PRECIPITATION AND RECRYSTALLIZATION OF AN AL-MG-SC-ALLOY

J. Røyset and N. Ryum
The Norwegian Institute of Technology, Department of Metallurgy, N-7034 Trondheim, Norway

Abstract

The precipitation kinetics of the alloys Al 4 wt.%Mg 0.2 wt.%Sc and Al 0.2 wt.%Sc at 250 and 300°C was studied by means of hardness measurements and transmission electron microscopy. Even after 170 hours at 300°C, there was only a small overageing of the alloys. The Mg addition had no marked effect on the precipitation behavior. In Al0.2Sc also the effect of ageing at 250°C, followed by ageing at 300°C was studied. The two-step ageing gave only a small increase in strenght, but a reduction of 60% in total ageing time to peak hardness level.

Compression tests were carried out to determine the mechanical properties of the alloys. Values of yield strenght, strain hardening coefficient and strain hardening exponent were evaluated from the stress/strain curves. When true strain exceeded approx. 0.15 deformation no longer followed the power law.

Al4Mg0.2Sc in the as-cast condition was cold rolled, and subsequentally annealed one hour at different temperatures. To verify the effect of Sc, the same experiment was made with a binary Al 4 wt.%Mg alloy. It appeared that Sc raised the recrystallization temperature with at least 100°C. When precipitates were present prior to the annealing, recrystallization was suppressed almost until the solidus temperature of the alloy.

Introduction

The second phase particles present in most technological Al-alloys can be divided into three broad classes:

1) Constituent particles, effecting the castability of the alloy when intensionally present.
2) Dispersoids, effecting the grain structure.
3) Precipitates, effecting the mechanical strength.

So far, different alloying elements have been used to produce dispersoids (Mn, Cr, Fe, Zr) or to produce precipitates (Cu, Mg, Si, Zn). The precipitates have a too low thermal stability to have any benificial effect on the grain structure. On the other hand, the dispersoids have a too low density (particle/volume) to have a marked and direct effect on the mechanical strength.

When analyzing the possible effect of different alloying elements in aluminium, it appears that scandium may have an effect both as a dispersoid-forming and a precipitate-forming element. So far, only rather scant investigation have been published on the effect of Sc on the structure and properties of Al-alloys. The russians are believed to be somewhat ahead, as the former Soviet Union started a research program in this field some 20 years ago. The main activity seems to have been investigating the binary Al-Sc system, but also some research on the ternary Al-Mg-Sc-system has been done.

A lot of interesting features of these alloys can be mentioned. There is a remarkable thermal stability of fully coherent strenghtening Al_3Sc precipitates. The Mg addition reduces the density, making the alloy system interesting for air- and spacecraft purposes. The ability of superplastic forming and mechanical properties at cryogenic temperatures has been investigated [1-2]. Scandium addition also seems to effectively prevent primary recrystallization in Al-Mg-alloy.

In this work, precipitation behavior and mechanical properties of Al0.2Sc and Al4Mg0.2Sc were characterized by hardness measurements and compression tests. The main emphasize has been put on recrystallization of cold-worked Al4Mg0.2Sc. The effect of Sc-addition has been demonstrated by comparing with the binary Al4Mg-alloy. Attempts has also been made to discover the mechanism by which Sc influences the recrystallization.

Experimental procedure

The alloys Al0.2Sc, Al4Mg and Al4Mg0.2Sc were prepared from 99.999% pure aluminum, an Al-35wt%Mg pre-alloy, and 98% pure scandium. Melting was made in nitrogen at approx. 800°C. Due to the numerous intermetallic phase transformations in the Al-Sc system, one needed to keep the melt at this high temperature for at least one hour. After stirring thoroughly, the melt was cast in rod-shaped graphite moulds.

Specimens for the age hardening experiments were prepared by cutting slices of 1.5 mm thickness from the rods. Ageing was performed from the as-cast condition at both 250 and 300°C. In addition possible effects of two-step ageing at these temperatures were investigated.

Stress/strain-curves for the alloys in the peak-hardness condition were obtained by performing compression tests in a servohydraulic MTS machine. The cylindric specimens had a diameter of 10 mm, and height of 15 mm. Deformation was performed at a constant rate of 2 mm/min at room temperature.

Rectangular rods of Al4Mg and Al4Mg0.2Sc in the as-cast condition were cold-rolled 25 and 75%. Also, a rod of Al4Mg0.2Sc was aged 10 hours at 300°C prior to 25% deformation. The response of subsequent heat-treatment was investigated by isochronal annealing. Specimens were kept at temperatures between 150 and 600°C, with steps of 25°C, for one hour. In addition, isothermal annealing was applied on specimens from Al4Mg0.2Sc aged prior to deformation. Annealing times ranged from 5 minutes to 300 hours at the temperatures 550 and 575°C.

Thin foils for TEM-microscopy were produced by the window technique in a solution of 20% $HClO_4$ and 80% ethanol at 15 V and -20°C. The foils were investigated in a JEOL JEM-200CX transmission electron microscope.

Results and discussion

Preparation of the alloys

Chemical analyses of the alloys containing scandium are given in Table I. It appears that there is a slight loss of both scandium and magnesium. This might be due to reaction with oxygen in the furnace atmosphere, and for scandium also improper dissolvation in the melt.

Table I. Chemical analyses of the alloys containing scandium

Alloy	wt% Sc	95% int. of conf.	wt.% Mg	95% int. of conf.
Al0.2Sc	0.162	[0.161 - 0.164]	-	-
Al4Mg0.2Sc	0.169	[0.157 - 0.181]	3.83	[3.71 - 3.94]

The microstructure of the material consisted of rather large columnar grains, grown from the mould walls towards the center of the rods. Consequently, there were some pores in the center of the rods. These defects were more frequently observed in the alloys containing Mg.

Age hardening

Figure 1 a) and b) displays the hardness measurements during ageing at 250 and 300°C, respectively. The stapeled lines with filled markers are the values from Al4Mg0.2Sc, subtracted the effect of Mg solution hardening. Thus one can directly compare the effect of precipitation hardening in the alloys. Peak values measured were 65.4 HV5 for Al0.2Sc after 48 hours at 250°C, and 98.7 HV5 for Al4Mg0.2Sc after 10 hours at 300°C. These values are in accordance with earlier works on this system [3-4].

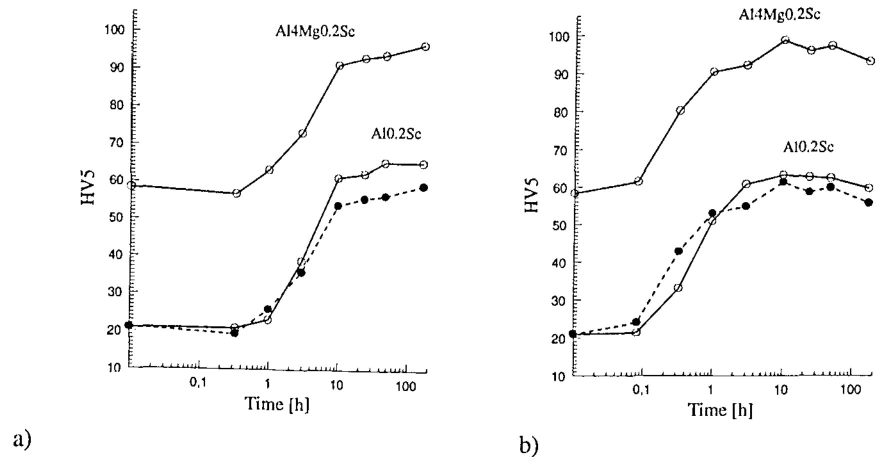


Figure 1 Hardness measurements from age hardening at a) 250 and b) 300°C.

As indicated by the curves, the effect of precipitation hardening is slightly smaller in the alloy containing magnesium, even though the chemical analysis shows a higher mean value of Sc-content. This may be due to a decrease in the driving force for precipitation. Such a decrease will occur if the solid solubility of Sc in α-Al increases when Mg is added. Thus, this observation indicates such an increase in the solubility of Sc.

In TEM one observed contrasts typical for elastic strain-fields caused by coherent precipitates. These contrasts are shown in Figure 2, from a specimen of Al4Mg0.2Sc aged 10 hours at 300°C. Superlattice spots in the diffraction pattern indicating coherent precipitates with a cubic primitive crystal structure were also easily observed. This is in accordance with earlier observations [5], indicating precipitates of the Al_3Sc-phase with $L1_2$ structure. Attempts of observing the precipitates in dark-field failed, probably due to the very small particle size.

In some alloy systems, e.g. Al-Si, a fine distribution of precipitates depends on the presence of excess vacancies. Grain boundaries effectively deplete the adjacent matrix for vacansies, and thus a precipitate free zone (PFZ) along the boundary is observed. In this alloy there were no PFZ's along the grain boundaries. This leads to the conclusion that excess vacancies are unimportant in the mechanism of nucleation of Al_3Sc precipitates.

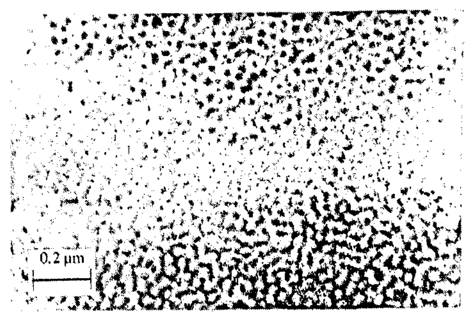


Figure 2. TEM-image of elastic strain fields in Al4Mg0.2Sc, aged at 300°C for 10 hours.

In the two-step ageing, two different ageing times at the first step of 250°C were applied; 3 hours, which should be in the growth stage of the precipitates, and 48 hours which is at the peak hardness. These two series are in the following designated series I and series II, respectively. Figure 3 shows the hardness measurements during the second step. Peak values were 66.1 for series I after 24 hours, and 66.7 for series II after 48 hours at 300°C.

The two-step routes gave only a small increase in the peak hardness values. But in series I a hardness of 64.2 is reached after only 1 hour in the second step. This is in exceed of the peak value from the one-step ageing at 300°C, reached after 10 hours. The total ageing time to reach this hardness is reduced 60 % by two-step ageing. By optimizing combinations of time and temperature, the total ageing time should be shortened even more.

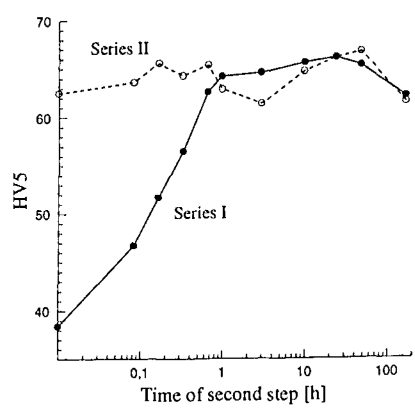


Figure 3. Hardness measurements from second step of two-step ageing.

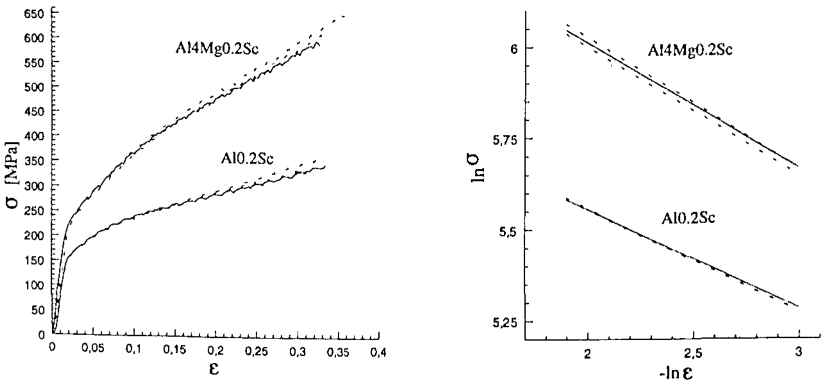
Compression tests

Both alloys were aged to peak hardness at 300°C (10 hours) before the compression tests. Three specimens of each alloy were used. True stress versus true strain for all the specimens are plotted in Figure 4 a). At low plastic strains, work hardening appears to follow the power law

$$\sigma = K \varepsilon^{n} \tag{1}$$

When true strains exceeds 0.15-0.20, true stress increases with true strain at a constant rate. Consequently the strain hardening exponent n is not a constant, but increases when $\varepsilon >$ ca. 0.2. Also, a marked deviation from the power law normally occurs at very low plastic strains. Thus, evaluating n for the alloys, only strains in the range 0.05 - 0.15 are included.

Figure 4 b) is a plot of $\ln\sigma$ versus $-\ln\varepsilon$ for all the alloys. The linear approximation is carried out according to the minimum square sum principle. From extrapolating the lines to $-\ln\varepsilon = 0$ the strain hardening coefficient K can be found, as $\ln K$ equals $\ln\sigma$ in this point. Values of yield strenght σ_y, K and n for the alloys are summarized in Table II.



a) b)

Figure 4. Stress/strain and mean $\ln\sigma/\ln\varepsilon$ -curves for the compression test specimens.

Table II. Values of σ_y, n and K for the alloys, with 95% interval of confidence.

Alloy :	Al0.2Sc		Al4Mg0.2Sc	
σ_y [MPa] :	138	[129 - 147]	194	[186 - 201]
n :	0.28	[0.26 - 0.30]	0.35	[0.33 - 0.37]
K [MPa] :	451	[433 - 469]	825	[778 - 872]

Recovery and recrystallization

Hardness measurements from isochronal annealed specimens are shown in Figure 5. Here, Al4Mg and Al4Mg0.2Sc are designated "Mg" and "Sc" respectively, followed by the rate of deformation. In addition, the rod peak-aged prior to deformation is designated PA. The temperatures for which the recrystallization reaction had started and finished after one hour, listed in Table III, were found by light microscopy.

Table III. Temperatures [°C] for beginning and end of recrystallization after 1 h. annealing.

Specimen	Mg 75%	Mg 25%	Sc 75%	Sc 25%	Sc PA 25%
Recr. starts	250	300	350	425	575
Recr. complete	300	350	400	575	600

When comparing figure 5 with Table III, one concludes that most of the drop in hardness value of Al4Mg is due to recovery processes with lower thermal activation than recrystallization. In the as-cast Al4Mg0.2Sc recovery and precipitation occurs simultaneously, giving competing contributions to the hardness value.

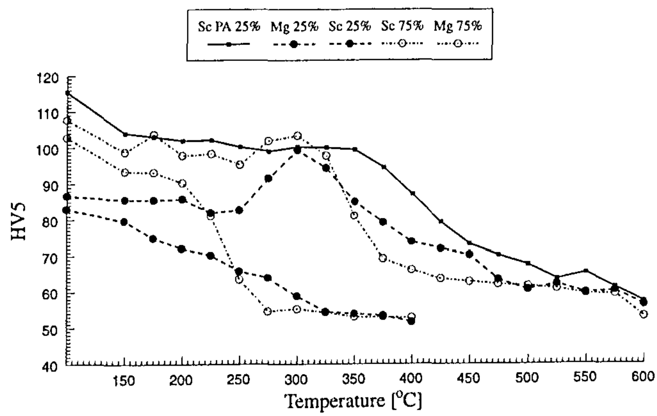


Figure 5 Hardness measurements from isochronal annealed specimens.

Both as-cast series containing scandium reach a peak value at 300°C. Above this temperature overageing takes place, and consequently the hardness value drops. One notices from Table III that Sc rises the recrystallization temperature significantly. In the 25% series, partially recrystallized material is observed over a wide temperature range.

When precipitates are already present in Al4Mg0.2Sc, like in the peak aged series, recrystallization seems almost impossible. After 1 hour at 575°C only recrystallization of very small areas can be seen. In the isothermal experiment only small recrystallized areas are found even after 300 hours at this temperature. After 1 hour at 600°C, the reaction is complete. This temperature is well above the solidus-line in the binary Al-Mg system [6]. Assuming that the small Sc-addition does not alter the solidus temperature too much, the material is in the partially molten condition during recrystallization. Indications of partial melting could also be found on some specimen surfaces.

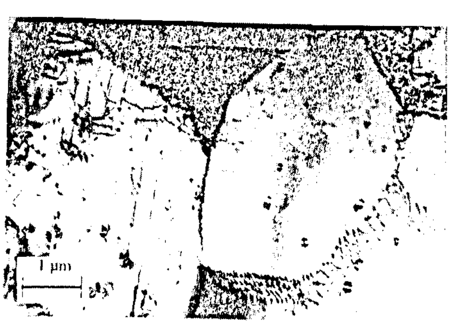
TEM-studies of the peak aged series revealed that the initial drop in hardness (1 hour at 175°C) is due to a reduction in dislocation density. This means that the recovery process starts with annihilation of dislocations of opposite Burgers vectors. As the temperature increases, hardness succesively approaches the value of the peak age hardness. This is probably due to the development of a subgrain-structure. As temperature exceeds 350°C there is a significant drop in the hardness value, caused by coarsening of the Al_3Sc-precipitates. For some reason, the resistance against coarsening seems to be a bit higher than in the as-cast series.

Figure 6 is a TEM-micrograph of peak-aged Al4Mg0.2Sc annealed at 400°C for one hour. Here, both a well developed subgrain structure and slightly coarsened precipitates can be observed. Typical subgrain sizes are in the range of 0.5-1 μm.

Figure 6. Subgrain structure in the peak aged series, annealed at 400°C for 1 hour.

After annealing one hour at 575°C, as shown in Figure 7 a), the subgrain size is somewhat larger. Typical values ranges from 2 to 4 μm. Also, there has been a dramatic coarsening of the Al_3Sc-precipitates to diameters of 100 - 150 nm. Diffraction patterns shows that there still is a coherency between the precipitates and Al-matrix. This is also indicated by the typical coffee-bean contrast displayed in Figure 7 a).

There is no tendency for the precipitates to be located on the subgrain boundaries. This means that there probably is only a negligable pinning action from the precipitates upon the movement of these boundaries. Figure 7 b) is a close-up of a subgrain boundary cutting a 150 nm Al_3Sc-particle. Assuming that the boundary is moving due to subgrain growth, the particle causes no distinct



a)

b)

Figure 7. Peak-aged series, annealed at 575°C for 1 hour. a) subgrain structure and coffee-bean contrasts from coherent Al_3Sc particles. b) subgrain boundary cutting a coarse Al_3Sc particle.

curvature of the boundary.

In this specimen one also found an area, limited by high-angle grain boundaries, in which there were no subgrains. Also, there were no coherency between the coarse Al_3Sc-particles and matrix. This observation indicates the area to be a recrystallized grain. The recrystallization front is showed in Figure 8. As can be seen, particles of 30-50 nm diameter are decorating the entire boundary. Since the effect of Sc is most accentuated when precipitates are grown prior to annealing, one believes that the high resistance against recrystallization is due to these particles. Still, a more thorough investigation is needed to clarify the mechanismes by which scandium impedes recrystallization.

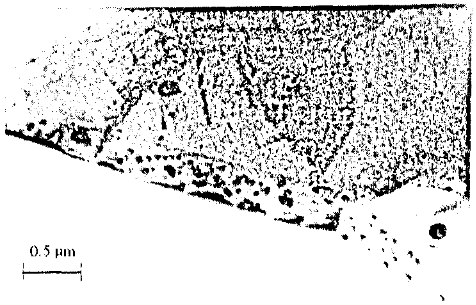


Figure 8. Particles of Al_3Sc on the recrystallization front.

Conclusions

The presence of magnesium does not affect the precipitation behavior of Al_3Sc. A slightly smaller effect of precipitation hardening is observed in the ternary alloy. Two-step ageing as performed in this work gives no marked increase in peak hardness, but a reduction of 60% of total ageing time to peak hardness level.

When deformed in the peak-aged condition, both alloys work-harden at a high rate. At true strain larger than ca. 0.2, work-hardening is higher than that predicted by the power law (1).

Scandium raises the temperatures needed for starting and ending recrystallization of cold-worked Al4Mg after 1 hour annealing with at least 100°C. When particles of Al_3Sc are present prior to the annealing, heating close to the solidus temperature is needed to recrystallize the material.

References

1. R.R. Sawtell and C.L. Jensen, Metall. Trans. A 21A, (1990), 421.

2. S.L. Verzasconi and J.W. Morris, Jr., Advances in Cryogenic Engineering (Materials), 36, (1990), 1127.

3. H.-H. Jo and S.-I. Fujikawa, Mater. Sci. Eng., A171, (1993), 151.

4. M.E. Drits et al., Russ. Metall., (3), (1984), 192.

5. N. Sano et al., Proceedings of the Second International Conference on Aluminium Alloys, oct. 9-13, Beijing, China, (1990), 549.

6. E. Butchers and W. Hume-Rothery, J. Inst. Metals, 71, (1945), 291.