

Effect of heat treatment environment on Li depletion and on mechanical properties in Al–Li alloy sheets

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The loss of lithium during solution heat treatment of various commercial Al–Li alloy sheets as a function of the heat treatment environment is investigated. The thickness of the soft layer formed due to solutionizing at 530 °C for 30 min in laboratory air, argon and salt bath environments is determined by microhardness measurements. Li loss from the surfaces is interpreted with the help of normalized microhardness profiles. Results showed that prior Li-depleted layer does not hinder further loss of Li from the surfaces and the heat treatment environment has relatively little effect on the loss of Li. The loss of tensile strength due to the Li depletion in the peak-aged condition is found to be more pronounced in 1 mm thick sheet than in 1.5 mm or 2 mm thick sheets. Possible reasons for loss of strength in 8090 series alloys after the heat treatment are discussed. Explanations for the observed ductile fracture features in Li-depleted layers is offered.

1. Introduction

Aluminium–lithium alloys have received a lot of attention in recent years for use in weight critical and stiffness critical structures, as they offer the promise of low density, improved specific strength and high stiffness to weight ratio over the presently used commercial Al alloys. One of the important drawbacks in Al–Li alloy systems, however, is that they suffer from solute depletion, as a result of selective oxidation of the reactive additions, such as Li and Mg, in the temperature range typically encountered during solution heat treatment [1]. Li has very high diffusivity through the aluminium alloy matrix at those high temperatures. The thermodynamic driving force for its preferential oxidation at the oxide–matrix interface is also very high [2, 3]. Solute depletion greatly reduces the age hardening capacity in Li-depleted surface layers as the major contribution to the strength of Al–Li alloys is from the formation of δ' (Al_3Li) precipitate on age hardening [4].

There are several reports [2–9] dealing with the elevated temperature oxidation of Al–Li alloys. However, much of the published work involves long exposure times, whereas in practice, heat treatment times for thin sheets are kept to a minimum to reduce Li depletion. Also, very little information is available about the effect of Li depletion on the mechanical properties of these alloys. A knowledge of variation of mechanical properties with the thickness of the Li-depleted layer is particularly important when thin sections are used. More quantitative data is required in order to relate the processing variables, like heat

treatment environment, temperature, etc., to the depth of Li depletion and its effect on degradation of mechanical properties of Al–Li alloys on age hardening. The strength reduction will mainly depend on the sheet thickness. The present study aims at establishing the relationship between the depth of Li depletion and its effect on tensile properties in the peak aged condition for some commercial Al–Li alloy sheets. The effect of various heat treatment atmospheres on the volume of Li depletion was also studied as a part of this programme.

2. Experimental procedure

Three Al–Li alloys; 8090 T6 (Lital A), 8090 T8 (Lital C) and 2090 T3, were used for the present study and were received in the form of sheets from the Aeronautical Development Agency (ADA), Bangalore. The 8090 T6 material was supplied in the form of 2 mm and 1 mm thick sheets and had an unrecrystallized microstructure, while the 8090 T8 material was in the form of 1.5 mm thick sheet and had a partially recrystallized structure. 2090 T3 material was received in the form of 2 mm thick sheet. All the materials were found to possess a soft layer to a certain depth on both the exposed surfaces. The thickness of the soft layer was determined by microhardness measurements. The nominal chemical composition (wt %) of the alloys is given in Table I.

As-rolled Al–Li alloys sheets in the unrecrystallized condition exhibit anisotropy in their mechanical properties with respect to the rolling direction [10], sheet

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TABLE I Chemical composition of various alloys (wt %)

Element	8090 T6	8090 T81	2090 T3
Cu	1.2–1.6	1.10	2.70
Li	2.5	2.32	2.20
Mg	0.8–1.2	0.80	1.10
Fe	0.10	0.09	0.12
Si	0.05	0.04	0.04
Zr	0.10	0.07	0.1
Others	0.15 max.	0.10 max.	0.10 max.
Al	balance	balance	balance

specimens with a gauge length of 12 mm and a width of 3 mm were milled from large sheets such that the tensile axis is always at right angles to the rolling direction. These samples were solutionized in a vertical tube furnace at 530 °C in enclosed air or flowing argon gas environments. In some cases solutionizing was carried out in an electrically heated salt (50–60% NaNO₃ + 40–50% KNO₃) bath furnace. The samples were drop-quenched in room temperature water (25 °C) after solutionizing. Quenched samples were dried and immediately transferred to an oil bath and were aged at 180 °C for 24 h. Simple tension testing was carried out on an Instron 8032 Universal servo-hydraulic testing machine at a strain rate of 10⁻³ sec⁻¹. Three samples were tested at each condition and their average values are reported here. In some samples, the soft layer formed due to solute depletion was removed by mechanical polishing prior to testing for comparison purposes. The fractured specimens were examined in a JEOL 840A scanning electron microscope (SEM) operating in secondary electron mode.

The gauge head portion of the tensile samples were cold mounted and polished in the standard way, as for the metallographic examination, and then microhardness profiles were evaluated across the short transverse section of the specimen using a 50 g load. Microhardness was measured at equal intervals on a line oblique to the rolling plane in order to improve the resolution. This was necessary to avoid having indentations too close to each other. All the hardness measurements were carried out on a Tukon microhardness tester. The hardness measurements were normalized by dividing each hardness value by the bulk hardness value.

3. Results and discussion

3.1. The effect of heat treatment environment on Li depletion

At high temperatures, Li oxidizes preferentially at the free surface of the Al–Li alloy and many oxidation reactions are possible depending on the alloy composition, the temperature of heat treatment and the atmosphere in contact with the free surface [5]. Using Auger electron spectroscopy (AES) and X-ray photoelectron spectroscopy (XPS) techniques, Ahmed [6] has shown that the rate of oxidation in these alloys is controlled by bulk diffusion of Li in the Al alloy matrix. The oxidation products of Al–Li alloys are

permeable to Li, leading to its unhindered loss with time at high temperatures [7]. It has also been shown that Li is lost to the environment at the free surface by sublimation in vacuum and in inert gas environments [7]. In the early stages of oxidation, significant variations in the amount of oxidation has been found between the grain boundary regions and the grain interiors and has been attributed to the greater rates of diffusion in the grain boundaries [7]. Although, lithium depletion from the surface of Al–Li alloys at high temperatures is controlled by the diffusion of Li in the bulk of the material [6], it also depends on the rate at which it is removed from the free surface. Hence, the composition and flow rate of the gaseous environment during the solutionizing heat treatment play an important role in determining the extent of the Li loss from the surfaces.

Fox *et al.* [8] determined the theoretical Mg concentration profiles in these alloys as a function of depth from the surface, assuming the surface to be an infinite sink for Mg, and found excellent agreement with the experimentally measured concentration profile. The theoretical Li profile calculated using similar assumptions correlated well with the normalized microhardness profile. Also, Li loss is very difficult to measure using conventional techniques, since Li is a very light element. It is well established now that the normalized microhardness profiles in the peak-aged condition fit very well with the theoretically calculated Li concentration profiles and hence represent lithium lost to the surroundings during high temperature heat treatment [6, 11]. A typical normalized microhardness profile (Fig. 1a) shows increasing hardness with depth until it reaches a constant value. The depth at which the maximum hardness is regained would therefore indicate the thickness of the layer from which Li is lost and the normalized microhardness profile would indicate a corresponding Li concentration profile within these layers. In the following analysis, variations in the normalized hardness close to the surface have therefore been directly interpreted in terms of the volume of Li depleted.

All the sheet materials in the as-received condition, already contained a certain Li-depleted zone. Since the primary aim of this investigation was to find out the effect of these solute depleted soft layers had on deterioration of mechanical properties of the sheet materials, these soft layers were not removed prior to the heat treatment. Normalized microhardness profiles in the as-received condition and peak-aged after solution heat treatment in air, argon or salt bath environments are shown in Fig. 1. With heat treatment the microhardness profiles receded deeper from the surface, indicating an increase in the thickness of the Li-depleted layer. Depletion of Li in spite of the existence of an already depleted layer shows that a prior Li-depleted layer causes no hindrance to further loss of Li to the environment on high temperature heat treatment. Papazian *et al.* [9] studied the loss of Li during the solution heat treatment of alloy 2090 clad with approximately 100 µm of Al alloy 7072. Their results showed that the cladding–base metal interface provided no impediment to the diffusion of

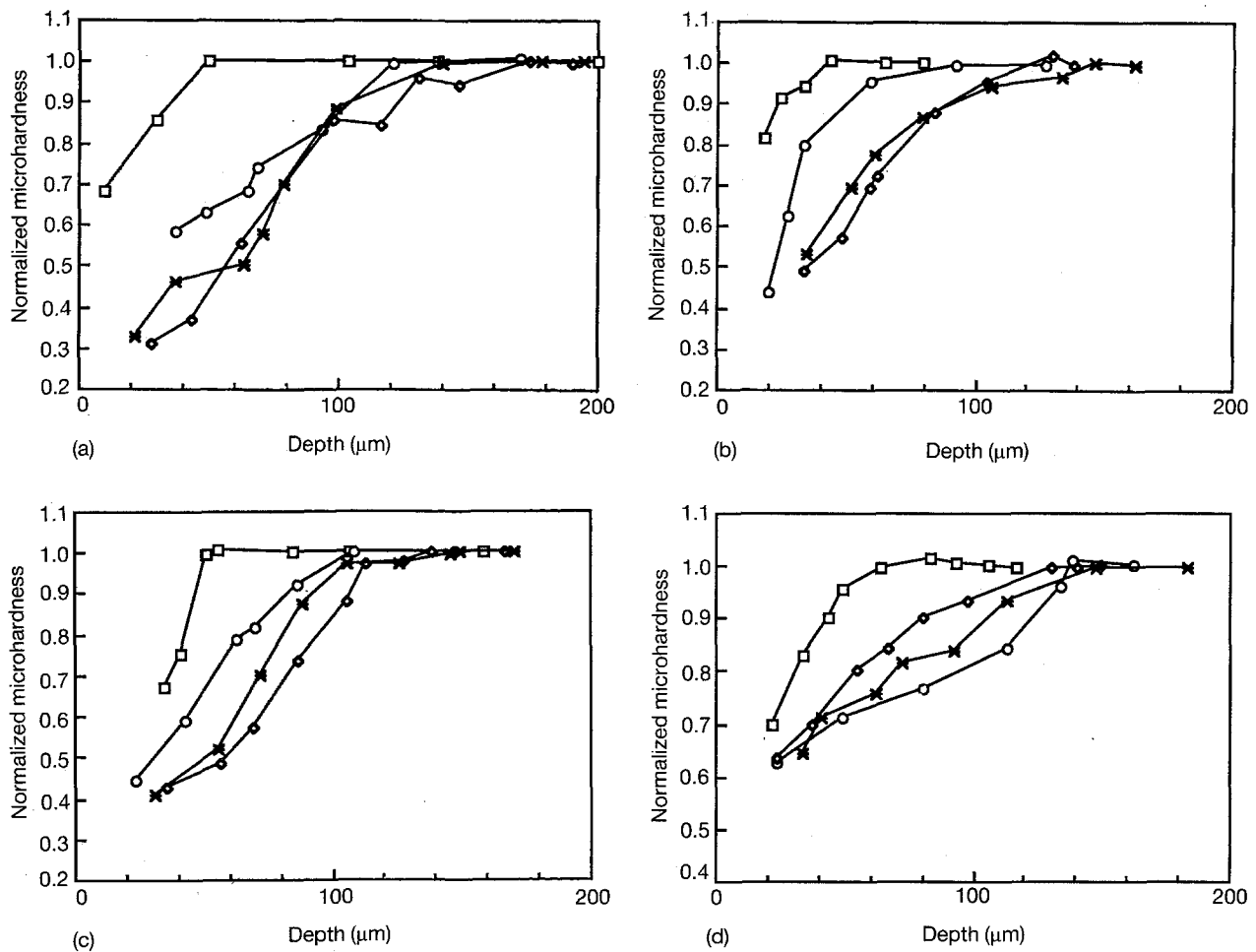
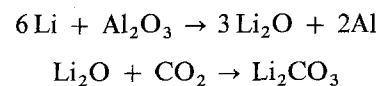


Figure 1 Normalized microhardness profiles of: (a) 8090 T6, 2 mm; (b) 8090 T6, 1 mm; (c) 8090 T8, 1.5 mm; (d) 2090 T3, 2 mm. □ As-received; ○ Air; ◇ Argon; * Salt bath.

Li and that the rate of diffusion in the cladding was similar to its rate of diffusion in base metal. Therefore, clad Al-Li alloys will be subject to the same Li loss and oxidation during solution heat treatment as the unclad Al-Li alloys. In fact, the prior Li-depleted layer itself will act as a cladding, making some external cladding to prevent Li depletion unnecessary. Also, the shape of normalized microhardness profiles in all the alloys did not change appreciably with heat treatment. This points out the fact that diffusion of Li from the bulk to the surface and its loss to the environment, either by oxidation or sublimation (in inert environments or under vacuum), is not influenced much by the composition of the surface layers and is primarily controlled by the concentration gradient alone. The depth of Li depletion after the heat treatments varied from 100 to 150 μm in all the alloy systems that were tested and is in good agreement with earlier studies by various investigators [4-7, 11].

In 8090 series alloys there is a small but appreciable change in the amount of Li lost to different environments used for the solution heat treatment. In all cases, solutionizing in air showed a minimum loss, whereas solutionizing in a salt bath or in a flowing argon atmosphere caused a relatively higher amount of Li loss. It is, in general, expected that inert environments will be more effective in reducing the loss of Li from the surface layers. The experimental results in the

present study are contradictory to the above expectation. This can be explained as follows: Ahmad [6] has shown that the dominant oxidation product in oxidising environments for Al-Li alloys at 530°C is Li_2CO_3 . Formation of Li_2CO_3 may occur in the following two step reaction proposed by Field *et al.* [5].



The Li_2CO_3 thus formed, and other oxidation products such as LiAl_2O_2 and Li_2O , form a porous and non-protective oxide layer on the surface [6]. It is physically plausible that the presence of an increasingly thick oxide layer may act as a barrier for diffusion of Li into the furnace atmosphere, thus increasing its concentration at the free surface. A non-zero surface concentration of Li would reduce the concentration gradient and hence decrease the rate of Li loss. When solutionizing was carried out in a flowing argon atmosphere there was no such chance to form any oxide layer. Also, Li that is lost to the furnace environment by sublimation at the free surface is carried away continuously by the flowing gas, maintaining a near zero concentration at the surface. Similarly, any oxidation products formed at the surface while heat treating in a salt bath will also be carried away by the

convection currents. This may be the reason for the relatively higher loss of Li when heat treated in flowing argon gas or in a salt bath.

The volume of Li depletion in the case of 2090 series alloys is not very dependent on the heat treatment environment (Fig. 1d). Also, the extent of Li depletion in these alloys was less than that in 8090 series alloys. 2090 series Al–Li alloys contain a lower amount of Li and a higher amount of Cu when compared to 8090 alloys. Hence, the effect due to the loss of strengthening precipitates associated with the loss of Li on the microhardness profiles will be relatively weaker. This might be the possible reason for the lower loss of Li that is observed (in terms of normalized microhardness) in 2090 series alloys when compared to the 8090 series.

3.2. Effect of Li depletion on mechanical properties

All the alloys studied in the present investigation were solutionized for 30 min at 530 °C and then artificially aged at 180 °C for 24 h. This corresponds to the typical industrial heat treatment procedure recommended for peak aging of Al–Li alloys. Also, the effect of Li loss is expected to be most pronounced in the peak-aged, high strength condition. An identical heat

treatment procedure was adopted for all the alloy systems studied, so as to compare the variations in mechanical properties after the heat treatment with the amount of Li depleted. Tables II–V give the tensile strength data for various alloys heat treated in different environments, with corresponding thicknesses of Li-depleted layer. On comparing the tensile properties after heat treatment in various environments it can be concluded that there is no major variation in mechanical properties with the heat treatment environments in general. However, in the as-received condition, as well as after the heat treatment, all the alloys showed consistently better mechanical properties when the Li-depleted soft layer was removed by mechanical polishing prior to tensile testing. This improvement was only marginal, except in the case of 1 mm thick sheets of 8090 T6 (Table III), where there was an improvement of about 12% in proof stress and 6% in ultimate tensile stress (UTS) after the Li-depleted soft layer was removed. This clearly shows that the effect of Li depletion is more pronounced in very thin sections. The results of the tensile tests conducted in the present investigation show that there is a considerable decrease in the strength of all the alloys of 8090 series after heat treatment. Probable reasons for this deterioration are discussed below.

Peel *et al.* [12] reported recrystallization in the

TABLE II Tensile properties of 8090 T6 (2 mm) under various conditions

Testing condition	Depth of Li depletion (μm)	0.2% proof stress (kg mm^{-2})	UTS (kg mm^{-2})	% Elongation
As-received (AR)	50	418	543	7.27
AR (soft layer removed)	0	424	552	6.88
SHT(air) + AA	120	381	498	6.88
SHT(salt bath) + AA	150	355	471	6.61
SHT(argon) + AA	140	368	479	6.61
SHT(air) + AA (soft layer removed)	0	387	498	6.88

TABLE III Tensile properties of 8090 T6 (1 mm) under various conditions

Testing condition	Depth of Li depletion (μm)	0.2% proof stress (kg mm^{-2})	UTS (kg mm^{-2})	% Elongation
As-received (AR)	45	429	541	6.26
AR (soft layer removed)	0	476	572	6.22
SHT(air) + AA	95	368	477	6.21
SHT(argon) + AA	120	367	474	6.61
SHT(salt bath) + AA	130	367	472	6.61
SHT(air) + AA (soft layer removed)	0	383	495	7.01

TABLE IV Tensile properties of 8090 T8 (1.5 mm) under various conditions

Testing condition	Depth of Li depletion (μm)	0.2% proof stress (kg mm^{-2})	UTS (kg mm^{-2})	% Elongation
As-received (AR)	50	323	443	8.33
AR (soft layer removed)	0	326	459	8.20
SHT(air) + AA	110	266	369	8.20
SHT(salt bath) + AA	130	250	340	8.20
SHT(argon) + AA	120	273	387	7.40
SHT(air) + AA (soft layer removed)	0	289	404	7.74

TABLE V Tensile properties of 2090 T3 (2 mm) under various conditions

Testing condition	Depth of Li depletion (μm)	0.2% proof stress (kg mm^{-2})	UTS (kg mm^{-2})	% Elongation
As-received (AR)	70	305	436	14.8
AR (soft layer removed)	0	308	443	14.3
SHT(air) + AA	150	344	485	14.1
SHT(salt bath) + AA	150	252	387	4.96
SHT(argon) + AA	150	341	472	14.3
SHT(air) + AA (soft layer removed)	0	362	496	12.6

surface layers due to solute depletion in Al–Li alloys. In this context, since the as-received material itself has a lithium-depleted zone which was not removed prior to solutionizing, there is a possibility for the recrystallized grains in the surface layers to grow into the interior of the sheet upon elevated temperature heat treatment. This kind of deterioration is more pronounced in 8090 T8 series Al–Li alloys, which have a partially recrystallized structure in the as-received condition. A particular case is the 8090 T8 alloy specimen, solution heat treated in a salt bath. The proof strength and UTS of this material were lower than that of the same material heat treated in air or argon under exactly similar conditions. The grain size in the as-received 8090 T8 alloy is typically $10\ \mu\text{m}$, whereas the grain size after heat treatment in the salt bath was $120\text{--}200\ \mu\text{m}$, indicating that there was a large grain growth upon heat treatment. Fig. 2a represents typical fracture features observed in the 8090 T8 alloy specimen which was solution heat treated in air and peak-aged prior to testing. It clearly shows that the fracture is intergranular in nature with a grain size approximately equal to the grain size in the as-received condition. Fracture features in a specimen which was solutionized in a salt bath, on the other hand, show a $600\ \mu\text{m}$ thick layer from the surface where extensive grain growth has taken place (Fig. 2b). Based on studies on the extent of recrystallization in a 50% cold-rolled 8090 Al–Li alloy sheet as a function of heating rate up to a temperature of $530\ ^\circ\text{C}$, Bowen [14] has concluded that increasing heating rate increases the amount of recrystallization. The heating rates to $530\ ^\circ\text{C}$ in a salt bath are very high compared to the heating rates in air or argon, which

might have resulted in higher recrystallization and grain growth leading to the relatively higher loss of strength in specimens which are heat treated in a salt bath. From Fig. 2b it can also be noted that there is no grain growth at the midsection of the sheet where the grain size is equal to the grain size in the as-received material. Using electron backscattering diffraction to map the grain-specific microtexture, Wismayer *et al.* [13] have shown that the centre section of a 1.6 mm Al–Li alloy sheet conforming to the composition of 8091 retains the deformed structure and texture, whereas at the surface, recrystallization and grain growth take place. In the present case, this may be the reason for the observed difference in the grain size at the centre section when compared with those of the surface regions.

There was a considerable increase in the proof strength and UTS, with a concomitant reduction in the ductility, in 2090 series alloys with heat treatment (Table V). The fracture features also changed with the heat treatment. In the as-received condition fracture features were dimple-like (Fig. 3a), indicating ductile failure. On the other hand, in samples tested after the heat treatment they were highly intergranular (Fig. 3c). Al–Li alloys are prone to inhomogeneous deformation due to planarity of slip in the coherent δ' precipitates (see for example [18]). This slip planarity increases with ageing time, leading to strain localization at the grain boundaries and hence loss of ductility. The as-received material, which was in an under-aged (T3) condition, therefore showed better ductility due to less strain localization. When re-solutionized and peak-aged, however, an increase in the slip planarity can be expected, which is possibly

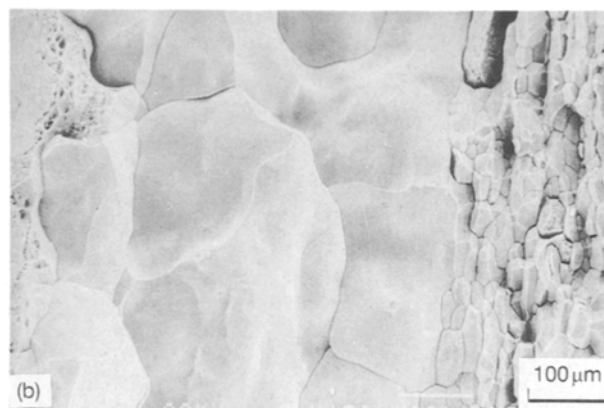
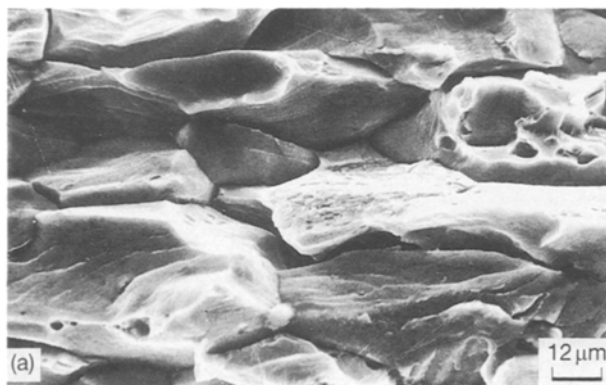


Figure 2 Scanning electron fractographs of 8090 T8 specimens tested after (a) SHT (air) and peak ageing and (b) SHT (salt bath) and peak ageing. See text for details.

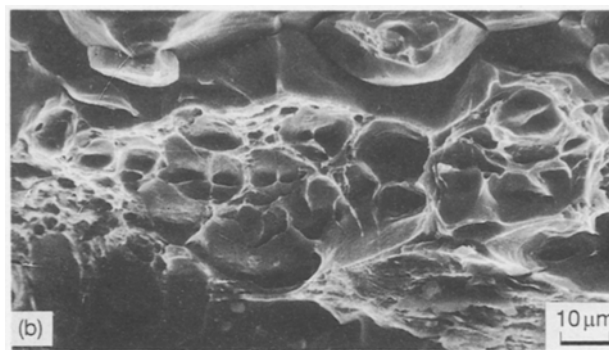
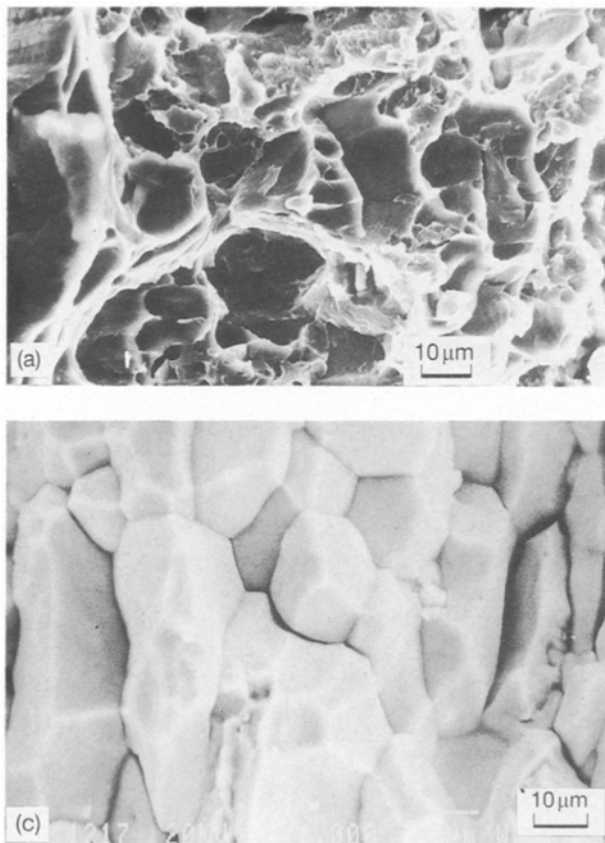


Figure 3 Fracture features in 2090 (a) in the as-received condition, (b) near the surface and (c) in the bulk. (b, c) From specimens tested after SHT (air) and peak ageing.

the reason for the observed loss of ductility. Specimens of the 2090 alloy, solution heat treated in salt bath showed poor mechanical properties relative to specimens heat treated in argon or air. This may be due to the strong exfoliation that was observed in this particular alloy sheet material when heat treated in sodium nitrate salt bath. The reasons for this exfoliation, however, are not known.

Extensive fractography has been carried out in order to observe the variations in fracture features in the Li-depleted zones with those in the bulk of the material. Figs 3(b) and 3(c) show the SEM fractographs in the Li-depleted layer and in the bulk of the material, respectively, of the 2090 (solutionized in salt bath and peak-aged). Whereas fracture in the bulk of the material (Fig. 3c) is highly intergranular, fracture in the Li-depleted layer is ductile with dimple-like features. This type of failure was observed to a depth of 60 μm , which is typically equal to the precipitate free zone. Similar differences in the fracture features were also observed in the 8090 T6 series alloys. Fig. 4(a) shows dimple-like fracture features near the free surface in these series alloys after the heat treatment and Fig. 4(b) shows the fracture in the corresponding bulk of the material, which is intergranular with some secondary cracking.

There are two conflicting reports about the sub-surface structure in the Li-depleted layers of Al–Li alloys. Papazian *et al.* [15] have reported the development of a band of sub-surface pores after heat treatment at 500 °C in 8090, 8091 and 2090 alloys in vacuum, air and hydrogen environments. They argued that the band of porosity formed is due to the differences in diffusivities of Al and Li in the Al alloy matrix.

At a typical solutionizing temperature of 550 °C Li diffuses 4 times faster than Al and this causes a significant flux of vacancies into the metal sub-surface via Kirkendall diffusion process and the pores are formed due to condensation of these vacancies. On the other hand, Dickenson *et al.* [16] observed coarse LiH particles and hydrogen porosity in the metal substrate of these alloys. They suggested that since LiH particles are soluble both in alcohol and water, these particles were mistakenly identified as pores in the studies relying on observation of metallographic cross-sections where alcohol or water were used for preparation of the samples, as observed by Papazian *et al.* [15]. Based on their observations, Dickenson *et al.* proposed that atomic hydrogen diffuses along the grain boundaries into the Al matrix and precipitates as LiH when it encounters a sufficient concentration of Li. When the Li concentration of the matrix surrounding the LiH particle falls below a critical level, the particle dissolves. While the liberated Li diffuses to the surface, the H diffuses in the opposite direction until it again forms LiH leading to a sub-surface precipitate-free zone. The formation of hydrogen porosity was also observed by Canaby *et al.* [17], who have shown a correlation between the formation of pores and hydrogen contamination of surface layers during solutionizing heat treatment and tungsten-inert gas and electron beam welding processes of 8090 Al–Li alloys.

In the present study the observation of dimple-like fracture features in both the 2090 and 8090 series alloys (Figs 3b and 4a, respectively) to a depth of 60 μm indicates ductile fracture within these Li-depleted surface layers. This type of fracture feature is

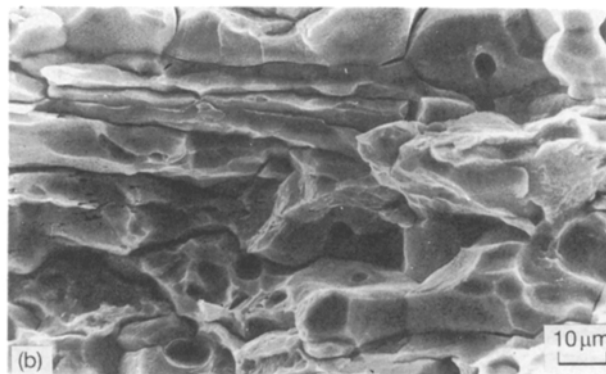
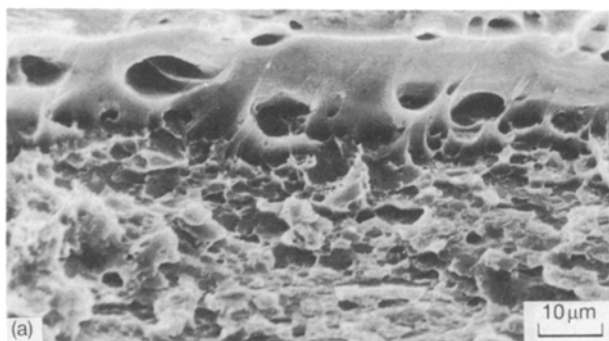


Figure 4 Fracture features in 8090 T6 after SHT (air) and peak ageing (a) at the free surface of the specimen and (b) in the bulk of the material.

observed, in general, in the un-aged (i.e. precipitate-free) condition of high strength Al alloys, indicating that these surface layers are indeed precipitate-free. Also, ductile fracture features were observed, approximately to a depth of 60 μm , which is in conformity with the observations of Dickenson *et al.* [16] confirming hydrogen-induced sub-surface porosity formation within the Li-depleted surface layers.

4. Conclusions

Based on the observations made in the present study, the following conclusions can be drawn about the behaviour of Li depletion and its effect on properties and fracture behaviour of Al–Li alloy sheets.

1. Evidence for Li depletion is found in both 8090 and 2090 series Al–Li alloys on heat treatment, as reflected in the microhardness profiles at the surface. Li depletion is found to be more in 8090 materials than in 2090 and the presence of a Li-depleted layer prior to the heat treatment does not hinder the loss of Li to the surroundings at the free surface.
2. The soft layers that are developed near the surface due to Li depletion have very little effect on the tensile properties of these alloy sheets in the peak-aged condition. However, some degradation of properties is observed in very thin sections.
3. The fracture features observed in Li-depleted layers are quite different from those observed in the bulk of the material. Dimpled fracture features near the surface indicate pore formation due to hydrogen contamination.
4. Extensive grain growth in the surface layers due to recrystallization and grain growth in the solute depleted layers might be a contributing factor to the loss of strength in 8090 series alloys.

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