

The mechanical properties and precipitation behavior of an Al-Cu-Li-(In,Be) alloy

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The effect of In and Be on the mechanical properties and precipitation behavior of an Al-Cu-Li alloy, which has applications in aerospace and aircraft, has been studied using hardness and tensile tests, differential scanning calorimetry (DSC), and transmission electron microscopy (TEM). The formation of GP zones and δ' phases was retarded, but the T_1 and θ' phases formation were promoted by the addition of In to Al-Cu-Li alloy. However the formation of T_1 and θ' phases, as well as the formation of GP zones and δ' phases, were accelerated by the combined addition of In and Be to Al-Cu-Li alloy. The additional precipitation of T_1 phases which was formed at 160°C for a long aging time, was a main reason for the secondary peaks of hardness and tensile strength in Al-Cu-Li alloys added Be and In, or In. The Al-Cu-Li-In-Be alloy was suggested as a material of good combination of tensile strength, elongation and toughness. © 2002 Kluwer Academic Publishers

1. Introduction

Al-Li-Cu alloys, one of the important Al-Li system alloys, show a characteristics of high tensile strength and toughness, and low density. The tensile strength of these alloys can be enhanced by cold working and aging treatments [1]. However, the use of cold working is precluded in some structural parts to achieve a full potential tensile strength, and in these cases it is desirable to make an alloy capable of achieving maximum tensile strength through the aging treatment alone.

Starke and Blackburn [2] showed that In addition to Al-Cu-Li alloy increases tensile strength, which is associated with an increase in the number of T_1 (Al_2CuLi) and θ' (Al_2Cu) particles, and their distribution. The formation of the precursor phase or δ' (Al_3Li) is suppressed, but G.P. zone and θ' formation is accelerated by the In addition to 2090 alloy (Al-Cu-Li) [3]. Luo and Youdelis [4] have shown that Be increases the density of the precipitation of metastable phases S' (Al_2CuMg), δ' , and GP zones in 8090 alloy (Al-Li-Cu-Mg) [4]. It has also been reported that the precipitation of the θ' and θ'' is enhanced by the addition of Be to 2090 alloy [5]. However the effects of a combined addition of In and Be to the Al-Li alloy system on the precipitation behavior and tensile strength has not been reported. In this study, the effects of In and Be addition to Al-Cu-Li alloy on the precipitation behaviors and mechanical properties were investigated using hardness and tensile tests, DSC, and TEM.

2. Experimental procedures

The Al-Cu-Li-(In,Be) alloys, which are specimens studied in the present work, were produced by the high

frequency (10 kHz) vacuum induction furnace under Ar atmosphere. The temperatures of melt and pouring were 870 and 770°C, respectively. The billets of 51mm diameter were homogenized for 24 h at 540°C under Ar atmosphere and then extruded the rods to 9.8 mm diameter. The temperature of billets and container was 420 and 340°C, respectively. Composition of the Al-Cu-Li-(In, Be) alloys studied are listed in Table I. The rods were heat treated for 1 h at 550°C, quenched in cold water, and aged at 160°C for various time intervals to analyze the precipitation behavior using a Rockwell hardness (Trade Sato Mark-3R : HRF) technique. Specimens (ASTM E-8, subsize) were prepared for tensile tests, and the tensile strengths were measured using the Shimazu tensile tester (DSS-10TS). TEM foils were double-jet electrothinned in a 30% HNO_3 -70%methanol electrolyte at -30°C, and the oxide was removed from the thin film using the Bollmann method. DSC measurement was performed for each sample of 30 mg in weight, using a DuPont-2000 thermal analyzer, with a constant heating rate of 10°C/min over a wide range of temperature 30 to 500°C. After the completion of DSC scan, the raw heat-flow data were converted to specific heat capacity using a DuPont program.

3. Results and discussion

3.1. DSC curves of the solution-heat-treated alloys

The precipitation sequence of Al-Cu-Li alloy with the ratio of Cu/Li is 1–2.5 can be represented by [6]:

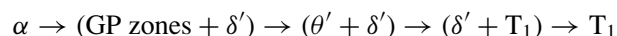


TABLE I Nominal chemical composition of Al-Cu-Li-(In,Be) alloys (wt%)

Alloys	Elements					Al
	Cu	Li	In	Be	Zr	
Alloy A	2.50	1.84	–	–	0.14	Bal.
Alloy B	2.53	1.85	0.182	–	0.14	Bal.
Alloy C	2.52	1.85	0.188	0.0035	0.14	Bal.

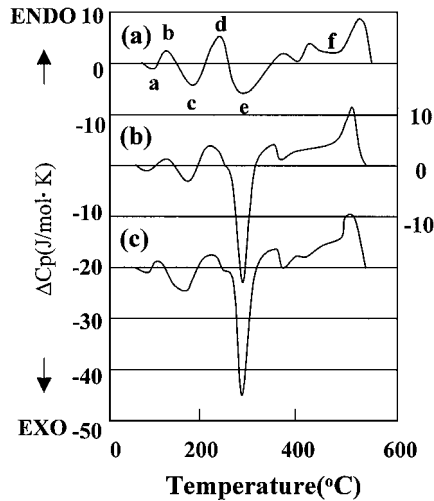


Figure 1 DSC curves of (a) A, (b) B and (c) C alloys as-quenched from 550°C.

Fig. 1 shows the DSC curves for the quenched specimens for alloys A, B and C. Three endothermic peaks, b, d and f, and three exothermic peaks, a, c, and e were observed. The existence of a precursor phase (peak “b”) [7, 8] can be seen for all specimens clearly in Fig. 1. According to Woo [9], Lee [3] and Eun [5], the peaks, a, c, and e are identified as the exothermic reactions due to the formation of GP zones, δ' and θ' (+ T_1) phases respectively, and the endothermic reactions, b, d and f are due to the dissolution of GP zones, δ' and θ' (+ T_1) phases respectively. The formation and dissolution of the GP zones have been shifted to a higher temperature compared to that of GP zones for Al-Cu base alloy [10].

Table II shows the DSC characteristics of quenched alloys. In alloy B, the “a” peak shifted to a higher temperature than the alloy A, and the exothermic energy, H (J/g), decreased with In addition to Al-Cu-Li alloy. This indicates that the formation of GP zones is suppressed by the addition of In to Al-Cu-Li alloy. This can

be explained by the fact the strain formed by quenching decreases by the addition of In, Cd and Sn to Al-Cu-Li alloy, which have the larger lattice constants than Al, and the formation of Cu cluster at the surrounding of the additional elements such as In and Cd reduces the strain energy. Therefore the number of Cu, which is needed to make GP zones [11], is reduced by existence of these elements. According to Silcock and Heal [12], the formation of GP zones is suppressed by the addition of these elements due to a segregation at the dislocations. These segregated elements interfere with the Cu diffusion down dislocations [12]. But the dislocations for the Al-Cu-Li-In alloy was not observed in the study of Starke and Blackburn [2] using a high-resolution electron microscopy. Therefore the dislocations seemed not an important factor for the control of the diffusion of Cu down dislocations. Another reason, which the formation of GP zones is retarded by the addition of In to Al-Cu-Li alloy, is due to the formation of In-Cu-vacancy clusters [13], but the formation of θ' phase is accelerated by these clusters. Quenched-in vacancies become to form the cluster with In because In-vacancy binding energy (0.39 eV) [14] is larger than that of Cu-vacancy [15]. The formation of GP zones is retarded because the density of free vacancies, a main factor of Cu diffusion, is reduced.

The enthalpy due to the precipitation of θ' and T_1 (peak “e”) increased with the addition of In or In and Be to the Al-Cu-Li alloy. This phenomenon means that the amount of θ' and T_1 precipitates is increased by the addition of In or In and Be to the Al-Cu-Li alloy. And the peaks “e” of the alloys B and C were found to be sharper compared with that of the Al-Cu-Li alloy, this means that the temperature range of precipitation becomes narrow by the addition of In or In and Be to the Al-Cu-Li alloy. But the effect of In to alloy A on the formation of GP zones is lessened by the combined addition Be and In to the Al-Cu-Li alloy. The effect of Be or In and Be to alloy A on the formation of δ' phase is similar to that for the formation of GP zones. The precipitation of δ' phases was interfered with addition of In, because of a high binding energy of In-vacancy. And if the Li-In binding energy becomes to be large, the formation of δ' phases will be retarded. If δ' is formed by the continuous ordering or spinodal decomposition [16], In affects the atomic fraction of Li which influences these reactions (the continuous ordering or spinodal decomposition) or changes the free energy (interface and coherent energy)

TABLE II DSC characteristics for the quenched alloys

Alloys	Reactions	a (GP zones: exo.)	c (δ' : exo.)	e (θ' + T_1 : exo.)
Alloy A	Temperature range (°C)	69.64–107.14	152.68–200.89	259.82–333.48
	T_p (°C) ^a	95.97	183.81	291.26
	H (J/g) ^b	0.48	5.94	7.63
Alloy B	Temp range(°C)	85.58–121.74	159.24–204.77	252.99–314.6
	T_p (°C)	104.35	190.58	288.26
	H (J/g)	0.30	3.76	14.39
Alloy C	Temp range(°C)	84.15–109.60	150.78–202.01	251.56–310.49
	T_p (°C)	103.09	184.88	282.54
	H (J/g)	0.23	4.46	14.09

^a T_p represents peaks temperature for the phases transformation.

^b H represents enthalpy for the phases transformation.

needed to these reactions. Another reason is that the formation of δ' phase is interfered by the addition of In to the Al-Cu-Li alloy.

The Be added to 8090 (Al-Li-Cu-Mg) alloy accelerates the δ' , S' phases or GPB zones, and increases the density of these phases [4]. The effect of In on the precipitation of δ' phases and GP zones of alloy A is suppressed by the addition of Be. This is because the atomic radius of Be is smaller, but that of In is larger than that of Al.

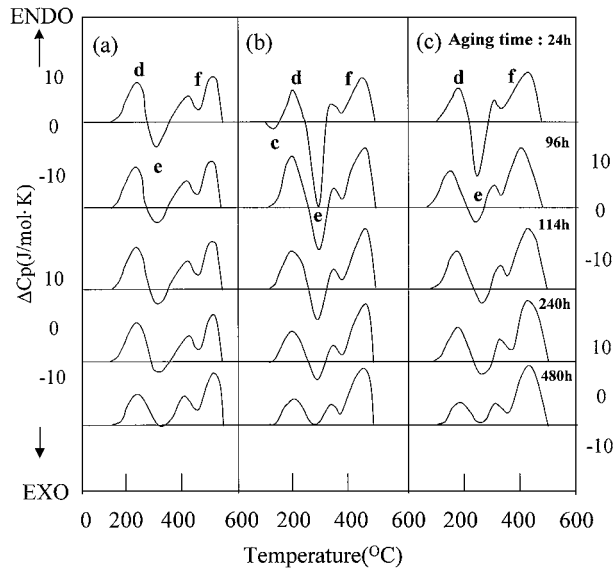


Figure 2 DSC curves of (a) A, (b) B and (c) C alloys aged at 160°C for 24–480 h.

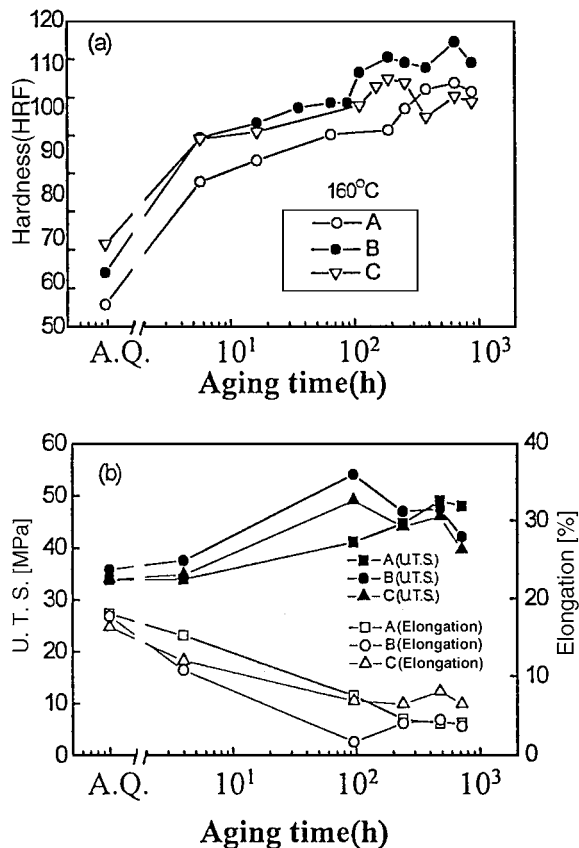


Figure 3 Variation of hardness (a), tensile strength and elongation (b) with the aging time of A, B and C alloys aged at 160°C.

Peak “e” shifted to the lower temperature by the addition of In or In and Be to alloy A. These results indicate that the precipitation of θ' and T_1 phases are accelerated by the addition of In, and by the combined addition of In and Be to Al-Cu-Li alloy (Table II). Be added to the alloy B promotes the effect of In on the precipitation of θ' and T_1 phases. The formation of δ' and T_1 phases is accelerated by the combined addition of Be and In to alloy A. This is because In(Be)-Cu-vacancy cluster, formed during quenching, becomes the preferential nucleation sites of δ' and T_1 phases. Another reason is that these elements affect the nucleation sites such as dislocation loop and In-rich phases, and affect the kinetics of nucleation of these phases. Silcock [11] suggested that the critical nucleation sizes of θ' or T_1 phases decrease due to a decrease in the interfacial energy between the matrix and precipitation by segregation at the interface.

3.2. DSC curves of alloys aged at 160°C

Fig. 2 shows DSC curves for A, B and C alloys aged at 160°C. Table III shows the changes of ΔH for the δ' and

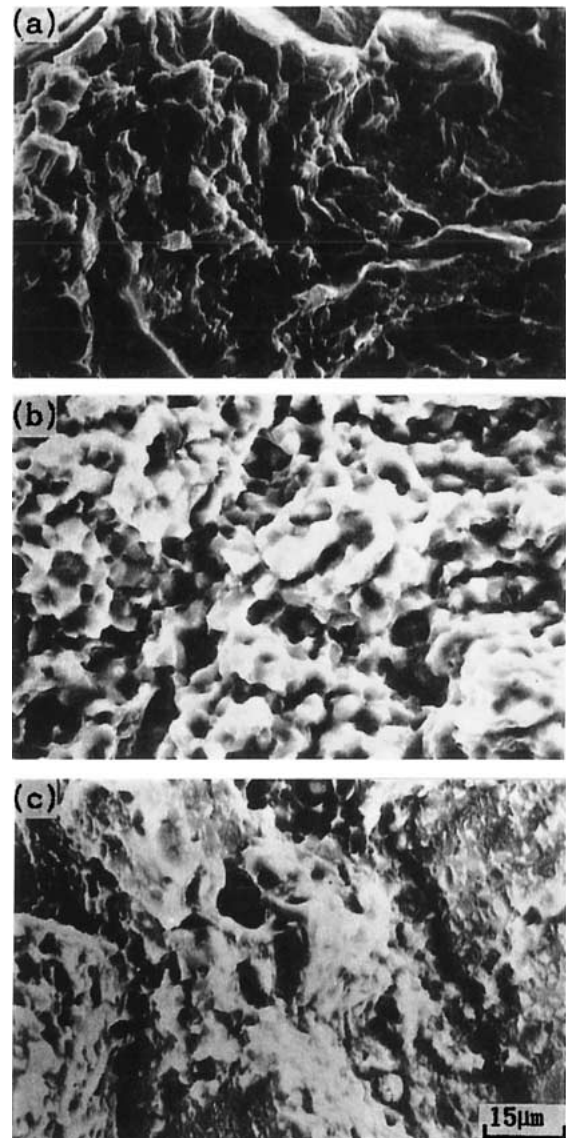


Figure 4 SEM fractographs of the tensile specimens (a) A, (b) B and (c) C aged at 160°C for 96 h.

$\theta' + T_1$ phases of the aged alloys. At the aging treatment for 24 h at 160°C, the precipitation of δ' phases was not finished in the alloys B(b), but finished in the alloys A(a) and C(c) (see Fig. 2). This results represent that In elements in Al-Cu-Li alloy retard the formation of δ' phase due to an acceleration of the precipitation of θ' and T_1 phases [2, 3].

From the Table III, the enthalpy change (ΔH) for the dissolution of δ' phases in all alloys increased with aging time until 48 h at 160°C, but that of δ' phases decreased after 72 h aging time at 160°C. And the enthalpy change (ΔH) for the formation of θ' and T_1 phases in all alloys decreased with aging time at 160°C. But the rate of decrease in ΔH for the formation of θ' and T_1 phases is higher for the alloys B and C than the alloy A. This is responsible for the higher rate of precipitation of θ' and T_1 phases for the alloys B and C. This represents the precipitation rate of θ' and T_1 phases of alloys B and C is faster than those of alloy A. In the alloy C, the

TABLE III Changes of ΔH for the δ' and $\theta' (+T_1)$ phases of alloys A, B and C aged at 160°C

Aging time	Alloy A		Alloy B		Alloy C	
	Phase ΔH (J)		Phase ΔH (J)		Phase ΔH (J)	
	δ' (endo.)	$\theta'(+T_1)$ (exo.)	δ' (endo.)	$\theta'(+T_1)$ (exo.)	δ' (endo.)	$\theta'(+T_1)$ (exo.)
A.Q.	3.37	7.63	3.18	14.39	1.91	14.09
24h	14.98	6.98	7.76	13.48	6.03	11.19
48h	16.98	6.28	15.48	10.85	14.15	10.24
96h	15.04	4.22	14.54	6.45	13.05	5.11
144h	14.23	3.84	12.97	6.10	10.41	4.59
240h	12.47	2.58	12.29	2.82	8.76	1.62
480h	10.21	0.12	9.53	0	5.82	0

precipitation rate of δ' , θ' and T_1 phases is accelerated rather than the In added alloy B. This is because the effect of In addition is decreased by the addition of Be to the Al-Cu-Li alloy.

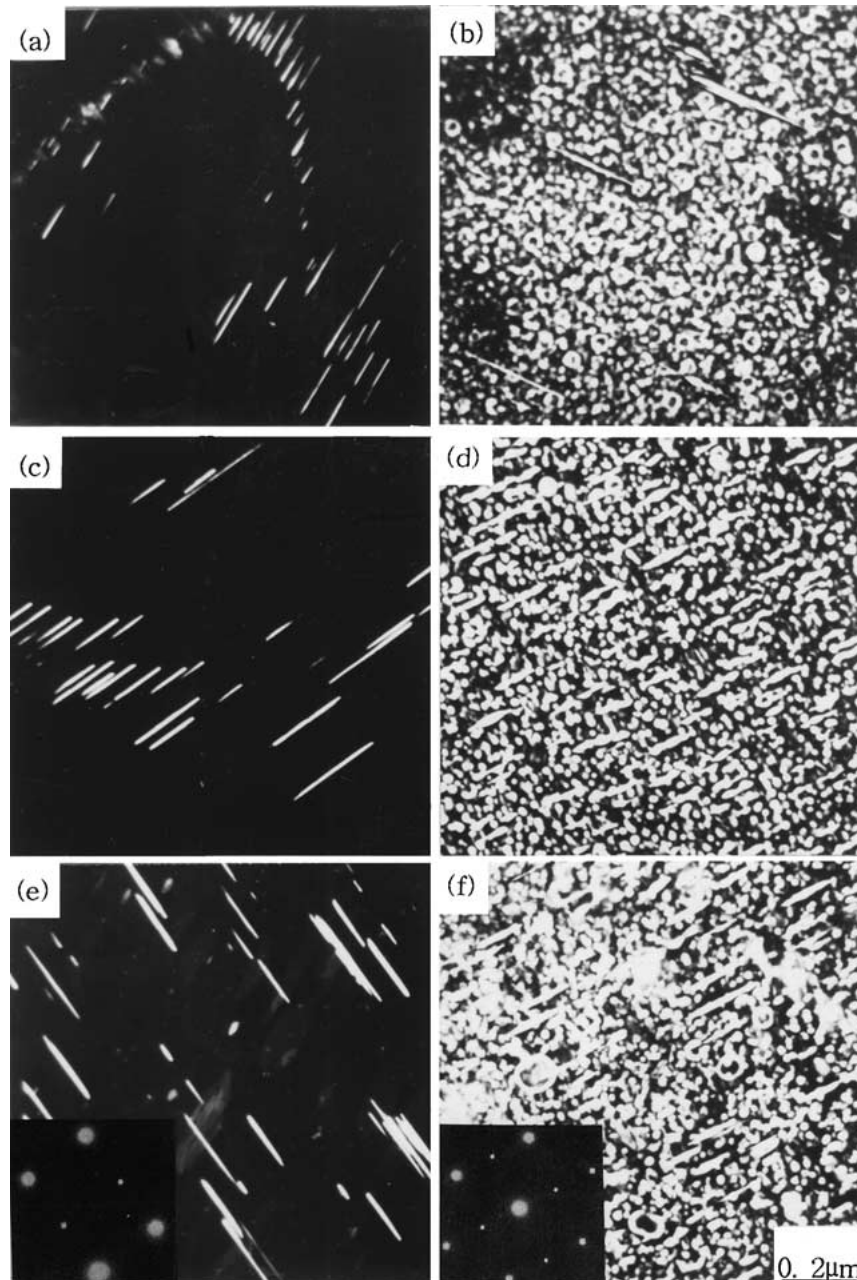


Figure 5 Transmission electron micrographs of A, B and C alloys aged at 160°C for 96 h: (a), (c), (e) DFI of T_1 phase at [112] zone axis of A, B and C alloys respectively, and (b), (d), (f) DFI of δ' and θ' phases at [110] zone axis of the A, B and C alloys respectively.

3.3. Mechanical properties of alloys aged at 160°C

Fig. 3 shows the hardness (a) and tensile strength (b) curves for A, B and C alloys aged at 160°C. The aging was accelerated by the addition of In and Be to the Al-Li-Cu alloy, and two peaks of hardness and tensile strength were observed at around 120 and 480 h for alloys B and C, while only one peak appeared at 480 h for alloy A. The hardness as well as tensile strength of alloys B and C were enhanced by the combined addition of In and Be to Al-Cu-Li alloy (alloy A). This enhancement might be explained by both the accelerated precipitation of θ' and T_1 phases and solution hardening effects by In and Be in the matrix.

The first peak of hardness and tensile strength appeared to the alloys B and C due to the precipitation of δ' , θ' and T_1 phases. But the second peak of hardness and tensile strength occurred due to the additional precipitation of T_1 phase. This is because δ' phase in alloys B and C might be easier to be resolved into the matrix rather than that of alloy A, thus Li content in the matrix increases, and helps the re-precipitation of T_1 phase (see Fig. 2 and Fig. 6). T_1 precipitation might depend on the excess Li content in the matrix supported by the dissolution of δ' phase. Therefore, the hardness and tensile strength of alloys B and C increase again because of the increase of T_1 phases content even though the ripening of δ' phase during aging for a long time. In this figure, the saddle point of hardness and tensile strength of alloy C was lower than that of alloy B, probably due to the low density and coarsening of δ' and θ' phases even though the high density of second

TABLE IV Mechanical properties of peak-aged alloys at 160°C

Alloys	Mech. Properties				
	UTS (MPa)	YS (MPa)	NTS (MPa)	NYR	Elongation (%)
Alloy A	379.9	403.9	424.1	1.05	7.5
Alloy B	530.4	507.9	521.9	1.03	1.6
Alloy C	480.7	451.3	505.5	1.12	6.8

T_1 phases. According to the Chung *et al.* [18], δ' phases grew rapidly by the addition of Be to Al-Li alloy, this is because Be makes the interfacial energy to be lower between matrix and δ' phase due to a segregation of Be at δ' /matrix interface or transformation of the structure of δ' phase by dissolution into the matrix. Especially, hardness and tensile strength decreased prominently by the decrease of θ' phase content (see Fig. 6d). This is main reason that the alloy C shows the lower second peak of hardness and tensile strength than any other alloy.

Table IV shows the tensile strength, notch tensile strength (NTS), and toughness. The NYR (notch tensile strength/yield strength ratio) value, which is able to use as a measure of the fracture toughness, increased with the combined addition of In and Be to the Al-Li-Cu alloy. This is because lots of T_1 and θ' phases suppress the planar slip.

Fig. 4 shows scanning electron microscope (SEM) fractographs of tensile specimens aged at 160°C for 96 h. The fracture surface of alloy B(b) shows a subgrain fracture. But the alloys A(a) and C(c) show mixture morphology of intergranular and transgranular.

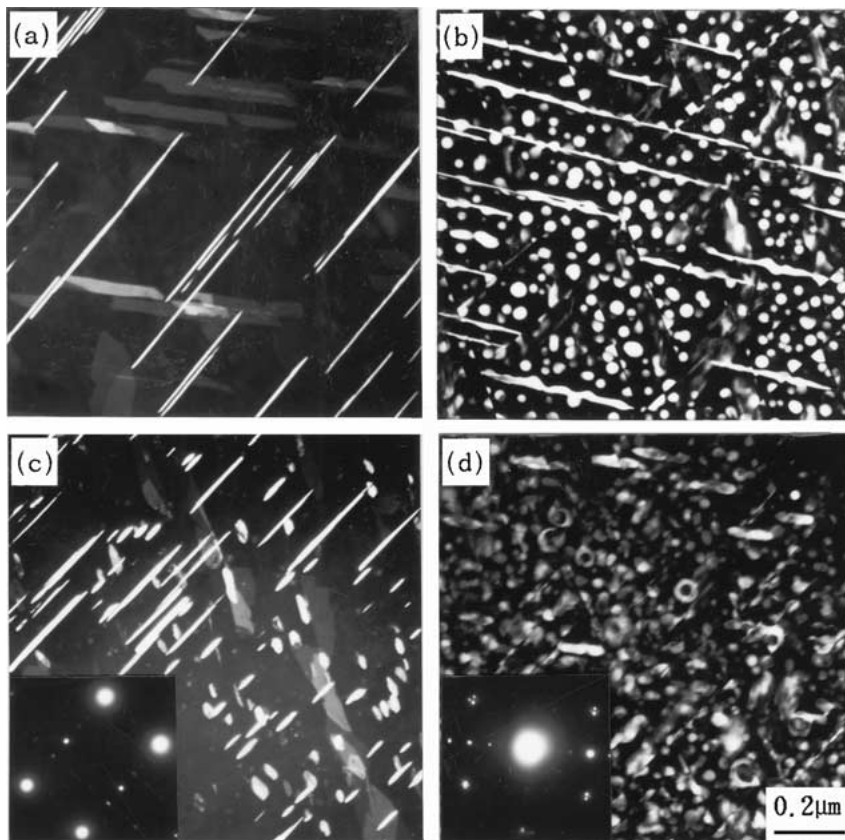


Figure 6 Transmission electron micrographs of A and C alloys aged at 160°C for 480 h: (a), (c) DFI of T_1 phase at [112] zone axis of A and C alloys respectively, and (b), (d) DFI of δ' and θ' phases at [110] zone axis of the A and C alloys respectively.

Therefore high tensile strength and elongation are obtained in alloy C due to a homogeneous distribution of lots of T_1 and θ' phases.

3.4. TEM microstructure observation of alloys

Fig. 5 and Fig. 6 show TEM micrographs for the alloys A(a), B(b) and C(c) aged at 160°C for 96 h and 480 h. In Fig. 5, the density of T_1 and θ' phases for the alloys B and C is higher than those of alloy A. The T_1 phases precipitated heterogeneously at the grain boundary for the alloy A, but precipitated homogeneously in the grains for the alloys B and C. The measurement of difference in the density and size of δ' phases among the alloys is found to be difficult due to a fine and high density of δ' phases. Fig. 6a and b show the T_1 phase, θ' ($+\delta'$) phases in alloy A, respectively. In this alloy, the coarse T_1 phases precipitated earlier are shown. In the alloy C, a coarse and fine precipitate of T_1 is found co-existence. The large precipitate of T_1 was nucleated at 160°C within a short aging times and was grown with increasing aging time, but the fine precipitates of T_1 phases were formed at 160°C for a long aging time. θ' phase was decomposed easily by the combined addition of Be and In to Al-Cu-Li alloy as shown in Fig. 6d. From these results, the second hardness peak seemed to occur because of an additional precipitation of T_1 phase.

4. Conclusions

The formation of GP zones and δ' phases were retarded, but the precipitation of θ' and T_1 phases were promoted by the addition of In to the Al-Cu-Li alloy. The effect on the formation of GP zones and δ' phases was diminished by the combined addition of Be and In to the Al-Cu-Li alloy. And the precipitation of θ' and T_1 phases was accelerated by the combined addition of In and Be to the Al-Cu-Li alloy. The additional precipitation of T_1 phase which was formed at 160°C for a long aging time, was a main reason for the secondary peaks of hardness and tensile strength in Al-Cu-Li-In-(Be) alloys. The Al-Cu-Li-In-Be alloy was suggested as a material of

good combination of tensile strength, elongation and toughness.

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