Fractional Recrystallization Behaviour of Al-Mg Alloy with Different Sc Addition Content

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Abstract—**Recrystallization kinetics of 75% cold rolled Al-6Mg alloy with ternary scandium is analyzed from the micro-hardness variation. Isothermally annealed aluminum alloy samples were also studied using JMAK type analysis to see if there exists any correlation between the methods. Recrystallization fraction between two methods, the scandium added alloys show the higher variation due to precipitation hardening and higher recrystallization behavior. From the microstructure it is also observed that the base Al-Mg alloy attained almost fully re-crystallized state after annealing at 400 °C for 60 minutes.**

Index Terms—**Al-Mg alloys, annealing, hardness, recrystallization, precipitate**

I. Introduction

Aluminum alloys with magnesium as the major alloying element constitute a group of non heat-treatable alloys with medium strength, high ductility, excellent corrosion resistance and weldability. Unfortunately, the strength of such Al-Mg system alloys is lower than precipitation-hardening Al alloys [1]. In recent years Al-Mg-Sc alloy has been studied systematically by many research groups [2]-[5]. The results show that, when different Sc contents are added, the effect of grain refinement of Al-Mg alloy is different remarkably. Adding Sc improves the strength, hardness, and other mechanical properties of Al-Mg alloy, and raises the thermal-stability of the alloy at the same time [6]. small amount of scandium has been found to significantly improve the strength of Al-Mg alloys, owing to the presence of coherent, finely dispersed $L1_2$ Al_3Sc precipitates that can be obtained at ahigh number density, thus preventing the dislocation motion. Also the Al3Sc precipitates have modest coarsening rates atelevated temperatures, leading to the effective suppression of recrystallization and the stabilization of fine grains, at high temperatures [7].

In the present study, recrystallization kinetics in directly cold rolled Al-Mg Alloy with scandium is studied via the methods of micro-hardness variation. Isothermal recrystallization kinetics can be represented by Johnson-Mehl-Avrami-Kolmogorov (JMAK) type behavior. In the present study the recrystallization kinetics for the alloys is analyzed by assuming a JMAK type behavior and the results were compared with that obtained from micro-hardness variation.

II. 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. 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 (98wt% Al + 2wt% Sc) were melted in a clay-graphite crucible, then magnesium ribbon (99.7% purity) was added by dipping in to the molten metal. The final temperature of the melt was always maintained at 780±15 °C with the help of the electronic controller. Then the melt was allowed to be homogenised under stirring at 700 °C. Casting was done in cast iron metal moulds preheated to 200 °C. Mould size was 12.5×51.0×200.0 in millimeter. All the alloys were analysed by wet chemical and spectrochemical methods simultaneously. The chemical compositions of the alloys are given in Table I. Cold rolling of the cast alloys were carried out with a laboratory scale rolling mill of 10HP capacity at 75% reduction. The alloys were pieces into 10×12×50mm and the deformation given was about 1.25mm per pass. Samples for the studying recrystallization kinetics, 2.5×12×15mm in size were obtained from the cold rolled sheet. The samples were isothermally annealed at 400 °C for different ageing times ranging from 1 to 240 minutes. Microhardness of the samples was measured with a Polyvermet microscope-cum microhardness tester, Reichert Jung Microduramet 4000E. A Knoop indenter was pressed on to the sample using a cycle time of 15 seconds and loading rate of 10gf/sec. The indentation tests were performed with 10gm load. Average results of fifteen tests are plotted. The optical metallography of the samples was carried out in the usual way. The specimens were polished finally with alumina and the etchant used was Keller's reagent (HNO_3-2.5cc, HCl-1.5cc, HF-1.0cc and H_2O-95.0cc). The washed and dried samples were observed carefully in Versamet-II-Microscope at different magnifications and some selected photomicrographs were taken.

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TABLE I. CHEMICAL COMPOSITION OF THE EXPERIMENTAL ALLOYS (WT%)

Alloy	Mg	Sc	Fe	Mn	Si	Zn	Al
1	6.10	0.000	0.382	0.155	0.380	0.136	Bal
2	5.90	0.200	0.345	0.132	0.360	0.174	Bal
3	6.02	0.600	0.293	0.086	0.320	0.126	Bal

Remarks:
Alloy 1 Al-6wt% Mg
Alloy 2 Al-6wt% Mg-0.2wt% Sc
Alloy 3 Al-6wt% Mg-0.6wt% Sc

III. RESULTS

A. Isothermal Annealing

Fig. 1 shows the isothermal annealing of the alloys at 400 ℃. When the alloys are isothermally annealed at 400 ℃, alloy 1 shows a very fast and steep decrease in hardness followed by a constant value due to recovery and recrystallisation. The rate and degree of initial softening is same for alloys 2-3. The age hardening peaks are observed in scandium added alloys 2 and 3.

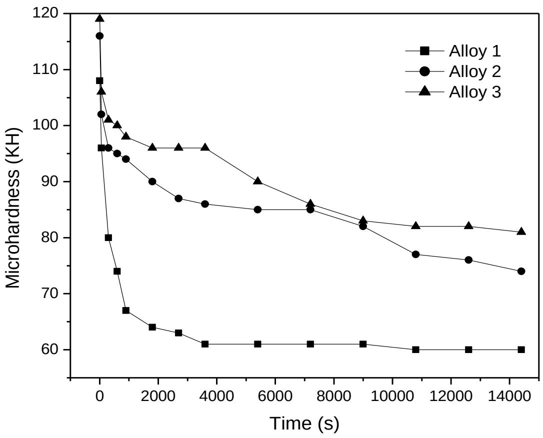

Figure 1. Isothermal annealing curve of the alloys, annealed at 400 ℃.

TABLE II. EXPERIMENTAL VALUE OF MAXIMUM, MINIMUM HARDNESS AND JMAK EXPONENT OF THE ALLOYS

Alloy No.	H max	H min	n	k
1	108	57	0.39557	0.001702
2	116	55	0.25608	0.000067
3	119	58	0.24796	0.000036

B. Recrystallization Kinetics from Microhardness Variation

The kinetics of recrystallization were determined from the microhardness values by considering the maximumand minimum values of microhardness, indicating deformed and completely recrystallized samples respectively. The maximum and minimum values for microhardness obtained in the present analysis are given in Table II. The fraction recrystallized is obtained from the microhardness value by using the following formula:

$$X = \frac{H_{max} - H_i}{H_{max} - H_{min}} \quad (1)$$

where H_{max} is maximum hardness corresponding to deformed sample (t=0), H_{min} is minimum hardness corresponding to fully recrystallized sample and *Hi* is microhardness after a given annealing time [8]. Fully recrystallied sample got hold of the alloys annealed at 500 for one hour. Fig. 2 shows the variation of microhardness and fraction recrystallized obtained from microhardness values for samples annealed at 400 ℃. The base alloy 1 shows the maximum values of recrystallization but scandium added alloy 2 and 3 show the minimum. Higher scandium shows the minimum recrystallization behaviour.

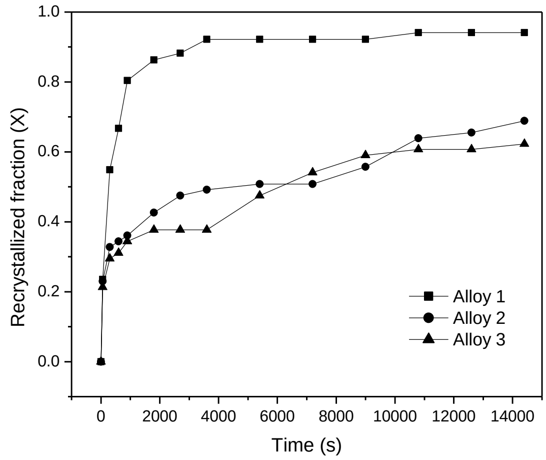

Figure 2. Recrystallization kinetics obtained from microhardness data.

The kinetics of recrystallization can be represented in a mathematical form by using the JMAK relationship [9], [10]. The variation of fraction recrystallized with annealing time in JMAK relationship is given as:

$$X = 1 - \exp[-(kt)^n] \quad (2)$$

where *n* and *k* are the JMAK exponent and temperature dependent constant, respectively. This equation can be rearranged to a linear relationship by using a logarithmic expression.

$$\ln[\ln(\frac{1}{1-X})] = n\ln(t) + n\ln(k) \quad (3)$$

The slope of this linear expression will yield the exponent n and the parameter k can be obtained from the ordinate as shown in Fig. 3.

The values of the JMAK exponent n and parameter k can be used to obtain recrystallization kinetics of the alloys annealed at 400 ℃ as shown in Fig. 4. Alloys 1-3 show the different slope for their different recrystallization behavior.

$$X = 1 - \exp[-(0.001702 \times t)^{0.39557}] \text{ for alloy 1} \quad (4)$$

$$X = 1 - \exp[-(0.000067 \times t)^{0.25608}] \text{ for alloy 2} \quad (5)$$

$$X = 1 - \exp[-(0.000036 \times t)^{0.24796}] \text{ for alloy 3} \quad (6)$$

Recrystallization fraction between two methods, the base alloy 1 shows the minimum variation. The scandium added alloy 2 and 3 show the higher variation due to precipitation hardening and higher recrystallization behavior.

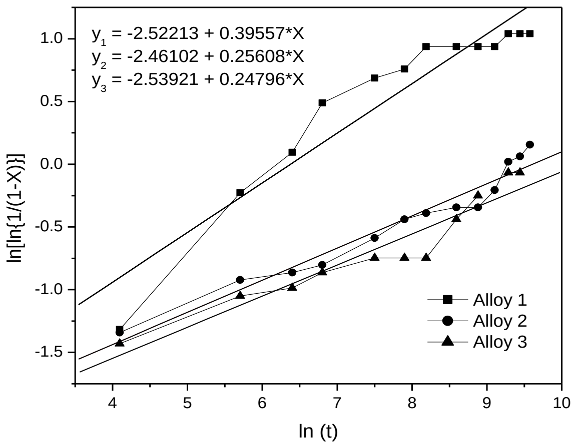

Figure 3. Plot of ln[ln{1/(1-X)}] Vs. ln(t), showing a linear relationship with a slope equal to the JMAK exponent.

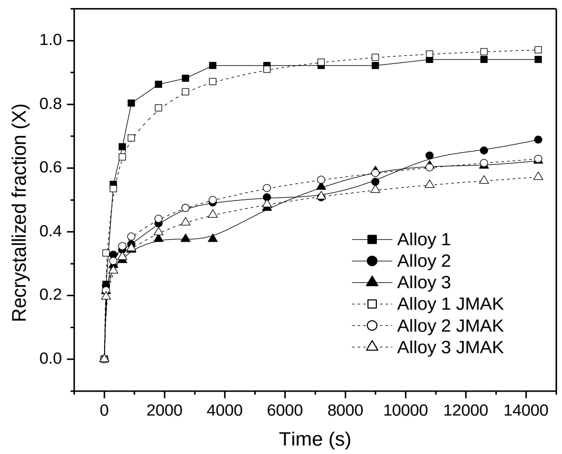

Figure 4. Recrystallization kinetics for the alloys from Micro-hardness data and JMAK analysis.

C. 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 (Fig. 5). Fragmented dendrites, elongated along the direction of rolling, are observed in Fig. 6, showing the microstructure of cold worked alloy 3.

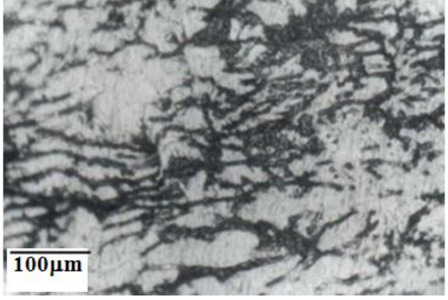

Figure 5. Optical micrograph of 75% cold rolled alloy 1.

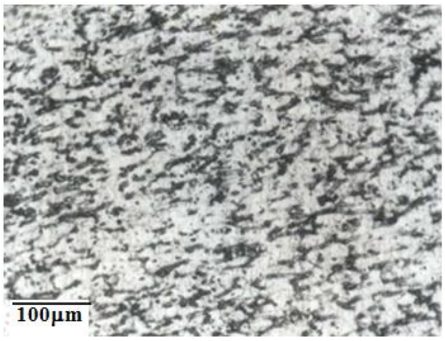

Figure 6. Optical micrograph of 75% cold rolled alloy 3.

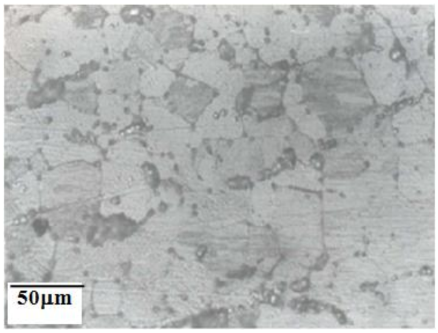

Figure 7. Optical micrograph of 75% cold rolled alloy 1, annealed at 400 ℃ for 1 hour.

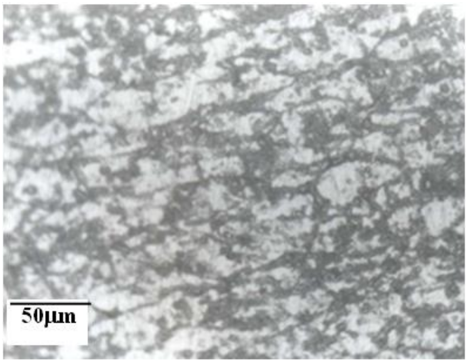

Figure 8. Optical micrograph of 75% cold rolled alloy 3, annealed at 400 ℃ for 1 hour.

If the alloys are annealed at 400 ℃ for one hour, the base alloy is seen to be recrystallised almost fully (Fig. 7). However, alloy 3 is recrystallised partially at the annealing treatment at 400 ℃ for one hour (Fig. 8).

IV. Discussion

The initial softening of the cold worked alloys during isothermal annealing is thought be due to rearrangement of dislocations at the annealing temperature. The age hardening of the alloys containing scandium is attributable to the formation of Al_3Sc precipitates. The maximum attainable hardness due to ageing the cold worked alloy has not exceeded the hardness values obtained due to cold working alone. This implies that the precipitation of Al_3Sc is not dislocation induced [3]. Moreover extensive cold working also generates large number of vacancies, which form vacancy-scandium atom complexes of high binding energy. The vacancysolute 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.

The microhardness variation as well as fractional recrystallization includes the contribution of both recovery and recrystallization processes to the overall decrease in microhardness. However, the decrease in hardness is also due to precipitation coarsening of the alloy. It was reported earlier that precipitation coarsening of Al_3Sc occurs beyond 300 ℃ [11]. Fig. 3 shows JMAK plot for recrystallization kinetics obtained from micro-hardness. Fig. 4 shows a plot for comparison of recrystallization kinetics as obtained from original micro-hardness data and from JMAK analysis. The overall kinetics behavior from the two methods of analysis is very similar. The higher difference in the curves for scandium added alloys can be attributed to the scale of analysis on each sample as well as precipitation hardening and recrystallization behaviour. The micro-hardness data represent an average behavior for recrystallization kinetics.

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 aluminium 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 aluminium used for the present experimentation [1]. The number of non-equilibrium segregation is dependent on the magnesium content and the concentration of other potential aluminide formers [11]. However, scandium forms an anomalous supersaturated solid solution, which decomposes to form Al_3Sc [12]. Though general observations under optical microscopy have not provided much information, the overall appearance of the microstructure resembles what are normally observed in cast aluminium alloy ingot [3]. The cold worked structures are comprised of elongated grains. The base alloy however has started recrystallising as it is known that recrystallisation of Al-6Mg alloy becomes completed at about 400 ℃. However alloy 3 has dispersion of fine precipitates of Al_3Sc. These precipitates are coherent with the matrix. It is reported that recrystallisation is almost impossible in aluminium alloys when such particles are already present [13]. Higher becomes the volume fraction of precipitates higher would be the recrystallisation start temperature. The precipitates hinder the movement of sub-boundaries and grain boundaries. On increasing the temperature to 400 ℃, 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 form Al_3Sc at around 300 ℃. 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 ℃ to 400 ℃ increases the size of Al_3Sc precipitates from 4nm to 13nm. The precipitates of Al_3Sc remain coherent with the matrix even when their size increases to 100 nm due to higher temperature of annealing [4]. In the present case however the precipitate size is around 15 nm when annealed at 400 ℃. Therefore dislocation pinning force is very large. As a result recrystallisation is not possible.

V. Conclusion

Al-Mg alloy shows the small difference between two methods. Scandium added alloys show the larger different between two methods due to precipitation of Al_3Sc, which age harden the alloys as well as recristallization inhibitor at higher temperature.

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