Association of Metallurgical Engineers of Serbia *Scientific paper*
AMES *UDC:669.715'721.065.53.040.2-174=20*

THE INFLUENCE OF THERMOMECHANICAL TREATMENT AND CHEMICAL COMPOSITION ON RECRYSTALLIZATION OF Al-Mg ALLOYS

LJ.RADOVIĆ[1,a], M.NIKAČEVIĆ[1,b], M.POPOVIĆ[2,c], E.ROMHANJI[2,d]

[1]*Military Technical Institute, R.Resanovića 1, 11135 Belgrade, Serbia,* [2]*Faculty of Technology and Metallurgy, Karnegijeva 4, 11120 Belgrade, Serbia*
[a]*nenrad@ptt.yu,* [b]*anikac@ptt.yu,* [c]*miljana@tmf.bg.ac.yu,* [d]*endre@tmf.bg.ac.yu*

ABSTRACT

Recrystallization behaviour of AlMg3, AlMg4.5Mn and AlMg6Mn alloy plates subjected to different thermomechanical treatments (TMTs), has been studied by means of optical microscopy and hardness measurements. Applied two-stage annealing at 565°C/6h + 450°C/4h simulated homogenization and heterogenization processes, respectively, and was followed by cold rolling with 20, 40 and 60%. Final annealing was performed for 3h at 250, 280 and 350°C, with slow and rapid heating rates (0.6 and 25°C/min). Increase of rolling reduction and temperature, either individually or in combination, promotes recrystallization during final annealing. The recrystallization process was found non-simply related to the chemical composition, but a rather complex recrystallization behaviour of these alloys is attributed to the effect of the size and distribution of the second phase particles formed during the thermomechanical treatment.

Key words: Al-Mg alloys, chemical composition, cold deformation, recovery, heating rate, recrystallization

INTRODUCTION

The control of microstructure development during thermomechanical processing is essentially important in the production of AlMg alloy sheet material. It is well known that the chemical composition and processing parameters affect the development of the microstructure in these alloys, determining their mechanical properties and formability [1-9].

Recrystallization characteristics and resulting grain sizes in Al alloy are closely related to the amount and distribution of second phase particles in the matrix established during preceding TMTs. [10-15]. At larger applied strains in particle containing alloys the increase of the stored energy due to particles increases the driving pressure for recrystallization. In addition, more particles become active sites for nucleation (particle stimulated nucleation-PSN), which also enhances the recrystallization [16]. On the other hand, particularly small and closely spaced particles may exert a significant pinning effect on grain boundaries and retard recrystallization. Therefore, the overall recrystallization behaviour of the particle containing material depends on difference between the driving force, due to increase in stored energy, and retarding force, due to particles pinning effect [16-19].

The objective of this paper was to assess the influence of TMTs and the content of Mg and Mn on recrystallization process and recrystallized grain size, AlMg3, AlMg4.5Mn and AlMg6Mn alloys.

EXPERIMENTAL

Three different Al-Mg alloys, produced by IMPOL-SEVAL Aluminium Rolling Mill AlMg3 (3.1 Mg, 0.03 Mn, 0.31 Fe, 0.09 Si); AlMg4.5Mn (4.1 Mg, 0.54 Mn, 0.36 Fe, 0.12 Si), AlMg6Mn (5.95 Mg, 0.57 Mn, 0.41 Fe, 0.16 Si) were tested in this work. The as received plates were 7.4, 15 and 8 mm thick in the as-received hot rolled condition.

The as-received plates were annealed at 565°C for 6h with heating rate of 35°C/h, than furnace cooled to 450°C, held for 4h, and finally furnace cooled to the room temperature. Following this process the samples were cold rolled by 20, 40 and 60% reductions. Subsequent annealing was applied at 250, 280 and 350°C for 3h with rapid heating -RH (25°C/min) and slow heating -SH (0.6°C/min). In both cases air-cooling was applied.

To reveal the grain structure, after electro-polishing, the samples were etched in Barker′s solution (25ml 40%HBF_4, 1000ml distilled water). The recrystallization behaviour during final annealing was assessed by both optical metallography and hardness measurement. The average grain size was determined by the linear intercept method. Hardness was measured by the Vickers hardness tester (HV5).

RESULTS AND DISCUSSION

The two-stage annealing was applied in order to develop as much as possible homogenous microstructure of the tested alloys. During the first step of annealing simulating homogenization, the dissolution of the intermetalic particles at 565°C takes place as it was found in previous papers [13,14,20]. During subsequent annealing at 450°C precipitation and particle coarsening should take place, leading to increased mobility of grain boundaries and the development of a rather homogenous grain structure [14,17,21]. Accordingly, this stage of annealing treatment can be treated as an heterogenization process.

Microstructure and particles distribution of the tested alloys after two-stage annealing are shown in Figs.1 and 2, respectively. In the AlMg3 alloy, the low Mn content enabled the high mobility of grain boundaries and the abnormal grain growth is the most expressed. In comparison with the AlMg3 alloy (Fig.1a) the grain growth in the AlMg4.5Mn was impeded (Fig.1b). The reason for this can be the higher content of the second phase particles, mostly due to the presence of $MnAl_6$ fine particles [10,11,22], and also due to the higher content of Mg-based second phase particles [13], which also can pin the grain boundaries and slow down their migration [11,15,17]. A higher Mg content and also the addition of Mn in the AlMg4.5Mn alloy contribute to the higher density of the second phase particles compared with AlMg3 alloy (Figs.2a and b).

In AlMg6Mn alloy, despite of significant coarsening of the particles (Fig.2c), the secondary recrystallization was impeded and homogenous structure with equiaxed grains with smooth boundary was obtained. This behaviour is attributed to the large amount of small dispersoids precipitated during slow cooling after heterogenization annealing, which effectively pinned the grain boundaries.

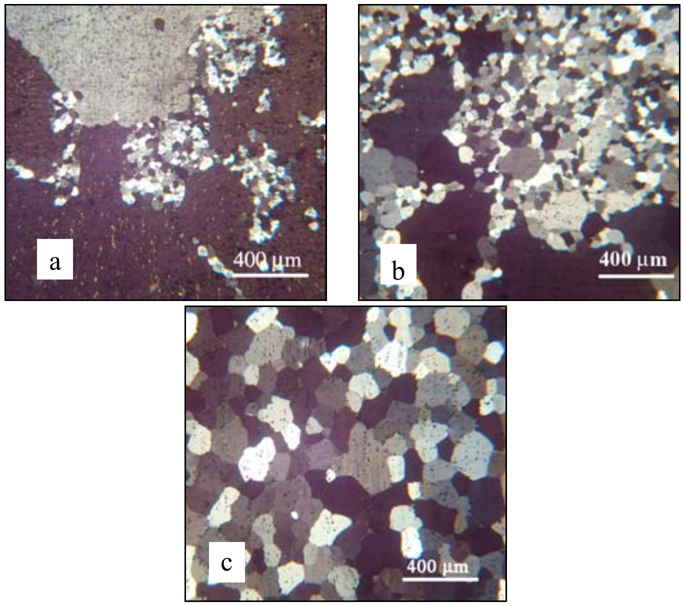

Fig. 1. Grain structure after two-stage annealing of the AlMg3 (a), AlMg4.5Mn (b) and AlMg6Mn (c) alloy sheets

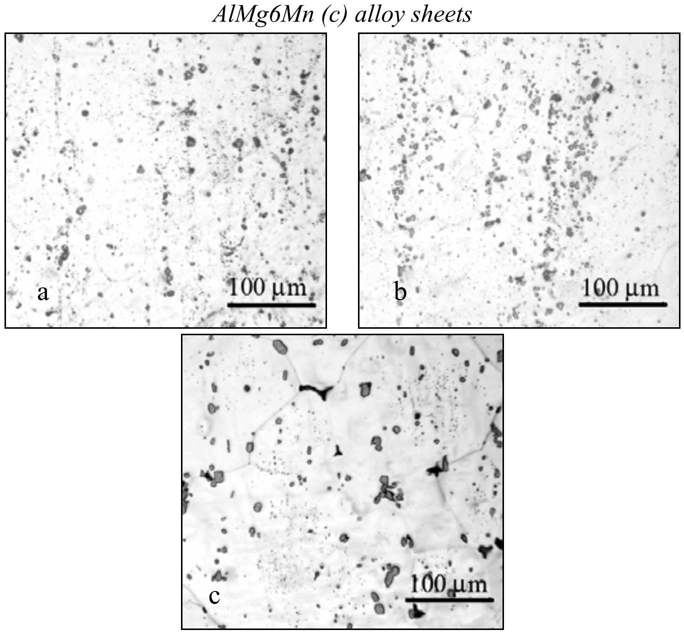

Fig. 2. Particle distribution in the tested alloys after two-stage annealing: (a) AlMg3, (b) AlMg4.5Mn and (c) AlMg6Mn

The results given in the Table 1 summarize the recrystallization behaviour of the previously cold rolled Al-Mg alloys after final annealing. Optical microscopy showed that the increase of rolling reduction or annealing temperature promote recrystallization. A higher rolling strain introduces higher stored energy thus promoting recrystallization (strain induced nucleation) [17, 19]. The effect of annealing temperature is equivalent to the effect of strain. The higher temperature facilitates the nucleation and grain growth [17, 19]. This results in the larger number of the recrystallized grains in the microstructures.

Table 1. Recrystallization behaviour of examined Al-Mg alloys after annealing for 3 hours

Alloy	t , °C	250		280		350	
	r (%)	SH	RH	SH	RH	SH	RH
AlMg3	20	□	□	□	□	▒	▒
	40	□	□	□	▒	■	■
	60	□	▒	■	■	■	■
AlMg4.5Mn	20	□	□	□	□	■	■
	40	▒	▒	■	■	■	■
	60	■	■	■	■	■	■
AlMg6Mn	20	□	□	□	▒	■	■
	40	□	□	▒	▒	■	■
	60	▒	▒	▒	▒	■	■

□ - recovered structures
▒ - partially recrystallized structures
■ - fully recrystallized structures.
SH - slow heating
RH - rapid heating

The results of Table 1 also implicate a strong influence of alloying additions on the recrystallization behaviour. In the AlMg4.5Mn samples the recrystallization temperature was found lower compared to the AlMg3 alloy. However, in the AlMg6Mn samples the considerably higher content of alloying elements increases the recrystallization temperature.

Increase in the volume fraction and the size of second phase particles in AlMg4,5Mn (Fig. 2b) was assumed to be favorable for the dislocation accumulation, creating high dislocation density around particles. Also, these particles served as grain nucleation sites during static recrystallization (PSN) [16]. It has been established that hard, non-deformable particles larger than a critical size can serve as a sites for recrystallization. For most Al alloys the critical particle size is around 1-2 μm [16]. On the bases of detailed metallographic examination, it was estimated that there was an increase in the amount of second phase particles larger than 1 μm (Fig. 4a) in AlMg4.5Mn. This value is in agreement with some earlier published results [3, 8, 11-13, 15]. The particles increase the dislocation density and stored energy of deformation, providing the driving force for recrystallization. However, another aspect of present particles is the Zener drag pressure, which sometimes leads to the opposite effect [8, 17, 18], *i.e.* particles of all sizes may exert a significant pinning effect on moving boundaries. This effect is more expressed if particles are small and particularly closely spaced [16-18].

Microstructure development in AlMg6Mn alloy after annealing at different temperatures is shown in Fig. 3. In this alloy the addition of Mg lowers the solid solubility of Mn in Al [1,6] leading to precipitation of higher volume fraction of Mn-bearing particles, which has been reported earlier [8, 12, 15].

Additionally, previous two-stage annealing significantly altered precipitate distribution resulting in highly coarser particles in AlMg6Mn alloy compared with particles in AlMg4.5Mn alloy (Figs. 4a and b). At the same time a slight increase in the number of very fine particles could be produced [4, 8, 11].

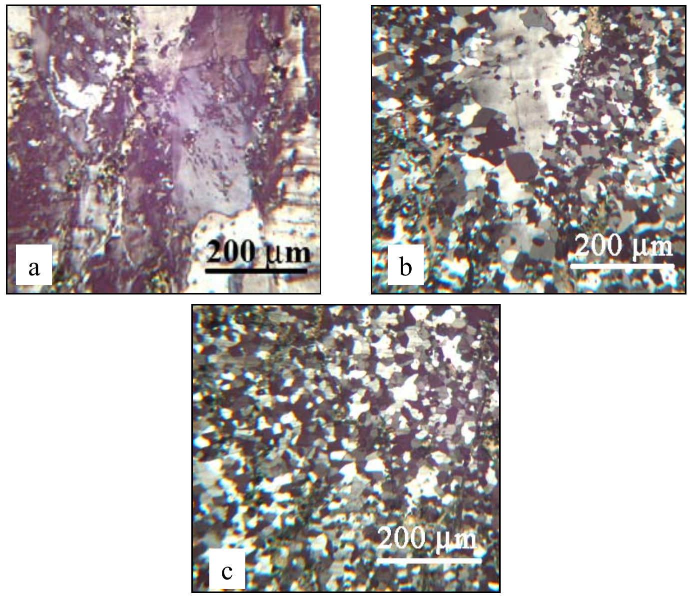

Fig. 3. Microstructures in AlMg6Mn alloy after 60% reduction and annealing at: (a) 250°C/3h, (b) 280°C/3h and (c) 350°C/3h; rapid heating

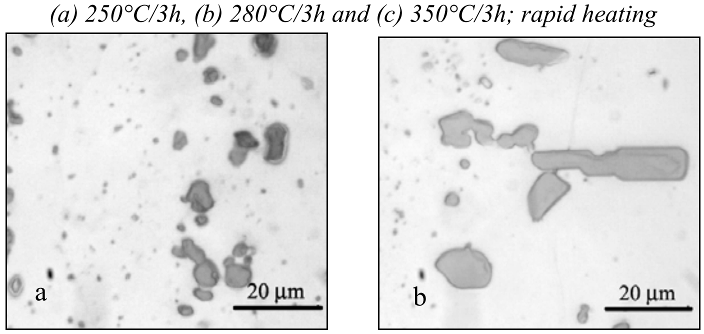

Fig. 4. Particles in the tested alloys after two-stage annealing: (a) AlMg4,5Mn and (b) AlMg6Mn

The particles' coarsening appears to minimize any beneficial effect noted in the AlMg4.5Mn alloy retarding static recrystallization in AlMg6Mn alloy. The coarsening leads to a smaller number of particles and thus the number of potential nuclei decreases. However, the coarse intermetalic particles were broken down during rolling, and this tendency significantly rises with increasing strain. This has been reported earlier in the non-heat treatable alloys, especially in Mn-containing alloys [23].

Despite being nucleated earlier, the new grains in the vicinity of particles are smaller in comparison to the other, probably because of the larger number of nuclei at particles (Fig.5).

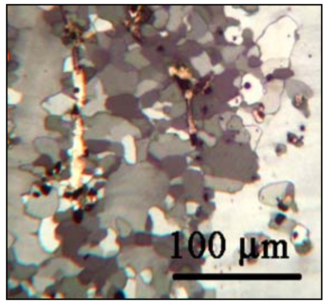

Fig. 5. A non-uniform grain development in AlMg6Mn

While AlMg3 alloy exhibits partial recrystallization within 3 hours at 280°C, AlMg4.5Mn alloy was recrystallized completely under the same conditions (Fig.6b). However, in AlMg6Mn alloy (Fig.6c) new recrystallized grains may be seen around large particles, but the fraction of recrystallized structure is smaller compared to AlMg3 alloy.

Results shown in Fig.7a indicate that increasing the cold rolling reduction prior to annealing reduced the recrystallized grain size. The results also suggest that the addition of Mg up to 4.5% and approximately 0.5% Mn reduced grain size. However, after higher reduction (60%), the influence of alloying additions on grain size was significantly reduced. Thus**,** the final grain size seems to be predominantly influenced by the rolling reduction. In the AlMg3 alloy at low strain, the low particles content allowed the grain growth. The addition of Mg and particle forming Mn, produced a larger amount of second phase particles and the preference for more nucleation sites. This leads a significant reduction in the final grain size, particularly under the higher strain [16].

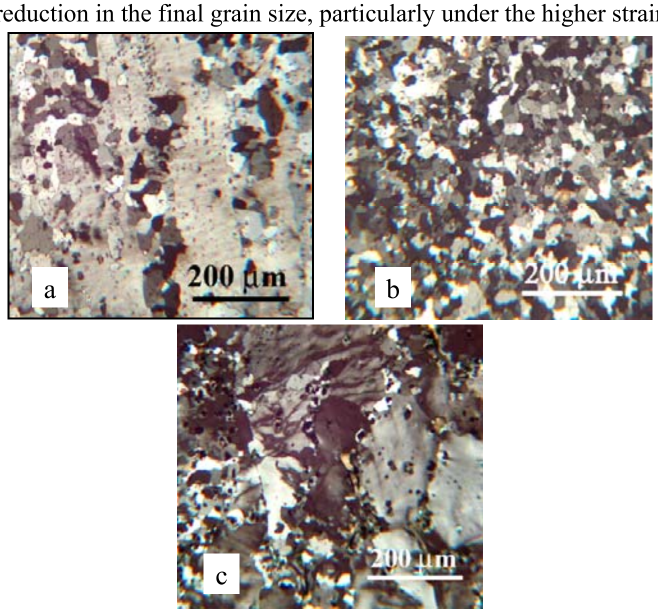

*Fig. 6. Microstructures of (a) AlMg3, (b) AlMg4.5Mn and (c) AlMg6Mn after 40% cold rolling followed by annealing at 280°C for 3 hours**;** rapid heating*

The heating rate was found to influence the onset of the recrystallization (Fig. 7b). It seems that slow heating rate promotes the recovery that occurs before recrystallization, so the driving pressure is reduced and recrystallization is retarded. This can be clearly noticed in the alloy without Mn, *i.e.* the AlMg3 alloy. In contrast, the rapid heating suppresses recovery, promotes recrystallization and induces the start of recrystallization at lower temperature, which are in accordance with previously published results [6,17,19]. Also, the more potential nucleation sites activated during rapid heating could influence the recrystallized grain size. This was confirmed by grain size measurements and illustrated in Fig 7b, for AlMg6Mn alloy.

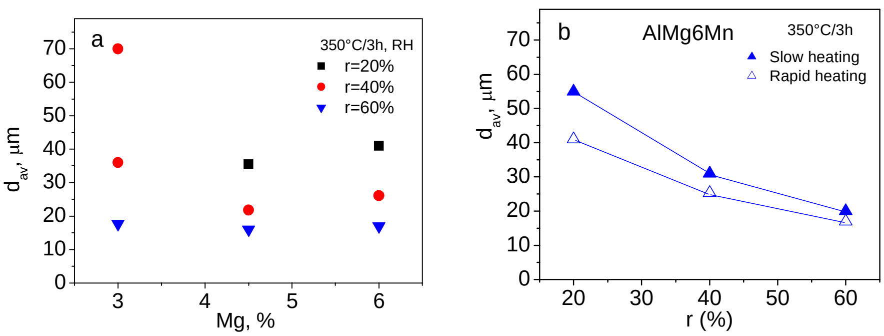

Fig. 7. The influence of (a) Mg content and (b) cold rolling reduction and heating rate, on recrystallized grain sizes

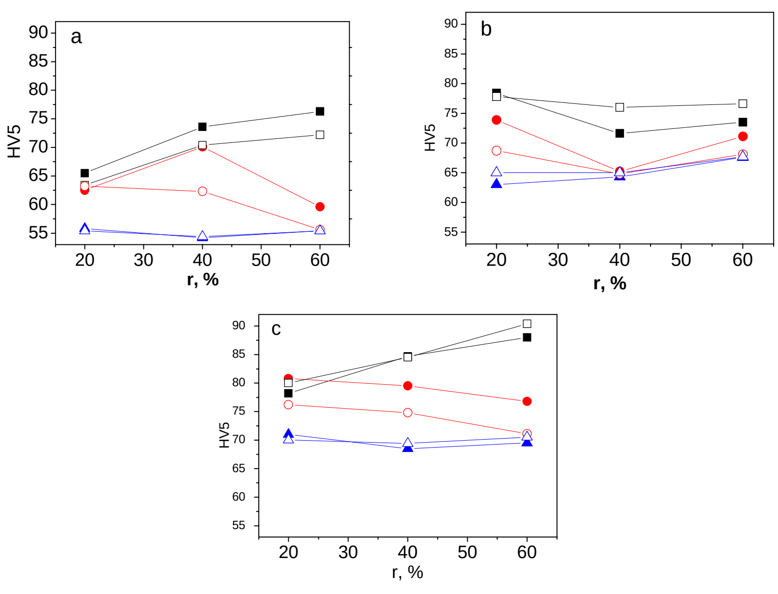

Fig. 8. The influence of cold rolling reduction on hardness after annealing for 3 hours at: ■ - 250°C, ● - 280°C, ▲ - 350°C, slow heating-closed symbols; rapid heating – opened symbols; (a) AlMg3, (b) AlMg4.5Mn and (c) AlMg6Mn

The variations in the hardness (Fig. 8) in Al-Mg alloys are in agreement with microstructure characteristics. The onset of the recrystallization, observed by metallography, was followed by softening. The hardness rises with increasing strain in recovered structure (AlMg6Mn at 250°C-Fig.8c) or in partially recrystallized structure when the fraction of recrystallized structure is small (AlMg3 60%+250°C-Fig.8a). When recrystallization starts the hardness continuously decreases with increase of the recrystallized grains fraction. In completely recrystallized structure (AlMg4.5Mn alloy 60%+350°C-Fig. 8b and Table 1.) hardness slightly increases with increasing strain as a result of grain size reduction (Fig. 7a).

SUMMARY

The influence of the chemical composition and processing conditions on structure development in AlMg3, AlMg4.5Mn and AlMg6Mn alloy sheets were investigated.

The alloying additions have a strong influence on the particle size and their distribution produced by two-stage annealing (565°C/6h+450°C/4h), and the recrystallization behaviour after subsequent annealing. The applied two-stage annealing was found to be followed with an abnormal grain growth in AlMg3 and AlMg4.5Mn alloys, while in AlMg6Mn it was suppressed.

The increase of the Mg content from 3 to 4,5%, coupled with addition of approximately 0,5% Mn, decreases both the recrystallization temperatures and the recrystallized grain size after final annealing. However, in the AlMg6Mn alloy in responce to the increase in the Mg content, the recrystallization temperature raises and the grain size slightly increases.

The increase of the rolling reduction prior to final annealing decreases the recrystallization temperatures in AlMg3 and AlMg4.5Mn alloys. On the other hand in the AlMg6Mn alloy the recrystallization temperature seems to be independent of the applied reductions. The recrystallized grain size is reduced with increasing reductions in all investigated alloys.

The heating rate has influence on the onset of the recrystallization and recrystallized grain size. It was assumed that the slow heating rate promotes the recovery that occurs before recrystallization, reducing the driving pressure and retarding recrystallization. In contrast, the rapid heating suppresses recovery and promotes recrystallization, reducing the recrystallized grain size.

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