EFFECTS OF ALKALI METAL IMPURITIES ON S-L FRACTURE TOUGHNESS OF 2090 Al-Li-Cu EXTRUSIONS

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Abstract

The effects of alkali metal impurity (AMI) content on the short-transverse fracture toughness of 2090-T8 alloy extrusions were studied. Material with < 2 ppm AMI's produced by vacuum refining had high S-L fracture toughness (up to 38 MPa.$\sqrt{m}$ for yield strengths ~440 MPa) compared with material with AMI's of 3-10 ppm which are characteristic of commercially available non-vacuum refined Al-Li alloys. The increase in fracture toughness with decreasing AMI content was associated with a decrease in the proportion of brittle islands on fracture surfaces. Both the present and previous studies indicate that these brittle islands result from liquid-metal embrittlement due to the presence of discrete alkali-metal rich liquid phases. The effects of temperature and strain rate on fracture toughness were also studied, and their effects could also be explained in terms of a liquid-metal embrittlement mechanism. The effects of grain structure on fracture toughness are also briefly discussed.

Introduction

One obstacle to the greater commercial acceptance of lithium-containing aluminium alloys (Al-Li) as a lower density alternative to conventional high strength aluminium alloys is their tendency to have a low and variable short-transverse (S-L) fracture toughness. Low S-L fracture toughness values are usually associated with the occurrence of low-energy intergranular fracture. This behaviour has been attributed to a number of causes including: i) coplanar slip which causes stress concentrations at grain boundaries [1, 2], ii) weak precipitate free zones adjacent to grain boundaries [2, 3], iii) coarse and/or large area fractions of grain boundary precipitates [2, 3], iv) the presence of hydrogen at grain boundaries, perhaps as a hydride [4, 5], v) lithium segregation at grain boundaries [6, 7] and vi) the presence of discrete alkali metal impurity (AMI) phases at grain boundaries [8, 9].

AMI's such as sodium and potassium are introduced into lithium-containing alloys through feedstock and pick up from refractories. In commercial alloys produced by conventional melting and casting techniques, these AMI's are typically present at levels ranging between 3-10 ppm. In most aluminium alloys, sodium and potassium are tied up in innocuous solid compounds by bismuth or silicon [10], but this is not the case in Al-Li alloys because lithium reacts preferentially with these elements. In Al-Li alloys containing high levels of AMI (>60 ppm), transmission electron microscopy (TEM) has revealed the presence of discrete liquid Na-K phases centred on grain boundary inclusions [8]. Surface analysis of fracture surfaces from alloys with AMI levels as low as 5 ppm have shown the presence of discrete sodium-rich areas [9]. Sodium-potassium phases are known to cause liquid metal embrittlement (LME) in aluminium alloys, as illustrated by embrittlement caused by the application of Na-K phases to the external surfaces of stressed specimens [11]. Evidence of LME of Al-Li alloys with as little as 7-10 ppm AMI's was found during studies of creep and overload cracking [9,12]. Examination of fracture surfaces revealed the presence of brittle intergranular and cleavage-like "islands" centred on grain boundary inclusions and surrounded by dimpled regions. These islands, but not adjacent areas, were covered by thin films of sodium and potassium.

Previous work on the effects of AMI content on fracture resistance has been limited to binary Al-Li alloys [13], or has used impact tests [14] which might not fully reveal the embrittling effects of AMI's. The aims of the present work were to: (i) Quantify the effects of Na+K content on the S-L fracture toughness of alloy 2090-T8 for a range of Na+K levels of < 1 to 40 ppm and, (ii) Obtain a better understanding of the mechanisms of embrittlement by determining the effects of temperature and crack-mouth-opening displacement (CMOD) on fracture toughness.

Experimental Procedure

Billets of varying sodium plus potassium contents in the range 0.5 to 40 ppm (Na < 0.5 to 20 ppm and K < 0.05 to 20 ppm) were produced by vacuum refining the melt and adding metallic sodium and a potassium-containing compound. AMI contents of each ingot were obtained using Glow Discharge Mass Spectrometry. The compositional ranges of the 2090 alloy were 1.8-1.9 wt% lithium, 1.9-2.3 wt% copper, 0.09-0.10 wt% zirconium, 0.02-0.09 wt% titanium, 0.04-0.07 wt% iron and 0.01-0.03 wt% silicon. Hydrogen contents were 0.1 to 0.3 ppm. Rectangular sections (60 x 14 mm) were extruded from homogenised and scalped billets which were then solution treated at 530°C for an hour, stretched 4% and aged for 24 hours at 20°C and then 24 hours at 150°C. Only the central 35 mm x 12 mm section of the extrusion was used for preparing specimens. Short-transverse fracture toughness was measured using ASTM E1304-89 chevron-notched short-bar specimens (B = 12.7 mm). Three or four specimens were tested for each condition. Yield strength was measured using tensile specimens prepared in the longitudinal direction.

Results

Optical micrographs of the grain structure of a low-impurity specimen are shown in Figure 1. This structure is typical of the extrusions used in this trial - the material retains the wrought pancake grain structure but is partially recrystallised. However, the grain structure and degree of recrystallisation varied somewhat from position to position within extrusions and from one extrusion to another. For a yield strength range of 428-456 MPa, the average fracture toughness at 20°C increased as the AMI content decreased (Fig. 2). For a given AMI content, the ratio of sodium to potassium appeared to have little effect on toughness, although further work is required on this point. SEM examination of selected fracture surfaces revealed that the number and size of brittle islands produced by liquid metal embrittlement decreased as the Na+K content decreased (Figures 3-5). The number of steps on fracture surfaces and the extent of secondary cracking increased with increasing AMI content, especially for contents > 25 ppm (Fig. 6).

Several kinds of dimpled fracture surfaces were observed in areas between brittle islands (and for specimens with low AMI's with no brittle islands) (Fig. 7). These variations in fracture-surface appearance were clearly associated with variations in grain structure. Relatively large dimples on intergranular and transgranular fracture surfaces were observed for partially recrystallised grain structures, and smaller, shallower dimples were observed on intergranular fractures along the boundaries of unrecrystallised and fully recrystallised grains. The proportions of those fracture modes varied somewhat from specimen to specimen and, for a given AMI content, specimens with greater proportions of partially recrystallised grains (along the fracture path) had higher toughness values. Interestingly, brittle islands were not observed in areas where fracture had occurred along the boundaries of unrecrystallised grains.

For specimens with high AMI contents (20 ppm Na + 17 ppm K), toughness increased with decreasing temperature, and lower CMOD rates resulted in lower toughness over the range +100°C to -100°C (Fig. 8a). The area fraction of brittle islands decreased with decreasing temperature and increasing CMOD rate, as indicated on figure 8(a). For specimens with low AMI's (1 ppm), limited data indicated that (i) there was little or no effect of CMOD rate on toughness, and (ii) decreasing temperature resulted in little or no change over the range 20°C to -60°C and a decrease in toughness (from ~40-45 to 30-35 MPa $\sqrt{m}$) from -60°C to -196°C (Fig. 8b). X-ray photo-electron spectroscopy and Auger-electron spectroscopy of fracture surfaces of specimens with 37 ppm Na+K (produced *in situ* under high vacuum at 20°C) indicated that there were high concentrations of sodium and potassium, and no significant amounts of other impurities.

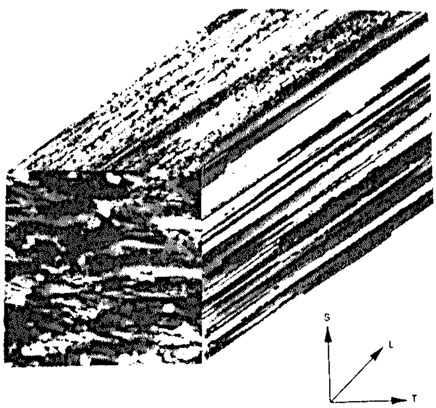

Figure 1. Optical micrographs showing grain structures

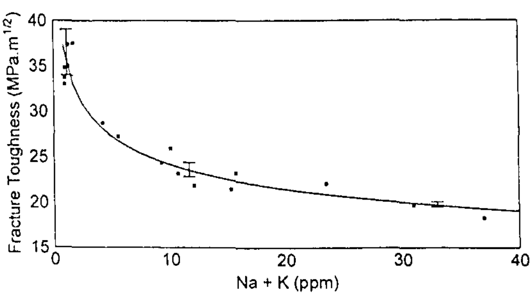

Figure 2. Effect of AMI content on fracture toughness

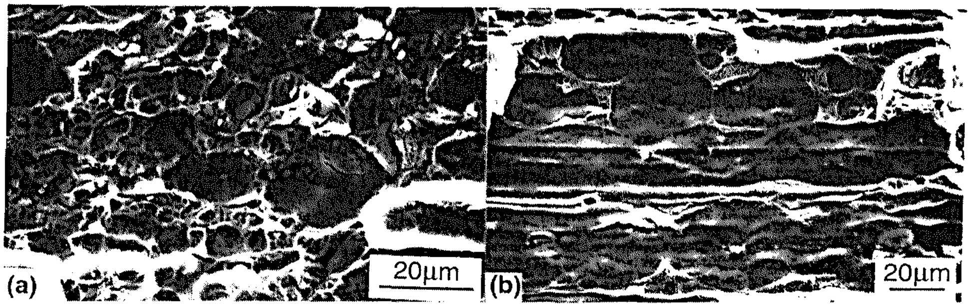

Figure 3. SEM of fracture surfaces of specimens with (a) 5.6 ppm and (b) 31 ppm AMI contents, showing brittle islands surrounded by dimples.

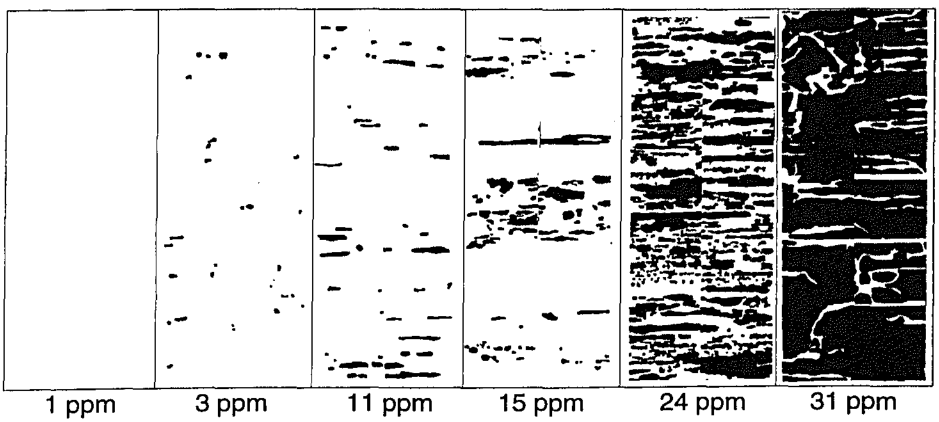

Figure 4. Image-analysis maps of the distribution of brittle islands for specimens with various AMI contents. Brittle islands were not observed at <1ppm AMI.

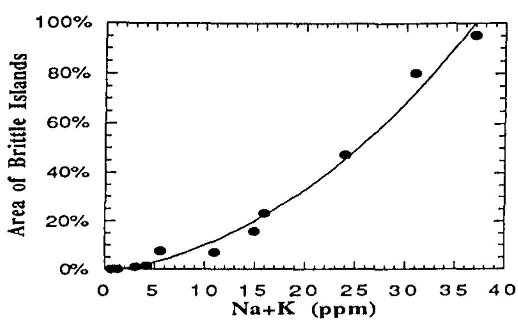

Figure 5. Plot of area fraction of brittle islands versus AMI content.

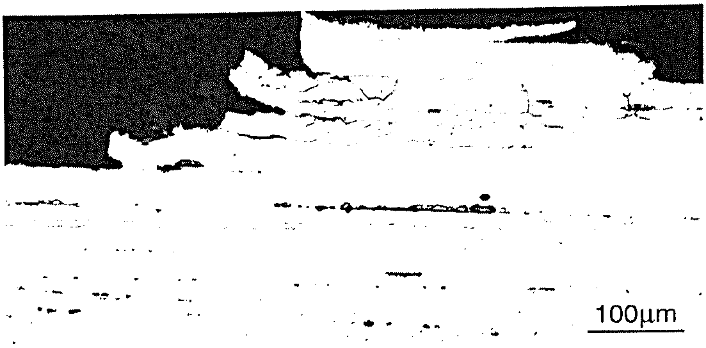

Figure 6. Optical micrograph of section through fractured specimen with 37 ppm AMI content, showing extensive steps and secondary cracking.

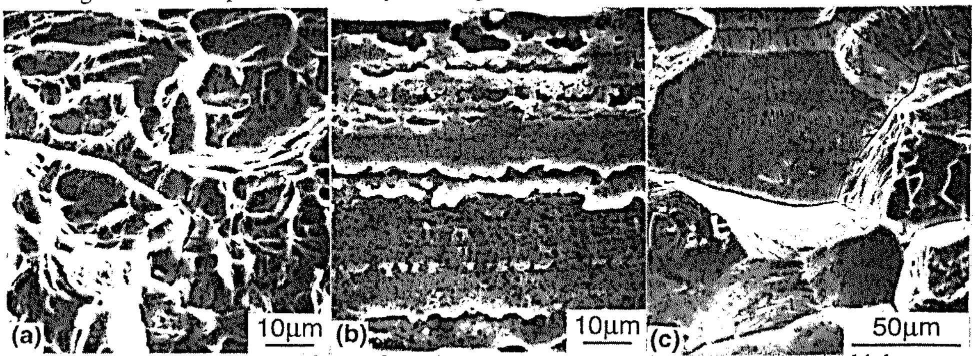

Figure 7. SEM of fracture surfaces of specimens (with <1 ppm Na + K) showing (a) high energy dimpled mode, (b) micro-dimpled mode associated with unrecrystallised grain boundaries, and (c) faceted intergranular mode associated with recrystallised grain boundaries.

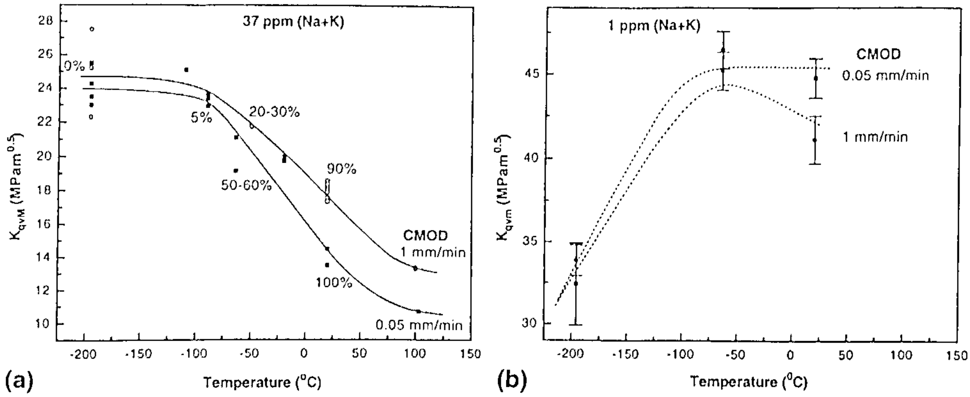

Figure 8. Effects of temperature and crack-mouth-opening displacement rate on fracture toughness for (a) high, and (b) low AMI contents. Area fraction of brittle islands noted in (a).

Discussion

Effect of Alkali Metal Impurity Content on Fracture Toughness

The increasing fracture toughness with decreasing AMI content (for testing at 20°C at CMOD rates of 1 mm/min) (Fig.2) can be explained partly on the basis that the number and volume of liquid AMI phases, and hence the extent of LME, decreases with decreases in AMI content. The volume of liquid phases is important because the supply of embrittling metal atoms to crack tips becomes exhausted after smaller extents of brittle cracking when the volume of liquid phases is smaller [9]. The shape of the plot of toughness versus AMI content (Fig. 2) does not, however, show a good match with the shape of the plot of area fraction of brittle islands versus AMI content (Fig. 5). Thus, there is only a relatively small change in the area fraction of brittle islands over the range 1-10 ppm AMI's but a large change in toughness, while there is a large change in the area fraction of brittle islands over the range 10-37 ppm AMI's but only a small change in toughness.

The small decrease in toughness over the range 10-37 ppm AMI's probably occurs because increasing embrittlement due to larger, more numerous AMI phases is partially offset by toughening effects associated with increasing extents of secondary cracking. Toughening results from the 'shielding' of the main crack tip by secondary cracks and from deformation associated with steps linking secondary cracks (Fig. 6). For material with AMI's in the range 1-10 ppm, the changes in toughness may be due not only to changes in number and volume of AMI phases but also to variations in grain structure. Partially recrystallised grain structures appear to promote a higher energy dimpled fracture mode than unrecrystallised and fully recrystallised grain structures. Quantifying the degree of recrystallisation has not yet been completed and, hence, the exact contribution of grain-structure variations to changes in toughness, especially over the range 1-10 ppm AMI content, has not been established. At higher AMI contents, toughness appears to be dominated by liquid metal impurity effects and the spread in results is correspondingly lower.

Commercial 2090 alloy extrusions produced by conventional casting and processing routes generally have unrecrystallised grain structures and AMI contents in the range 3-10 ppm. The present fractographic observations suggest that lower AMI contents are required to prevent significant reductions in toughness due to LME for material with partially recrystallised grain structures than for material with unrecrystallised grain structures. Greater numbers and volumes of AMI phases are probably present on the boundaries of partially recrystallised grains because AMI's are swept from grain interiors to grain boundaries during grain-boundary migration. The high toughness of vacuum refined alloys (S-L K_V up to 38 MPa$\sqrt{m}$ at yield strengths 440 MPa) is probably therefore due to both low AMI contents and partially recrystallised grain structures.

Effects of Temperature and Crack-Mouth-Opening-Displacement Rate on Toughness

The effects of temperature on fracture toughness of material with high AMI's (Fig. 8a) can be explained along the lines discussed for the effects of AMI content. Thus, decreasing temperature will result in a decrease in the number and volume of embrittling liquid phases and, hence, a decrease in the extent of LME. Bulk melting points of pure sodium, pure potassium, and the Na-K eutectic, are 98, 64, and -12°C, respectively. The fact that toughness increases over the range 120 to -100°C suggests that either considerable undercooling of Na-K phases occurs, embrittlement occurs when the phases are solid or that other impurities such as cesium, which could lower the melting points of Na-K rich phases, are present. Cracking due to solid-metal induced embrittlement (SMIE) occurs at lower rates than that for LME because atoms are transported to crack tips by surface self-diffusion — a slower process than capillary flow of liquid to crack tips [15]. Crack growth at normal CMOD rates (1 mm/min) was rapid and, hence, SMIE is unlikely to contribute to cracking. At slow CMOD rates, however, SMIE could well occur. Lower toughness values are observed at slower CMOD rates probably not only because SMIE contributes to cracking but also because cracks run out of **liquid** phase after smaller increments of crack growth when crack velocities are higher [9]. The occurrence of more secondary cracking at higher crack velocities would also increase toughness.

For material with low AMI's (1 ppm), fracture toughness shows little (if any) change from 20°C to -60°C and decreases with decreasing temperature over the range -60°C to -196°C probably because (i) LME is not involved, (ii) strength increases with decreasing temperature, and (iii) lithium segregation at grain boundaries results in greater embrittlement at lower temperatures. At -196°C the toughness values of the 37ppm and 1ppm AMI samples were about the same. Studies of the effects of temperature and CMOD rate on fracture toughness of material with 'intermediate' AMI contents (3-10 ppm) are in progress, and a detailed discussion of the results will be presented in a future paper.

Conclusions

1. Significant improvements in the room temperature S-L fracture toughness of alloy 2090-T8 can be achieved by vacuum refining to reduce AMI levels to < 2 ppm (compared with the 4-9 ppm typically present in commercially available non vacuum-refined material). For yield strengths ~440 MPa, chevron-notch fracture toughness values as high as 38 MPa.$\sqrt{m}$ were observed.

2. Increasing AMI's above 3-4 ppm results in increasing extents of brittle intergranular fracture and decreasing fracture toughness due to an increasing number and volume of embrittling liquid Na-K rich phases. Brittle islands were not observed for material with < 1-2 ppm AMI's.

3. Fracture toughness increased with decreasing temperature (over the range 100 to -100°C) for material with high AMI contents, but decreased with decreasing temperature for material with low AMI contents.

4. Fracture toughness decreased with decreasing crack-mouth-opening displacement (CMOD) rates (over the range 0.05 to 1 mm/min) for material with high AMI contents, but there was no effect of CMOD rate on toughness for material with low AMI contents.

5. The effects of temperature and CMOD rates on toughness can be explained in terms of the mechanisms and kinetics of liquid (and solid) metal induced embrittlement.

6. The variation of fracture toughness with AMI content (and proportion of brittle islands) is complicated by varying extents of secondary cracking, and by variations in the proportions of other fracture modes which depend on the degree of recrystallisation. Partially recrystallised grain structures promoted a high-energy fracture mode.

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