Mechanical Properties of Hot-Extruded Products from Centrifugally Atomized Powders of Al-Li and Al-Li-Fe Alloys

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We present in this paper the method developed for the sintering of powders of Al-Li and Al-Li-Fe alloys, obtained as reported in Part I of this work. The main microstructural characteristics of compacted samples, after solution heat treatment and after aging at different temperatures for different periods of time, are described. We also report some mechanical properties of the compacted and heat treated alloys, obtained by means of microhardness and tensile stress measurements. Finally, a discussion is made on the main effects associated with the introduction of Fe to the Al-Li alloys.

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I. Introduction

The addition of lithium to aluminum, with the corresponding increase in Young's modulus and decrease in density makes this alloys very useful in several structural applications⁽¹⁾⁽²⁾. However, as mentioned in Part I of this work, the cast alloys are very brittle and techniques like those involving the production of powders by rapid solidification have to be used, which reduce the fragility of the alloys by reducing the grain size and improving the solute elements distribution. On the other hand, sintering of the powders to produce useful geometries introduces undesirable stress concentrations, which reduces the mechanical response of the alloys. Heat treatments are then needed to eliminate those stress concentrations and optimize the mechanical behavior of the alloys, by a controlled increase in size of δ' (Al₃Li) precipitates. But even in this case, features like precipitates-free-zones (PFZ) are present close to the grain boundaries, which reduces again the mechanical response of the alloys⁽³⁾⁽⁴⁾.

In order to correct for the decrease in ductility due to these PFZ's, one possibility considers the introduction of a second alloying element, which leads to the formation of small incoherent precipitates homogeneously distributed into the matrix and stable enough during heat treatments. Such stability can be reached by considering intermetallic compounds of the type Al_xI_y, where I_y are elements with low solubility and diffusivity in aluminum. Looking at the different phase diagrams, it becomes clear that these stability conditions are fulfilled by the following elements: Ca, Co, Cr, Dy, Er, Fe, Mo, Nd, V, W, Y

and Zr. It should be noted that in cast alloys the addition of these elements leads to the formation of big precipitates, which increases even more their fragility⁽¹⁾. In contrast, rapid solidification is expected to produce small precipitates homogeneously distributed into the matrix and hopefully stable during heat treatments. Moreover, this method should allow for an increase in concentration of the second alloying element and therefore in the volume fraction of the precipitates.

In the present work, iron was introduced as a third element in Al-Li alloys, and the characteristics of the resulting precipitates were studied. Also, the effects of heat treatments on the microstructure and the mechanical properties of compacted powders of Al-Li and Al-Li-Fe alloys are presented, which are expected to be of some interest for further work in the field.

II. Sintering of the Powders

Powders of the Al-Li and Al-Li-Fe alloys, with mean diameters of about 50 μm , (and standard deviation of about 30 μm), were produced by the method already explained in Part I of this work⁽⁵⁾⁻⁽⁷⁾. The powders were degassed before sintering by using the device shown in Fig. 1. This consists in an evacuated chamber with a collecting capsule in duralumin at the bottom. The powder on the supporting plate is heated in vacuum (6.7×10^{-4} Pa) at 473 K for 50 h with an electrical furnace. After degassing, the supporting plate is push down from the outside and the powder falls into the collecting capsule at the end of the collecting tube. The tube is strangled mechanically on its upper side and separated from the system by sawing it at the strangled zone. The sawed end was welded before sintering in order to prevent the admission of air into the collecting tube. By this procedure the vacuum is maintained inside the tube during the rest of the process, preventing the reaction of the powders with air.

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Fig. 1 Schematic of the device used for the degassing of the powders. Thermocouple T, vacuum pipe V, stainless steel chamber A, powders P, electrical furnace F, supporting plate N, collecting tube U and collecting capsule C.

Fig. 2 Section of the rod of compacted powders (R). The outside shield (S) corresponds to the duralumin collecting capsule after extrusion.

Sintering of the powders was made by hot extrusion at 693 K of the filled collecting capsule. With this method, rods of 10 mm in diameter and 1.5 m in length, with a core of sintered Al-Li or Al-Li-Fe powders (6 mm in diameter), covered with a shield of duralumin, were obtained, as shown in Fig. 2. The shield in duralumin was separated mechanically and tensile samples were cut from the inner rod of compacted powders. Figure 3 shows the geometry and normalized dimensions of the tensile samples. Densities of $2.5 \pm 0.05 \text{ Mg/m}^3$ were measured for both compacted alloys, which represents about 100% and 96% of the values that we have measured for cast Al-Li (2.5 Mg/m^3) and Al-Li-Fe (2.6 Mg/m^3) alloys respectively.

Figure 4 shows an optical micrograph of a typical zone

Fig. 3 Geometry and dimensions of the samples. Dimensions are in millimeters.

Fig. 4 Optical micrograph of a typical zone in compacted powders of the Al-Li alloy.

Fig. 5 TEM micrograph of an Al-Li alloy after sintering, showing dislocation tangles around $\delta(\text{AlLi})$ precipitates.

in compacted powders of the Al-Li alloy⁽⁸⁾⁽⁹⁾, where grains $2\text{--}8 \mu\text{m}$ large can be clearly seen. Similar grain sizes were measured in Al-Li-Fe alloys.

Transmission electron microscopy observations of Al-Li powders, after sintering at 693 K, revealed the presence of high dislocation densities (not measured) and the growing of large $\delta\text{-AlLi}$ precipitates ($0.01\text{--}0.02 \mu\text{m}$). Figure 5 is a TFM micrograph showing those δ -precipitates surrounded by dislocation tangles, which produces high local stress concentrations.

TEM observations of Al-Li-Fe powders after sintering

Fig. 6 TEM micrograph of an Al-Li-Fe alloy after sintering, showing: (a) Al_3Fe (needle shape-N) and Al_6Fe (globular shape-G) precipitates. (b) Small Al_3Li and one large AlLi (δ) precipitates.

(Fig. 6(a)) evidenced the presence of two types of Al-Fe precipitates, that we have identified as Al_6Fe and Al_3Fe . The first ones (Al_6Fe) are larger (200–250 nm) and had a globular shape; they were observed to form as well at the interior of the grains and at the grain boundaries. The second ones (Al_3Fe) are smaller (50–150 nm) and needle-shaped, and were formed at the interior of the grains. The size of both these precipitates was larger than expected, and resulted probably from the high temperature heat treatment experienced by the samples during the sintering of the powders. Also, AlLi (δ) and Al_3Li (δ') precipitates were observed within the grains and are shown in Fig. 6(b).

III. Heat Treatments of the Compacted Alloys

A solution heat treatment of the tensile samples of both alloys was made at 813 K for 30 min, followed by quenching into water at room temperature. This treatment leads to the elimination of internal stresses (dislocations) by recrystallization from the arms of the dendrites. Straight edge grains, as the ones shown in Fig. 7, with sizes between 2–10 μm were observed after recrystallization. Also, δ - AlLi precipitates were observed to disappear after this treatment; however, Al_6Fe and Al_3Fe precipitates were still present in the grains.

The tensile samples were then aged at 453, 473 and 493

Fig. 7 Straight edge grains in solution heat treated Al-Li alloys.

Fig. 8 δ' (Al_3Li) precipitate free zones (PFZ) in Al-Li alloys after aging at 473 K, for 16 h.

Fig. 9 Al_3Fe precipitates (N) in an Al-Li-Fe alloy after aging at 473 K for 16 h. Note the presence of these precipitates into the δ' -PFZ.

K for 12, 16, 20 and 24 h. These treatments resulted in the growing of the small δ' precipitates obtained during the quenching, and in the formation of δ' -PFZ (100–120 nm wide) along the grain boundaries. The δ' -PFZ in the Al-Li alloy are shown in the TEM micrograph of Fig. 8. In addition, Al_3Fe needle shape precipitates (50–150 nm long) appeared homogeneously distributed in the whole grain of the Al-Li-Fe alloy, as shown in Fig. 9. No Al_6Fe

precipitates were observed after aging. It is interesting to note that the size of the Al_3Fe precipitates does not change significantly during these heat treatments. This fact can be understood by considering the low solubility limit of Fe in Al at these temperatures. As mentioned before, the measured final size of these precipitates was probably obtained during the compaction of the powders, where the heating to higher temperatures (693 K) was necessary. It is worthwhile noting that no significant changes in the subgrain size were observed by the addition of Fe.

The mean diameter of the Al_3Li precipitates after aging at 493 K for 20 h was found to be comprised between 30–40 nm; this value was only about 2–4 nm after the solution heat treatment.

IV. Mechanical Properties of the Compacted Powders

Microhardness measurements were made on the just compacted powders and in the solution heat treated and aged ones. Figure 10 shows microhardness curves as a function of the aging time at temperatures of 453, 473 and 493 K for compacted powders of the Al-2.4 mass% Li and Al-2.4 mass% Li-1 mass% Fe alloys. Also shown in this figure are the measured values of microhardness for the samples just after compaction and also after solution heat treatment (i.e. unaged samples). Each point in these curves represents the mean value of a series of ten measurements. It is clear from the curves in Fig. 10, that for both alloys the maximum mean value in microhardness is obtained for an aging temperature of 493 K. For almost all the aging temperatures the best aging time was 20 h, except for the samples of Al-Li-Fe alloy aged at 493 K, where a 16 h time was found to produce the highest mean value of microhardness.

Tensile stress-strain curves were obtained at room temperature for tensile samples of both alloys at a constant elongation rate of 0.3 mm/min, which correspond to a constant deformation rate of $2.5 \times 10^{-4} \text{ s}^{-1}$. For each alloy three kind of samples were deformed to fracture: 1) Just compacted samples, 2) solution heat treated samples

Table 1 Yield stress at 0.2% of deformation ($\sigma_y(0.2)$), maximal stress (σ_m) and maximal strain (ϵ_r) for several Al-Li-X alloys.

Alloy	$\sigma(0.2)$ (MPa)	σ_m (MPa)	ϵ_r (%)	References
Al-2.4 mass% Li(C)	223	259	2.4	This work
Al-2.4 mass% Li (JC)	231	224	13.5	This work
Al-2.4 mass% Li-1 mass% Fe (JC)	300	278	14.5	This work
Al-2.4 mass% Li (SHT)	264	263	15.5	This work
Al-2.4 mass% Li-1 mass% Fe (SHT)	232	205	15.5	This work
Al-2.4 mass% Li	312	423	6.2	This work
Al-2.7 mass% Li	296	396	5.5	(7)
Al-2.4 mass% Li-1 mass% Fe	366	432	5.2	This work
Al-2.78 mass% Li-0.49 mass% Fe	286	372	8.2	(7)
Al-2.95 mass% Li-0.27 mass% Zr	296	409	8.3	(7)
Al-3.56 mass% Li-0.36 mass% Fe-0.48 mass% Ni	378	464	4.8	(10)
Al-3.8 mass% Li-0.48 mass% Fe-0.43 mass% Co	391	468	4.5	(10)

(C) Cast alloy, (JC) just compacted, (SHT) solution heat treated.

and 3) samples aged at 493 K for 20 h. In each case, at least 10 samples were tested and the mean values of the yield stress at 0.2% of deformation $\sigma(0.2)$, the maximal stress σ_m and the deformation to fracture ϵ_r were obtained. These values are listed in Table 1, together with the same values for other Al-Li based alloys, as reported in the literature. It is to be noted that the values obtained in this work are comparable with those reported for other powder processed alloys.

V. Discussion

Several remarks can be made following the different observations and results in this work. First, it should be noted that the centrifugal atomizing method reported in Part I of this work and the sintering process and heat treatments described above appears as good alternative methods for the preparation of Al-Li and Al-Li-Fe alloys. As shown in Table 1, the mechanical properties of the alloys are similar with those exhibited by other powder processed alloys. Particular interest has to be given to the similar values in mechanical properties shown by Al-Li alloys having addition impurities in concentrations of about 1%, indistinctly of whether these are only Fe or combinations of Fe and Co or Ni.

The centrifugal atomizing method produced an adequate distribution of diameters of the powder particles, and the sintering method lead to the obtention of rods whose density was close to the theoretical one. Also, the diameter of the Al_3Li precipitates, measured after the solution heat treatment, appears as an adequate starting value for further growing in a controlled way during aging; then allowing for the optimization of the mechanical properties.

Concerning the effects of the addition of Fe to the Al-Li alloys, it should be noted that small Al_3Fe precipitates were observed to distribute homogeneously within the grains and remain stable in the course of heat treatments. These are important characteristics of Al_3Fe precipitates

Fig. 10 Microhardness curves as a function of the aging time at different temperatures for Al-2.4 mass% Li and Al-2.4 mass% Li-1 mass% Fe. JC=just compacted, SHT=solution heat treated.

since, as stated before, their main function was to compensate the undesired effects of the δ' -PFZ's on the mechanical properties of the alloys. Measurable increases in $\sigma(0.2)$ and σ_m were obtained in the alloy with Fe impurities. It is left as a further work the study of the effect of higher Fe contents in the mechanical properties of the Al-Li alloys.

The addition of Fe to the alloys also results in a different mechanical behavior of the solution heat treated samples, as it can be deduced from the hardness values in Fig. 10, but also from the values of stresses in Table 1. This can be explained by considering that during solution heat treatment two contributions with different sign actually determines the values of microhardness, yield stress and maximal stress of the alloys: 1) a decrease in those values is obtained by stress relaxation associated with the elimination of the dislocations introduced during sintering of the powders, and 2) an increase in those values is obtained by the coarsening of δ' -Al₃Li precipitates. In the case of Al-Li alloys the second mechanism becomes more important, and therefore slightly higher values of microhardness, $\sigma(0.2)$ and σ_m are measured after solution heat treatment. In contrast, in Al-Li-Fe alloys the presence of Al₆Fe and Al₃Fe precipitates probably inhibits the growing of δ' precipitates and the corresponding solution hardening; then, in Al-Li-Fe alloys, hardness and stress levels are effectively decreased by the elimination of dislocations during the solution heat treatment.

VI. Conclusions

(1) Good quality rods of Al-Li and Al-Li-Fe alloys (6 mm in diameter and 1.5 m in length) were produced by hot extrusion (693 K) under vacuum conditions.

(2) Solution heat treatment at 813 K for 30 min leads to the elimination of large (0.01–0.02 μm) δ -AlLi precipitates obtained during the hot extrusion. In the Al-

Li-Fe alloys Al₆Fe and Al₃Fe precipitates were still present and uniformly distributed.

(3) Aging at 693 K for about 20 h was found to produce the best mechanical properties. After this treatment Al₃Li precipitates with a 30–40 nm mean diameter were observed; this value was only 2–4 nm after solution heat treatment. δ' -PFZ (100–120 nm wide) were also observed along the grain boundaries after aging. In Al-Li-Fe alloys no Al₆Fe precipitates were detected after the same treatment, but Al₃Fe needle shape precipitates (50–150 nm long) appeared distributed homogeneously in the whole grains (including the δ' -PFZ); no significant change in size was observed for these precipitates after solution heat treatment and aging.

(4) In Al-2.4 mass% Li-1 mass% Fe we have measured values as high as 366 MPa for the yield stress at 0.2% of deformation, 432 MPa for the maximal stress and 5.2% for the maximal strain.

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