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Influence of lithium on the mechanical properties of metallic composite materials based on the eutectic Al–5.7% Ni alloy

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The effect of lithium addition was examined on the structure and the mechanical properties of metallic composite materials, prepared from the binary eutectic Al–5.7 wt% Ni alloy by casting and isostatic extrusion. A small amount of lithium addition significantly increases the tensile strength. The beneficial effect of lithium addition is due to solid-solution hardening for materials with low lithium content, i.e. those with less than about 1.7 wt%; in addition, precipitation hardening due to the formation of small spherical particles of δ′-Al_3Li phase in the aluminium matrix has an increased effect for materials of higher lithium content.

1. Introduction

We have developed in our previous work [1–4] a new type of metallic composite material based on the eutectic Al–5.7 wt % Ni alloy. These materials are prepared as follows. A eutectic alloy is first cast under argon gas in a copper mould and solidified with a relatively high cooling rate. We thus obtain a eutectic structure consisting of a ductile aluminium matrix containing fine, strong Al_3Ni fibres. The alloy is then severely deformed by isostatic extrusion or swaging. The fibrous intermetallics are aligned in the extrusion direction and they are broken to form fine, dispersed particles in the ductile matrix. These materials possess good mechanical tensile strength, nearly 300 MPa, comparable to that achieved by the unidirectionally solidified eutectic Al–Al_3Ni alloy. The mechanical strength of these materials is further remarkably increased by the addition of a small amount of copper, manganese [3] or magnesium [4]. It attains about 400 MPa by the addition of 2 to 3 wt % Cu or of 1 to 2 wt % Mg, and more than 500 MPa by the addition of 3 wt % Mn.

The development of Al–Li alloys and technologies resulting therefrom is proceeding at a remarkably rapid pace during this decade, as documented by the proceedings of successive conferences on these materials [5–8]. Lithium is recognized as a promising element for addition to engineering aluminium alloys by its low density and strengthening effect due to the formation of fine precipitates of δ′-Al_3Li and other phases. We consequently investigated in this work the influence of lithium addition on the structure and mechanical properties of eutectic Al–5.7 wt % Ni composite materials.

2. Experimental procedure

2.1. Preparation of the composite materials

The alloy specimens were prepared by casting and isostatic extrusion in a way similar to that described previously [3]. In the case of lithium addition, however, some technical difficulties appear because of its low fusion temperature and high reactivity. After several attempts, we used the following procedures: the Al–Li mother alloys were first prepared from an aluminium and lithium mixture by induction heating in a crucible under argon gas and casting in a copper mould. The dimensions of the rectangular cast ingots were 28 mm × 37 mm × 135 mm. The lithium content in the obtained ingots varied from 2 to 6.5 wt %. By using these mother alloys, the Al–Ni–Li alloys were prepared by fusion and casting under argon gas.

Table I summarizes the five kinds of alloy used in this work. Alloy Nos 1 to 4, prepared in a graphite crucible, contain a small amount of carbon, but No. 5 was prepared in an alumina crucible and is fairly clear of carbon. The other minor impurity elements were not analysed.

The cylindrical cast ingots of 22 mm diameter were placed in a copper capsule and then severely deformed by isostatic extrusion. The extrusion ratio R, defined by the ratio of sectional areas of the ingot before and after extrusion, was 4.8.

2.2. Characterization of structure and mechanical properties

The mechanical properties of our composite materials were evaluated by the tensile test, using test pieces of cylindrical form, at room temperature and high temperature (200 and 260 °C). The test pieces had a gauge length of 30 mm with 5 mm diameter. The 0.2% offset yield strength σ_E (MPa), the ultimate tensile strength σ_R (MPa), the uniform and fracture elongation, A_U and A_R (%), were measured respectively on engineering stress–strain diagrams. We used an extensometer with 25 mm gauge length for tensile tests at room

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TABLE I Composition of prepared Al–Ni–Li alloys

Alloy No.	Composition (wt %)			
	Ni	Li	C	Al
1	6.0	0.44	0.09	Balance
2	5.9	0.76	0.07	Balance
3	6.1	1.36	0.06	Balance
4	5.9	1.67	0.07	Balance
5	6.1	2.35	–	Balance

temperature and 200 °C in order to measure the yield strength with a higher precision. The tensile velocity was small, 0.02 cm min^{-1}, during a first stage of deformation and then it was increased after the yield stress to 0.1 cm min^{-1} until fracture of the test pieces.

The Vickers microhardness was measured at room temperature on polished surfaces with a weight of 100 g. We observed that the microhardness is approximately the same on both transverse and longitudinal sections of the cylindrical test pieces.

The microstructure of the alloys was examined by optical and electron microscopic observations, and X-ray diffraction using a Philips Norelco diffractometer with automatic recording. The cubic lattice parameter of the δ'-Al_3Li phase is almost the same as that of the aluminium matrix, but the $L1_2$ type of ordered structure gives superlattice reflections on its diffraction diagram, by which the presence of δ'-Al_3Li can be identified.

3. Experimental results

In the first part of this work, we examined the mechanical properties of the Al–Ni–Li system on the five kinds of different composition, as shown in Table I. The age-hardening effect was then examined on three alloys: Al–2.36% Li, Al–6.1% Ni–2.35% Li and Al–5.9% Ni–1.67% Li.

3.1. Mechanical properties of the Al–Ni–Li system

Fig. 1a, b and c summarize the results of tensile tests carried out on the five kinds of alloys in Table I at room temperature, 200 and 260 °C, respectively, as a function of lithium content. The experimental data for zero lithium content are those obtained for the Al–5.7% Ni alloy reported previously [3]. The results of microhardness measurements at room temperature are also shown in Fig. 1a.

At room temperature, lithium addition hardens the composite materials and this effect becomes more remarkable at high lithium content. We obtained an ultimate tensile strength σ_R of about 400 MPa for lithium content 0.5 to 1.7 wt % with the fracture elongation being fairly large, $A_R = 4$ to 7%. At high lithium content, 2.35%, σ_R nearly attains 500 MPa but the alloys become less ductile. At high temperature, 200 and 260 °C, the mechanical resistance increases at relatively low lithium content, less than 1.5 wt %, and fracture elongation decreases, while the strength decreases and the elongation increases for higher lithium content.

3.2. Age-hardening of Al–2.36% Li

The age-hardening of lithium-containing aluminium-base alloys has been investigated by many researchers [5–14]. In the case of the binary Al–Li system, it is now recognized that the formation of very small particles of the metastable δ'-Al_3Li phase is responsible for this phenomenon.

We first examined the effect of ageing on the Vickers microhardness of Al–2.36% Li. This alloy was prepared by using a graphite crucible and consequently it contains a small amount of carbon, 0.03 wt %. The cast ingot was rolled from 28 to 5 mm thickness at about 400 °C. The Vickers microhardness of this rolled plate was 58. X-ray diffraction analysis and microscopical observation showed that this alloy contains the δ-AlLi phase, distributed as small particles with irregular form. After solution treatment in a lead bath at 530 °C for 3 h, followed by quenching into brine at − 3 °C, the amount of δ-AlLi phase decreased considerably but still persisted as a small content. Until this stage the presence of the δ'-Al_3Li phase was not detected by X-ray diffraction.

The alloy was then heated at 200 °C, with the ageing time being variable from 2 to 100 h, and cooled in air. By this heat-treatment, the δ'-Al_3Li phase clearly becomes visible on the X-ray diffraction diagram by the appearance of its superlattice reflections. Curve 1 in Fig. 2 shows the results for the variation of Vickers microhardness as a function of ageing time. The hardness increases rapidly from 51 by a short time of heating at 200 °C, and it attains the maximum value of about 115 after 5 to 10 h and then decreases slowly.

3.3. Age-hardening of Al–6.1% Ni–2.35% Li composite materials

We examined the effect of ageing on the mechanical properties of Al–6.1% Ni–2.35% Li (alloy No. 5 in Table I). Tensile test specimens, similar to those used previously, were heated in a lead bath at 530 °C for 1 h and quenched into cooled brine at − 3 °C. They were then heated at 200 °C with an ageing time variable from 3 to 20 h, and cooled in air. In spite of the same order of lithium content in this alloy as in the previous Al–2.36% Li alloy, the presence of δ-AlLi was not detected by X-ray diffraction nor by microscopic observation. This suggests that a fairly large amount of lithium is dissolved in the Al_3Ni phase.

The Vickers microhardness of this composite alloy was 125 in its initial cast state, but it increased up to about 145 after isostatic extrusion with $R = 4.8$. Fig. 3 summarizes the results on Vickers microhardness H_V, the ultimate tensile strength σ_R, the 0.2% offset yield strength σ_E, and the fracture and uniform elongation A_R and A_U, respectively, as a function of ageing time. These composite materials show in their initial state after 1 h heating at 530 °C and quenching into cooled brine the following results: $H_V = 96$, $\sigma_E = 150$ MPa, $\delta_R = 309$ MPa, $A_U = 9.8\%$ and $A_R = 12.3\%$. The

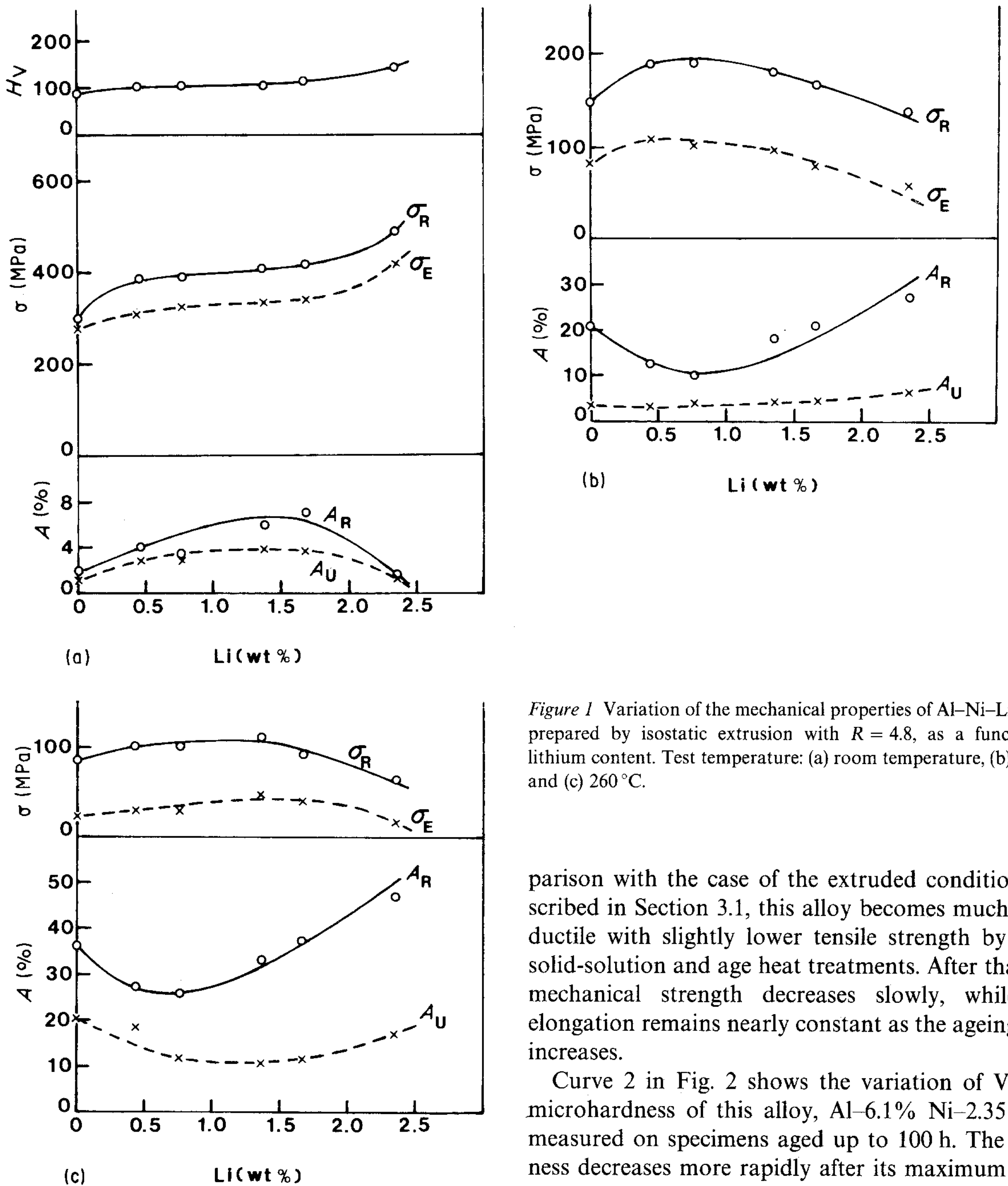


Figure 1 Variation of the mechanical properties of Al–Ni–Li alloys, prepared by isostatic extrusion with $R = 4.8$, as a function of lithium content. Test temperature: (a) room temperature, (b) 200 °C and (c) 260 °C.

mechanical strength is increased remarkably by a short time of ageing at 200 °C and it attains the maximum value after ageing of about 3 h, while the elongation decreases: $H_V = 131$, $\sigma_E = 318$ MPa, $\sigma_R = 447$ MPa, $A_U = 6.0\%$ and $A_R = 6.9\%$. In comparison with the case of the extruded condition, described in Section 3.1, this alloy becomes much more ductile with slightly lower tensile strength by these solid-solution and age heat treatments. After that, the mechanical strength decreases slowly, while the elongation remains nearly constant as the ageing time increases.

Curve 2 in Fig. 2 shows the variation of Vickers microhardness of this alloy, Al–6.1% Ni–2.35% Li, measured on specimens aged up to 100 h. The hardness decreases more rapidly after its maximum value than the previous Al–2.36% Li alloy for a long time of ageing.

The experimental results for microhardness on the Al–5.9% Ni–1.67% Li, (alloy No. 4 in Table I) is also shown by curve 3 in Fig. 2. The age-hardening effect for this lower lithium-content alloy is small. These differences depending on the lithium content will be

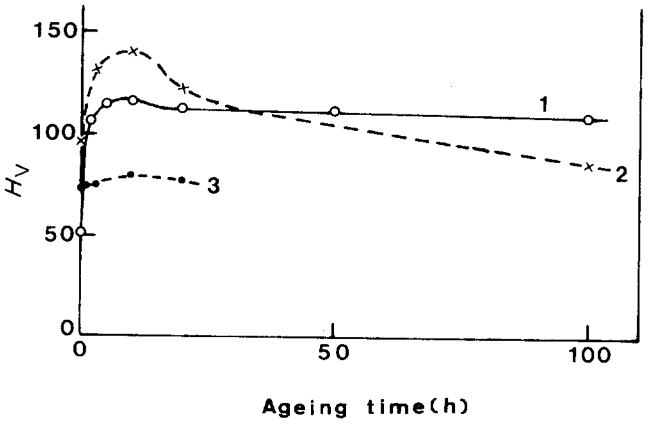


Figure 2 Variation of Vickers microhardness as a function of ageing time at 200 °C of alloys prepared by solution treatment at 530 °C and water-quenching: (1) Al–2.36 % Li, (2) Al–6.1% Ni–2.35% Li, (3) Al–5.9% Ni–1.67% Li.

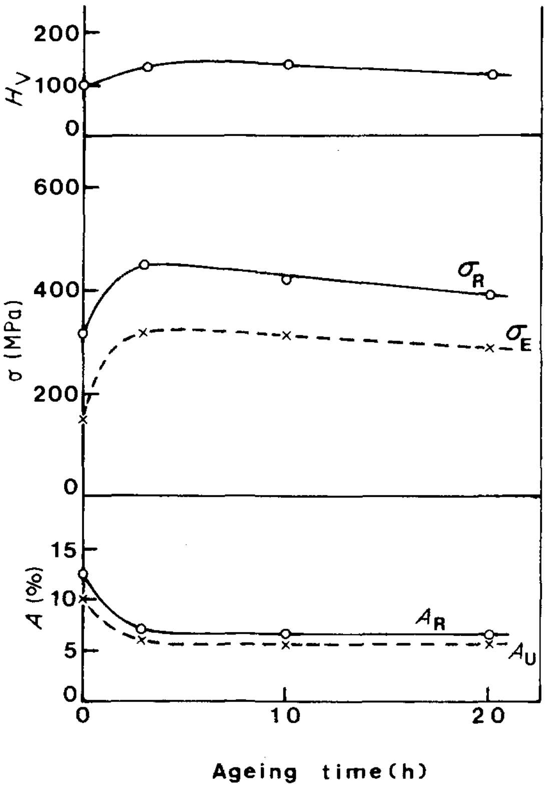


Figure 3 Mechanical properties of Al–6.1% Ni–2.35% Li composite alloy, heated for 1 h at 530 °C and then water-quenched, as a function of ageing time at 200 °C.

discussed hereafter by referring to the microstructure observation by transmission electron microscopy.

3.4. Microstructure observation by transmission electron microscopy

The specimens for transmission electron microscopy (TEM) were prepared by mechanical polishing and electrolytic polishing, using a solution whose composition was 500 cm^3 ethanol, 88 cm^3 70% perchloric acid, 12 cm^3 water and several drops of ethylene glycol. We first examined the Al–2.36% Li alloy described in Section 3.2, and then the alloys of the Al–Ni–Li system in Table I.

In the case of the Al–2.36% Li alloy, electron diffraction showed the presence of the σ'-Al_3Li phase in the cast and rolled plates. The superlattice reflections are very weak and diffuse, but they appear more clearly after solution treatment of 3 h at 530 °C, followed by water-quenching, even though the σ'-Al_3Li phase was not detected by X-ray diffraction. The crystallographic orientation is the same as that of the fcc aluminium matrix. Fig. 4a and b are the electron diffraction diagram of the hot-rolled Al–2.36% Li plate and that of the same alloy after solution treatment, respectively.

Electron microscopic images show that the σ'-Al_3Li phase consists of very small spherical particles, as reported by others [10–16]. The dimensions and the amount of these particles increase by ageing at 200 °C, and superlattice diffraction spots such as (1 0 0), (1 1 0), (2 1 0), . . . become more intense and sharper as the ageing time increases. Fig. 5 shows the growth of δ'-Al_3Li particles whose diameter is about 10 to 20 nm after ageing of 10 h, and 40 to 60 nm after 100 h.

In the case of the Al–6.1% Ni–2.35% Li (alloy No. 5 in Table I) the superlattice reflections of the δ'-Al_3Li phase are observed by electron diffraction with very weak intensity in both cast and extruded alloys, but they become more intense after 1 h ageing at 530 °C and water-quenching, as shown in Fig. 6. By this heat-treatment, the plastically deformed matrix by isostatic extrusion is recrystallized and the spheroidization of fragmented fibres of Al_3Ni phase occurs. This structural change leads to an increase of ductility and decrease of strength as described in Section 3.3.

The spherical precipitates of δ'-Al_3Li are still very small in Fig. 6a and difficult to identify. However, their dimensions and quantity increase by ageing at 200 °C. The diameter of spherical particles of the δ'-Al_3Li phase becomes about 10 to 20 nm after ageing for 3 h, and 10 to 30 nm after ageing for 20 h. Fig. 7 shows the microstructure of this alloy after ageing for 20 h at 200 °C. There appears a bright region of reduced precipitate density of δ'-Al_3Li phase on either side of the grain boundary. The electron diffraction diagram corresponding to the microstructure is almost the same as Fig. 5c, the superlattice reflections being fairly strong.

Contrary to our expectation, the size of δ'-Al_3Li precipitates does not further increase but becomes smaller during a prolonged ageing of 100 h; their diameter is nearly 4 to 5 nm. The δ'-Al_3Li particles in Fig. 7 change to finer ones and their amount also decreases, as shown in Fig. 8.

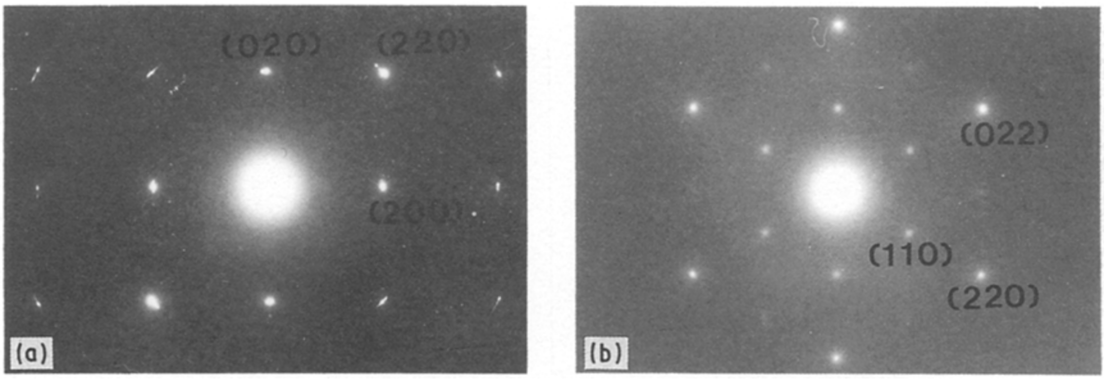
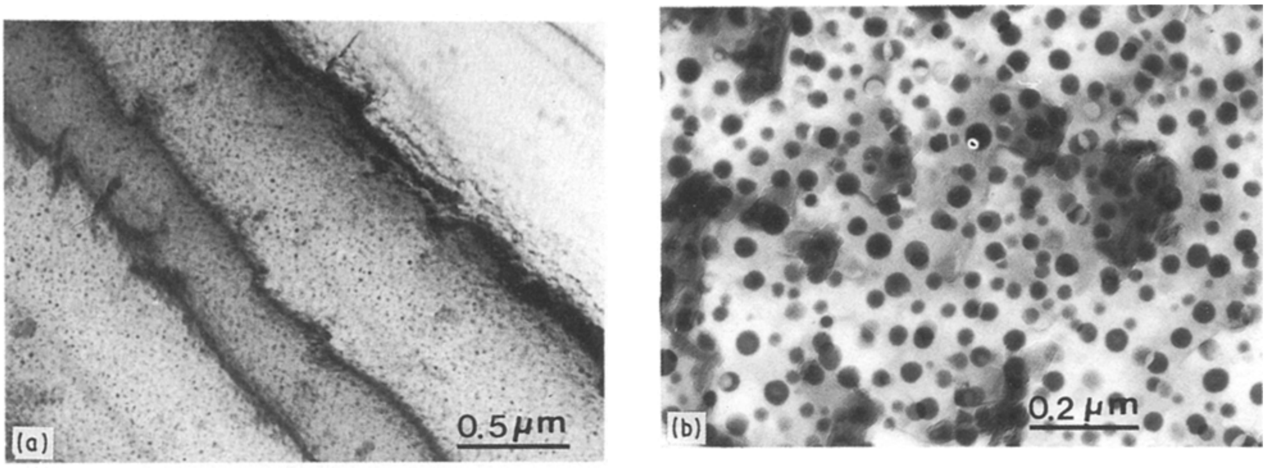


Figure 4 Electron diffraction diagrams of Al–2.36% Li: (a) rolled plate and (b) after solution treatment at 530 °C for 3 h, followed by water-quenching.



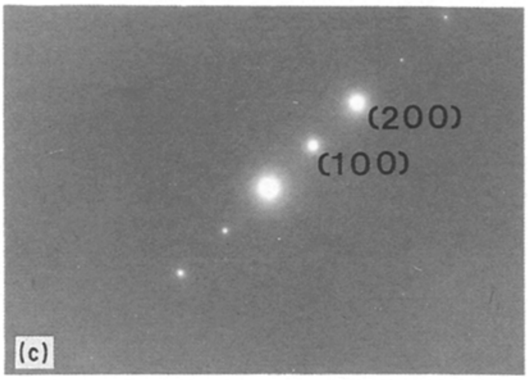


Figure 5 TEM micrographs and electron diffraction diagram of Al–2.36% Li alloy, heated for 3 h at 530 °C and water-quenched: (a) after 10 h ageing, (b) and (c) after 100 h ageing at 200 °C.

We also examined the other composite materials, Nos 1 to 4 in Table I, prepared by isostatic extrusion with $R = 4.8$, but we never observed the presence of the δ'-Al_3Li phase in these lower lithium-containing alloys. This result shows that the formation of δ'-Al_3Li phase is possible only for the higher lithium-containing materials with concentrations above approximately 1.7 wt %.

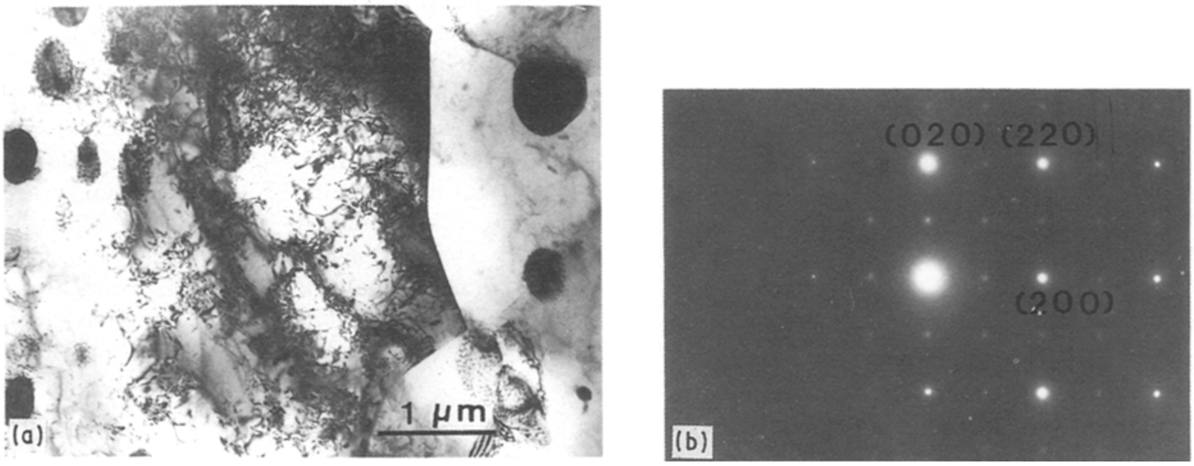


Figure 6 Al–6.1% Ni–2.35% Li alloy, extruded with $R = 4.8$ and then heated for 1 h at 530 °C and water-quenched: (a) TEM micrograph, (b) electron diffraction diagram.

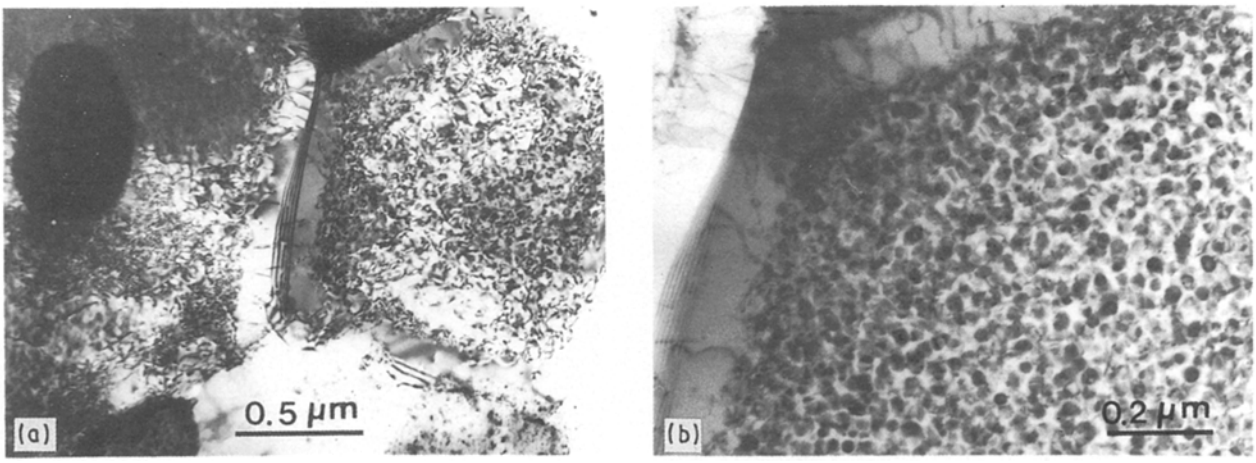


Figure 7 TEM micrographs of Al–6.1% Ni–2.35% Li alloy, prepared by isostatic extrusion with $R = 4.8$, and heated for 1 h at 530 °C and water-quenched, then aged for 20 h at 200 °C. (a) and (b) are obtained at the same place but with different magnification.

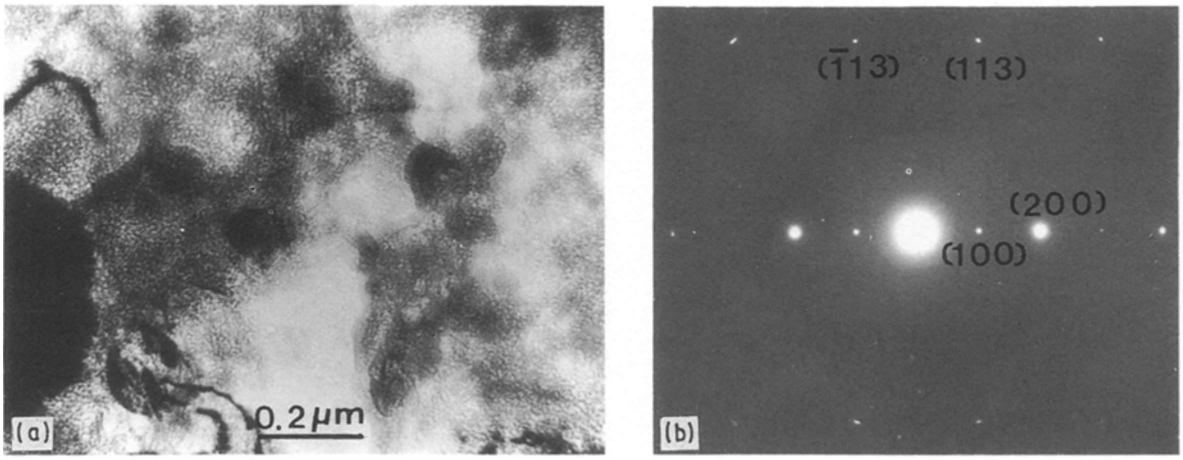


Figure 8 Al–6.1% Ni–2.35% Li alloy, extruded with $R = 4.8$ and heated for 1 h at 530 °C and water-quenched, then aged for 100 h at 200 °C: (a) TEM micrograph, (b) electron diffraction diagram.

4. Concluding remarks

Our experimental results show that the addition of a small amount of lithium considerably improves the mechanical properties of the metallic composite materials, based on the eutectic Al–5.7% Ni alloy and developed in our previous work [1–4].

The ultimate tensile strength at room temperature increases from 300 MPa to about 400 MPa by lithium additions of 0.5 to 1.7 wt %, and at the same time the fracture elongation also increases from about 2% to 4 to 6%. This increase of mechanical strength is almost equivalent to the effect of 2 to 3 wt % copper addition [3] or 1 to 2 wt % magnesium addition [4]. The mechanical strength further increases with lithium additions of more than 1.7 wt %, and we obtained about 500 MPa with the addition of 2.35 wt % Li, which is comparable with the effect of 3 wt % Mn addition [3].

At higher temperatures, 200 and 260 °C, the relatively high mechanical strength is still conserved at about 200 and 100 MPa, respectively. The fracture elongation decreases for low lithium content but it increases remarkably for higher lithium content, as shown in Fig. 1.

The microstructure observation of these materials by TEM shows that the strengthening by lithium addition is due to solid-solution hardening for a lithium content lower than about 1.7 wt %, while precipitation hardening by the δ'-Al_3Li phase is added to this effect for materials containing lithium with more than about 1.7 wt %. This difference due to the lithium content is in accordance with the phase diagram of the metastable δ'-Al_3Li phase in dilute Al–Li alloy [10–12, 17], i.e. the formation of the δ'-Al_3Li phase occurs due to the aluminium matrix dissolving lithium with a higher content than about 1.7 wt % by ageing at 200 °C.

The δ'-Al_3Li phase, a cubic $L1_2$ type of ordered structure, precipitates as small spherical particles in the aluminium matrix. Its lattice parameter is almost the same as that of aluminium and the crystallographic orientations of these two are identical, and this leads to the strengthening of the aluminium matrix by the precipitation of δ'-Al_3Li crystals. Consequently, the mechanical properties of the Al–Ni–Li alloys depend on the behaviour of the lithium-containing aluminium matrix and that of the δ'-Al_3Li precipitates.

In the case of Al–2.36% Li alloy, water-quenched from 530 °C and then aged at 200 °C, the particle size of δ'-Al_3Li precipitates increases as a function of the ageing time and it reaches 40 to 60 nm diameter after 100 h. On the other hand, the δ'-Al_3Li precipitates formed in the Al–6.1% Ni–2.35% Li alloy reach their maximum size of about 10 to 30 nm after ageing periods of 20 h at 200 °C, but after that their size and amount decrease during longer ageing periods. These observations suggest that the lithium content in the aluminium matrix of Al–6.1% Ni–2.35% Li alloy is less than that in the Al–2.36% Li alloy although the total content of lithium in each of these alloys is almost the same. It may be possible that one part of lithium, initially contained in the δ'-Al_3Li crystals in Al–6.1% Ni–2.35% Li, further diffuses into the Al_3Ni phase during a long time of ageing at 200 °C.

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