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Characterization by TEM of a Supersaturated P/M Al-Mg-Zr Alloy after Thermal Treatments

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Abstract. Light materials have been studied thoroughly and used in components and parts in the automobile, naval and aerospace industries in the last decade. Their application makes possible: to reduce the mass, to increase the load capacity, increase of the speed and, and improvement in the mechanical properties when it is possible. Among those materials, the aluminium - magnesium alloys have special attention due, not only to the lightness of the material, but also to certain mechanical properties and reciclability.

The addition of zirconium in the Al-Mg alloys make it less susceptible to fatigue corrosion crack and it aids in the control of the natural aging, that causes the loss of the ductility. However the solubility of the zirconium in the Al-Mg alloys is low, not exceeding 0,28% for alloys manufactured by conventional metallurgy; and the use of not conventional techniques (powder metallurgy) it can increase to 2,5 to 5 times the limit of normal solubility. Previous works show that the addition of 0,1% in weight in an Al-10Mg alloy, it also increases the mechanical properties, being pronounced the superplasticity in these alloys.

The aim of this work is the microstructural characterization by transmission electron microscopy (TEM), of an Al-2Mg-0.6Zr (weight values) alloy, produced by powder metallurgy (hot compactation and extrusion technique), 79% cold worked, and them annealed at 623 K for several different times. Microhardness measurements were made to verify possible mechanical improvements. Differential Scanning Calorimetric (DSC) was made to verify possible range of phase transformation temperature.

Introduction

Light materials have been studied thoroughly and used in components of pieces in the automobile, naval and aerospace industries in the last decade. Their application makes possible: to reduce the mass, to increase the load capacity, increase of the speed and, and improvement in the mechanical properties when it is possible.

The principal criteria for selection of those materials for structural applications are: specific mechanical resistance (resistance-weight ratio) and the specific rigidity, that differs among the several materials of light alloys. [1,2]

Among those materials, the aluminium-magnesium alloys have special attention due, not only to the lightness of the material, but also to certain mechanical properties and reciclability. [1-3]

The aluminium has CFC structure, lattice parameter of 0,4041 nm, and show low density, high values of thermal and electric conductivity, ductility and corrosion resistance. The common impurities found in the aluminium: Fe, Si, Ti, Cu and Zn, in general, they worsen the physical properties previously mentioned. However, the inclusion of certain concentrations of impurities and mechanical treatments (plastic deformation, for instance) can improve the mechanical resistance and the hardness of the aluminium and their alloys.

The magnesium, with HCP structure, lattice parameter of 0,5199 nm, shows good machinability and capacity of absorbing mechanical loads (impact and vibrations, for example). Compared to the aluminium, has inferior values of thermal conductivity (33% of that), electric (50%), elastic module

(33%), but practically the same specific rigidity, and ductility and corrosion resistance decreases much more than for the aluminium, in presence of the same type of impurities. In pure form, the magnesium suffer oxidation very intensely when warm, what takes to problems in the fusion and molding of their alloys. In the powder form, it can begin ignition in to the air in usual temperatures (20 °C). [3]
The addition of zirconium in the Al-Mg alloys make it less susceptible to fatigue corrosion crack and it aids in the control of the natural aging, that causes the loss of the ductility. However the solubility of the zirconium in the Al-Mg alloys is low, not exceeding 0,28% for alloys manufactured by conventional metallurgy; and the use of not conventional techniques (powder metallurgy) it can increase to 2,5 to 5 times the limit of normal solubility. Previous works show that the addition of 0,1% in weight in an Al-10Mg alloy, it also increases the mechanical properties, being pronounced the superplasticity in these alloys. [2,4]

Experimental Procedure

The material in study, a bar (ϕ = 15 mm) of Al-2%Mg-0.6%Zr was produced by powder metallurgy techniques in the Laboratory of Powder Metallurgy of the Materials Science and Technology Center of IPEN. The processes included the previous mixes of the elements in high-energy mill by 2 hours in recipient of Polyethylene of the type UHMW (Ultra-High Molecular Weight) and with steel spheres.
For the hot compaction process a high purity aluminium 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 the insertion of lubricants in the mixture to minimize the abrasive effect of the powder with the inner wall of compaction tool.
The temperature for both processes was 450 °C and the pressures were 400 MPa in hot compaction process and 60 MPa in extrusion process.
After the process of compaction and extrusion the alloy was 79% cold worked, and them annealed at 623 K for several different times as show the table 1.

Table 1. Temperature and anneal treatment time for sample of Al-2Mg-0,6Zr alloy in study

Sample identification	Treatment time (s)	Temperature (K)
Al2Mg1	—	—
Al2Mg2	60	623
Al2Mg3	600	623
Al2Mg4	6000	623

Following the process of analysis, microhardness measurements were made in to verify possible mechanical improvements.
Samples were prepared for observation by transmission electron microscopy, which process of preparation followed the usual route, being obtained as final result fine disks with 3 mm in diameter, and thickness in the area of transmission of the order of dozens µm. The samples were observed to the microscope JEOL available JEM200C in the Centro de Ciência e Tecnologia de Materiais do IPEN.
With samples without thermal treatments, prepared as like for TEM observation, were analyzed by Differential Scanning Calorimetric (DSC), in the Laboratório de Análises Térmicas do Instituto de Química da Universidade de São Paulo.

Results and Discussion
The transmission electron microscopy shows an evolution on the microstructure in the samples of Al2Mg 0,6 Zr in study. Figure 1.a (Al2Mg1 sample) show fine precipitates distributed inside the

grains and crystalline defects, like dislocations, interacting with precipitates, The figure 1.b (Al2Mg2 sample) shows bands of dislocations interacting both with precipitates and each other. In Figure 1.c (Al2Mg3 sample) is observed a less degree of precipitates inside the grains and much greater degree of distribution in the between grains region; a less degree of crystalline defects is also observed. Figure 1.d (Al2Mg4 sample) is possible observe almost the same structure as in figure 1.c, with less degree of crystalline defects and more precipitates between grain. Is observed, also in the micrographies, an evolution on the shape and grain size.

Figure 2 shows the curve obtained from the Differential Scanning Calorimetric analysis, which no significant phase transformation is observed between the interval of time of the isothermal.

The figure 3 shows the curve from microhardness of the samples in study. Is observed a loss of hardness with the increase of time of thermal treatment.

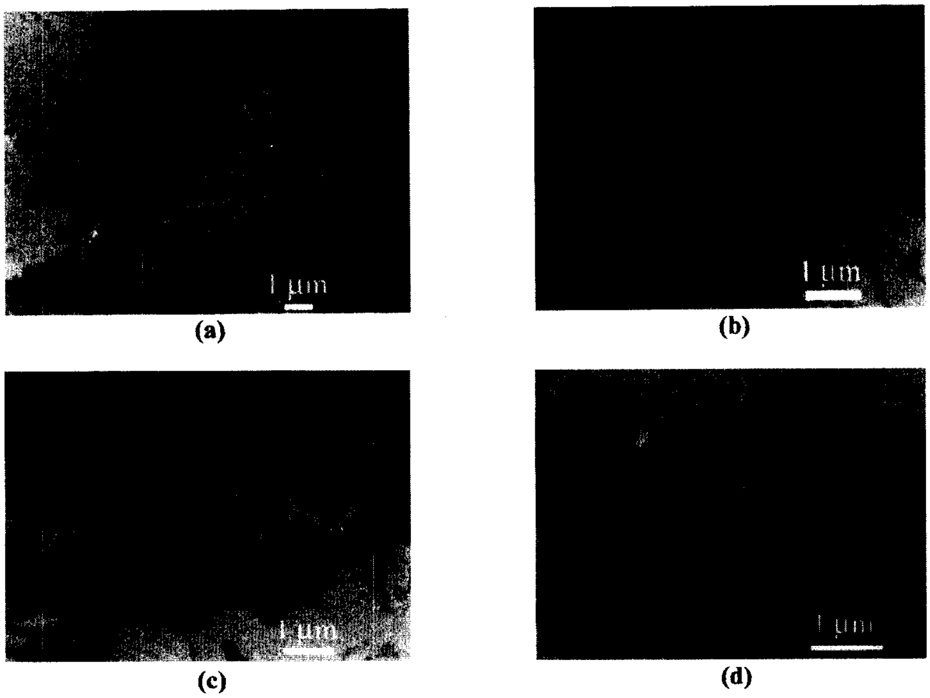

Figure 1. *Transmission electron microscopy of Al-2Mg-0.6Zr (weight values) alloy, produced by powder metallurgy (hot compactation and extrusion technique), 79% cold worked, and them annealed at 623 K for several different times: (a) sample Al2Mg1(without thermal treatment) showing fine precipitates inside the grains and crystalline defects, (b) sample Al2Mg2 (60 s) showing bands of dislocations interacting both with each other and precipitates, (c) sample Al2Mg3 (600 s) showing precipitates between grains region and crystalline defects and (d) sample Al2Mg4 (6000 s) showing precipitates between grains region and little less crystalline defects than (c).*

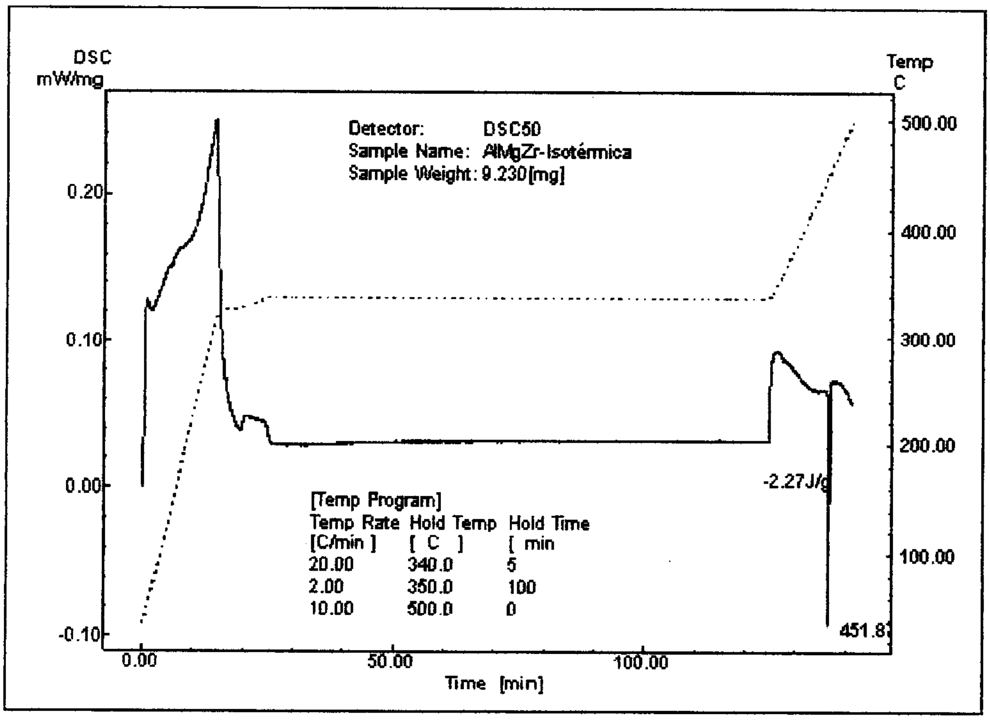

Figure 2. *Curve of Differential Scanning Calorimetric analysis of sample of Al-2Mg-0.6Zr (weight values) alloy, produced by powder metallurgy (hot compactation and extrusion technique), 79% cold worked.*

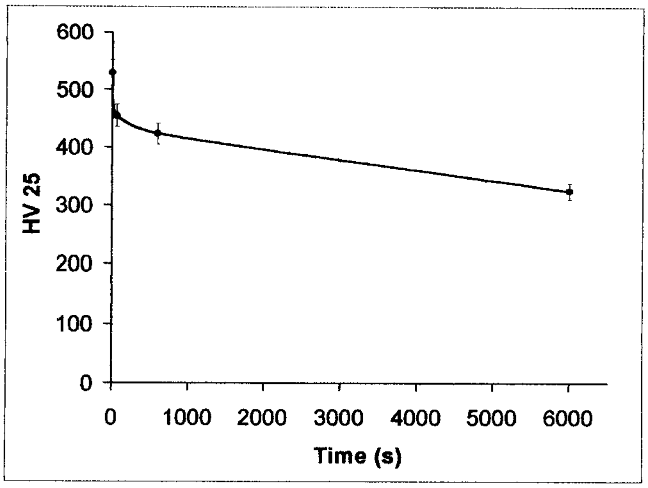

Figure 3. *Curve of microhardness analysis of samples of Al-2Mg-0.6Zr (weight values) alloy, produced by powder metallurgy (hot compactation and extrusion technique), 79% cold worked, and them annealed at 623 K for several different times*

Conclusions

The analysis of DSC curve does not show any phase transformation in the time of isothermal analysis. However changes in the microstructure were observed by TEM as expected. The micrographies shows a process of distribution of precipitates interacting with crystalline defects, crystalline defects annihilation and evolution in the shape and grain size.
These processes, with the fact that the alloy was produced by p/m in supersaturated state, can be the cause of the slightly loss of hardness observed in the microhardness curve.

Acknowledgements

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