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Effect of Mechanical Alloying and Ti Addition on Solution and Ageing Treatment of an AA7050 Aluminium Alloy

Kátia Regina Cardoso[a], Dilermando Nagle Travessa[b], Asunción García Escorial[c], Marcela Lieblich[c]*

[a]*IP&D, UNIVAP, Av. Shishima Hifumi, 2911, 12244-000, São José dos Campos - SP, Brazil*
[b]*Embraer-Empresa Brasileira de Aeronáutica S.A., Av. Brigadeiro Faria Lima, 2170, 12227-901, São José dos Campos - SP, Brazil*
[c]*CENIM, CSIC, Avenida Gregorio del Amo, 8, 28040 Madrid, Spain*

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In this work, solution heat treatments at different temperatures were performed in a commercial based AA7050 aluminium alloy, with and without titanium addition, produced by mechanical alloying and hot extrusion with the aim to investigate the effect of titanium addition and mechanical alloying in the precipitates stability. The same heat treatment conditions were used in a reference sample obtained from a commercial AA7050 alloy. Solution heat treated samples were characterised by differential scanning calorimetry (DSC), optical microscopy and hardness test. Once the better temperature for the solution treatment of modified alloys was defined, the ageing curve at 120 °C was obtained to verify the effect of milling and Ti addition in the precipitation and in the maximum values of hardness obtained for the alloys.

Keywords: *mechanical alloying, aluminium alloy, heat treatment*

1. Introduction

Structural applications of aluminium alloys at high or moderate temperatures require a fine, homogeneous and stable distribution of particles, to guarantee dispersion hardening up to the temperature of use. High melting point intermetallic phases are good candidate for that. Al_3Ti is very attractive among all intermetallics, because it has a high melting point (~1350 °C) and relative low density (~3.3 $g.cm^{-3}$)[1]. Although particle dispersion can be obtained by conventional ingot metallurgy, problems as formation of coarse intermetallic particles and segregation during the ingot solidification usually take place[2,3]. These problems can be overcome by using powder metallurgy and more specifically mechanical alloying as processing route[1,4-6]. Mechanical alloying is a solid-state process that enables refining of the microstructure, homogenisation and extension of solid solubility limits[7,8]. The possibility of higher solubility could be interesting to enhance precipitation of equilibrium phases in the heat treatable aluminium alloys.

The highest strength aluminium alloys with application in the aircraft industry are those of 7XXX series, a heat treatable alloy. As reported in previous works[9,10], a commercial AA7050 based aluminium alloy was produced by mechanical alloying with titanium addition with the aim to produce a dispersion-hardened alloy by Al_3Ti particles. Mechanical alloying, in the same process conditions used to produce the dispersion-hardened alloy, was also utilised to obtain a reference powder of the AA7050 aluminium alloy without titanium addition. The precursor powders produced by mechanical alloying were hot extruded and the consolidated samples in the as-extruded condition were characterised. Results showed that mechanical alloying increased the mechanical properties of the AA7050 aluminium alloys when compared to the commercial one due to strengthening mechanisms resulting from the milling such as grain size refinement to nanometric scale and increase in dislocation density due to severe deformation caused by the process. The addition of titanium produced additional strengthening due to Al_3Ti formation. The formation of Al_3Ti phase also had a pronounced effect on the elastic modulus that attained the highest value among the three 7050 alloys[9,10], commercial, mechanical alloyed and mechanical alloyed with Ti addition.

In this work, samples of AA7050 aluminium alloy with and without titanium addition that were produced by mechanical alloying and hot extrusion were submitted to solution heat treatment at different temperatures, and aged at 120 °C, a typical temperature for maximum strength on commercial AA7XXX alloys. The aim of the heat treatment was to evaluate the effect of titanium addition and milling in the precipitates stability and in the precipitation. The same heat treatment conditions were used in a reference sample obtained from a commercial AA7050 alloy.

2. Experimental

As reported elsewhere[9], powder of an AA7050 based alloy with nominal composition Al -2.4 Cu-2.4 Mg-6.2 Zn (wt. (%)) with addition of 1 wt. (%) of titanium (7050 MA1%Ti) was produced by mechanical alloying using pure elemental powders as starting materials. The same alloy, without titanium addition (7050 MA), was produced at the same milling conditions. Mechanical alloying was carried out using a planetary ball mill (Fritsch Pulverisette 5) for 100 hours using a ball to powder ratio of 20:1. Further details of milling are reported in[9]. The precursor powders were consolidated by hot extrusion at 400 °C in a horizontal press with an extrusion ratio of 37:1 and extrusion rate of 0.3 mm/s. Results of microstructural and mechanical characterisation of extruded bars are reported in[10].

Samples were submitted to solution heat treatments at 440, 478 and 500 °C for one hour followed by water quench. The temperature of 478 °C is recommended for solution heat treatment of the commercial alloy[11]. The two other temperatures were chosen in order to evaluate eventual changes on the ideal temperature for precipitates dissolution as the alloy is submitted to both MA and Ti addition. Hardness tests using the Rockwell B scale were performed immediately after quenching in order to evaluate the efficiency of

*e-mail: katiarc@univap.br

solution heat treatments. At least five indentations were done on each sample. Differential scanning calorimetry was performed on a DSC 200 F3 Maya from Netzsch with a heating rate of 10 °C/min. The solution heat treated alloys were prepared for optical microscopy analysis by conventional metallographic techniques.

Based on the results of optical microscopy analysis, a fourth solution heat treatments temperature of 455 °C was chosen for samples 7050 MA and 7050 MA1%Ti. All samples were aged at 120° at different times (from 10 to 2430 minutes) and ageing curves based on hardening measurements were obtained.

3. Results and Discussion

Table 1 shows results of hardness tests on samples in the as-extruded and solution heat treated conditions. Comparing the hardness values of samples in the as extruded condition with that of solution treated alloys, it can be observed a pronounced softening of the commercial alloy, while only a small softening is observed in the modified and mechanically alloyed alloys. Evidences of eutectic melting during solution heat treatment of 7050 MA1%Ti at 478 °C did not encourage the treatment of such alloy at 500 °C. It is well established that MA results in grain size refining, work hardening and intermetallic dispersion, when intermetallic precursors are present. Consequently, the high hardness of the 7050 MA and 7050 MA1%Ti alloys in the as extruded condition and after the solution heat treatment can be result of the same hardening mechanisms described above, once Al_3Ti phase particles are stable until the solution temperatures employed, and probably appreciable grain growth did not occur during heat treatment. Al powder oxidation occurring during MA can also result on additional dispersion hardening due the formation of Al_2O_3 particles[12,13.]

Figure 1 presents the DSC curves obtained after the solution treatments. Exothermic peaks related to precipitation events are observed. Values of enthalpy for each individual peak and the total enthalpy until 300 °C are presented in Table 2. The enthalpy releases for modified and mechanically alloyed alloys are considerable smaller than that for commercial alloy. Besides that, the two precipitation peaks that are overlapped in the DSC curve of the commercial alloy are separated in the mechanically alloyed samples, and the first peak is found at lower temperature. For commercial alloy these two peaks correspond typically to the precipitation of metastable η' $MgZn_2$ and stable η $MgZn_2$ phases[14]. These results show that for commercial alloy, as expected, the better temperature of solution treatment, among the three used in this work, is that indicated by commercial practices[11], 478 °C. However, the same is not valid for the other two alloys, as the efficiency of the solution heat treatments at the three different temperatures is much lower, as showed by the small values of enthalpy. The lower enthalpies in the Ti containing alloy can be attributed to reduction of stability of solid solution by nucleation of new Zn-Mg phase on the surface of the intermetallic particles[15]. As particles of Zn-Mg phase are precipitated mainly on the matrix-particle interface, the density of the Zn-Mg particles in the bulk of the matrix decreases sharply. The reduction of enthalpy can also be attributed to the Al_3Ti particles that act as vacancy sink, resulting in a quenched alloy with a low concentration of excess vacancies, which are preferential sites to nucleation of GP zones, thus reducing precipitation capacity of the 7050 alloy[16]. Al_2O_3 small particles originating from the layer that always cover the surface of aluminium and become broken during the high energy mechanical alloying process also may act as vacancy sinks and as reinforcing elements. Dislocations can act as vacancy annihilation sites too. An increased density of these defects can be present in these mechanically alloyed samples and additional dislocations can be resulting from the differential thermal contraction between the intermetallic/ceramic particles and the surrounding matrix, which is produced during solution heat treatment[17].

Table 3 shows optical micrograph images of 7050 commercial, 7050 MA and 7050 MA1%Ti alloys after solution heat treatments at 440, 478 and 500 °C. The commercial alloy presents after solution heat treatment at 440 and 478 °C an elongated structure of grains typical of a material deformed by a process as rolling. The equiaxial structure of grains after the solution treatment at 500 °C evidences that recrystallisation has taken place. It can be observed an intercrystalline failure in the sample treated at 500 °C that can be result of partial melting of the alloy. The mechanical alloyed 7050 MA and the modified 7050 MA1%Ti samples, presented a very fine microstructure not resolved by optical microscopy, as can be observed in Table 3. The failures, probably resulting from partial fusion, already appear after

Table 1. Rockwell B Hardness testing results (HRB) and standard deviations (s), for samples in the as-extruded condition and after solution heat treatment at three temperatures.

	As-extruded		Solution heat treatment					
			440 °C		478 °C		500 °C	
	HRB	s	HRB	s	HRB	s	HRB	s
7050 commercial	83*	-	45	1.52	44	3.8	43	3.3
7050 MA	84	0.38	84	0.75	80	1.17	71	0.99
7050 MA1%Ti	89	0.92	88	0.89	76	1.09	-	-

* Typical Rockwell B hardness for AA7050 aluminium alloy at T7451 condition.

Table 2. Enthalpy releases in $J.g^{-1}$ during DSC analyses of solution heat treated alloys.

	Solution heat treatment								
	440 °C			478 °C			500 °C		
	ΔH_1	ΔH_2	ΔH_T	ΔH_1	ΔH_2	ΔH_T	ΔH_1	ΔH_2	ΔH_T
7050 commercial	–14.7	–2.0	–16.7	–14.3	–2.9	–17.2	–12.1	–2.6	–14.7
7050 MA	–0.7	–5.8	–6.5	-1.1	–5.0	–6.1	–0.7	–6.0	–6.7
7050 MA1%Ti	–0.6	–3.5	–4.1	–0.8	–4.6	–5.4	-	-	-

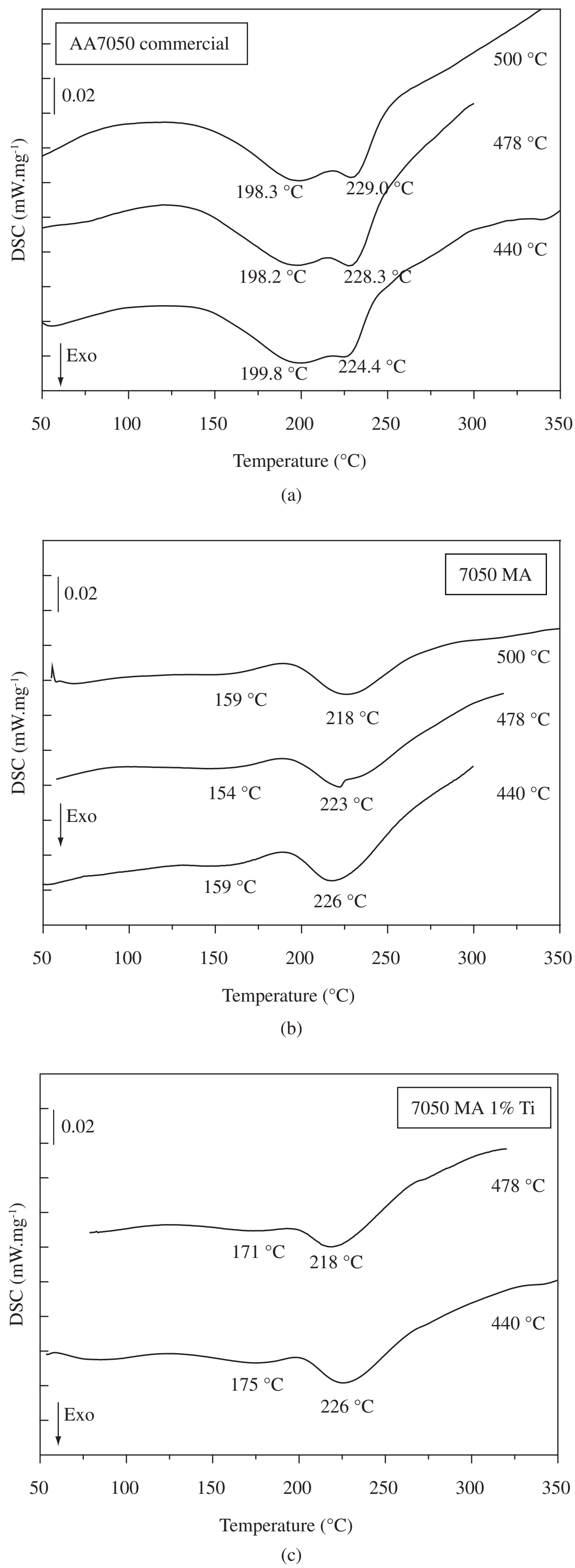


Figure 1. DSC curves of 7050 commercial, 7050 MA and 7050 MA1%Ti alloys after solution heat treatments at 440, 478 and 500 °C.

the solution heat treatments at 478 °C. Thus, mechanical alloying and titanium addition in the AA7050 alloy restricts the maximum solution temperature to below 478 °C.

Taking into account these results, the 7050 MA and 7050 MA1%Ti alloys were solution heat treated at 455 °C and aged at 120 °C. Figure 2 shows the ageing curves for these alloys, together with results of commercial alloy also aged at 120 °C, after solution heat treatment at 478 °C. The mechanical alloyed alloys show higher hardness than the commercial one in the aged condition, being the highest value obtained in Ti containing alloy. Although the hardness values of modified alloys are higher than that of commercial alloy, the gain in this property with the ageing treatment is very low, as shown in the Figure 2. These results, together with the DSC results, indicate that mechanical alloying efficiently improves the hardness of the 7050 aluminium alloy in the as-extruded condition due to grain refining and probably the reinforcing effect of Al_2O_3 dispersoids. Moreover, hardness of the MA materials in the as-extruded condition, i.e. without any further heat treatment, can be as high as that of commercial alloy in peak age condition, i.e. after solution treatment and ageing. On the other hand, this processing hinders the age hardening capacity of the alloy, probably due to the increase in vacancy and solute sinks. Based on this, it is evident that traditional heat treatments employed on aluminium commercial alloys are not suitable for these new MA alloys and optimised conditions for heat treatments need to be attained, which will require a better understanding on the effect of mechanical alloying in the precipitation kinetics and products.

4. Conclusions

Mechanical alloying and Ti addition increased the hardness of AA7050 aluminium based alloy in the as-extruded condition as well as after all heat treatments that these alloys were submitted to in this work.

The low enthalpy associated to precipitation after solution heat treatment in the modified and mechanically alloyed alloys could be result of Al_3Ti particles and Al_2O_3 dispersoids that act as vacancy/solute sinks reducing precipitation hardening capacity of the alloy. Furthermore, the small reduction in hardness after solution heat treatment at 440 °C and partial melting observed of 478 to 500 °C evidences that a satisfactory thermal processing was not attained on these alloys.

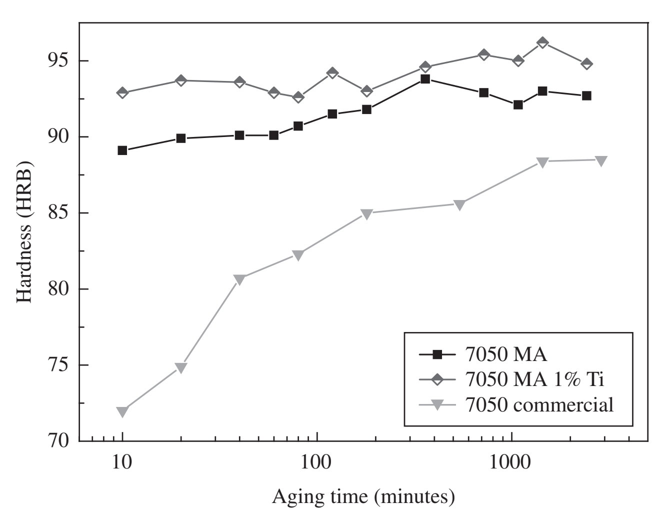
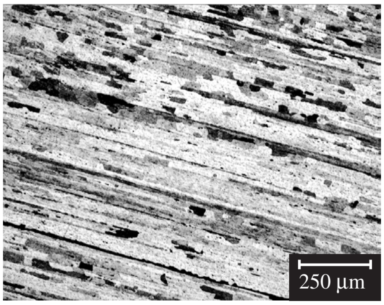
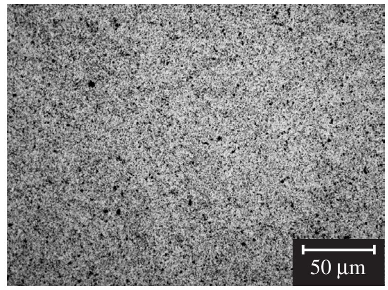
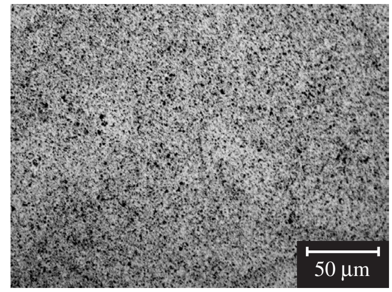
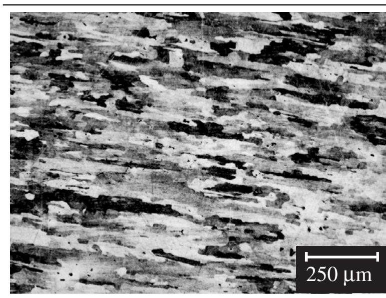
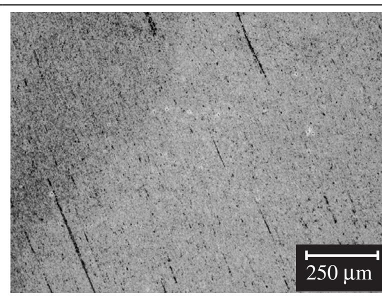
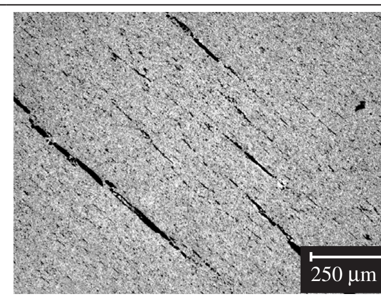
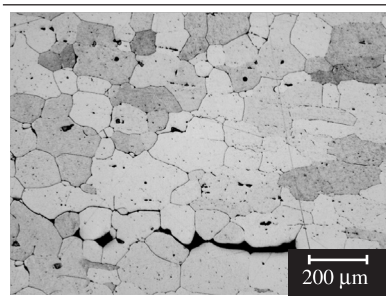
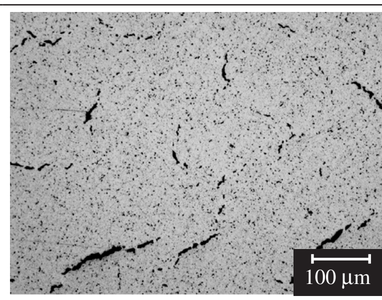


Figure 2. Ageing curves at 120 °C of 7050 commercial, 7050 MA and 7050 MA1%Ti alloys. 7050 commercial was solution treated at 478 °C and 7050 MA and 7050 MA1%Ti at 455 °C.

Table 3. Optical micrographs of the 7050 commercial, 7050 MA and 7050 MA1%Ti alloys after solution heat treatments at 440, 478 and 500 °C.

7050 commercial	7050 MA	7050 MA1%Ti	
			440 °C
			478 °C
			500 °C

The precipitation was affected by mechanical alloying as shown by DSC curves: precipitation peaks were separated and the first event was accelerated.

Optimised conditions for heat treatment of these alloys could be attained with a better understanding on the effect of mechanical alloying in the precipitation kinetics and products.

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