

## EFFECT OF SCANDIUM ON THE GRAIN REFINING AND AGEING BEHAVIOUR OF CAST Al-Si-Mg ALLOY

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**Abstract:** Microstructure and properties of the Al-6Si-0.3Mg alloys containing scandium (0.2 to 0.6wt %) were investigated. The microstructure was observed by optical microscopy, the hardness was determined by Vickers tester and phase transformation was investigated by differential scanning calorimetry (DSC) technique. The results showed that scandium can refine dendrites, enhance hardness in the aged alloys and suppress softening effect during prolonged ageing treatment.

**Keywords:** Al-Si-Mg alloys, age hardening, grain-refinement, precipitates, recrystallization.

### 1. INTRODUCTION

Aluminium alloys are the lightest structural alloys commercially available. They have great potential for applications in automotive, aerospace and other industries. Significant improvement in properties of aluminium alloys can be achieved through addition of scandium. A few numbers of works regarding the use of scandium in aluminum alloys have already been published [1-5]. Scandium is an effective grain refiner and increases the recrystallization temperature in aluminium alloys [6, 7]. Scandium addition in pure aluminium and its alloys enhances not only their mechanical and thermal properties, but also increases their corrosion resistance and weldability; and reduces the hot cracking susceptibility of these alloys [3]. Scandium forms a stable LI2 phase, (i.e. Al<sub>3</sub>Sc) with aluminum. The interface of Al<sub>3</sub>Sc with the matrix is coherent. Presence of fine coherent precipitates of Al<sub>3</sub>Sc impedes the migration of dislocations and increases the recovery temperature by stabilising the substructure. The kinetics of recrystallisation is also retarded by scandium addition [1, 3]. Scandium does not form any second phase intermetallic compound with other alloying elements such as iron, magnesium, manganese, silicon and chromium [3].

In this work, the alloying effects of Sc on

microstructures and mechanical properties of Al-6Si-0.3Mg cast alloys were investigated for improving their mechanical properties.

### 2. EXPERIMENTAL

Four classes of alloys were produced by melting certain amounts of (i) pure aluminium (98.5% purity), (ii) piston head of Toyota car engine (11.2% Si with small amount of magnesium and other trace elements) and (iii) aluminium-scandium master alloy (2%Sc) in a clay-graphite crucible in a natural gas fired pit furnace under suitable flux cover (degasser, borax etc.). In all cases the final temperature of the melt was maintained at  $760 \pm 15$  °C with the help of an electronic temperature controller. The melt was homogenised by stirring at 700 °C. Casting was performed in preheated (i.e. 200 °C) cast iron moulds [20.0 mm in diameter x 200.0 mm in length] All the alloys were analysed by wet chemical and spectra-chemical methods simultaneously. The chemical compositions of the alloys are given in Table 1.

Cylindrical samples with dimensions of 16.0 mm in diameter and 10.0 mm in length were cut from the cast alloys. The samples in the as-cast condition were subjected to (i) natural ageing for 60 days, (ii) isochronal ageing for 60 minutes at different temperatures up to 500 °C, and (iii) isothermal ageing at various temperatures up to

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

| Alloy | Si   | Mg    | Sc    | Pb    | Ti    | Cu    | Fe    | Mn    | Ni    | Zn    | Cr    | Sn    | Al  |
|-------|------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-----|
| 1     | 5.73 | 0.282 | 0.000 | 0.006 | 0.020 | 0.765 | 0.406 | 0.074 | 0.402 | 0.043 | 0.011 | 0.027 | Bal |
| 2     | 5.57 | 0.305 | 0.180 | 0.007 | 0.025 | 0.838 | 0.404 | 0.069 | 0.404 | 0.040 | 0.011 | 0.029 | Bal |
| 3     | 5.87 | 0.291 | 0.360 | 0.007 | 0.027 | 0.839 | 0.459 | 0.080 | 0.454 | 0.044 | 0.012 | 0.027 | Bal |
| 4     | 5.88 | 0.289 | 0.550 | 0.008 | 0.034 | 0.875 | 0.409 | 0.077 | 0.393 | 0.039 | 0.012 | 0.026 | Bal |

**With Remarks**

Alloy 1 Al-6 wt% Si-0.3 wt% Mg

Alloy 2 Al-6 wt% Si-0.3 wt% Mg-0.2 wt% Sc

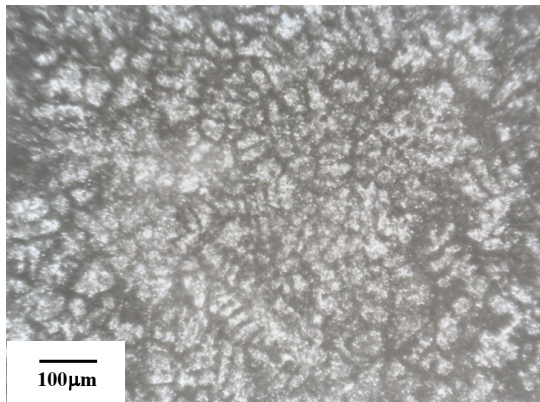
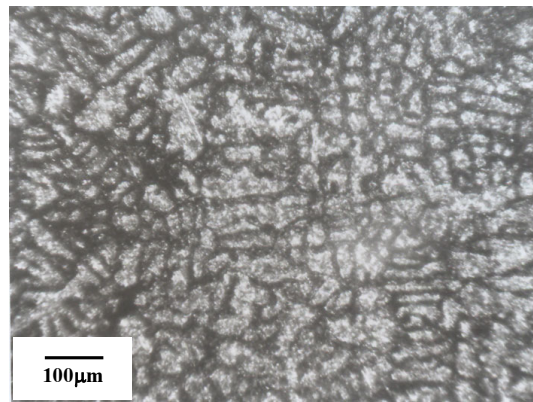
Alloy 3 Al-6 wt% Si-0.3 wt% Mg-0.4 wt% Sc

Alloy 4 Al-6 wt% Si-0.3 wt% Mg-0.6 wt% Sc

400 °C for different period of time ranging from 30 to 240 minutes. Then the hardness's of the treated samples were determined. A Rockwell F scale [60kg load, 1/16// steel ball indenter] was used for this purpose and an average of ten concordant readings was taken as the representative hardness of a sample. The microstructures of the specimens were investigated using optical microscopy. An etchant reagent Keller and optical microscope Versamet II were used for this purpose. Pieces of the alloys, in the form of lumps of 10 to 15mg in weight, have also been subjected to DSC at a temperature range of 50 to 600 °C under inert gas N<sub>2</sub>, using a Du Pont 900 instrument. A fixed heating rate of 10o/min was used in all DSC scans.

**3. RESULTS****3. 1. Optical Microscopy**

Optical micrograph of as-cast alloy1 shows black second phase particles within interdendritic regions (Fig. 1). Addition of 0.2 wt% scandium to the base alloy (Alloy 2) showed a diminution in the amount of second phase particles. It was also observed that secondary dendrite arm spacing decreased in alloys 2 and 3 resulting to refinement of dendrites (Figs. 2,3). The structural fineness was seen to increase with increasing scandium content. In the alloy with 0.6 wt% Sc, the amount of second phase particles reduced to a great extent. The dendrite fragments were seen to have refined remarkably (Fig. 4).

**Fig. 1.** Optical micrograph of cast alloy 1.**Fig. 2.** Optical micrograph of cast alloy 2.

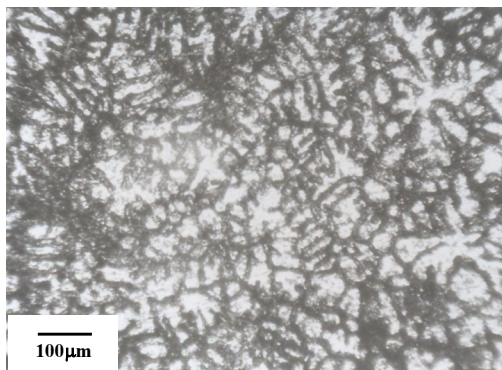


Fig. 3. Optical micrograph of cast alloy 3.

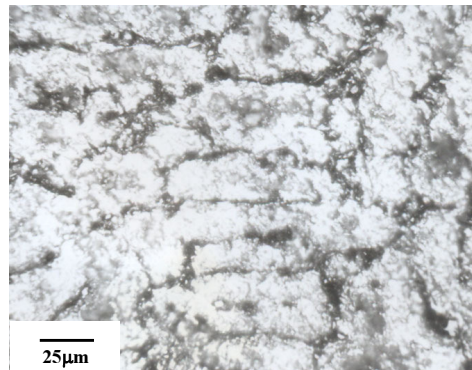


Fig. 6. Optical micrograph of cast alloy 2, aged at 400 °C for 1 hour.

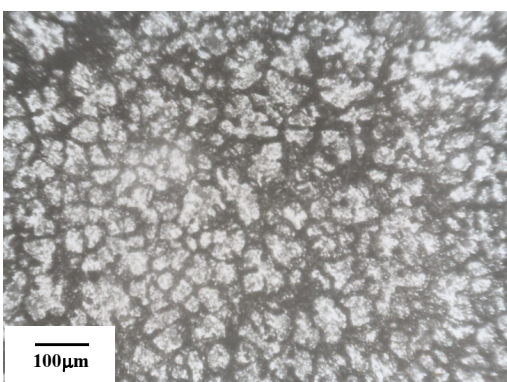


Fig. 4. Optical micrograph of cast alloy 4.

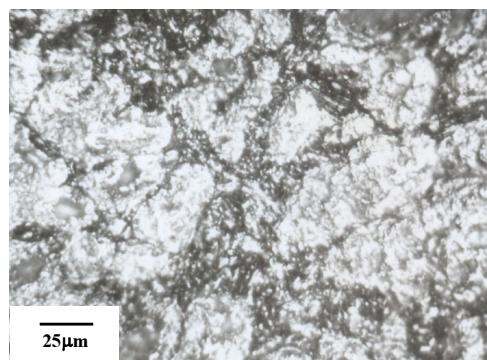


Fig. 7. Optical micrograph of cast alloy 3, aged at 400 °C for 1 hour.

When annealed at 400 °C, the base alloy was seen to be recrystallised almost fully (Fig. 5). The scandium added alloys, on the other hand did not recrystallise even when annealed at 400 °C (Figs. 6-8).

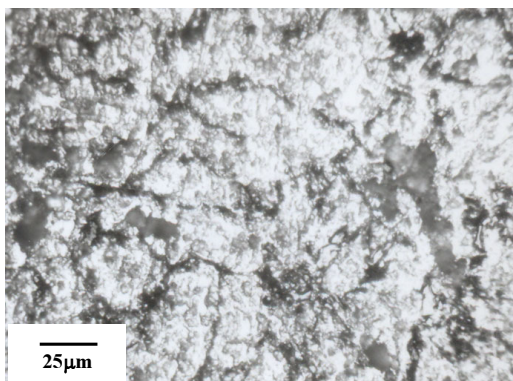


Fig. 5. Optical micrograph of cast alloy 1, aged at 400 °C for 1 hour.

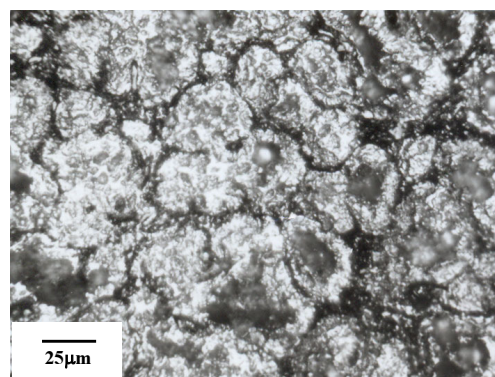


Fig. 8. Optical micrograph of cast alloy 4, aged at 400 °C for 1 hour.

### 3. 2. Ageing

Fig. 9 shows the variation in hardness of the different alloys under natural ageing condition. All the alloys age harden to some extent in 10 days. The Sc containing alloys initially showed

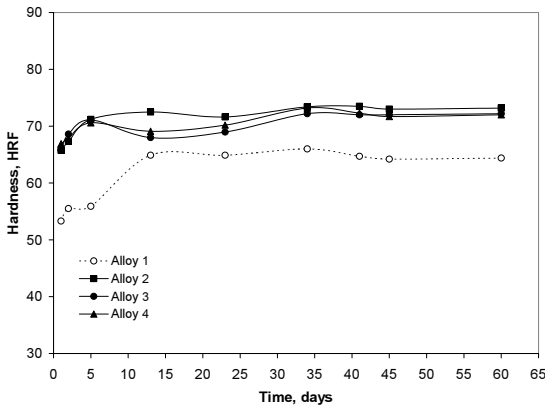


Fig. 9. Natural ageing curve of the cast alloys for 60 days.

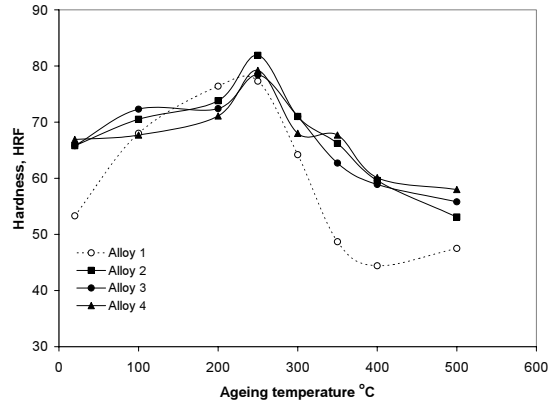


Fig. 10. Isochronal ageing curve of the cast alloys, aged for 1 hour.

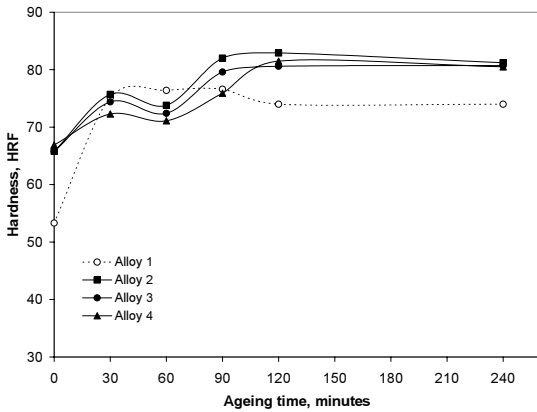


Fig. 11. Isothermal ageing curve of the cast alloys, aged at 200 °C.

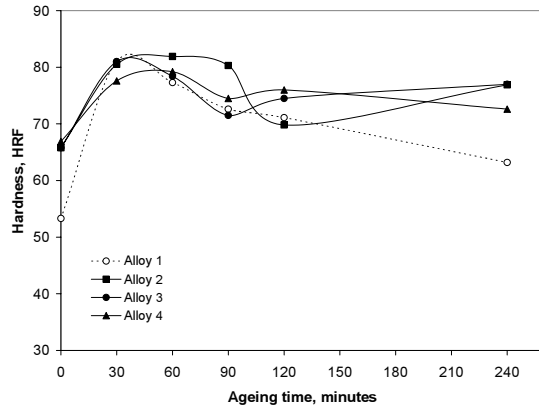


Fig. 12. Isothermal ageing curve of the cast alloys, aged at 250 °C.

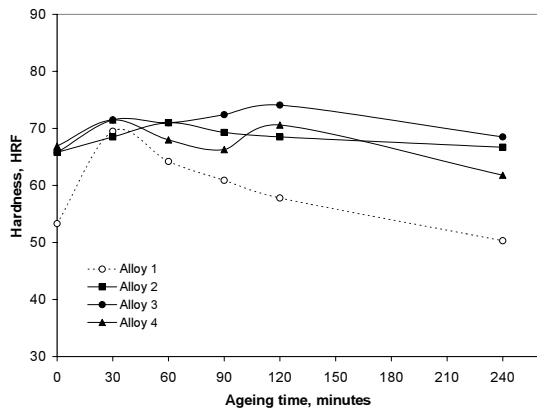


Fig. 13. Isothermal ageing curve of the cast alloys, aged at 300 °C.

higher hardness and over the period of holding the hardness did not vary significantly.

Fig. 10 shows the isochronal ageing behavior of the different alloys. For all the alloys the peak ageing condition was attained at 250 °C. Although hardness of the base alloy was lower than the Sc bearing alloys in the as cast condition, the softening effect above 250 °C was found to be much more prominent in the base alloy than in the Sc containing alloys.

Figures 11 to 14 show the isothermal ageing behavior of different alloys at different levels of temperature. At 200 °C the base alloy attains the peak condition after 30 min. In the case of Sc bearing alloys the peak hardness is maintained without any appreciable softening during ageing.

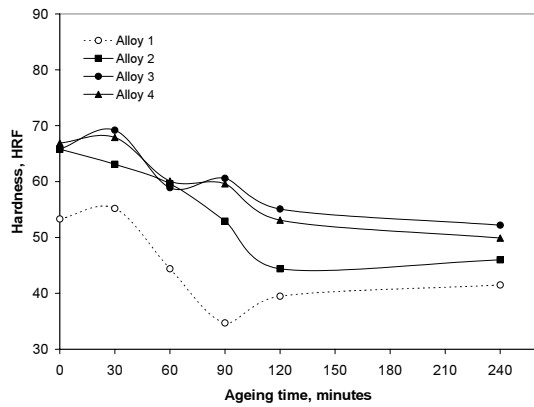


Fig. 14. Isothermal ageing curve of the cast alloys, aged at 400 °C.

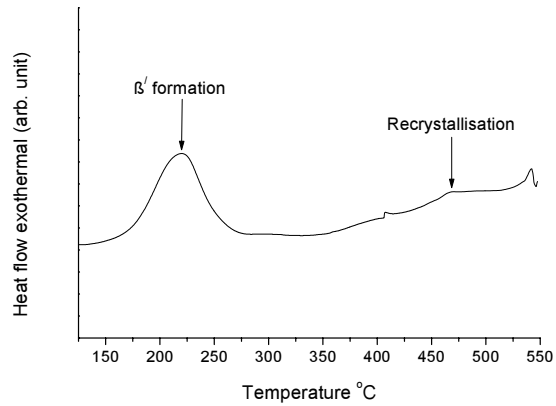


Fig. 15. DSC heating curve of cast alloy 1.

For ageing in the temperature range 250 - 400 °C the peak ageing condition for the base alloy was attained in 30 min. The decrease in hardness became more pronounced with increasing ageing temperature in the range 250 – 400 °C. However, the Sc bearing alloy exhibits stronger resistant to softening in comparison to the base alloy.

### 3. 3. Differential scanning calorimetry (DSC)

The DSC curve for the base Al-6Si-0.3Mg alloy (Fig. 15) showed a broad exothermic at 220 °C. This has been attributed to the dissolution of some phase already present in the cast alloy [8]. This is followed by another exothermic peak at 460 °C. The exothermic at 460 °C corresponds to recrystallisation. The DSC heating curve of alloy

containing 0.2 wt% Sc is shown in Fig. 16. An exothermic at 225 °C corresponds to the dissolution of some second phase particles, presumably  $\beta$ -phase, present in the microstructure. Following this a broad exothermic peak is seen to appear at 475 °C in the heating curve. This indicates that the recrystallisation takes place at a higher temperature.

The DSC thermogram of the cast alloy containing 0.4 wt.% Sc indicates a superimposition of two exothermics (Fig. 17). The preceding peak occurs at 225 °C and seems this is an indicative of the formation second  $\beta$ -phase. There is a peak at about 475 °C to support recrystallisation.

The DSC heating curve of the alloy containing 0.6 wt% Sc shows an exothermic peak at 225 °C and another exothermic peak present there at



Fig. 16. DSC heating curve of cast alloy 2.

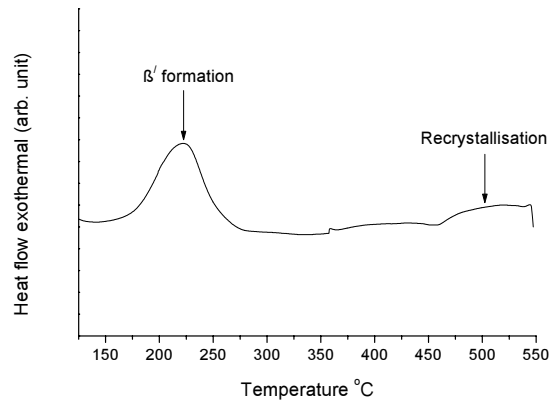


Fig. 17. DSC heating curve of cast alloy 3.

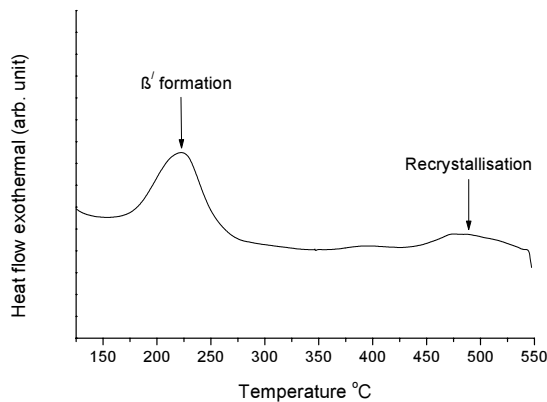


Fig. 18. DSC heating curve of cast alloy 4.

475°C, shows the occurrence of recrystallisation (Fig. 18).

#### 4. DISCUSSION

Observations under optical microscopes did not provide much information; nevertheless the overall appearance of the microstructure resembles what are normally observed in cast aluminium alloy ingot [9]. The dendrites of the cast base alloy are refined significantly with the addition of scandium. It has been reported [3] that 0.2 wt.% Sc in the alloy does not provide much grain refinement, but refines the primary dendrites of  $\alpha$  with consequent diminution of dendrite arm spacing. The arm spacing in scandium treated alloys were found to lie within a range of 20  $\mu\text{m}$  to 40  $\mu\text{m}$  in comparison to the untreated base alloy having a grain size of about 45  $\mu\text{m}$ . This was related to the modification of solidification speed by scandium during the growth of the dendrite structure [10]. Also scandium-containing alloys contained less amount of intermetallic compounds in comparison to those exist in the base alloy.

Due to increase in solidification speed, the super-cooling effect was weakened. The faster solidification rate led to decrease in the amount and size of the second phase constituents with scandium addition. Faster solidification rate aids in the retention of more solute in solution. Since dendrites were refined with scandium addition, the size of individual second phase region

became smaller as these phases were formed within the interdendritic spaces.

Increasing the amount of scandium led to a greater increase in solidification speed. It is reported that recrystallisation in Al-6Si-0.3Mg alloy becomes complete at 400 °C [12]. In good agreement with this observation the base alloy (Fig.5) has started recrystallising. In alloys containing scandium the supersaturated solution decomposes to form  $\text{Al}_3\text{Sc}$  at around 300 °C. These precipitates are known to be resistant to coarsening. At 300 °C, the fine dispersion of  $\text{Al}_3\text{Sc}$  in alloy 2 inhibits recrystallisation fully.

Several research works have shown that an increase in the annealing temperature of Al-Mg-Si-Sc alloy from 300 °C to 400 °C increases the size of  $\text{Al}_3\text{Sc}$  precipitates from 4 nm to 13 nm. The precipitates of  $\text{Al}_3\text{Sc}$  remain coherent with the matrix even when their size increases to 100 nm due to higher temperature of annealing [2, 3]. In the present case however the precipitate size is around 15 nm when annealed at 400 °C. Therefore it seems dislocation pinning force is very large. As a result one can not expect any recrystallization to take place.

Hardening peaks appeared in the aging curves of the alloys in about 10 days. This is due to formation of hexagonal  $\text{Mg}_2\text{Si}$  [3]. Scandium added alloys show high values of hardness due to relatively fine grains [5].

In isochronal ageing for one hour, maximum hardness was found at 250 °C for all the alloys. The peak hardness of alloy 1 falls more quickly than the other alloys. This was due to recrystallisation of alloy 1 as the temperature increased. But because of the formation of  $\text{Al}_3\text{Sc}$ , recrystallization was delayed in alloys 2-4. This seemed to be due to formation of  $\text{Al}_3\text{Sc}$  which resulted to formation of finer grains and increase in hardness. On ageing at 200 °C hardening peak appeared after 90 minutes. This is also because of formation metastable  $\beta'$  phase which precipitate in Al-Si-Mg alloy [3]. Hardness value due to aging of alloy 1 fell quicker than those of the other alloys, because of its quicker recrystallisation relative to other alloys. When alloys are aged at 250 °C maximum hardness was found in 30 minutes. In other word under this condition  $\beta'$  was formed in less time due to

higher aging temperature.

The effect of  $Al_3Sc$  formation is clearly seen the right section of Fig. 12 (where hardness of alloy 1 decrease due to recrystallisation). Ageing at 300 °C gives maximum hardness in 30 minutes. But this maximum hardness is lower than the previous maximums. Because at higher temperature the precipitates tend to become coarser and coarse precipitates are not as effective as finely dispersed precipitates to resist the movement of dislocations. Coarse precipitates also do not offer enough resistance to the recrystallisation [7]. Effect of high temperature and coarse precipitates are also seen in Fig.14.

Alloy 1 contains some metastable phases. It is reported that metastable  $\beta'$  phase in Al-Si-Mg alloy gives way to the formation of  $\beta$ -phase [12, 13]. Two separate peaks appeared in DSC curve of the alloy 1 can be an indication of the probable dissolution of  $\beta'$  phase and subsequent formation of  $\beta$ -phase at 220 °C.

Recrystallisation temperature was found to be similar to what has been reported earlier [3, 8]. DSC plot of cast alloys 2-4 are almost similar to cast alloy 1. An exothermic peak denoting the formation of metastable  $\beta'$  appeared at 225 °C, because the presence of dislocations might have induced the formation of a metastable phase in higher scandium alloy. This implies that with the greater volume fraction of finely distributed  $Al_3Sc$  precipitates, the dislocation movement can be restricted resulting to sub-structural stability.

Recrystallisation takes place at a high temperature viz. 475 °C although it is reported that the favorable temperature for recrystallisation is 400 °C. Thus kinetics of recrystallisation is greatly delayed in Al-6Si-0.3Mg-0.4Sc alloy. This is so because fine coherent precipitates of  $Al_3Sc$  have high coherency strains. This severely impedes the migration of dislocations. This may be due to misfit dislocations which can be partially annihilated at high temperatures after sufficient degree of particle coarsening [14].

## 5. CONCLUSION

Scandium led to significant grain refinement in cast Al-6Si-0.3Mg alloy. By increasing the

amount of scandium one can obtain a greater dendrite refinement and lower fraction of secondary phase. Scandium addition was effective in respect of improving the hardness during ageing. However scandium addition was most effective in suppressing the softening effect during prolonged ageing treatment. The precipitates delayed the recrystallisation in scandium bearing alloys. The higher the volume fraction of precipitates, the higher became the recrystallisation starting temperature.

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