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The Effect of Slip Mode on Brittle Intergranular Fracture of Al Alloys

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The slip-mode of age-hardened aluminium alloys depends on precipitate size and coherency, grain size, temperature, strain and other factors. There is a widespread view that a planar-slip mode, favoured by small, coherent, easily sheared matrix precipitates, promotes brittle intergranular fracture. A review of previous work, along with some new observations, suggest that there is little or no correlation between slip mode and the tendency for brittle intergranular fracture in Al alloys, and that other factors, such as grain-boundary segregation and grain-boundary microstructure, are much more important.

Keywords: *Slip-Mode, Fracture-Mode, Slip-Planarity, Brittle Intergranular Fracture, Grain-Boundary Segregation.*

1. Introduction

Slip is often characterised as planar or wavy, and both types can occur on a coarse or fine scale, depending on the material, stacking fault energy, lattice ordering of solute, precipitate coherency, grain size, and other variables. Slip characteristics may, of course, lie between the extremes of coarse planar slip and fine homogeneous slip, and can vary from one grain to another in the same material [1]. Quantifying slip characteristics in terms of slip-band waviness, slip-band width and spacing, and heights of slip steps on surfaces is difficult, and is not often attempted [2]. Coarse planar slip (with widely spaced, localised slip bands) occurs especially in underaged Al alloys, where precipitates are coherent and readily sheared by dislocations [3,4]. Once precipitates along a band are sheared, subsequent slip is facilitated. Fine, homogeneous slip tends to occur in overaged alloys when precipitates are incoherent and are by-passed by dislocations.

It is often suggested that coarse planar slip leads to brittle intergranular fracture, due to dislocations in slip bands piling up at grain boundaries so that locally high stress concentrations are produced [5-12]. This hypothesis has been proposed for Al-Li alloys in particular since these alloys are especially prone to brittle intergranular fracture (especially for short-transverse orientations of plate), and exhibit more marked planar-slip characteristics than most other alloys due to the presence of easily sheared, coherent δ' (Al_3Li) age-hardening precipitates. However, an alternative hypothesis for the propensity for brittle intergranular fracture in Al-Li alloys, based on Li segregation at grain boundaries, has also been proposed [13-17]. The presence of grain-boundary precipitates (GBPs) and soft precipitate-free-zones (PFZs) also needs to be considered.

In the present paper, the planar slip hypothesis vis-à-vis the segregation hypothesis (and other considerations) for brittle intergranular fracture in a number of Al alloys are summarised, drawing on studies in the literature and on recent, previously unpublished work. Comparisons of the fracture behaviour of Al alloys with other materials are made where appropriate. It appears that, contrary to widespread views, there is generally little correlation between slip mode and the occurrence of brittle intergranular fracture, and that grain-boundary microstructure/segregation is much more important.

2. Ductile-to-Brittle Fracture Transitions with Decreasing Temperature in Al-Li alloys

Remarkably sharp ductile transgranular fracture to brittle intergranular fracture transitions have been observed with decreasing temperature in very underaged Al-Li-Cu-Mg-Zr (8090) alloys, with the ductile to brittle transition temperature (DBTT) depending on the ageing treatment (Fig.1a)[15,16]. For these very underaged alloys, there were no GBPs or PFZs present to complicate matters (Fig.1b), but lithium segregation has been observed by parallel-electron-energy-loss spectroscopy [14]. The slip characteristics on specimen surfaces were similar in ductile and brittle temperature regimes although, if anything, slip was somewhat finer at -196°C than at 20°C (Fig. 2a). TEM observations of dislocation arrangements in specimen interiors also suggested that slip was somewhat more homogeneous at -196°C than at 20°C (Fig.2b), as has been found in other studies [18-20]. More importantly, slip characteristics would not be expected to change significantly over the 20°C-50°C temperature range corresponding to the transition from ductile to brittle behaviour.

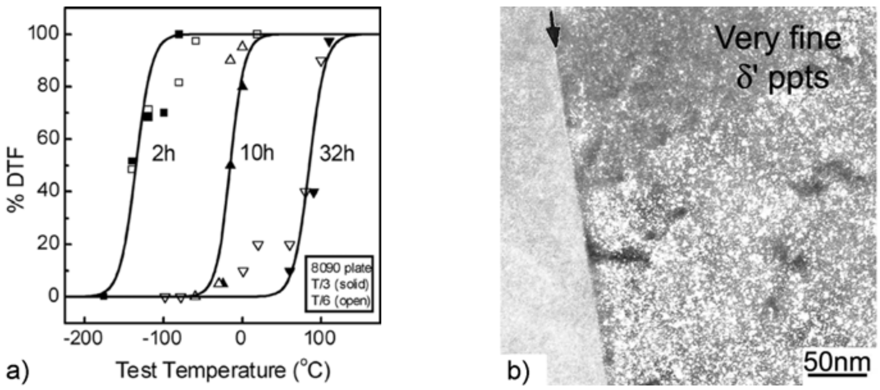

Fig. 1 (a) Plot of the percentage ductile transgranular fracture (DTF) (where 0% represents completely brittle intergranular fracture) versus test temperature for an 8090 alloy plate (S-L crack-plane orientation) aged for 2, 10, and 32h at 60°C, showing sharp ductile-to-brittle transitions with decreasing temperature [15], (b) TEM of typical microstructure of very underaged 8090 alloys showing very fine δ' precipitates with no GBPs or PFZ. [16]

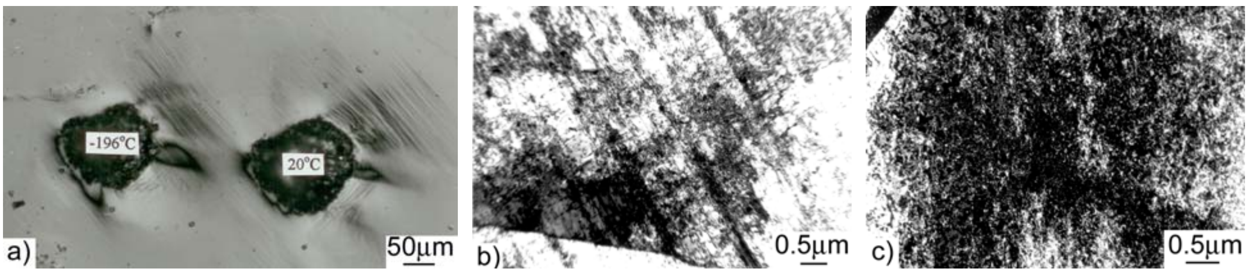

Fig. 2 8090 alloy aged 2h at 60°C (a) Optical micrograph showing slip lines around hardness impressions made in the same grain at -196°C and 20°C and (b),(c) TEM of dislocation arrangements produced after 9% strain at 20°C and -196°C, respectively, showing somewhat more homogeneous slip at -196°C. [15]

The planar-slip hypothesis for explaining brittle intergranular fracture in Al-Li alloys is obviously not consistent with the above observations, which can best be explained in terms of a 2-D grain-boundary structural phase change that occurs with decreasing temperature when lithium segregation is present [16]. The higher DBTT in alloys aged for longer times (resulting in more lithium segregation at grain boundaries) probably occurs because the transition to a brittle grain-boundary structure occurs at higher temperatures when lithium-segregation levels are higher.

The occurrence of 2-D phase changes at grain boundaries (and surfaces) is well established [21-25], but the structural changes (to polyhedral structural units) which result in embrittlement by facilitating decohesion across grain boundaries or by facilitating dislocation emission from crack tips, are not well understood [16].

3. Effects of Re-aging on Intergranular Fracture and Slip Mode in 8090

For an Al-Li-Cu-Mg (8090 T-8771) plate with an elongated grain structure (Fig.3a), there are fine dispersions of δ' precipitates and PFZs and GBPs at grain boundaries after ageing to near peak hardness (32h at 170°C) (Fig.3b). Fracture at 20°C for the short-transverse orientation produced a mixed brittle-intergranular fracture/dimpled-intergranular fracture mode (Fig.3c). Re-ageing specimens for 5min at 200°C doubled the room-temperature fracture toughness (Fig.4a) and eliminated the brittle-intergranular-fracture mode, so that just dimpled intergranular fractures were observed [26]. This re-ageing treatment produced only a small decrease in strength and little change in microstructure except for some reversion of the finest δ' precipitates and, as would be expected, did not result in a significant change in the degree of slip planarity (Fig.4b).

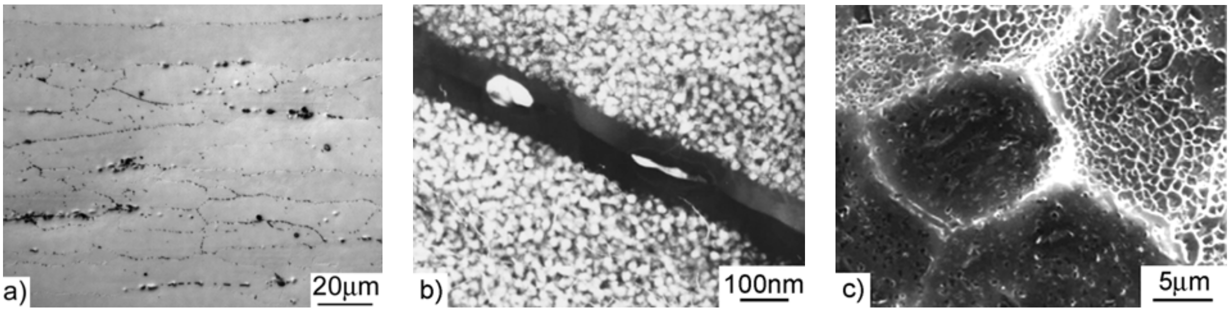

Fig. 3 8090 near peak-aged alloy (a) optical micrograph showing grain structure and constituent particles [27], b) TEM showing GBPs and PFZ, and (c) SEM of fracture surface produced at 20°C showing dimpled intergranular and brittle intergranular facets for S-L specimens [15].

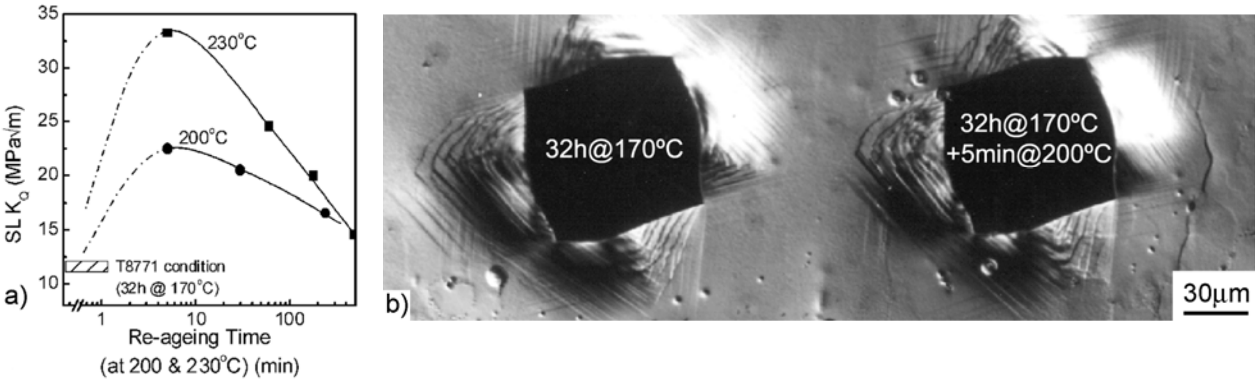

Fig. 4(a) Plot of room-temperature S-L fracture toughness versus re-ageing time at 200°C and 230°C for an 8090-T8771 alloy plate (initially aged 32h at 170°C) showing large increases in toughness after very short re-ageing times [13], and (b) optical micrograph of slip around hardness impressions for the T8771 condition and re-aged condition in the same grain, showing no significant difference in slip characteristics [14].

These observations are also not consistent with the planar-slip hypothesis for brittle intergranular fracture, and can best be explained in terms of embrittlement by grain-boundary 2-D lithium-rich phases (in the T8771 condition) and their reversion during subsequent ageing for short times at somewhat higher temperatures. Longer re-ageing times allow the re-establishment of grain-boundary lithium-rich phases (and growth of GBPs) so that fracture toughness decreases along with the re-appearance of brittle intergranular fracture. Without grain-boundary embrittlement due to segregation, completely dimpled intergranular fractures would be expected since the PFZ widths and GBP spacings were such that strain-localisation in PFZs and around GBPs should result in void nucleation and growth producing well-defined dimples, as observed for the re-aged condition.

The variation in fracture mode from one grain to another (Fig.3c), or along the same (curved) grain boundary, is probably associated with variations in grain-boundary misorientation/structure, resulting in different concentrations of lithium segregation at grain boundaries – a hypothesis that is supported by (i) observations that 8090 material with strong textures (where low-angle grain boundaries predominate) exhibit less brittle intergranular fracture than material with weaker textures, and (ii) expectations based on observations in other materials that segregation levels would be lower for low-angle grain boundaries than high-angle grain boundaries [15].

4. Intergranular Fracture and Slip Mode in a 7079 Alloy

For peak-aged Al-Zn-Mg-Cu (7079-T651) alloys, slip is relatively homogeneous compared with near-peak-aged Al-Li-Cu-Mg (8090-T8771) alloys since the former is strengthened by semi-coherent η' precipitates, which are not as easily sheared by dislocations as the coherent δ' precipitates in 8090. There are also coarse, incoherent Mn/Cr based dispersoid particles in the 7079 alloy which helps disperse slip – unlike the small, coherent Zr-based dispersoids in 8090. Despite the fairly homogeneous slip mode, a predominantly brittle intergranular fracture mode was observed at 20°C and -196°C for the 7079 T-651 alloy [28]. Like the 8090-T8771 alloy, there was generally little sign of plasticity/dimples on many grain-boundary facets despite the presence of PFZs and GBPs (Fig.5). Thus, segregation of Mg and Zn is probably responsible for brittle intergranular fracture of the 7079-T651 alloy. Segregation of these elements has been observed [29-31], and theory predicts that, like Li, they should embrittle aluminium alloys [32].

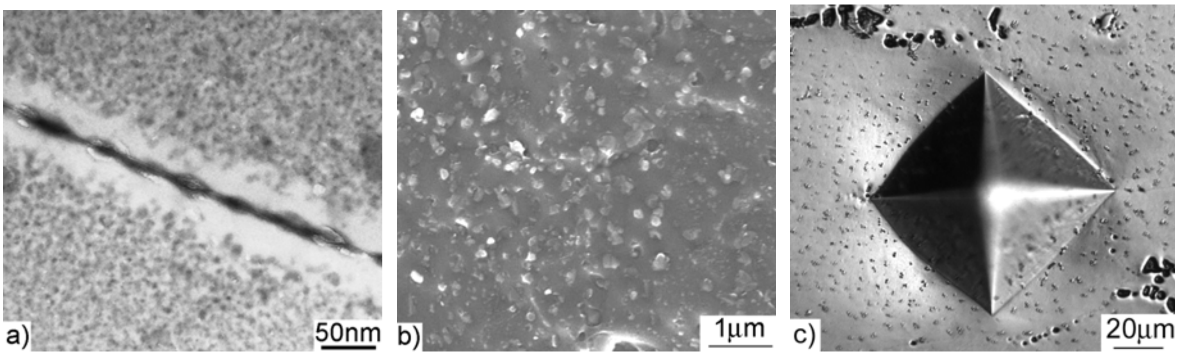

Fig. 5 7079 T651 alloy (a) TEM of grain-boundary microstructure, (b) SEM of brittle intergranular fracture for S-L specimens tested at 20°C [25], and (c) hardness impression on polished surface showing absence of slip lines indicating that slip was much finer/more homogeneous than in the 8090 alloy (cf. Fig. 4b) [27].

5. Fracture Modes and Slip Mode in 5091 (Al-Li-Mg-C-O) alloy

The 5091 alloy (Al-4.11Mg-1.29Li-1.17C-0.41O wt%) has a fine grain size (~500nm) as a result of processing by severe plastic deformation (mechanical-alloying) and the presence of fine dispersions of oxides and carbides, which pin grain boundaries and prevent grain growth during subsequent heat treatment [33]. Very fine δ' precipitates are also present, but the alloy does not undergo significant age-hardening. As would be expected, due to the oxide and carbide dispersions plus the fine grain size, deformation at 20°C and -196°C produces fine, fairly homogeneous slip. There is, however, a transition from dimpled transgranular fracture to brittle intergranular fracture with decreasing temperature (Fig.6). Thus, this is another example where brittle intergranular fracture occurs in the absence of coarse planar slip, and probably involves segregation of Li plus Mg to grain boundaries and a 2-D phase transition with decreasing temperature (as for the very underaged 8090 alloy where relatively planar slip occurred) – again suggesting that slip-mode is incidental in this regard.

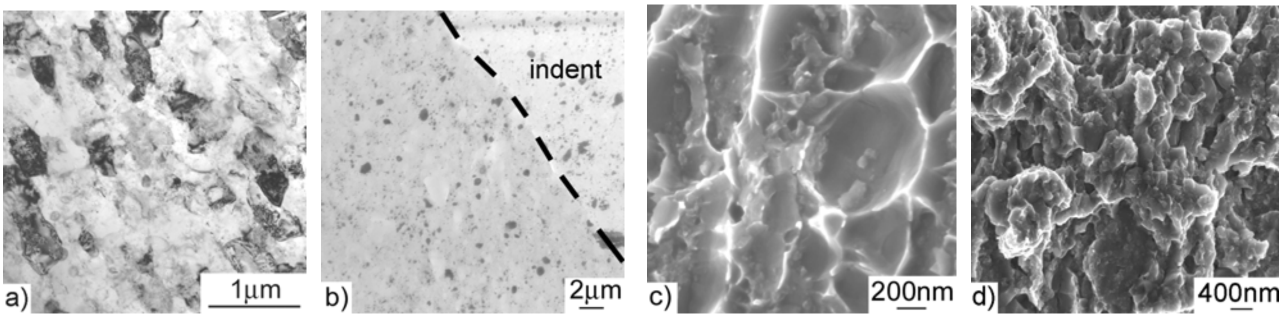

Fig. 6 Mechanically alloyed Al-Mg-Li-C-O (5091) alloy: (a) TEM of microstructure showing ultra-fine grain structure, (b) optical micrograph of electropolished surface showing coarse oxides/carbides, and absence of slip lines at the edge of a hardness impression at 20°C, and (c),(d) SEM of fracture surface produced at 20°C and -196°C showing ductile transgranular fracture and brittle intergranular fracture, respectively [27].

6. Effects of Microstructural Refinement of an 8090 Alloy on Fracture and Slip Modes

Friction-stir processing (FSP) of an initially coarse-grained 8090 Al-Li-Cu-Mg alloy plate (immersed in an anti-freeze (<20°C) bath before and during processing) resulted in an equi-axed grain size ~500nm, with mostly high angle grain boundaries (Fig. 7a), as a result of severe plastic deformation and dynamic recrystallisation [27]. In addition, large constituent particles in the as-received condition were fragmented during FSP resulting in a fine dispersion of these particles (Fig. 7b). In the peak-aged condition, δ' precipitates, grain-boundary PFZs, and some GBPs were present (Fig. 7c), as observed in the as-received material (Fig. 3b). For this material, a dimpled fracture was observed after testing at 20°C and -196°C (Fig. 7d) – in contrast to as-received coarse-grained material which exhibited mixed ductile-intergranular and brittle-intergranular fracture at 20°C and a completely brittle intergranular fracture at -196°C.

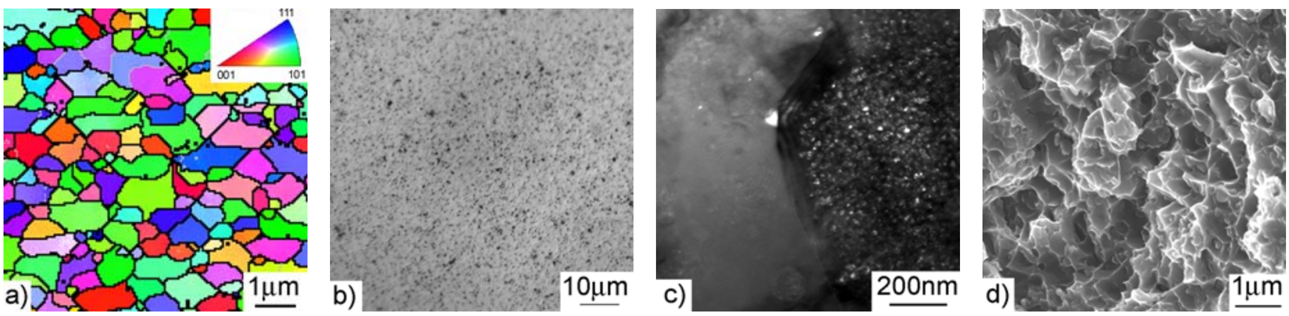

Fig. 7 FSP 8090 alloy (peak-aged condition): (a) EBSD image, (b) Optical micrograph of electropolished surface showing fine dispersion of fragmented constituent particles (cf. Fig.3a), (c) TEM showing fine δ' precipitates and PFZ adjacent to grain boundary, and (d) SEM of fracture surface produced at -196°C. (The fracture surface produced at 20°C was similar). [27]

It could be argued that producing a fine dispersion of fragmented constituent particles and reducing grain size could result in a more homogeneous slip mode or, if planar slip did occur, slip-band lengths would be short. Stress concentrations due to dislocation pile-ups at grain boundaries would then be much smaller than in coarse-grained material, thereby resulting in a dimpled fracture surface rather than a brittle intergranular fracture. However, given the lack of correlation between slip mode and fracture mode described in the previous sections, an alternative explanation seems more likely. Thus, the small, dispersed constituent particles within grains (and at some grain boundaries) nucleate voids at lower strains than grain-boundary precipitates (which are smaller), and coalescence of these voids produces predominantly dimpled transgranular fracture surfaces – with a dimple size sometimes greater than the grain size. Once voids have been nucleated at the particles, strains become concentrated between them rather than at grain boundaries. Larger strains are required for coalescence of particle-nucleated voids than for coalescence of aligned voids nucleated at grain-boundary precipitates, so that toughness is higher for transgranular fracture compared with intergranular fracture.

7. Observations of Slip and Fracture Modes in Other Materials

For Al-Mg-Si alloys, a change from intergranular fracture to transgranular fracture, and increasing toughness, is produced by the addition of manganese to form dispersoid particles [9,10]. It has been argued that slip may be more homogeneous when dispersoids are present, so that the stresses at grain boundaries due to dislocation pile-ups are reduced, thereby producing the change in fracture mode. However, the alternative explanation suggested for the FSP 8090 alloy could be applicable, i.e. adding dispersoid particles in Al-Mg-Si alloys could cause the primary void-nucleation sites to change from grain-boundary precipitates to the dispersoids within grains.

For Al-Mg alloys, a transition from transgranular fracture to intergranular fracture occurs when the magnesium content increases above ~9wt.% [27,34]. The slip mode should be more homogeneous in solid-solution strengthening Al-Mg alloys than in Al alloys strengthened by fine, coherent precipitates, and a significant change in slip mode between alloys with 8 and 10% magnesium would not be expected. Thus, the most likely explanation for the change to brittle intergranular fracture with increasing Mg content is that segregation of Mg at grain boundaries increases with increasing Mg content, and that a 2-D phase change occurs at a critical Mg content around ~9wt.%. Magnesium segregation and indications of a 2-D phase have indeed been observed by TEM [29-31].

Comparisons of Al alloys with other materials, where marked planar slip occurs without promoting brittle intergranular fracture, also raise doubts regarding the planar-slip hypothesis (and support for the segregation hypothesis) for brittle intergranular fracture. For example, a nickel-based superalloy (Waspaloy – Ni 19.3Cr 13.9Co 4.0Mo 3.1Ti wt.%) is strengthened by coherent γ' (Ni_3-(Al,Ti)) ($L1_2$) precipitates [35], analogous to δ' (Al_3Li) ($L1_2$) precipitates in Al-Li alloys. It also exhibits planar slip (Fig.8a) since precipitates in the underaged condition are easily sheared by dislocations. For underaged conditions, there are also no grain-boundary precipitates or PFZs adjacent to grain boundaries – again similar to underaged Al-Li alloys. For Waspaloy, ductile transgranular fracture occurs at 20°C and -196°C (Fig. 8b,c). The difference in fracture modes between Al-Li and Waspaloy most likely occurs because there are no embrittling segregants at grain boundaries in Waspaloy, whereas Li segregation weakens grain boundaries in Al-Li alloys.

The case for a grain-boundary-segregation hypothesis for brittle intergranular fracture in Al alloys is supported by comparisons of the effects of re-ageing of Al-Li alloys with the effects of re-tempering a martensitic steel. For this steel, initially tempered at 425°C for 24h, re-tempering for 5 minutes at 525°C produced a substantial increase in fracture toughness (impact energy) and a decrease in the extent of brittle intergranular fracture, whereas decreases in toughness occurred at longer times (Fig.9a) [36]. This behaviour is remarkably similar to that observed for re-ageing of the 8090 alloy (Fig.4a), and it would not be surprising if there were similar explanations. For tempered-martensitic steels, it is well established that brittle intergranular fracture is promoted by segregation of metalloid impurities to grain boundaries. The toughening effects of re-tempering for short times were explained in terms of a reversion of a 2-D phosphorus-rich grain-boundary phase [36]. For Al-Li alloys, analyses have shown that impurity segregation is not involved, but that lithium segregation is probably present. Thus, the explanation for brittle intergranular fracture in Al-Li alloys in terms of lithium segregation, and reversion of 2-D lithium rich phases during re-ageing, is supported by the comparisons with the temper-embrittled steels.

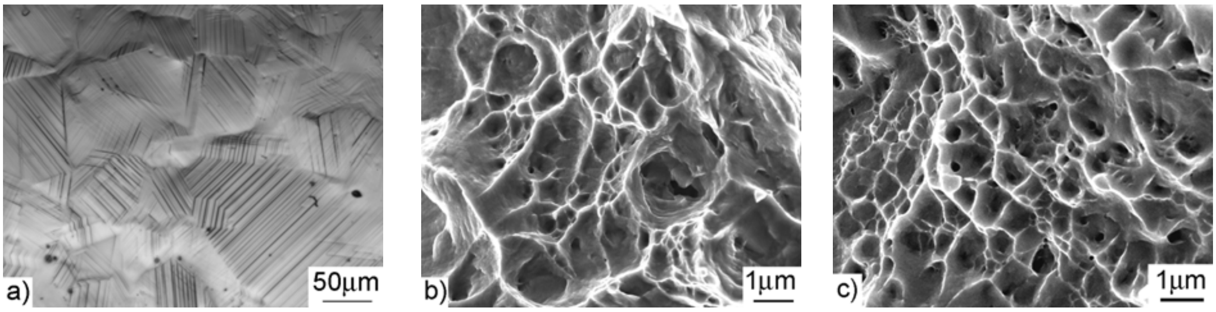

Fig. 8(a) Optical micrograph of electropolished surface of Waspaloy (2h 1040°C oil quench + 6h 730°C air cool) after 2.5% strain showing planar slip, and (b,c) SEM of fracture surfaces produced at 20°C and -196°C showing transgranular dimpled fracture surfaces [27].

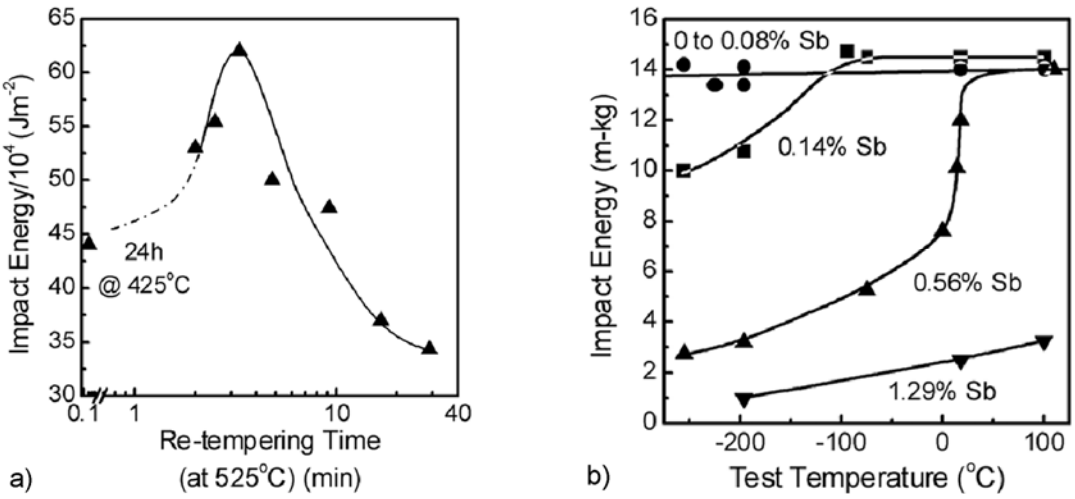

Fig. 9 (a) Plot of impact energy versus re-tempering times for a martensitic steel where low impact energies are associated with phosphorus segregation at grain boundaries. (Re-plotted from the work of Shinoda et al. [36]) and (b) Plots of impact energy versus test temperature for Cu-Sb alloys, showing increasing ductile-to-brittle transition temperature with increasing Sb content. (Replotted from data given by McLean [37])

The transitions from ductile transgranular fracture to brittle intergranular fracture observed with decreasing temperature in the 8090 alloy are also analogous to the behaviour observed in some other materials embrittled by impurity segregation to grain boundaries. For example, for Cu-Sb alloys, the ductile-to-brittle transition temperature increases with increasing Sb content, with the transitions also occurring over a narrow temperature range in some cases (Fig. 9b) [37]. Such observations further strengthen the argument that brittle intergranular fracture in Al-Li alloys is associated with segregation at grain boundaries and that the slip-mode is incidental. High strains probably occur at grain boundaries regardless of slip-mode due to general strain incompatibilities in adjacent grains so that, if grain boundary cohesion is low, intergranular fracture will occur.

8. Conclusion

A review of previous work, along with some new observations, suggest that (i) there is little or no correlation between slip mode and the tendency for brittle intergranular fracture in Al alloys, and (ii) low grain-boundary cohesion is probably associated with segregation of alloying elements such as Li and Mg, which results in specific, temperature/concentration-dependant structural changes, i.e. 2-D grain-boundary phase transitions.

References

[1] E. Hornbogen and K-H. Zum Gahr: Metallography 8 (1975) 181-202.
[2] J. C. Williams, A. W. Thompson and R. G. Baggerly 8 (1974) 625-630.
[3] A Luft: Pro. Mat. Sci. 35 (1991) 97-204.
[4] I. J. Polmear: Light Alloys (3rd edition, Arnold, London, 1995) 35.
[5] A. A. Csontos and E. A. Starke: Inter. J. Plasticity 21 (2005) 1097-1118.
[6] P. J. Gregson and H. M. Flower: Acta metall. 33 (1985) 527-537.
[7] B. Noble, S. J. Harris and K. Dinsdale: Materials Science Forum 331-337 (2000) 1353-1358.
[8] P. D. Pitcher, R. J. Stewart and S. Gupta: Scripta Metall. 26 (1992) 511-516.
[9] J. Dowling and J. W. Martin: Acta Metall. 24 (1976) 1147-1153.
[10] K. C. Prince and J. W. Martin: Acta Metall. 27 (1979) 1401-1408.
[11] L. Edwards: Proceedings of the Riso International Symposium on Metallurgy and Materials ` Science (1983) 237-242.
[12] G. Terlinde and G. Luetjering: Met. Trans. A. 13A (1982) 1283- 1292.
[13] S. P. Lynch: Mat Sci. Engng. A136 (1991) 25-43.
[14] S. P. Lynch, A. R. Wilson and R. T. Byrnes: Mat. Sci. Engng A172 (1993) 79-93.
[15] S. P. Lynch, B.C. Muddle and T. Pasang: Acta Mater. 49 (2001) 2863-2874.
[16] S.P. Lynch, B. C. Muddle and T. Pasang: Phil Mag. 82 (2002) 3361-3373.
[17] W. S. Miller, M. P. Thomas D. J. Lloyd and D. Creber, Aluminium Lihtium Alloys III, (1986) 584-594.
[18] K. Welpmann, Y. T. Lee, M. Peters: Mat. Sci. Engng A129 (1990) 21-34.
[19] K. S. Sohn, Y. T. Lee, N. J. Kim: Scripta Metall. Mater. 32 (1995) 1255-1260.
[20] J. Glazer, S. L. Verzasconi, R. R. Sawtell and J. W. Morris: Met. Trans. A. 18A (1987) 1695-1701.
[21] M. Guttman: Met Trans. A., 8A (1977) 1383-1401.
[22] C. Rottman: Journal De Physique 49 (1988) C5-313 – C5 326.
[23] J. W. Cahn: Journal De Physique 43 (1982) C6-199- C6-213.
[24] E. W. Hart: Scripta Met. 2 (1969) 179-182.
[25] E. L. Maksimova, E. I. Rabkin, L. S. Shvindlerman and B. B. Straumal: Acta Metall. 37 (1989) 1995-1998.
[26] C. P. Blankenship and E. A. Starke: Met. Trans. 24A (1993) 833-841.
[27] S. Moutsos, PhD thesis, Monash University, to be published 2010.
[28] S. Knight: SCC of Al-Zn-Mg-Cu Alloys, PhD Thesis, Monash University (2008).
[29] D. C. Paine, G. C. Weatherly and K. T. Aust: J. Mat. Sci. 21 (1986) 4257-4261.
[30] J. M. Chen T. S. Sun, R. K. Viswanadham, J. A. S. Green: Met. Trans. 8A (1977) 1935-1940.
[31] T. Malis, M. C. Chaturved: J. Mat. Sci. 17 (1982) 1479-1486.
[32] M. P. Seah: Acta Metall. 28 (1980) 955-962.
[33] H. R. Last and R. K. Garrett Jr: Met. Mat. Trans. A. 27A (1996) 737-745.
[34] Masahiro Yanagawa: J. Japan Inst. Light Metals 44 (1994) 492-497.
[35] R. E. Stoltz and A. G. Pineau: Mat. Sci. Engng 34 (1978) 275-284.
[36] T. Shinoda, Y. Mishima, A. Kobayashi, T. Suzuki: Metallk. 77 (1986) 433-441.
[37] D. J. Mc Lean: J. Inst. Metals 81 (1952-1953) 121-123.