Chapter 4

PLANE STRAIN FRACTURE TOUGHNESS OF Al–6Mg ALLOY DOPED WITH SCANDIUM

4.1 Objective

Toughness of aluminium alloys is known to be appreciably influenced by the second phase particles present in the microstructure [1, 2]. The fracture toughness of aluminium alloys is equally influenced by the size and shape of grains. In majority of aluminium alloys coarse insoluble inter metallic compounds of impurity elements like iron and silicon are inherently present [3]. Reportedly, fracture of coarse insolubles leads to deterioration of fracture toughness with increasing yield strength of age-hardenable alloys [4]. Commonly present inherent dispersoids exert complex effect on the fracture toughness of aluminium alloys. They tend to
enhance fracture toughness when present as fine particles which inhibit recrystallisation and grain growth [5]. Sub microscopic dispersoids on the other hand adversely affect the fracture toughness by way of creating micro pores due to decohesion at the particle-matrix interfaces [6]. Plain strain fracture toughness of aluminium alloys is greatly improved by the non-recrystallized grains obtained due to aluminides of impurity elements inherent to the system [7]. Fine dispersoids also enhance homogeneous deformation of the matrix through Orowan bypassing mechanism, thereby improving fracture toughness. Fine coherent ageing precipitates in age hardenable alloys significantly influence the fracture toughness and lead to slip band formation during deformation, thereby deteriorating the fracture toughness of the alloy [8]. Moreover, a wide precipitate free zone near the grain boundaries aids in crack formation at the grain boundary triple points due to grain boundary slip [9]. In a number of previous works it has been demonstrated that the extent of dislocation pile-up is reduced by grain refinement and this leads to enhancement of fracture toughness [10]. Minor addition of scandium in aluminium alloys can serve to refine grains by way of forming \( \text{Al}_3\text{Sc} \) particles within the solidifying melt due primarily to the strong interaction between aluminium and scandium atoms [11–13]. These \( \text{Al}_3\text{Sc} \) particles also act as dispersoids controlling the final grain structure of the alloys. Although minor additions have influenced the fracture toughness of age-hardenable aluminium alloys [14], the plain strain fracture toughness behaviour of Al–6Mg alloys due to dispersion of \( \text{Al}_3\text{Sc} \) in the matrix is not well documented in literatures. Therefore, the present investigation is carried out with the objective to understand the influence of inherent dispersoids and other micro structural features on the plain strain fracture toughness of Al–6Mg
4.2 Experimental Procedures

The experimental aluminium-magnesium alloys were prepared as detailed in the previous chapter 3. The chemical composition of the alloys under investigation is given in Table 4.1.

1) Alloy 1: Al – 6 wt.% Mg
2) Alloy 2: Al – 6 wt.% Mg – 0.2wt.%Sc
3) Alloy 3: Al – 6 wt.% Mg – 0.6wt.%Sc

4.3 Results

4.3.1 Microstructures

The optical microstructure of the cast Al – 6Mg alloy shows the presence of $\alpha + \beta$ eutectic within the inter-dendritic spaces of primary [15]. $\beta$ – phase is known to be comprised of aluminides of magnesium and other impurity elements [16]. Figure 4.1 shows the microstructure at a plane just below the fractured surface of the FT specimen of binary alloy annealed at 200°C. A good amount of second phase insolubles is present in the micro structure. The fractograph of the above sample shows lamellar tearing without ev-
idence of appreciable plastic deformation (Figure 4.2). When the base alloy annealed at 300°C is viewed at higher magnification, cracks are seen to have originated from second phase insolubles and a grain-boundary crack of size about 10 \( \mu \text{m} \) is noted in the same photo–micrograph (Figure 4.3). The fractographs of the base alloy annealed at higher temperatures have given evidence of micro void coalescence. Fine particles are also seen to be seated at the dimple base. Long cracks are due to the fracture of second phase particles present in the annealed alloys (Figure 4.4). The microstructure of alloy 2 containing 0.2 wt.% Sc exhibits grain boundary segregation along with intragranular second phase particles. Fracture of grain boundary phases is noted in microstructure of the alloy 2 annealed at 300°C (Figure 4.5). Moreover, the evidence of micro cracks at the grain interiors is discernible in the same micrograph. Extensive planar slip band (PSB) formation is evidenced in the same photograph. Similar observation could be made for alloy 2 aged at 400°C.

The fractograph of alloy 2 aged at 200°C shows grain boundary separation (Figure 4.6). The presence of shear ridges is indicative of over–load failure. Crack branching as noted in the above fractograph is supposed to be due to the presence of brittle constituents in its microstructure. The fractograph of peak aged alloy 2 tested for fracture toughness gives evidence of micropores within the material. It seems, that these micropores act as the source of the cracks (Figure 4.7).

The microstructure beneath the fracture surface of FT sample of cast alloy 3 (∼0.6% Sc) once again gives the evidence of grain boundary segregation of small aluminide particles (Figure 4.8). However, a small amount of intra–granular particles of size ∼ 0.5 \( \mu \text{m} \) is also seen in the same figure. The fractograph of this
4.3. RESULTS

Figure 4.1: SEM image of Al–6Mg alloy annealed at 200°C for 1 h

Figure 4.2: SEM image of Al–6Mg alloy annealed at 200°C for 1 h
Figure 4.3: SEM image of Al–6Mg alloy annealed at 300°C for 1 h

Figure 4.4: SEM image of Al–6Mg alloy annealed at 400°C for 1 h
4.3. RESULTS

Figure 4.5: SEM image of Al–6Mg–0.2Sc alloy annealed at 300°C for 1 h

Figure 4.6: SEM image of Al–6Mg–0.2Sc alloy annealed at 200°C for 1 h (Fracture Surface)
Figure 4.7: SEM image of Al–6Mg–0.2Sc alloy annealed at 300°C for 1 h (Fracture Surface)

Figure 4.8: SEM image of Al–6Mg–0.6Sc alloy (As cast)
4.3. RESULTS

cast alloy shows grain boundary lamination and the failure is seen to be typically inter–crystalline in nature (Figure 4.9).

The subsurface microstructure from FT specimens of alloy 3 annealed at 200°C has delineated the formation of slip bands (Figure 4.10). While cracking at slip bands is quite discernible in the above microstructure, microcracks emanating from the second phase insolubles are also noticeable. It is observed from the fractograph of the above alloy that the cracks have moved both intra as well as intergranularly before failure has taken place (Figure 4.11). Under peak aged condition, slip band formation has been quite extensive (Figure 4.12). The cracks are seen to have formed due to grain boundary slipping and void formation at grain boundary triple points.

From the Differential Thermal Analysis plots of alloys under in-
vestigation it is observed that the area under the DTA peak of alloy 2 is lower than that for the alloy 3 (Figure 4.13). The activation energy of precipitation reaction is found to be 180 KJ/mole in either case. Thus the area under the curves would fairly give the ratio of the amount of precipitates formed in each of the two alloys. It appears that higher amount of precipitate is formed in alloy 3.

4.3.2 Fracture toughness

Variation of plane strain fracture toughness ($K_{IC}$) of the experimental alloys against isochronal annealing temperature is shown in Figure 4.14. It is evident from Figure 4.14 that the fracture toughness of binary Al–6Mg alloy increases marginally with increase in the ageing temperature ($\sim$4 MPam$^{-1/2}$). However $K_{IC}$ of
4.3. RESULTS

Figure 4.11: SEM image of Al–6Mg–0.6Sc alloy annealed at 200°C for 1 h (fracture surface)

alloy 2 with 0.2 wt% scandium is found to be quite sensitive to ageing. Figure 4.14 demonstrates the enhancement of $K_{IC}$ of alloy 2 with a progressive increase in ageing temperature up to 300°C. Further ageing beyond this temperature has led to a reduction in its fracture toughness value. An overall increase of fracture toughness by about 15 MPam$^{-1/2}$ has been recorded for alloy 2 aged at 300°C. On the contrary, alloy 3 containing 0.6 wt% Sc exhibits a different trend of variation of fracture toughness with temperature of isochronal ageing. The $K_{IC}$ of alloy 3 decreases with increasing ageing temperature and attains its minimum value at an ageing temperature of 300°C. Notably, a further increase in ageing temperature causes a significant improvement in the fracture toughness of the alloy ($\sim$ 35 MPam$^{-1/2}$).

The isochronal ageing curves of the concerned alloys are shown in Figure 4.15. It is revealed that the ageing response of alloy 1 is
Figure 4.12: SEM image of Al–6Mg–0.6Sc alloy annealed at 300°C for 1 h

Figure 4.13: DTA curves alloy 2 and 3
Figure 4.14: Fracture Toughness vs. Temperature of alloys

very low. A small rise (~15 VPN) in hardness may be noted after ageing the binary alloy at 300°C; however, ageing beyond this temperature leads to a lowering of its hardness value. On the other hand, a prominent age-hardening effect is exhibited by alloy 2 with its peak ageing hardness occurring at 300°C. It is to be noted from Figures 14 and 15 that, the pattern of variation of hardness and fracture toughness of alloy 2 with isochronal ageing temperature is almost the same. The age-hardening behaviour of alloy 3 is similar to that of alloy 2. A strong ageing response of alloy 3 is evidenced by a rise of hardness value to the tune of 40 VPN at the peak-aged condition. However, in contrast with alloy 2, the fracture toughness of alloy 3 is seen to decrease with increasing ageing temperature up to 300°C. In fact, fracture toughness of alloy 3 becomes minimum after ageing at a temperature of 300°C which is the peak age hardening temperature of this alloy.
Fracture toughness values of each of the experimental alloys are plotted against the hardness values obtained after various ageing treatments (Figure 4.16). It appears from the figure that the fracture toughness property of alloy 1 and alloy 3 varies in a similar manner with the corresponding micro hardness values. Fracture toughness value, $K_{IC}$, is seen to increase with increasing microhardness value up to a certain extent; it then records a decline with increasing microhardness, till a certain value of microhardness is reached, beyond which it starts rising once again with increasing microhardness. Alloy 2 is, however, different in the sense that its fracture toughness initially decreases with increasing microhardness up to a certain value. This is then followed by a continuous increase in $K_{IC}$ with microhardness values. It is clear from Figure 4.16, that the fracture toughness behaviour of the experimental
alloys cannot be related to the variation of micro hardness of the alloys with one to one correspondence because of strong sensitivities of fracture toughness to the microstructures. In other words, Figure 4.16 is indicative of the fact that all the causes, which are responsible for enhancement of micro hardness values of the present alloys, do not necessarily act in the same way while influencing the fracture toughness property.

### 4.4 Discussions

#### 4.4.1 Microstructures

The microstructure of Al–6Mg alloy in its cast condition [17] has been found to be similar to that of previous observation [16]. Number density and distribution of interdendritic eutectic ($\alpha + \beta$) depend upon the solidification speed which is known to be signifi-
cantly influenced by scandium addition [18]. The microstructure of the alloys after annealing has shown grain boundary segregation. This is due to the fact that the existing eutectics go in solution at the annealing temperatures and the soluble dispersoids are precipitated at grain boundaries during slow cooling from the annealing temperature. The coarse insolubles and dispersoids present in the base alloy annealed at 200°C (Figure 4.1) are seemingly responsible for giving rise to notch effects which lead to easier formation of cracks during fatigue loading. Due to annealing at high temperature, the growth of second phase particles takes place, resulting in the presence of different sized particles from 0.5 µm to 3.5 µm. These second phase particles are composed of different kinds of aluminides and their size increases with increasing annealing temperature.

The lamellar tearing in fractograph of Fracture Toughness (FT) sample aged at 400°C (Figure 4.4) owes its origin to overload failure after fatigue cracking. Fatigue loading used for pre-cracking the FT samples has created alternately stressed regions within the material due to continuous flow of stress wave. Strain hardening due to Mg interferes with these stressed regions and leads to fracture. As stated, annealing at a high temperature (400°C) leads to appreciable precipitation at the grain boundary. This results in wider precipitate free zones (PFZ).

Delamination along grain boundary (Figure 4.4) is thought to be related to PFZ in the alloy annealed at 400°C as it is stated that wider PFZ facilitates dislocation pile-ups during fatigue loading. These pile-ups lead to the formation of microcracks, which upon loading, propagates and branches out if precipitates are encountered during their progress.

Fracture through separation of grain boundaries stems from the
occurrence of grain boundary phases. Upon increasing the annealing temperature, second phase insolubles dissolve (Figure 4.5). At the same time, Al₃Sc precipitates out at the grain interiors. This leads to toughening of the matrix and so, the material fractures intergranularly (Figure 4.7).

Scandium is known to enhance solidification speed in Al–6Mg alloy [19]. Higher scandium content in alloy 3 has led to structural fineness alongwith diminution in second phase constituents. However, it is known that due to strong interaction between aluminium and scandium atoms, the solidified alloy forms extremely fine Al–Sc atom clusters [11]. The presence of grain boundary phase in Figure 4.8 together with a tough matrix accounts for intergranular crack propagation as noticed in the fractograph of cast alloy 3 (Figure 4.9). When this alloy is annealed at 200°C, precipitates of Al₃Sc form on the pre-existing Al–Sc segregations [11]. Alongwith this, second phase dispersoids consisting of aluminide of magnesium and other impurity elements are also formed. Upon loading the samples, the dislocations come across the fine coherent precipitates in abundance and can move through the active slip planes by cutting mechanism. This results in the formation of persistent slip bands (PSB) as noted in Figure 4.10. In this connection, it may be noted that among all the resolved shear stresses which correspond to every possible slip direction on every possible slip planes, there must be a single resolved shear stress whose magnitude exceeds all others. Crystals oriented so will deform initially by slip on only one set of parallel slip planes. Moreover, most aluminium alloys are characterised by pronounced cube texture and thus, the grains have preferred orientation. Hence, the favourable slip system as described above remains parallel across the grains. This is why the PSBs are seen to have a single orientation in Fig-
ures 4.5, 4.8 and 4.10. These PSBs are very prone to intragranular cracking, which is quite prominent in Figure 4.10. The corresponding fractograph in Figure 4.11 also substantiates the above view. When annealed at 300°C, extensive precipitation of coherent Al$_3$Sc takes place. These are responsible for slip band formation in the deformed alloy (Figure 4.12). Simultaneous with the formation of Al$_3$Sc precipitates, localized enrichment of magnesium takes place. The magnesium atoms in the near vicinity of grain boundary can easily migrate to grain boundaries at this temperature. It may be noted that the temperature of $\beta$–phase formation is around 327°C in alloy 3 [15]. Extensive precipitate formation at grain boundary leads to a wider PFZ near grain boundaries (Figure 4.12). Thus, dislocations pile up at these PFZs, thereby causing grain boundary slipping [9]. This gives rise to cracking at the triple points of the grain boundaries and the material fails intergranularly.

### 4.4.2 Fracture toughness (FT)

Fracture toughness of high strength aluminium alloys is largely determined by the coarse intermetallics, since the cracks do not easily nucleate within the matrix of the above high strength alloys. Fracture of coarse intermetallics without much plastic deformation quite often gives rise to the formation of intragranular cracks, which tends to lower the fracture toughness of the alloys. Under such situation, FT is seen to diminish with increasing yield stress, owing to the reduction in critical flaw size [4]. In case of alloy 1, FT does not change much with the ageing temperature, although the ageing curve of alloy 1 records a small increase in hardness value at 300°C followed by a decrease. It is known that Al–Mg alloy does not age-harden and therefore, hardness increment as ob-
4.4. DISCUSSIONS

served in the present case may be explained by the dissolution of pre-existing \( \beta \)-phase which increases the amount of magnesium in solution in aluminium. Magnesium increases strain hardening in aluminium-magnesium alloy [20] and hence, improves fracture toughness. This is reflected in the marginal improvement of FT of Alloy 1 beyond an annealing temperature of 300°C. However, during this time, the ageing hardness is seen to decrease.

It may be noted that FT vs. ageing temperature curve of alloy 2 is quite similar to its age-hardening curves (Figures 4.14 and 4.15). That the FT of alloy 2 is higher than the base alloy at all ageing temperatures seems to follow from the grain-refining effect of Sc. Fine particles of Al\(_3\)Sc stabilises substructure. Because of small degree of misorientation across the sub-boundaries, the dislocation pile-ups at those get reduced. This leads to improvement in fracture toughness. The coherent precipitates of Al\(_3\)Sc envisage cutting mechanism to be operative during deformation. As a result, a difference in strength arises between the activated and non-activated slip planes. Although yield strength is increased, due to fine particle size, the deformation mode of the alloy promotes the formation of planar slip bands and dislocation pile-ups at the grain boundaries. It may therefore be anticipated that the FT should decrease with increasing ageing temperature. But in the present study, toughening of the matrix by fine coherent precipitate of Al\(_3\)Sc has made crack nucleation difficult. Stabilized sub-structure also acts similarly. The cracks cannot move through the toughened matrix. Since Sc-content is small in alloy 2, the amount of precipitates is also small. Therefore, slip band formation is not as extensive as to damage the material to any significant extent. Also grain-refinement helps to improve FT as it does not allow grainboundary slipping at the triple points. As mentioned earlier, addition of Sc
leads to the reduction of second phase constituents in terms of size and amount. Because of structural refinement, second phase particles virtually act as fine dispersoids to aid in slip–dispersion. Thus, slip band formation is counteracted. Cracking is to initiate only at the grain-boundaries by the fracture of grainboundary phases. However, amount of grainboundary phases is also reduced by Sc addition. Due to low Sc-level and finer grainsize, the PFZ width will be small. All the above stated factors are favourable for improving FT. Over-ageing beyond 300°C leads to particle coarsening. Along with this, fresh precipitates of Mg-aluminide also appear at the grain-boundsaries. With increase in the size and amount of grainboundary precipitates due to ageing at higher temperature, the width of PFZ increases. Shear within this PFZ leads to crack-nucleation at grain boundary triple points and so FT goes down in the overaged condition of alloy 2.

Fracture toughness of alloy 3 is found to be minimum at 300°C at which the ageing hardness value is found to be maximum. Owing to high Sc content in alloy 3, higher density of precipitates is realized at 300°C. This results in a higher degree of inhomogeneous deformation promoting planar slip. Excessive slip band formation leads to considerable deterioration in FT. At 300°C, the precipitate density is maximized and hence, FT is seen to be minimum. This behaviour is different from alloy 2 because the precipitate density in alloy 2 has not reached that extent which makes it structurally fatal. Although particle coarsening above 300°C enables fracture toughness of alloy 3 to improve due to homogenous deformation, the yield strength of the alloy goes down. Again, initial rise in fracture toughness in alloy 3 as noted in Figure 4.14 may be attributed to reduction in size and number density of second phase insolubles, which lead to prevention of slip band formation and
4.4. DISCUSSIONS

thus, counteracts planar slip.

The increase in fracture toughness of alloy 1 at low values of hardness in Figure 4.16 is seemingly due to the dissolution of insolubles, which leads to higher magnesium content in the alloy. Thus, solid solution strengthening is enhanced. The increase in Mg-content promotes strain hardening of the matrix, thereby enhancing fracture toughness. The lowering of fracture toughness beyond a definite hardness value stems from the fracture of coarse insoluble particles as because the matrix is not strong enough to inhibit crack nucleation. The final rise in FT with hardness value is rather small. Though the initial rise in hardness is ascribed to the increasing solid solution hardening in alloy 1 as stated earlier, the appearance of small amounts of ageing precipitate might also have minor role in increasing the hardness value of alloy 3. The ageing precipitates toughen the matrix and make the crack nucleation difficult. Further increase in hardness corresponds to appearance of some kind of aluminide precipitates which fracture and deteriorate fracture toughness. A still higher value is a manifestation of structural refinement. This promotes homogenous deformation in the alloy, and hence, fracture toughness is improved. In case of alloy 2, the increase in hardness has led to higher FT due mainly to matrix toughening accompanied by grain refinement (Figure 4.16). Initial drop in FT at low hardness level may be attributed to the fact that at this level of hardness, matrix toughening is not good enough in general. The increase in hardness is more due to the presence of second phase insolubles whose fracture has initiated cracks and lowered FT. This is particularly true as it was reported earlier that in many aluminium alloys, fracture toughness decreased with increasing yield stress. This occurs due to the lowering of critical flaw length and initiation of failure by cracking of insolubles.
4.5 Conclusions

i) Minor addition of scandium significantly improves the plane strain fracture toughness of Al–6Mg alloy by way of modifying the structure of dispersoids in the microstructures.

ii) A higher density of ageing precipitates due to high scandium content in the alloy promotes slip band formation and hence deteriorates fracture toughness.

iii) However, promoting homogenous deformation through particle coarsening improves the fracture toughness of Al–6Mg (Sc) alloy.

References


REFERENCES


