, February 12-15, 2001. The manuscript contains outstanding papers on:

- Current understanding of coarsening of precipitates in novel aluminum alloys
- Deformation, fracture and fatigue and corrosion resistance of aluminum-lithium alloys
- Nano-crystalline aluminum alloys
- Processing, fatigue and fracture and relationships to microtexture in Ti
- Status of gamma titanium aluminides
- Microstructure and properties of metal matrix composites

ACKNOWLEDGEMENTS

The organizers of the symposium would like to extend their sincere appreciation to Professors Erhard Homborgen, Gary Shiflet, Thomas Sanders, Hugh McQueen, Arun Gokhale and to Drs. Jonathan Paul, Kamran Nikbin and Awadh Pandey for participating in the symposium as invited speakers and to Dr. Mary C. Juhas and Prof. O.S. Es-Said who helped in chairing the sessions. The organizers would like to thank all the participants who made this symposium a big success.

Kumar V. Jata  
Air Force Research Laboratory, USA  
June 2001
Precipitation hardening of aluminum was discovered about 100 years ago by Dr. Alfred Wilm [1,2]. Using aluminum alloys as example a survey is given on mechanism and limits of precipitation hardening. It is discussed how hard, nanometer-size particles (nanos, greek, the dwarf) can form as an ultra fine dispersoid. A simple example for optimum conditions is provided by diamond cubic particles (Si, Ge) in the f.c.c. Al-Matrix. The role of a sequence from more to less metastable phases is discussed, as well as the effects of additional (and trace) alloying elements. The amount of precipitation hardening is limited, besides by the volume fraction of particles, by their strength. This in turn determines the critical diameter above which the transition from passing to by-passing by dislocations takes place. Simple models for the calculation of this microstructural parameter are discussed.

From combinations of precipitation hardening with other hardening mechanisms the limits for ultra high strengths are defined.
The story of a discovery

It is now about 100 years ago that Dr. Alfred Wilm started experiments with a wide range of aluminum alloys at the metallurgical department of the Central Institute for Scientific and Technological Studies in Neubabelsberg (close to Berlin). Major motivation for his work was to increase the insufficient strength of this then still relatively young material. By 1906 he had developed a new type of alloy with 3.5-5.5% Cu and less than 1% Mg and Mn with a strength of more than 400 MPa. It was soon well known by its trade name Duralumin. The prefix contains a possibly intended ambiguity: *durus* (latin, hard), but also *Dürener Metallwerke*, (Rhineland) the industrial firm, where the alloy was produced and shaped. [1,2]

There are the options for evolutionary, predictable progress in science and technology or revolutionary developments. The discovery of precipitation hardening was unpredicted. Alfred Wilm knew all the physical metallurgy of his days. He knew that metals could be solid solution hardened and work hardened. He also knew that the carbon steels had to be quenched for hardening. Therefore he melted a large number of alloys which he investigated in an as-quenched, slowly cooled and worked state. To his great frustration he found out that – different from steels – some alloys became even softer by quenching. However, one day, many quenching experiments were performed on saturday morning. Not all the hardness measurements were completed the same day. The sun was shining and Wilm went out for the week-end to go sailing. Next monday morning the hardness measurements were completed, and surprisingly hardness as well as all strength properties had increased considerably. At first, the technician was blamed for sloppy work, but then all the measurements were repeated carefully. In addition, duration and temperature of aging was varied systematically.

1. Wilm had to follow the wrong hypothesis of the analogy between steel and aluminum to expect hardening by rapid quenching.
2. He had to be lazy, not to complete his measurements immediately, to realize the importance of aging.
3. He had to recognize the completely unexpected result, reproduce, optimize and apply it. He could, however, not understand that the production of a fine dispersoid of nm-size particles is the physical cause of precipitation hardening. So he did not know that he discovered the first nano-technology.

Lattice correspondences

Considerable strength of Al-alloys is always due to precipitation hardening. This requires the formation of an even and ultra-fine dispersoid of hard particles. The optimum would be achieved by hard diamond cubic particles in the fcc Al-based matrix. Size and spacing should be in the nm-range. In fact, often either a fine microstructure of soft particles emerges or a coarser one of strong particles. Both do not lead to the desired hardening effect. (Fig. 1)

This paper will discuss the mechanisms of precipitation of second phases, using fcc solid solutions of Al as examples. They have been well explored in the past 50 years [3, 4]. The results can be interpreted by a generalized Ostwald step rule. In addition dislocation theory provides the principles on how to obtain optimum strengthening.

Starting condition is a supersaturated solid solution α_s, which is often obtained by quenching from a temperature of high solubility. This implies that not only solute atoms are obtained in excess, but also lattice vacancies (Fig. 3). Higher super-supersaturations α_{ss} can be obtained by rapid solidification of liquids or vapour deposition - which may lead either to a glassy, or quasi-crystalline, or crystalline (massive crystallization) phases. Not only vacancies have to be considered for the analysis of precipitation reactions in Al-alloys, but also 0 < d ≤ 3-dimensional defects, such as dislocations (d = 1), grain boundaries, stacking faults (d = 2).
density of dislocations \( \rho_d \), for example, may be varied by many orders of magnitude \( (\rho_d \leq 10^{15} \text{ m}^{-2}) \) by plastic deformation of supersaturated crystals \( \alpha_{sd} \).

Figure 1: Anomalies of different physical properties due to nm-size particles

![Figure 1](image1)

Figure 2: Types of coherency between matrix \( \alpha \) and particle \( \beta \), (a) coherent, ordered, (b) constrained coherent (shear), (c) partially coherent, (d) non coherent

![Figure 2](image2)

Table I Nucleation mechanisms in Al-Cu- alloys

<table>
<thead>
<tr>
<th>crystal structure</th>
<th>stability</th>
<th>coherency with fcc</th>
<th>defects geom. dimension</th>
<th>0-d</th>
<th>1-d</th>
<th>2-d</th>
</tr>
</thead>
<tbody>
<tr>
<td>cluster, GP-zones</td>
<td>↓</td>
<td>c</td>
<td></td>
<td>+</td>
<td>+</td>
<td>+</td>
</tr>
<tr>
<td>( \Theta^\circ )</td>
<td>↓</td>
<td>c</td>
<td></td>
<td>+</td>
<td>+</td>
<td>++</td>
</tr>
<tr>
<td>( \Theta^\prime )</td>
<td>↓</td>
<td>pc</td>
<td></td>
<td>+++</td>
<td>++</td>
<td>+</td>
</tr>
<tr>
<td>( \Theta )</td>
<td>max</td>
<td>nc</td>
<td></td>
<td>+++</td>
<td>++</td>
<td>+</td>
</tr>
</tbody>
</table>

\( \Theta \) coherent, \( \text{pc} \) partially \( \Phi \), \( \text{nc} \) non \( \Phi \), + low activation barrier for nucleation, +++ high activation barrier for nucleation
Finally a comparison must be made with respect to coherency between the structure of the matrix crystal $\alpha$ and the newly formed phases $\beta_i$. This relationship may imply:
a) non-coherency, b) partial coherency, c) constrained coherency, d) full coherency (Figs. 2, 3). These possibilities provide a decreasing amount of the structural term of the interfacial energy $\gamma_{\alpha\beta}$, which essentially controls the nucleation behavior of $\beta_i$ from $\alpha$ [5, 6, 7] (Table 1). The crystal structures of strong particles (termodynamically stable and high resistance to dislocation motion: $\Theta$-$\text{Al}_2\text{Cu}$, $\delta$-$\text{AlLi}$, Si) do not fulfill the prerequisites for coherency with fcc Al (Fig. 3).

![Figure 3: Formation of non-coherent Si from fcc Al (Si) solid solution](image)

**Metastable phases**

The sequence of the precipitate phases $\beta_i$ for the Al-Cu-alloys implies a decreasing coherency with increasing thermodynamical stability (Fig. 4): $\Theta'' \rightarrow \Theta' \rightarrow \Theta$.

![Figure 4(a): Sequence of metastable, coherent $\rightarrow$ non-coherent $\Theta$-$\text{Al}_2\text{Cu}$ in AlCu-alloys](image)
At a certain amount of undercooling the relatively smaller driving force becomes sufficient for a quicker formation of the metastable phase, relative to the more stable one. Its nucleation is favoured by the formation of a low interfacial energy $\gamma_{\alpha\beta}$ with $G_N$ activation energy of nucleation, $g_\beta$ specific free energy change $Jm^{-3}$, $i$ number of atoms in the nucleus, [6]:

$$G_N(T) = g_\beta(T) i + \gamma_{\alpha\beta} i^{2/3}$$  \hspace{1cm} (1a)$$

$$\frac{dg_\Theta}{di} < \frac{dg_\Theta}{di} < \frac{dg_\Theta}{di}$$  \hspace{1cm} (1b)$$

$$\gamma_{\alpha\Theta} < \gamma_{\alpha\Theta} < \gamma_{\alpha\Theta}$$  \hspace{1cm} (1c)$$

Thus crystallographic coherency and classical nucleation theory explain why the less stable phase forms first and not the one which leads to the most stable state. There are two reasons why multi-stage reactions cannot be expected: 1. No metastable phases exist (Al-Si, Al-Ge, Al-Zn) [8], 2. The most stable phase can form coherently. Examples for the second case (as (γ + γ)-Ni alloys) do not exist for Al-alloys.

**Extrinsic defects**

Structural correspondences are also evident between lattice defects and types of interfaces: $d = 0$, lattice vacancy-coherency, $d = 1$, lattice dislocation-interfacial dislocation- partial coherency, $d = 2$, grain boundary - non-coherent interface.

This in turn leads to particular combinations of lattice defects and interfaces [5, 6, 7, 8], which provide minimum activation barriers and consequently maximum rates of formation (Table 1). Such favourable nucleation processes occur only at sites where lattice defects are preexisting. Consequently the bulk rate depends on grain size or dislocation density. This leads to the phenomenon that different phases form simultaneously, but at the various sites inside the alloy. During particle growth small particles dissolve in favour of large ones. But even more important, less stable phases are dissolved in the environment of more stable phases due to the differences in local solubilities. Consequently particle-free zones and uneven distribution of particles in the interior of grains develop (Fig. 5).
At any defect: \[ G_N(T) = g_\beta(T) + (\gamma_{\alpha\beta} - \gamma_{\text{def}})T^{2/3} \] (2a)

At dislocation: \[ G_{N_{\text{min}}} = g_{\Theta'}(T) + (\gamma_{\alpha\Theta'} - \gamma_{\alpha})T^{2/3} \] (2b)

At boundary: \[ G_{N_{\text{min}}} = g_{\Theta}(T) + (\gamma_{\Theta'} - \gamma_{\Theta})T^{1/3} \] (2c)

Besides lattice defects some trace elements are known to have favorable effects on the dispersoid microstructure. Wilm reported already the effect of Mg on the binary Al-Cu-alloy. An additional effect of Ag was found later on [4]. Such elements should favor the formation of a finer dispersoid. This can be explained by solubility of the trace element in the coherent metastable phase, while the stable non-coherent phase shows no solubility [9]. This in turn retards anomalous coarsening by the in-situ-transformation, coherent \( \rightarrow \) non-coherent.

**Rules for the creation of nano-size dispersoids**

Unconstrained coherent nucleation comes closest to homogeneous nucleation and therefore to the formation of the finest possible dispersoid of the second phase \( \beta_i \) [10]. However, a dispersoid of an incoherent, hard phase is desired.

For Al-alloys there is usually a multitude of options for reactions which lead closer to equilibrium at a wide range of rates (Equ. 1, 2). For Al-Cu the following possibilities exist: The stable phase \( \Theta \) and three metastable phases, combined with three types of lattice defects and eventually a continuous and a discontinuous mode; i.e. there is a competition between 13 options. The fastest ones will win and produce the microstructure. The thermodynamical principle behind this phenomenon seems to be the maximization the initial rate of entropy production. The eventual creation of structural order \( -S_{\text{str}} \) is overcompensated by the fast production of thermal entropy \( +S_{\text{th}} \). This in turn is a generalisation of Ostwald's step rule [10].
\[ \frac{d(S_h - S_m)}{dt} \rightarrow \text{max.} \] \hspace{1cm} (3)

The initial, microstructure can be interpreted in terms of Equ. 3. This relation is favored by the following set of sometimes conflicting properties:

1. Enthalpy of the formation for precipitate phases \( \rightarrow \text{MAX.} \),
2. Size of its elementary cell, i.e. min. nucleus size \( \rightarrow \text{MIN.} \),
3. Coherency with fcc lattice \( \rightarrow \text{MAX.} \),
4. Correspondence between structure of defect and interface \( \rightarrow \text{MAX.} \),
5. Size of prenucleation clusters* \( \rightarrow \text{MAX.}^*, \)
6. Diffusion path \( \rightarrow \text{MIN.} \).

* this is favored in ternary alloys by the ±-effect, i.e. one solute larger, the other smaller than Al (Table 2).

<table>
<thead>
<tr>
<th>Smaller</th>
<th>Larger</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cu</td>
<td>Ti</td>
</tr>
<tr>
<td>Zn</td>
<td>Sn</td>
</tr>
<tr>
<td>Si</td>
<td>Cd</td>
</tr>
<tr>
<td>Ni</td>
<td>Ge</td>
</tr>
<tr>
<td>Co</td>
<td>Li</td>
</tr>
<tr>
<td>Fe</td>
<td>Mg</td>
</tr>
<tr>
<td>Mn</td>
<td>Ga</td>
</tr>
<tr>
<td>Cr</td>
<td></td>
</tr>
</tbody>
</table>

Table II Atomic size ratio of solutes in Al

Hardening

The formation of a fine dispersoid of particles is not sufficient for hardening \([11,12]\). Necessary for the validity of the OROWAN-equation (Figs. 6, 7, 8) are strong particles which sustain the stress exerted by moving dislocations, \( d > d_c \):

\[ \Delta \sigma_p = \frac{Gb}{S_{\text{eff}}} = C \frac{Gb}{S - d} \] \hspace{1cm} (4)

there is always a critical particle diameter \( d_c \) below which the particles are sheared. This diameter should be as small as possible (Table 3). The critical diameter \( d_c \) defines the maximum hardening effect by a certain volume fraction of particles (Fig. 6). It can be estimated by examining the force \( F \) which a single particle is able to exert on a looping dislocation. For \( F < Gb^2 \), shearing occurs and by-passing for \( F \geq Gb^2 \) (Figs. 7, 8).

Particle strengthened Al-alloys may contain a dispersoid of particles: ordered or disordered, coherent or non-coherent with the fcc-matrix. For coherent ordered particles the critical diameter \( d_c \) follows from \( Gb^2 = \gamma d = F \) where \( \gamma \) is the antiphase domain boundary energy (APB) and \( d \) the particle diameter:

\[ d_c = C \frac{Gb^2}{\gamma} \] \hspace{1cm} (5a)

and \( C=1 \) is a geometrical factor depending on the shape of the particle. \( G \) and \( b \) are shear
modulus and Burgers vector of the fcc-matrix solid solution. For disordered coherent particles no APB has to be created, but the amount of the difference in critical shear stress between matrix α and particle β \((τ_α-τ_β)\) becomes relevant for modest hardening:

\[
d_c = C \frac{Gb}{(τ_α-τ_β)} \tag{5b}
\]

Pores or liquid inclusions are sheared at any size, inspite of their strong hardening effect (Equ. 4):

\[
d_c = \infty \tag{5c}
\]

Particles β with a different crystal structure from the matrix α require the theoretical shear stress to create a dislocation \(b_β\). Consequently only very small incoherent particles are sheared:

\[
d_c = C \frac{4πbG_α}{G_β} \tag{5d}
\]

Low \(d_c\)-particles like Si will provide the highest hardening pro volume fraction. These equations have to be modified if not one but a pair or more dislocations interact with a particle. It follows that the “art” of causing precipitation hardening implies the production of even and \((S \to \text{min}, \text{Equ. 4})\) fine dispersoids of particles with small critical sizes \(d_c\) (Table 3).

![Figure 6: Calculated (Equ. 4) upper limit of hardening by different volume fractions of particles in Al.](image)

Usually, it is easy to form small subcritical particles \(d < d_c\). As they are sheared they must cause less hardening than the by-passing mechanism. Also consequences on localization of strain have to be considered, for example on initiation and propagation of cracks under fatigue or stress corrosion conditions.
Finally it has to be considered that ultra high strength cannot be obtained by precipitation hardening $\Delta \sigma_0$ alone. A high yield stress $\sigma_y$ must be built up from contributions of additional hardening mechanisms. They can be systematically discussed by considering the 0- to 2-dimension of obstacles to the motion of dislocations [12].

![Diagram showing the relationship between Particle Diameter $d_p$ (Å) and localized homogeneous plastic strain.](image)

Figure 7: Definition of size ranges and critical particle diameters $d_c$, for 1% particles

$$1000 \text{MPa} \approx \frac{G}{2\pi} \approx \sigma_y = \sigma_0 + \Delta \sigma_1 + \Delta \sigma_d + \Delta \sigma_b + \Delta \sigma_p$$  \hspace{1cm} (6)

Where $\sigma_0$ is the very low strength of pure Al, and $\Delta \sigma_i$ the contribution of solid solution hardening, $\Delta \sigma_d$ of a dislocation forest, $\Delta \sigma_b$ of grain boundaries. The different terms are not independent of each other. For our discussion it is important that for $d > d_c$, fine grain hardening $\Delta \sigma_b$ becomes irrelevant. $\Delta \sigma_b$ and $\Delta \sigma_d$ are used to built up high strength of Al-alloys. However, precipitation hardening $\Delta \sigma_p$ contributes usually the biggest share to high strength. This in turn is always due to strong particles in the size range between 1 and 10 nano-meters (Table 3).

<table>
<thead>
<tr>
<th>Examples</th>
<th>Coherency</th>
<th>$d_c$/nm</th>
<th>Notes</th>
</tr>
</thead>
<tbody>
<tr>
<td>Si, Ge</td>
<td>n, p</td>
<td>2</td>
<td>diamond structure</td>
</tr>
<tr>
<td>$\Theta$-Al$_2$Cu</td>
<td>n, p</td>
<td>3-10</td>
<td>intermetallic compounds</td>
</tr>
<tr>
<td>T-Al$_2$CuLi</td>
<td>n, p</td>
<td>3-10</td>
<td></td>
</tr>
<tr>
<td>Zn</td>
<td>n, p</td>
<td>20</td>
<td>non coherent, solid solutions</td>
</tr>
<tr>
<td>Al$_3$Li</td>
<td>c</td>
<td>&gt;50</td>
<td>coherent ordered, depending on coherency stress, $\gamma_{APB}$</td>
</tr>
</tbody>
</table>
Figure 8: Geometrical features and particle-dislocation interactions for dispersoid microstructures

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2. C. Kammer, "Success for aluminium thanks to 75 years of materials research", *Aluminium*, 75 (1999) 753-775
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ALUMINUM ALLOYS

The Role of Ledge Nucleation/Migration in Ω Plate Thickening Behaviour in Al-Cu-Mg-Ag Alloys

C.R. Hutchinson, X. Fan, S.J. Pennycook and G.J. Shiflet

Pgs. 13-23
THE ROLE OF LEDGE NUCLEATION/MIGRATION IN Ω PLATE THICKENING BEHAVIOUR IN Al-Cu-Mg-Ag ALLOYS

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3Solid State Division, Oak Ridge National Laboratory, Oak Ridge, TN, 37831, USA.

Abstract

The thickening kinetics of Ω plates in an Al-4Cu-0.3Mg-0.2Ag (wt. %) alloy have been measured at 200 °C, 250 °C and 300 °C using conventional transmission electron microscopy techniques. At all temperatures examined the thickening showed a linear dependence on time. At 200 °C the plates remained less than 6nm in thickness after 1000h exposure. At temperatures above 200 °C the thickening kinetics are greatly increased. Atomic resolution Z-contrast microscopy has been used to examine the structure and chemistry of the (001)_Ω || (111)_α interphase boundary in samples treated at each temperature. In all cases, two atomic layers of Ag and Mg segregation were found at the broad face of the plate. The risers of the growth ledges and the ends of the plates were free of segregation. No significant levels of Ag or Mg were detected inside the plate at any time. The necessary redistribution of Ag and Mg accompanying a migrating thickening ledge occurs at all temperatures and is not considered to play a decisive role in the excellent coarsening resistance exhibited by the Ω plates at temperatures up to 200 °C. Plates transformed at 200 °C rarely contained ledges and usually exhibited a strong vacancy misfit normal to the plate. A large increase in ledge density was observed on plates transformed at 300 °C, concomitant with accelerated plate thickening kinetics. The high resistance to plate coarsening exhibited by Ω plates at temperatures up to 200 °C, is due to limited ledge nucleation under these conditions. The prohibitively high barrier to coherent ledge nucleation on the broad faces of plates aged at 200 °C arises from the contribution to the total free energy change attending nucleation from elastic interactions between the misfitting coherent ledges and the significant strain field that can exist normal to the broad face of the Ω plate.

Acknowledgments

CRH and GJS greatly acknowledge the support of the Southeastern Universities Research Association 1999 Summer Cooperative Research Program and the National Science Foundation under grant number DMR-9904034. The work at Oak Ridge National Laboratory was supported by the Division of Materials Sciences, US Department of Energy under contract No. DE-AC05-00OR22725 with UT-Battelle, LLC. Dr. Simon Ringer of the Electron Microscopy Unit at the University of Sydney, Australia is thanked for kind provision of materials used in this study.

Lightweight Alloys for Aerospace Applications
Edited by Kumar Jata, Eui Whee Lee, William Frazier and Nack J. Kim
TMS (The Minerals, Metals & Materials Society), 2001

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1. Introduction

The addition of trace amounts of Ag to Al-Cu-Mg alloys with high Cu:Mg ratios (e.g., 10:1) significantly alters the precipitation sequence usually observed in these alloys [1-4]. The most notable change is the appearance of a thin, hexagonal-shaped phase, designated Ω, that forms as platelets on the \{111\}_\alpha slip planes of the matrix. Several structures for the Ω phase have been proposed [5-7] although the most widely accepted structure is orthorhombic (Fmmm, a=0.496nm, b=0.859nm, c=0.848nm) [8, 9]. The orientation relationship between Ω and the α matrix is (111)_α || (001)_Ω and [110]_α || [010]_Ω. The appearance of the Ω phase promotes greater hardening, and alloys based on the Al-Cu-Mg-Ag system have shown promising creep properties at temperatures up to 200 °C because of the apparent resistance of the Ω phase to particle coarsening [10]. The reported coarsening resistance of Ω has been confirmed by Ringer et al. [11]. Those researchers directly measured the changes in plate thickness using conventional transmission electron microscopy (CTEM) techniques as a function of time for temperatures between 200 °C and 300 °C. The plates examined in that study remained less than 6nm in thickness after 1000h exposure at 200 °C. At temperatures above 200 °C the thickening was greatly accelerated.

It is now generally accepted that rationally oriented plate-like precipitates thicken by a ledge mechanism [12] (Fig. 1). The thickening of plate-like precipitates therefore depends on the kinetics of the nucleation and growth of thickening ledges on the broad faces of the plates. Previous studies on precipitate thickening kinetics in Al-alloys [13-15] have concluded that the overall thickening kinetics are ultimately restricted by limited ledge nucleation.

Ω plates differ from other \{111\}_α precipitate plates in at least two important aspects. The first concerns the relatively large lattice misfit between the precipitate and the matrix that exists normal to the (111)_α || (001)_Ω interphase boundary. Other examples of \{111\}_α plates include T_1 (Al2CuLi) in Al-Cu-Li alloys, η' (MgZn2) in Al-Zn-Mg and γ' (γ) (AlAg2) in Al-Ag. Each of these phases has a hexagonal structure whereas Ω is usually assumed to be hexagonal. The misfit normal to the precipitate for each of these phases is 0.12% for T_1, 0.03% for η', 1.46% for γ' and 9.3% for Ω. The misfit normal to the Ω plate is accordingly considered large. On the basis of this large misfit, Fonda et al. [16] initially postulated that the source of the enhanced thermal stability of Ω may be the relationship between ledge nucleation and propagation and the elastic strain field. Fonda et al. [16] investigated the accommodation of misfit strain surrounding Ω plates and found the plates consistently exhibit a vacancy type strain field normal to the habit plane (Fig. 2), independent of plate thickness. Two types of thickening ledges were observed, coherent 1/2Ω unit cell high ledges and less commonly, larger ledges which contain a misfit compensating dislocation of the type \( b=1/3<111>_\alpha \). Similar dislocations were also
observed at the ends of the plates with an average spacing of 2 1/2 or 3Ω unit cells, which produces a minimum strain normal to the plate.

The second characteristic that differentiates Ω from other {111}_α plates is the well reported segregation of Ag and Mg to the broad face of the Ω plate. This segregation was first detected by Muddle and Polmear [9] and most recently by Reich et al. [17] using 3D-APFIM. The 3D-APFIM work of Reich et al. has provided evidence to warrant careful consideration of the usual assumption that ledge nucleation controls the overall plate thickening rate in Al-based alloys. In Reich et al.'s atom probe study, they captured an Ω plate thickening ledge in an Al-1.9Cu-0.3Mg-0.2Ag (at. %) alloy aged 10h at 180 °C. Their observations show the presence of a monoatomic layer of Ag and Mg at the Ω plate/matrix (001)_Ω || (111)_α interface but no Ag or Mg was detected within the plate itself or at the riser of the ledge. The motion of the thickening ledge must then involve the simultaneous flux of Cu from the matrix to the riser of the ledge and the redistribution of Ag and Mg from the original broad face of the Ω plate to the terrace of the migrating thickening ledge. Fig. 1 is a schematic illustration of this process1. This complicated diffusion geometry raises two interesting questions. (a) What interaction (if any) is there between the redistributing Ag and Mg and the incoming flux of Cu? and (b) If an interaction is expected, could it be sufficient to retard ledge migration to the point where it becomes the rate controlling process for plate thickening instead of ledge nucleation?

![Figure 2](image-url)

Figure 2. Schematic illustration of (a) vacancy and (b) interstitial strain fields normal to the broad face of a precipitate plate.

![Figure 3](image-url)

Figure 3. Mean Ω plate thickness (nm) as a function of time at 200 °C, 250 °C and 300 °C, in an Al-4Cu-0.3Mg-0.4Ag (wt. %) alloy.

The present work addresses the need for a systematic study of the structure and chemistry of the Ω plate/matrix (001)_Ω || (111)_α interface as a function of time and temperature to examine the respective roles of ledge nucleation and migration in accounting for the excellent coarsening resistance of Ω plates at temperatures up to 200 °C.

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1. The diffusion path for Ag and Mg redistribution from the broad face of the plate to the terrace of the migrating thickening ledge shown in Fig. 1 is only one of several possible diffusion paths. This schematic is not intended to imply that this is the path of solute redistribution, only that some interaction between the flux of Cu and the Ag and Mg may be expected.
2. Experimental Procedure

The composition of the alloy used is Al-4.0Cu-0.3Mg-0.4Ag (wt. %). Strips of material 0.5-1mm thick were solution treated (ST) at 525 °C for 1h, water quenched (WQ) and aged in molten salt baths at 200 °C ± 2°C, 250 °C ± 2°C or 300 °C ± 2°C for various times up to 1000h.

Specimens for transmission electron microscopy (TEM) were punched mechanically from the strips and twin-jet electrolytically polished in a solution of 33 vol. % nitric acid and 67 vol. % methanol at -25 °C. The microstructural evolution was monitored using CTEM techniques with a 200 kV microscope. High resolution phase contrast microscopy was performed using a top entry HREM operating at 400 kV. The high resolution phase contrast simulation was performed using the Crystalkit and MacTempas software packages [18]. Atomic resolution Z-contrast microscopy [19, 20] was used for the systematic examination of the composition and structure of the Ω plate/matrix (001)Ω // (111)α interface as a function of time and temperature. This technique is capable of providing two-dimensional intuitively interpretable images of atomic structures with compositional sensitivity without the need for model structures and simulations associated with the phase contrast imaging techniques. A Z-contrast image is formed by scanning an electron probe of atomic dimensions across a specimen and collecting the high angle scattered electrons with an annular dark-field (HAADF) detector. Since the scattering is incoherent at high scattering angles, the image is essentially a map of the total scattering intensity of each atomic column, which is approximately proportional to the square of the atomic number (Z). This technique is especially well suited to the investigation of Ag in the Al-Cu-Mg-Ag system due to the relatively high atomic number of Ag. The microscopy was performed using a VG Microscope HB603U scanning transmission electron microscope operating at 300kV which is capable of forming an electron probe size of 0.126nm. The EDS analysis was carried out using a ATEM operating at 200 kV equipped with a field emission gun and an EDSX system.

Measurements of the thickness of Ω precipitate plates were made from CTEM micrographs recorded with the electron beam parallel to the precipitate habit plane (i.e. parallel to <112>α/ [100]Ω or [110]Ω). In each case, the "edge-on" thickness of between 70 and 100 precipitates was measured from the negatives magnified using a 4x graticule.

3. Results

3.1 Conventional Transmission Electron Microscopy (CTEM)

Observations of the <100>α, <110>α and <112>α zone axes of the Al matrix were made to ensure a true representation of the precipitate distribution was obtained. At all times and temperatures examined, the Ω phase was found to be present. At 200 °C and 250 °C, the Ω phase co-exists with Θ' (Al2Cu) and S (Al2CuMg) phases. At 300 °C, the Ω phase was the only precipitate found at all times observed. The thickening kinetics of Ω plates were measured at each temperature. A plot of plate thickness as a function of time is shown in Fig. 3. At all temperatures examined the precipitate thickness shows a linear dependence on time. At 200 °C, the Ω plates reach a thickness of approximately 5.5nm after 100h exposure, after which there is no detectable change in average thickness. At 300 °C the rate of thickening is rapid and thicknesses greater than 30nm are reached within 50h at 300 °C. At 250 °C, thicknesses of 25-30nm are obtained after 1000h exposure. These observations are qualitatively consistent with those of Ringer et. al. [11].
3.2 High Resolution Electron Microscopy

3.2.1 Z-Contrast Microscopy

Atomic resolution Z-contrast microscopy was used to examine the structure and chemistry of the Ω plate/matrix (001)\textsubscript{Ω} \parallel (111)\textsubscript{α} interface in samples transformed at each temperature.

A low magnification Z-contrast image of an Ω plate (right) and a Θ' plate (left) is presented in Fig. 4(a). The intensity in a Z-contrast image is approximately proportional to \( Z^2 \) and the bright bands bounding each side of the Ω plate are interpreted as preferential segregation of at least Ag to the Ω plate/matrix (001)\textsubscript{Ω} \parallel (111)\textsubscript{α} interphase boundary, consistent with previous investigations [9,17]. Ω plates in this orientation (Fig. 4(a)) were found to be very long, straight and typical of Ω plates observed in samples transformed at 200 °C. An atomic resolution Z-contrast image of a 4 unit cell thick Ω plate is shown in Fig. 4(b). Two atomic layers of enhanced intensity are seen at the Ω plate/matrix (001)\textsubscript{Ω} \parallel (111)\textsubscript{α} interface. These correspond to two layers of segregation. This is in contrast to the monoatomic layer reported by Reich et. al. [17]. The layers of enhanced intensity within the plate parallel to the habit plane are separated by 0.424nm and correspond to layers enriched in Cu. This is qualitatively consistent with the projection of the proposed orthorhombic Ω structure down zone axes parallel to the habit plane. EDS was used to determine that the interfacial segregation contained both Ag and Mg, consistent with recent analytical investigations [17]. No significant quantities of Ag or Mg were detected within the Ω plate or the adjacent matrix.

![Figure 4](image)

**Figure 4.** Z-contrast images of sample transformed 100h at 200 °C. (a) Low magnification image of Ω plate (right) and Θ' plate (left). (b) Atomic resolution image of an Ω plate illustrating two atomic layers of interfacial segregation.

Fig. 5(a) is a Z-contrast image of an Ω plate thickening ledge in a sample exposed for 70h at 250 °C. The ledge is 1/2 Ω unit cell high and coherent with the matrix. The image shows a double layer of interfacial segregation to the terraces of the growth ledge but not at the riser of the ledge. The lack of segregation at the riser of the thickening ledge is consistent with the APFIM observations of Reich et. al. [17]. Energy dispersive spectra were obtained from the matrix, the (001)\textsubscript{Ω} \parallel (111)\textsubscript{α} interphase boundary and wholly within the Ω plate. They showed segregation of both Ag and Mg to the interface. As was the case for the sample treated at 200 °C, no significant quantities of Ag or Mg were detected within the Ω plate or the adjacent matrix. At 300 °C the Ω plates thickened at a greatly enhanced rate, and the Z-contrast image in Fig. 5(b) shows thickening ledges are plentiful on Ω plates transformed at this temperature. Two atomic layers of segregation to the Ω plate/matrix (001)\textsubscript{Ω} \parallel (111)\textsubscript{α} interface were again observed. EDS analysis