The Effect of Reversion Treatments on Precipitation Mechanisms in an Al-1.35 at. pct Mg_2Si Alloy

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The effect of reversion treatments on an Al-1.35 at. pct Mg_2Si alloy fully age hardened for 24 h at 160°C was studied by electron microscopy and tension tests. This alloy aged to full strength at 160°C did not show true reversion when heated 15 min at 200 to 300°C. The G.P. zones did not dissolve rapidly at a particular temperature but instead were replaced by the more stable phase, β' (the intermediate partially coherent form of Mg_2Si). After reheating the fully age hardened alloy 15 min at 250°C, a slight increase in strength was obtained, but the ductility was slightly lowered. Reversion treatments at higher temperatures (275 to 300°C) gradually decreased the strength of the alloy. Two simultaneous reactions are believed to occur during the reversion treatments: 1) the growth of some of the G.P. zones and the dissolution of others and 2) the formation of needles of β'.

ONE important aspect of the study of precipitation hardening in alloys is the reversion phenomenon. In general reversion may be defined as the resolution of G.P. zones or precipitates in an aged alloy by heating at a temperature above the original aging temperature but below the equilibrium solvus. According to their effect on precipitation mechanisms in alloys, reversion treatments can be divided into two groups:

Type 1: (True Reversion) With this reversion treatment G.P. zones dissolve in aged alloys at or above a metastable G.P. zone solvus in a short time without an excessive amount of intermediate, more stable precipitates being formed during the dissolution of the G.P. zones. Associated with the dissolving of the G.P. zones in this process is a rapid decrease in tensile strength.

For example, Herman and Fine[1] found that the reversion process for G.P.1 zones in an Al-1.7 at. pct Cu alloy was over in roughly $\frac{1}{2}$ min at 205°C. Graf[2] observed in an Al-1.7 at. pct Cu alloy that G.P.2 zones formed at 150°C redissolved completely on heating for 5 min at 250°C. Other reversion experiments have been made on Al-Zn alloys[3-5] and a G.P. zone solvus has been determined[4] which corresponds to the boundary above which the G.P. zones dissolve. Polmear[6] applied reversion treatments to a series of Al-Zn-Mg alloys and determined a G.P. zone solvus surface for this alloy system.

Type 2: With this reversion treatment G.P. zones dissolve much more slowly than those of Type 1 and simultaneous precipitation of an intermediate metastable phase occurs. That is, a more stable intermediate phase is formed before the resolution of the G.P. zones has been completed. For example, in Al-Ag alloys reversion treatments remove the hardening due to the formation of the G.P. zones but small platelets of γ' form simultaneously.[7]

Many experimental investigations have been carried out recently on Al-Mg-Si alloys[8-11] but little attention has been paid to reversion treatments. The general sequence of aging in a supersaturated solid solution of Mg_2Si in aluminum is well established:[12-17]

G.P. Zones (Shape = ?)	$\longrightarrow$	Intermediate Precipitate (Needles along ⟨100⟩ of the aluminum lattice)	$\longrightarrow$	Equilibrium Phase Mg_2Si (Platelets)

The morphology of the G.P. zones, however, is still a controversial matter since Cordier and Gruhl[18] believe them to be approximately spherical while other workers[10,16,17] believe them to be needle-like.

The purpose of this research was to investigate the effects of reversion treatments on an Al-1.35 at. pct Mg_2Si alloy by relating the tensile properties to the microstructures observed in the electron microscope.

EXPERIMENTAL PROCEDURE

A high-purity Al-Mg_2Si alloy consisting of 0.90 at. pct Mg and 0.45 at. pct Si (0.81 wt pct Mg and 0.47 wt pct Si) with copper and iron impurities less than 0.01 pct was used in this research. Cast ingots were homogenized at 570°C for 24 h, scalped and hot rolled to 0.10 in. slabs at 300 to 400°C. The 0.01 in. slabs were then cold rolled with intermediate anneals to the thickness required for each experiment.

1) Electron Transmission Microscopy

For these experiments thin foils were made by cold rolling and annealing until the metal was 0.003 in. thick. This material was then cut into thin strips and solution heat treated at 565°C in a furnace which was controlled to ±5°C. All samples were water quenched at 20°C and aged in a bath of Ucon heat transfer fluid which was controlled to ±1°C. The foil samples were thinned by standard techniques such as described by Hirsch *et al.*[19] Examination of the thin foils was made using an Hitachi HU-11E electron microscope operated at 100 kV.

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2) Tension Tests

Samples for the tension tests were made from blanks $2.0 \times 0.25 \times 0.025$ in. from which a 0.5×0.125 in. gage portion was machined. After heat treatment, the samples were tested for ultimate tensile strength and 0.2 pct offset yield strength using an Instron machine (0.1 in. per min). Elongation was measured using an Instron strain gage extensometer.

RESULTS

Isochronal Reversion Studies

(a) STRUCTURE AND TENSILE PROPERTIES OF THE FULLY AGED ALLOY

The ultimate tensile strength of the Al-1.35 at. pct Mg_2Si alloy was raised from an average of 17.2 ksi for the as-quenched samples after solution heat treatment to 34.9 ksi after single-stage aging for 24 h at 160°C. The latter heat treatment provided the highest strengths using a single-stage aging process.

Selected electron transmission micrographs of this alloy given the 24 h at 160°C aging treatments are shown in Fig. 1(*a*) and 1(*b*). The geometric shapes of the precipitates after this treatment are difficult to discern even at a magnification of 88,000 times. However, two types appear to predominate: one which is approximately spherical and another which is needle shaped.

The shape of the G.P. zones in Al-Mg_2Si alloys is a controversial topic. Cordier and Gruhl[18] determined from electron transmission microscopy that the G.P. zones in an Al-1.38 pct Mg_2Si alloy were approximately spherical and that their shape depended on the temperature and time of aging. However, earlier work by Lutts[16] led to the conclusion that the G.P. zones were needle-like and were formed in two stages. His experiments were made using diffuse X-ray scattering. However, Cordier and Gruhl[18] pointed out that because of the small differences in the scattering factors of magnesium and silicon in comparison with aluminum, it would be difficult to make a precise X-ray analysis of the formation of G.P. zones in the Al-Mg_2Si alloys. The results of this present work appear to agree more with Cordier and Gruhl's work in that the G.P. zones are believed to be spherical-like and the intermediate β' precipitate needle-like.

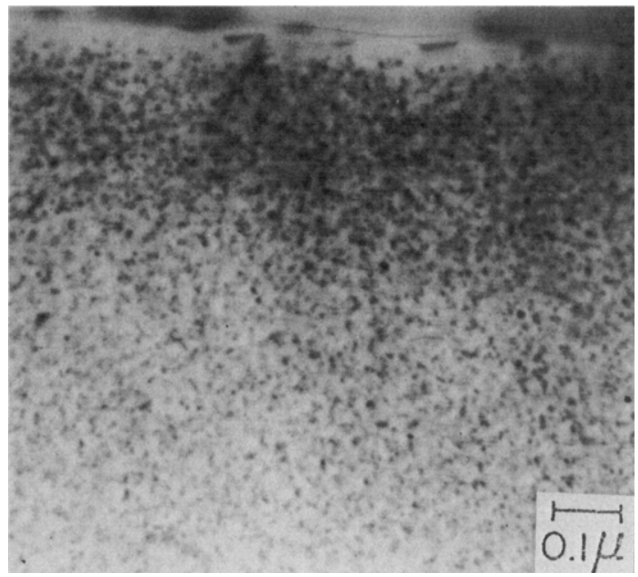
Although Al-Mg_2Si alloys are not known for having precipitate-free zones like Al-Zn-Mg alloys,[20] this alloy clearly shows one of about 0.1 μ. One of the earlier theories to explain the formation of these precipitate-free zones was the vacancy theory first presented by Taylor.[21] According to this theory the precipitate-free zones were formed because there was a low concentration of vacancies near the grain boundaries which would inhibit precipitation. As a result of more recent experiments, precipitate-free zones are now believed to be formed by a combination of a low concentration of vacancies *and* solute atoms in the region adjacent to the grain boundaries.[22]

(b) STRUCTURE AND TENSILE PROPERTIES OF THE FULLY AGED ALLOY AFTER REVERSION TREATMENTS

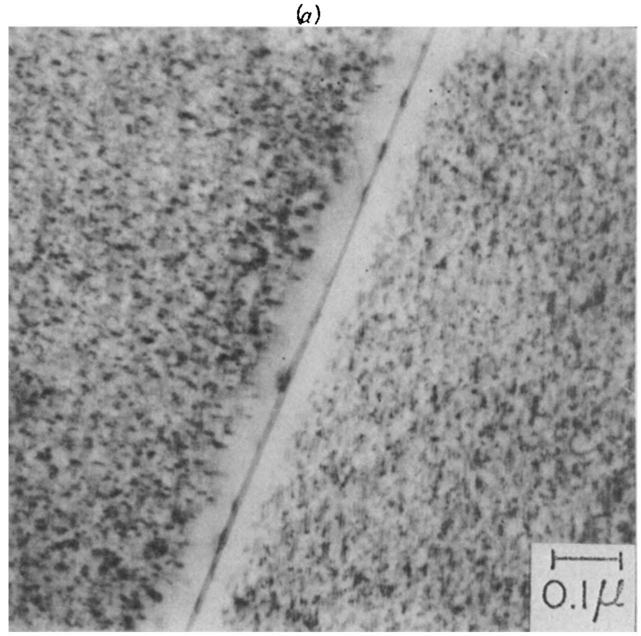
The effects of the reversion treatments on the fully aged alloy for 15 min at 200 to 300°C are shown graphically in Fig. 2. The effects of these treatments on the microstructure are shown in the electron transmission micrographs of Figs. 3 to 6.

When a reversion treatment of 15 min at 200°C is given to the alloy aged at 160°C, the ultimate tensile strength decreased slightly to 32.0 ksi and the ductility decreased to 6 pct (Fig. 2). The microstructure of the aged alloy after this heat treatment is coarsened as is shown in Figs. 3(*a*) and 3(*b*). Distinct needle-like precipitates are visible at *A* and *B* marked on the microstructure (Fig. 3(*b*)).

Raising the reversion treatment temperature to 250°C for 15 min causes the needles to grow along preferred crystallographic ⟨100⟩ directions[17] (Fig. 4). The



(*a*)



(*b*)

Fig. 1—Electron transmission micrographs (magnification about 88,000 times) of an Al-1.35 at. pct Mg_2Si alloy fully age hardened showing (*a*) duplex nature of precipitate: spherical-like and needle-like types, and (*b*) precipitate-free zones. (Alloy was solution heat treated for 30 min at 565°C, water quenched at 20°C, and aged 24 h at 160°C.)

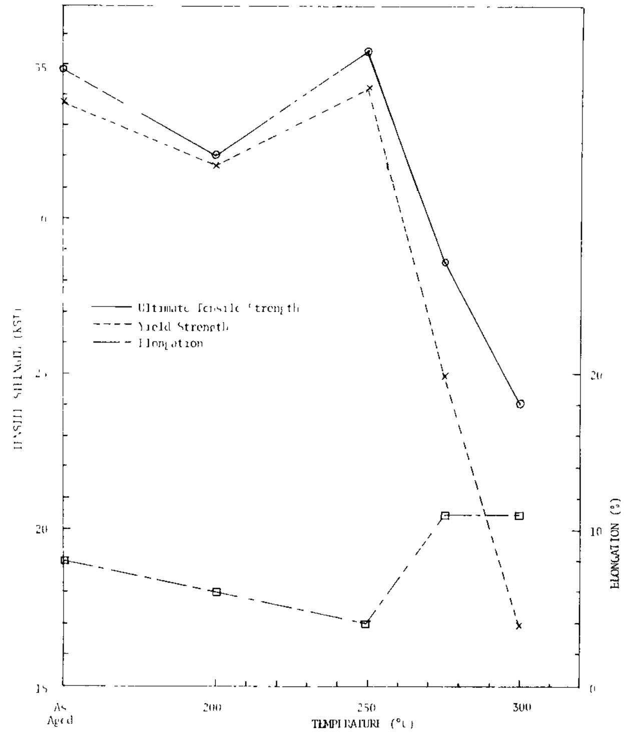
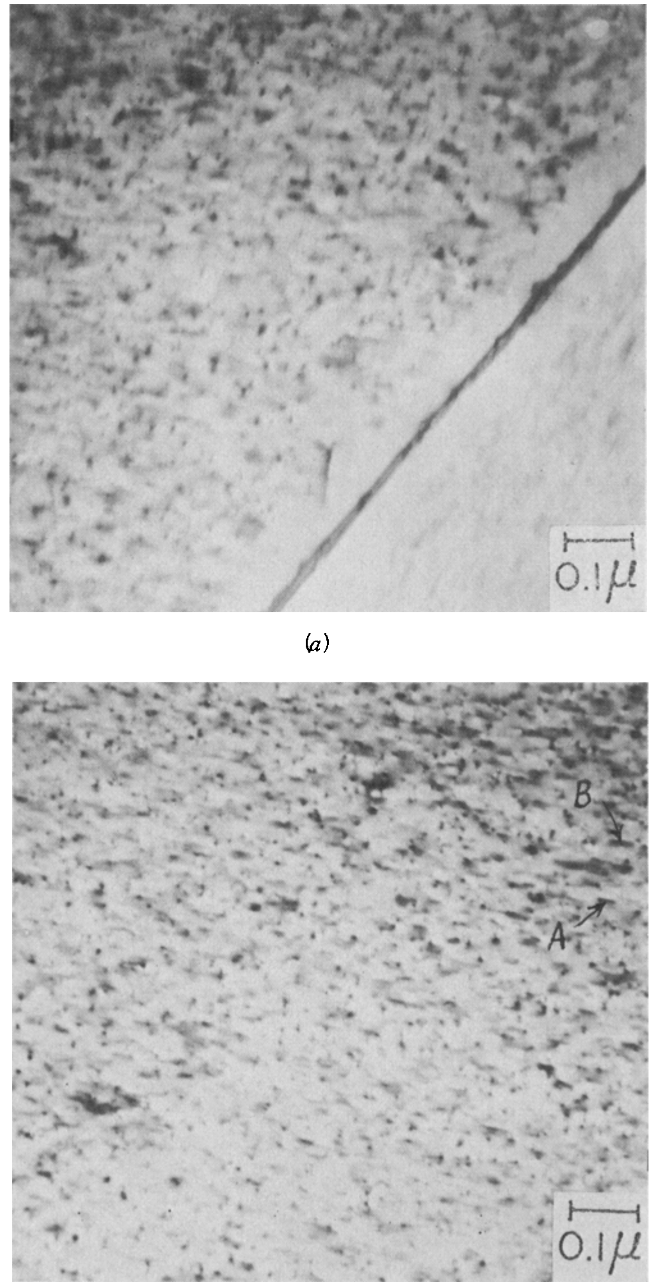


Fig. 2—Effect of 15 min reversion treatments at temperatures from 200 to 300°C on the tensile properties of a fully age hardened Al-1.35 at. pct Mg_2Si alloy. (Alloy was solution heat treated for 30 min at 565°C, water quenched at 20°C, aged 24 h at 160°C, and reheated 15 min at the temperatures indicated.)



(a)

(b)

Fig. 3—Electron transmission micrographs (magnification about 88,000 times) of an Al-1.35 at. pct Mg_2Si alloy after reversion treatment of 15 min at 200°C. After this reversion treatment the alloy shows (*a*) coarsening of the precipitates, and (*b*) some distinct needles of β' intermediate phase, such as at *A* and *B*. (Alloy was solution heat treated for 30 min at 565°C, water quenched at 20°C, aged 24 h at 160°C, and reheated 15 min at 200°C.)

duplex nature of the precipitation can be seen by the presence of large needles such as at *A* along with a fine precipitate as shown, for example, at *B*. The alloy with this duplex structure showed superior strength properties (UTS = 35.5 ksi) but a decreased ductility (4 pct).

Increasing the reversion treatment temperature still higher to 275°C for 15 min finally lowers the strength to 28.6 ksi UTS and increases the ductility to 11 pct. The microstructure in this case shows a distinct network of the needle-like precipitates, but the fine precipitate appears to have dissolved (Fig. 5).

Applying a reversion treatment at an even higher temperature of 300°C for 15 min coarsens the needle-like network of precipitates (Fig. 6). Correspondingly, the tensile strength of the alloy decreased to 24.2 ksi with the ductility remaining at 11 pct.

Isothermal Reversion Studies

It was observed from the constant time experiments that at 275°C the strength of the alloy decreased considerably. Therefore, this temperature was chosen for varying the time at temperature to investigate the changes in microstructure and properties of this alloy isothermally.

After applying a reversion treatment of 5 min at 275°C to the alloy aged 24 h at 160°C, the strength (UTS) of the alloy decreased from 34.9 to 33.3 ksi (Fig. 7). The corresponding microstructure for this alloy is shown in Fig. 8(*a*). Some needle-like precipitate can be seen, for example, at *A* and *B*. A fine precipitate can also be observed between the needles which shows a low degree of partial coherency and hence does not appear clearly.

A reversion treatment of 10 min at 275°C applied to the alloy after aging 24 h at 160°C lowers the strength (UTS) to 32.1 ksi. The needles appear more distinct in the structure (Fig. 8(*b*)) and the fine precipitates appear to have lost most of their contrast.

After reverting the aged alloy for 15 min at 275°C, the tensile strength (UTS) decreased to 28.6 ksi and the ductility increased to 11 pct. The 0.2 pct offset yield strength also decreased considerably from 34.2 ksi for 10 min at 275°C to 28.6 ksi for 15 min at 275°C. In the microstructure the network of needles appears even more distinct (Fig. 8(*c*)) than after 10 min (Fig. 8(*b*)) due to its becoming more incoherent. The areas between the needles also are observed to have cleared up.

DISCUSSION

On the basis of these experiments, it is clear that Type 2 reversion, as defined in the introduction of this paper, takes place in Al-Mg_2Si alloys. That is, the G.P. zones are not completely dissolved by the reversion process without some simultaneous precipitation of the partially coherent intermediate phase of Mg_2Si. Even after the fully age hardened Al-Mg_2Si alloy was reverted for 15 min at 250°C, which is 90°C above the temperature at which it was age hardened, the tensile strength did not *decrease* as one would expect if true (Type 1) reversion occurred. Instead, this alloy was

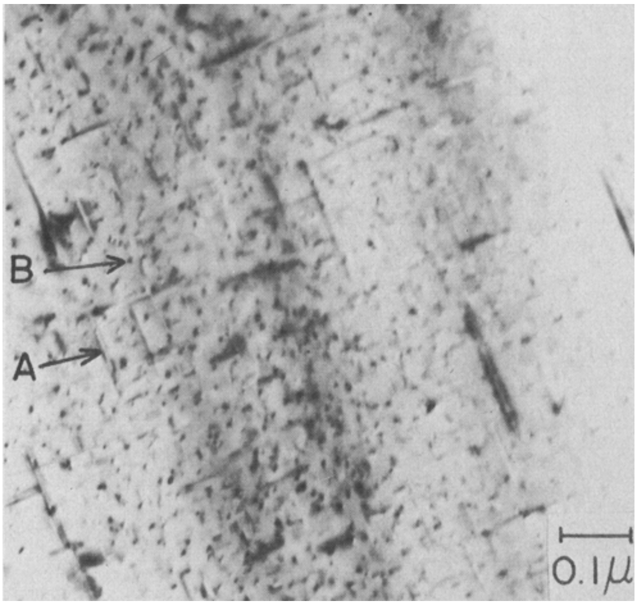


Fig. 4—Electron transmission micrograph (magnification about 88,000 times) of an Al-1.35 at. pct Mg_2Si alloy after reversion treatment of 15 min at 250°C. After this reversion treatment the alloy still showed a duplex microstructure. For example, large needles at *A* and a fine precipitate at *B*. (Alloy was solution heat treated for 30 min at 565°C, water quenched at 20°C, aged 24 h at 160°C, and reheated 15 min at 250°C.)

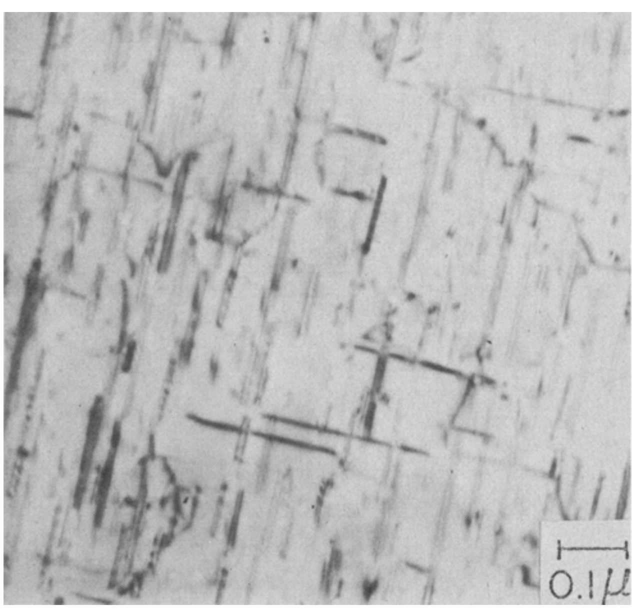
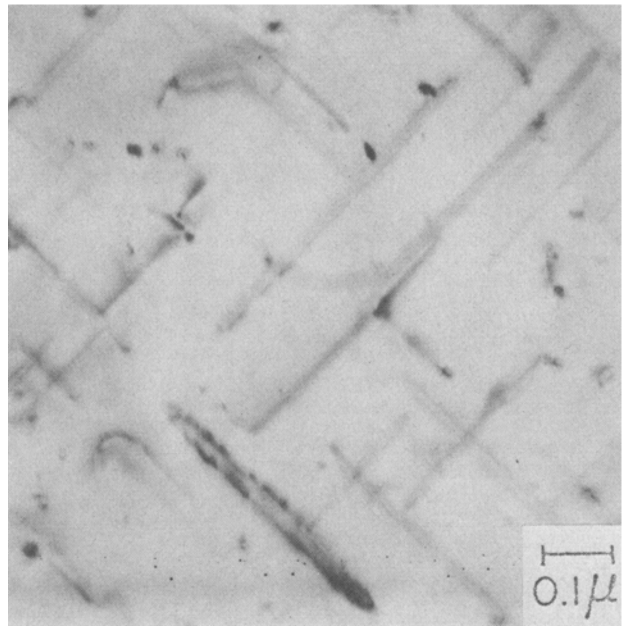
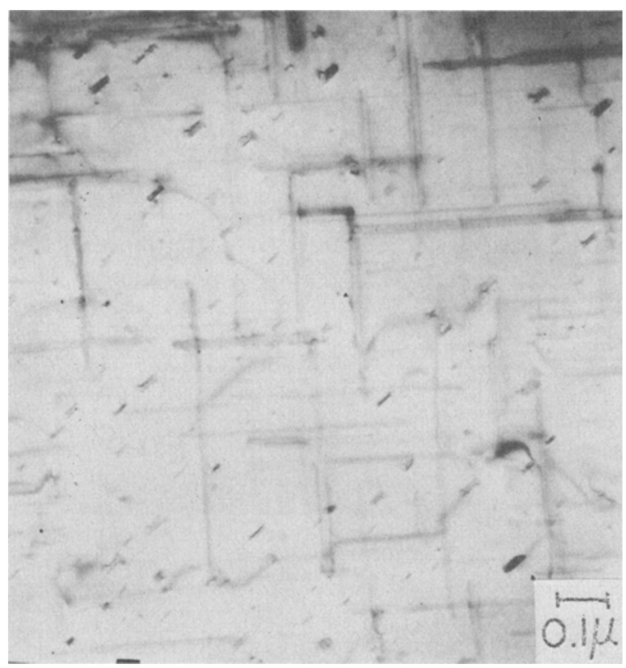


Fig. 5—Electron transmission micrograph (magnification about 88,000 times) of an Al-1.35 at. pct Mg_2Si alloy after reversion treatment of 15 min at 275°C. After this reversion treatment, alloy shows needles of β' intermediate phase only. (Alloy was solution heat treated 30 min at 565°C, water quenched at 20°C, aged 24 h at 160°C, and reheated 15 min at 275°C.)



(*a*)



(*b*)

Fig. 6—Electron transmission micrographs of an Al-1.35 at. pct Mg_2Si alloy after reversion treatment of 15 min at 300°C. Structure shows coarse needle-like precipitate of β' intermediate phase in (*a*) which is at magnification about 88,000 times, and (*b*) which is at magnification about 51,000 times. Lower magnification shows needle-like network more distinctly. (Alloy was solution heat treated for 30 min at 565°C, water quenched at 20°C, aged 24 h at 160°C, and reheated 15 min at 300°C.)

slightly strengthened (34.9 to 35.5 ksi) by the reheating, but its ductility was lowered from 8 to 4 pct. This effect could be explained by the coarsening of the intermediate β' precipitate and the simultaneous coarsening of some of the G.P. zones and the dissolution of others.

This situation whereby two precipitation reactions occur simultaneously is not uncommon. For example, while platelets of θ' phase in an Al-1.7 at. pct Cu alloy are being dissolved by a reversion process, the equilibrium phase θ appears in its place.[23] In the case of some aged Al-Ag alloys reversion treatments must remove the hardening effects of at least two decomposition products.[7] Thus, for Type 1 or normal reversion to take place there must exist a *discontinuity* between the two successive forms of segregate. There appears

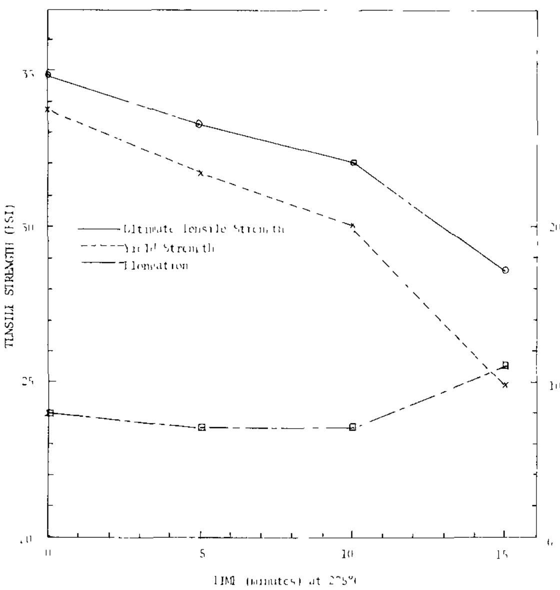
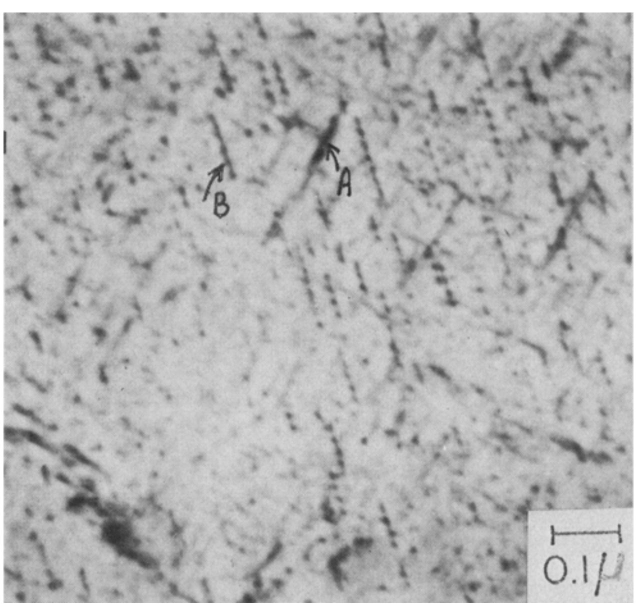


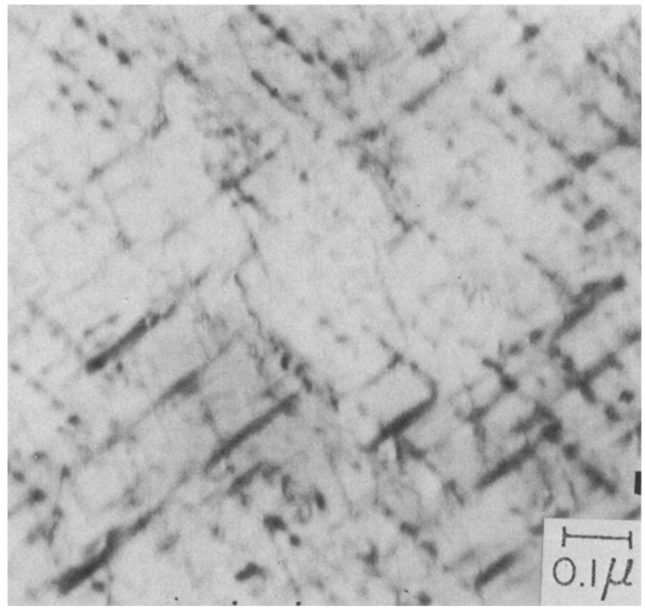
Fig. 7—Effect of reversion time at 275°C on the tensile properties of a fully age hardened Al-1.35 at. pct Mg_2Si alloy. (Alloy was solution heat treated for 30 min at 565°C, water quenched at 20°C, aged 24 h at 160°C, and reheated for different time intervals at 275°C.)



(a)

to be no such discontinuity in this Al-Mg_2Si alloy between the G.P. zones and the intermediate β' precipitate.

However, a critical temperature (T_c) below which homogeneous precipitation occurs and above which heterogeneous precipitation takes place can be determined for this alloy system upon cooling (quenching and aging) as has been shown by Pashley *et al.*[24] For their alloy (Al-1.20 wt pct Mg_2Si) a value of T_c of 225 ± 5°C was determined for samples quenched directly into a salt bath and aged. However, as Pashley points out, T_c is kinetically sensitive and its value will change



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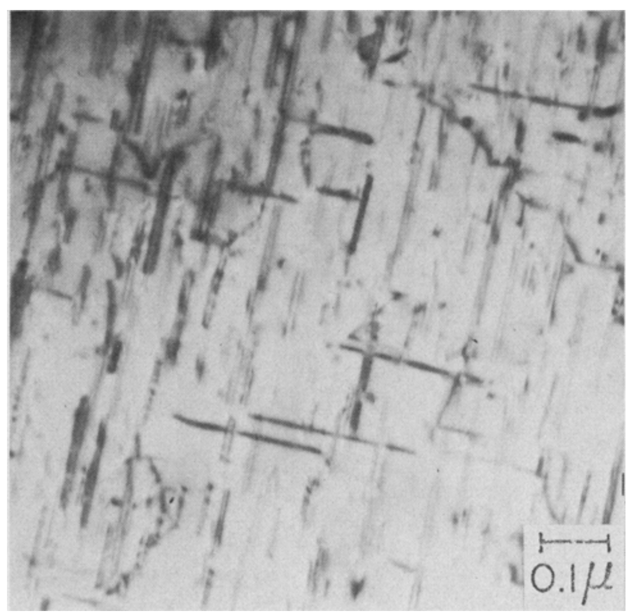

(c)

Fig. 8—Electron transmission micrographs of an Al-1.35 at. pct Mg_2Si alloy after reversion treatments at 275°C for (*a*) 5 min, (*b*) 10 min, and (*c*) 15 min. Needles of β' precipitate can be seen at *A* and *B* in (*a*). The partially coherent precipitate between the needles in (*a*) is cleared up in (*b*) and (*c*). (Alloy was solution heat treated for 30 min at 565°C, water quenched at 20°C, aged 24 h at 160°C, and different samples were reheated at 275°C for 5, 10, and 15 min respectively.)

with variation in quench rate and vacancy concentration.

The behavior of the Al-Mg_2Si system is in sharp contrast to the Al-Zn-Mg system which shows a discontinuity in the form of a recognizable G.P. zone solvus which can be determined by reversion experiments.[6] Homogeneous precipitation changes to heterogeneous precipitation over a narrow temperature range in some Al-Zn-Mg alloys and can be observed by electron microscopy.[20]

The results of this investigation indicate that both the G.P. zones and the intermediate partially coherent precipitate needles strengthen Al-Mg_2Si alloys. Even after a relatively high temperature reversion treatment of 15 min at 300°C, the ultimate tensile strength of the alloy is still only reduced to 24.2 ksi. With this treatment the electron micrographs indicate that the G.P. zones had dissolved. By comparison, the strength of this alloy in the as-quenched condition was 17.2 ksi. It does not appear that the conclusion reached by Lutts[16] that the G.P. zones are the only hardening agent in Al-Mg_2Si alloys is correct.

One reason for the high degree of stability of the G.P. zones in Al-Mg_2Si alloys as compared to those in Al-Zn-Mg alloys might be attributed to the relatively high heat of formation and melting point of the equilibrium phase, Mg_2Si. This phase has an enthalpy of formation of -6.4 kcal per g-atom[25] and a melting point of 1085°C. On the other hand, the equilibrium phase in the Al-Zn-Mg alloy system, $MgZn_2$, has a much lower enthalpy of formation of -2.6 kcal per g-atom[26] and a lower melting point of 590°C.[27] Larger exothermic heats of formation are generally associated with higher degrees of ionicity[28] and stabler bonds. Hence diffusion would be more difficult in the Al-Mg_2Si alloys. According to recent work by Kovacs *et al.*,[11] diffusion in the Al-Mg_2Si system is slow because there are strong binding forces between magnesium and silicon atoms. These factors would also favor a high activation energy for the dissolution of the G.P. zones in aged Al-Mg_2Si alloys and hence would lead to the reversion properties shown by them.

CONCLUSIONS

1) Reversion treatments when applied to a fully aged Al-1.35 pct Mg_2Si alloy indicate that true reversion of the G.P. zones formed by the original aging treatment did not occur. Instead they were gradually replaced by β', the partially coherent form of the equilibrium phase, Mg_2Si.

2) After a reversion treatment of 15 min at 250°C (90°C above the original age hardening temperature), the tensile strength of the alloy was increased slightly instead of being decreased, but its ductility was lowered. This effect could be explained by the simultaneous formation of β' while the G.P. zones were coarsening. The decrease in ductility would be due to the development of a coarser precipitate structure.

3) Higher temperature reversion treatments (275 to 300°C) resulted in a gradual decrease in strength and ductility of the alloy which would be caused by the gradual dissolution of the G.P. zones and coarsening of the β'. However, the strength of the alloy did not return to the as-quenched value even after 15 min at 300°C, which would indicate that the coarse needles of the β' precipitate have some strengthening effect.

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