UDK: 620.18:669.715'5'721'3
COSATI: 11-06

Microstructure and Properties of Cold Rolled and Annealed Al-Mg Alloys

Ljubica Radović, MSc (Eng)[1)]
Milutin Nikačević, MSc (Eng)[1)]

The effect of Mg content and thermomechanical processes on microstructural evolution, mechanical and physical properties of cold rolled and annealed Al-Mg alloys containing 3-6% Mg and around 0.5wt.% Mn, have been investigated by means of optical microscopy, hardness and electrical conductivity measurements. For a given composition, it was found that the final grain size of the recrystallized material decreases with increasing strain as well as with the increasing Mg content. In alloys with 4.5 and 6% Mg the grain size at critical strain decreases with addition of Mn.

Key words: **Al-Mg alloys, microstructure, cold rolling, annealing, hardness, electrical conductivity, critical strain, grain size.**

Introduction

Al-Mg alloys are an important group of commercial Al alloys with good combination of strength and formability and therefore they have various applications in transport, packaging and general engineering industries. They belong to the group of non-heat treatable Al alloys which derive their strength mainly from solution strengthening and work hardening during deformation. Cold deformation is particularly effective in strengthening since restoration processes are thermally activated.

Strength of Al-Mg alloys strongly increases with addition of Mg. Mg has a high solubillity in solid solutions and therefore it provides the most effective enhancement of strength among all alloying additions in the aluminium solid solution [1-5]. Mg, like other solute elements, has a strong influence on recrystallization and grain growth. It also serves to reduce both the rate of growth and the final grain size [1, 6, 7].

Commercial Al-Mg alloys also contain elements such as Mn, Fe, Si or Cr and Ti. These elements form the second phase particles due to a limited solubility in solid solutions. Although these elements have a minor influence on strength of Al alloys, they have a significant influence on annealing behaviour, i.e. they are an important parameter in the grain size control. Addition of Mn increases strength no matter it is in a solid solution or in the precipitated intermetalic phase. Mn usually precipitates as a dispersoid during preheating and hot processing increasing toughness, and, at the same time, decreasing susceptibility to intergranular cracking and stress corrosion. Mn is mainly added for the grain size control due to its grain growth inhibition. Although the predominant reason for alloying is to increase strength, alloying has important effects on the other characteristics of Al alloys: physical and electrochemical properties and corrosion resistance. Electrical conductivity is one of the most sensitive properties of Al alloys, being particularly responsive to changes in composition and temper. Electrical conductivity of Al-Mg alloys depends on whether the element is in a solid solution or a second phase is formed. Elements in a solid solution are always more harmful to electrical conductivity. The electrical conductivity of these alloys is mainly dependent on the amount of magnesium dissolved and decreases almost linearly with concentration of Mg in a solution [1]. The effects of other elements are submerged by Mg. During the cold deformation, electrical conductivity decreases due to the increase of the number of vacancies and the density of dislocations. A higher electrical conductivity after annealing is a result of the combined effects of recovery and recrystallization, and the dissolution of precipitate phases.

The grain size is one of the most important microstructural factors controlling strength and formability. A fine grain size is extremely important factor to minimize surface roughening during forming and to provide ductility during forming operation and fracture toughness. On the other hand, to avoid stretcher strain markings, the material should be formed in the fully soft temper, i.e. in this case it is necessary to avoid a grain size which is too fine. For these reasons, it is important to obtain an optimal grain size, and to have a good combination of strength and formability. The grain size is controlled by an appropriate combination of hot and cold rolling and annealing [8-12].

The grain size in Al-Mg alloys with Mn after annealing depends on chemical composition, initial grain size, heating rate, precipitation, second-phase particles, Mn dispersoid, content Mg in solid solution and annealing time. Magnesium serves to reduce both the rate of growth and the final grain size but also, to give a much greater non-uniformity in the scale of the recrystallized structure [4]. The grain size in alloys for a given composition is the most significantly effected by cold rolling reduction before annealing.

1) Military Technical Institute (VTI), Ratka Resanovića 1, 11132 Belgrade, SERBIA

For a given annealing temperature there is a critical amount of strain required to initiate recrystallization. Below the critical strain, formation of recrystallization nuclei is hampered by the low driving force and the lack to region of large lattice curvatures and complete recrystallization does not occur. Just above the critical strain the annealed grain size is very large due to the activation of only nucleation sites [2, 13, 14], i.e. the critical strain gives raise to the largest grains after annealing. If the strain is higher than critical, the grain size decreases.

The aim of this work was to determine the relation between the applied deformation and the grain size of annealed Al-Mg alloys, as well as their mechanical and physical properties.

Experimental

The chemical compositions of commercial Al-Mg alloys which were investigated are presented in Table 1.

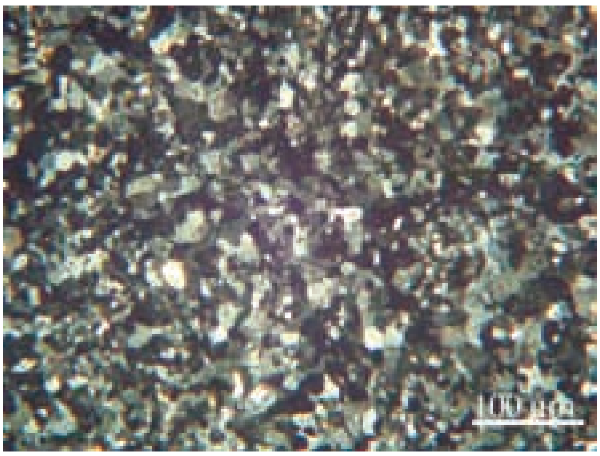
Table 1. Chemical compositions of Al alloys (wt.%)

	Mg	Si	Cu	Mn	Fe	Zn	Ni	Ti	Al
AlMg3	3.1	0.09	0.01	0.03	0.31	0.04	0.01	0.01	Bal.
AlMg4.5Mn	4.1	0.12	0.015	0.54	0.36	0.07	0.01	0.01	Bal.
AlMg6Mn	5.95	0.16	0.02	0.57	0.40	0.02	0.01	0.04	Bal.

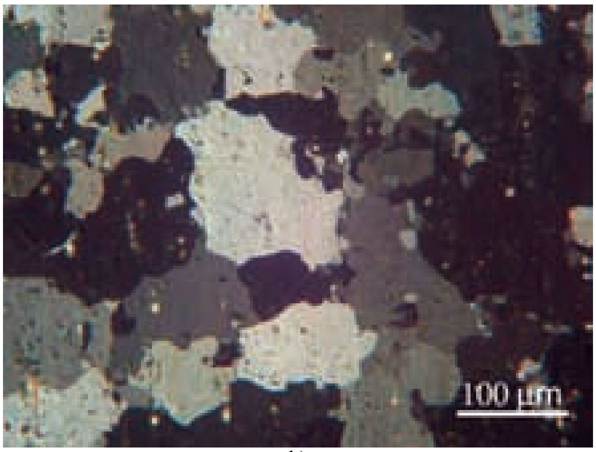
Pre-deformation treatment was performed and it consisted of pre-rolling and annealing for 3h at 320°C. The final rolling included reductions of 10, 15, 20, 25 and 50%, and was followed by the final annealing for 3h at 320°C. The final thickness of all samples was 3 mm, since they will be used for further spinnability testing on the industrial scale. To reveal a grain size, the standard procedure included mechanical and electro-polishing, etching using Barker's solution (25ml HBF_4 (40%), 1000ml distilled water). The grain size was observed by the optical microscope under the polarized light. The average grain size was determined by the linear intercept method. Hardness was measured by the Vickers hardness test (HV5) and electrical conductivity was measured using the Sigmatest D. 2.068.

Results and Discussion

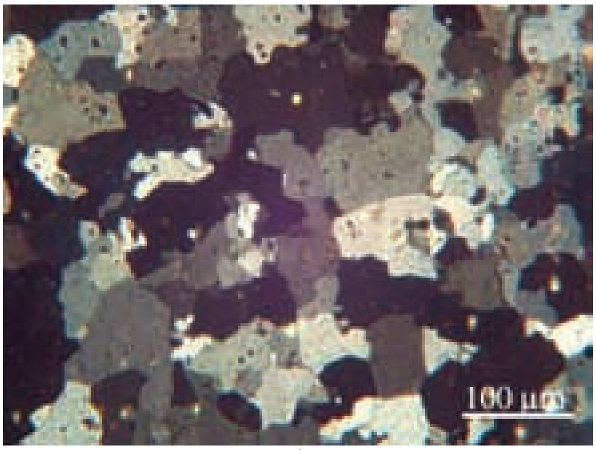
The microstructures obtained after annealing of AlMg3 are shown in Fig.1a-e. The effect of strain on the average grain size in the annealed specimens is shown in Fig.1f.



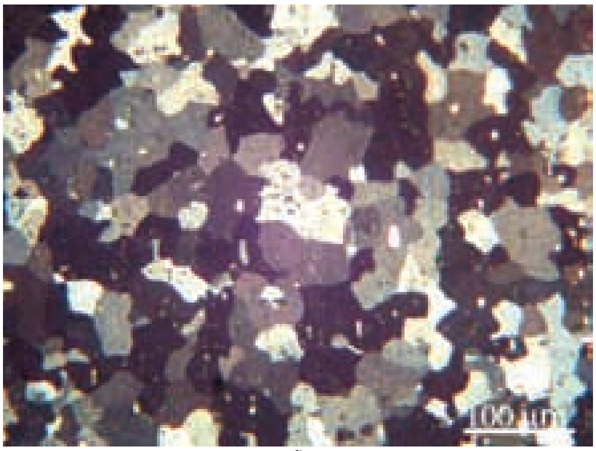
a)



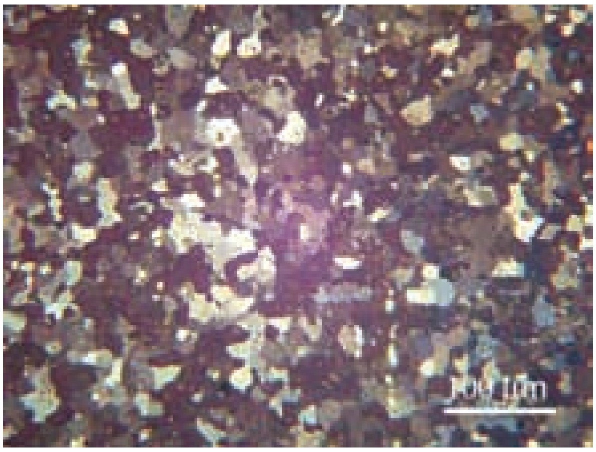
b)



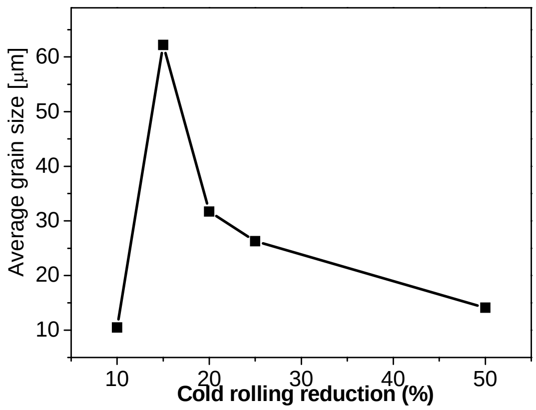
c)



d)



e)

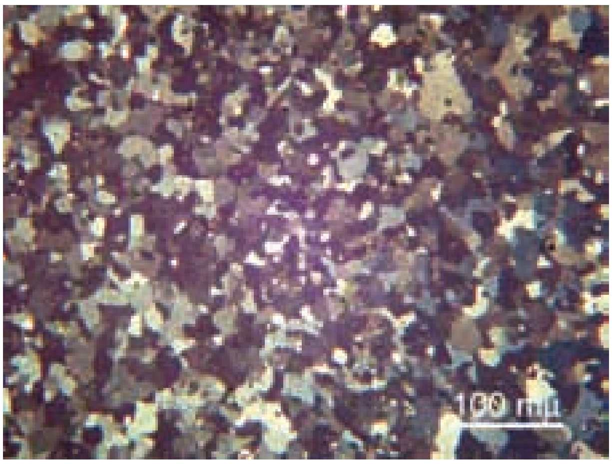


f)

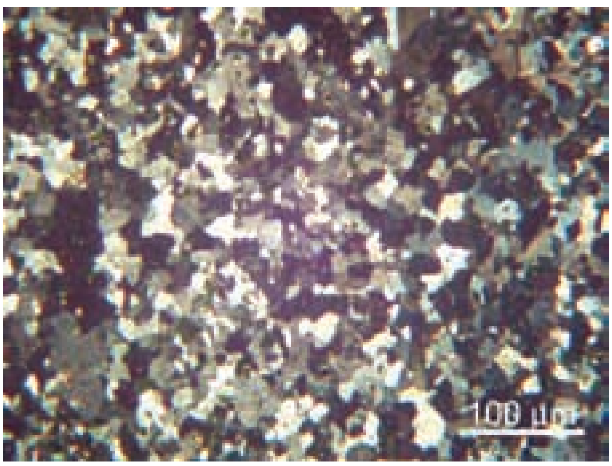
Figure 1. Microstructure of AlMg3 alloy annealed for 3h at 320°C after (a) 10% reduction; (b) 15% reduction; (c) 20% reduction; (d) 25% reduction; (e) 50% reduction; (f) the effect of strain on the average size grain

Polygonal grains with almost uniform size distribution characterize the microstructure in all samples. It was noted that the grain size increases with the rolling reduction up to the maximum value, and then decreases. The maximum grain size is related to the critical strain for recrystallization [13]. For this alloy the critical strain is about 15%. Similar dependence was obtained for the two other alloys.

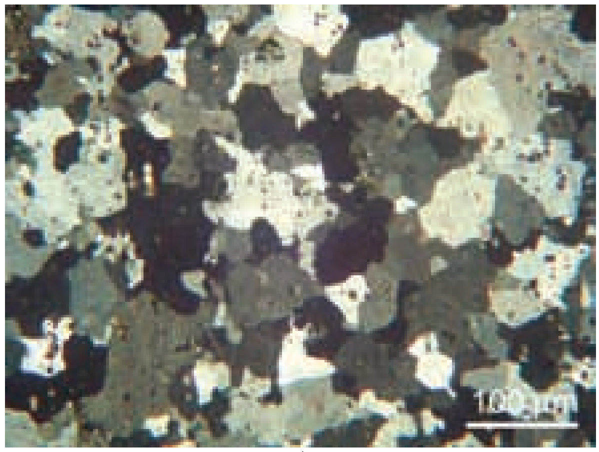
The typical microstructures obtained after annealing of AlMg4.5Mn and AlMg6Mn are shown in Figures 2a-e and 3a-e. The effect of strain on the average grain size of these alloys is shown in Figures 2f and 3f.



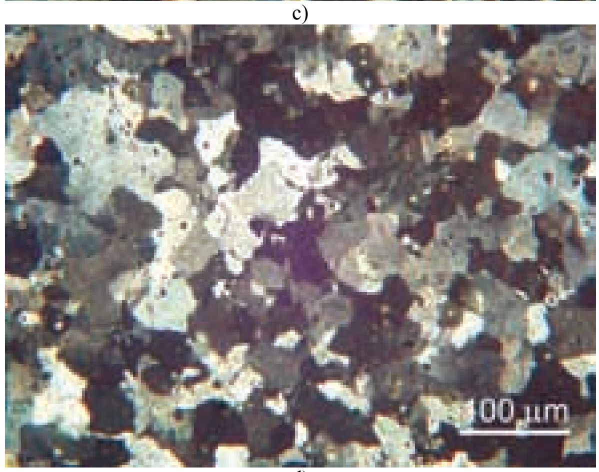
a)



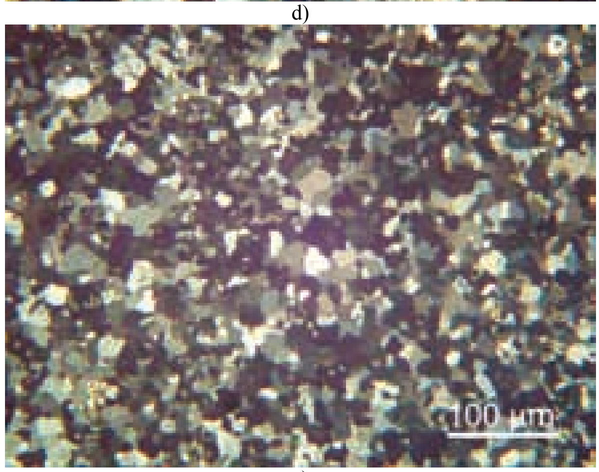
b)



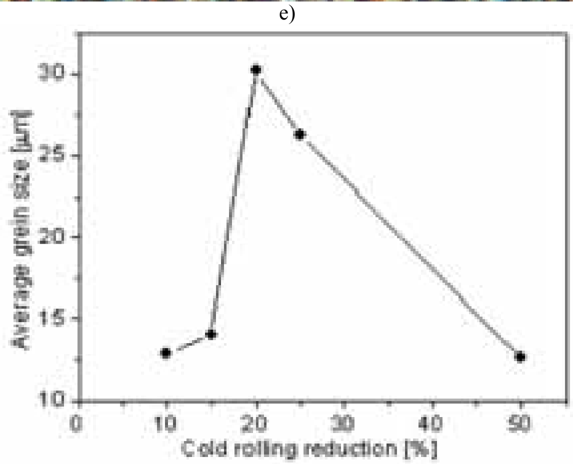
c)



d)



e)



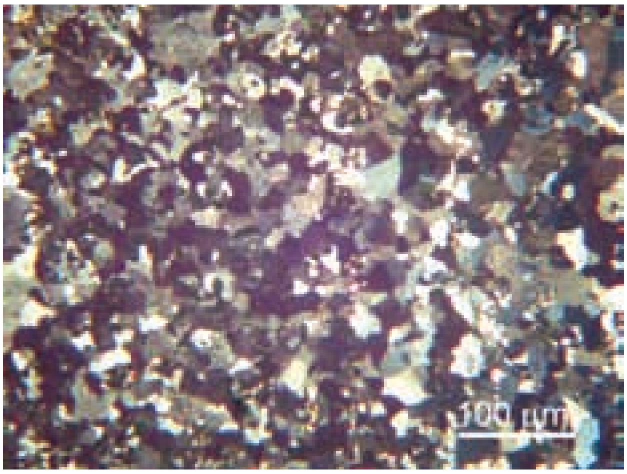
f)

Figure 2. Microstructure of AlMg4.5Mn alloy annealed for 3h at 320°C after (a) 10% reduction; (b) 15% reduction; (c) 20% reduction; (d) 25% reduction; (e) 50% reduction; the effect of strain on the average size grain.

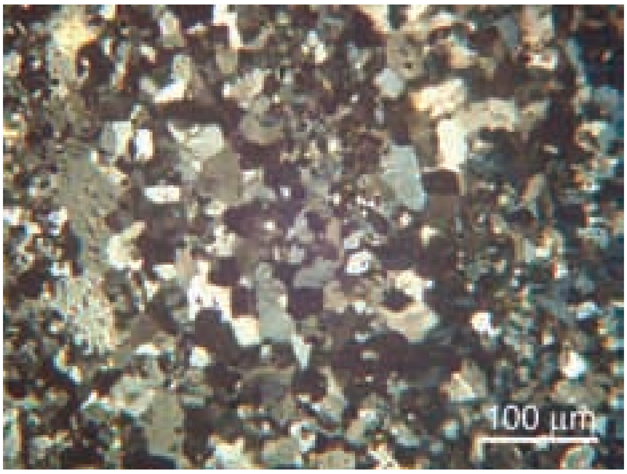
For AlMg4.5 Mn alloy the maximum grain size of approximately 30 μm was obtained after 20% reduction and then the grain size decreases and for AlMg6Mn, the maximum grain size, approximately 25 μm, was obtained after 25% reduction and as in AlMg4.5Mn the grain size decreases after achieving the critical strain.

The effect of strain on the average size grain in the annealed specimens of all tested alloys is shown in Fig.4.

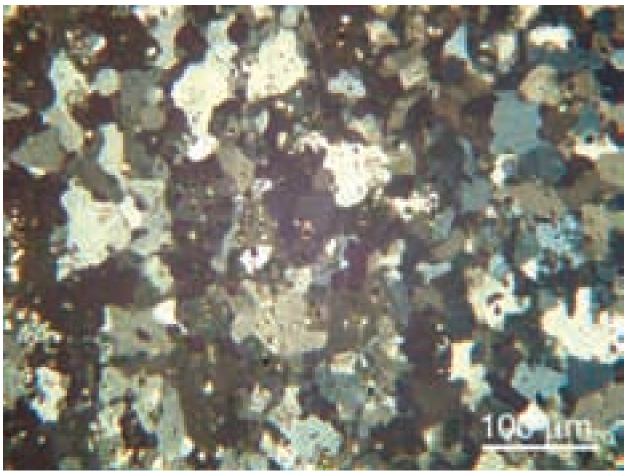
For all tested alloys the average grain size increases with reduction up to the maximum value, and then decreases. The critical strain values for AlMg3, AlMg4.5Mn and AlMg6Mn were 15, 20 and 25% respectively. The maximum grain size is related to the critical strain for recrystallization.



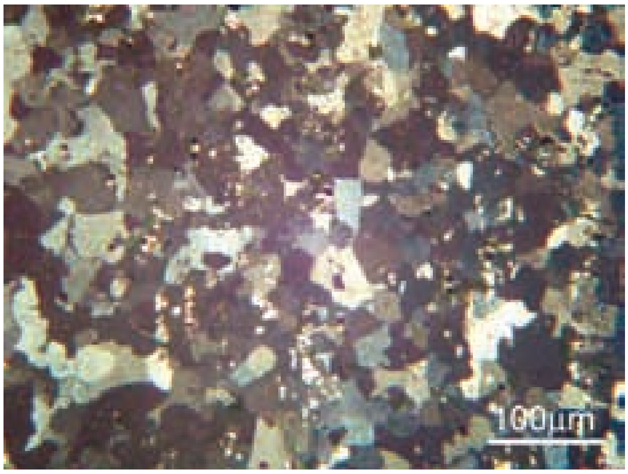
a)



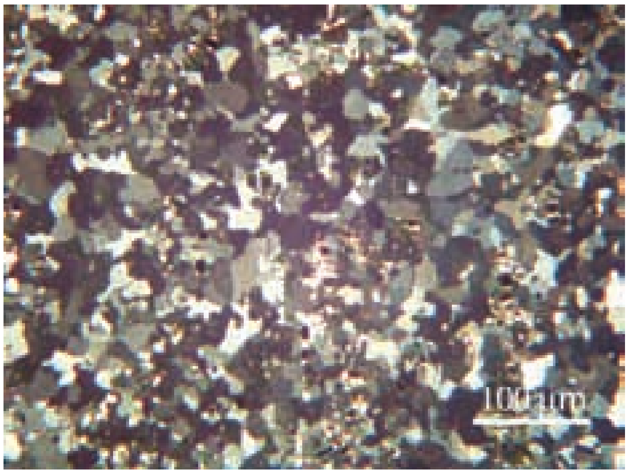
b)



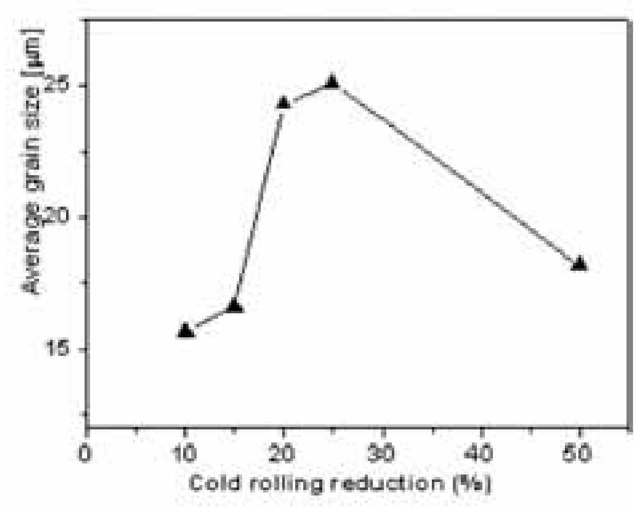
c)



d)



e)



f)

Figure 3. Microstructure of AlMg6Mn alloy annealed for 3h at 320°C after (a) 10% reduction; (b) 15% reduction; (c) 20% reduction; (d) 25% reduction; (e) 50% reduction; the effect of strain on the average grain size

Since the critical strain for recrystallization has been reached, the average grain size decreases as the percentage of cold deformation increases. A further increase of strain results in the decrease of the grain size. The recrystallized grain size depends on the nucleation rate and the growth rate [2, 7, 8, 14]. The nucleation rate increases with strain. At low strain, the deformation is distributed heterogeneously close to the grain boundaries and particles. Consequently, the nuclei are widely distributed in the deformed region resulting in a smaller number of nuclei per unit volume and in coarse grains. As the strain increases, more energy stored from cold work is available for the nucleation of new grains and the recrystallized structure is finer. Also, the critical strain increases due to the increase of Mg content in alloy, because a higher Mg content requires a higher driving force for the structural rearrangement (recovery and recrystallization). The Mn has the main role in the grain size control. The addition of the Mn in AlMg4.5Mn and AlMg6Mn alloys inhibits grain growth and strongly decreases the grain size at the critical strain compared to the AlMg3 alloy.

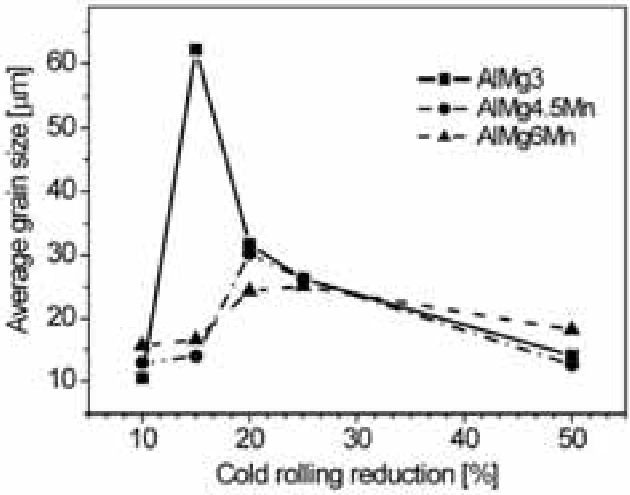


Figure 4. Effect of strain on the grain size in annealed specimens

The effect of the Mg content on the critical strain for static recrystallization and the related grain size is summarized in Fig.5. Higher Mg content increases the critical strain for static recrystallization, while decreasing the related grain size. The different slopes on d_{max}/%Mg curve are directly related to the addition of Mn in alloys with 4.5% and 6% Mg.

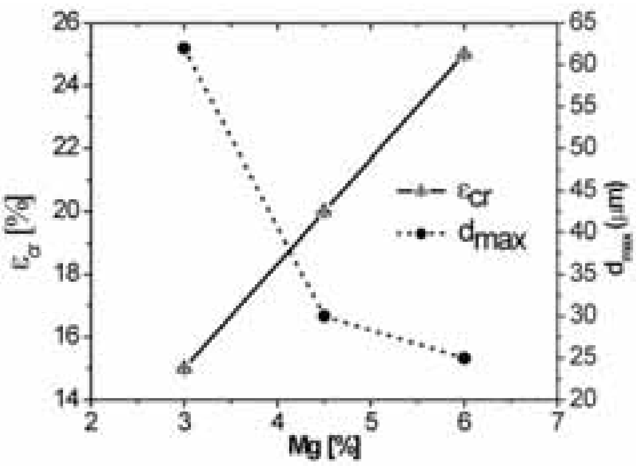


Figure 5. Effect of the Mg content on the critical strain for static recrystallization and related grain size

The effect of strain on hardness is shown in Fig.6, for both strained (closed symbols) and annealed (open symbols) specimens. In the strained state, hardness increases as the amount of deformation increases. Also, the differences in hardness between AlMg6Mn and AlMg3 increase as the amount of deformation increases. This is in agreement with the assumption that the Mg content has a greater influence on hardness at higher strain [4].

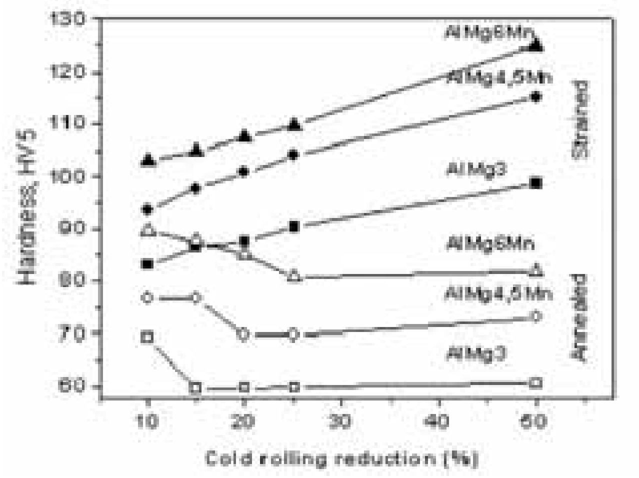


Figure 6. The influence of cold rolling reduction on hardness

In the annealed condition, hardness curves for all alloys have a similar shape. The hardness of annealed specimens decreases up to some specific strain and then slightly increases with further increase in strain. The minimum hardness values were obtained after 15, 20 and 25% cold rolling for AlMg3, AlMg4.5Mn and AlMg6Mn, respectively. The results in Fig.6 are comparable with those in Fig.4. It can be noted that the largest drop in hardness is related to the critical strain of all alloys. Also, the highest hardness drop was obtained for AlMg3 alloy, medium for AlMg4.5Mn and the lowest for AlMg6Mn alloy. This behaviour is in good agreement with the grain sizes shown in Fig.4.

Also, the results in Fig.6 show that the Mg content has a great influence on hardness, it increases the hardness in both deformed and annealed condition, as it was found earlier [1-5]. The clear Mg content effect on hardness is illustrated in Fig.7.

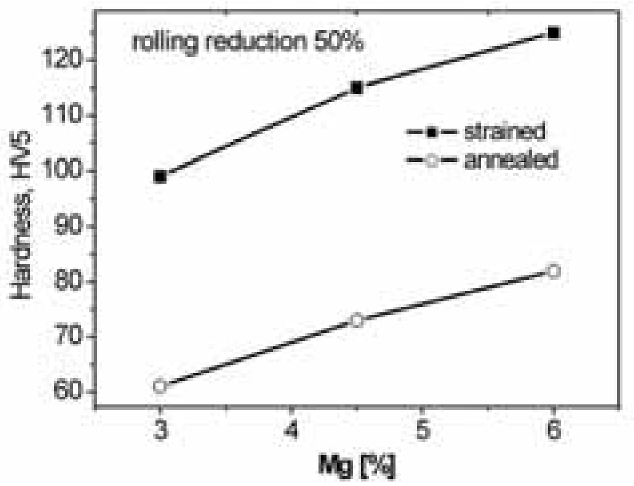


Figure 7. Effect of the Mg content on hardness

The results of the electrical conductivity measurements are shown in Fig.8. The results indicate that the addition of Mg has a strong influence on the electrical conductivity, in both strained and annealed conditions. On the other hand, it seems that the strain has a very weak influence, e.g. at 50% deformation the electrical conductivity decreases from 2 to 2.5%, which is in accordance with some previously reported results [1, 15].

Electrical conductivity slightly decreases with increasing strain as a result of an increase of the dislocation density and vacancies concentration [1, 14-16]. However, the decrease of the electrical conductivity induced by cold deformation is the most intensive at the cold rolling reduction lower than applied in this work, as stated earlier [16, 17]. It can explain the small changes in the electrical conductivity in respect the applied strain. After annealing the electrical conductivity level is slightly higher than in strained condition. Two opposite effects contribute to the variations in electrical conductivity. The annihilation of vacancies during the recovery and the decrease of dislocation density during the recrystallization increase conductivity. On the other hand, the dissolution of soluble precipitate phases (i.e. Mg removing to solution) [18] decreases conductivity. After annealing at 320°C/3h the structures of these alloys are fully recrystallized and the grain growth still does not start. It can be assumed that the differences in the structures, i.e. grain sizes, have no influence on conductivity.

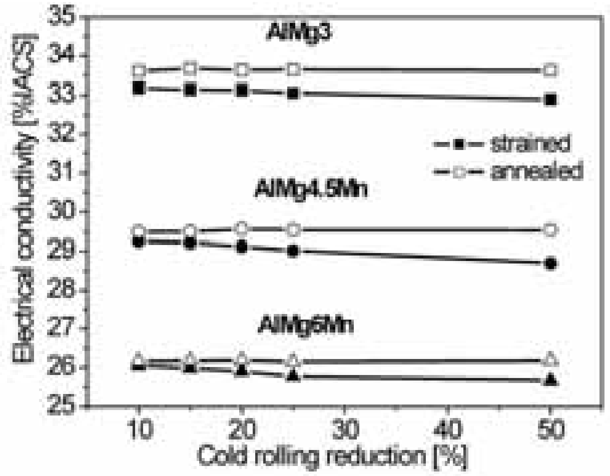


Figure 8. The influence of cold rolling reduction on electrical conductivity

Fig.9 illustrates the effect of the Mg content on electrical conductivity in both 50% deformed and annealed condition.

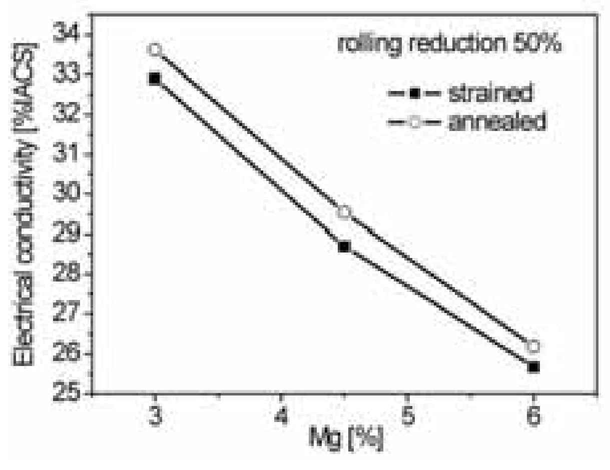


Figure 9. Effect of the Mg content on electrical conductivity

The increase of the Mg content decreases the electrical conductivity in both deformed and annealed conditions. The deviation from the linear relationship between the electrical conductivity and the Mg content is probably related to the absence of Mn in the AlMg3 alloy.

Conclusion

The effect of cold rolling reduction on the grain size, hardness and electrical conductivity of annealed Al-Mg alloys with different content of Mg was studied.

The grain size is strongly influenced by strain prior to annealing. The critical strain gives rise to the largest grains after annealing. The critical strain is dependant on the Mg content, so the values for AlMg3, AlMg4.5Mn and AlMg6Mn were 15, 20 and 25% respectively. The grain sizes which related to the critical strain were 62, 30 and 25 μm, respectively. The grain size decreases after achieving the critical strain for all tested alloys, thus the influence of the Mg content on the recrystallized grain size diminishes at higher strains. The addition of approximately 0.5% Mn in alloys with 3 to 4.5% Mg greatly decreases the grain size at the critical strain.

The hardness of Al-Mg alloys increases with Mg addition, as well as with strain, while in the annealed condition the hardness was observed to decrease to a minimum, and then to increase slightly. That minimum value of hardness corresponds to the critical strain.

Electrical conductivity slightly decreases with increasing strain, but pre-strain has no influence on electrical conductivity of annealed specimens. It can be assumed that changes of the concentration of vacancies and the dislocation density during deformation/annealing have the main influence on the electrical conductivity while the grain size has minor influence.

References

[1] MONDOLFO,L.F.: *Aluminium Alloys: Structure and Properties*, Boston, Butterworths, 1976.

[2] SANDERS,JR.,R.E., BAUMANN,S.F., STUMPF,H.C.: *Aluminium Alloys-Physical and Mechanical Properties*, EMAS, West Midlands, UK, 1986, Vol.III, pp.1441-1484.

[3] HATCH,J.E.: *Aluminium: properties and Physical Metallurgy*, ASM, Metals Park, Ohio, 1984.

[4] SANDERS,Jr.R.E.et al.: *Meterials Forum*, ed. by J.F.Nie et al., Institute of Mat.Eng. Australasia Ltd, 2004, Vol.28.

[5] BURGER,G.B., GUPTA,A.K., JEFFREZ,P.W., LLOYD,D.J.: Mat.Characterization, 35, 1995, pp.23-29.

[6] RYUM,N., EMBURY,J.D., SCAND.J.: Metall 11 (1982) 51.

[7] HUMPHREYS,F.J., HATHERELY,M.: *Recrystallization and Related Annealing Phenomena*, New York, NY: Elsevir Science Inc., 1996.

[8] GRIMES,R.: *Grain control in aluminium*, Paper No.MS 353. “Recrystallization in the Control of Microstructure”, London, 1973

[9] CHADWICK,R., HOOPER,W.H.L.: *J.Inst. Metals*, 1951-52, Vol.80, pp.17-22.

[10].BAE,D.H., GHOSH,A.K.: *Acta Mat.* 48, 2000, pp.1207-1224.

[11] GIRARD,S.X. et al.: *Met. and Mat.Trans A*, 2004, Vol.35A, pp.949.

[12] WEI WEN, MORRIS,J.G.: *Mat.Sci.Eng A354*, 2003, pp.279-285.

[13] WILLIAMS,W.M., EBORALL,R.: *J.Inst.Metals,* 81, (1952/53), pp.501.

[14] DROBNJAK,Dj.: *Fizička metalurgija*, TMF, Belgrade, 1986.

[15] ASM Metals Handbook, Vol.2, Metals Park, Ohio, 1979.

[16] STANOJEVIC,S. et al.: Proc. of the 12th Int. Research/Expert Conf., TMT 2008, Istanbul, Turkey, 2008, in press

[17] RADOVIC,Lj. et al: Proc. of the 2th Int. Conf. Deformation Processing and Structure of Materials, eds. E.Romhanji et al., Belgrade, 2005, pp.231-236.

[18] VERMA,R.et al.: Mat.Sci.Eng. A191, 1995, pp.143-150.

Received: 10.03.2008.

Mikrostruktura i osobine hladno valjanih i žarenih Al-Mg legura

Ispitan je uticaj sadržaja Mg i parametara termomehaničke prerade na mikrostrukturu, mehaničke i fizičke osobine hladno valjanih i žarenih Al-Mg legura sa sadržajem Mg od 3-6% i dodatkom oko 0,5% Mn. Korišćene su metode optičke mikroskopije, merenja tvrdoće i električne provodljivosti. Pokazano je da za leguru datog hemijskog sastava veličina rekristalisanog zrna opada sa povećanjem stepena deformacije. Povećanje sadržaja Mg u leguri takođe smanjuje veličinu rekristalisanog zrna. u legurama sa 4,5 i 6% Mg dodatak Mn značajno smanjuje veličinu zrna pri kritičnom stepenu deformacije.

Ključne reči: **legure Al-Mg, mirkostruktura, hladno valjanje, žarenje, tvrdoća, električna provodljivost, kritični stepen deformacije, veličina zrna.**

Микроструктура и свойства холодно прокатыванных и обожённых сплавов Al и Mg

Здесь испытано влияние содержания Mg и параметров термомеханической переработки на микроструктуру, а в том роде и механические и физические свойства холодно прокатыванных и обожённых сплавов Al и Mg со содержанием Mg из 3-6% с добавлением около 0,5 % Mn. Здесь использованы методы оптической микроскопии, измерения жёсткости и электрической проводимости. Тоже показано, что для сплава данной химической структуры размер рекристаллизованной зерно снижается с увеличением степени деформации. Увеличение содержания Mg в сплаве тоже уменьшает размер рекристаллизованной зерно. В сплавах со 4,5% и 6% Mg добавление Mn значительно уменьшает размер зерно при критической степени деформации.

Ключевые слова: сплавы Al и Mg, микроструктура, холодное прокатывание, обжигание, жёсткость, электрическая проводимость, критическая степень деформации, размер зерно.

Microstructure et les propriétés des alliages Al-Mg laminés à froid et recuits

Dans ce papier on présente les recherches sur l'effet du contenu du Mg et les paramètres du traitement thermo mécanique à la microstructure, les propriétés mécaniques et physiques des alliages Al-Mg laminés à froid et recuits avec le contenu de Mg de 3 à 6% et l'addition de Mn de 0,5% environ. On a utilisé les méthodes de microscopie optique, le mesurage de la dureté et la conductibilité électrique. On a démontré que pour l'alliage de cette composition chimique la dimension de grain recristallisé diminue si le degré de déformation augmente .L'augmentation du contenu de Mg dans l'alliage diminue aussi la dimension du grain recristallisé. Dans les alliages qui contiennent 4,5 et 6% du Mg l'addition du Mn diminue considérablement la taille du grain quand le degré de déformation est critique.

Mots clés: **alliages Al-Mg, microstructure, laminage à froid, recuit, dureté, conductibilité électrique, degré critique de déformation, dimension de grain.**