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EFFECT OF SCANDIUM ON THE CAST Al-Si-Mg ALLOY

M. S. Kaiser[1], M. R. Basher[2], and A. S. W. Kurny[2]

[1]Directorate of Advisory, Extension and Research Services,
[2]Department of Materials and Metallurgical Engineering, Bangladesh University of Engineering and Technology, Dhaka-1000, Bangladesh

ABSTRACT

Ageing of Al-6Si-0.3Mg alloy doped with varying concentration of scandium ranging from 0.2wt% to 0.6wt% has been carried out. Attempts were made to understand the grain refining effect of scandium in Al-6Si-0.3Mg alloy. As cast samples were naturally aged for 60 days. Some samples were aged isochronally for 60 minutes at different temperature up to 500^oC and also isothermally up to 400^oC for different periods ranging from 30 to 240 minutes. Hardness of the alloys was measured to study the age hardening effect due to scandium addition. Moreover, a kinetic analysis for precipitation in experimental alloys has been carried out by Differential Scanning Calorimetric Technique to gain a clear understanding about the kinetics of precipitation and recrystallization in Al-6Si-0.3Mg alloy with or without scandium additions.

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

1. INTRODUCTION

Scandium is an effective grain refiner and increases the recrystallization temperature in aluminium alloys [1, 2]. Scandium addition in pure aluminium and its alloys also enhance the mechanical properties, thermal properties, corrosion resistance and its weldability and reduce hot cracking susceptibility [3]. Scandium forms a stable LI_2 phase, Al_3Sc with aluminium. The precipitation of Al_3Sc is coherent with the matrix. Presences of fine coherent precipitates of Al3Sc impede the migration of dislocations and increase the recovery temperature by stabilizing the substructures. The kinetics of recrystallization is also delayed by scandium addition [1, 3]. Scandium does not form any second phase intermetallic compound with other alloying elements such as iron, manganese and chromium [3].

This article discusses the results of investigations on the ageing behavior of Al–6Si-0.3Mg alloys containing varying amounts of scandium. It also presents the results on the evolution of microstructures in the experimental alloys due to change in their chemistry, mechanical and thermal history. Differential Scanning Calorimetry has also been used to understand the precipitation and recrystallization in Al–6Si-0.3Mg alloys.

2. EXPERIMENTAL

Four alloys were produced by melting in a natural gas fired pit furnace under suitable flux cover (degasser, borax etc.). Commercially pure aluminium (98.5% purity) was taken as the starting material. First the aluminium, aluminium-silicon and aluminium- scandium master alloy (2%Sc) were melted in a clay-graphite crucible. Piston head of Toyota car engine was used as aluminium-silicon master alloy (11.2% Si with small amount of magnesium and other trace elements). The final temperature of the melt was always maintained at 760 ± 15^oC with the help of an electronic temperature controller. Then the melt was homogenized by stirring at 700^oC. Casting was done in cast iron metal moulds [20.0 mm in dia x 200.0 mm in length] preheated to 200^oC. All the alloys were analyzed by wet chemical and spectrochemical methods simultaneously. The chemical compositions of the alloys are given in Table 1. The cast alloys were cut to pieces of suitable size [16.0 mm dia x 10.0 mm length]. As cast samples were aged isochronally for 60 minutes at different temperatures up to 500^oC. The samples in the as cast condition were isothermally aged at various temperatures up to 500^oC for different ageing times ranging from 30 to 240 minutes. All cast alloys were naturally aged up to 60 days. The aged alloys were then put to hardness testing to assess the age hardening behaviour. A Rockwell F scale [60kg load, 1/16” steel ball indenter] was used and an average of ten concordant readings has been taken as the representative hardness of a sample. The heat treated cast alloys were subjected to optical metallographic studies. The specimens were polished with alumina, etched with Keller’s reagent and observed under a Versa met-II Microscope. The alloys, in the form of lumps of 10 to 15mg in weight, have also been subjected to DSC using a Du Pont 900 instrument. Inert N_2 gas atmosphere was used and the DSC scan was conducted over a temperature range from 50^oC to 600^oC. A fixed heating rate of $10^oK/min$ was used in all scans.

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	**0.00**	0.006	0.020	0.765	0.406	0.074	0.402	0.043	0.011	0.027	Bal
2	5.57	0.305	**0.20**	0.007	0.025	0.838	0.404	0.069	0.404	0.040	0.011	0.029	Bal
3	5.87	0.291	**0.40**	0.007	0.027	0.839	0.459	0.080	0.454	0.044	0.012	0.027	Bal
4	5.88	0.289	**0.60**	0.008	0.034	0.875	0.409	0.077	0.393	0.039	0.012	0.026	Bal

3. RESULTS

3.1. Optical Microscopy

Optical micrograph of alloy 1 shows dendrites with black second phase particles within inter-dendritic spaces (Fig. 1). Addition of 0.2 wt% scandium to the base alloy showed a diminution in the amount of second phase particles. It further appears that dendrite arm spacing is decreased in alloy 2 and 3 with the consequent refinement of dendrites (Figs. 2-3). The structural fineness is seen to increase with increasing scandium content. In the alloy with 0.6 wt% Sc, the amount of second phase particles is reduced to a great extent. The dendrite fragments are seen to have refined remarkably (Fig. 4).

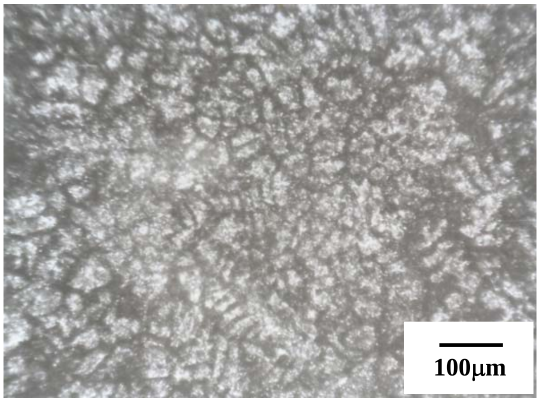

Fig 1**.** Optical micrograph of cast alloy 1.

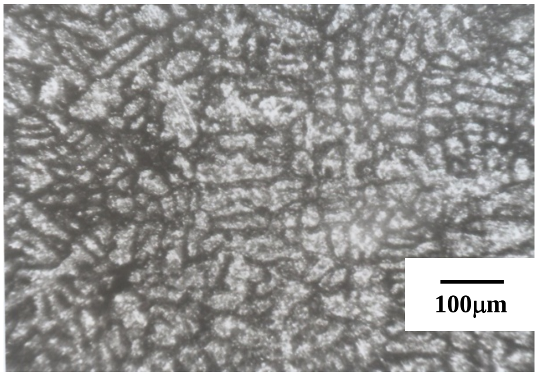

Fig 2**.** Optical micrograph of cast alloy 2.

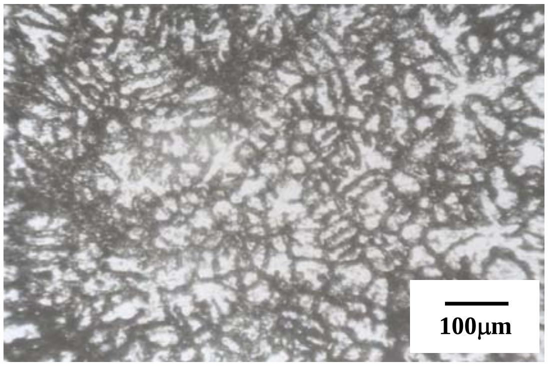

Fig 3**.** Optical micrograph of cast alloy

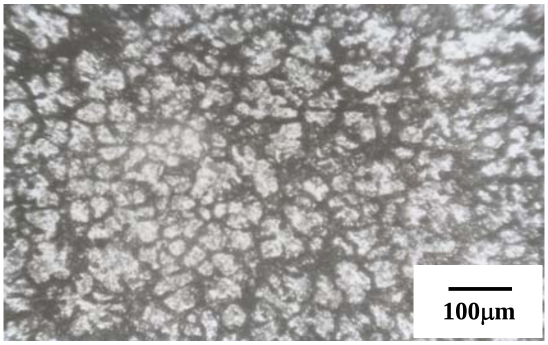

Fig 4**.** Optical micrograph of cast alloy 4.

If the alloys are annealed at 400°C, the base alloy is seen to be recrystallized almost fully (Fig. 5). The scandium added alloys, on the other hand, do not recrystallize even when annealed at 400°C (Figs. 6-8).

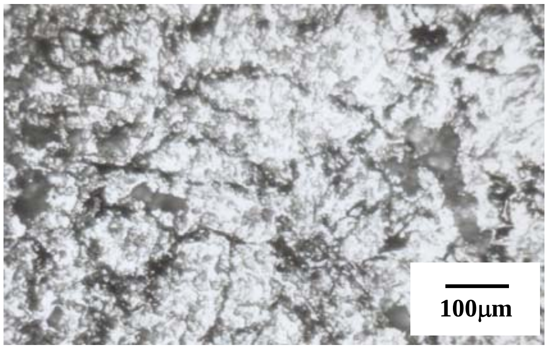

Fig 5**.** Optical micrograph of cast alloy 1, aged at 400°C for 1 hour.

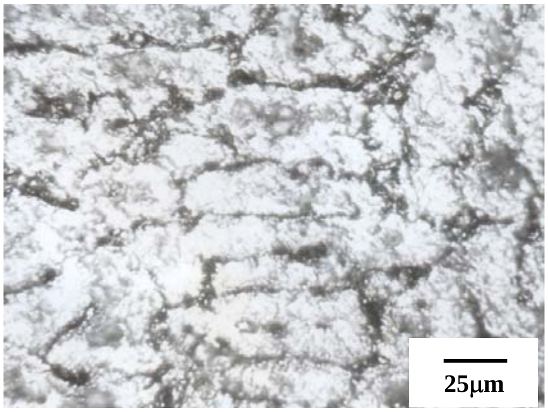

Fig 6**.** Optical micrograph of cast alloy 2, aged at 400^oC for 1 hour.

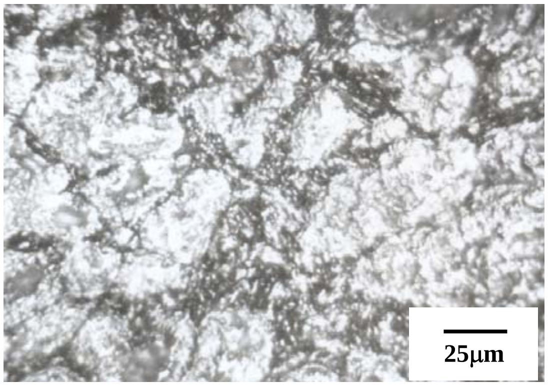

Fig 7**.** Optical micrograph of cast alloy 3, aged at 400^oC for 1 hour.

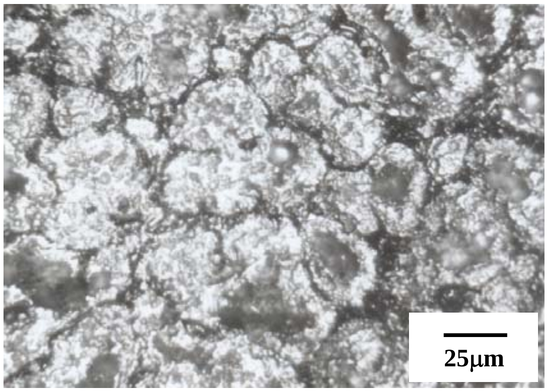

Fig 8**.** Optical micrograph of cast alloy 4, aged at 400^oC for 1 hour.

3.2. Ageing

Fig. 9 shows the variation of hardness of the different alloys under natural ageing condition. It is apparent that all the alloys achieve some extent of age hardening after 10 days. However, the Sc containing alloys initially show higher hardness. In the case of Sc bearing alloy the hardness in as cast condition over the period of holding does not vary significantly. Fig. 10 shows the Isochronal ageing behavior for different alloys. For all the alloys the peak ageing condition has been attained at 250^o C. Although hardness of the base alloy is lower than the Sc bearing alloys in the as cast condition, the softening effect above 250^{0}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. Up to 200^{o}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 through the duration of ageing. For ageing within the temperature range 250 to 400^{o}C the peak ageing condition has been attained after 30 min. The ageing effect is gradually reduced with increase in temperature above 250^{0}C. However, the Sc bearing alloy exhibits stronger resistant to softening in comparison to the base alloy.

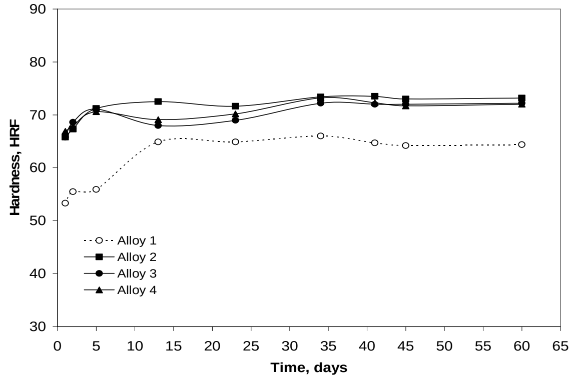

Fig 9. Natural ageing curve of the cast alloys for 60 days.

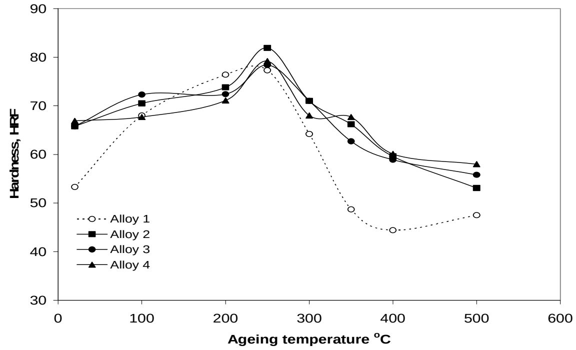

Fig 10. Isochronal ageing curve of the cast alloys, Aged for 1 hour.

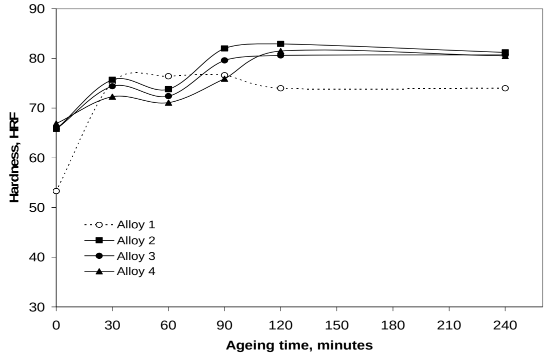

Fig 11. Isothermal ageing curve of the cast alloys, Aged at 200^{o}C.

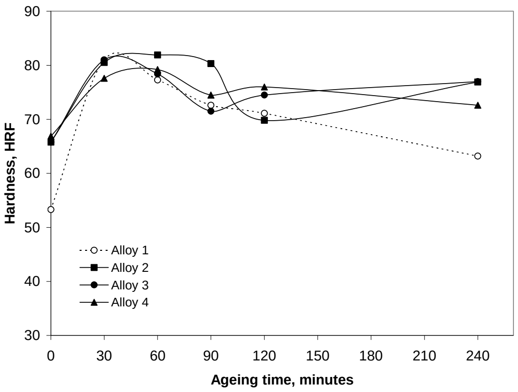

Fig 12. Isothermal ageing curve of the cast alloys, Aged at 250^0C.

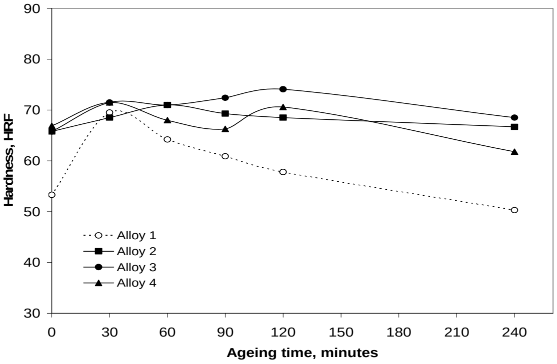

Fig 13. Isothermal ageing curve of the cast alloys, Aged at 300^oC.

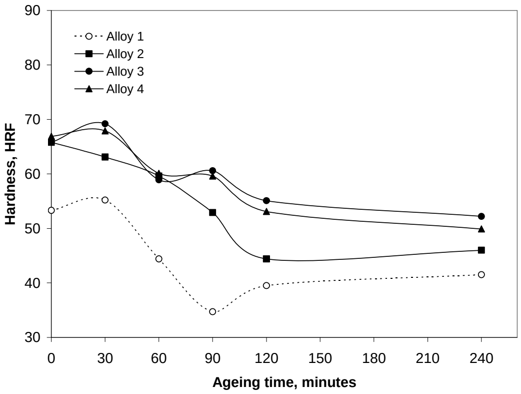

Fig 14. Isothermal ageing curve of the cast alloys, Aged at 400^oC.

3.3. Differential scanning calorimetry (DSC)

The DSC curve for the base Al–6Si-0.3Mg alloy is shown in Fig. 15. It may be noted that a broad exothermic occurs at 220^oC. This is indicative of dissolution of some phase already present in the cast alloy [8]. This is followed by another exothermic peak at 460^oC. The exothermic at 460^oC corresponds to recrystallization. The DSC heating curve of alloy containing 0.2 wt% Sc is shown in Fig. 16. An exothermic at 225^oC corresponds to the dissolution of some kind of second phase particle presumably β-phase present in the microstructure. Following this a broad exothermic peak is seen to appear at 475^oC in the heating curve. This signifies the recrystallization taking place at a higher temperature. The DSC thermogram of the cast alloy containing 0.4 wt% Sc records a superimposition of two exothermics (Fig. 17). The preceding peak occurs at 225^oC and is indicative of the formation second β-phase. There is a peak at about 475^oC to support recrystallization. The DSC heating curve of the alloy containing 0.6 wt% Sc shows an exothermic peak at 225^oC signifying the precipitation of hexagonal Mg_2Si and another exothermic presents there at 475^oC for the recrystallization (Fig. 18).

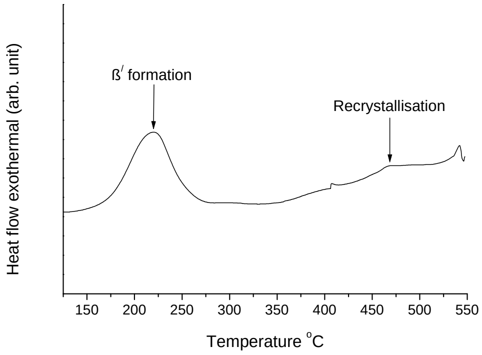

Fig 15. DSC heating curve of cast alloy 1.

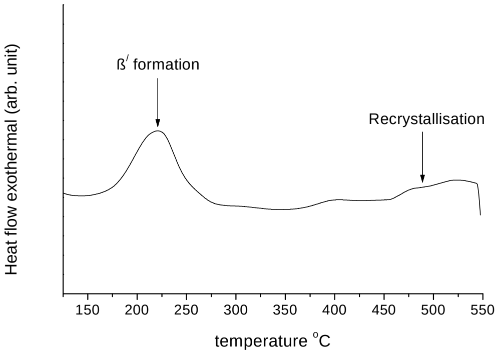

Fig 16**.** DSC heating curve of cast alloy 2.

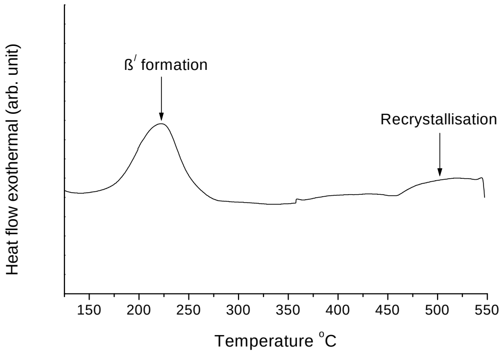

Fig 17. DSC heating curve of cast alloy 3.

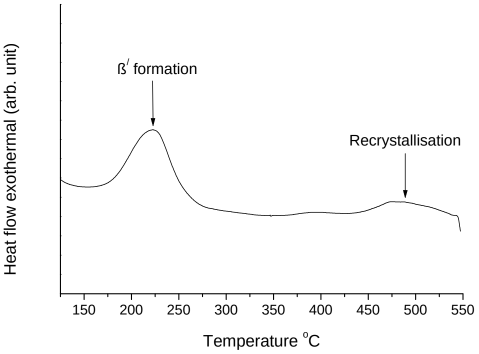

Fig. 18: DSC heating curve of cast alloy 4.

4. DISCUSSION

Observations under optical microscopes have not provided 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 binary alloy are seen to have refined significantly with the addition of scandium. Reportedly [3], alloy with 0.2 wt% Sc does not provide much grain refinement, but refines the primary dendrites of α with consequent diminution of dendrite arm spacing. The arm spacing in scandium treated alloys were found to lie within a range of 20 μm to 40 μm against a value of around 45 μm in case of base alloy. This is ascribed to the modification of solidification speed by scandium during the growth of the dendrite structure [10]. Also scandium-containing alloys are seen to contain fewer amounts 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. Increasing amount of scandium leads to a greater increase in solidification speed and hence the related effects on dendrite refinement and fraction and number density of second phase constituents have been realized more as evidenced in the optical microstructures of alloys 2-4. The base alloy however has started recrystallizing as it is known that recrystallization of Al-6Si-0.3Mg alloy becomes completed at about 400°C. However alloys 2-4 have dispersion of fine precipitates of Al_3Sc. These precipitates are coherent with the matrix. It is reported[11], that recrystallization is almost impossible in aluminium alloys when such particles are already present. However at 300°C, the finely dispersed Al_3Sc in alloy 2 is sufficient to inhibit recrystallization fully. The precipitates hinder the movement of sub-boundaries and grain boundaries. On increasing the temperature to 400°C, the second phase constituent is almost dissolved in base alloy and there is nothing to hinder dislocation movement. As a result recrystallization becomes complete. In alloys containing scandium the supersaturated solution decomposes to form Al_3Sc at around 300°C. These precipitates are known to be resistant to coarsening. There are reports saying that increasing the annealing temperature of Al-Mg-Si-Sc alloy from 300°C to 400°C 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 [2, 3]. In the present case however the precipitate size is around 15 nm when annealed at 400°C. Therefore dislocation pinning force is very large. As a result recrystallization is not possible.

Hardening peak was found for all alloys in 10 days. This is due to formation of hexagonal Mg_2Si. Scandium added alloys show higher hardness due to relatively fine grain. In isochronal ageing for one hour maximum hardness was found at 250°C for all alloys. The peak hardness of alloy 1 falls more quickly than the other alloys. This is because with the increase of temperature alloy 1 starts to recrystallize. But due to the formation of Al_3Sc recrystallization is delayed in alloys 2-4. This is also because of formation of Al_3Sc which results in finer grains and increase of hardness. When alloys are aged at 200°C hardening peak was found in 90 minutes. This is also because of formation of $\beta^{/}$. Again hardness of alloy 1 falls quicker than the other alloys because of early recrystallization. When alloys are aged at 250°C maximum hardness was found in 30 minutes. $\beta^{/}$ is formed in less time due to higher temperature. The effect of Al_3Sc formation is clearly seen the right portion of the graph where hardness of alloy 1 decrease due to recrytallization. Ageing at 300°C gives maximum hardness in 30 minutes. But this maximum hardness is less than the previous maximums. Because in higher temperature the precipitates tend to become coarser and coarse precipitates are not as effective as finely dispersed precipitates to resist the movement of dislocation. Coarse precipitates also do not offer enough resistance to the recrystallization. Effect of high temperature and coarse precipitates are also seen here and in the following graph.

Alloy 1 contains some metastable phase. It is reported that metastable $\beta^{/}$ phase in Al-Si-Mg alloy gives way to the formation of β-phase [12, 13]. Two separate DSC peaks noted for the alloy is suggestive of the probable dissolution of $\beta^{/}$ phase for subsequent formation of β-phase at 220°C. The following exothermic occurs at 460°C. Recrystallization temperature is found 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 $\beta^{/}$ is noticed 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 is restricted. Hence sub structural stability is increased.

Recrystallization takes place at a high temperature viz. 475°C although it is reported that the favorable temperature for recrystallization is 400°C. Thus kinetics of recrystallization 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 due to misfit dislocations which are partially annihilated only at high temperatures after sufficient degree of particle coarsening [14].

5. CONCLUSION

1. The dendrites of the cast base alloy are refined significantly with the addition of scandium. Increasing amount of scandium leads to a greater dendrite refinement and fraction of second phase constituents is reduced.
2. The age hardening effect shown by the alloys are due to formation of metastable $\beta^{/}$ phase.
3. Sc addition is effective in respect of improving the hardness during ageing. However Sc addition is most effective in suppressing the softening effect during prolonged ageing treatment.
4. The precipitates delay the recrystallization in scandium bearing alloys. Higher is the volume fraction of precipitates; higher becomes the recrystallization start temperature.

6. REFERENCES

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7. MAILING ADDRESS

Md. Rashedul Basher
House: Y-16(1st floor), Road: 1, Block: A, Chandgaon
C.D.A Residential Area, Chandgaon, Chittagong.
Phone: +8801913484165
E-mail: rashed.mme.buet@gmail.com