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Microstructural Aspects by Electron Microscopy Observed in Al-Mg Based Alloys after Special P/M Processes

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Abstract. In the last decade light materials have been studied thoroughly and used in components of pieces in the automobile, naval and aerospace industries. Their application makes possible mass reduce, load capacity increase, improvement in the mechanical properties when it is possible. Aluminium-magnesium alloys present good mechanical properties at moderate mechanical efforts (400 to 700 MPa) and good corrosion resistance. The alloys in study (Al-2Mg-0.6Zr and Al-2Mg-1Nb) were made by powder metallurgy (P/M) techniques, employing hot compactation and extrusion processes followed by cold work and thermal treatments. The analysis by SEM and TEM shows an evolution in the microstructure of precipitates with the increase of the time of thermal treatment, according to literature. The distribution of the precipitates in both alloys was observed and identified by EDS microanalysis (SEM and TEM).

Introduction

Most industrial alloys contain second phase particles which have a strong influence on the recrystallization kinetics, microstructure and texture. Convenient combination of alloying and materials processing could control the distribution of second-phase particles in the microstructure. Consequently, with the understanding of the effects of these particles on the annealing behaviour, it is possible to use second-particles as a method of controlling the grain size and texture during thermomechanical processing.
The Al-Mg alloys have special attention due, not only to the lightness of the material, but also to certain mechanical properties and reciclability. They are classified as "non-heat-treatable", i.e., that cannot achieve higher strength by heat-treatment. These alloys obtain higher strength either by strain-hardening or by solid solution. However, some concentrations of magnesium (above 7%) increase your mechanical resistance with thermal treatment. Such alloys cause good mechanical properties in moderate mechanical efforts (400 to 700 MPa) and good resistance to the corrosion [1-5].
The aim of this work is the microstructural characterization by electron microscopy of an Al-Mg based alloys (values in weight) produced by powder metallurgy (hot compaction and extrusion technique) after thermal treatments. As complement, electrical resistivity and mechanical measurements were made. The employed P/M processing in this work (laboratory scale) shows to be more homogeneous comparing with the conventional metallurgy processing.

Theoretical Aspects

Several materials properties are strongly dependent of microstructure, such as resistance limit, elongation, toughness, ductile - fragile transition temperature, resistance to the

impact, wear resistance. Those properties are also influenced by the temperature, deformation rate in the processing and the way as the material is deformed. The nucleation of new grains is a non stability of the deformed microstructure, depending on subgrain size heterogeneities present as potential embryos in the deformed state adjacent to high local misorientation. The growth of the new grains depends on both the mean stored energy and on the frequency of new grains re-acquiring a low mobility boundary by meeting similar orientations in the deformed state [5 - 7].
During deformation, particles will affect the deformation microstructure and texture through effects such as an increase in dislocation density, the production of large deformation heterogeneities at larger particles and the alteration of the homogeneity of slip, e.g. shear bands. During annealing, the main effect of closely spaced particles is to hold grain boundaries, Zener pinning, but the deformation heterogeneities at large particles may be sites at which recrystallization originates ("particle stimulated nucleation"). It is significant the effect of second-phase particles on recrystallization and how to control the resulting microstructure and texture by the use of particles [5, 8].
In highly deformed polycrystal most work shows the nuclei to be of almost random orientation. It is possible to control the amount of random texture in the material through controlling the occurrence of "particle stimulated nucleation". It may be a preferential growth in the early stage due to their local environment or a selection of certain orientations from among those produced by particles stimulated nucleation or a preferential nucleation at particles in favoured sites such as grain boundaries [5, 8].
It has established that recrystallization is prevented or delayed by a dispersion of closely spaced second-phase particles, due to the pinning (Zener drag) of both high and low angle boundaries [5]. The influence of the particles depends on a number of factors and, in particular, on whether the particles are present during deformation or whether they form during the subsequent anneal before recrystallization occurs.

Materials and Methods

The P/M process utilized in this work was developed by Coelho [9], with material processing, in form of particles. Aluminium powder of was supplied with medium granulometry (25µm). The three alloy components (magnesium, zirconium and niobium) were carefully reduced in very small fragments without oxidation and after this, a final reduction, in a special P/M ball mill of high energy with argon atmosphere, taking in account the shavings removal, getting a powder with size of the order of millimetres. The processes included a previous mixes of the alloy elements in high-energy mill by 2 hours in recipient of UHMW Polyethylene and with steel spheres.
For the hot compactation process a high purity Al 1100 alloy made cup was used together filled mixed powder alloying elements, whose masses were previously measures and in mixture. This process does not need lubricants insertion in the mixture to minimize the powder abrasive effect with the inner wall of compaction tool. The final product of this process was 15 mm in diameter and 20 cm in length bar. It was necessary machining to remove 1 mm superficial layer due to the cup of aluminium, extruded during the process.
Concerning the first part of this work, the alloy Al-2Mg-0.6Zr (values in weight) was made by powder metallurgy techniques in our laboratory, employing hot compactation and extrusion processes (400 and 60 MPa pressures respectively, and 723K of temperature in both processes). After those processes were employed two cold rolling steps (79% and 90% in area reduction) for the recrystallization studies. Three isothermal heating (623, 723K and 873K) were utilized during 60, 600 and 6000 s. In the second

part, Al-2%Mg-1%Nb (values in weight) was also produced in laboratory by P/M techniques. After the processes of compaction and extrusion, the alloy was 89% cold worked and thermal heated at 373, 473, 573, 673 and 873K, each batch was air cooled after arrive at the specific temperature

The analysis of the degree of crystalline defects is also indirectly observed through mechanical tests such as microhardness analysis (in our case with a SHIMADZU type H machine) and electrical resistivity (Hewlett Packard model 4338 B) [10, 11].

The direct observation was made in a JEOL JEM 200C TEM and the chemical microanalysis with a Philips TEM (200kV) and XL30 SEM. The samples preparation followed the usual route of metallographic specimen preparation.

Results and Discussion

The analysis by SEM shows an evolution in the microstructure of precipitates with the increase of the time of thermal treatment, according to literature [4]. However the distribution of the precipitates in Al-2Mg-0.6Zr samples shows to be more homogeneous than those obtained in literature.

The scanning electron microscopy aspects of the Al-2Mg-0.6Zr alloy were earlier presented. Similarly was observed in Al -2Mg-1Nb. The microstructures show a great dispersion of fine precipitates of probably $NbAl_3$, showing little influence of the treatment temperature in the structural condition in the Al-2Mg-1Nb alloy an evolution of the microstructure and precipitates interactions in the micrographs of the samples of Al-2Mg-0.6Zr [1, 2].

The precipitates present in the Al-2Mg-1Nb alloy show round format, with very different dimensions to each other, not presenting appreciable amounts of niobium. The EDS analyses of several precipitate didn't detect presence of niobium in none of them. The occurrence of this phenomenon is owed to a very fine precipitation of Nb in the contour of these larger precipitates observed by TEM. However in the Al-Mg-Zr alloy, this rounded shape does not occur, but a formation of a complex structure with an aggregation of one or more fine precipitates, making fine “islands” of precipitates [10, 11].

The transmission electron microscopy shows an evolution of the microstructure and precipitates interactions in the samples of Al-2Mg-0.6Zr in study at both of isothermal heating. Initially shows a high degree of dislocation density interacting with precipitates at sample without treatment. The figure 1 show microstructures of P/M Al-2Mg-0.6Zr alloy, cold worked (79% reduced in area), the recovery process occurs at 623K during 60 s (fig. 1a) and 600 s (fig. 1b). The beginning of the recrystallization process occurs after 6000 s at 623K and also at 723K during 60 s (fig. 1c). At 823K during 6000 s shaped grains with high precipitates dispersion is observed (fig. 1d).

The figure 2 shows TEM micrographs of sample of the P/M Al-2Mg-0.6Zr alloy, cold worked (90% reduced in area.): At 623 K for 60 s recrystallization process still happens (fig. 2a) and also with 623 K for 6000 s (fig.2b). The development of the recrystallization processes influenced by the presence of precipitates with grain size refinement is also observed in figures 2c (T = 723 K after 600s) and 2d (T = 823 K after 60 s). The influence of higher cold work utilized in PM Al-2Mg-0.6Zr sample is remarkable observed in these micrographs.

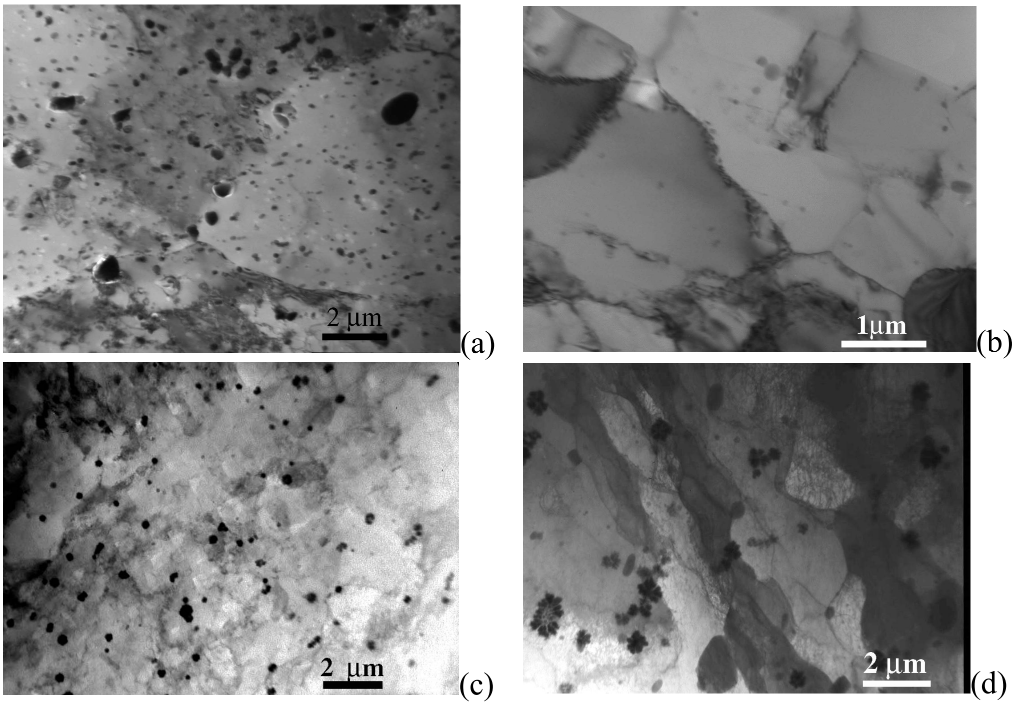

Figure 1 - TEM micrographs of sample of the P/M Al-2Mg-0.6Zr alloy, cold worked (79% reduced in area): a) 60 s, 623 K; b) 600 s, 623 K; c) 60 s, 723 K; d) 6000 s, 823 K.

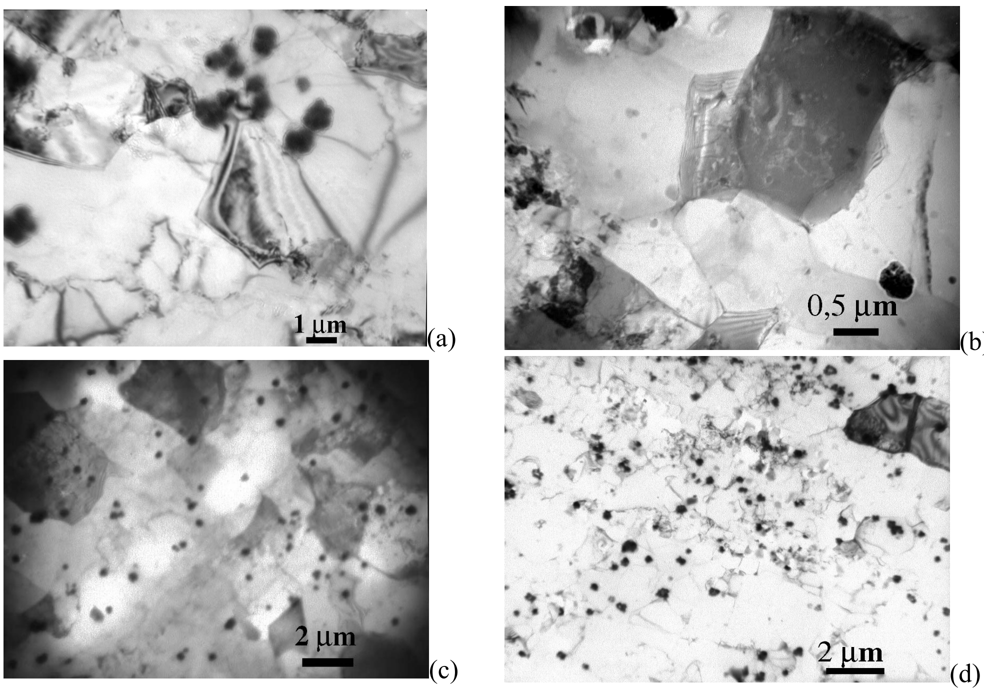

Figure 2 - TEM micrographs of sample of the P/M Al-2Mg-0.6Zr alloy, cold worked (90% reduced in area.): a) 623 K, 60 s; b) 623 K, 6000 s; c) 723 K, 600; d) 823 K, 60 s

The figure 3 shows the structural development after thermal treatments in Al-2Mg-1Nb alloy. Initially the microstructure presents a high density of dislocations, with intense dispersion of small size precipitates (with Al and Mg components). Figure 4 shows a detail of some of these precipitations surrounded by Nb precipitation (size of 20 nm) that was identified by EDS in TEM. The figures 5 and 6 also show the presence of small precipitates. The understanding of the observed phenomena depends of the fact that materials produced by powder metallurgy present complex interface reactions in a great amount of nucleation sites, and a subtle change in the structure of the material can mean an important variation in your properties.

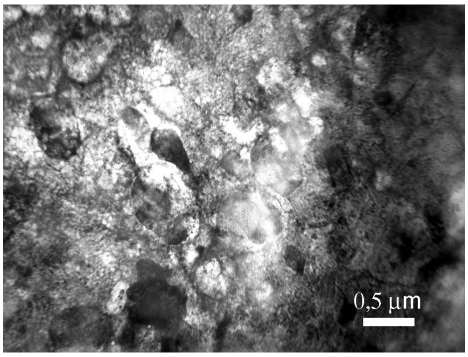

Figure 3. TEM of Al-2Mg-1Nb heat treated at 373K.

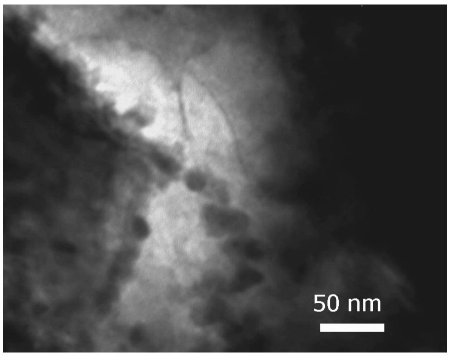

Figure 4. TEM of Al-2Mg-1Nb heat treated at 373K.

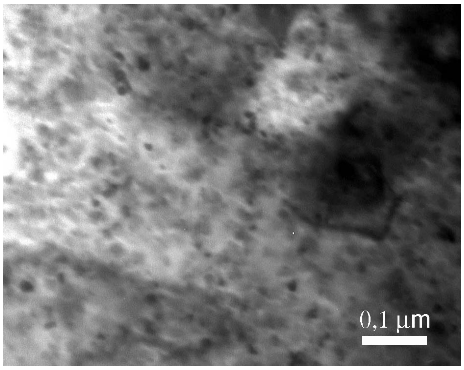

Figure 5. TEM of Al-2Mg-1Nb heat treated at 573K.

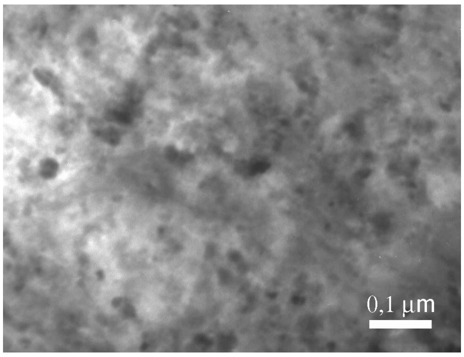

Figure 6. TEM of Al-2Mg-1Nb heat treated at 773K.

The mechanical properties of the Al-Mg-Nb alloys, in laboratory scale [10], are very superior to register for most of the aluminium alloys. This fact happens due to the great mechanical deformation imposed to the samples, causing great hardening, and due to the presence of fine precipitate containing niobium dispersed in the metallic matrix. Niobium participates of the constitution of a large amount of precipitates with small dimensions and its low mobility in the matrix causes the predominant mechanism in the hardening of the material.

The condition of thermal treatment brought great variations in the resistivity measurements, occurrence that it is not verified in alloys produced by conventional metallurgy; consequently it is considered valid the rule of Matthiessen that affirms that the electric resistivity of an alloy just depends of the chemical composition and the temperature. So, the effect of the microstructure of the studied alloy would have strong influence (in order of 3% in the value of the resistivity).

When the amount of crystalline defects is reduced, a decrease in the electric resistivity is expected, due to a reduction of the amount of points that benefits a larger “scattering" of electrons, improving their mobility when a difference of electric potential is applied. In Al-Mg-Nb alloys a reduction is observed in the value of the resistivity, larger than it is expected, due to the subtle structural transformations occurrence. In the temperature of treatment of 773K, an abrupt increase is observed in the value of the resistivity, due to the structural changes in this temperature [10].

The DSC analyses [10, 11] show different endothermic peaks for the Al-Mg-Zr and Al-Mg-Nb alloys. The relative curve to the Al-2Mg-1Nb alloy presents stable behaviour until the temperature of 503K, where the peak occurs, probably due to the beginning of $NbAl_3$ precipitation. Already the peak of Al-2Mg-0.6Zr occurs beyond the 723K, probably due to the phase transformations that can occur next to that temperature. That process in the Al-Mg-Zr alloys in study shows more notable, leading to fine grain structure after the thermal treatment. The understanding of the these observed phenomena depends of the fact that materials produced by powder metallurgy present complex interface reactions in a great amount of nucleation sites, and a subtle change in the structure of the material can mean an important variation in your properties.

Summary

Alloy in study presented interesting values of properties, with evident technological potential. The relationship between physical characteristics and thermal treatments, in all the done measures, suggests that materials produced by powder metallurgy present important changes in your properties with minimum alterations in your structural conditions, a study that it should be object of future works. The presence of both niobium and zirconium delays the recrystallization process, due to the formation of small precipitates which interact with the crystalline defects distributed in the matrix.

References

[1] S. J. Buso, W. A. Monteiro, Proc. Int. Conf. on Advanced Materials Processing Technologies (AMPT'01), Leganés, Madrid, Spain, 2001.

[2] S. J. Buso, W. A. Monteiro, Proc. Int. Conf. on Processing & Manufacturing of Advanced Materials (THERMEC'2003), Spain, 2003.

[3] B. N. Arzamasov, et al.: *Materials Science*, cap. 12. Mir Publishers Moscow, Moscow, 1989.

[4] J. E. Hatch: *Aluminum Properties and Physical Metallurgy*, ASM, USA, 1984.

[5] F. J Humphreys, and M. Hatherly: *Recrystallization and Related Annealing Phenomena*, Elsevier Science Ltd., 2nd edition, chapters 2-5 and 8, U.K., 1996.

[6] R. W. Cahn: *Recrystallization, Grain Growth and Textures*, Ed. H. Margolin, ASM, Metals Park, Ohio, 1966.

[7] R. D. Doherty, in: F. Haessner (Ed.), Recrystallization of Metallic Materials, Dr. Rieder Verlag, Berlin, 1978, p. 23.

[8] R. D. Doherty, D. A. Hughes, F. J. Humphreys, J. J. Jonas, D. Juul Jensen, M. E. Kassner, W. E. King, T. R. McNelley, H. J. McQueen, A. D. Rollett, "Current issues in recrystallization: a review", Materials Science and Engineering A238, 219–274, 1997

[9] R. E. COELHO, PhD Thesis, IPEN – USP, 2001 (in Portuguese).

[10] A. Almeida Filho, PhD Thesis, IPEN-USP, 2005 (in Portuguese).

[11] S. J. Buso, PhD Thesis, IPEN-USP, 2003 (in Portuguese).

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