



International Journal of Cast Metals Research

ISSN: 1364-0461 (Print) 1743-1336 (Online) Journal homepage: https://www.tandfonline.com/loi/ycmr20

Tensile properties of cast and mushy state rolled Al-4.5Cu alloy and in situ Al4.5Cu-5TiB₂ composite

M. A. Herbert, G. Das, R. Maiti, M. Chakraborty & R. Mitra

To cite this article: M. A. Herbert, G. Das, R. Maiti, M. Chakraborty & R. Mitra (2010) Tensile properties of cast and mushy state rolled AI-4.5Cu alloy and in situ AI4.5Cu-5TiB₂ composite, International Journal of Cast Metals Research, 23:4, 216-224, DOI: 10.1179/136404609X12580240349018

To link to this article: https://doi.org/10.1179/136404609X12580240349018



Published online: 18 Jul 2013.



📝 Submit your article to this journal 🕑





Q View related articles 🗹



Citing articles: 2 View citing articles 🗹

Tensile properties of cast and mushy state rolled Al-4.5Cu alloy and in situ Al4.5Cu-5TiB₂ composite

M. A. Herbert¹, G. Das², R. Maiti³, M. Chakraborty⁴ and R. Mitra^{*3,5}

A comparative study has been carried out on tensile properties and failure mechanisms of Al-4.5 wt-%Cu alloy and in situ Al-4.5Cu-5TiB₂ composite in as cast or mushy state rolled conditions. For the alloy, mushy state rolling at temperature for 30 vol.-% nominal liquid content to 2.5% thickness reduction leads to marginally higher strength but poorer ductility, while both these properties worsen significantly on 5% reduction because of grain growth and intergranular solute segregation. Contrarily, mechanical properties of the composite are significantly improved on mushy state rolling at temperatures for 10-30 vol.-% liguid to 2.5-5% reduction. Improvement in mechanical properties of the composite on mushy state rolling is attributed to evolution of globular and finer grains. Examination of fracture surfaces by scanning electron microscope has shown evidence of ductile failure in as cast alloy and in cast or mushy state rolled composite, but that of brittle failure in mushy state rolled alloy.

Keywords: Al-Cu alloy, In situ composite, Mushy state rolling, Tensile properties, Fracture

Introduction

Discontinuously reinforced aluminium (DRA) composites have received significant attention for last few decades, because of their isotropic character as well as attractive mechanical properties including high specific strength, elastic modulus and wear resistance.¹⁻⁷ The research on DRA has been driven by potential automotive, military and aerospace applications.^{6–9} It has been reported¹⁰ that the interfaces in *in situ* Al–TiB₂ composites are clean and free of reaction products, ensuring direct contact on atomic scale and excellent bonding between particle and matrix. Significant improvement in mechanical properties with respect to those of monolithic alloy has been noticed in the in situ processed Al-TiB2 based DRA composites.11,12 The strength of these composites are found to be higher than that of monolithic alloy due to both matrix grain refinement as well as presence of well bonded reinforcement/matrix interfaces, which ensures efficient load transfer from matrix to particle.¹⁰⁻¹⁴

One of the major limitations of the metal matrix composites is their lower tensile elongation to failure and fracture toughness^{2,15-18} than those of the monolithic alloy, which in turn is responsible for their poor formability. Metal forming in mushy state (partly liquid state) is promising for manufacturing of the Al alloy based components, because of the following reasons:¹⁹⁻²⁵

- (i) the constraints imposed by grain boundaries to deformation in solid state is absent in mushy state, and this reduces the flow stress and improves the formability significantly
- (ii) microstructural refinement with solute redistribution occurs, accompanied by formation of an equiaxed grain structure.

Thus, mushy state rolling process is particularly relevant for working of materials, which are either difficult to form through conventional methods, or require refinement of microstructure.

There are quite a few reports in the literature²⁶⁻³¹ on thixo formed Al alloys and composites, indicating that their strength and ductility are greater than those found in as cast materials. Such improvement in mechanical properties is attributed to reduction in the porosity content along with microstructural refinement and increase in chemical homogeneity. The evolution of structure and size distribution of the matrix grains as well as the accompanying changes in hardness of the Al-4.5Cu alloy or Al-4.5Cu-5TiB₂ (wt-%) composite plates mushy state rolled to different thickness reductions at temperatures corresponding to 10-30 vol.-% nominal liquid content f_1 , has been discussed in detail in a previous publication by the authors.³² It has been reported that dendritic structure in as cast alloy and composite is readily converted to grains with globular

216

¹Department of Mechanical Engineering, National Institute of Technology, Surathkal 575025, Karnataka, India ²National Metallurgical Laboratory, Jamshedpur 831007, India

³Central Research Facility, Indian Institute of Technology, Kharagpur 721302, India

⁴Indian Institute of Technology, Bhubaneswar, India

⁵Department of Metallurgical and Materials Engineering, Indian Institute of Technology, Kharagpur 721302, India

^{*}Corresponding author, email rahul@metal.iitkgp.ernet.in

shape on mushy state rolling. However, one of the major hurdles for mushy state rolling of Al alloy-TiB₂ composite is alligatoring caused by inhomogeneous deformation and strain localisation, which could be overcome to a significant extent by prior hot rolling. Experimental measurements^{33,34} have shown that both hardness and wear resistance increase in the following order: as cast composite<mushy state rolled composites<prehot rolled mushy state rolled composites, indicating that prehot rolling before mushy state rolling is beneficial. Furthermore, it should be noted that tensile properties of these mushy state rolled composites have not been investigated so far, but should be evaluated to judge the suitability of these materials for various structural applications. The present paper is focused on the comparative study of the tensile properties and failure mechanisms of cast Al-4.5Cu alloy and in situ Al- $4 \cdot 5Cu - 5TiB_2$ composite, some samples of which have been subjected to mushy state rolling in either as cast or in prehot rolled conditions. The major objectives include correlation of microstructure (grain size and morphology) and chemical heterogeneities with tensile strength, elongation and failure mechanism; as well as determination of process parameters for achieving optimum mechanical properties.

Experimental procedure

Liquid metallurgy processing

Al-4.5 wt-%Cu alloy was prepared by melting Al-33 wt-%Cu master alloy with suitable amount of Al to obtain the alloy with the desired composition. Furthermore, Al-4.5Cu-5TiB₂ (wt-%) composites were manufactured by a process, in which the TiB₂ particles were formed *in situ* inside the molten Al through a mixed salt route^{32,35} involving the reaction of K₂TiF₆ and KBF₄ salts inside the melt. A graphite crucible housed inside a pit furnace was used to melt process the composite, which was subsequently stir cast into plate shaped cast iron moulds.

Mushy state rolling

The specimens with dimensions of $40 \times 25 \times 6$ mm were soaked in a portable furnace at temperatures (accuracy $\approx \pm 2^{\circ}$ C) corresponding to mushy zone and subsequently rolled in a two high rolling mill, using a procedure explained in detail in an earlier publication.³² The operating conditions including temperature (for different values of f_i) and thickness reduction amounts used for mushy state rolling of alloy and composite samples used in this study are shown in Table 1. The temperatures for mushy state rolling were selected by using lever rule applied to the Al–Cu binary phase diagram,³⁶ for the alloy and adding the amount of TiB₂ particles to solid volume fraction for the composites. In



1 Dimensions of specimen used for tension test

an earlier study, the average liquid volume fractions at the temperatures chosen for mushy state rolling were experimentally confirmed through quenching of samples after isothermal soaking following the procedure used by Tzimas and Zavalingos,³⁷ followed by quantitative image analyses.³² The experimentally obtained values of average liquid content were found to be reasonably close to those estimated from the binary phase diagram, and considered as nominal in this study. However, it should be noted that the liquid is inhomogeneously distributed in the as cast alloy and composite during heating in mushy zone, because of the uneven solute (Cu) concentration in the dendritic Al alloy matrix, and due to preferred melting at grain boundaries and triple points. Some of the as cast composite plates were hot rolled at 370°C to 2.5% reduction in thickness, before mushy state rolling for achieving greater homogeneity in solute distribution, which in turn is expected to increase the degree of uniformity of liquid content during mushy state rolling.

Microstructural characterisation

The as cast and mushy state rolled samples of alloy and composite were sectioned along the long transverse plane, metallographically polished and etched with Keller's reagent for microstructural examination. Images showing the matrix grains were recorded on the optical microscope (model: Leica DFC320, Germany) and were subsequently analysed using the Leica QWin V3 software.³² Mean grain sizes and standard deviations were measured by considering ~100 grains for each of the investigated materials using image analysis following the procedure in ASTM E-112-96.³⁸

Tensile testing

Tensile testing was carried out on a Hounsfeld Materials Testing Machine (model: the S-series H10KS/05, UK) at a nominal strain rate of 5.0×10^{-4} s⁻¹, and elongation was measured with a non-contact type laser extensometer (model: 500LC, Tenius Olsen Ltd, USA. Tests were carried out on flat specimens with dimensions shown in Fig. 1, following ASTM standard E-8M.³⁹ Subsequently,

Table 1 Conditions of mushy state rolling used for AI-4.5Cu alloy and AI-4.5Cu-5TiB₂ composite

	Mushy state rolling parameters						
Material	Reduction <i>R</i> , %	Temperature of rolling, $^{\circ}$ C	Nominal liquid content f _l , %				
Al-4.5Cu alloy	0, 2.5, 5	631	30				
As cast Al-4.5Cu-5TiB ₂	0, 2.5, 5	632	30				
Prehot rolled Al-4·5Cu-5TiB ₂	5	610, 626 and 632	10, 20 and 30				



2 Optical micrographs showing a dendritic structure in as cast alloy and b rosette like grain structure in as cast composite

the fractured surfaces were examined on a scanning electron microscope (SEM) (model: JEOL JSM 5800, Japan) using both secondary (SE) and backscattered electron imaging techniques. Furthermore, chemical compositions at specific locations were determined using energy dispersive spectroscopy (EDS) facility (Oxford Instruments, UK) attached to the SEM.

Results and discussion

Microstructure

Optical micrographs depicting the typical microstructure of the as cast alloy and composite are shown in Fig. 2. The optical micrograph of the alloy (Fig. 2*a*) exhibits a typical dendritic structure, while that of as cast composite (Fig. 2*b*) reveals a rosette-like irregular grain structure. The growth of dendrites in the composites is obstructed due to the presence of TiB₂ particles. Using image analysis, the average size and volume fraction of TiB₂ particles in the composite samples have been found as $1.5 \ \mu m$ and 3% respectively. Coarse particles of CuAl₂ could be observed at interdendritic locations in the as cast alloy and composite, when observed at higher magnification.³²

Optical micrographs depicting the typical microstructure of the alloy and composite, subjected to mushy state rolling with $f_1=30\%$ and thickness reduction of 5% are shown in Fig. 3. Microstructure of the mushy state rolled alloy (Fig. 2a) shows both irregular and equiaxed grains, while that of the composite (Fig. 2b) shows grains with globular shape. Microstructural studies³² of the sections along the transverse direction in the long transverse plane



3 Optical micrographs showing microstructure of a Al-4⋅5Cu alloy subjected to mushy state rolling to thickness of 5% at 631°C (f₁=30%) and b Al-4⋅5Cu-5TiB₂ composite, subjected to mushy state rolling to thickness reduction of 2⋅5 at 632°C (f₁=30%)

(perpendicular to the plane of rolling and containing the rolling direction) have shown presence of bimodal grain size distributions. Coarsening of unmelted grains during mushy state rolling contributes to formation of the relatively larger grains, while rapid solidification of the liquid, dynamic recrystallisation and grain fragmentation lead to formation of the finer grains. The average sizes of coarse and fine grains having bimodal distribution in the investigated alloy and composite samples, subjected to mushy state rolling under the conditions mentioned in Table 1, are shown in Tables 2–4. The average grain sizes of the composite are much smaller than those observed in the alloy, as the TiB₂ particles effectively pin the grain boundaries, inhibiting grain growth during soaking and deformation in mushy state.

Tensile properties

The tensile properties, namely yield strength (YS), ultimate tensile strength (UTS), ratio of UTS to YS (UTS/YS) and elongation to failure (El) of the investigated alloy and composite samples in either as cast or mushy state rolled conditions are shown in Tables 2 and 3 respectively, while those of the composite mushy state rolled after prior hot rolling are presented in Table 4. The tensile properties shown are average of the results from two tests. The Vickers hardness data from an earlier publication are shown for the purpose of comparison. The increments in mechanical properties of the alloy and composites on subjecting to mushy state

Table 2 Grain size, hardness and tensile properties of as cast and mushy state rolled AI-4.5Cu alloy*

R, %	f _I , %	Grain size, μm		Hardness, HV	YS, MPa	UTS, MPa	UTS/ YS	Elongation, %
0		Dendritic		72±1	116 <u>+</u> 2·4	169±2·5	1.46	7.2 ± 0.3
2.5	30	329 ± 204 (coarse)	158±91 (fine)	$77 \pm 2.5 (+6.9\%)$	124±2·8 (6·9%)	174±2·3 (3·0%)	1.40	6·3±0·4 (-13·2%)
5	30	363 ± 225 (coarse)	157 ± 86 (fine)	63±2 (-12·5%)	97±2·9 (-16·4%)	147±2·4 (-13·0%)	1.51	5·1±0·4 (-29·4%)

*Increments in mechanical properties with respect to those of the as cast alloy are shown in parenthesis. '*R*' and '*f*_i' stand for thickness reduction and nominal volume fraction of liquid respectively.

Table 3 Grain size, hardness and tensile properties of *in situ* Al-4·5Cu-5TiB₂ composite subjected to mushy state rolling in as cast condition*

R, %	f _I , %	Grain size, μm		Hardness, HV	YS, MPa	UTS, MPa	UTS/ YS	Elongation, %
0		Dendritic		78±1	178±2·9	236 ± 2.8	1.33	9.7 ± 0.3
2.5	30	66 ± 15 (coarse)	37±10 (fine)	88±2·5 (12·8%)	195±2·8 (9·6%)	264±2·3 (11·9%)	1.35	12·1±0·3 (25·3%)
5	30	55 ± 14 (coarse)	32 ± 10 (fine)	96±2 (23·1%)	224±2·4 (25·8%)	277±2.6 (17.4%)	1.24	14·1±0·4 (45·0%)

*Increments in mechanical properties of the composite due to mushy state rolling are shown in parentheses. '*R*' and '*f*₁' stand for thickness reduction and nominal volume fraction of liquid respectively.

rolling are also shown in Tables 2–4. The effect of precipitation hardening by natural aging has been ignored in the present study as little change in hardness of matrix could be observed even after different periods of exposure of the samples at room temperature.³² In general, the tensile properties of the alloy and composite in as cast and mushy state rolled condition are in agreement with the broad range of values for thixocast or rheocast Al alloys and composites with similar compositions and volume fractions of particulate reinforcement respectively.^{28–30} Higher strengths have been reported for heat treated Al alloys and composites with similar matrix compositions,^{26,27} which indicates that the mechanical properties could be further improved by aging of the investigated composites.

Al-4.5Cu Alloy

On examination of the data in Table 2, the following can be inferred about the effects of mushy state rolling on tensile properties of the alloy:

- (i) the YS and UTS increase marginally on 2.5% thickness reduction, but are significantly reduced in the samples subjected to 5% reduction, in comparison to the values recorded for the as cast alloy
- (ii) the variations of YS and UTS with thickness reduction are similar to that of hardness
- (iii) the ratio of UTS to YS lies between 1.4 and 1.5
- (iv) the elongation decreases with increasing thickness reduction.

The marginal increase in strength on 2.5% thickness reduction is attributed to refinement of dendritic

microstructure to formation of globular grains. The decrease in both strength and elongation with 5% thickness reduction is because of grain growth during mushy state rolling, as is obvious from the grain size data in Table 2. Hence, the above results lead to the inference that mushy state rolling is detrimental for the Al–4.5Cu alloy, and this has been further discussed using the results of SEM fractography in the section on 'Fracture surfaces of alloy'.

Al-4.5Cu-5TiB₂ composite

On examination of the data in Tables 3 and 4, the following can be inferred about the trends observed for the YS, UTS and % El of the composites:

- (i) these are significantly higher in the composites mushy state rolled in either as cast or prehot rolled condition than those in the as cast composite samples
- (ii) on rolling at the temperature for $f_1=30\%$, these properties are improved as the thickness reduction is increased from 0 to 5%
- (iii) for 5% reduction in thickness, the YS and UTS of the composite mushy state rolled at the temperature corresponding to $f_1=20\%$ is the highest, while % El increases with volume fraction of liquid present during mushy state rolling.

Correlation of the mechanical properties with grain size is made complicated by the bimodal distribution of grain sizes in the α -Al matrix. It is intuitive that improvement in mechanical properties of the composite by mushy state rolling is caused by microstructural refinement

Table 4 Grain size, hardness and tensile properties of *in situ* Al-4·5Cu-5TiB₂ composite subjected to mushy state rolling after prior hot rolling*

R, %	f _l , %	Grain size, μm		Hardness, HV	YS, MPa	UTS, MPa	UTS/ YS	Elongation, %
0 5	 10 20	Dendritic 42 ± 16 (coarse) 41 ± 15 (coarse)	26±11 (fine) 25±12 (fine)	78±1 104±2 (33·3%) 112±1·5 (43·6%)	178±2·9 189±2·5 (6·2%) 219±2·4 (23·1%)	236±2·8 239±2·7 (1·3%) 276±2·0 (16·9%)	1·33 1·26 1·26	9·7±0·3 11·3±0·3 (16·1%) 13·5±0·3 (39·3%)
	30	46 ± 17 (coarse)	24 ± 11 (fine)	99±2 (26·9%)	214±2 (20·2%)	273±2·9 (15·7%)	1.27	14·4±0·3 (48·1%)

*Increments in mechanical properties of the composite due to mushy state rolling are shown in parentheses. '*R*' and '*f*₁' stand for thickness reduction and nominal volume fraction of liquid, respectively.

involving evolution of globular grain shape (Figs. 2 and 3) and decrease in the average grain sizes. Comparison of the data in Table 3 suggests that hardness, YS, UTS and % El are higher in the composite samples with smaller average grain sizes, while examination of the data in Table 4 indicates that there are exceptions. Such exceptions are observed because strain hardening caused by deformation of the grains remaining unmelted during mushy state deformation also leads to increase in hardness and strength of the investigated composites, provided grain growth is prevented. For a given thickness reduction, the composite mushy state rolled with $f_1=20\%$ exhibits maximum hardness, YS and UTS (Table 4), probably due to optimum combination of the effects of work hardening of solid skeleton and fine grains formed by the rapid solidification of the liquid. However, the % El increases continuously with increasing the volume fraction of the liquid during mushy state rolling. This increase in ductility may be attributed to increase in volume fraction of fine grains formed by rapid solidification of the higher liquid content.

The UTS/YS ratio is found to be 1.33 for the as cast composite and is reduced marginally to the range of 1.24–1.27 on mushy state rolling. Examination of the data on UTS/YS ratios indicates that this parameter decreases with decreasing the average matrix grain size, and is lower in the composite than in the alloy for similar thermal or mechanical treatments. Low values of UTS/YS ratios imply that the process of recovery sets in at a lower strain, and counteracts against work hardening during the tension test. The smaller matrix grain size and the presence of undeformable reinforcements is responsible for a smaller mean free path for dislocation movement and increase in the dislocation density, which ensures that the process of recovery sets in at a lower strain.

Comparison of tensile data of alloy and composite

The values of YS, UTS and %El of the alloy and composite in as cast and mushy state rolled conditions are compared in the plots shown in Fig. 4. On examination of the plots (Fig. 4), it is clear that:

- (i) the mechanical properties of the composite are superior to that of alloy
- (ii) the difference between the YS, UTS and %El of composite and alloy increases with increasing thickness reduction during mushy state rolling.

This increase in difference of the tensile properties with reduction during mushy state rolling is attributed partly to reduction of the average matrix grain size of the composite and its increase in case of the alloy (Tables 2–4). The TiB_2 particles play a significant role in strengthening, presumably both by acting as obstacles to dislocation motion and by restricting grain growth in the composite during mushy state rolling. Interestingly, the %El of the composite is two and three times that of the as cast alloy for mushy state rolling at the temperature for $f_1=30\%$ for 2.5 and 5% thickness reductions respectively. This is an unexpected observation, as ductility of the composites is usually found to be lower than that of the unreinforced alloy, and this can be explained on the basis of both finer average grain size in the former material and results of fractographic studies discussed in the section on 'Study of fracture surfaces'.



4 Plots showing variation of a YS, b UTS and c elongation to failure with thickness reduction for mushy state rolled Al-4.5Cu alloy and its composite

Study of fracture surfaces

Fracture surfaces of alloy

Typical SEM-SE images depicting the fracture surfaces of the tension tested as cast alloy are shown in Fig. 5. While Fig. 5a shows tear ridges and dimples confirming ductile fracture, Fig. 5b indicates the presence of dendritic nodules inside a typical shrinkage cavity at a relatively higher magnification. Hence, it may be



a dimples; b shrinkage cavity and dendritic tip
Images (SEM-SE) of tensile fracture surfaces in Al-4.5Cu alloy tested in as cast condition (one of dimples is marked 'D' in a, while dendritic tip and cavity are marked as 'DT' and 'C' in b)

inferred that shrinkage cavities are responsible for failure initiation, and cracks propagate preferentially through interdendritic separation in as cast alloy.

In contrast to the dimpled fracture surface morphology (Fig. 5a) observed for the as cast alloy, SEM examination of the fracture surfaces of the mushy state rolled alloy samples has shown evidence of brittle failure, with the morphology of fracture surface dependent on amount of thickness reduction. A typical SEM-SE fractograph of the alloy subjected to mushy state rolling at 631°C (f_1 =30%) for 2.5% thickness reduction, as presented in Fig. 6, shows presence of facets and river patterns as evidence of transgranular brittle failure. However, Fig. 6 also shows the presence of fine dimples at a few places indicating that ductile fracture has occurred locally. Typical SEM fractographs of the alloy samples mushy state rolled at 631°C $(f_1=30\%)$ for thickness reduction of 5%, presented in Fig. 7a and b at low and high magnification respectively, show distinct evidence of complete intergranular fracture. The presence of secondary cracking along the grain boundaries, is obvious in the higher magnification micrograph (Fig. 7b). The intergranular failure observed in case of Al-4.5Cu alloy mushy state rolled to 5% thickness reduction, suggests that grain boundary embrittlement has taken place and is primarily responsible for the decrease in its % El to failure by $\approx 30\%$.

The cause for embrittlement of the alloy mushy state rolled to 5% thickness reduction has been investigated



6 Images (SEM-SE) of tensile fracture surfaces of mushy state rolled AI–4·5Cu alloy at 631°C (f₁=30%) to 2·5% thickness reduction (evidence of transgranular failure is observed; river patterns and grain facets, characteristics of transgranular cleavage fracture are shown as 'R' and 'F'; localised dimples are surrounded by an ellipse)

further through SEM and EDS analysis of the matrix grain boundaries, and the results are shown in Fig. 8. A typical grain boundary triple point in the fracture surface of the Al-4.5Cu alloy subjected to mushy state rolling at 631°C (f_1 =30%) to 5% thickness reduction is



7 Images (SEM-SE) of tensile fracture surfaces of mushy state rolled AI-4.5Cu alloy at 631°C (f_i=30%) to 5% thickness reduction at *a* low and *b* high magnifications (location encircled in *a* is magnified in *b*; evidence of intergranular failure is observed)





a SEM-SE image showing grain boundary triple point; b SEM-SE image showing magnified view of eutectic present in triple point; c EDX spectrum taken from spot marked as 'X' in b

8 Tensile fracture surfaces of mushy state rolled Al–4.5Cu alloy at 631 $^{\circ}$ C (f_{i} =30%) to 5% thickness reduction

shown in Fig. 8*a*. A magnified view of the phases present at the intergranular locations (Fig. 8*b*) clearly shows a eutectic structure. The corresponding EDS spectrum from the location marked as X in Fig. 8*b*, is shown in Fig. 8*c*. The EDS quantitative analysis based on the spectrum (Fig. 8*c*) shows ~ 50 wt-%Cu, suggesting a hypereutetectic composition with primary CuAl₂ and eutectic CuAl₂– α -Al phases. It may be noted that the eutectic point in the binary Al–Cu phase diagram³⁶ occurs at the composition with 32 wt-%Cu at 548·2°C. Thus, it may be inferred that presence of primary CuAl₂ and eutectic between the α -Al grains and at triple points leads to grain boundary embrittlement in composites mushy state rolled at temperature for f_1 =30% to 5% thickness reduction.

The enrichment of Cu and presence of primary CuAl₂ and eutectic at the grain boundaries and triple points



9 Images (SEM-SE) of tensile fracture surfaces of as cast AI-4.5Cu-5TiB₂ composite at a low and b high magnifications

can be explained on due consideration of the thermomechanical history of the alloy samples. Before rolling, the alloy was soaked in mushy zone for 10 min, which allows the unmelted grains in solid skeleton to grow in an unabated manner with solute rejection into the liquid. The formation of high volume fraction of the brittle intermetallic compound, CuAl₂ and trapped eutectic at the grain boundaries and triple points is attributed to solidification of solute rich hypereutectic liquid. The presence of α -Al–CuAl₂ eutectic at interdendritic locations of as cast Al–4·5Cu alloy has also been reported in an earlier study.³¹

Fracture surfaces of composite

Investigation of tensile fracture surfaces of both as cast and mushy state rolled composite samples has shown evidence of ductile failure. Typical SEM fractographs of the composite tested in as cast and mushy state rolled conditions are shown in Figs. 9 and 10 respectively at different magnifications. The SEM images recorded at low (Fig. 9a) and high magnifications (Fig. 9b) of fracture surfaces of the as cast composite show dimples and does not show dendrite tips, unlike the features observed in course of fractographic studies of the as cast alloy (Fig. 5b). Figure 10a and b shows SEM images of fracture surfaces generated by tension tests of the composites mushy state rolled to 2.5 and 5% reduction respectively. These SEM images show dimples as well as tear ridges as evidence of ductile failure. The dimpled morphology in the composite mushy state rolled to 5% reduction is shown at a higher magnification in Fig. 10c.



a mushy state rolled at 632°C (f_1 =30%) to 2.5% thickness reduction; *b*,c mushy state rolled at 631°C (f_1 =30%) to 5% thickness reduction

10 Images (SEM) depicting tensile fracture surfaces of AI-4.5Cu-5TiB₂ composite

In the higher magnification SEM images of Figs. 9*b* and 10c, it is possible to observe large dimples containing TiB₂ or CuAl₂ particles, as well as smaller dimples in the alloy matrix, resulting in bimodal size distribution of dimples. Observation of either TiB₂ or CuAl₂ particles inside the dimples suggest that the voids are initiated at particle matrix interfaces. The smaller dimples form in the matrix between the TiB₂ particles due to extensive stress concentration, and flow localisation at casting defects such as porosities. The presence of smaller dimples inside the larger ones suggests that dimple growth has taken place by coalescence of smaller dimples. The mechanism of ductile fracture through void nucleation around inclusions or defects, followed

by their linkage is well established.^{15,40,41} Interestingly, examination of the fracture surfaces at low magnification (Fig. 10*b*) indicates that many of the relatively larger dimples in the fracture surfaces of the mushy state rolled composites appear elliptical. The elliptical shape of voids could be due to two possible reasons as follows:

- (i) coalescence of smaller voids formed around the TiB₂ or CuAl₂ particles present in the form of networks at the matrix grain boundaries
- (ii) presence of elongated grains formed during mushy state rolling.³²

The ductile, dimpled fracture surfaces observed for all types of mushy state rolled Al-4.5Cu-5TiB₂ composite are morphologically in sharp contrast to the brittle intergranular fracture surfaces observed in case of the alloy, mushy state rolled to 5% reduction in thickness. It is interesting that extensive segregation of Cu could not be observed at the grain boundaries unlike that found in case of the alloy. Similar observations have been reported in previous studies³² involving isothermal soaking in the mushy zone and quenching. In the composite, the dendrite arms tend to be shorter and closer to one another than those in the alloy, with only limited Cu segregation at the tips. Martinez and Flemings⁴² have reported that solute concentration is increased sharply only beyond a radial distance of 20 µm from the centre of solid particles, in the case of the Al-4.5 wt-%Cu alloy. The results of studies on microstructure and composition have shown that the distribution of solute is more uniform after hot or mushy state rolling than in as cast condition.^{32,35}

Conclusions

The tensile properties and fracture mechanisms of the Al-4·5Cu alloy and Al-4·5Cu-5TiB₂ composite samples have been compared. The major conclusions from the present study are:

1. mushy state rolling causing thickness reduction in the range of 2.5-5% at the temperature for $f_1=30\%$ is beneficial for improvement in mechanical properties of the *in situ* composite reinforced with TiB₂ particles, but is severely detrimental for the alloy

2. for thickness reduction of 5%, the hardness and strength are found to be the highest on mushy state rolling at the temperature corresponding to $f_1=20\%$, indicating optimised contributions of work hardening and volume fraction of fine grains

3. while ductile failure has been observed in the as cast alloy and composite specimens, as well as in mushy state rolled composites, brittle transgranular and intergranular fracture has been found in case of mushy state rolled alloys.

The tensile properties in Al-4·5Cu alloy are deteriorated on mushy state rolling due to grain growth and grain boundary embrittlement by segregation of Cu and formation of primary CuAl₂ and CuAl₂– α -Al eutectic at intergranular and triple point locations. On the other hand, spherodisation of the originally rosette shaped grain structure, prevention of grain growth by TiB₂ particles during mushy state processing, reduction in average grain size by rapid solidification of liquid and dynamic recrystallisation are responsible for higher YS, UTS and % El in the composite samples than in the alloy. The *in situ* formation of only 5 wt-% (or 3 vol.-%) TiB₂ serves the twin objectives of particle strengthening as well as inhibition of grain growth, and hence contributes to significant improvement in mechanical properties on mushy state rolling. The enhancement of both strength and ductility on mushy state rolling of composites is encouraging, as it indicates an increase in toughness of the composite. Hence, mushy state rolling is a viable process for deformation processing of cast Al alloy based composites with thermodynamically stable ceramic reinforcements for further improvement in mechanical properties.

Acknowledgements

The authors wish to acknowledge the valuable technical assistance rendered by Mr K. Saw for processing of the materials. The authors would also like to thank Dr R. N. Ghosh, NML, Jamshedpur for extending tensile testing facility for carrying out the tensile experiments.

References

- 1. K. L. McDanels: Metall. Trans. A, 1985, 16A, 1105-1115.
- S. V. Nair, J. K. Tien and R. C. Bates: Int. Met. Rev., 1985, 30, (6), 275–290.
- A. V. Smith and D. D. L. Chung: J. Mater. Sci., 1996, 31, 5961– 5973.
- K. K. Chawla: 'Composite materials science and engineering', 2nd edn, 164; 1998, New York, Springer.
- 5. T. Clyne and P. Withers: 'An introduction to metal matrix composites'; 1993, Cambridge, Cambridge University Press.
- 6. D. B. Miracle: Compos. Sci. Technol., 2005, 65, 2526–2540.
- W. H. Hunt, Jr: 'Metal matrix composites: applications', in 'Encyclopedia of materials: science and technology', (ed. K. H. Jürgen Buschow et al.), 5442–5446; 2008, New York, Elsevier.
 G. Hirt, R. Cremer, T. Witulski and H.C. Tinius: *Mater. Design*,
- G. Hirt, R. Cremer, T. Witulski and H.C. Tinius: *Mater. Design*, 1997, 18, (4/6), 315–321.
- 9. J. E. Allison and G. S. Cole: JOM, 1993, 45, (1), 19-24.
- R. Mitra, W. A. Chiou, M. E. Fine, J. R. Weertman: J. Mater. Res., 1993, 8, 2380–2392.
- A. K. Kuruvilla, K. S. Prasad, V. V. Bhanuprasad and Y. R. Mahajan: Scr. Metall. Mater., 1990, 24, (5), 873–878.
- J. V. Wood, P. Davies and J. L. F. Kellie: *Mater. Sci. Technol.*, 1993, 9, 833–840.
- 13. K. L. Tee, L. Lu and M. O. Lai: Wear, 2000, 240, 59-64.
- 14. R. Mitra and Y. R. Mahajan: Bull. Mater. Sci., 1995, 18, (4), 405-434.
- 15. D. L. Davidson: Metall. Trans. A., 1987, 18A, 2115-2128.
- M. Manoharan and J. J. Lewandowski: *Mater. Sci. Eng. A*, 1992, A150, 179–186.

- A. F. Whitehouse and T. W. Clyne: Acta Metall. Mater., 1993, 41, (6), 1701–1711.
- 18. M. Song and D. Xiao: Mater. Sci. Eng. A, 2008, A474, 371-375.
- M. Kiuchi, S. Sugiyama and K. Arai: J. Jpn Soc. Technol. Plast., 1982, 23, 915–923.
- M. Kiuchi: 'Metal forming in mushy state', in 'Plasticity and modern metal-forming technology', (ed. T. Z. Blazynski), 289–313; 1989, London, Elsevier Applied Science.
- 21. M. Kiuchi: Ann. CIRP, 1991, 40, (1), 259-262.
- 22. W. Lapkowski: J. Mech. Work. Technol., 1989, 18, 305-313.
- 23. W. Lapkowski and J. Sinczak: J. Mater. Proc. Technol., 1992, 32, 271–277.
- W. Lapkowski, J. Sinczak and S. Rusz: J. Mater. Proc. Technol., 1997, 63, 260–264.
- 25. M. C. Flemings: Metall. Trans. A, 1991, 22A, 957-981.
- W. G. Cho and C. G. Kang: J. Mater. Proc. Technol., 2000, 105, 269–277.
- S. W. Youn, C. G. Kang and P. K. Seo: J Mater. Process. Technol., 2002, 130–131, 574–580.
- S. V. Kamat, J. P. Hirth and R. Mehrabian: *Acta Metall.*, 1989, 37, (9), 2395–2402.
- 29. S. Skolianos: Mater. Sci. Eng. A, 1996, A369, 76-82.
- E. J. Zoqui and M. H. Robert: J. Mater. Proc. Technol., 1998, 78, 198–203.
- J. L. Jorstad, Q. Y. Pan and D. Apelian: *Mater. Sci. Eng. A*, 2005, A413–A414, 186–191.
- M. A. Herbert, C. Sarkar, R. Mitra and M. Chakraborty: *Metall.* Mater. Trans. A, 2007, 38A, 2110–2126.
- M. A. Herbert, R. Maiti, R. Mitra and M. Chakraborty: Solid State Phenom., 2006, 116–117, 217–220.
- M. A. Herbert, R. Maiti, R. Mitra and M. Chakraborty: Wear, 2008, 265, (11–12), 1606–1618.
- 35. M. A. Herbert: 'Some studies on the mushy state rolling of Al-4·5Cu alloy based in-situ composites reinforced with TiB₂ or TiC particles', PhD thesis, Indian Institute of Technology, Kharagpur, West Bengal, India, 2007.
- 36. in 'ASM handbook', Vol. 3, 1·23–2·44; 1992, Materials Park, OH, ASM International.
- 37. E. Tzimas and A. Zavaliangos: J. Mater. Sci., 2000, 35, 5319-5329.
- 'Methods for determining the average grain size', E112-96, ASTM, Philadelphia, PA, USA, 1996.
- 'Standard test methods for tension testing of metallic materials', E8M-08, ASTM, Philadelphia, PA, USA, 2008.
- 40. G. E. Dieter: 'Mechanical metallurgy', 3rd edn, 586; 1986, New York, McGraw-Hill Book Co.
- L. M. Brown and J. D. Embury: 'The initiation and growth of voids at second phase particles', Proc. 3rd Int. Conf. on 'Strength of metals and alloys', 164–169; 1973, London, Institute of Metals.
- R. A. Martinez and M. C. Flemings: *Metall. Mater. Trans. A*, 2005, 36A, 2205–10.