EFFECT OF SCANDIUM ON THE SOFTENING BEHAVIOUR OF DIFFERENT DEGREE OF COLD ROLLED AL-6MG ALLOY ANNEALED AT DIFFERENT TEMPERATURE

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ABSTRACT

The softening behavior of different cold rolled Al-6Mg alloys containing scandium 0.2 wt% and 0.6 wt% have been investigated by means of microscopy, hardness and electrical conductivity measurements. It is found that the scandium added alloys attend the higher hardness at every state of cold rolling at higher annealed temperature due to the precipitation of scandium aluminides. Electrical resistivity of the scandium added alloys show higher than base alloy due to grain refining. It is seen from the microstructure that scandium refine the grain structure and inhibit recrystallization.

KEYWORDS

Al-Mg alloys, microstructure, cold rolling, annealing, electrical resistivity

1. INTRODUCTION

Technically pure aluminium has good formability, good thermal conductivity but very little strength. Increasing the strength of aluminium can be achieved in several ways: by solid-solution-hardening, precipitation-hardening or by refinement of grains, creating a fine and even ultrafine grains microstructure [1-3]. However, precipitation-hardening is the most effective method. This type of hardening is often used in aluminum alloys containing copper, magnesium, chromium, lithium, zinc etc. [4, 5]. Precipitations coherent with the matrix which are located inside the grains are the most effective. Due to the different methods of production of aluminum components casting, extrusion, rolling or processing the creation of an optimum microstructure in a single technology process is extremely difficult and additional heat treatment is necessary [6-8]. The most commonly given information applies only to the temperature range of super saturation and aging, but it does not give time specifications of the aging process. It is very important to note that too long artificial aging can lead to so-called over aging i.e. decreased strength and hardness of the material as a result of mostly coagulation of the precipitates. The knowledge of the recommended treatment temperature as well as the time of this process are essential for optimum hardening results. With regard to the ranges of the heat treatment temperature, deviations from specified ranges and the impact of the implementation of treatment at a slightly higher or slightly lower temperature should be known. Mg is the principal alloying element and is added for solid solution strengthening [9]. The Al-Mg alloys have a favorable formability, as due solution hardening they can achieve high strength and high strain hardening ability, which enable a stable behavior in the complex forming operation, reducing the further material flow in the locally strained regions [10, 11].

The use of scandium as an alloying element in aluminium has gained an increasing interest even though scandium is difficult to extract, which makes the metal very expensive. The three principle effects that can be obtained by adding scandium to aluminium alloys are (i) grain refinement during casting or welding, (ii) precipitation hardening from Al_3Sc particles and (iii) grain structure control from Al_3Sc dispersoids [3].

Al-Mg alloy is normally used in a work-hardened condition and will undergo softening during use. But the softening behavior of the alloy system under the influence of Al_3Sc particles pre-existing in the matrix has not been studied elaborately. The aim of this work was to determine the softening behavior of cold rolled Al-6Mg alloys doped with scandium 0.2 wt% and 0.6 wt%. Since cold working introduces huge dislocations, it is a question to resolve if the presence of dislocations has any role on the precipitation behavior of Al_3Sc during annealing of the Al-Mg-Sc alloy.

2. EXPERIMENTAL

Melting was carried out in a resistance heating pot furnace under the suitable flux cover (degasser, borax etc.). Several heats were taken for developing base Aluminium-Magnesium alloy and Aluminium-Magnesium alloy containing scandium at various levels. In the process of preparation of the alloys the commercially pure aluminium (99.5% purity) was taken as the starting material. First the aluminium and aluminium-scandium master alloy (2%Sc) were melted in a clay-graphite crucible, then magnesium ribbon (99.7% purity) was added into solution. The final temperature of the melt was always maintained at 780±15^{o}C with the help of the electronic controller. Variation of the scandium percentage was accomplished by its respective additions of master alloy. Casting was done in 12.5mm x 50mm x 200mm cast iron metal mould preheated to 200^{o}C. The alloy was analysed by chemical and spectrochemical methods simultaneously. The chemical compositions of the alloys are given in Table 1. The cast samples were first ground properly to remove the oxide layer from the surface. Cold rolling of the alloy, as cast was carried out with a laboratory scale rolling mill of 10HP capacity at deferent percentages of reduction. The samples in as cast and cold rolled were annealed isochronally at various temperatures for one hour ranging from 100^{o}C to 500^{o}C. Vickers hardness of differently processed alloys was measured with a 5 kg load for assessing the softening effect of the alloy. An average of seven consistent readings was accepted as the representative hardness value of an alloy. Electrical conductivity of the alloys as cast and cold worked with different deformation percentages, were measured. Surface finished samples of area 12 mm x 12 mm, were subjected to conductivity test using a Conductivity Meter, Type 979. Electric resistivity was then calculated from the measured values of conductivity. The cold rolled alloys after different heat treatments are subjected to optical metallographic studies. The specimens were polished with alumina and etched with Keller's reagent and observed under a Versamet-II- Microscope. Scanning electron microscopy of the selected samples was carried out by a Jeol Scanning Electron Microscope, JSM-5200. Transmission Electron Microscopic studies of the alloy was carried out in Philips (CM12) Transmission Electron Microscope at an accelerating voltage 160 KV.

Table 1. Chemical Composition of the Experimental alloy (wt%)

Alloy	Mg	Sc	Zr	Ti	Cu	Fe	Mn	Ni	Si	Zn	Al
1	6.10	0.000	0.000	0.001	0.081	0.382	0.155	0.003	0.380	0.136	Bal
2	5.90	0.200	0.001	0.002	0.081	0.345	0.132	0.003	0.360	0.174	Bal
3	6.02	0.600	0.001	0.003	0.061	0.293	0.086	0.002	0.320	0.126	Bal

3. RESULTS

3.1. Isothermal Annealing

The results of isochronal annealing of all the three cast alloys at different temperature for 1 hour are shown in Figure 1. It is seen that all the alloys except base alloy (alloy 1) have shown appreciable precipitation hardening response. Alloy 1 has however shown a continuous softening at increasing annealing temperatures, with a steeper hardness drop beyond 400^{o}C. However a distinct rise in peak hardness of the alloys is observed for increase in scandium content from 0.2 wt% and 0.6 wt%. In all cases the peak hardness is obtained at around 300^{o}C. Beyond peak hardness value the usual softening due to annealing takes place. On close observation of the variation of hardness in over aged situation, it appears that alloy 3 with 0.6 wt% Sc possesses maximum resistance to age softening.

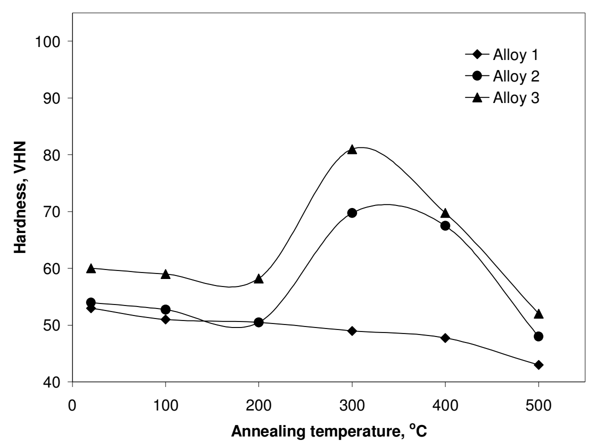

Figure 1. Isochronal annealing curve of the cast alloys annealed for 1 hour.

The results of isochronal annealing of the cold worked alloys at different temperature for 1 hour show identical nature of precipitation hardening at different deformation percentages (Figures 2-4). The initial hardness is higher for higher amount of deformation for all the alloys. The base alloy (alloy 1) shows a continuous softening due to recovery and recrystallisation of the strained grains. All other alloys demonstrate precipitation hardening response with peak hardness value at 300^{o}C. An initial softening to the tune of 5 VHN is noted in almost all the alloys. The extent of age hardening varies with the composition of the alloy; however alloy 3 shows maximum hardness at all deformations. The hardness value of all the alloys at peak-aged condition is seen to be increased with increasing amount of deformation. The extent of precipitation hardening does not change with varying deformation percent. At high deformation no extra benefit in maximum value of hardness could be noted. Most of the alloys show softening during initial period of annealing and increase in hardness after annealing finally enables the maximum hardness to reach a magnitude which is comparable to the initial hardness of cold worked alloys. When the alloys are annealed at higher temperature a sharp decrease in hardness is observed for all the alloys. Thus appreciable drop in hardness values are noted at annealing temperatures beyond 300^{o}C. Figure 5 shows the peak hardness of the alloys at different cold rolled state. It is shown that all the hardness increases with increasing of the cold rolling. Higher scandium added alloy attended the higher hardness.

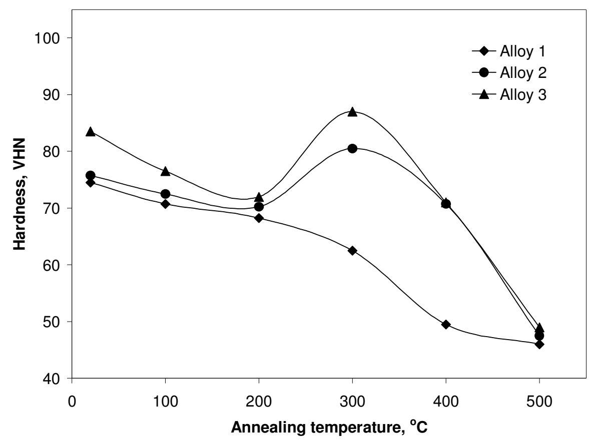

Figure 2. Isochronal annealing curve of the 35% cold rolled alloys annealed for 1 hour.

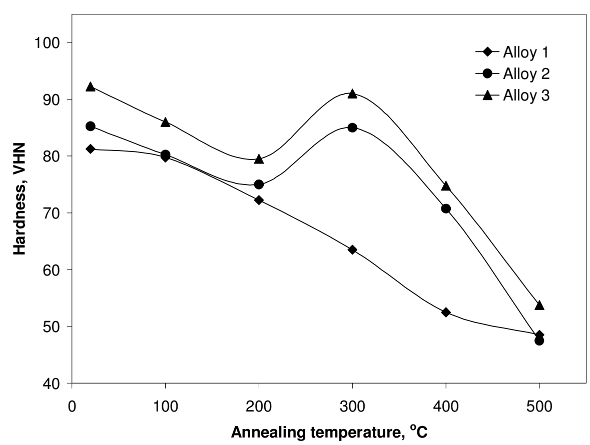

Figure 3. Isochronal annealing curve of the 50% cold rolled alloys annealed for 1 hour.

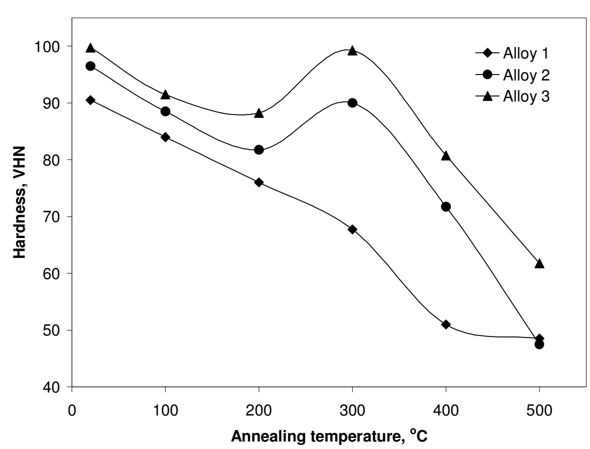

Figure 4. Isochronal annealing curve of the 75% cold rolled alloys annealed for 1 hour.

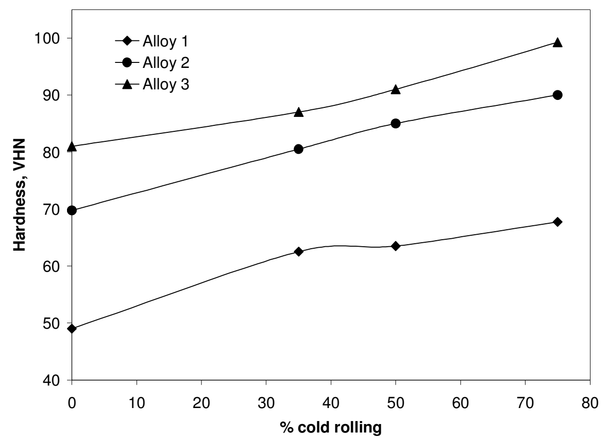

Figure 5. Variation of hardness of different cold rolled alloys annealed at 300^{o}C for one hour

Figure 6 shows the electrical resistivity of the alloys at different cold rolled state annealed at 300^{o}C for one hour. Electrical resistivity decreases with the increase of the cold deformation. From the resistivity curve the initial resistivity of the scandium added alloys show higher than base alloy due to grain refining.

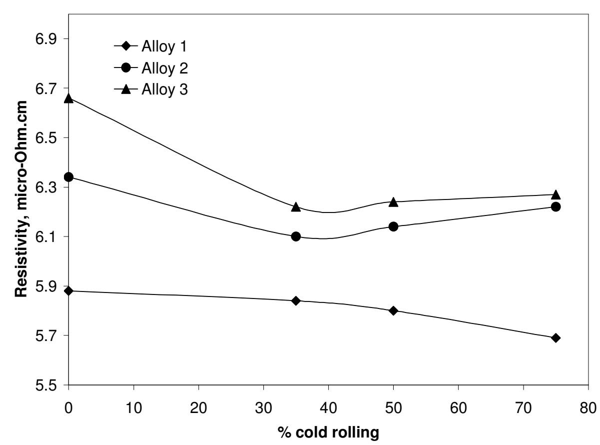

Figure 6. Variation of electric resistivity of different cold rolled alloys annealed at 300^{o}C for one hour

3.2. Isothermal Micrographs

3.2.1 Scanning Electron Micrographs

Scanning electron microstructures of the base alloy shows coarse dendrites with high quantity of second phase constituents. The second phase is found to be contained in the inter-dendritic space (Figure 7). In 0.6Wt % scandium added alloy 3, not only there happens refinement of dendrites but also the second phase constituent has been reduced in amount (Figures 8).

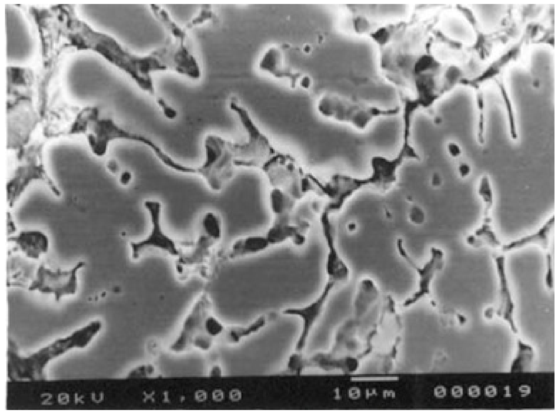

Figure 7. SEM micrograph of cast alloy 1.

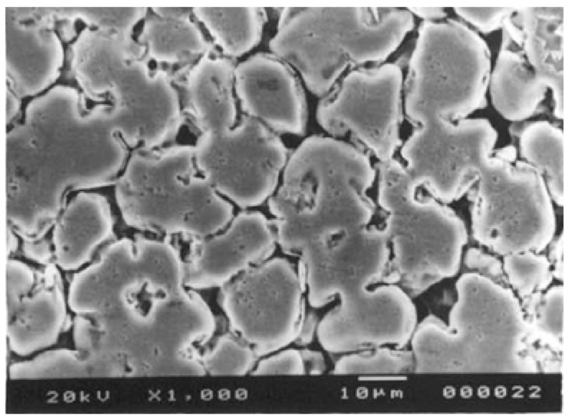

Figure 8. SEM micrograph of cast alloy 3

3.2.2 Optical Micrographs

The cold worked alloy shows relatively coarse non-uniform grain structure. The overall appearance is columnar grains with second phase particles remaining aligned along the grain boundaries. The microstructure of base alloy annealed at 300^{o}C after 75% cold rolling shows partially recrystallised grains with ample second phase particles at the grain boundaries (Figure 9). In case of alloy 2 (0.2 wt% Sc) there is no evidence of recrystallisation after annealing at 300^{o}C (Figure 10). Recrystallisation could not be effected also in alloys 3 after annealing at 300^{o}C. It may be noted that a circularity of grains is maximum in alloy 2, which contains large elongated grains. On the contrary higher scandium containing alloy 3 has exhibited elongated grains of smaller size (Figure 11). If the alloys are annealed at 400^{o}C, the base alloy is seen to be recrystallised almost fully (Figure 12). The scandium added alloy 3, on the other hand do not recrystallise even when annealed at 400^{o}C (Figure 13). A good number of elongated grains are present in the microstructure.

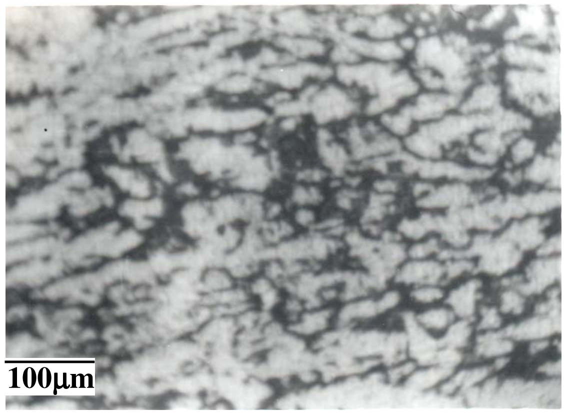

Figure 9. Optical micrograph of 75% cold rolled alloy 1 annealed at 300^{o}C for one hour.

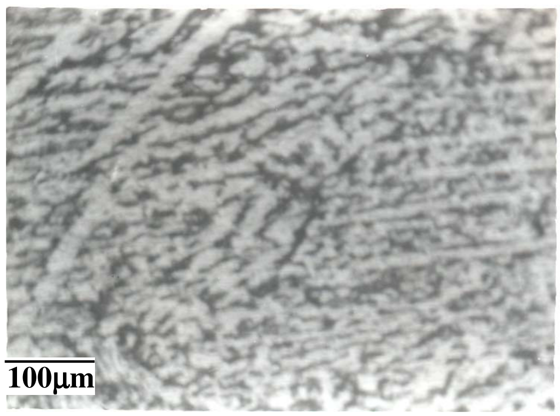

Figure 10. Optical micrograph of 75% cold rolled alloy 2 annealed at 300^{o}C for one hour.

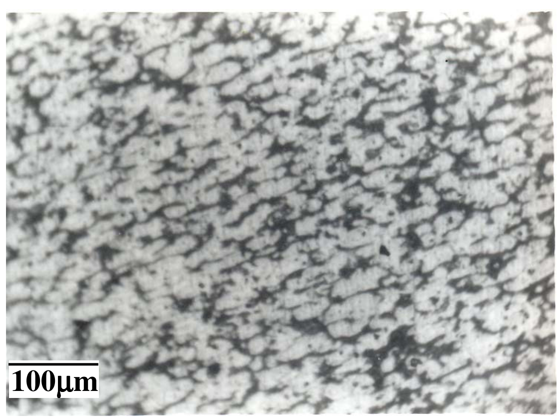

Figure 11. Optical micrograph of 75% cold rolled alloy 3 annealed at 300^{o}C for one hour.

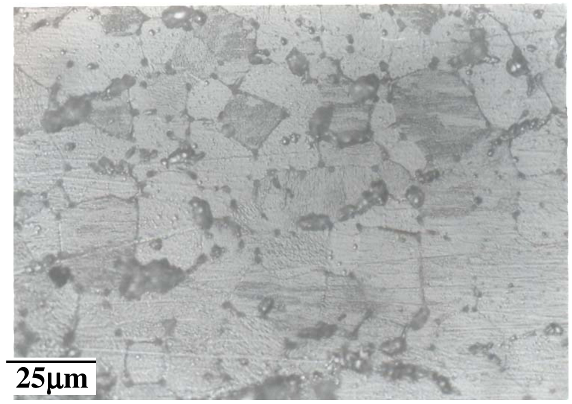

Figure 12. Optical micrograph of 75% cold rolled alloy 1 annealed at 400^{o}C for one hour.

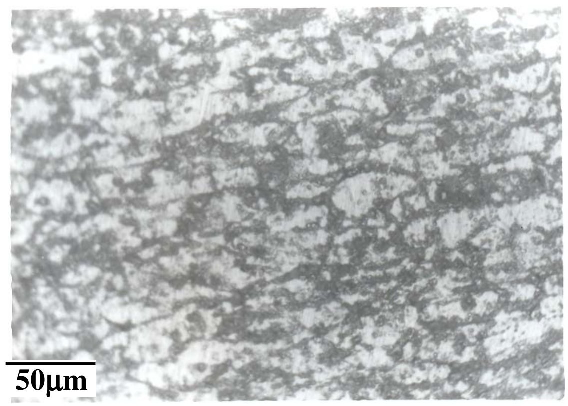

Figure 13. Optical micrograph of 75% cold rolled alloy 3 annealed at 400^{o}C for one hour.

3.2.3 Transmission Electron Micrographs

High resolution micrograph of alloy 2 (Figure 14) showing needles of Al_3Sc; strain field around the needles is visible. The morphology demonstrates that the coherent precipitates of Al_3Sc are needle shaped in their early stage.

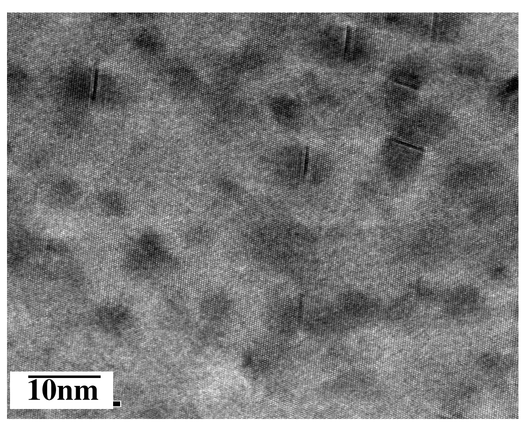

Figure 14. High resolution micrograph of alloy 2 showing needles of Al_3Sc

4. DISCUSSION

The initial softening of the cast and cold worked alloys during isochronal annealing is thought be due to rearrangement of dislocations at the annealing temperature. The precipitation hardening of the alloys containing scandium is attributable to the formation of Al_3Sc precipitates. Higher scandium added alloys shows higher strength due to higher volume fraction of precipitates. The strengthening is found to be greater for alloys with higher deformation because a higher degree of strain hardening resulted from higher dislocation density. But the extent of precipitation hardening has not improved. This means that extra advantage is not accruable by working scandium treated alloys. Moreover there has not been any change in the peak hardness temperature due to cold working. This signifies that scandium precipitation is not dislocation induced. Moreover extensive cold working also generates large number of vacancies, which form vacancy-scandium atom complexes of high binding energy. The vacancy-solute atom complexes reduce the mobility and availability of solute atoms at low temperature to form G P zones. Hence hardening takes place only at a temperature high enough to decompose the complexes thereby making solute scandium atoms available for precipitate formation. Beyond peak hardness, over ageing effect due to coarsening of the precipitates is seen to have taken place. At higher annealing temperature there is ample scope for dislocation annihilation and this softens the material. The precipitates formed by trace element hinder dislocation movement and thus limit the softening. The major drawback of Al-6Mg alloy in respect of undergoing softening during use is overcome by scandium addition. It is found from hardness plots that pinning of dislocation by formation of Al_3Sc onto them is unlikely. Nevertheless once formed, the precipitates hinder the motion of dislocations and hence lessen softening [12].

The effect of grain refinement is clearly evident from the resistivity curves, which show a significant difference of resistivity values of the scandium added alloy with that of the base alloy. The initial high resistivity of scandium treated alloy is indicative of high electron scattering sites viz. grain boundary area to mean that grains in all those alloys are finer. Formation of supersaturated solid solution assures a high precipitation hardening effect upon decomposition of this solid solution with the formation of fine coherent equilibrium Al_3Sc precipitates [12].

From the phase diagram of the alloy it is found that the present alloys would contain α + β eutectic within the primary dendrites of α. Here 'α' is the aluminum rich solid solution and β is composed of intermetallics, primarily Al_8Mg_5 along with aluminides of other metals like iron, chromium, zirconium, manganese, which are present in small quantities in the aluminum used for the present experimentation. The number of non-equilibrium segregation is dependent on the magnesium content and the concentration of other potential aluminide formers. However, scandium forms an anomalous supersaturated solid solution which decomposes to form Al_3Sc [13]. The dendrites of the cast binary alloy are seen to have refined significantly with the addition of scandium. This is ascribed to the modification of solidification speed by scandium during the growth of the dendrite structure [14]. Also scandium-containing alloy is seen to have contained less amount of intermetallic compounds. Due to increase in solidification speed, the super-cooling effect is weakened. The consequential faster solidification leads to decrement in the amount and size of the second phase constituents with scandium addition. The faster solidification also aids in the retention of more solutes in solution. Since dendrites are refined with scandium addition, the size of individual second phase region becomes smaller as these phases are formed within the inter-dendritic spaces. Though general observations under optical microscopy have not provided much information, the overall appearance of the microstructure resembles what are normally observed in cast aluminum alloy ingot [15]. The cold worked structures are comprised of elongated grains. When annealed at 300^{o}C no alloy except base alloy shows the sign of recrystallisation. The base alloy however has started recrystallising as it is known that recrystallisation of Al-6Mg alloy becomes completed at about 400^{o}C. However alloys 2 and 3 have dispersion of fine precipitates of Al_3Sc. These precipitates are coherent with the matrix. It is reported that recrystallisation is almost impossible in aluminum alloys when such particles are already present [2, 3]. Higher becomes the volume fraction of precipitates higher would be there crystallisation start temperature. The precipitates hinder the movement of sub-boundaries and grain boundaries. On increasing the temperature to 400^oC, the second phase constituent is almost dissolved in base alloy and there is nothing to hinder dislocation movement. As a result recrystallisation becomes complete. In alloys containing scandium the supersaturated solution decomposes to formAl_3Sc at around 300^oC. These precipitates are known to be resistant to coarsening. There are reports saying that increasing the annealing temperature of Al-Mg-Sc alloy from 300^oC to 400^oC increases the size of Al_3Sc precipitates from 4 nm to 13 nm. The precipitates of Al_3Sc remain coherent with the matrix even when their size increases to 100 nm due to higher temperature of annealing [1]. In the present case however the precipitate size is around 15 nm when annealed at 400^oC. Therefore dislocation pinning force is very large. As a result recrystallisation is not possible.

High resolution micrograph shows the presence of fine needles of 10 nm size surrounded by strain field (Figure 14). These are supposed to be Al_3Sc precipitates which are known to form in aluminium alloys containing scandium. The morphology demonstrates that the coherent precipitates of Al_3Sc are needle shaped in their early stage. In base alloy the monoclinic phases are likely to exhibit specific habit of ά being parallel to {110} planes. This causes difficulty for slipping along <110> direction of the fcc matrix. Hence critical resolved shear stress apparently increases and nucleation of micro twins becomes inevitable [16].

5. CONCLUSIONS

The scandium added alloys show the precipitation hardening behavior during annealing. It is attributable to the formation of Al_3Sc precipitates. The initial softening is thought to be due to the recovery in the cold worked alloys. The strengthening is found to be greater for alloys with higher deformation because a higher degree of strain hardening resulted from higher dislocation density. The trace element precipitates of Al_3Sc hinder dislocation movement and thus limit the softening. During the cold deformation, electrical conductivity decreases due to the increase of the number of vacancies and the density of dislocations. The microstructure shows that dendrites of the cast base alloy are refined significantly with the addition of scandium. The addition of scandium severely retards the recrystallization of the alloys when it is present in the form of fine Al_3Sc precipitates.

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