THERMAL ANALYSIS AND KINETICS OF THE PRECIPITATION IN WROUGHT Al-Mg, Al-Mg-Sc AND Al-Mg-Sc-Me (Me=Zr, Ti) ALLOYS

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Abstract: Precipitation behaviour of wrought Al-6Mg alloys with ternary scandium and quaternary zirconium and titanium has been studied. Hardness measurements and resistivity studies are employed to assess the precipitation behaviour of scandium doped Al-6Mg alloy without or with quaternary additions of zirconium and titanium. Further, the kinetics of precipitations are studied by differential scanning calorimetric technique. Scandium has been observed to form fine coherent Al_3Sc precipitates during ageing and these are responsible for strengthening of the alloys. The precipitation kinetics of Al_3Sc depends on the diffusion of scandium in aluminium. Presence of fine coherent precipitates of Al_3Sc impedes the migration of dislocations and increase the recovery temperature. The kinetics of recrystallisation is also delayed.

Keywords: Trace addition, precipitation hardening, resistivity and activation energy.

1. INTRODUCTION

Differential scanning calorimetry (DSC) has often been used for characterization of precipitates in Al-alloys [1-3]. This technique has been suitably employed for the study of precipitation kinetics in Al-Mg alloys [4]. Although DSC analysis could not ascertain the precipitation mechanism of β/ phase in Al-Mg alloys, that for the equilibrium phase β could be identified in the above work as a process taking place anywhere within the bulk and ultimately leading to the growth of existing nuclei. In a binary Al-Sc system the precipitation kinetics was studied by earlier workers [5]. Any possibility of clustering before precipitate formation has been ruled out by the previous workers [6]. By employing microscopic techniques it has been conjectured that the formation of Al_3Sc is a process of nucleation and diffusion growth. Again there are reports on using kinetic analysis of hardness variation in aged Al-Mg-Sc for describing the precipitation kinetics of Al_3Sc in the ternary alloy [7]. Further studies with transmission electron microscope elaborated the growth kinetics of Al_3Sc precipitates in Al-Mg-Sc during the course of ageing the solidified alloy [8]. It was also reported that the maximum rate of Al_3Sc formation occurs at 300°C in a decomposing Al-

Mg-Sc alloy. On the other hand trace quaternary addition of zirconium in Al-Mg-Sc alloy has slowed down its phase decomposition process. The completion of precipitation is found to have been delayed by 50°C in the above quaternary alloy [9]. Recrystallisation kinetics is found to be linked with precipitation coarsening kinetics in Al-Mg-Sc alloy. It has been observed earlier that the temperature for rapid growth of Al_3Sc precipitates is almost co-incident with the recrystallisation temperature of a ternary Al-Mg-Sc alloy [10]. However microscopic techniques have mainly been used by the above workers while analysing the recrystallisation behaviour. Recently kinetic analysis of recovery and recrystallisation processes in Al-Mg alloys are studied through DSC experiments [11]. It has been demonstrated that recovery process is diffusion controlled with associated activation energy of 120 kJ/mol, however the temperature and activation energy for recrystallisation in the same alloys have been found to be higher.

Form the available information, it appears that no systematic study has been carried out on the thermal analysis and kinetics of the precipitation behaviour of Al-Mg-Sc alloys with scandium content and with potent quaternary additions of zirconium or titanium. Moreover, reports on precipitation kinetics of Al_3Sc within a deformed matrix is almost lacking in literatures although

one may reluctantly presume an influence of existing dislocations onto the precipitation behaviour of Al₃Sc in ternary as well as quaternary alloys. Hence in the present investigation a detailed kinetic analysis for precipitation in cold rolled experimental alloys has been made through the employment of differential scanning calorimetric technique. DSC study has also been used to gain a clear understanding about the kinetics of recrystallisation and recovery in Al-Mg-Sc alloys with or without quaternary additions.

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, aluminium-magnesium alloy containing scandium and with or without zirconium and titanium. 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. Powder Zr and Ti (99.98% purity) packed and covered in an aluminum foil were added to the melt with the use of a plunger. The final temperature of the melt was maintained at 780±15°C. Casting was done in 12.5mm x 50mm x 200mm cast iron metal moulds preheated to 200°C. All the alloys were analysed by chemical and spectrochemical methods simultaneously. The chemical compositions of the alloys are given in Table 1.

Cold rolling of the alloys in as cast state was carried out in a laboratory scale rolling mill of 10HP capacity at 75% reduction percentages. The sample sizes were 9 x 12 x 50 mm and the deformation given was about 1.25 mm per pass. Cold rolled samples were aged isochronally for 60 minutes at different temperature up to 500°C followed by natural cooling. Hardness of different alloys were measured in Vickers hardness testing machine at 5 kg load to assess the age hardening effect of the alloys. Electrical conductivity of the alloys under various conditions were carried out with an electrical conductivity meter, type 979. 12 mm x 12 mm samples surface finished by grinding and

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

Alloy	Mg	Sc	Zr	Ti	Cu	Fe	Mn	Ni	Si	Zn	Cr	Sn	Al
1	6.10	0.000	0.000	0.001	0.081	0.382	0.155	0.003	0.380	0.136	0.002	0.002	Bal
2	5.97	0.400	0.000	0.002	0.071	0.314	0.107	0.002	0.335	0.124	0.002	0.002	Bal
3	5.85	0.400	0.185	0.003	0.069	0.335	0.112	0.001	0.345	0.170	0.003	0.002	Bal
4	6.06	0.400	0.000	0.175	0.080	0.306	0.104	0.002	0.335	0.170	0.002	0.002	Bal

Remarks:

Alloy 1   Al-6 wt% Mg

Alloy 2   Al-6 wt% Mg-0.4 wt% Sc

Alloy 3   Al-6 wt% Mg-0.4 wt% Sc-0.2 wt% Zr

Alloy 4   Al-6 wt% Mg-0.4 wt% Sc-0.2 wt% Ti

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Table 2. Results of DSC heating run

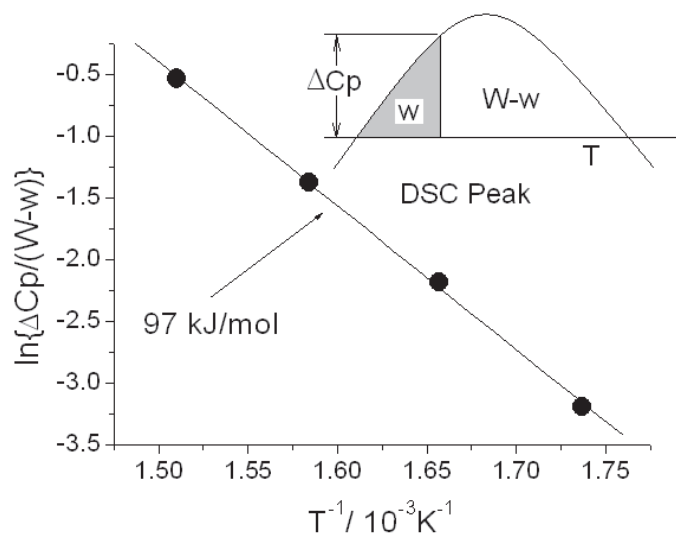| Alloy no.	History	Transformation	Peak temp. (°C)	Actv. energy (kJ/mol)
1	75% cold deformation	Dissolution of some phases	280	139
		β formation	327	231
		Recrystallisation	460	238
2	75% cold deformation	Dissolution of some phases	215	120
		Precipitation of Al_3Sc	310	174
		β formation	385	222
		Recrystallisation	450	219
3	75% cold deformation	Precipitation of Al_3Sc	345	97
		Recovery	420	209
		Recrystallisation	490	286
4	75% cold deformation	Precipitation of Al_3Sc	290	113
		Precipitation of Al_3Ti	310	302
		Recrystallisation	470	220

Fig. 1. Example of Activation Energy calculation from the DSC curve.

polishing were prepared for this measurement. Electrical resistivity was calculated from those conductivity data. The cold rolled alloys have been subjected to DSC heating run in a Du Pont 900 instruments. Inert N_2 gas atmosphere was used during DSC experiments. The samples for DSC studies were lump of 10 to 15 mg in weight. The DSC scan was conducted over a temperature range from 50°C to 650°C. A fixed heating rate of 10°C/min was used in all scans. The activation energy of transformations for the alloys in different conditions was calculated from the DSC curves by using the method of Nagasaki-Maesono analysis [12], is based on the well know Arrhenius equation.

For the Nagasaki-Maesono analysis, the following equation was used.

$$\ln \{ \Delta Cp/ (W\text{-}w)\} = \ln A - E / RT$$

where, ΔCp is the heat capacity, W and w are the total and shades area respectively and T is the temperature. As an example $\ln \{ \Delta Cp/ (W\text{-}w)\}$ Vs $1/T$ plot is shown in Fig. 1. The plot was done for Alloy 3, where precipitation of Al_3Sc occurred having peak temperature 345°C. The slope for four individual points of the DSC peak was measured manually using a standard graph

paper where ΔCp is the height of the point from the abscissa, W is the total area and w is the area of the shaded region. The results of the DSC studies are furnished in Table 2.

3. RESULTS

3. 1. Isochronal Ageing

The results of isochronal ageing of the cold worked alloys at different temperature for 1 hour is shown in Fig. 2. The base alloy (alloy 1) shows a continuous softening due to recovery and recrystallisation of the strained grains. All other alloys demonstrate age hardening response with peak hardness value at 300°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 2 shows maximum hardness. Most of the alloys show softening during initial period of ageing and increase in hardness after ageing finally enables the maximum hardness to reach a magnitude which is comparable to the initial hardness of cold worked alloys (Fig. 2). When the alloys are aged at higher temperature a sharp decrease in hardness is observed for all the alloys. Thus appreciable drop in hardness values
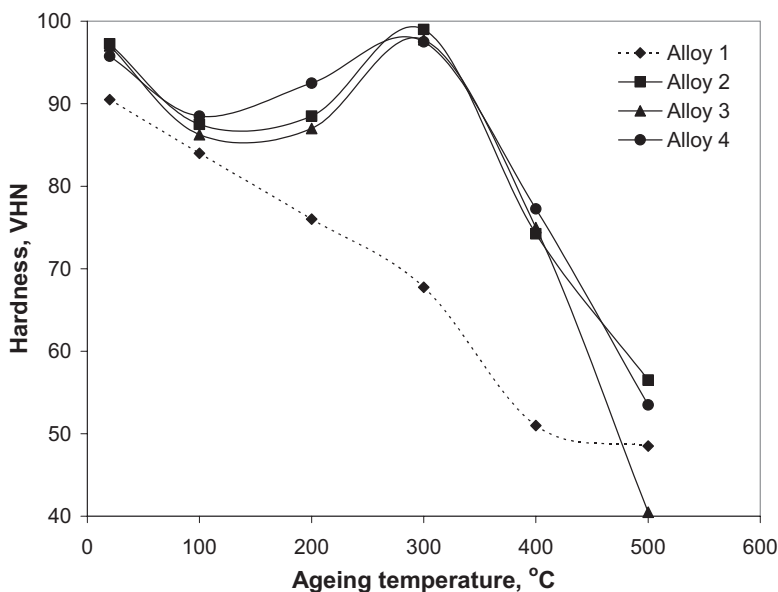

Fig. 2. Isochronal ageing curve of the 75% cold rolled alloys, Aged for 1 hour.
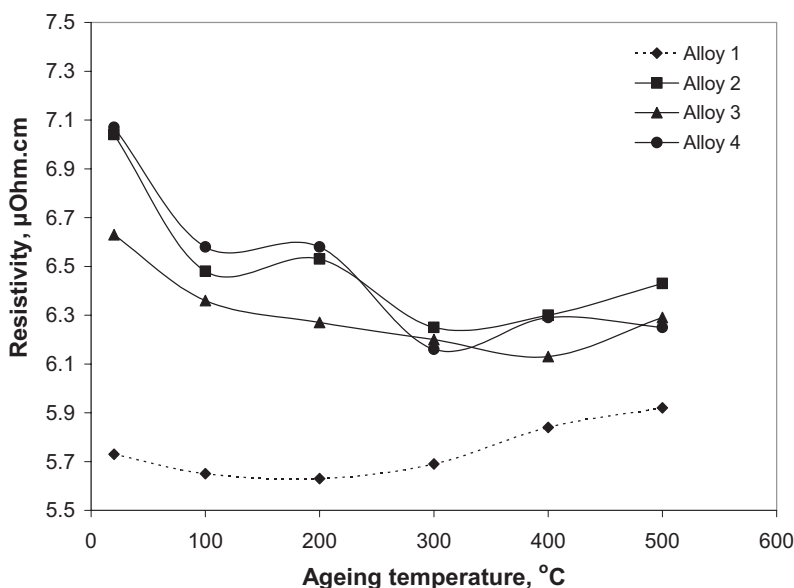
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Fig. 3. Variation of resistivity due to ageing of the 75% cold rolled alloys, Isochronally aged for 1 hour.

are noted at ageing temperatures beyond 300°C.

From the resistivity curve (Fig. 3) the initial resistivity of the scandium added alloys show higher than base alloy due to grain refining. The initial drop in resistivity during isochronal ageing of the experimental alloys is indicative of dislocation rearrangement taking place within the cold worked alloys. The striper decrease in resistivity is found to be much higher in all alloys than Alloy 1. The resistivity of the base alloy remains fairly unaltered over the entire period of ageing. For zirconium and titanium added alloys the behaviour is different. Following a sudden drop at higher ageing temperature there is an increase in resistivity due to recrystallisation of the base alloys and precipitation coarsening for the scandium added alloys.

3. 2. Differential Scanning Calorimetry

The base Al-6Mg alloy when cold rolled by 75% has given rise to the DSC heating curve as shown in the Fig. 4 In the figure it is noted that a broad endothermic occurs at 280°C. This is indicative of dissolution of some phase already present in the cast alloy. Following the method described elsewhere [12], the activation energy concerning dissolution reaction was calculated to

be 139 kJ/mol. This is followed by two exothermic peaks. The first one is the peak due to the formation of β. An activation energy of 231 kJ/mol was calculated. The following recrystallisation peak takes place at 460°C and the associated activation energy is 238 kJ/mol.

The DSC curve of the alloy 2 cold worked by 75% reduction records an endothermic at 215°C characterizing the dissolution of existing second phase particles (Fig. 5) This is followed by a formation peak at 310°C to indicate that Al_3Sc forms at this temperature at the maximum rate. This exothermic pick is associated with activation energy of 174 kJ/mol. The highly cold worked alloy shows a formation peak at 385°C with activation energy 222 kJ/mol. It seems that this alloy forms Mg-aluminides at this temperature during DSC heating. Another exothermic peak appearing at 450°C is seen to be associated with activation energy of 219 kJ/mol. This exothermic peak is representative of recrystallisation.

When the alloy 3 is cold worked by 75% the precipitation takes place at higher temperature, 345°C with much reduced activation energy of magnitude 97 kJ/mol (Fig. 6). This means that prior presence of dislocations facilitates the nucleation process. But this has not been
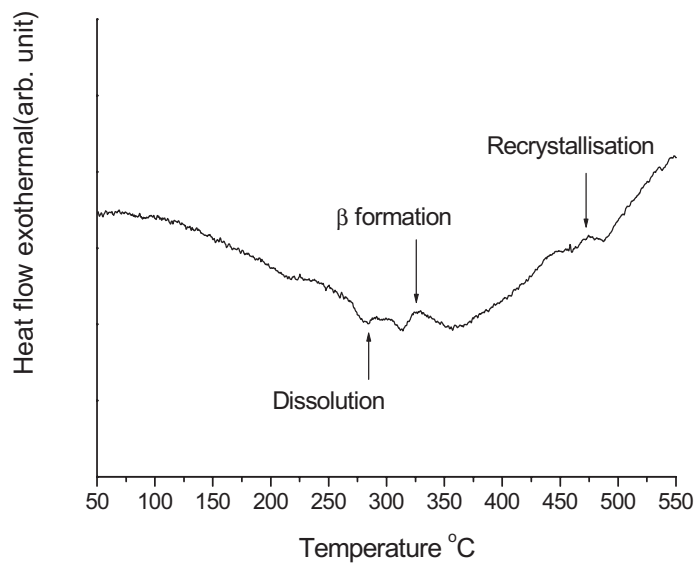
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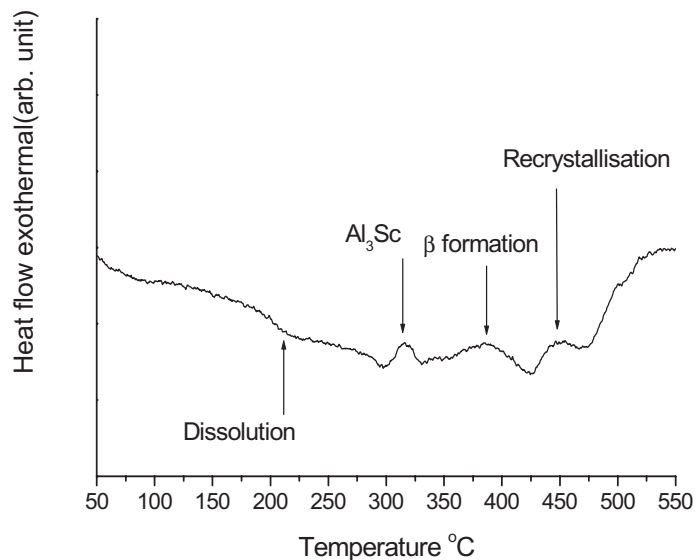**Fig. 4.** DSC heating curve of alloy 1 with 75% cold deformation.

Fig. 5. DSC heating curve of alloy 2 with 75% cold deformation.

observed in ternary Al-Mg-Sc alloy. The recovery peak occurs at 420°C with the associated activation energy of 209 kJ/mol. On the contrary, the recrystallisation process is greatly retarded as its peak is seen to occur at a temperature higher than 490°C. The value of activation energy is also unusually as high as 286 kJ/mol.

In cold worked alloy 4 two precipitate forming peaks are noted (Fig.7). The preceding small

peak at 290°C with an activation energy 113 kJ/mol. denotes Al_3Sc formation where as the adjacent larger peak at 310°C is due to the formation of Al_3Ti. The activation energy of larger peak is calculated to be 302 kJ/mol. Another prominent exothermic occurs at about 470°C indicating that recrystallisation of the cold worked alloy takes place at this temperature with an activation energy 220 kJ/mol.
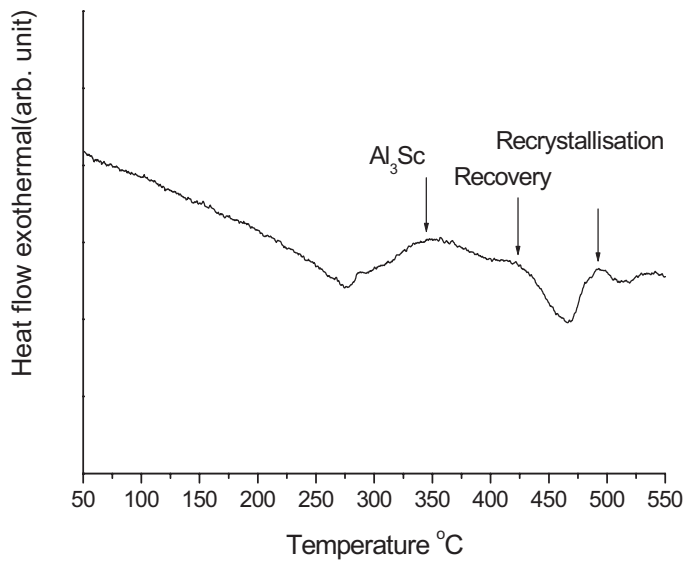
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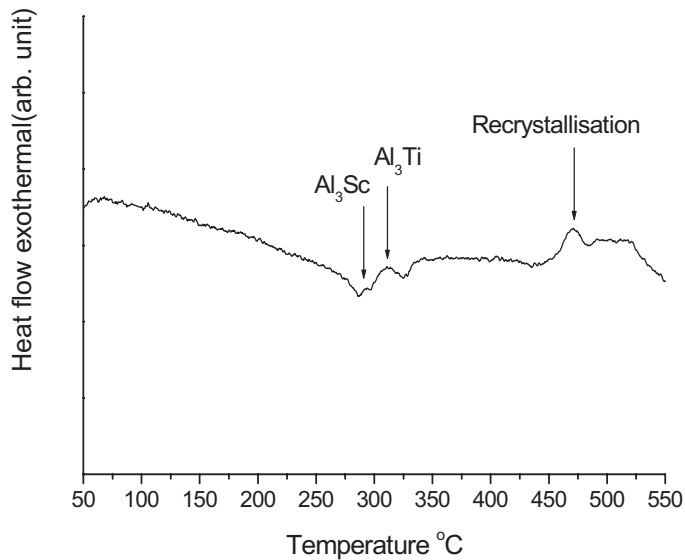**Fig. 6.** DSC heating curve of alloy 3 with 75% cold deformation.

Fig. 7. DSC heating curve of alloy 4 with 75% cold deformation.

4. DISCUSSION

The initial softening of the cast and cold worked alloys during isochronal ageing is thought be due to rearrangement of dislocations at the ageing temperature. The age hardening of the alloys containing scandium is attributable to the formation of Al_3Sc 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 age 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. However peak temperature in zirconium bearing alloy is responsive to cold working. It is conjectured that Al_3Zr is formed at dislocations. The Al_3Zr being isomorphous with and soluble in Al_3Sc, the nucleation of Al_3Sc is facilitated indirectly by the presence of higher dislocation density. 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 ageing temperature there is ample scope for dislocation annihilation and this softens the material. In respect of resisting softening due to overageing titanium is found to be more effective than zirconium.

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 Al3Sc precipitates [13]. The initial drop in resistivity during isochronal ageing of the experimental alloys is indicative of dislocation rearrangement taking place within the cold worked alloys. At lower temperature ageing, drop in resistivity due to recovery is counteracted by its increase due to ongoing precipitation process. As a result, depending upon the dominance of particular event, the resistivity either remains constant or marginally increases when the alloys are aged at lower temperature. About 300°C ageing temperature the scandium added alloys show small increase of resistivity due to precipitate formation of Al_3Sc. At beyond this temperature

the increase of resistivity due to precipitate coarcening of scandium added alloys and recrystallisation of base alloy [14].

Alloy 1 with 75% cold deformation contains some metastable phase. It is reported that metastable $\beta^/$ phase in Al-Mg alloy gives way to the formation of β-phase [15]. Two separate DSC peaks noted for the alloy is suggestive of the probable dissolution of $\beta^/$ phase for subsequent formation of β-phase. Thus the endothermic noted at 280°C in the base alloy seems to correspond to the dissolution of $\beta^/$-phase. The activation energy of the process, 139 kJ/mol, is close to the activation energy for diffusion of magnesium in aluminium, which has been reported to be 130 kJ/mol [4]. Owing to the availability of huge dislocations, the subsequent formation of β-phase could be facilitated. Its activation energy is found to be similar to the earlier reported value, 240 KJ/mol [4]. Higher deformation percent with fine precipitates of β onto the dislocation have hindered their movement thereby displacing recovery and recrystallisation temperatures to higher values. This is why activation energy for recrystallisation is found to be 238 KJ/mol against a reported value of 190 kJ/mol for an otherwise homogeneous alloy [16].

In Fig. 5, the exothermic at 310°C corresponds to the formation of Al_3Sc. It may be mentioned that activation energy for diffusion scandium in aluminium is reported to be 175 kJ/mol. Also activation energy for the formation of Al_3Sc in scandium bearing aluminium alloy has been found elsewhere to be 169 kJ/mole. This implies that precipitation kinetics of Al_3Sc is decided by the diffusion of scandium in aluminium. In the present case the activation energy for the aforesaid exothermic is found to be close to the previously reported value (174 kJ/mole). Upon cold working by 75% a $\beta^/$ -metastable formation peak is noticed at 215°C because the presence of dislocations might have induced the formation of a metastable phase in higher scandium alloy. The $ScAl_3$ formation peak with activation energy 174 kJ/mole is usually noted at 310°C. But only a faint recovery peak is observed in this alloy. This implies that with the greater volume fraction of finely distributed Al_3Sc precipitates, the

dislocation movement is restricted. Hence substructural stability is increased. The same reasoning of nonavailability of vacancies holds good for a shift of β-formation temperature to a higher value. However its activation energy 222 kJ/mole remains more or less comparable with other observations. Recrystallisation takes place a high temperature viz. 450°C although it is reported that the favourable temperature for recrystallisation is 400°C. Also it is noticed that in this particular case the activation energy for recrystallisation is also rather high. Thus kinetics of recrystallisation is greatly delayed in Al-6Mg-0.4Sc alloy. This is so because fine coherent precipitates of Al_3Sc have high coherency strains. This severely impedes the migration of dislocations. The activation energy for migration of dislocation in such alloy is 100 kJ/mole [17] whereas that for recrystallisation is 190 kJ/mole [11]. So the activation energy for strain free grains should be ~ 90 kJ/mole. In the present case however it is found that situation is entirely different. This may be explained only if we give consideration to misfit dislocations which may be partially annihilated only at high temperature after sufficient degree of particle coarsening.

The higher temperature of Al_3Sc type precipitate formation may be ascribed to the difference in the character of the precipitate in alloy 3. It is known that zirconium forms Al_3Zr of LI_2 structure similar to Al_3Sc. Al_3Sc and Al_3Zr are isomorphous and mutually soluble. Therefore the precipitate in this alloy is $Al_3(Sc_{1-x}Zr_x)$ type. The formation of this precipitate involves the diffusion of both scandium and zirconium. Activation energy for diffusion of zirconium in aluminium is 250 kJ/mole, which is higher than that of scandium in aluminium. However the process of precipitation is led by Al_3Zr, which is kinetically more sluggish than Al_3Sc. Hence transformation temperature is seen to be in higher. However once the Al_3Zr particles are formed, they induce the formation of Al_3Sc onto the existing Al_3Zr particles. This reduces the overall activation energy. Dislocation induced Al_3Zr formation further reduces the activation energy for formation of $Al_3(Sc_{1-x}Zr_x)$ precipitate. Thus Al_3Zr is formed first preferably by heterogeneous nucleation. The activation energy

for vacancy formation is known to be 70 kJ/mole. The migration energy of scandium thus comes out to be nearly 100 kJ/mole since the activation energy for diffusion of scandium in aluminium is statedly ~170kJ/mol. It then appears that precipitation kinetics in alloy 3 is principally controlled by the migration of scandium through aluminium as its activation energy ~100 kJ/mole is found to be close to the calculated activation energy from the experimentally observed peak (97 kJ/mole) [14]. However the recovery and recrystallisation processes are not only delayed but also are associated with higher activation energy. This is because majority of dislocations are pinned by Al_3Zr containing precipitations. Having had a higher dislocation density, the nucleation sites become numerous and hence fine precipitates are formed. For this reason, the dislocation rearrangement and annihilation becomes difficult and thus the kinetics of recovery processes become slower.

In alloy 4 the precipitate formation peak occurs with lower activation energy (113 kJ/mole). In this case Al_3Ti leads the process of nucleation and acts as the nucleation sites for Al_3Sc. Thus nucleation being heterogeneous, the kinetics is determined by migration of scandium atoms due to reasons stated earlier. Al_3Ti however forms on dislocations and the activation energy of 302 kJ/mole is comparable with the activation energy for diffusion of titanium in aluminium (270 kJ/mole). Thus the precipitation of Al_3Ti is controlled by diffusion of titanium through aluminium. The character of recrystallisation peak does not change and its activation energy, 220 kJ/mole is nearly equal to that in binary alloy [4]. Recrystallisation takes place a little earlier (470°C) due to higher driving force caused by increased amount of stored energy. Since the precipitates are formed and coarsened at the recrystallisation temperature, they do not interfere much in the recrystallisation of the matrix, which virtually resembles a solute Sc/Ti free binary system. For this reason the recrystallisation behaviour appears to be identical with the binary alloy though the temperature, 470°C is higher than the reported [11] value for binary alloy (370-400°C).

5. CONCLUSIONS

1. Availability of dislocations facilitates the formation of β'-metastable phase in Al-Mg alloys. This precipitation onto the dislocations hinder the process of recrystallisation.

2. The precipitation kinetics of Al_3Sc depends on the diffusion of scandium in aluminium. Presence of fine coherent precipitates of Al_3Sc impedes the migration of dislocations and increase the recovery temperature. The kinetics of recrystallisation is also delayed.

3. The precipitation temperature of Zr-bearing alloy is increased due to higher activation energy for diffusion of zirconium in aluminium. In case of dislocated matrix the dislocations aided precipitation of Al_3Zr results in a lowering of peak temperature for precipitate formation. For Ti-bearing alloy, Al_3Ti precipitates act as the nucleation sites for precipitation of Al3Sc and thus lowers the activation energy of precipitation.

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