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THE AGING CHARACTERISTICS OF ALUMINUM ALLOYS
ELECTRON TRANSMISSION STUDIES OF
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The Aging Characteristics of Aluminum Alloys

Electron Transmission Studies of

Al-Mg-Si Alloys

G. Thomas* (Ordinary Member)

ABSTRACT

Transmission electron microscope studies of an Al-Mg-Si alloy using the thin foil technique have revealed many interesting features in the quenched and aged conditions. The concentration of vacancies annealed out to form dislocation loops in quenched specimens was about $3 \times 10^{-6}$, and the loop density about $10^{14}/\text{cm}^3$. The zones which form first upon aging were resolved as needles on $\{110\}$ with $\langle 100 \rangle$ growth directions; their density was about $2-5 \times 10^{15}/\text{cm}^3$. Prolonged aging revealed the aging sequence as a continuous change: needles-ropes-plats; the rods being an intermediate form of the equilibrium plates of $\text{Mg}_2\text{Si}$. It is suggested that the change zones-ropes occurs as a result of Mg-Si-vacancy groups diffusing to zones and that the final transformation to $\text{Mg}_2\text{Si}$ is diffusionless. Age hardening in this alloy is due chiefly to chemical effects.

INTRODUCTION

The use of the thin foil technique for transmission electron microscopy has already proved to be a valuable method for studying the morphology of aging reactions in light alloys$^{(1-3)}$ and is particularly superior

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to the average diameter is, however, about 100Å. The average density of dislocation loops was determined from many micrographs and was found to be about $10^{14}$/cm$^2$ assuming a foil thickness of 1000Å. If these loops are formed as a result of the condensation of vacancies into collapsed discs, the required vacancy concentration (equal to $\pi r^2 bN$ where $r$ is the average loop radius, $b$ the Burgers vector and $N$ the number of loops per cm$^3$), to produce these lattice defects works out to be about $3 \times 10^{-6}$. Quenching experiments on pure aluminum$^{(16,19)}$ have shown that the concentration of vacancies is $10^{-4}$ after quenching from 550°C and that these have all gone towards the formation of loops$^{(16)}$. This now leads to two possibilities in regard to the Al-Mg-Si alloy where a lower annealed-out vacancy concentration has been found, viz. (i) not all the vacancies have gone to form loops, or (ii) there is a lower vacancy concentration in the ternary alloy than in the pure metal. However, other quenching experiments on binary aluminum alloys$^{(17)}$ have shown that the concentration of quenched-in vacancies actually increases with alloying (e.g., in dilute Al-Cu alloys concentrations of $10^{-3}$ were found$^{(17)}$. This indicates that the major fraction of vacancies are retained in solution, probably as a result of interaction with solute atoms. Because vacancies have strain energy as well as surface energy, it is to be expected that they would be attracted to regions of strain in the lattice, e.g., to solute atoms of different size from solvent atoms. In the alloy used in this investigation, magnesium atoms are larger and silicon atoms are smaller than the parent aluminum atoms, so that it is only those vacancies in supersaturation and free from solute atoms which can condense, collapse, and form dislocation loops upon quenching. If this is
so, then since the concentration of annealed-out vacancies is of the order of $3 \times 10^{-6}$ (neglecting those condensed on incipient dislocations), and as it is likely that a total concentration of at least $10^{-4}$ are quenched in, it is concluded that most of the vacancies remain in solution after quenching Al-Mg-Si alloys.

An interesting feature which was observed in quenched foils was that loops tended to coalesce together. Some isolated examples of this are shown in A in Fig. 1. There were no anomalous features (i.e., streaks or extra spots) observed in the diffraction patterns taken from quenched samples, indicating zones were not present immediately after quenching. It was not possible to determine what planes the dislocation loops formed in because they were very small and had irregular shapes (Fig. 1).

Precipitates were sometimes observed at the grain boundaries in the quenched samples but were more readily resolved after a slight amount of aging, e.g., Fig. 2. It is observed that these particles lie mainly in the regions adjacent to the grain boundary rather than in the boundary plane. This form of grain boundary precipitation is a unique feature in Al-Mg-Si alloys and has not been observed in thin foil studies of other light alloys\(^1\).

Foils exhibiting this form of precipitation were examined in an R. C. A. electron diffraction camera. Patterns were obtained in which there were a few spots matching the d-spacings of randomly oriented silicon. Owing to the diffuseness of the diffraction spots however, it is
not certain that this evidence is conclusive. However, similar results have been obtained by Stumpf (private communication) so that the precipitates in fig. 2 are tentatively identified as pure silicon. There was no evidence for precipitation of Mg2Si.

2. Aged Alloys

The first evidence of a change in the supersaturated solid solution was obtained from the electron diffraction patterns. Very faint streaks were observed parallel to the [100] and [010] Al directions with the origins at 110 Al, as shown in fig. 3. The pattern is redrawn in fig. 4 for the sake of clarity. From this it can be seen that the habit planes are 110 with <100> growth directions. These are forbidden reflections for a f.c.c. lattice and could correspond to a superlattice structure of small dimensions with axes perpendicular to the electron beam, i.e., in <100> orientation. This diffraction effect shows that a change in composition of atoms in the 110 planes of the lattice has occurred, and thus corresponds to the formation of zones in this alloy (20). This was observed after one hour at 71°C, twenty-four minutes at 149°C and five minutes at 204°C. The streaks were not observed in specimens aged above 204°C. At longer aging times at lower temperatures, the streaks became more intense (but still very much weaker than the matrix reflections) but remained continuous through the [110] reciprocal lattice points. This was interpreted in terms of further growth of zones in <100> with little or no increase in thickness. An example of the microstructure corresponding to these diffraction effects after the 204°C aging treatment, is shown in fig. 5. In this, one observes thin dark lines parallel to [100] and [010] which are revealed only when the foil is exactly in the [100] or [110] orientations. Thus the resolution of these effects is extremely
difficult and sharp pictures can be obtained only from very thin foils viewed exactly in the right orientation. There were no contrast effects around these precipitates as has been observed in Al-Cu alloys\(^2\), so that it is concluded that these precipitates are exactly coherent with the matrix with only small elastic coherency strains involved. It is unlikely that zones of such small dimensions are noncoherent. The streaks on the diffraction patterns indicate that the zones could be plates or needles. A close examination of fig. 5 shows that there are black spots, e.g., at A, of diameter 60Å corresponding to the width of the black lines at B, and appear to be needles viewed end-on. If this is so, then there should be about one-half as many spots as lines in micrographs taken from foils in \([001]\) orientation since the three \(<001>\) orientations are equally frequent. Estimates of the number of spots and lines in fig. 5 (and other similar areas) shows that this is the case. This must mean that the zones in Al-Mg-Si Alloys are needle-shaped and that the spots in fig. 5 are needles lying normal to the foil surface along \([001]\). The long dimensions of the needles vary from about 200Å to over 1000Å as the aging time and temperature is increased but the diameter of the needles stays fairly constant at about 60Å. The average density of zones was found to be about \(2 - 5 \times 10^{15} / \text{cm}^3\), and they were always uniformly distributed throughout the alloy.

At aging temperatures above 200°C the needles were observed to change into rods by an increase both in their length and diameter whilst the density decreased to \(\sim 10^{13} / \text{cm}^3\). At the same time the diffraction patterns changed from a system of streaks to a cross-grid pattern of spots. Figure 6 shows an early stage in the formation of rods. The three \(<100>\) orientations are clearly visible; the foil orientation is
approximately [311] and rods in [100] are inclined at 25° to the foil surface whilst those in [010] and [001] are inclined at 72° to the surface. This proves conclusively that the second stage of aging corresponds to the formation of rods and not platelets. The dotted contrast along the precipitates in [010] and [001] is due to an electron interference effect.

At higher aging temperatures the rods thicken to about 1000Å diameter, e.g., as shown in Fig. 7 in which the black spots are rods observed end-on in the [001] direction. From the cross grid spot patterns obtained by selected area electron diffraction the orientation relationship was found to be <100> Al//<110> rod; a result which was previously known from x-ray investigations (9) (10). The diffraction patterns corresponding to the rods were found to be consistent with a f.c.c. lattice in which \( a = 6.42 \pm 0.07\,\text{Å} \).

Since the equilibrium phase \( \text{Mg}_2\text{Si} \) is f.c.c. (CaF$_2$ structure) with \( a = 6.39\,\text{Å} \), the rods can be considered as an intermediate \( \text{Mg}_2\text{Si} \) phase which has a structure corresponding to a highly ordered unit cell of \( \text{Mg}_2\text{Si} \). The formation of \( \text{Mg}_2\text{Si} \) can thus occur directly from the zones and will be discussed in more detail later.

At this stage the grain boundary precipitates are concentrated mainly in the plane of the grain boundary, e.g., Fig. 8, and in which the precipitates are still predominantly silicon. With prolonged aging, however, they change to \( \text{Mg}_2\text{Si} \). Figure 8 shows a structure in which there are rods apparently oriented at random. Some of these have bent shapes. It was not possible to distinguish between these rods and the rods lying along <100> and there were no extra spots in the diffraction patterns, so that this effect remains unexplained. In Fig. 8 the grain A is in poor
contrast, and this demonstrates the necessity for orienting the foil into a strong diffracting position in order to obtain good contrast.

The equilibrium $\text{Mg}_2\text{Si}$ phase was observed to form after three hours at $260^\circ\text{C} (500^\circ\text{F})$. The structure after this time is predominantly the intermediate phase, e.g., fig. 9, but at (A) small plates of $\text{Mg}_2\text{Si}$ were observed. Figure 9 is taken from an oxide replica—the $\text{Mg}_2\text{Si}$ particles are not oxidized appreciably, so they may remain in the oxide film*. In most cases the $\text{Mg}_2\text{Si}$ plates grow out of the intermediate rod structure, e.g., at (A) fig. 9, but occasionally others seem to nucleate independently, e.g., at (B). Aging for 2-1/2 hours at $316^\circ\text{C} (600^\circ\text{F})$ resulted in the formation of large plates of $\text{Mg}_2\text{Si}$. Figure 10 shows clearly how the plates develop chiefly from the rods by growth in a direction normal to the length of the rod. This process is accompanied by dissolution of nearby rods, e.g., at (A).

Precipitation was never observed to occur on dislocations (e.g., see fig. 7 at A) and at all stages of aging the precipitates were uniformly distributed throughout the matrix. This means that incipient dislocations and dislocation loops have no effect on the morphology of precipitation in this alloy, whereas in Al - Cu and Al - Ag alloys preferential precipitation on dislocations is commonly observed$^{(1)}$.

Dislocations were observed to move through the precipitates with apparently little or no difficulty. Figure 11 shows dislocation traces in the same area as that shown in fig. 6 after the foil had been exposed to a fine focus beam ~10 $\mu$ diameter for 2 - 3 minutes. Unfortunately, after this time, owing to the deposition of carbonaceous material on the foil surface, the resolution is considerably worsened

*The replicas are better than the thin foils at this stage because the particles tend to be leached out of the foils during preparation. This fact leads to the conclusion that the formation of $\text{Mg}_2\text{Si}$ involves a complete loss of coherency.
so that it is not possible to clearly recognize the precipitates, except possibly at A. However, it can be seen that the slip traces are continuous throughout the length of the grain and that there is no cross-slip. This shows that the dislocations must cut through all the precipitates which intersect the glide plane.

SUMMARY OF RESULTS

1. In quenched alloys the concentration of vacancies annealed out to form dislocation loops was found to be \( \sim 3 \times 10^6 \) indicating that a large fraction of vacancies remain in solution. The average number of loops was \( \sim 10^{14} / \text{cm}^3 \).

2. In aged alloys the sequence of decompositions of the supersaturated solid solution depended on the aging temperature and was found to be continuous and of the form:

   Zones \( \rightarrow \) intermediate \( \text{Mg}_2\text{Si} \) \( \rightarrow \) equilibrium \( \text{Mg}_2\text{Si} \)

   The zone stage exists only at temperatures below 220°C.

3. The average number of zones was \( 2 - 5 \times 10^{15} / \text{cm}^3 \).

4. The zones can be resolved only in thin foils orientated such that one of the \( <100> \) axes lies perpendicular to the incident electron beam.

5. The orientation relationships found from electron diffraction confirmed the x-ray results of other workers\(^{(9)}\) \(^{(10)}\) and were:

   (a) \( <100> \) needles parallel \( <100> \) matrix in a superlattice structure. The habit planes of needles are the \( \{110\} \) matrix planes.

   (b) \( <110> \text{Mg}_2\text{Si} \) parallel \( <100> \) matrix.

6. The intermediate \( \text{Mg}_2\text{Si} \) is f.c.c. and may correspond to a superlattice of the equilibrium \( \text{Mg}_2\text{Si} \).
(7) Precipitation near the grain boundaries follows the sequence:

pure silicon adjacent to boundaries → Mg₂Si in boundaries.

DISCUSSION

1. The Aging Sequence

In the present electron microscopic investigation the new features observed during the quenching and aging of an Al-Mg-Si alloy were (1) the resolution of the quenched structure, (2) the resolution of the zone stage, and (3) the morphology of the matrix and grain boundary precipitates. As a result of obtaining this information it is possible to consider the influence of quenching on subsequent precipitation during aging. In the first stages, i.e., zone formation, the needles nucleate on \( \{110\} \) planes and grow along \(<100>\).

Guinier and Lambot \(^{10}\) suggested that because of the ionic nature of the Mg₂Si phase, zones formed prior to precipitation of Mg₂Si should consist of atom rows along \(<100>\) such that one row of silicon atoms is bounded by two rows of magnesium atoms. Looking into this picture more closely and considering what sort of strains arise from such a zone, it is found that the least possible elastic strains occur if the zones (or needles) are in fact composed of atom rows in the ratio one \(<100>\) silicon to two \(<100>\) magnesium. This can be seen from the following: the atomic diameters (distance of closest approach) of aluminum, silicon and magnesium atoms are 2.862Å, 2.351Å and 3.196Å respectively. Assuming the atoms in zones occupy the same space as they do normally, then a single layer zone of three atoms wide, i.e., one silicon and two magnesium rows, would thus occupy a width of

\[
2 \times 3.196 + 2 \times 2.351 = 8.743\text{Å}, \text{ and this would replace three rows of aluminum atoms in which the width would be } (3 \times 2.862) = 8.586\text{Å}. \text{ This}
\]
means the formation of a needle along \langle100\rangle on \{110\} involves an expansion of about two percent. The needles are thus in a state of cylindrical compression. Fitting in other possible combinations of Mg:Si ratios it can easily be shown that a ratio of 2 Mg:1 Si involves the least amount of elastic strain. This argument only holds if the binding in the zone is metallic and not ionic. The arrangement of planes in the zone would then appear as a superlattice as shown in Fig. 12a and b. The coherency strains due to the zones are illustrated schematically in Fig. 12b.

The formation of the disc-shaped G. P. \{1\} zones in Al-4% Cu alloys involves elastic strains corresponding to about a 10% contraction also along \langle100\rangle directions normal to the plane of the discs, i.e., \{100\}. These strains have been observed to show up as contrast effects in thin foils\(^2\). The fact that no such effects were observed in the present work, even though careful tilting of the foils was done, may indicate that the estimated 2% expansion associated with the needles is too low a strain to show up any contrast effects in the electron micrographs. The magnitude of elastic strains is thus important in thin foil work where many features are revealed only as a result of coherent scattering of electrons by the strain field around the feature being examined (e.g., dislocations, phase interfaces, etc.), and this alone may set a limit on resolution. This might explain why it was not possible to resolve needles thinner than 50Å.

The electron diffraction results (see Fig. 3) show that the habit planes of the needles are \{110\}, which are also the most highly favored planes on which needles can be nucleated, since they are the least
close-packed in the f. c. c. lattice. Since each \{110\} plane contains only one \langle100\> direction, e. g., (011) has \{100\}, (101) has [100] and (110) has [001], only three orientations of zones parallel to \langle100\> are possible. Thus once a zone is nucleated on any one \{110\} plane, it can grow in only one of the \langle100\> directions. The resultant precipitation pattern is Widmanstatten, as demonstrated in Figs. 5-8. The reason for \langle100\> being preferred growth directions may be inferred from the model proposed in Figs. 12a and b. In 12 (a), a section is shown containing two (011) planes for the 2 Mg: 1 Si atom rows in the zone. The open squares and circles represent atoms in the next plane above or below those represented by closed squares and circles. The growth direction in this case is [100], i.e., along AA and BB (Fig. 12b); the zone is in a state of cylindrical compression as indicated by the shape of the surrounding matrix planes (shown as broken lines). Thus the 'interfaces' AB would be the most preferred places for atoms to become attached. The other possible growth directions are those normal to AA and BB, i.e., [011], but these are close packed directions already, so that it is unlikely that the zones would expand much radially, as has been shown by the experimental results. This could mean that the zone diameters are fixed by the size of the initial nuclei.

It is interesting to note that the zones are uniformly distributed throughout the matrix and no precipitation was observed on loops or dislocation lines. This is consistent with previous results (1-3) and shows that the zones are nucleated as a result of the movement of excess vacancies retained by quenching after solution heat-treatment and occurs independently of the dislocation substructure in the alloy.
If the atoms in solution are attached to vacancies, as seems highly likely, then vacancy-assisted diffusion of solute atoms would be expected to occur readily towards the positions at AB (Fig. 12 b) because vacancies would be attracted to these regions since they are in compression. Vacancies could then be lost at the zones themselves, thereby relieving the internal strains. This process can continue only as long as vacancies and solute atoms are available for diffusion. The growth rate should then stop when all the vacancies are lost in the precipitate or at other sinks. The final stage in diffusion is thus the formation of the rod structure. The electron micrographs and diffraction patterns showed that the growth of zones into rods seems to be an intermediate step in the change; zones - Mg₂Si. This change is thus continuous and the equilibrium phase can readily be produced by only minor atom movements within the zones as illustrated in Figs. 12 c and d. This shows that the final transformation is diffusionless. In Fig. 12 c a section of Mg₂Si is drawn along two adjacent (011) planes. The Mg₂Si phase is an ionic structure of the CaF₂ type. By comparing Figs. 12 a and c, it can be seen that the Mg₂Si structure can be produced by silicon atoms in positions 1 moving to 1' and magnesium atoms in positions 2, 3 moving to 2', 3' respectively. Alternatively it is only necessary for magnesium atoms at 2 and 3 to move to a halfway position in [100] thereby dropping into position to form the center of a tetrahedron between silicon atoms in the two (011) planes. This latter mechanism then requires only a small readjustment of silicon atoms. Either process results in a CaF₂ type structure. This change then brings the magnesium and silicon rows along [100] into equidistant positions with respect to each other (Fig. 12d).
It will be seen from Fig. 12 that the transformation of zones to Mg$_2$Si involves a contraction. At first sight this seems to be a real difficulty because the \{110\} planes of the zones are already close packed. However, if it is assumed that the binding between atoms in the zones (and rods) is metallic, then the number of nearest neighbors remains at 12 as in the surrounding matrix, whereas in the ionic structure of Mg$_2$Si, the magnesium atoms have only four nearest neighbors, and silicon eight. Furthermore, since magnesium atoms have a much smaller diameter in the ionic state, then the Mg$_2$Si structure occupies less space than the zones and a contraction is therefore to be expected.

The Mg$_2$Si phase has been observed to form as plates which grow only by dissolution of nearby precipitates (e.g., at A, Fig. 10). This means that there are not enough solute atoms left in supersaturation in the matrix to cause further growth once they are formed, and this supports the view that the final phase transformation is diffusionless. The growth of Mg$_2$Si as plates will occur to produce the minimum increase in strain energy. Figure 12c shows that the restricted direction is that normal to the (011) faces as is expected, because atoms are more easily attached along [100] and [011] than along [011]. The micrographs (Figs. 9 and 10) show that [100] and [011] are in fact the growth directions.

To sum up, the aging process in this alloy can be considered as a sequence involving (1) elimination of free vacancies into collapsed discs producing dislocation loops with simultaneous formation of zones, (2) growth into rods by vacancy assisted diffusion of solute atoms to zones with simultaneous elimination of associated vacancies at the precipitates, and (3) transformation to the equilibrium Mg$_2$Si structure.
Finally, it is interesting to note that in aluminum alloys where there is a large size factor difference between solute atoms and aluminum atoms, the zones are never spherical and the preferred directions of precipitation are \(<100>\) with \([100]\) or \([110]\) habit planes, e.g., Al-Cu and Al-Mg-Si, whereas in cases where there is only a small size factor difference, the zones are spherical and subsequent precipitation occurs on \([111]\) matrix planes, e.g., Al-Ag, Al-Zn, Al-Mg-Zn\(^1\). This confirms Guinier's suggestion made in 1949\(^4\).

Thus, although much is now known about zone formation in light alloys, more detailed information is required on the role of vacancies in nucleating zones and the effects of vacancy-solute atom interactions in any subsequent diffusion progress.

2. **Hardening**

Although no age-hardening data was obtained in the present work, Stumpf (private communication) has shown that maximum hardening in this alloy occurs when it is aged to produce the needle structure. Since there appears to be only a small elastic interaction at the needle-matrix interface, it is unlikely that coherency strains play an important part in hardening. Since dislocations have been observed to cut through the precipitates during glide, the chief resistance to dislocation movement must arise from the gain in chemical energy which is involved in the intersection process, as this would result in the breaking of Mg-Si bonds. This mechanism has been extensively developed by Kelly and Fine\(^{22}\) and Kelly\(^{23}\) to account for zone hardening in Al-Cu and Al-Ag alloys.
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Fig. 1. Al-Mg$_2$Si alloy quenched after 2 hrs. 550°C, showing dislocation loops and networks. Note coalescence at A. X 30,000.
Fig. 2. Al-Mg$_2$Si alloy aged 5 mins. 204°C, showing precipitates adjacent to grain boundaries. X 45,000.
Fig. 3. Selected area electron diffraction pattern of Fig. 5 showing streaks in <100> from {110}. Foil orientation [001].
Fig. 4. Schematic representation of electron diffraction pattern shown in Fig. 3.
Fig. 5. Al-Mg$_2$Si alloy aged 4 hrs. 204°C. The three <100> orientations of needles are visible at A and B. The foil surface is (001). X 30,000.
Fig. 6. Al-Mg$_2$Si aged 5 hrs. 220°C; the intermediate Mg$_2$Si stage. Foil orientation [311]. Showing the three <100> orientations of rods and dislocations at A. X 40,000.
Fig. 7. Al-Mg$_2$Si alloy aged 5 hrs. 288°C. Foil orientation [001]; note rods seen end on, i.e., in [001], X 15,000.
Fig. 8. Al-Mg$_2$Si alloy aged 10 mins. 288°C. X 20,000.
Fig. 9. Oxide replica taken from Al-Mg$_2$Si alloy after aging 3 hrs. at 260°C. X 30,000.
Fig. 10. Oxide replica taken from Al-Mg$_2$Si alloy after aging 2-1/2 hrs. at 316°C showing development of plates of Mg$_2$Si from rods at A. X10,000.
Fig. 11. Same area as Fig. 6 after dislocations have been moved across the foil. X40,000.
Fig. 12a, b. Proposed model for zones in Al-Mg-Si alloys.
Fig. 12c, d. The equilibrium Mg₂Si structure formed from the atom movements shown in (a).
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