Comparison of the Effect of Individual and Combined Zr and Mn Additions on the Fracture Behavior of Al-Cu-Li Alloy AA2198 Rolled Sheet

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The effect of individual and combined addition of dispersoid-forming alloying elements Zr and Mn on the fracture behavior of the Al-Cu-Li alloy 2198 has been investigated by the Kahn tear test. Overall, the standard baseline 2198 alloy containing only Zr exhibited the best performance, while the alloy with the combined presence of Zr and Mn was slightly inferior. The lowest properties were seen for a Zr-free 2198-0.4Mn alloy variant. In the T351 temper fracture initiated at coarse constituent particles that formed large cavities and microvoid sheets linked the initial sites of void growth. In the Mn-containing alloys microvoids clearly nucleated at the coarser $\text{Al}_{20}\text{Cu}_2\text{Mn}_3$ dispersoids within the microstructure, while this was not identifiable for the finer coherent Al_3Zr dispersoids. However, this difference in the mechanism of cavity linkage had little effect on the overall toughness of the materials, which was more closely related to the effect of Mn and Zr on the level of recrystallization. Extended artificial aging promoted grain boundary decohesion due to the precipitation of high densities of T_1 particles on GBs and favored a cleavage fracture mode. Particle decohesive fracture was also promoted by T_1 precipitation on the Mn dispersoids.

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I. INTRODUCTION

THE fracture behavior of the first two generations of Al-Li alloys has been studied over many years.^[1–6] A main conclusion from this research is that there is a detrimental effect of the metastable δ' phase, because it causes intense slip localization. This results in stress concentration at grain boundaries which, when combined with the presence of grain boundary phases that have poor matrix cohesion (*e.g.*, δ and T_2) and their associated precipitate-free zones (PFZs), readily leads to intergranular fracture and a low toughness.^[7,8] As a result, such alloys have only found limited application in aircraft design, despite the potential they offer for mass reduction.^[1–4] More recently, there has been renewed interest in the, so called, “3rd generation” of Al-Li alloys because of the improved mechanical properties they can provide, of high strength combined with significantly better damage tolerance.^[5,9] The main reason for the improved performance of these newer materials lies in their lower lithium concentration (1.0 to 1.8 wt pct),^[3] which does not exceed the metastable δ' phase solvus.^[4,10] In peak aged tempers suppression of δ' causes dominance of the T_1 phase (together with some S and θ') which leads to a reduction in slip localization and, when combined with less detrimental grain boundary precipitation, virtually eliminates the intergranular

fracture issues associated with earlier alloy generations.^[11,12]

Microstructural factors that have a strong influence on the fracture behavior of Al alloys include: the state of matrix and grain boundary precipitation, which is related to the aging condition, the presence of coarse constituent particles, trace impurities, PFZs, the grain size and structure, level of recrystallization, the dispersoids present within the material, and crystallographic texture.^[7,8,12–20] In older, δ' -containing Al-Li alloys the main fracture mode is intergranular ductile fracture, which is exacerbated by poor grain boundary cohesive strength and the concentration of slip in shear bands and within grain boundary PFZs.^[7,8] In comparison, fracture in more recently developed Al-Li alloys has been reported to involve several synergistic factors including: (i) initiation due to cracking at coarse Fe- and Si-containing constituent particles at grain boundaries, and the subsequent growth of associated voids, (ii) the spread of intergranular fracture by microvoid formation at finer grain boundary particles, such as dispersoids and equilibrium precipitates formed during age hardening, and (iii) transgranular linkage in void sheets nucleated at matrix dispersoids, which coalesce within shear bands.^[8] This is frequently still associated with strain localization, the intensity of which depends on the state of matrix precipitation, and increases the stress concentration at grain boundaries, aiding crack initiation.^[7,8] Although not as important as in earlier Al-Li alloys, the presence of PFZs can also still assist crack initiation by concentration of slip at grain boundaries and triple junctions.^[13]

The joint addition of Zr and Mn dispersoid-forming elements is now established industrial practice in damage

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tolerant aerospace plate products, such as AA2050.^[21] The rationale behind their combined addition is based on the opposite microsegregation patterns they form during casting.^[2,3] In theory, this should increase recrystallization resistance by leading to a greater uniformity of dispersoid coverage in rolled products,^[4–8] consequently improving fracture toughness.^[9–11] However, their synergistic effect on the fracture behavior of AA2198 used for sheet products has not been previously reported. Indeed, in such sheet material the current authors have previously found there can be a disadvantage of joint Zr and Mn additions, in terms of recrystallization resistance, relative to the sole use of Zr.^[22,23]

In Al-Li alloys zirconium forms a high density of fine coherent Al₃Zr precipitates,^[24] while manganese forms a lower density of coarse and semi-coherent Al₂₀Cu₂Mn₃ dispersoids that become incoherent after rolling.^[25] In addition, manganese, together with Cu, Fe, and Si, can form coarse insoluble constituent phases.^[22,26] The main beneficial effect of Al₃Zr dispersoids is to reduce the length of shear bands by preventing recrystallization, due to the finer grain size and subgrain structure present in an unrecrystallized material.^[27] The dispersoids themselves are non-shearable and thus also homogenize slip.^[27] Due to their small size and coherency, nucleation of voids at the Al₃Zr dispersoids is more difficult than for Mn dispersoids, which perform the same role in terms of slip dispersal, but have more complicated and contradictory effects on the fracture behavior. An important benefit of their addition is that they can change the fracture mode from intergranular to transgranular,^[28,29] with microvoids forming around them.^[30] They are also reported to reduce the crack propagation rate more effectively than the finer Al₃Zr dispersoids.^[31] However, on increasing their volume fraction there is competition between the effects of slower microvoid nucleation due to slip homogenization, thus improving fracture toughness, and accelerated microvoid coalescence owing to the reduced particle spacing.^[32]

The present paper aims to systematically compare the differences in fracture behavior caused by single and joint additions of Zr and Mn to a typical 3rd generation Al-Li alloy, such as the AA2198, in terms of the direct and indirect effects of the dispersoid particles they form. The case of combined Zr and Mn additions is particularly interesting, since it has not been previously studied in detail in the context of fracture toughness. After presenting key aspects of the materials' microstructures, the alloys' toughness behavior, with respect to orientation relative to the rolling direction (RD) and aging condition, will be discussed using data obtained from Kahn tear tests. In addition, fracture surfaces from the test samples have been characterized by SEM in order to determine the operating fracture modes.

II. EXPERIMENTAL

A. Materials

The materials studied in this work were provided by Constellium from the Centre de Recherches de Voreppe,

France. Four experimental alloy variants were compared in the form of 6-mm-thick sheet, with compositions based on AA2198 for Cu, Mg, Li, and Ag, but with varying amounts of Zr and Mn. The nominal composition of the base alloy is given in Table I, where the Zr and Mn levels in the alloy variants measured by chemical analysis are also defined. Trace elements in the microstructure were 0.05 wt pct Fe, 0.03 wt pct Si, 0.02 wt pct Zn, and 0.02 wt pct Ti. Care was taken in ensuring tight control for the base composition, with variability between castings kept to less than 3 pct for each element. The alloy variants are identified according to their dispersoid element content (in wt pct) in Table I as: 2198-0.1Zr, 2198-0.1Zr-0.3Mn, 2198-0.05Zr-0.3Mn, and 2198-0.4Mn. In the as-received temper (T351), the rolled sheet materials had undergone solution treatment, stretching and natural aging. Samples were also aged at 428 K (155 °C) with a heating rate of 75 K/h for 14 and 100 hours prior to the fracture tests, to produce slightly underaged and overaged microstructures with a similar hardness level.

B. Fracture Toughness Measurements

Kahn tear tests were employed to measure the alloys' notch toughness characteristics, since this technique is suitable for sheet materials.^[33] Testing was carried out according to the ASTM specification B 871-01 at a displacement rate of 1.3 mm/min.^[34] All samples were tested in the three different starting temper conditions, T351, and after aging for 14 and 100 hours at 428 K (155 °C), as well as in two different orientations relative to the RD (L-T, notch normal to RD; T-L, notch parallel to RD). Selected samples were also tested at 45 deg to the RD. The sample dimensions were 57.17 mm × 36.51 mm × 2.95 mm. Before testing, each notch was checked individually under the optical microscope to ensure a smooth and accurate tip radius.^[33] The following four parameters were extracted from the measured load curves, averaged over three tests for each condition: (i) tear strength, (ii) unit initiation energy (UIE), (iii) unit propagation energy (UPE), and (iv) unit total energy (UTE), calculated from Eqs. [1] through [4] below:

$$\text{tear strength} = \frac{4 \cdot F_{\text{MAX}}}{b \cdot t} \quad [1]$$

$$\text{UIE} = \frac{\text{initiation area}}{b \cdot t} \quad [2]$$

$$\text{UPE} = \frac{\text{propagation area}}{b \cdot t} \quad [3]$$

$$\text{UTE} = \text{UIE} + \text{UPE} \quad [4]$$

where the initiation area is the area under the load-displacement curve up to peak load (F_{MAX}), the propagation area is the area below the curve from maximum load until failure, b is the length from the notch tip to the sample edge, t is the specimen thickness,

Table I. Alloy Compositions Used in this Study (Weight Percent)

Alloy	Cu	Li	Mg	Ag	Zr	Mn
2198-0.1Zr	2.9–3.5	0.8–1.1	0.25–0.8	0.1–0.5	0.110	—
2198-0.1Zr-0.3Mn	2.9–3.5	0.8–1.1	0.25–0.8	0.1–0.5	0.111	0.30
2198-0.05Zr-0.3Mn	2.9–3.5	0.8–1.1	0.25–0.8	0.1–0.5	0.053	0.31
2198-0.4Mn	2.9–3.5	0.8–1.1	0.25–0.8	0.1–0.5	—	0.40

The Zr and Mn concentrations were directly measured. Nominal compositions are given for other elements.

and F_{MAX} is the maximum force required to initiate a crack.

C. Characterization

SEM with an integrated EBSD system was used to determine the grain size, level of recrystallization, and observe the particle distributions, as well as to study the fracture surfaces following the Kahn tear tests. The instruments employed were an FEI Sirion FEG-SEM and an FEI Magellan 400 XHR FEG-SEM. For EBSD, the Sirion microscope was operated at 20 kV, while 15 kV was selected for secondary electron imaging. Electron backscatter imaging was carried out at 15 kV in the Magellan microscope in order to make use of its high contrast annular detector. An FEI Tecnai F30 FEG-TEM, operating at 300 kV, was also used to quantify the dispersoid distributions, check subgrain sizes and observe the age hardening precipitates. Imaging was carried out in bright field and scanning transmission mode with a high angle annular dark field detector (HAADF-STEM).

III. RESULTS AND DISCUSSION

A. Material Characterization

Microstructural characterization, of parameters important to understanding the fracture resistance of the different AA2198 alloy variants, has been performed prior to testing: including their primary and dispersoid particle distributions, fraction of recrystallization, grain structure, and texture.

1. Primary particles and dispersoids

An overview of the density and size of the coarse constituent particles in the AA2198 alloys, with different Zr and Mn contents, is provided in Figure 1. The constituent particles are strongly aligned in the RD, but were only of the order of 1 to 4 μm in size and had a low volume fraction, owing to the low iron content in the base composition (<0.1 wt pct). In the standard 2198-0.1Zr alloy (Figure 1(a)), constituent particles are formed mainly due to the presence of iron and copper.^[35] Although the addition of Mn, together with Cu and Fe, can lead to the precipitation of additional phases that also contain Mn,^[36–38] there was a negligible increase in the volume fraction of constituent particles in the Mn-containing alloy variants (Figures 1(a) through c)).

Higher magnification imaging (Figure 1) showed that in the Mn-containing alloys there was a high density of

$\text{Al}_{20}\text{Cu}_2\text{Mn}_3$ dispersoids present in the size range 100 to 500 nm, whereas the Al_3Zr dispersoids have much smaller dimensions (~20 nm). In Figure 2, HAADF-STEM images of the typical dispersoid distributions in each alloy are presented. It is evident from these images that the dispersoids were concentrated in discrete bands aligned in the rolling plane. Where Zr and Mn were both present their respective dispersoid families formed alternating bands in the sheet normal direction (ND), owing to the inverse microsegregation patterns of these two elements in the original casting.^[23,39]

Table II summarizes the average dispersoid sizes and densities measured from the STEM images, where it is obvious that the $\text{Al}_{20}\text{Cu}_2\text{Mn}_3$ dispersoids are an order of magnitude larger and have a lower number density than the Al_3Zr particles. However, alloying with Mn resulted in the presence of some Mn-containing dispersoids, at the top end of their size distribution with dimensions above 1 μm , which could be considered to be overlapping with the bottom end of the size range of the constituent particles (Figures 1(b) and (c)). In addition, it can be noted from Table II and Figure 2 that in the 2198-0.1Zr and 2198-0.1Zr-0.3Mn alloys the Al_3Zr dispersoids exhibited a significant difference in their size distribution, despite the fact that both alloys contained the same Zr content and had received an identical homogenization treatment. This observation has been explained previously^[22] and, in brief, results from a small loss of Zr supersaturation from the matrix to Mn-containing phases, which reduce the density and increases the size of the Al_3Zr dispersoids that precipitate during the homogenization treatment.

2. Level of recrystallization and texture

A higher level of recrystallization and factors such as grain size and shape can greatly influence fracture toughness, although they are interrelated with the dispersoid content and degree of recrystallization.^[8,13,15–20,40,41] Stronger textures in unrecrystallized microstructures are also associated with lower misorientations between neighboring grains, which can stimulate easier slip transmission from one grain to another.^[8,20] Hence, their effect on fracture cannot be easily separated from that of recrystallization.

The recrystallized fractions for each alloy are given in Table III, along with EBSD maps in Figure 3, that show the grain structure and distribution of main rolling texture components within the alloy variants. Reducing the Zr content, in parallel with an increasing addition level of Mn, can be seen to be detrimental to the AA2198 base alloy's recrystallization resistance. The volume fraction of recrystallization was negligible in the

Fig. 1—SEM images illustrating differences in the density of coarse constituent particles in the alloy variants in the T351 condition, with accompanying higher magnification views comparing the distributions of larger scale dispersoids: (a) 2198-0.1Zr, (b) 2198-0.1Zr-0.3Mn, and (c) 2198-0.4Mn.

base alloy that contained only Zr, and increased to 100 pct in the 2198-0.4Mn sheet that contained no Zr. It is also noted that the alloy that combined 0.3 wt pct Mn, with the same standard 0.11 wt pct Zr concentration used in the AA2198 base alloy, showed a significantly greater level of recrystallization (~14 pct). This reduction in recrystallization resistance, seen when Mn is combined with Zr, has previously been shown to be

caused by its strong influence on the Al_3Zr dispersoid-free band width within a sheet, when a small level of Zr is removed from the matrix into Mn-bearing phases.^[22]

Figure 3 indicates that Brass $\{011\}\langle 112 \rangle$ and S $\{123\}\langle 634 \rangle$ components prevailed in unrecrystallized regions, while on recrystallization the texture formed during rolling was generally replaced by random orientations. The textures thus became weaker with increasing

Fig. 2—HAADF-STEM images showing the dispersoid distributions in the (a) 2198-0.1Zr, (b) 2198-0.1Zr-0.3Mn, (c) 2198-0.05Zr-0.3Mn, and (d) 2198-0.4Mn alloy variant (T351 condition).

Table II. Summary of the Average Dispersoid Particle Sizes and Densities seen in the Alloy Variants, Measured in the TEM for Both Dispersoid Types at their Mid-thickness Plane (T351 Condition)

	2198-0.1Zr	2198-0.1Zr-0.3Mn	2198-0.05Zr-0.3Mn	2198-0.4Mn
Al_3Zr				
Diameter (nm)	18.8 ± 0.2	21.3 ± 0.3	25.3 ± 0.8	—
Number density (μm^{-3})	252 ± 60	174 ± 23	NM	—
$\text{Al}_{20}\text{Cu}_2\text{Mn}_3$				
Length (nm)	—	223.0 ± 4.0	264.6 ± 7.6	221.7 ± 4.0
Width (nm)	—	102.8 ± 0.8	107.6 ± 1.2	107.9 ± 0.8
Equiv. spherical diameter	—	152.4	166.2	157.0
Number density (μm^{-3})	—	2.6	2.6	3.6
NM not measured.				

Mn and reducing Zr content, as the recrystallized fraction increased. It can also be seen from Table III that the 2198-0.1Zr-0.3Mn alloy, with its largely unrecrystallized fibrous structure, had the finest average HAGB spacing in ND as well as the smallest overall subgrain size. This was closely followed by the virtually fully unrecrystallized

2198-0.1Zr alloy, which had a slightly coarser grain width. In contrast, the largest values of HAGB spacing were exhibited by the fully recrystallized 2198-0.4Mn alloy, which was devoid of substructure, and the partially recrystallized 2198-0.05Zr-0.3Mn alloy had an intermediate average grain size.

Table III. Recrystallized Volume Fraction and Average Grain Size Data in Each Alloy Variant Measured at the Mid-thickness Plane by EBSD and TEM

	2198-0.1Zr	2198-0.1Zr-0.3Mn	2198-0.05Zr-0.3Mn	2198-0.4Mn
Recrystallized fraction (pct)	1.5	13.5	62.5	100
HAGB spacing ND (μm)	7.36 ± 0.66	5.76 ± 0.38	18.38 ± 1.49	50.6 ± 4.27
LAGB spacing ND (μm)	2.19 ± 0.07	0.96 ± 0.02	1.36 ± 0.05	—
LAGB spacing RD (μm)	3.24 ± 0.15	1.54 ± 0.12	1.72 ± 0.06	—

Fig. 3—EBSD maps showing the grain structures and distribution of the main rolling texture components in the T351 sheet for (a) the 2198-0.1Zr, (b) 2198-0.1Zr-0.3Mn, (c) 2198-0.05Zr-0.3Mn, and (d) the 2198-0.4Mn alloy variant. The texture components are given in the key where white indicates randomly orientated grains.

3. Age hardening behavior

As well as performing toughness tests in the T351 condition, samples were aged at 428 K (155 °C) to slightly underaged and overaged tempers to better understand the effect of the matrix precipitation state on the toughness behavior. In Figure 4, it can be seen that there was little difference in the alloys' aging responses, as a result of varying their Zr and Mn content. After 2 hours of isothermal treatment at 428 K (155 °C), the hardness of all the materials began to rise until it reached a plateau after about 20 hours, which can be attributed to precipitation of the maximum volume fraction of the main strengthening phase, T_1 , and a low volume fraction of θ' .^[9,42] In the same heat treatment condition, the matrix microstructures were identical for all the aged alloys and example TEM images are shown in Figure 5 for both aging times. The

$[100]_{\text{Al}}$ zone axis diffraction pattern in Figure 5(c) shows four elongated reflections, corresponding to the T_1 phase surrounding the matrix $\{110\}$ position, along with weaker horizontal and vertical streaks from the θ' phase—confirming the presence of the dominant T_1 phase and a more minor volume fraction of θ' . The microstructure for the overaged condition (100 hours) indicates the presence of larger T_1 plates, although their volume fraction is known to remain nearly constant during the hardness plateau region.^[43] A slight increase was recorded in the hardness measured between the two under and overaged conditions (14 and 100 hours) used in the fracture tests of only 10 HV. The immunity of the 2198 alloys to T_1 precipitation irrespective of their largely different grain structures resulting from the type of dispersoid additions is owing to the fact that precipitation surpasses the influence of grain structure

Fig. 4—Age hardening curves on artificial aging at 428 K (155 °C), showing a similar behavior for all the alloy variants.

in terms of strength in the aged condition. In addition, it is known that upon aging the alloy strength is controlled by precipitation within the grains, while the grain size only affects fracture toughness due to preferential precipitation on the GBs.^[44] Since hardness is proportional to yield strength, it is reasonable that no differences are observed in the aging curves of the present alloys.

B. Kahn Tear Test Results

The results of the parameters measured from the Kahn tear tests (tear strength, UIE, UPE, UTE, Eqs. [1] through [4]) are compared in Figure 6, as a function of heat treatment condition in both the L-T and T-L sample orientations. Although it is theoretically known that the UIE correlates best to the plane strain fracture toughness, K_{IC} ,^[45] the other parameters measured from the Kahn tests have been included as they can provide a more complete view of the materials' fracture behavior.

From Figure 6, it can be seen that despite some scatter in the results, the data showed consistent trends with respect to the relative performance of the alloy variants across the different aging conditions. Starting with the tear strength data, after aging for 14 hours at 428 K (155 °C), a broadly similar increase in tear strength could be seen for all the alloys, compared to the T351 starting temper. However, on extended aging (100 hours) the tear strengths in the T-L orientation dropped much more significantly than the respective decrease seen in the L-T orientation. In the L-T orientation, over the three different heat treatments, the standard 2198-0.1Zr alloy that contained no Mn performed best and the lowest values were consistently measured for the Zr-free 2198-0.4Mn alloy (Figure 6(a)). It can be further seen that the 2198-0.1Zr-0.3Mn alloy, with dual additions of Zr and Mn, performed second best in terms of tear strength, but was only marginally worse than the standard 2198-0.1Zr baseline material. Finally, the 2198-0.05Zr-0.3Mn alloy

Fig. 5—Typical matrix precipitate microstructures imaged in the $\langle 011 \rangle$ zone axis for the 2198 alloy after aging at 428 K (155 °C) for (a) 14 hours, and (b) 100 hours, with (c) an accompanying $\langle 001 \rangle$ zone axis diffraction pattern for the 14 hours condition.

Fig. 6—Average toughness parameters measured from Kahn tear tests (tear strength, UIE, UPE, UTE), plotted as a function of aging time in (a) L-T, and (b) T-L sample orientations.

that contained Mn and a reduced Zr level had the second worst performance. In the T-L orientation (Figure 6(b)), the overall trend in the order of the materials' tear strengths was the same as that for the L-T tests, but the materials became more separated into two groups: the alloys with the higher Zr content (2198-0.1Zr and 2198-0.1Zr-0.3Mn) showing better tear strength than the 2198-0.05Zr-0.3Mn and 2198-0.4Mn samples.

In contrast to the tear strength results, the UTE diminished with aging time across all the alloys and the differences between the alloy variants were less pronounced in the artificially aged conditions for both L-T and T-L orientations. In the L-T orientation the UTE gave consistent results, with respect to the order of each alloy's performance noted above, and the 2198-0.1Zr alloy again exhibited the highest values in all three aging conditions. The 2198-0.4Mn alloy also had the lowest UTE level in the T351 and artificially aged tempers and nearly the lowest in the overaged temper in the L-T orientation. There was again a similar, but slightly less clear, trend in the T-L orientation.

When the UTE was separated into UIE and the UPE data, for both test piece orientations in the T351 temper the 2198-0.1Zr and 2198-0.1Zr-0.3Mn alloys showed the highest energy to initiate a crack and the 2198-0.4Mn again showed the minimum. However, a steeper drop in UIE was observed on aging for the 2198-0.1Zr-0.3Mn

and 2198-0.1Zr alloys and the range in the data for the overaged condition became smaller than the error in the test, so that differences between the alloy variants became more marginal. The UPE gave more scatter in the results than tear strength and UTE, but again showed that the standard 2198-0.1Zr alloy performed best overall. In addition, it was not possible to measure UPE data for the T-L orientation after 100 hours artificial aging because the samples fractured abruptly at maximum load when the notch was aligned with RD.

If all the Kahn test results are considered together, the 2198-0.1Zr and 2198-0.1Zr-0.3Mn alloys were consistently found to exhibit the best fracture properties, with the former being marginally superior, while the Zr-free 2198-0.4Mn variant was the worst performing material. Overall, the results therefore indicate that when the 2198-0.1Zr alloy is compared with a material with a comparable Zr content that also contains an additional set of Mn dispersoids, this can have a slight negative impact on sheet toughness. To further verify this result, these two alloy variants were also compared in the 14 hours at 428 K (155 °C) artificially aged condition, using samples machined with the notch orientated at 45 deg to RD. The results from these additional tests are presented in Table IV, where it can be seen that the 2198-0.1Zr alloy again exhibited a slightly higher tear strength than the 2198-0.1Zr-0.3Mn material.

C. Fracture Behavior

The reasons for the differences observed in the toughness data have been explored further by examining the fracture surfaces of all the materials at the initiation area of the Kahn test samples. However, because of the large number of test conditions only selected examples are referred to below, which illustrate the role of specific important material variables.

1. Effect of Zr and Mn dispersoid distributions on fracture behavior

The effect of the particles generated by the Zr and Mn alloying additions on microvoid formation and the

fracture resistance of the AA2198 base alloy will first be considered by examining the materials in the T351 temper. This test condition has been selected to avoid the influence of additional grain boundary precipitation that might occur as a result of artificial aging.^[7,8,12,46,47] In this condition, the 2198-0.1Zr and 2198-0.1Zr-0.3Mn alloys exhibited higher Kahn tear strengths and fracture energies than the other Mn-containing variants that had a lower Zr content, with the Mn-free standard 2198-0.1Zr alloy performing marginally better than the 2198-0.1Zr-0.3Mn alloy (Figure 6) and the 2198-0.4Mn variant being consistently worst overall. As anticipated, the alloys with sole Zr (2198-0.1Zr) and sole Mn (2198-

Table IV. Kahn Tear Test Results for the 2198-0.1Zr and 2198-0.1Zr-0.3Mn Alloys Aged for 14 hours at 428 K (155 °C), Tested with the Notch Direction Oriented at 45 deg to the RD

	Tear Strength (MPa)	UIE (N/mm)	UPE (N/mm)	UTE (N/mm)
2198-0.1Zr	698.2 ± 7.8	120.7 ± 1.8	136.9 ± 21.6	260.7 ± 20.4
2198-0.1Zr-0.3Mn	675.0 ± 1.7	116.3 ± 3.4	148.7 ± 15.7	258.3 ± 14.3

Fig. 7—L-T fracture surfaces for the different 2198 alloy variants in the T351 temper, showing the overall topography at a low magnification, exhibiting coarse dimples initiated by large constituent particles, and fine dimples within void sheets at higher magnifications for (a) the 2198-0.1Zr alloy, (b) the 2198-0.1Zr-0.3Mn, and (c) the 2198-0.4Mn alloy.

0.4Mn) additions yielded quite different fracture behaviors. However, it should be remembered that the dispersoid density in the 2198-0.4Mn variant was insufficient to prevent recrystallization during solution treatment, whereas the 2198-0.1Zr alloy had a nearly fully fibrous grain structure (Figure 3; Table III).

In Figure 7, SEM images of the L–T fracture surfaces are presented. The 2198-0.1Zr alloy (Figure 7(a)) shows a mixture of features including large dimples around coarse constituent particles and sheets of very fine dimples resulting from ductile transgranular fracture in regions devoid of large particles. In this base alloy there was no sign of intergranular fracture and, when viewed at a higher magnification, the small coherent Al_3Zr dispersoids did not appear to nucleate fine-scale voids within the large cavities formed at primary particles, which were generally very smooth in appearance. Equally, Al_3Zr dispersoids could not be identified in the fine dimples seen in the transgranular microvoid sheets (Figure 7(a)). Hence, the fracture mode was one of mainly ductile rupture with fine void sheets linking large-scale void growth at the coarse constituent parti-

cles that acted as nucleation sites. In this alloy, it should be noted that the Al_3Zr dispersoids also contributed to the higher fracture resistance by preventing recrystallization (Figure 3(a)) and maintaining a fibrous grain structure, which resulted in a small effective grain size in the ND-TD plane.

In comparison to the 2198-0.1Zr alloy, the 2198-0.4Mn samples produced a greater area of fine dimples on the fracture surface that were clearly initiated by $\text{Al}_{20}\text{Cu}_2\text{Mn}_3$ dispersoids (Figure 7(c)). The fracture surface also showed large-scale rough faceting, which reflects the coarser recrystallized grain size of this material, and this is probably more detrimental to crack propagation than the stronger texture present in the unrecrystallized 2198-0.1Zr alloy.^[8,13,20,40,41] While in this alloy the primary particles again initiated failure forming large cavities, the smaller dimples caused by the $\text{Al}_{20}\text{Cu}_2\text{Mn}_3$ dispersoids would be expected to accelerate the link-up of the initiation sites. The microvoids themselves were coarser than the corresponding ones in the Zr-containing alloy. It can also be noted that after initiation occurred, the large voids grew to a certain size

Fig. 8—L–T fracture surfaces for the 2198-0.1Zr-0.3Mn alloy as a function of aging time at 428 K (155°C), showing the overall topography at a low magnification and typical regions containing dimples and cleavage steps at higher magnifications: (a) T351, (b) 14 hours, and (c) 100 hours.

with a relatively smooth surface before the surface changed to show many fine dimples. This occurs because the nucleation of finer voids at the $\text{Al}_{20}\text{Cu}_2\text{Mn}_3$ dispersoids requires a larger strain intensity to be generated at the crack tip.^[15,48] However, by comparing Figures 7(a) and (c), it can be noted that cavity growth from the primary particles was more limited in the 2198-0.4Mn alloy than in the 2198-0.1Zr material, which indicates that microvoid nucleation was more difficult in the Mn-free base alloy.

It was shown earlier that the 2198-0.1Zr-0.3Mn alloy had only a slightly inferior fracture resistance to the 2198-0.1Zr base alloy. Due to the presence of Mn, which reduced the density of the Al_3Zr dispersoids (Table II),^[22] this alloy partially recrystallized to a volume fraction of 13.5 pct, which is a low level but still higher than that of the base material (Table III). The fracture surface from this material is shown in Figure 7(b) and at low magnification looks more similar to that seen for the unrecrystallized 2198-0.1Zr alloy than the 2198-0.4Mn alloy. However, at higher magnification the larger cavities formed at the primary particles are again smaller and the fine-scale dimples can be seen to contain $\text{Al}_{20}\text{Cu}_2\text{Mn}_3$ dispersoids.

From the above discussion, it can be concluded that in the T351 temper the overall failure mechanism was similar for each alloy variant, being controlled by void initiation at primary particles and linkage by the growth of transgranular microvoid sheets. However, clear differences were apparent in (i) the macroscopic roughness of the fracture surfaces, owing to the materials' different grain structures, and (ii) void nucleation in the microvoid sheets at $\text{Al}_{20}\text{Cu}_2\text{Mn}_3$ dispersoids, when they were present. Despite these differences, the $\text{Al}_{20}\text{Cu}_2\text{Mn}_3$ dispersoid-containing 2198-0.1Zr-0.3Mn alloy only exhibited a marginally lower performance in the Kahn tear tests compared to the Mn-free 2198-0.1Zr base alloy, and the largest difference in toughness was seen between these two alloys and the 2198-0.4Mn alloy. Therefore, the differences seen in the toughness results are most closely related to changes in grain structures across the alloy variants, while microvoid formation at the $\text{Al}_{20}\text{Cu}_2\text{Mn}_3$ dispersoids is a second-order effect.

Hence, although the $\text{Al}_{20}\text{Cu}_2\text{Mn}_3$ dispersoids have been claimed to have a positive impact on toughness by homogenizing slip, and thus preventing slip localization,^[15,29,49] in the results above this effect is clearly not sufficiently significant to overcome the detrimental consequence of a lower Zr level on the alloys' recrystallization resistance. Furthermore, when joint Zr and Mn additions are made to the 2198 alloy, the additional $\text{Al}_{20}\text{Cu}_2\text{Mn}_3$ dispersoids do not appear to sufficiently alter the energy required for linkage of the constituent particle initiation sites, causing only a marginal reduction in toughness between the 2198-0.1Zr and 2198-0.1Zr-0.3Mn alloys.

2. Effects of the aging treatment on fracture

An alloy's yield strength, tendency for shear localization, and the precipitation of additional coarse second phase particles are affected by artificial aging and, hence, heat treatment can greatly influence fracture toughness.^[46,50,51] In the Kahn tear tests, the most important

effect of aging at 428 K (155 °C) was to increase the tear strength of all the alloy variants, due to the rise in yield stress (Figure 4). However, on extending the aging time (100 hours) the tear strength started to reduce again, even though the hardness was slightly greater. In addition, the crack initiation and propagation energies progressively decreased and differences between the alloy variants reduced with artificial aging time (Figure 6). Finally, in the cross-grain T–L orientation the crack propagation energy was particularly severely affected and premature failure meant it could not be measured after aging for 100 hours.

In Figure 8, a series of images demonstrate the change in fracture mode seen with aging time from the T351 temper up to 100 hours at 428 K (155 °C), in the 2198-0.1Zr-0.3Mn alloy in the L–T orientation. A reduction in the extent of plastic deformation during crack growth is evident from the progressively fewer characteristic features of dimple ductile rupture and the prevalence of

Fig. 9—TEM images of grain boundary precipitation in the 2198-0.1Zr-0.3Mn alloy after artificial aging at 428 K (155 °C) for (a) 14 hours and (b) 100 hours.

a cleavage fracture mode with aging time. This behavior is clearly responsible for the loss of UIE and UTE found at longer aging times (Figure 6). The difference in tear strength seen between long aging times and short aging times is related to the larger influence of the materials' different grain structures.

The role of grain boundary decohesion was also enhanced during aging, as shown by a TEM investigation. From Figure 9, it can be seen that there are only low levels of grain boundary precipitation after 14 hours aging, whereas in the overaged 100-hour sample the grain boundary plane is heavily decorated with T_1 precipitates. However, there is little evidence of the development of accompanying significant PFZs. Such extensive precipitation will clearly allow cracks initiated by coarse primary particles to readily propagate along grain boundaries with little energy absorption. Another effect of extended aging was that decohesive rupture of the $\text{Al}_{20}\text{Cu}_2\text{Mn}_3$ dispersoid particles was observed in all the Mn-containing alloys, but the same observation was rather vague for the Al_3Zr dispersoids. This behavior is illustrated in Figure 10(a) where shallow dimples are present on the fracture

surface of the 2198-0.4Mn alloy after aging for 100 hours at 428 K (155 °C), with lath-shaped $\text{Al}_{20}\text{Cu}_2\text{Mn}_3$ dispersoids protruding from them. TEM imaging indicates that this phenomenon might be likely caused by preferential precipitation of the T_1 strengthening plates on the incoherent interface of the Mn dispersoids at longer aging times. This behavior could be similar to the preferential nucleation on GBs, judging by the short and thick morphology of the T_1 plates in Figure 10(a), since the same morphology occurs in that case.^[52,53] On the other hand, although Figure 10(b) shows that T_1 plates might also precipitate preferentially on Al_3Zr dispersoids,^[54] by SEM imaging of the fracture surface it could not be firmly concluded that such aggregates of particles were responsible for the fine dimples observed in regions rich in Zr dispersoids.

In addition, areas with fine cleavage steps were seen to develop on the fracture surfaces with artificial aging time (Figure 8) that were not present in the T351 temper (Figure 7). The formation of such cleavage steps is probably a direct consequence of slip localization, due to the T_1 plates being sheared on specific slip planes during deformation. In the present Al-Cu-Li alloy, the

Fig. 10—Evidence of the particle decohesive fracture mode after aging for 100 hours (L–T orientation): (a) SEM image of $\text{Al}_{20}\text{Cu}_2\text{Mn}_3$ particles on the fracture surface of the 2198-0.1Zr-0.3Mn alloy, with a corresponding TEM image illustrating the possible signs of preferential T_1 precipitation at the incoherent interface; and (b) SEM image of fine-scale dimples in the 2198-0.1Zr alloy that do not appear to be formed by the Al_3Zr dispersoids, with a corresponding TEM image showing the affiliation of T_1 particles with the interface of an Al_3Zr particle.

T_1 phase is known to be still sheared during plastic deformation, even after prolonged aging treatments, because the T_1 plates do not thicken with aging time at temperatures below 433 K (160 °C).^[43]

IV. CONCLUSIONS

The present work has compared the effects of dispersoids, formed by sole and combined Mn and Zr additions, on the fracture resistance of an Al-Cu-Li 2198, rolled, sheet material with the aid of the Kahn tear test. The type and size of particles present in the microstructure was found to have a significant effect on the fracture behavior, although this occurred mainly indirectly through their influence on the level of recrystallization, which was increased by the addition of Mn to the 2198 base alloy and on reducing the Zr concentration. Overall, the standard 2198-0.1Zr alloy exhibited the best performance, while the combined presence of Zr and Mn in the 2198-0.1Zr-0.3Mn alloy was not found to offer any particular benefits in terms of fracture toughness. The lowest properties overall were seen for the fully recrystallized, Zr-free, 2198-0.4Mn alloy variant.

In the T351 temper, the fracture mode was predominantly ductile rupture by void nucleation and coalescence. Fracture was initiated at coarse constituent particles that formed large dimples on the fracture surface, while microvoid sheets linked the initial sites of void growth. In the Mn-containing alloys microvoids nucleated at the coarser $\text{Al}_{20}\text{Cu}_2\text{Mn}_3$ dispersoids, while it was not possible to draw the same conclusion for the finer coherent Al_3Zr dispersoids, due to limitations arising from their small size. However, this difference in the cavity linkage process had little effect on the toughness values measured for the materials. Other interesting observations include the promotion of grain boundary decohesion due to the precipitation of high densities of T_1 on the GBs with extended aging times and the development of fine cleavage steps with aging time. In addition, it is possible that preferential precipitation of the T_1 phase on the incoherent interface of Mn dispersoids favored a particle decohesive fracture mode.

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