Hybrid aluminum matrix composites containing carbon nanotubes and zirconium diboride particles: fractography, microstructure and mechanical performance

Anas Farooqi¹ · Masood Ahmed¹ · Tayyab Subhani² · Syed Wilayat Husain¹

Received: 12 July 2019 / Accepted: 22 November 2019 / Published online: 27 November 2019
© Springer Nature Switzerland AG 2019

Abstract

Hybrid aluminum matrix composites (HAMCs) containing carbon nanotubes (CNTs) and zirconium diboride (ZrB_2) particles were prepared via solid-state powder metallurgy route. The constituents of HAMCs were initially ball milled for uniform dispersion, and subsequently composite mixtures were consolidated by cold compaction, which was followed by pressureless sintering. The fractions of nanotubes and ceramic particles in HAMCs were 0.5 wt% and 5.0 wt%, respectively. Individually reinforced composites containing 0.5 wt% MWCNTs and 5.0 wt% ZrB_2 particles were also prepared for reference together with pure aluminum. X-ray diffraction identified the crystalline phases while optical and scanning electron microscopy revealed uniform dispersion of reinforcements without their agglomeration. Mechanical characterization showed a significant rise in the properties of HAMCs over unreinforced aluminum and composites containing individual reinforcements. The improvements of ~ 26%, ~ 34% and ~ 19% in hardness, compressive strength and flexural strength were observed in comparison with pure aluminum, respectively. The examination of fractured surfaces revealed the strengthening mechanisms responsible for the improvement in mechanical performance of HAMCs.

Keywords Aluminum · Hybrid composite · Multiwalled carbon nanotubes · Zirconium diboride · Mechanical properties · Fractography

1 Introduction

Metal matrix composites (MMCs) have always been considered as potential and prospective candidates for advanced engineering applications due to their superior mechanical and functional properties in comparison with monolithic metals and alloys [1]. Among different metallic matrices, aluminum and its alloys are ideal materials for MMCs due to their light weight, cost-effectiveness, good workability and recyclability together with high thermal and electrical conductivities [2]. Aluminum matrix composites (AMCs)

have therefore gained acceptance for a variety of applications in automotive and industrial engineering due to their unique combination of properties like high specific strength and stiffness, good tribological properties, electromagnetic shielding capability and improved thermal stability [3].

In preparing AMCs with enhanced mechanical properties, the role of an appropriate reinforcement selection and manufacturing technique cannot be underestimated. The properties of AMCs are principally dependent on the nature, geometry, mass fraction and dispersion quality of

Electronic supplementary material The online version of this article (<https://doi.org/10.1007/s42452-019-1767-7>) contains supplementary material, which is available to authorized users.

✉ Masood Ahmed, masoodmaqsood@gmail.com | ¹Department of Materials Science and Engineering, Institute of Space Technology, Islamabad, Pakistan. ²Department of Mechanical Engineering, College of Engineering, University of Hail, Ha'il, Saudi Arabia.

SN Applied Sciences (2019) 1:1702 | <https://doi.org/10.1007/s42452-019-1767-7>

reinforcement. A wide range of reinforcements in the form of fibers, particles and whiskers are incorporated in aluminum to prepare AMCs. However, particulate-reinforced AMCs are comparatively economical to produce and offer better isotropy in mechanical properties. A range of reinforcements including oxides (Al_2O_3 , ZrO_2), carbides (SiC , B_4C , TiC , WC), nitrides (Si_3N_4 , AlN), borides (TiB_2 , ZrB_2), intermetallics (Al-Fe , Al-Ni , Al-Nb), graphite and fly ash have been incorporated in aluminum, by liquid- and solid-state processing techniques [4].

Among different particulate reinforcements, zirconium diboride (ZrB_2) is considered as an efficient ceramic reinforcement due to its high elastic modulus (~ 489 GPa), high melting point (~ 3245 °C), good thermal shock resistance and high thermal and electrical conductivities [5]. ZrB_2 particles significantly enhance the wear resistance and corrosion protection of AMCs [6]. On the other hand, the novel form of graphite, i.e., multiwalled carbon nanotubes (MWCNTs), has been identified as an ideal reinforcement for AMCs due to their low density, superior mechanical properties, high aspect ratio and better thermal and electrical conductivities [7].

Despite an attractive package of properties, AMCs still face the challenges of high cost, reduced ductility, low fracture toughness and unpredicted corrosion behavior. However, the recent trend of preparing hybrid aluminum matrix composites (HAMCs) containing more than one reinforcements in metallic [8], ceramic [9] and polymeric [10] matrices offers an option to produce composites with improved performance. Typically, in aluminum matrix, different combinations have been reported including SiC /graphite [11], $\text{SiC}/\text{Al}_2\text{O}_3$ [12], SiC/mica [13], $\text{Al}_2\text{O}_3/\text{B}_4\text{C}$ [14], $\text{Si}_3\text{N}_4/\text{AlN}/\text{ZrB}_2$ [15], $\text{ZrB}_2/\text{Al}_3\text{Zr}$ [16], MWCNTs/graphene [17], $\text{ZrB}_2/\text{TiB}_2$ [18] and SiC/ZrO_2 [19]. For the preparation of hybrid composites, a range of manufacturing techniques have also been used such as direct melt reaction

[20], stir casting [21], friction stir processing [22, 23] and powder metallurgy [23].

A methodical search of relevant literature yielded that ZrB_2 particles have not been incorporated along with MWCNTs in aluminum matrix to explore their synergistic effect. The combination of two physically and chemically different reinforcements may produce novel properties of the developed composites. The aim of present work is therefore to investigate the combined effect of MWCNTs and ZrB_2 particles on the microstructural and mechanical properties of HAMCs. The investigation of the strengthening mechanisms in HAMCs due to the incorporation of binary reinforcements is another aim together with the change in the fracture morphology attributed to two morphologically different reinforcements.

2 Experimental

2.1 Materials

Commercially available pure aluminum powder with purity level $> 99.9\%$ and particle size $6\text{--}10\text{ }\mu\text{m}$, procured from Merck, Germany, was employed as the matrix. Surface morphology of the as-received Al particles is shown in Fig. 1a. Both MWCNTs and ZrB_2 particles were procured from Guangzhou Jiechuang, China, which were used as reinforcements. MWCNTs had length and diameter of $5\text{--}20\text{ }\mu\text{m}$ and $20\text{--}30\text{ nm}$, respectively, while ZrB_2 particles had a size of $2\text{--}5\text{ }\mu\text{m}$, as evident from SEM images shown in Fig. 1b and c, respectively.

2.2 Manufacturing

Four types of composite mixtures were prepared: (a) pure Al (P-Al) without any reinforcement to be used as

Fig. 1 SEM micrographs of composite constituents: **a** Al particles, **b** MWCNTs and **c** ZrB_2 particles

reference, (b) Al containing 0.5 wt% MWCNTs (0.5M–Al), (c) Al containing 5 wt% ZrB₂ particles (5Z–Al) and (d) Al containing both 0.5 wt% MWCNTs and 5 wt% ZrB₂ particles to (0.5M/5Z–Al) produce desired HAMCs (Table 1).

Figure 2 shows powder metallurgy (PM) route for preparing AMCs, wherein ball-milled constituents were followed by cold compaction and pressureless sintering for consolidation. The composite constituents were mixed thoroughly in milling process using a high-capacity laboratory jar mill (Model: RZ-04149-05, Cole-Parmer Instrument Co. USA) at 180 rpm for 6 h at room temperature. Zirconia balls with diameter of 5 mm and weight of 5 g were used as milling media, and ball-to-powder ratio was kept at 1:1. Same milling media and conditions were used to mill reference aluminum to produce the same effect of mechanical working as experienced by aluminum powder in composite mixtures.

For the consolidation of composite mixtures, uniaxial cold pressing followed by pressureless sintering was performed. Ball-milled mixtures were initially filled in a steel die of inner diameter of 20 mm and cold-compacted at 150 MPa using a manual hydraulic press (15-Ton, Atlas Series, Specac Ltd, UK). As-pressed compacts called as

green bodies were later sintered in a tube furnace (KJ-1600VF, Zhengzhou Kejia Furnaces, Co. China) under argon atmosphere. Sintering was carried out in two steps: (a) 400 °C for 1 h to remove the moisture followed by 630 °C for 6 h, which was followed by furnace cooling. The sintered pellets had average diameter and thickness of 20 mm and 5 mm, respectively.

2.3 Characterization

The densities of samples after sintering were measured by Archimedes principle using a densimeter (AU-900S, Dong Guan Hong Tuo Instrument Company, China) having accuracy of 10^{−3} g, while the densities of green bodies were measured by geometrical method. For microstructural observation, samples were cut parallel to pressing direction using a low-speed diamond blade saw (ISOMET, Buehler Ltd. USA). Grinding and polishing of samples were performed on a lapping/polishing machine (Forcipol 2V, Metkon Instruments, Turkey). For grinding, silicon carbide emery papers of grit sizes 600, 800, 1000, 1200, 1500 and 2000 were used at rotational speeds of 120–200 rpm. A mirror-like and scratch-free surface was obtained through polishing using alumina paste of 20 and 5 μm on polishing cloth at a speed of 60–80 rpm. To reveal the surface morphology, the polished specimens were etched for 35 s in Keller's reagent (2 mL HF, 5 mL HNO₃, 3 mL HCL and 190 mL distilled water). Optical microscopy (OM) was performed by using a metallurgical microscope (IMM-901, Metkon Instruments, Turkey) at magnifications of 100× and 500×. Microstructural observation of the constituents in composites and their polished and fractured surfaces was performed under field emission gun scanning electron microscope (FEG-SEM, MIRA-III, TESCON, Czech Republic) in secondary electron imaging mode at an accelerating

Table 1 Compositions of individually reinforced and hybrid composites

No.	Composites	Fraction of constituents (wt%)		
		MWCNTs	ZrB ₂ (%)	Aluminum
1	Pure aluminum (P-Al)	–	–	100
2	0.5 wt% MWCNTs–Al (0.5M–Al)	0.5	–	99.5
3	5 wt% ZrB ₂ –Al (5Z–Al)	–	5	95
4	0.5 wt% MWCNTs/5 wt% ZrB ₂ –Al (0.5M/5Z–Al)	0.5	5	94.5

Fig. 2 Schematic showing the processing steps for the manufacturing of AMCs containing MWCNTs and ZrB₂

voltage of 10 kV. X-ray diffraction (XRD) technique was employed for phase identification in composites using Rigaku X-ray diffractometer with Cu K α radiation over 2θ scanning range of 20° – 90° .

Hardness was measured by Vickers micro-hardness tester (VLS 3853, SHIMADZU, Japan) equipped with pyramid diamond indenter at a load of 500 g for 15 s. At least, ten readings of hardness values were acquired to obtain the average value of each sample. For measuring hardness, the composite samples were polished up to a surface finish of 5 μm . Compressive and flexural strengths of composites were measured on an universal testing machine (WDW-30, Jinan Testing Equipment, China) at a strain rate of 2.0 mm/min and 0.02 mm/min, respectively. At least five specimens were prepared for each type of composites, and their average value along with standard errors has been presented.

3 Results and discussion

3.1 Density

The theoretical, actual and relative densities of pure Al and composite samples after sintering are presented in Table 2. It can be seen that all the composites showed relative densities $> 97\%$ indicating that the processing of composites was suitable to consolidate the specimens to near theoretical density values.

Figure 3 shows relative density comparison, wherein pure Al sample showed the density $> 98\%$, which was reduced by incorporating individual and dual reinforcements but still $> 97\%$. The density results are also in good agreement with earlier studies [24, 25], while the samples containing MWCNTs resulted in comparatively lower densities [24].

Table 2 Theoretical, actual and relative density values of pure Al and composites

No.	Composites	Density		
		Theoretical (g/cm ³)	Actual (g/cm ³)	Relative (%)
1	Pure Al	2.7	2.65 ± 0.31	98.5 ± 0.2
2	0.5 wt% MWCNTs–Al	2.69	2.61 ± 0.32	97.2 ± 0.4
3	5 wt% ZrB ₂ –Al	2.86	2.80 ± 0.23	97.7 ± 0.3
4	0.5 wt% MWCNTs/5 wt% ZrB ₂ –Al	2.86	2.77 ± 0.45	97.1 ± 0.4

Fig. 3 Relative densities of pure Al and composites containing MWCNTs and ZrB₂ particles

3.2 Microstructural characterization

3.2.1 X-ray diffraction

Figure 4 shows XRD patterns of pure Al and composite samples. Typical Al peaks can be observed in all samples, while ZrB_2 peaks are prominent in 5Z–Al and 0.5M/5Z–Al samples. Peaks of MWCNTs and any other interfacial phase due to the reaction between Al and MWCNTs such as Al_4C_3 were not detected in 0.5M–Al and 0.5M/5Z–Al samples. It may be due to the presence of MWCNTs in small fractions, i.e., 0.5 wt% or the limitation of XRD machine, as also observed elsewhere [25]. Similarly any other phase due to the possible reaction between Al and ZrB_2 such as ZrAl_3 or AlB_2 was not identified in 5Z–Al and 0.5M/5Z–Al samples. In contrast, the formation of ZrAl_3 was reported in a different study [18, 26].

The reason for the absence of AlB_2 and ZrAl_3 in the present study may be due to relatively low processing temperature of 630 °C during pressureless sintering as compared to melting techniques. The absence of inter-metallic compounds of Al with ZrB_2 is usually reported in ZrB_2 –Al composites when synthesized by conventional powder metallurgy [6] and microwave [27] sintering routes.

3.2.2 Optical microscopy

Optical micrographs of pure Al and composite samples are shown in Fig. 5. Homogenous distribution of the two reinforcements in matrix was observed without the presence of any visible agglomeration. Although MWCNTs are not detectable at the present magnification, any sign of their agglomeration was likewise not found [24]. Moreover, due to optimized sintering procedure, the defects such as porosity and voids were minimum.

In the succeeding sections, it will be emphasized that the uniform dispersion of dual reinforcement and minimum of porosity plays a vital role to prepare hybrid composites with improved mechanical properties. The compressed grains can be observed in pure Al and the composite containing solely MWCNTs (0.5M–Al) attribute to the uniaxial pressing, while the composites containing ZrB_2 particles alone (5Z–Al) or in combination with MWCNTs (0.5M/5Z–Al) revealed equiaxed grains (Fig. 5). If the uniaxial pressing of composite powders is taken as the reason for the flattening of aluminum particles, then it should have been observed in all composite samples. A possible cause for the elongation of Al grains in two of the samples, i.e., P–Al and 0.5M–Al (Figs. 5a, b, 6a, b) has been discussed in the following section.

Fig. 4 XRD patterns of pure Al and composites showing the characteristic peaks of Al and ZrB_2

Fig. 5 Optical micrographs of prepared samples: **a** pure Al, **b** 0.5 wt% MWCNTs–Al composite, **c** 5 wt% ZrB₂–Al composite and **d** 0.5 wt% MWCNTs/5 wt% ZrB₂–Al composite

3.2.3 Scanning electron microscopy

Figure 6 displays SEM images of pure Al and composite samples. It can be seen that pure Al has coarse grains, which are also elongated (Fig. 6a), while Fig. 6b shows image of 0.5M–Al composite sample wherein comparatively smaller size grains can be seen but still elongated. Coarsening may be suppressed due to the addition of MWCNTs. This effect has also been reported in an earlier study [20]. The presence of ZrB₂ particles (light color) in Al matrix (dark color) also plays a role in grain refinement, as shown in Fig. 6c, d. It was recognized in a previous work that ZrB₂ particles have the tendency to lock grain boundaries [18]. Nevertheless, a significant effect of MWCNTs and ZrB₂ as grain refiners was not observed in the present study as the composites still had large grains though grain refining in comparison with pure Al was witnessed. Finally, Al particles can be seen along with small ZrB₂ particles due to their homogeneous distribution.

It has been pointed out throughout the mechanical mixing process that the ductile Al underwent deformation while the brittle ZrB₂ particles fragmented [6]. By mixing Al with ZrB₂ particles, the grain growth was restricted resulting in equiaxed grain morphology.

Additionally, most of the ZrB₂ particles were present near grain boundaries; however, their fewer fractions were also found within grains (Fig. 6c). This type of dispersion was also revealed in a parallel study [28]. The detection of MWCNTs in SEM images of the present magnification was difficult, especially on the polished surfaces. Presence of MWCNTs was spotted in SEM images of the fractured surfaces of the composites in Fig. 10, discussed further below.

3.3 Mechanical characterization

The combined addition of micrometer- and nanometer-scale particulate reinforcements imparts modification in the overall mechanical properties of a matrix material. The improvement in mechanical strength depends on the individual characteristics of reinforcements and matrix along with their interaction at the interface [9]. Likewise, significant improvement in the mechanical properties of the prepared HAMCs (0.5M/5Z–Al) has been observed in the present study as compared to reference aluminum (P–Al).

Fig. 6 SEM images of polished surfaces of samples: **a** pure Al, **b** 0.5 wt% MWCNTs–Al composite, **c** 5 wt% ZrB₂–Al composite and **d** 0.5 wt% MWCNTs/5 wt% ZrB₂–Al composite

3.3.1 Hardness

Figure 7 and Table 3, shows the improvement in Vickers hardness due to the addition of reinforcements in pure Al. It is evident that the composites have remarkably higher hardness values than pure Al. Upon the addition of 0.5 wt% MWCNTs to pure aluminum, an increase of about ~ 7% in hardness was observed, i.e., from 33.1 ± 0.6 to 35.4 ± 0.5 HV. However, in case of 5 wt% ZrB₂ particles as reinforcement, the average hardness attained was 40.1 ± 0.6 HV, which is a rise of ~ 22%. The HAMCs (0.5M/5Z–Al) revealed the maximum hardness value of 41.7 ± 0.7 HV showing an increase of ~ 26% as compared to pure Al.

The manifested increase in the hardness value of 0.5M/5Z–Al may be attributed to dispersion hardening. The presence of both micrometer- and nanometer-scale reinforcements induced the pinning effect and constrained grain growth [29]. Moreover, dispersion strengthening effect attributed to uniform distribution of MWCNTs and ZrB₂ particles [7] and Orowan strengthening mechanism by dislocation looping around reinforcement particles [30] are believed to be supplementary mechanisms responsible for the increase in the mechanical strength, which was revealed in the rise of hardness values.

Fig. 7 Vickers hardness values of pure Al and composite samples **Table 3** Vickers hardness values of pure Al and composites

No.	Composites	Vickers hardness (HV)
1	Pure Al	33.1 ± 0.6
2	0.5 wt% MWCNTs–Al	35.4 ± 0.5
3	5 wt% ZrB ₂ –Al	40.1 ± 0.6
4	0.5 wt% MWCNTs/5 wt% ZrB ₂ –Al	41.7 ± 0.7

Table 4 Compressive strength values of pure Al and composites

No.	Composites	Compressive strength (MPa)
1	Pure Al	77 ± 3
2	0.5 wt% MWCNTs–Al	85 ± 2
3	5 wt% ZrB ₂ –Al	98 ± 4
4	0.5 wt% MWCNTs/5 wt% ZrB ₂ –Al	103 ± 6

3.3.2 Compression strength

Like hardness, compressive strength of pure Al was markedly improved by incorporating MWCNTs and ZrB₂ particles individually and combined (Table 4). Figure 8a and b shows the stress–strain diagram and compressive

strength values, respectively. The 0.5M/5Z–Al containing 0.5 wt% MWCNTs and 5 wt% ZrB₂ particles exhibited an increase in compressive strength of ~ 34% from 77 ± 3 MPa to 103 ± 6 MPa. On the other hand, composites having single reinforcements displayed relatively lower enhancements. 0.5M–Al composite had compressive strength of 85 ± 2 MPa showing an improvement of ~ 10%, and 5Z–Al reinforced with 5 wt% ZrB₂ particles displayed compressive strength of 98 ± 4 MPa, which is an increase of ~ 27%.

It can be inferred that the strengthening effect was influenced by the presence of reinforcements at two different scales in 0.5M/5Z–Al composites. The uniform dispersion of hard and stiff reinforcements led to efficient load distribution in the bulk composite and enhanced dislocation density effect [31].

HAMCs exhibited higher compressive strength values due to simultaneous operation of two types of strengthening mechanisms. One effect was Orowan strengthening mechanism attributed to the presence of hard ceramic micrometer-sized ZrB₂ particles. When composites are under compressive stress, the stiff ZrB₂ particles impede the dislocation motion during plastic deformation of aluminum. The second strengthening phenomenon was grain refining due to the pinning effect of MWCNTs and ZrB₂ particles, which hindered the movement of dislocations across the grains, owing to higher grain boundary

Fig. 8 Compressive strength of pure Al and composites: **a** stress–strain graph and **b** compressive strength values

Fig. 9 Flexural strength of pure Al and composites: **a** stress–strain plot for three-point bend test and **b** flexural strength values

population. Similar correlation has also been established in prior studies [9, 16].

3.3.3 Flexural strength

The flexural test results as measured by three-point bending method are presented in Fig. 9 and Table 5. The stress–strain plots in Fig. 9a show the behavior of pure Al and composites under flexural loading. The incorporation of reinforcements improved the flexural strength of composites, i.e., 0.5M–Al composite displayed a rise of ~8%, 5Z–Al composite exhibited ~15% increase and 0.5M/5Z–Al composite showed the highest improvement of ~19%. The flexural strength was improved because of

Table 5 Flexural strength values of pure Al and composites

No.	Composites	Flexural strength (MPa)
1	Pure Al	525 ± 8
2	0.5 wt% MWCNTs–Al	570 ± 9
3	5 wt% ZrB ₂ –Al	605 ± 11
4	0.5 wt% MWCNTs/5 wt% ZrB ₂ –Al	622 ± 10

the strengthening mechanisms, as discussed above and also observed elsewhere [29].

Fig. 10 SEM images of the fractured surfaces of pure Al and composites: **a** pure Al, **b** 0.5 wt% MWCNTs-Al composite (yellow arrows indicate MWCNTs), **c** 5 wt% ZrB₂-Al composite (white arrows indi-

cate ZrB₂ particles) and **d** 0.5 wt% MWCNTs/5 wt% ZrB₂-Al HAMCs. Insets show SEM images at higher magnifications

On the other hand, composites displayed relatively lower strain-to-failure values (~ 0.85 to $\sim 1.55\%$), which may be related to increased strength along with lower relative density in the range of 97–98% or attributed to the absence of secondary processing, which could produce compacted composites without any defects; an analogous effect has also been noticed in a previous work [24].

It has been reported that the incorporation of stiff ZrB₂ particles resulted significant decline in the failure strain, which was attributed to higher weight fractions of ZrB₂ and their inability to deform plastically, thus consequently

leading to easy debonding and early failure [16, 32]. 5Z-Al composite exhibited strain-to-failure of about $\sim 0.85\%$, as compared to $\sim 1.55\%$ of pure aluminum. However, 0.5M-Al composite displayed a remarkable feature of increase in flexural strength, with marginal decrease in failure strain owing to the flexible nature of MWCNTs and their sliding during deformation, as also observed elsewhere [33]. In case of 0.5M/5Z-Al composite, strain-to-failure was close to that of 5Zr-Al composite due to the dominant role of ZrB₂, as discussed above and also pointed out in a parallel research [8].

3.4 Fractography

The fractured surfaces of pure Al and composite samples were observed to evaluate the distribution quality of MWCNTs (pointed out with yellow arrows) and ZrB₂ particles (pointed out with white arrows) and their influence on fracture mode (Fig. 10). It is evident from the SEM images that reinforcement clustering, interfacial cracks, porosity, debonding between matrix and reinforcements are the common problems found in composites, which in this study are significantly less due to optimum reinforcement content and their uniform distribution in the matrix.

It has been observed in previous studies that higher reinforcement content resulted in their aggregation [6, 34]. Although no chemical interface was identified between reinforcements and ductile Al matrix, the presence of a mechanical interface was found due to the absence of reaction between the matrix and the reinforcements. The irregular shape of ZrB₂ particles in ductile aluminum matrix was enough to transfer the stress from the matrix to the reinforcement. In case of MWCNTs, their flexible nature and bended contours incorporated them firmly in ductile matrix for effective load transfer and thus improved performance.

Intergranular fracture is observed in unreinforced pure aluminum sample (Fig. 10a), as prepared through the same processing route as composites. After the addition of MWCNTs with large aspect ratio, the fracture mode changed from purely intergranular to a mixture of intergranular and transgranular (Fig. 10b). However, the fracture mode was predominately intergranular attributed to the bendable nature of MWCNTs during plastic deformation, as discussed above.

Unlike MWCNTs, the addition of rigid micrometer size ceramic particles of ZrB₂ was located along the surface of aluminum particles; therefore, their presence around grain boundaries in sintered samples resulted in stronger grain boundaries. Due to this phenomenon, the crack could not be propagated along the grain boundaries and deflected toward the grains, thus showing predominantly the transgranular fracture mode (Fig. 10c). While in hybrid composite, both kinds of fracture modes, i.e., intergranular and transgranular, can be observed (Fig. 10d) due to the presence of two dissimilar types of reinforcements, i.e., MWCNTs and ZrB₂ particles, and their different behavior under applied load.

4 Conclusions

The solid-state processing technique comprising ball milling, cold compaction and pressureless sintering was employed to develop hybrid aluminum matrix composites containing 0.5 wt% MWCNTs and 5.0 wt% ZrB₂ particles. Uniform dispersion of MWCNTs and ZrB₂ particles was observed in the matrix without any indication of agglomeration and reinforcement-rich and reinforcement-depleted areas. The addition of ZrB₂ particles developed fine and equiaxed aluminum grains in composites. The prepared HAMCs showed sintered density near theoretical, which is prerequisite for achieving improved mechanical properties. XRD confirmed the absence of any interfacial phase between matrix and reinforcements, which suggested the development of a mechanical interface. The mechanical properties including hardness, compressive and flexural strengths were improved over reference aluminum by ~ 26%, ~ 34% and ~ 19%, respectively. This improvement in mechanical properties was attributed to the strengthening mechanisms and the enhanced dislocation density owing to the presence of dual reinforcements, i.e., MWCNTs and ZrB₂ particles.

Acknowledgements Authors are grateful to Umar Farooq and Bilal Ghafoor for their technical support.

Compliance with ethical standards

Conflict of interest Authors declare that there is no actual or potential conflict of interest in relation to this article.

References

1. Mortensen A, Llorca J (2010) Metal matrix composites. *Annu Rev Mater Res* 40:243. <https://doi.org/10.1146/annurev-matsci-070909-104511>
2. Thirumoorthy A, Arjunan TV, Senthil Kumar KL (2018) Latest research development in aluminum matrix with particulate reinforcement composites—a review. *Mater Today Proc* 5:1657. <https://doi.org/10.1016/j.matpr.2017.11.260>
3. Kumar GV, Rao C, Selvaraj N (2011) Mechanical and tribological behavior of particulate reinforced aluminum metal matrix composites—a review. *J Miner Mater Charact Eng* 10:59
4. Bodunrin MO, Alaneme KK, Chown LH (2015) Aluminium matrix hybrid composites: a review of reinforcement philosophies; mechanical, corrosion and tribological characteristics. *J Mater Res Technol* 4:434
5. Guo S-Q (2009) Densification of ZrB₂-based composites and their mechanical and physical properties: a review. *J Eur Ceram Soc* 29:995. <https://doi.org/10.1016/j.jeurceramsoc.2008.11.008>
6. Fogagnolo J, Robert M, Ruiz-Navas E, Torralba J (2004) 6061 Al reinforced with zirconium diboride particles processed by

- conventional powder metallurgy and mechanical alloying. *J Mater Sci* 39:127
7. Bakshi SR, Agarwal A (2011) An analysis of the factors affecting strengthening in carbon nanotube reinforced aluminum composites. *Carbon* 49:533. <https://doi.org/10.1016/j.carbon.2010.09.054>
 8. Khan M, Zulfaqar M, Ali F, Subhani T (2017) Hybrid aluminium matrix composites containing boron carbide and quasicrystals: manufacturing and characterisation. *Mater Sci Technol* 33:1955
 9. Ahmad I, Ahmed S, Subhani T et al (2016) Synergic influence of MWCNTs and SiC nanoparticles on the microstructure and properties of Al₂O₃ ceramic hybrid nanocomposites. *Curr Appl Phys* 16:1649. <https://doi.org/10.1016/j.cap.2016.10.009>
 10. Subhani T, Latif M, Ahmad I, Rakha SA, Ali N, Khurram AA (2015) Mechanical performance of epoxy matrix hybrid nanocomposites containing carbon nanotubes and nanodiamonds. *Mater Des* 87:436. <https://doi.org/10.1016/j.matdes.2015.08.059>
 11. Krishna MV, Xavier AM (2014) An investigation on the mechanical properties of hybrid metal matrix composites. *Procedia Eng* 97:918
 12. Devaraju A, Kumar A, Kumaraswamy A, Kotiveerachari B (2013) Influence of reinforcements (SiC and Al₂O₃) and rotational speed on wear and mechanical properties of aluminum alloy 6061-T6 based surface hybrid composites produced via friction stir processing. *Mater Des* 51:331
 13. Rajmohan T, Palanikumar K, Ranganathan S (2013) Evaluation of mechanical and wear properties of hybrid aluminium matrix composites. *Trans Nonferrous Met Soc China* 23:2509
 14. Ramnath BV, Elanchezian C, Annamalai R et al (2014) Aluminium metal matrix composites—a review. *Rev Adv Mater Sci*, p 38
 15. Kumar NM, Kumaran SS, Kumaraswamidhas L (2016) Aerospace application on Al 2618 with reinforced—Si₃N₄, AlN and ZrB₂ in situ composites. *J Alloys Compd* 672:238
 16. Gautam G, Mohan A (2015) Effect of ZrB₂ particles on the microstructure and mechanical properties of hybrid (ZrB₂ + Al₃Zr)/AA5052 *insitu* composites. *J Alloys Compd* 649:174
 17. Moghadam AD, Omrani E, Menezes PL, Rohatgi PK (2015) Mechanical and tribological properties of self-lubricating metal matrix nanocomposites reinforced by carbon nanotubes (CNTs) and graphene—a review. *Compos B Eng* 77:402
 18. Zhao D, Liu X, Liu Y, Bian X (2005) *In-situ* preparation of Al matrix composites reinforced by TiB₂ particles and sub-micron ZrB₂. *J Mater Sci* 40:4365
 19. Arif S, Alam MT, Ansari AH, Siddiqui MA, Mohsin M (2017) Study of mechanical and tribological behaviour of Al/SiC/ZrO₂ hybrid composites fabricated through powder metallurgy technique. *Mater Res Express* 4:076511
 20. Yang X, Zou T, Shi C, Liu E, He C, Zhao N (2016) Effect of carbon nanotube (CNT) content on the properties of in situ synthesis CNT reinforced Al composites. *Mater Sci Eng A* 660:11
 21. Kala H, Mer K, Kumar S (2014) A review on mechanical and tribological behaviors of stir cast aluminum matrix composites. *Procedia Mater Sci* 6:1951
 22. Khan M, Rehman A, Aziz T, Shahzad M, Naveed K, Subhani T (2018) Effect of inter-cavity spacing in friction stir processed Al 5083 composites containing carbon nanotubes and boron carbide particles. *J Mater Process Technol* 253:72. <https://doi.org/10.1016/j.jmatprotec.2017.11.002>
 23. Khan M, Amjad M, Khan A, Ud-Din R, Ahmad I, Subhani T (2017) Microstructural evolution, mechanical profile, and fracture morphology of aluminum matrix composites containing graphene nanoplatelets. *J Mater Res* 32:2055
 24. Khan M, Zulfaqar M, Ali F, Subhani T (2017) Microstructural and mechanical characterization of hybrid aluminum matrix composite containing boron carbide and Al–Cu–Fe quasicrystals. *Met Mater Int* 23:813
 25. Basariya MR, Srivastava V, Mukhopadhyay N (2014) Microstructural characteristics and mechanical properties of carbon nanotube reinforced aluminum alloy composites produced by ball milling. *Mater Des* 64:542
 26. Kumar N, Gautam RK, Mohan S (2015) In-situ development of ZrB₂ particles and their effect on microstructure and mechanical properties of AA5052 metal-matrix composites. *Mater Des* 80:129. <https://doi.org/10.1016/j.matdes.2015.05.020>
 27. Ghasali E, Yazdani-Rad R, Rahbari A, Ebadzadeh T (2016) Microwave sintering of aluminum–ZrB₂ composite: focusing on microstructure and mechanical properties. *Mater Res* 19:765
 28. Selvam JDR, Dinaharan I (2017) In situ formation of ZrB₂ particulates and their influence on microstructure and tensile behavior of AA7075 aluminum matrix composites. *Eng Sci Technol Int J* 20:187
 29. Sanaty-Zadeh A (2012) Comparison between current models for the strength of particulate-reinforced metal matrix nanocomposites with emphasis on consideration of Hall–Petch effect. *Mater Sci Eng A* 531:112. <https://doi.org/10.1016/j.msea.2011.10.043>
 30. Zhang Z, Chen DL (2008) Contribution of Orowan strengthening effect in particulate-reinforced metal matrix nanocomposites. *Mater Sci Eng A* 483–484:148. <https://doi.org/10.1016/j.msea.2006.10.184>
 31. Casati R, Vedani M (2014) Metal matrix composites reinforced by nano-particles—a review. *Metals* 4:65
 32. Kumar NM, Kumaran SS, Kumaraswamidhas L (2015) An investigation of mechanical properties and corrosion resistance of Al2618 alloy reinforced with Si₃N₄, AlN and ZrB₂ composites. *J Alloys Compd* 652:244
 33. Kondoh K, Threrujirapapong T, Imai H, Umeda J, Fugetsu B (2009) Characteristics of powder metallurgy pure titanium matrix composite reinforced with multi-wall carbon nanotubes. *Compos Sci Technol* 69:1077. <https://doi.org/10.1016/j.compscitech.2009.01.026>
 34. Kai X, Zhao Y, Wang A, Wang C, Mao Z (2015) Hot deformation behavior of in situ nano ZrB₂ reinforced 2024Al matrix composite. *Compos Sci Technol* 116:1

Publisher's Note Springer Nature remains neutral with regard to jurisdictional claims in published maps and institutional affiliations.