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Failure analysis of a hot extrusion die used for Al–Li alloy processing

U RAMA MURTY, G S AVADHANI and E S DWARAKADASA
Department of Metallurgy, Indian Institute of Science, Bangalore 560012, India

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Abstract. Al–Li alloys being developed as lighter substitutes for conventional high strength Al alloys are to be processed by routine methods. During extrusion of a 8090 Al–Li alloy, the extrusion die container failed causing some alarm. This failed die container was analysed to examine if the failure was caused by interaction of Li diffusing out of Al–Li alloy with the carbides of die steel. The evidence, although not conclusive, is sufficient to exercise caution during such processing.

Keywords. Al–Li alloys; extrusion; die failure; diffusion; Li-carbide interaction.

1. Introduction

Al–Li alloys are being developed as low density direct substitutes for currently used high strength Al alloys in the aerospace industry. The potential reduction in weight and hence operating costs predicted for Al–Li alloy structures have led to extensive investigations for specific applications of these alloys by the aerospace industry. The possibility of integrating Al–Li alloys into existing production facilities gives an advantage to this approach over composites, the other class of competing materials. To facilitate a smooth transition, aspects of the behaviour of these new alloys which may differ from that of traditional alloys, and their influence, if any, on other materials especially in view of the presence of lithium, must be considered. It is a tribute to the aluminium industry that many of the problems associated with the manufacture of Al–Li alloys have now been overcome (Grimes *et al* 1987).

One particular problem which is associated with Al–Li alloys is the depletion of Li from the surface during high temperature processing due to the high diffusivity of Li in aluminium matrix (Papazian *et al* 1986). The Li that diffuses to the surface from the bulk may react with the atmosphere or the material in contact with it which might lead to a premature failure of the component that is both expensive and hazardous. Since Li is not an engineering material, nothing much is known about its reaction with and effect on other engineering materials. The use of Al–Li alloys is a new situation which has brought this particular problem to focus. Study of a failed steel extrusion die container that was used for processing indigenously developed Al–Li alloys is a case for consideration. The effect of Li diffusing out from the Al–Li alloy and causing this failure is of particular interest to foundries and extrusion industries, handling Al–Li alloys.

2.1 *History of the failed component*

The cross-section of the die container is shown in figure 1a. The container material was a hot work die steel and the composition range of the die and the punch materials are given in table 1. The container, die and the punch were heat-treated according to details given in table 2, which conformed to the data given by the supplier.

The die container was in use for the extrusion of Al, Pb, Cd and Mg base alloys over a period of 4 years and for extruding Al–Li alloys (mostly 8090) during the last one-year period. All extrusions were carried out in an electrically operated 250 ton hydraulic press. During one such hot extrusion experiment, the die failed by cracking into pieces while processing an Al–Li alloy having the following composition (in

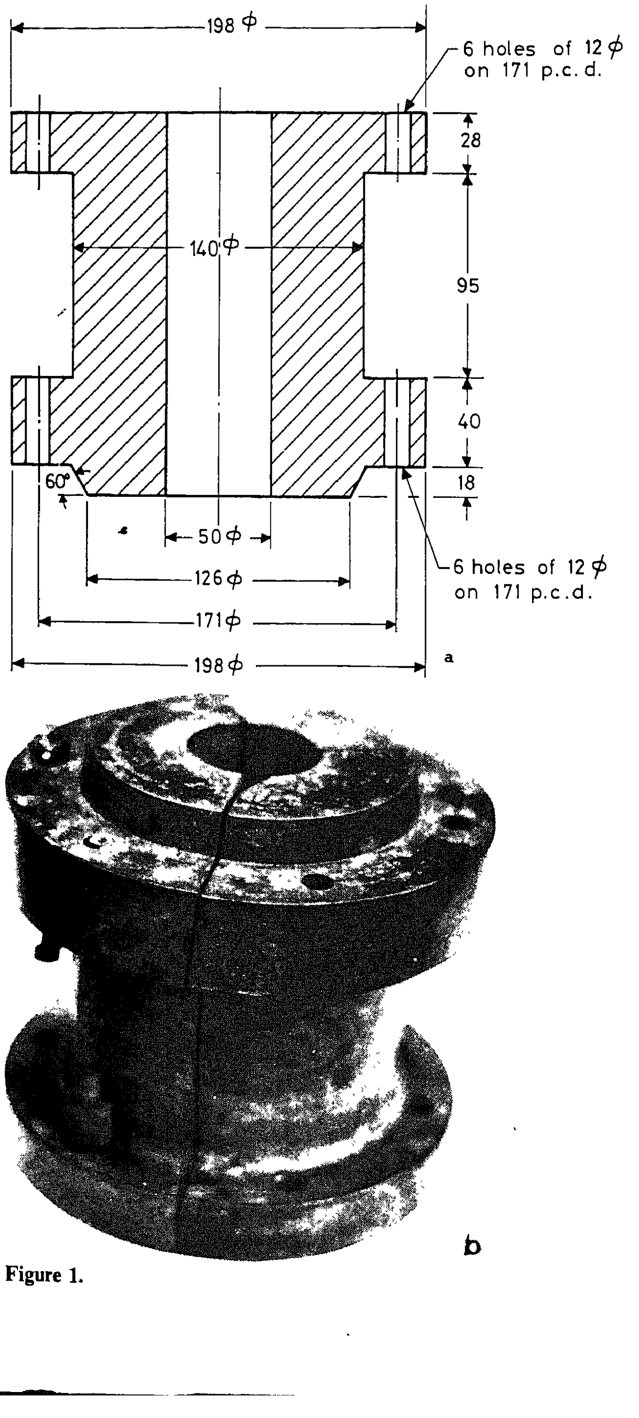
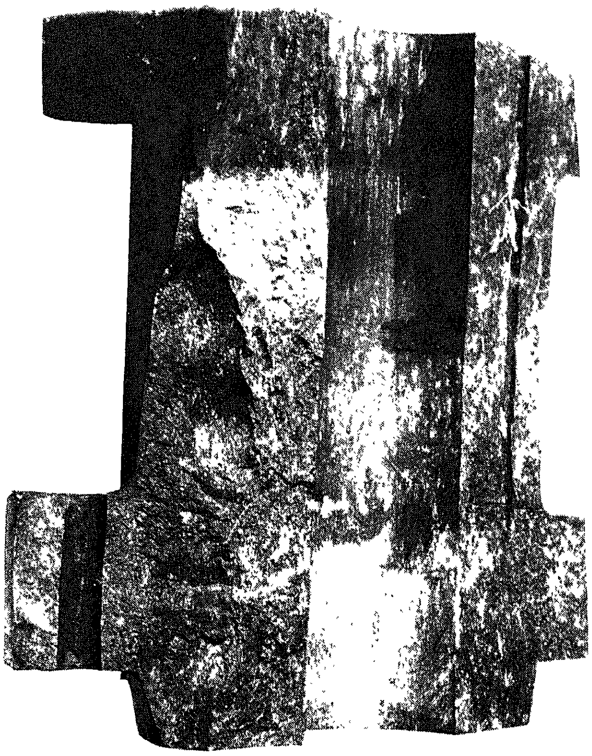


Figure 1.



c

Figure 1. **a**. Cross-section of the die container (schematic). **b**. picture of the failed die container and **c**. fractured surfaces.

Table 1. Chemical compositions (in wt%) of various die components.

Component	Die	Container	Punch
C	0·30–0·40	0·33–0·43	0·26–0·36
Mn	0·20–0·5	0·20–0·5	0·15–0·4
Si	0·80–1·2	0·80–1·2	0·15–0·5
Cr	4·75–5·5	4·75–5·5	3·0–3·75
Ni	0·3 max	0·3 max	0·3 max
Mo	1·25–1·75	1·10–1·60	—
W	1·0–01·7	—	8·5–10·0
V	0·5 max	0·3–0·6	0·3–0·6

wt.%) 2·5–2·6 Li, 0·8–1·0 Cu, 0·8–1·0 Mg, 0·09–0·1 Zr, 0·15 max. Fe and Si, 0·05 max. impurities, balance Al. About 50 Al–Li alloy extrusions had been completed prior to failure. The dimensions (in mm) and extrusion ratios involved were: Billet — 50 dia. and 75 length; extrusion ratios: 4, 9, 16 and 25; extrudant shape: cylindrical with sizes 10, 16, 20 and 25 mm, rectangular with a size of 25 × 10 mm.

Table 2. Various heat treatments given to the die components.

Component	Heat treatment	Rockwell hardness
Die	1253 K 1 h, oil quench (343 K) and tempered at 450 K for 2 h	60
Container	1223 K 2 h, oil quench (343 K) and tempered at 450 K for 6 h	59
Punch	1223 K 1 h, oil quench (343 K) and tempered at 450 K for 4 h	58/59

2.2 *Extrusion conditions at the time of failure*

Cast billets of Al–Li alloy 8090, 50 mm in dia., cut from 180–200 mm long ingots to a size of 75 mm, and externally heated in a muffle furnace for 2–4 h, at 673 K, were placed in the die container. To prevent the punch sticking to the alloy, billets were topped with a die washer. The whole set of die and container was externally heated by an electrical resistance furnace to maintain a uniform temperature of 523–533 K at the outer surface. The extrusion temperatures were 523–653 K; but most extrusions were carried out with a billet temperature of 623 K. The billet cools by about 10 K before the start of the extrusion but reaches 653–663 K during the final stages. The extrusion period is about 10–20 min from the placing of billet in the container until the removal of the extrudant and the discard.

2.3 *Fracture*

The die was in good use over its life period of about 5 years. Even after starting the extrusion of Al–Li alloys, many Al–Li extrusions had been successfully completed. At a time when extrusion of an alloy conforming to 8090 Al–Li alloy composition was being made, at an extrusion ratio of 16, the die failed with an accompanied audible sound. The presence of mind of the operator prevented the pieces from flying off but ended up with the pieces of the failed die falling apart on the bed of the press. Nearly all the pieces could be collected and were checked for the completeness of the die. There was no damage on the punch whatsoever.

The die failed into three large fully fractured pieces (figure 1b) and several smaller fragments from areas close to the die lip. The major fracture surfaces (figure 1c) did not reveal any signs of brittleness or the presence of any prior cracked regions which might occur due to bad heat-treatment. The fracture surface was quite granular indicating a very ductile failure. The nature of failure indicated that the cracking initiated from the inner surface of the die and opened outwards. It is known that elemental Li reacts with carbides of iron and other elements, and can cause embrittlement; also during high temperature treatment of Al–Li alloys, there occurs Li diffusion to the surface and loss by sublimation. This prior knowledge prompted this investigation to determine if Li was responsible for the failure of the die.

3. Microstructure and fractography

A piece of the failed die was prepared and examined metallographically to determine the heat-treated structure of the steel (figure 2a) which contained a bimodal distribution of spheroidal carbides of size 0·5–1 μm and 2–3 μm. In some locations there also existed some amount of lamellar carbide, fairly coarse in size (figure 2b). However, it was confirmed that this situation did not alter the fracture behaviour of the steel.

Representative areas of the fracture were examined with SEM to determine the fracture behaviour. Fracture always appeared to be ductile (figures 3a and b), and indicated a predominant carbide/matrix decohesion as the mechanism of fracture for both spheroidal carbide and the lamellar carbide. The fracture morphology near the inner edge of the die that was in contact with the Al–Li alloy extrudant was completely different (figures 4a and b). Areas close to the inner edge of the die revealed the presence of surface irregularities in an otherwise well-polished surface. There occurred visible areas of interaction, about 90–100 μm deep. These are considered to be the regions in which Li reacted with the steel to generate nucleation of the failure. In several locations adjacent to the interaction zone the stronger carbide exhibited fracture by cleavage (figure 4a). Figure 5 exhibits the presence of a deep crack separating the regions of failure that are ductile in nature on the one side and what appears to be cleavage on the other side.

4. Discussion

The high strength steel die, which was in good service for the hot extrusion of various metals, failed on being continuously used for extruding Al–Li alloys. Reference to the mechanical properties of the particular steel (Metals 1980), confirmed that the container material exhibited the most optimum properties and perhaps the best crack growth resistance in the heat-treated condition. This also corroborates with the observation that the fracture surface did not show step-wise growth of crack which is an indication of a brittle fatigue failure. The fracture appeared to be more static in nature. This necessitated a serious examination of the correctness of using high strength, hot-worked die steel containers for extruding Li-containing alloys.

During high temperature processing of Al–Li alloys, a certain amount of Li loss has been observed, which takes place by Li diffusion to the surface and depletion from the surface. The rate of Li loss at elevated temperatures is considered to be relatively independent of environment but limited primarily by the diffusivity of Li in the alloy (Ahmad 1987). This Li being highly reactive will immediately react with the atmosphere. In oxidizing atmospheres, a number of compounds may form due to a variety of reactions (Wefers and Mozelewski 1988). If the Al–Li alloy is in intimate contact with another material, the elemental Li and Li compounds may react at the interface and, in addition to this, elemental Li may penetrate into the surface layers and react with the constituent phases of the adjacent component, damaging it. This can be appreciated well from the fact that Li metal has been shown to react even with a stable ceramic compound like alumina (Konys and Borgstedt 1985).

Wu *et al* (1990) studied the chemical interactions between metallic Li; Li_2CO_3 and Li_2O with AISI 316 steel using secondary ion mass spectroscopy (SIMS) to find that the steel was penetrated by Li^+ ions together with the corresponding anions via

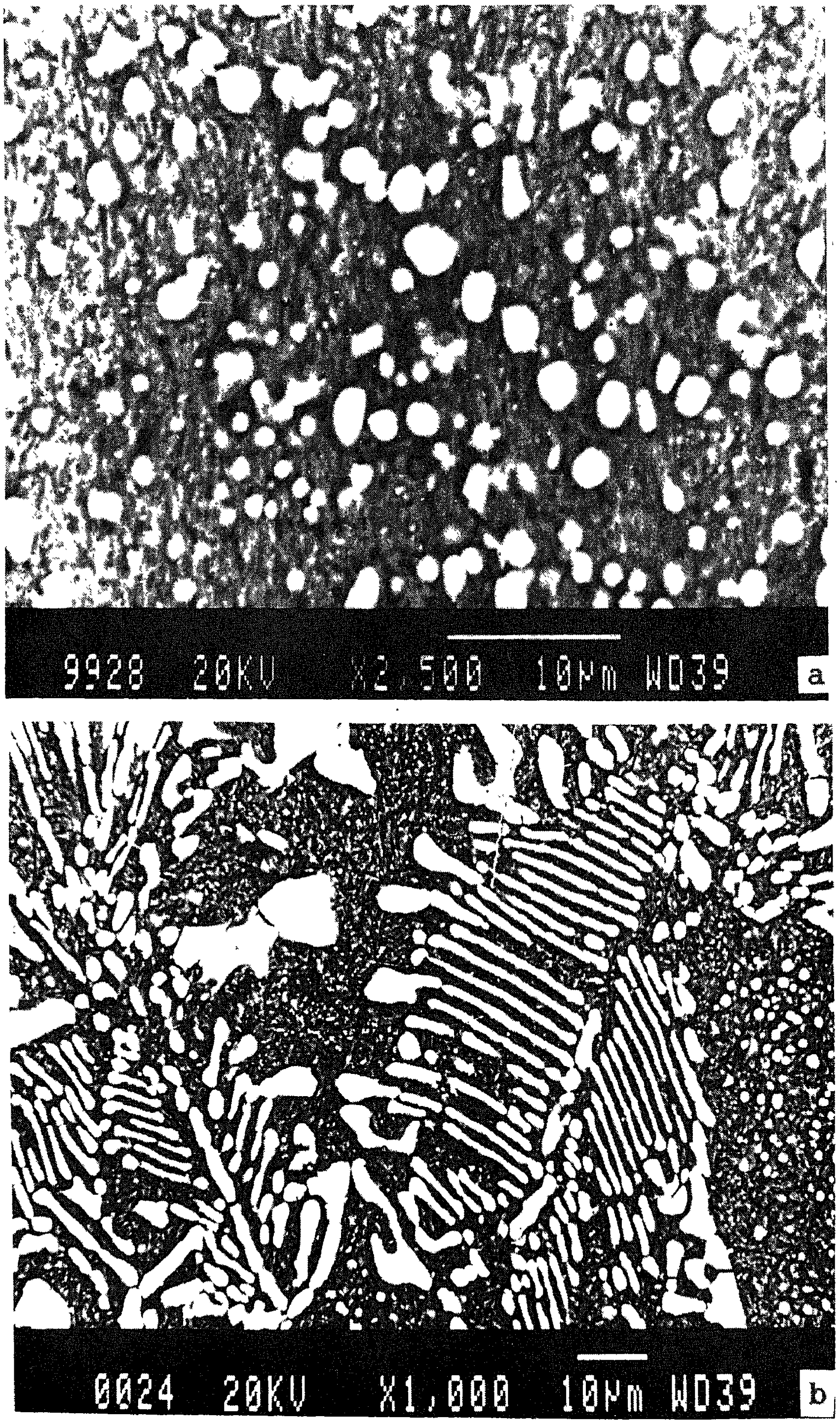


Figure 2. Microstructure of the die container steel showing **a.** a bimodal distribution of spheroidal carbides and **b.** lamellar carbide.

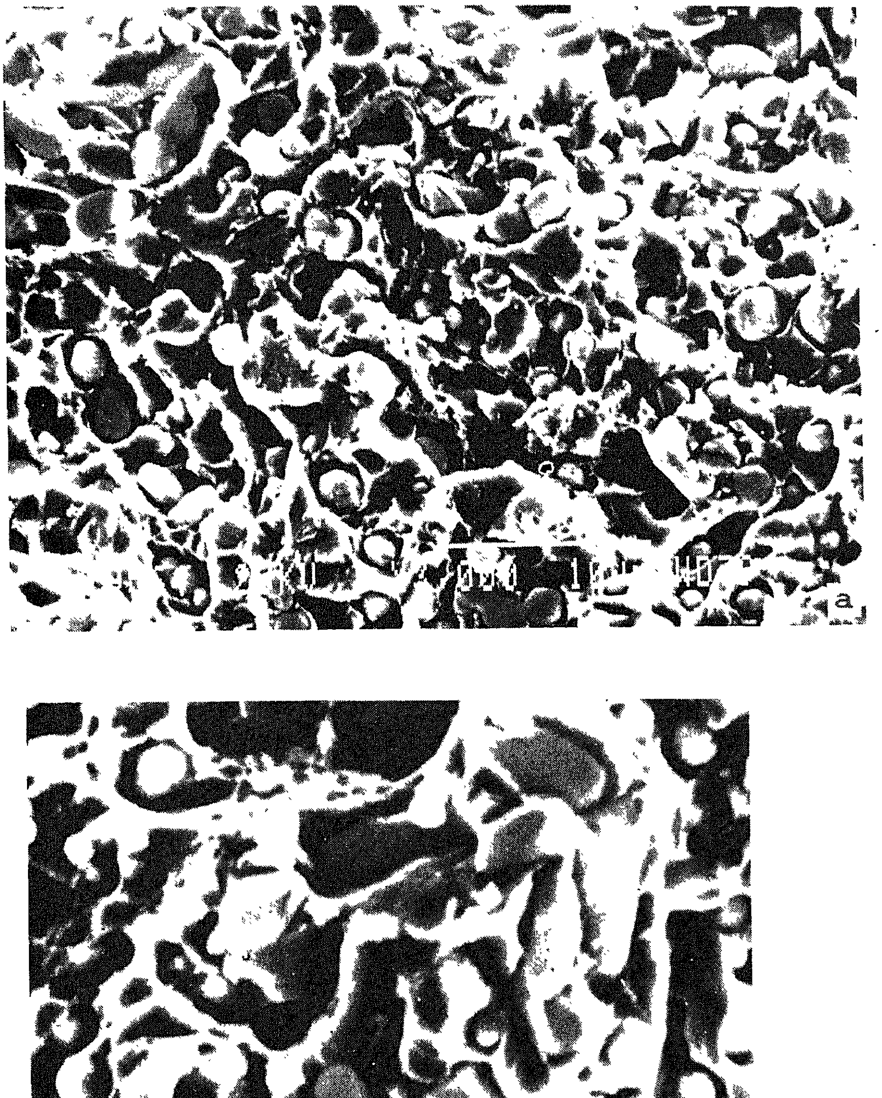


Figure 3. **a** and **b**. Fracture features in the bulk of the die container steel showing carbide/matrix decohesion.

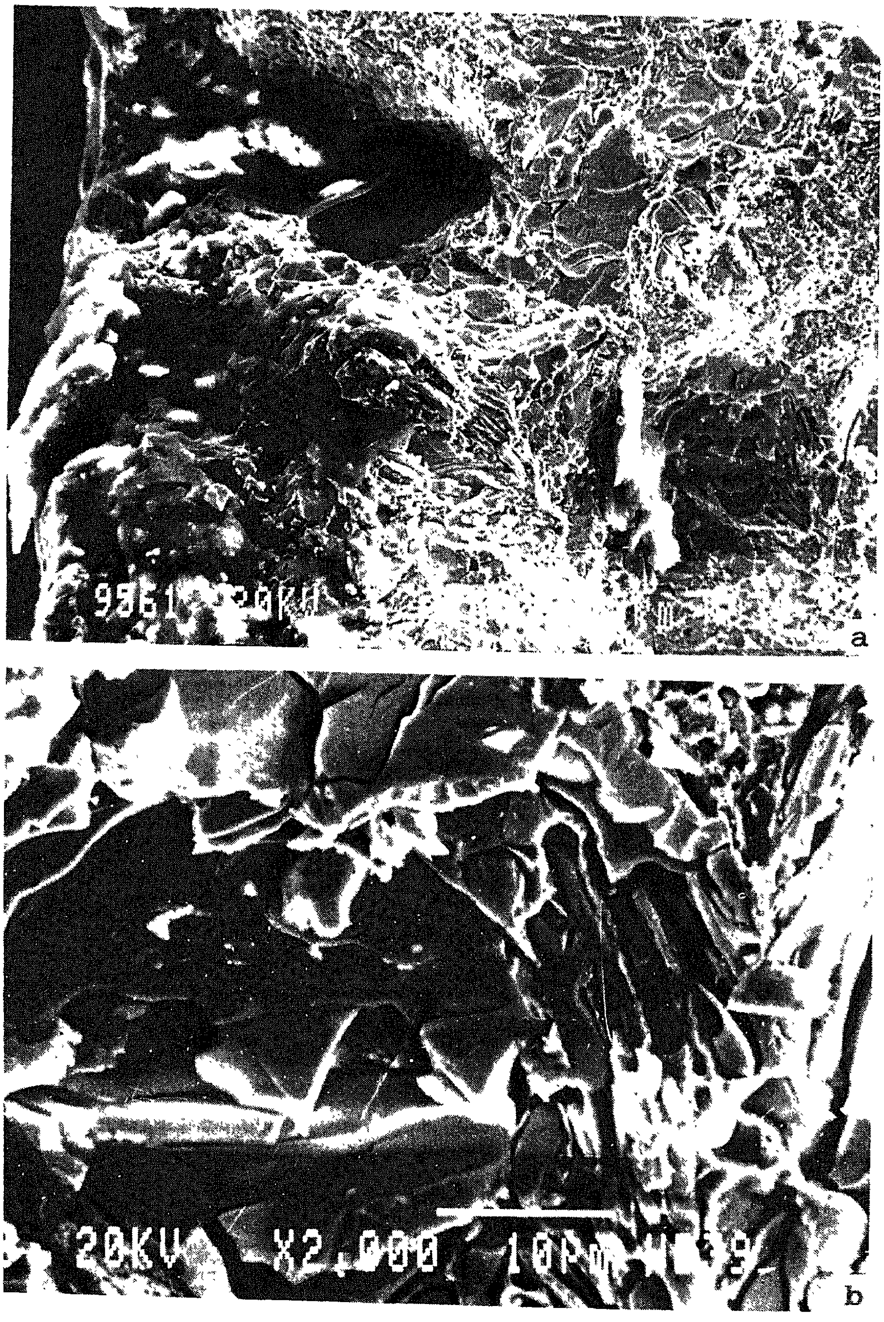


Figure 4. a. SEM fractograph taken close to the edge of the failed container showing interaction zone and fracture by carbide cleavage and **b.** more clearly seen at higher magnification.

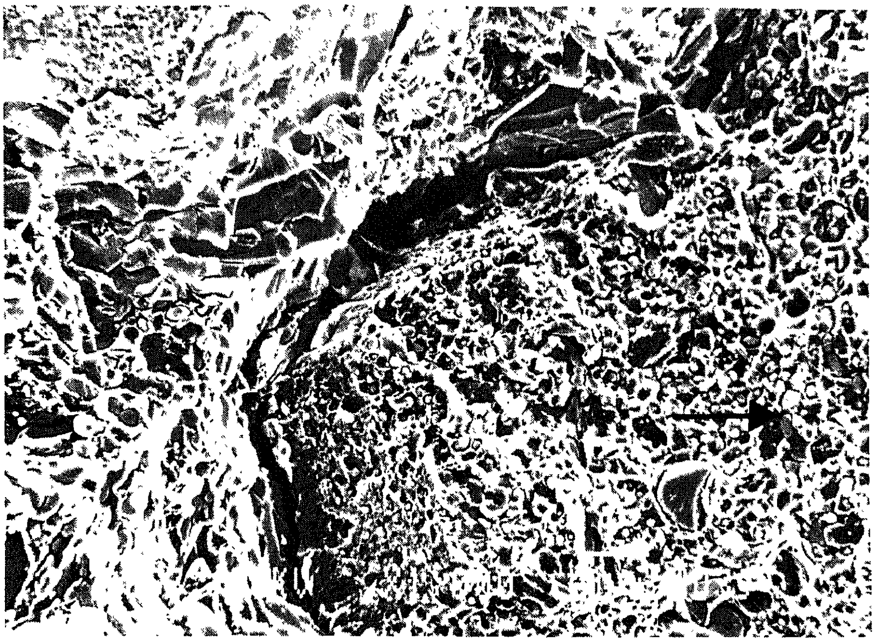


Figure 5. A deep crack near the surface of the failed container showing carbide/matrix decohesion on one side (away from the interaction zone, indicated by the arrow) and carbide cleavage on the other (towards the interaction zone).

grain boundaries. Chromium is extracted from the grains to give chromium-depleted steel. Wu *et al* also reported the formation of stable ternary compounds at grain boundaries and at the steel surface according to the following reactions,

$$\mathrm{Cr} + 2\,\mathrm{Li_2O} \rightarrow \mathrm{LiCrO_2} + 3\,\mathrm{Li}\uparrow. \tag{1}$$

$$\mathrm{Fe} + 4\,\mathrm{Li_2O} \rightarrow \mathrm{Li_5FeO_4} + 3\,\mathrm{Li}\uparrow, \tag{2}$$

$$5\,\mathrm{Li_3N} + \mathrm{Cr} \rightarrow \mathrm{Li_9CrN_5} + \mathrm{Li}\uparrow, \tag{3}$$

$$\mathrm{Cr_2O_3} + \mathrm{Li_2O} \rightarrow 2\,\mathrm{LiCrO_2}. \tag{4}$$

Barker *et al* (1988) reported that the corrosion of 316 steel by lithium is more pronounced in the presence of oxygen impurities.

In the present context, since the Al–Li billet is homogenized in a muffle furnace for 4 h in normal furnace atmosphere, it is probable that considerable amount of Li compounds such as $\mathrm{Li_2CO_3}$, $\mathrm{Li_2O}$ and $\mathrm{Li_2N}$ etc would have formed on the surface due to the preferential oxidation of Li depleting from the bulk (Field *et al* 1981). Most of these compounds have low densities, form in a fluffy manner and loosely adhere to the base material. On introducing the billet into the die container, much of these extraneous compounds would deposit on the die container surface. That such a material transfer took place was confirmed by the observation of the bright die surface getting coated with a white powder rendering the surface to become quite dull. During extrusion, these compounds may interact with the die container material at the surface.

The elemental Li diffusing out from the bulk of Al–Li alloy billet and the Li released from the surface reacting according to any one or all of the reactions (1), (2) and (3) will penetrate into the bulk of the die material. The high temperatures and pressures that exist at the die/billet interface, make the reaction kinetics suitable for this. The surface irregularities observed in the lower portion of the die container were probably initiated by the interaction of the carbides with the elemental Li and Li^+ thus penetrated.

From figure 4a, it may be observed that fracture by cracking of carbides is only up to a depth of about 150–170 μm. It is interesting to note that this depth matches approximately well with the depth of Li penetration in 316 steel that has been observed (about 140 μm) by Wu *et al* (1990). Also it is observed that while the fracture mechanism in general in the steel is by carbide/matrix interface decohesion (figures 3a and b), close to the surface the decohesion does not appear to be predominant. On the other hand a number of larger carbide particles have shown tendency to fracture by cracking. This type of fracture is considered to occur due to the interaction of Li with carbides.

In order to get confirmatory evidence, a pair of well-polished carbon steel plates were interlined with a thin sheet of 8090 Al–Li alloy and clamped together using a C-clamp. This composite was then soaked at a temperature of 673 K (same as the temperature of extrusion) for 4 h in a vertical tube furnace. After soaking, the clamp was removed, and the plates were attempted to be separated. In a number of cases the Al–Li alloy plate and the steel plates got stuck to each other and required considerable force to separate. On being separated, a certain amount of material transfer on to the steel surface was observed. SEM observations revealed that the steel surface was attacked and severely pitted (figure 6). A transverse section in the attacked region of the steel did not show any variation in the microstructure. However microhardness measurements (figure 7) indicated a small decrease in hardness to a depth of about 150 μm. This obviously should be due to the attack of Li on the carbides.

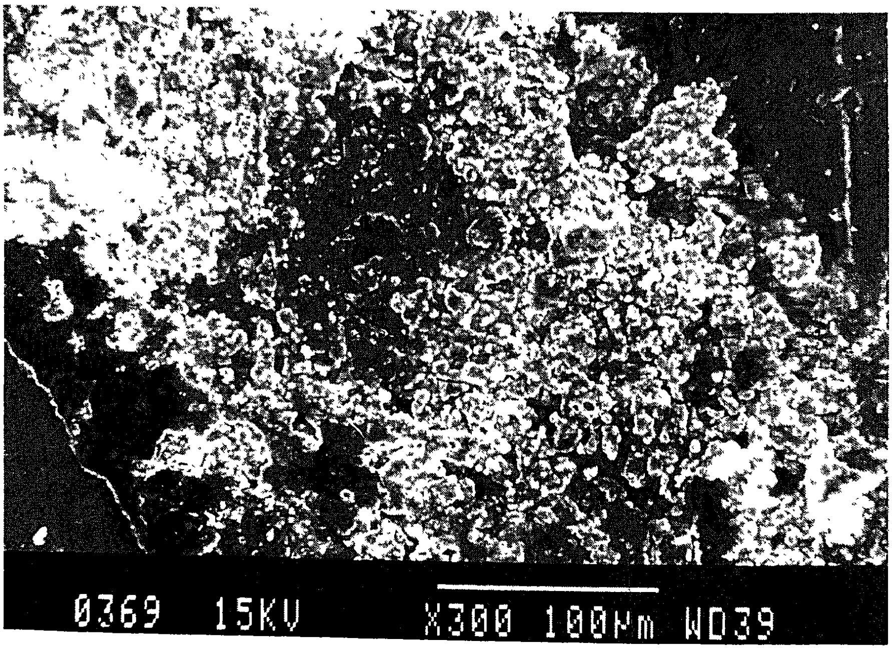


Figure 6. SEM picture of the steel surface after diffusion anneal of Al–Li alloy/steel couple showing deep pitting.

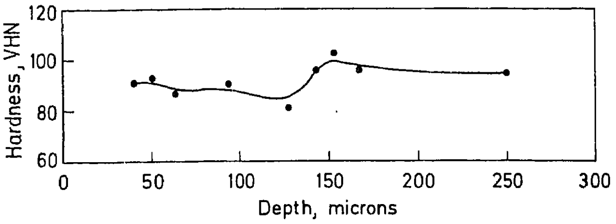


Figure 7. Microhardness profile of the transverse section of the steel after heat treatment in combination with Al–Li alloy.

Close to bottom region of the die container (figure 5), a crack about 3–5 μm wide appears as though formed by a penetrating fluid. This crack also separates regions of ductile failure on the right (interior) and cracked carbides on the left (closer to the surface). This was an abnormal observation not generally found in heat-treated H11 steel. It is therefore thought that this kind of failure may be due to interaction with Li. Further experiments are in progress to determine the exact nature of the effect of Li on high strength steels.

5. Conclusions

(i) A high strength steel die container used for hot extrusion of Al–Li alloy fractured in a ductile manner during an extrusion experiment.
(ii) In the interaction zone, fracture mechanism near the surface was by cracking and fracturing of carbides as against interface decohesion at the carbide/steel matrix interface that is observed in the bulk.
(iii) Li diffusing out from the Al–Li alloy billet is thought to interact with carbides in steel and this may have caused the observed deterioration. Steel dies for handling Li containing Al alloys should be used with caution.

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