JOURNAL OF MATERIALS SCIENCE LETTERS 11 (1992) 452–456

Fracture toughness evaluation of aluminium 4% Mg–Al_2O_3 liquid-metallurgy particle composite

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Composites are a class of modern engineered materials that can be tailored to exhibit certain desired properties required for engineering applications. Development in the area of composite materials [1] has taken rapid strides in the past decade or so for a variety of applications which not only aim at increasing the strength-to-weight ratio, but also are very energy-efficient. In addition, it is encouraging to note that there is no need [2] for any special techniques to be adopted for production; conventional routes can be made use of in a profitable manner.

Composite materials may contain any of polymer, metallic, intermetallic or ceramic matrix and fibres, whiskers or particles of a variety of strong dispersoids. Among metal-matrix composites (MMCs) the final mechanical property combinations depend on the nature and strength of the matrix–particle interface bonding. Among other things, methods have to be developed to characterize this new class of materials. Since the main application for lightweight composite materials is in the aircraft industry [3], there is urgent need for developing methods of evaluating the fracture toughness, K_{Ic}, of these materials. The presence of distributed second-phase particles may pose some problems in evaluating the K_{Ic} of these materials.

Other the past two decades or so very elaborate methods have been developed to characterize a material for its fracture toughness [4, 5]. Many of these methods are quite complicated and require very accurate machining of specimens and measurement techniques. Precracking is an important necessity. Often it becomes necessary to use simpler and/or extrapolation techniques to determine the fracture toughness quickly so that valuable time and effort can be saved. One such extrapolation has been tried by Tada *et al.* [6] and Hahn and Rosenfield [7] for aluminium-based alloys to relate the notch tensile behaviour to the fracture toughness. There have been attempts to obtain the same from impact tests or even from simple tensile tests [8]. Since the extent of application of such extrapolations has not been established, it is not known whether such evalutions apply to other systems. As a part of a larger programme on the evaluation of composites, this work was undertaken to determine the applicability of the indirect method of evaluation of fracture toughness for particle-dispersed aluminium matrix composites and to determine the credibility of such evaluations by actual experimental determinations.

Commercial-grade high-purity aluminium (> 99.97%) was used to make the composite materials for the test. Levigated Al_2O_3, normally used as an abrasive in metallography, was used as the dispersoid material. The individual particles were therefore angular in nature. The as-received sample was subjected to sieving and the −200 mesh fraction retained on +270 mesh was isolated and used. The minimum and maximum particle sizes of this fraction were 54 and 110 μm, respectively, and the average particle size was 73 μm. The as-sieved powder was preheated to the same temperature as the melt in each case before adding it to the melt. Mg of 99.9% minimum purity was taken in the form of cut pieces from ingots. All melts were made in an electrical resistance furnace using clay graphite crucibles, and were cast in preheated cast iron chill moulds coated with China clay. Both cylindrical (62 mm diameter × 200 mm long) and rectangular (90 mm × 35 mm × 150 mm) moulds were used. The melt was superheated to a temperature range of 740–780 °C.

Composite material preparation was done by the vortex method [9–13] by forming the vortex with an impeller and in 5 kg batches of various compositions. The melt was degassed with hexachloroethane tablets. The dross formed was skimmed out before Mg was added. The crucible was then transferred to the holder in the stirrer assembly to obtain a deep vortex. A weighed quantity of Al_2O_3 was added at the periphery of the vortex in a continuous stream while stirring was continued. The stirrer was then switched off and the impeller withdrawn from the melt. Using a preheated graphite rod, the melt was stirred manually before pouring into the moulds. The ingots thus produced were proof machined to check for the presence of any major casting defect. Analysis was carried out at three different points of the casting. The ingots were then homogenized at 500 °C for 30 h.

Cylindrical castings were machined into slugs 50 mm × 100 mm and were hot-extruded in a 250 t hydraulic press at a speed of 3.2 mm s^{-1} into 20 and 16 mm diameter extrudants to yield extrusion ratios of 6.5 and 10, respectively. A suitable length from the leading edge was discarded and the homogeneous central portion alone was used for property evaluation. Before evaluation the extruded material was solutionized at 480 °C for 6 h and then peak

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aged at 120 °C for 12 h [14].

Composites with various weight fractions of Al_2O_3 were characterized by the following tests: (a) tensile strength, (b) notched tensile strength, (c) fracture toughness on compact tension (CT) specimens and (d) hardness.

Tensile tests were performed on a 2 t Hounsfield tensometer using the specimen geometry shown in Fig. 1a. From the load–displacement curves, the ultimate tensile strength (UTS), 0.2% yield stress (YS) and percentage elongation were evaluated.

The residual strength of the material containing a notch was determined by performing notch tensile strength (NTS) tests on the specimens shown in Fig. 1b. The NTS values were calculated from the recorded load–displacement curve.

The fracture toughness behaviour of the material was determined by using a (CT) specimen geometry as shown in Fig. 1c. These tests were conducted on a servohydraulic Instrom testing machine model 8032 of capacity 10 t (dynamic). The specimen was fatigue precracked using a peak load of 500 kg at 35 Hz. A crack was initiated at the root of the chevron notch and allowed to propagate for a distance of 3 mm. The crack growth was carefully monitored by following the crack tip with a microscope. A 10 mm displacement gauge was fitted at the mouth of the notch. The specimen was then fractured by applying a continuously increasing tensile load. The crack length was measured at the centre of the crack front, midway between the centre and the end of the crack on each side using vernier calipers. The average

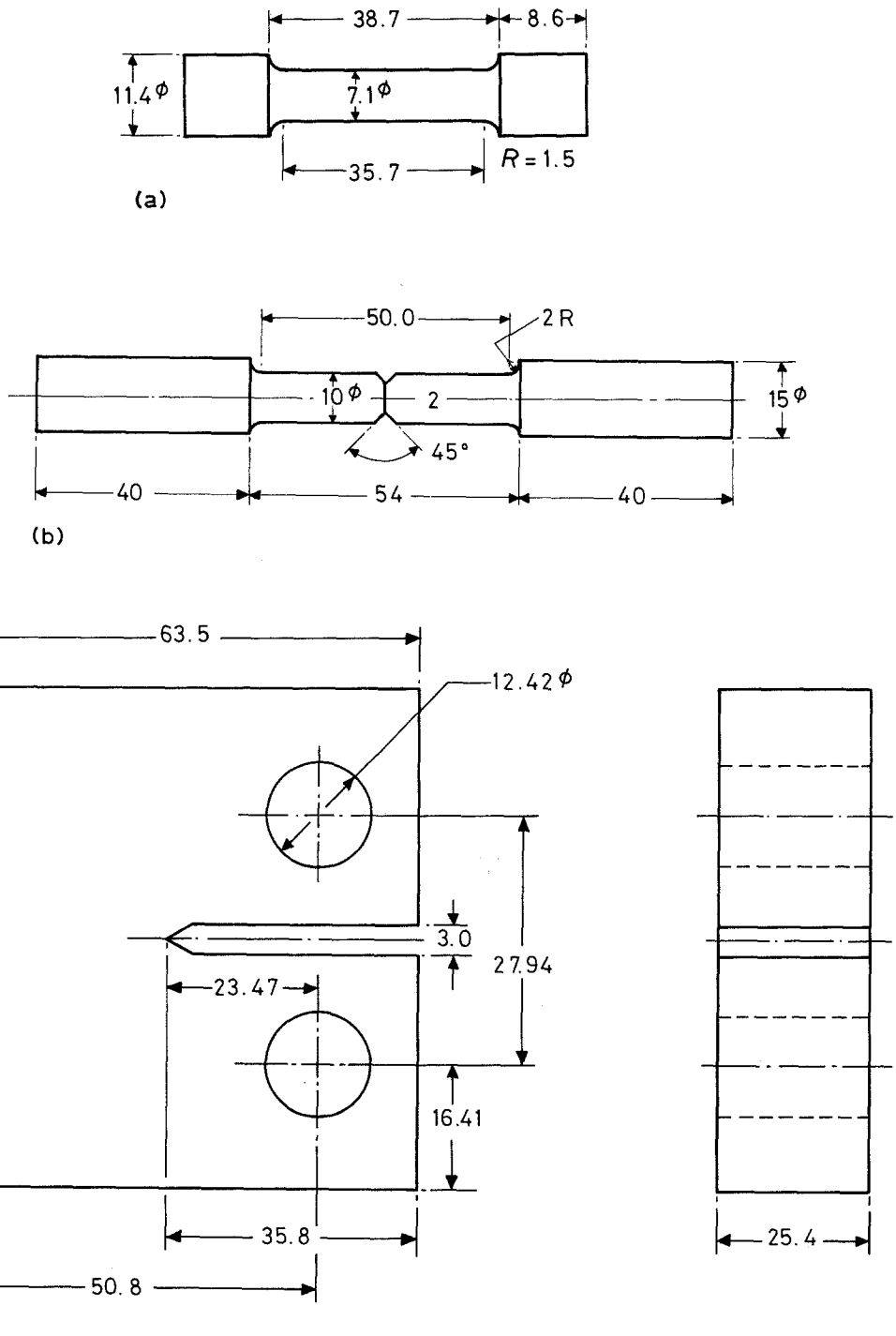

Figure 1 Specimen geometry: (a) Hounsfield specimen, (b) notch tensile specimen and (c) compact tension specimen.

values of these measurements was used as the crack length in subsequent calculations of the fracture toughness.

The following formula was used [15–17] to calculate K_Q, the crack resistance:

$$K_Q = \frac{P_Q}{B\,W^{1/2}}(2 + a/w)\,[0.886 + 4.64\,(a/w) - 13.32(a/w)^2 + 14.72(a/w)^3] - \frac{5.6(a/w)^4}{(1 - a/w)^{3/2}} \quad (1)$$

where a is the average crack length, w is the breadth of the specimen and P_Q is the 5% tangent load.

Brinell and Rockwell hardness values were determined for the as-cast material. As the size of the specimen was very small in the case of the extruded and heat-treated material, only Vickers hardness number (VHN) values were determined.

In spite of rigorous degassing of the melt, castings contained pinhole porosity. The inevitability of the occurrence of micropores has been reported by several investigators [10–12, 18]. Microscopic and macroscopic examination of the castings revealed that the distribution of alumina was reasonably good and uniform at both micro- and macro-levels (Fig. 2). This is also supported in the investigations of Mehrabian *et al.* [19] and Akira Sato and Mehrabian [20]. The tendency for coagulation or segregation was found to become prominent with increasing alumina content. The composites were easily extrudable with little or no difficulty. A striking feature observed in the extrusion of composites was that the optimum temperature for extrusion decreased with increasing alumina content until about 7%. Alumina particles act as potential sites for stress concentration and cracks originate at the particle–matrix interface.

On a systematic analysis of the mechanical behaviour, the following features were observed. Hardness values gradually increased with increasing alumina content. The reasonably uniform distribution of alumina was also a contributing factor for this increase. Fig. 3 shows the variation of hardness with Al_2O_3 content.

A better comparison of the material can be effected by analysing the mechanical properties. The values of percentage elongation measured in a tensile test are plotted in Fig. 4 for three experimental conditions. Ductility in the as-cast condition was very poor in all cases and decreased with increasing Al_2O_3 content. However, after extrusion the ductility increased significantly although the trend in the variation with Al_2O_3 was the same. It is to be noted that the hot-extrusion process breaks down the cast structure in the matrix, thereby increasing the ductility. It is significant to note that

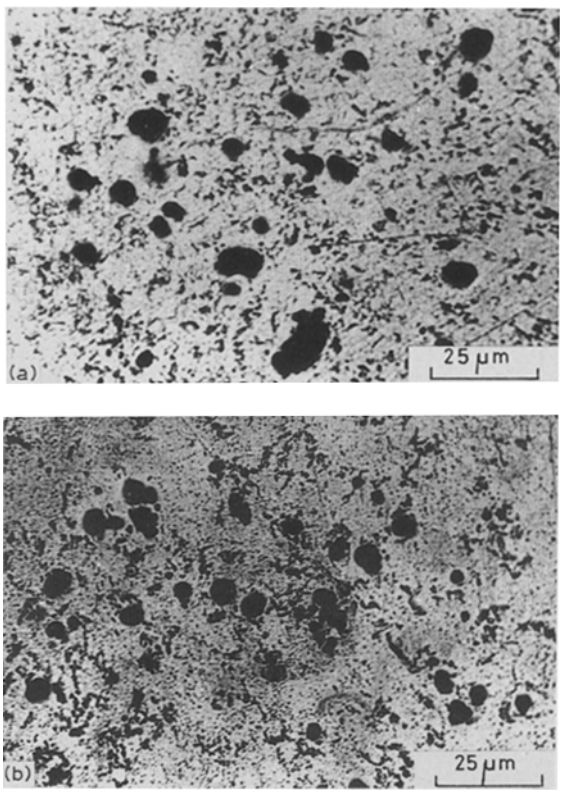

Figure 2 Typical microstructures of the Al–Al_2O_3 composite, showing good microscopic distribution of the particles: (a) 2% Al_2O_3 and (b) 3% Al_2O_3.

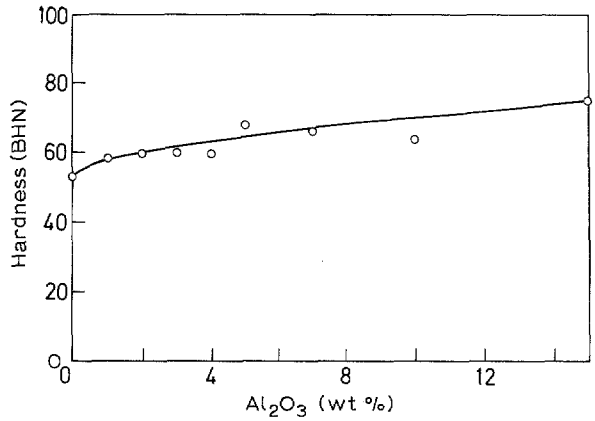

Figure 3 Variation of hardness as a function of Al_2O_3 content.

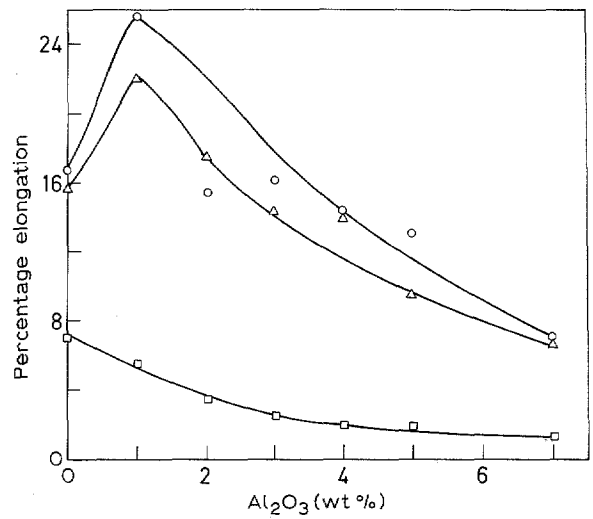

Figure 4 Variation of percentage elongation in (□) as-cast, (△) extruded and (○) extruded and heat treated condition as a function of the Al_2O_3 content.

the composite containing 1% Al_2O_3 showed maximum ductility, and this was observed to be higher than that exhibited by the matrix Al–4 Mg alloy. This was true even for the mechanical properties (Fig. 5), and the values of the UTS and NTS of the material in the three conditions. Considering only the UTS, the values are quite low for the as-cast condition. For the extruded material, in every composite the UTS observed was very large compared with that in the as-cast condition. The strength values increased up to 5% Al_2O_3 content and dropped considerably even in the extruded condition. The effect of solution heat treatment and ageing only increased the UTS marginally. This is because Al–4Mg alloy is not heat-treatable in the real sense. The NTS values are significant in that all of the composites show a fairly high value. The values after extrusion and heat treatment were again higher than those observed in the as-cast condition. However, the NTS/UTS ratio was greater than unity in all composites. This is explained from the fact that in all cases failure takes place by deformation and fracture of the matrix, which is quite ductile. Although the presence of a bonding phase at the particle–matrix interface is discernible, the Al_2O_3 particles do not apparently play on active part in supporting the applied stress. It is quite possible for the notched cross-section to have a lower particle density for fracture than the fractured area in the unnotched specimen. These two factors seem to be responsible for the NTS/UTS ratio being larger than unity.

Values of K_{Ic} have been calculated using the Tada *et al.* [6] and Hahn and Rosenfield [7] equations to make a comparison. In Table I a comparison is made of fracture toughness values obtained from experiments on CT samples. On comparing the experimental values with the calculated values, it is seen that the calculated values are higher but are of the same order of magnitude. The yield strength of the extruded material shows better mechanical properties than the as-cast material. However, since these values agree within experimental error, a check on this is possible only if Al–Al_2O_3 composite material is available in plate form. This is presently being attempted. K_{Ic}-values reported in Table I were obtained through the Tada–Irwin–Hahn [6, 7] equation and the experimental R-curve for the as-cast material, whereas for the extruded and heat-treated conditions the values of K_{Ic} have been evaluated only through the Tada *et al.* [6] and Hahn and Rosenfield [7] equation. This was because the geometry of the extrusion did not permit making a CT specimen. Fracture toughness values obtained from the use of the Tada *et al.* [6] and Hahn and Rosenfield [7] equations for the extruded and heat-treated conditions as well as those obtained experimentally for the as-cast specimen using CT specimens are given in Table I.

The experimental values agree with the calculated values within experimental error, supporting the use of either the R-curve method or the Tada–Irwin–Hahn [6, 7] equations for calculating K_{Ic}-values. The calculated values were found to be marginally higher than the experimental values. In terms of the fracture toughness, the composites containing 1–7% Al_2O_3 exhibit nearly the same K_{Ic}-values. This only confirms the fact that the dispersed particles are not

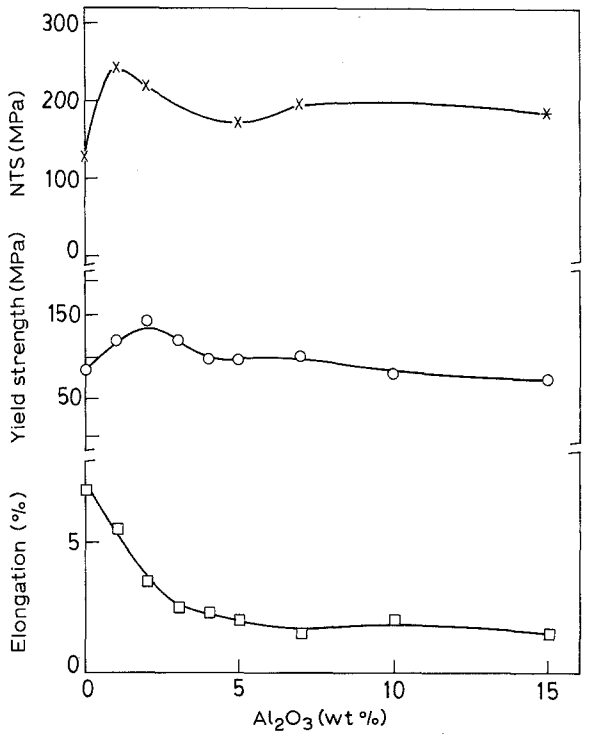

Figure 5 Mechanical properties of the Al–Al_2O_3 composite with varying Al_2O_3 content: notch tensile strength, yield strength and percentage elongation.

TABLE I Calculated values of fracture toughness for extruded and heat-treated, as-cast Al–4 Mg–Al_2O_3 composite material

Extruded and heat-treated						As-cast					
Al_2O_3 (wt %)	σ_{NTS} (MPa)	0.2 σ_{YS} (MPa)	$\frac{\sigma_{NTS}}{\sigma_{YS}}$ (NYR)	K_{Ic} from Tada equation (MPa $m^{1/2}$)	K_{Ic} from Hahn and Rosensfield equation (MPa $m^{1/2}$)	σ_{NTS} (MPa)	0.2% σ_{YS} (MPa)	$\frac{\sigma_{NTS}}{\sigma_{YS}}$ (NYR)	K_{Ic} from Tada equation (MPa $m^{1/2}$)	K_{Ic} from Hahn and Rosenfield equation (MPa $m^{1/2}$)	K_Q experimental (MPa $m^{1/2}$)
0	284	131	2.17	30.63							
1	349	164	2.13	38.31	33.32	241	75	3.2	18.77	22.53	15.5
2	280	141	1.99	32.75	34.68	217	87	2.5	21.31	21.62	10.5
5	268	136	1.97	31.55	39.68	168	81	2.1	19.00	17.9	10.35
7	130	191	1.43	20.59	34.33	192	72	2.7	17.00	16.00	11.95

taking part in the crack growth process. What is being measured on the CT specimens is perhaps the K_{Ic}-value for the matrix material with a certain amount of porosity. A recent study [21] shows that up to about 7% porosity had no effect on the K_{Ic}-values of weldments in 15 CDV steel.

In conclusion, in composite materials containing up to 15% Al_2O_3 in Al–4 Mg matrix successfully prepared by a liquid-metallurgy technique using levigated Al_2O_3 the presence of Mg in the matrix leads to a certain amount of bonding.

The mechanical properties in terms of percentage elongation, UTS and NTS increase slightly up to 2% addition of Al_2O_3 and decrease thereafter. The NTS/UTS ratio for all composites is more than unity. Use of the Tada–Hahn equations yields a slight overestimate of K_{Ic}, but of the same order of magnitude as experimental values.

Acknowledgements

The authors thank the Chairman, Department of Metallurgy, Indian Institute of Science, Bangalore, for the facilities provided and the Department of Mines for a financial grant under grant no. 14/28/89-Met. V of 11 June 1990, under which part of this work was carried out.

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Received 4 April
and accepted 25 April 1991