# Synthesis and mechanical characterisation of self-lubricating Al7075/$MoS_2$/$ZrB_2$ hybrid composite

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**Abstract:** In the current study, Al7075-based hybrid composites reinforced with zirconium diboride ($ZrB_2$) and molybdenum disulphide ($MoS_2$) particles were synthesised through a two-step stir casting process. Three distinct compositions Al7075 alloy, Al7075 + 3 vol.%$MoS_2$ and Al7075 + 3 vol.%$MoS_2$ + 3 vol.%$ZrB_2$ have synthesised using the stir casting process. X-ray diffraction (XRD), optical microscopy, scanning electron microscopy (SEM) along with energy dispersive spectroscopy (EDS), transmission electron microscopy (TEM), and mechanical characterisation were performed on all synthesised samples to identify reinforcement particles, grain refinement, particle morphology, hardness, tensile properties and failure mode. Microstructural examination reveals refined grain structure, uniform distribution of particles, along with clear interface and good bonding. Mechanical characterisation reveals Al7075/$MoS_2$/$ZrB_2$ hybrid composite with highest hardness, strength, and ductility among all the compositions examined. Increased strength along with good ductility of composite over the unreinforced alloy is an important finding of the present study in contrast with many previous studies. Microstructure and mechanical features were correlated with results obtained for enhanced performance and life span of the hybrid composites.

**Keywords:** hybrid composite; Al7075; zirconium diboride; $ZrB_2$; molybdenum disulphide; $MoS_2$; stir casting; mechanical properties.

**Reference** to this paper should be made as follows: Dubey, M., Kumar, N. and Mohan, S. (2023) 'Synthesis and mechanical characterisation of self-lubricating Al7075/$MoS_2$/$ZrB_2$ hybrid composite', *Int. J. Materials and Product Technology*, Vol. 66, No. 1, pp.1–16.

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## 1 Introduction

Many lightweight materials like magnesium, titanium, aluminium, and their respective alloys are widely utilised in automobile industries for the manufacturing of major components to achieve improved performance and fuel economy. Owing to the lowest density metal next to the magnesium alloy, aluminium alloy has a wide scope of applications whereas, explosive hazards and the flammable nature of magnesium alloy restricts its use in many applications. Moreover, titanium alloy also has limited applications in the manufacturing of automobile components due to its high cost and casting difficulty. The scope of aluminium alloys can be extended further by tailoring their characteristics making aluminium matrix composites (AMCs). Tailored characteristics can be obtained as per the requirement, by adding a variety of ceramic reinforcement particles into the aluminium matrix via in-situ or ex-situ technique (Kaczmar et al., 2000). Owing to simplicity, mass production capability, and cost-effectiveness, liquid state processing is widely used for synthesising composites (Ranjan and Shanmugasundaram, 2019). In contrast to ex-situ, in-situ technique is favourable to achieving a fine and homogeneous distribution of particles and a reaction-free clean interface with good bonding. Moreover, reinforcement particles do not react with molten aluminium alloy at 860°C temperature and show thermodynamic stability at this temperature. A thermodynamic stable compound has a negative standard Gibbs free energy of formation. Thermodynamically stable compounds do not decompose into their elements.

Since last few decades, AMCs have emerged as potential candidates for applications in aerospace, transportation, and marine sectors through their enhanced high specific strength, high strength to cost ratio, higher hardness, enhanced wear and corrosion resistance characteristics (Sudhakar et al., 2016; Ranjan and Shanmugasundaram, 2019). Major components in automobile industries like brake rotors, pistons, connecting rods, cylinder blocks, suspension systems, etc. are manufactured by AMCs (Kumar et al., 2015). Among the aluminium alloys, Al7075 exhibits good strength, good corrosion resistance, good fatigue resistance and relatively low density. A substantial number of reports are available in the literature for improving the mechanical characteristics of Al7075 alloy by developing it as a composite through a variety of synthesis techniques. Carbides, oxides, nitrides, borides ceramic reinforcement, and their combined form are favourably incorporated into the Al7075 alloy matrix for achieving better mechanical properties and extending its application areas (Jiang and Wang, 2015; Selvam and Dinaharan, 2017; Haq et al., 2020). Hybrid composite dispersed with different ceramics amounts exhibits incredible performance characteristics over single reinforcement composite (Raghavendra and Ramamurthy, 2015; Sambathkumar et al., 2017; Devaganesh et al., 2020). Mostly, ceramics used as the reinforcement enhance the mechanical properties of composites like hardness and strength of alloy at the expense of a reduction in ductility (Keshavamurthy et al., 2013; Kumar and Girish, 2016; Selvam and Dinaharan, 2017; Kumar et al., 2021). However, some authors reported an improvement in ductility also in addition to the mechanical properties (Tian et al., 2014; Kumar et al., 2015). The use of traditional carbon-based lubricating oil in dry machining has been determined to be problematic in terms of environmental effects (Khanna et al., 2021). Wear-resistant coatings (TiN and AlTiSiN) are also being developed because of their benefits (extend life span and enhance surface quality) when utilised on metal equipment that is exposed to abrasive and damaging conditions (Colombo-Pulgarín et al., 2021). On the other hand, many authors reinforced the ceramic particles along with the solid lubricants to achieve the optimum properties. Ramanan et al. (2019) stir cast Al7075/$Al_2O_3$/$MoS_2$ hybrid composites with good tensile strength, hardness, and wear resistance properties along with uniformly distributed reinforcement particles. A novel self-lubricating AA7075-based composite dispersed with silicon nitride and varying molybdenum disulphide ($MoS_2$) amount was stir cast by Haq et al. (2020). Their study stated that solid lubricant ($MoS_2$) affects microhardness, compression and microstructural behaviour. A decreasing trend of compressive strength and micro hardness was reported with increasing $MoS_2$ content. Liu et al. (2019) explored $B_4C$ and $MoS_2$ particles influence on mechanical behaviour. They observed the improvement in mechanical properties through the combined effect of reinforcements. Shankar and Basavarajappa (2015) also investigated the tensile behaviour of Al/$B_4C$/$MoS_2$ hybrid composites processed by stir casting and discovered that the tensile strength of the base material is larger than the secondary reinforcing $MoS_2$. They also found that Al 2219 has the lowest microhardness as well as density, whereas Al + 3% B4C + 5% $MoS_2$ has the highest. Devaganesh et al. (2020) explored the influence of solid lubricants $MoS_2$, hBN and graphite along with SiC particles on mechanical and tribological behaviour of Al7075 alloy. They observed that hBN reinforcement has better wear attributes than other reinforcements, but when examining tensile and wear properties, Al7075 alloy reinforced using SiC and graphite inorganic nanoparticles is more lucrative, with good mechanical and wear behaviour. Recently, Beyanagari and Kandasamy (2021) performed a comparative study on mechanical and tribological behaviour of Al7075/hBN and Al7075/hBN/$MoS_2$ squeeze cast composites and concluded that the AA7075/1.0% hBN/0.5% $MoS_2$ combination had a greater hardness value and superior tribology behaviour than the AA7075/h-BN and AA7075 alloy.

In view of the literature reviewed, it seems that a large number of studies are available on strengthening Al7075 with numerous ceramic particles along with solid lubricants. It is also observed that the addition of solid lubricants enhances tribological properties of AMCs but simultaneously, reduction in hardness and strength is also reported (Vinoth et al., 2012). Therefore, to improve the mechanical properties along with good tribological properties ceramic particles are added with solid lubricants by some authors (Baradeswaran and Perumal, 2014; Karvanis et al., 2016; Omrani et al., 2016). Various authors in the literature to synthesise the composites have, also established the feasibility of in-situ process and stir casting. Further, enhanced hardness and strength are noticeable in composites; however, reduction in ductility is also significant. Simultaneously, authors were attracted to many self-lubricating materials like graphite, hexagonal boron nitride and $MoS_2$ (Omrani et al., 2016).

Also, when approaches to self-lubricating abilities, $MoS_2$ endures more loads when it is compared with other self-lubricating materials (Baradeswaran and Perumal, 2014). Moreover, self-lubricating materials can reduce the utilisation of toxic lubricants and reduce energy consumption in industries (Omrani et al., 2016). In contrast to graphite, $MoS_2$-based solid lubricant has the distinctive edge of being utilised in vacuum (Kanthavel et al., 2016). Therefore, to improve the ductility along with hardness and strength in composites and to minimise toxic lubrication and energy consumption in the manufacturing of industrial components, a new potential hybrid composite material Al7075/$MoS_2$/$ZrB_2$ is produced by using in-situ stir casting route. This material finds a wide scope of application with excellent mechanical and wear properties along with self-lubricating properties. Therefore, the aim of the present work is to synthesise a self-lubricating hybrid Al7075/$MoS_2$/$ZrB_2$ composite. Meagre detailed systematic study of Al7075/$MoS_2$/$ZrB_2$ composites are reported in the literature, hence, in-situ zirconium diboride ($ZrB_2$) particles in the aluminium matrix need more detailed and systematic analysis for its synthesis, morphology, microstructural, and mechanical properties. Moreover, $ZrB_2$ has high-temperature performance characteristics and the most important is that it is unreactive with aluminium (Zhang et al., 2008; Sambathkumar et al., 2017). Hence, to get the synergic effect of $ZrB_2$, $MoS_2$ as the reinforcement hybrid composites are synthesised through a stir casting process. The process is employed for the fabrication of hybrid composites because of its simplicity and being economical. Microstructure and mechanical properties are also correlated for enhanced performance and life span of the hybrid composites.

## 2 Experimental details

### *2.1 Materials and composites synthesis*

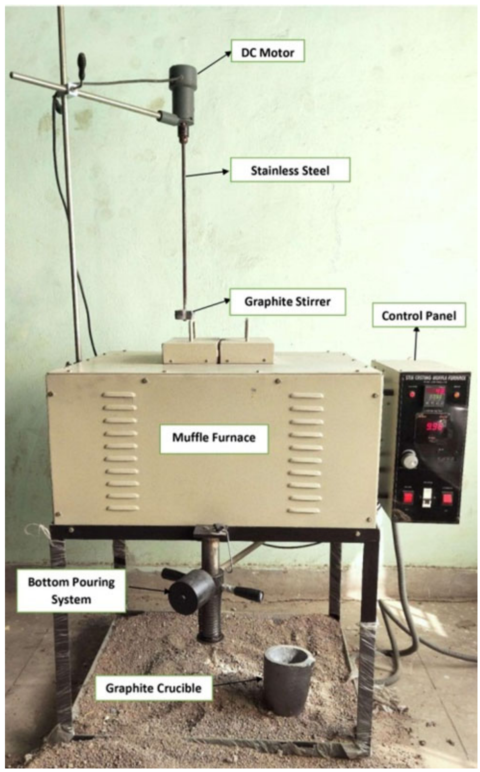
Al7075 aluminium alloy, as matrix material and two inorganic powders $K_2ZrF_6$ and $KBF_4$ were used for in-situ formations of $ZrB_2$ reinforcement. The chemical composition of the matrix alloy was determined using a spectroscopy test at Met Lab Laboratory Services, Mumbai, India. Table 1 shows the chemical composition of matrix alloy.

Stoichiometric amounts of $K_2ZrF_6$ and $KBF_4$ powders were calculated, weighed, and placed in an electric oven at 300°C for three hours for removing moisture. Dehydrated powders $K_2ZrF_6$ and $KBF_4$ were mixed in a mass ratio of 52:48 (Tian et al., 2014). Graphite crucible with Al7075 alloy was kept inside the vertical stir casting muffle furnace shown in Figure 1. A vertical muffle furnace is used to melt the aluminium alloy until the temperature reaches 860°C. The temperature of the molten alloy was measured using a K-type thermocouple. Al7075 + 3 vol.%$MoS_2$ composite was synthesised by adding ex-situ $MoS_2$ particles in the semi-solid slurry of molten alloy.

**Table 1** Chemical composition of Al7075 alloy

| *Element* | *Zn* | *Mg* | *Cu* | *Si* | *Fe* | *Mn* | *Cr* | *Ti* | *Al* |
|---|---|---|---|---|---|---|---|---|---|
| Wt. % | 5.323 | 2.435 | 1.389 | 0.127 | 0.105 | 0.111 | 0.222 | 0.112 | Balance |

**Figure 1** Stir casting muffle furnace for the preparation of composites (see online version for colours)




To synthesise Al7075 + 3 vol.%$MoS_2$ + 3 vol.%$ZrB_2$ hybrid composite $K_2ZrF_6$ and $KBF_4$ powders were added at a temperature of 860°C for in-situ formations of $ZrB_2$ particles and melt were intermittently stirred for 30 minutes. During the in-situ synthesis time period, several reactions take place as per the following sequence (Dinaharan et al., 2011).

$$K_2ZrF_6 + \frac{13}{3}Al = Al_3Zr + \frac{4}{3}AlF_3 + 2KF \tag{1}$$

$$2KBF_4 + 3Al = AlB_2 + 2AlF_3 + 2KF \tag{2}$$

$$Al_3Zr + AlB_2 = ZrB_2 + 4Al \tag{3}$$

Once in-situ reaction was completed, temperature of melt was reduced to 750°C, then preheated $MoS_2$ particles were inserted into the semi-solid melt and stirred for further five minutes. Finally, hexachloroethane ($C_2Cl_6$) tablets were used for degassing and pouring done into the preheated metallic mould. Similar casting procedure was performed for casting Al7075 alloy as shown in Table 2. Casting samples were machined and prepared for various examinations.

**Table 2** Different compositions

| *Material developed* | *Vol.% of $ZrB_2$* | *Vol.% of $MoS_2$* | *Vol.% of Al7075* |
|---|---|---|---|
| Al7075 alloy | 0 | 0 | 100 |
| Al7075 + 3 vol.%$MoS_2$ | 0 | 3 | 97 |
| Al7075 + 3 vol.%$MoS_2$ + 3 vol.%$ZrB_2$ | 3 | 3 | 94 |

### *2.2 Equipment used for characterisation*

Different phases formed in the composites were identified using X-ray diffraction (XRD) (Bruker, D-8 Advance 206890). Optical microstructures were obtained by using Leitz Metallux-3 optical microscope.

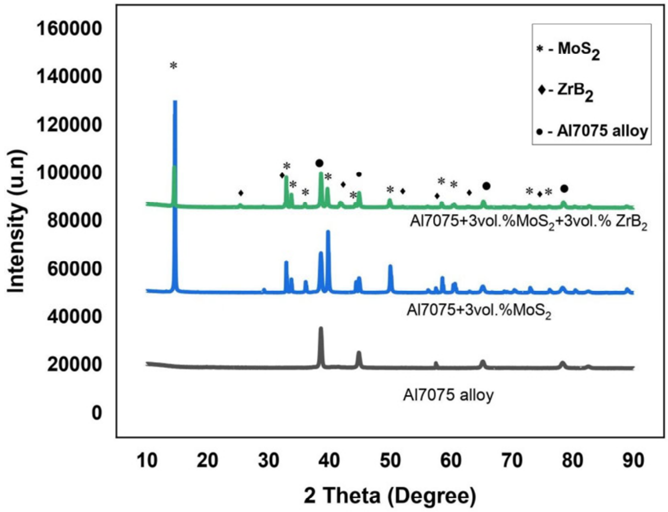
Tungsten-electron microscope (W-SEM JSM-6010LA) examined energy dispersive spectroscopy (EDS) analysis, reinforcement particle size, morphology, and their distribution in the matrix. Fractured surface morphology, and failure mode were also examined using scanning electron microscopy (SEM). Dislocations, morphology and particle-matrix interfacial bonding were observed by using transmission electron microscopy (TEM) (TECNAI G2 20). The density of all compositions was determined using Archimedes principle. The porosity of composites was determined by using rule of mixture. Brinell hardness of all compositions was measured at 500 kgf load and 30 seconds dwell time. Tensile test specimens were prepared in accordance with BS 12-1950 British Standards in the cylindrical form. Tests were carried out at ambient temperature on computerised 100 kN InstronTM UTM, model 4206, at a strain rate of $1.07 \times 10^{-3}$ $s^{-1}$ (Kumar et al., 2015).

## 3 Results and discussion

### *3.1 XRD analysis*

In order to confirm the completion of in-situ reaction and phases present XRD analysis was done. Incomplete reaction or insufficient reaction time may lead to the formation of undesirable intermediate compounds like $AlB_2$ and $Al_3Zr$. Figure 2 shows the XRD pattern of different composites. XRD peaks were identified and analysed by using JCPDS software. Diffraction peaks of $ZrB_2$ and $MoS_2$ with the Al matrix are clearly visible which confirm the presence of reinforcements $ZrB_2$ and $MoS_2$ without formation of any intermetallic compound. Absence of intermetallic compounds implies that the interface between aluminium alloy and reinforcement particles is clean and the reinforcement particles are thermodynamically stable.

**Figure 2** XRD patterns for the prepared composites (see online version for colours)




## *3.2 Density and porosity measurement*

Table 3 shows the density and porosity of prepared compositions. Variation in density was observed in composites as compared with the base alloy. Porosity was observed within a limit of 4 to 6 vol.% of cast aluminium alloy Al7075 and composites as well.

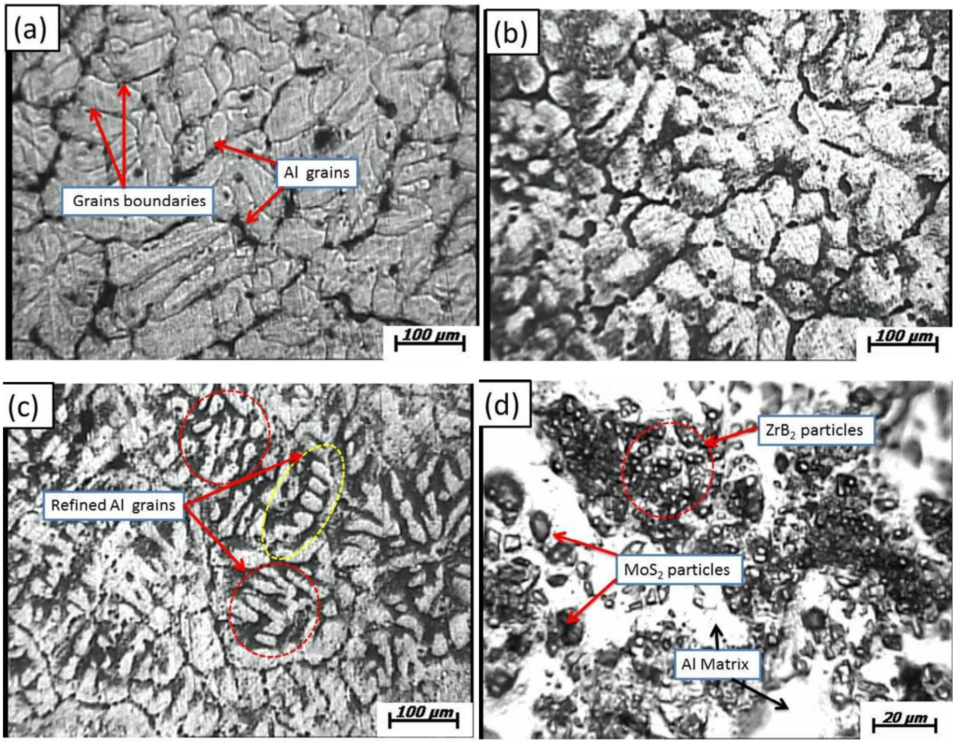
**Table 3** Density and porosity of different compositions

| *Material developed* | *Theoretical density (g/cm³)* | *Experimental density (g/cm³)* | *Porosity (vol.%)* |
|---|---|---|---|
| Al7075 alloy | 2.81 | 2.73 | 2.8 |
| Al7075 + 3 vol.%$MoS_2$ | 2.88 | 2.71 | 5.9 |
| Al7075 + 3 vol.%$MoS_2$ + 3 vol.%$ZrB_2$ | 2.98 | 2.79 | 6.3 |

## *3.3 Optical microscopy*

Figures 3(a)–3(c) show the optical micrographs of Al7075 alloy, Al7075/$MoS_2$ and Al7075/$MoS_2$/$ZrB_2$ composites, respectively. The images clearly show the distribution of $MoS_2$ and $ZrB_2$ particles in the matrix. It can be observed that reinforcement particles are located along the grain boundaries. In-situ generated $ZrB_2$ particles act as grain refiner and aluminium matrix grains have been refined significantly [Figure 3(c)]. The average aluminium grain size was reduced from 143 µm to 55 µm. Grain refinement contributes to the improved mechanical properties of composites. At higher magnification, particles are visible [Figure 3(d)].

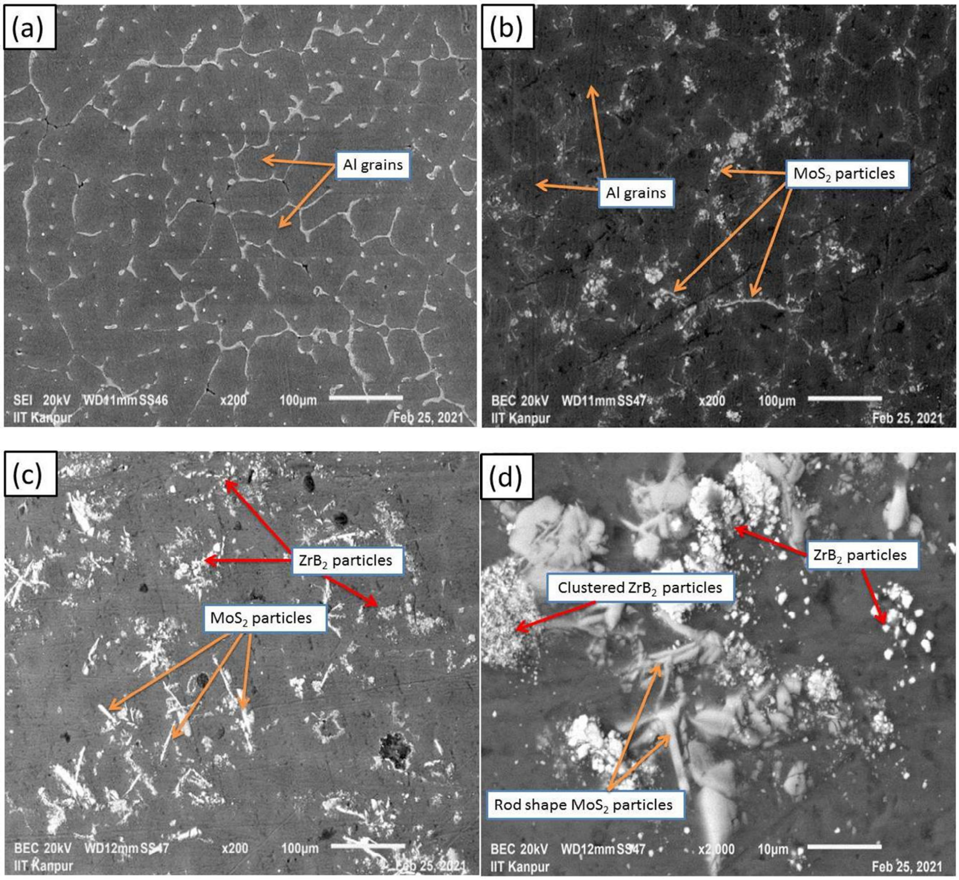
**Figure 3** Optical microscopy of (a) Al7075 alloy (b) Al7075+3vol.%$MoS_2$ (c) Al7075+3 vol. % $MoS_2$+3vol. % ZrB2 (d) microscopy of particles at higher magnification (see online version for colours)




### *3.4 SEM and EDS examination*

Figures 4(a)–4(c) show SEM pictures of Al7075 alloy and fabricated composites. Micrographs show the uniformly distributed in-situ generated $ZrB_2$ particles and externally added $MoS_2$ particles. In-situ formed $ZrB_2$ particles were found in varying sizes ranging from 55 nm to 2.5 µm. It is well documented that density difference between the matrix alloy and reinforcement particles significantly affect the distribution of the second phase particles. Since the density difference between Al7075 alloy and both reinforcements is more than 2 g/cm$^3$, the particles may differ for much time in the molten liquid during solidification (Rajan et al., 2014; Selvam and Dinaharan, 2017). Moreover, $MoS_2$ particles are added in the semi-solid state of the melt for the longer suspension of particles. Good wettability and semi-solid melt restricted the free movement of reinforcement particles which leads to the uniform distribution of particles throughout the matrix (Dinaharan et al., 2011; Selvam and Dinaharan, 2017). However, a cluster of particles [Figure 4(d)] is also observed in some places having good interfacial bonding with the matrix. EDS spectrum (Figure 5) of the reinforcement particles is evident of $ZrB_2$ and $MoS_2$ particles in the matrix. Further SEM mapping reveals the uniform distribution of various elements present in the matrix (Figure 6).

**Figure 4** SEM micrographs of composites with, (a) Al7075 alloy (b) Al7075 + 3 vol.%$MoS_2$ (c) Al7075 + 3 vol.%$MoS_2$ + 3 vol.%$ZrB_2$ (d) morphology of particles at higher magnification (see online version for colours)




## 3.5 TEM examination

To reveal the morphology of reinforcements, interfacial features, and dislocations present, TEM examinations were performed. TEM micrograph [Figures 7(a) and 7(b)] shows the hexagonal shape and dislocation density present in the composite. Clear interface of $ZrB_2$ with matrix is also evident from Figures 7(a) and 7(b) for improved properties.

## 3.6 Mechanical properties

Mechanical properties of all compositions are influenced by ceramic reinforcement $ZrB_2$, solid lubricant $MoS_2$ and their combination. Table 4 shows values of different mechanical properties, discussed in detail under the following section.

**Figure 5** EDS spectrum of $ZrB_2$ and $MoS_2$ particles (see online version for colours)

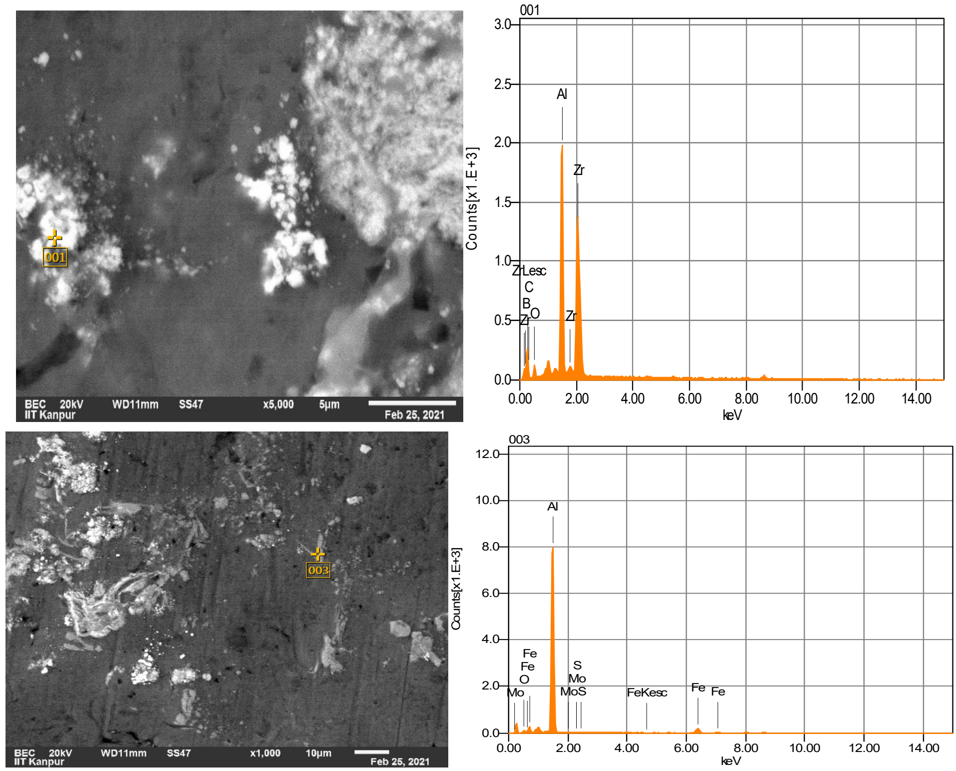


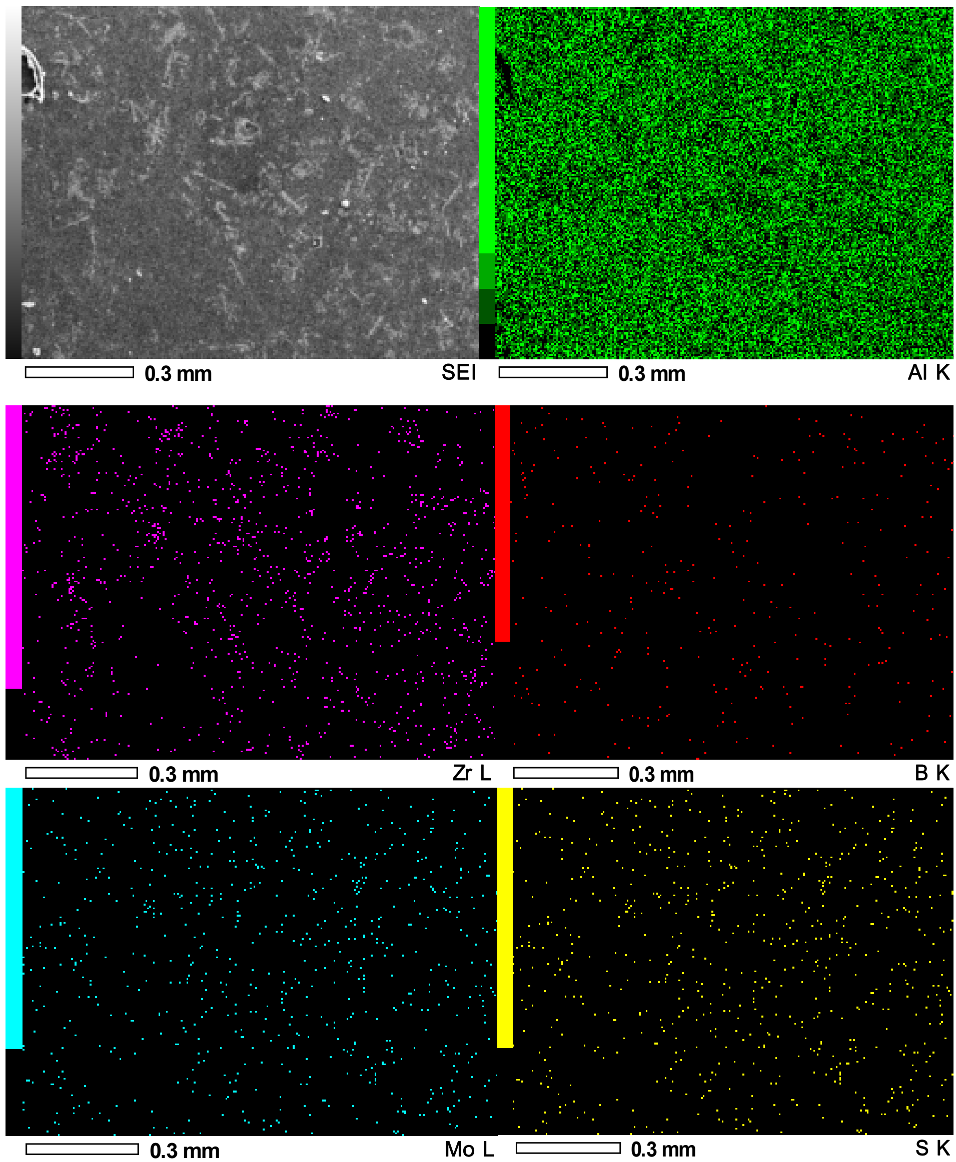

**Table 4** Mechanical properties of Al7075 alloy and composites

| *Developed material* | *Hardness (HB)* | *YS (MPa)* | *UTS (MPa)* | *% elongation* |
|---|---|---|---|---|
| Al7075 alloy | 56 | 113 | 140 | 7.3 |
| Al7075 + 3 vol.%$MoS_2$ | 51 | 109 | 137 | 13.0 |
| Al7075 + 3 vol.%$MoS_2$ + 3 vol.%$ZrB_2$ | 67 | 108 | 166 | 15.4 |

### *3.6.1 Hardness*

It is clear from Table 4, that among all compositions, composite with $MoS_2$ particles exhibit lowest hardness value due to its soft and self-lubricating behaviour while its hardness increased to maximum value with the addition of $ZrB_2$ particles. Hybrid composite, with $MoS_2$ and $ZrB_2$ particles, exhibit highest hardness value because of large number of dislocations generation around the particles. Figure 7(b) shows increased dislocation density causing the enhanced hardness. Different thermal expansion coefficient values of aluminium and $ZrB_2$ particles may introduce excessive dislocation density causing enhanced hardness (Mandal et al., 2008; Dinaharan et al., 2011). High hardness of hybrid composite may also be attributed to clear interface with good bonding, thus improving the load bearing capacity (Ramesh et al., 2010, 2011).

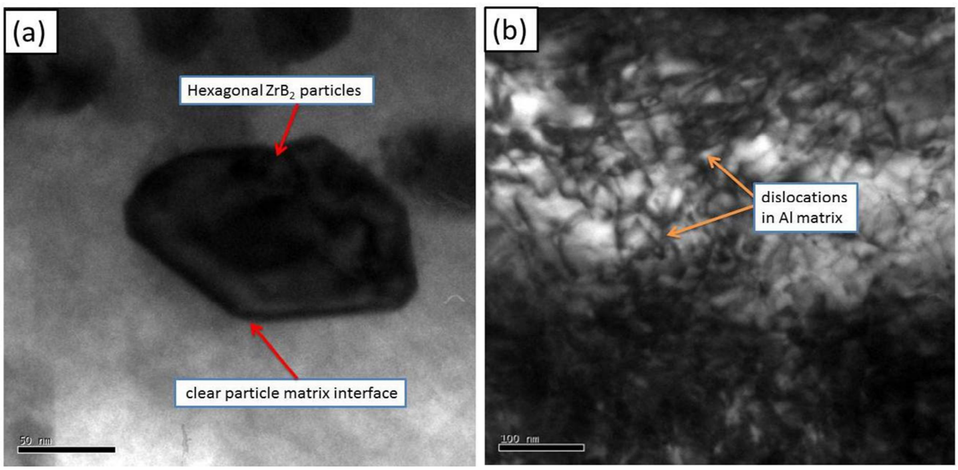
**Figure 6** SEM mapping of elemental distribution in the composite (see online version for colours)




### 3.6.2 *Tensile properties*

Tensile tests were performed at ambient conditions for evaluating the tensile properties of composites. UTM machine data was utilised to determine 0.2% offset yield strength (YS), ultimate tensile strength (UTS) and percentage elongation (ductility). It is explored that UTS of composite gets decreased with addition of soft $MoS_2$ particles however, ductility was increased while $ZrB_2$ particles increased the strength and ductility of the composite through grain refining tendency and excessive dislocation density. Good interfacial bonding also contributes to enhanced strength and thus transferring the load to the reinforcement particles effectively from the matrix (Hong-zhan et al., 2006; Khorramie et al., 2013; Ramanan et al., 2019). In contrast with many other composites, increased ductility along with good strength and hardness is the main finding of the present work. Therefore, expanding application area of Al7075 alloy in manufacturing of industrial components is needed where toughness is the main requirement.

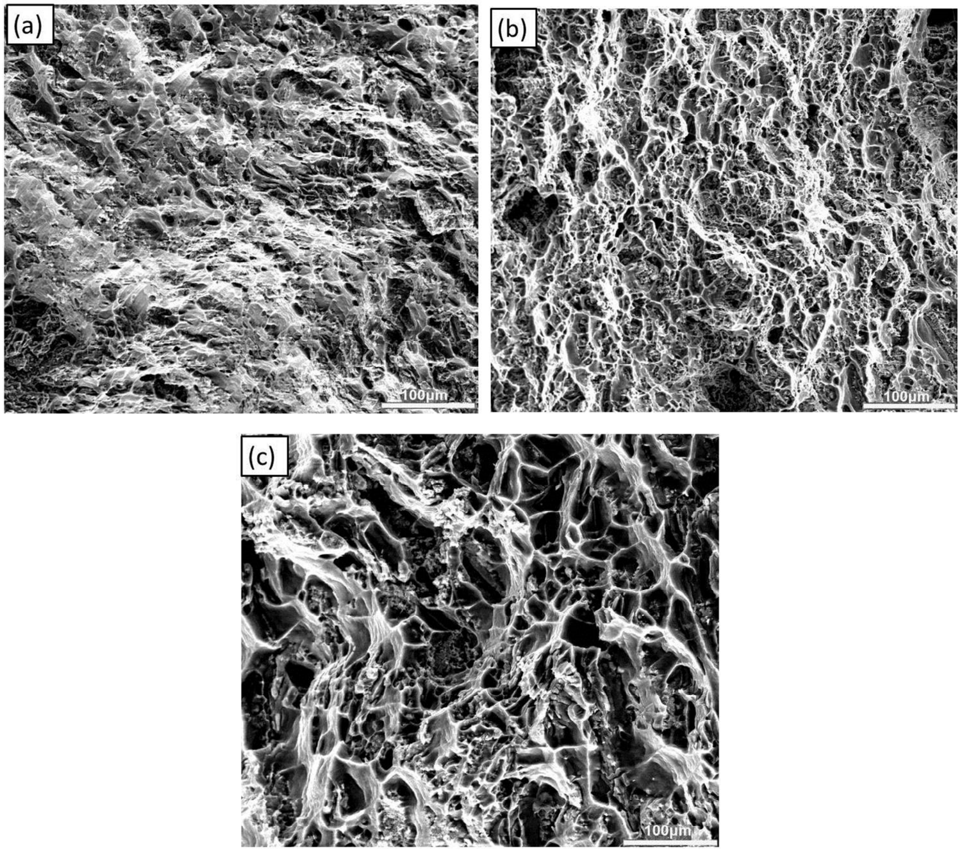
**Figure 7** TEM micrographs showing, (a) hexagonal shape of $ZrB_2$ (b) dislocations (see online version for colours)




## 3.7 *Fractography*

Fractographic analysis helps to decide failure mode and its cause in materials. Figures 8(a)–8(c) show the fractography images of Al7075 alloy and its composites. Al7075 alloy fractured surface morphology reveals few dimples, and more plane areas, indicating as mixed mode fracture [Figure 8(a)]. Whereas morphology of the fracture surface of $MoS_2$ composite shown more dimples, less plane area featured as ductile mode fracture [Figure 8(b)]. Hybrid composite having $ZrB_2/MoS_2$ particles show ductile failure with large dimpled features. Hybrid composite show maximum ductility because of the grain refiner $ZrB_2$ and soft, self-lubricating $MoS_2$ particles. However, clustered particles within the fractured surface are also visible at few places [Figure 8(c)]. Fractography surface morphology is in well agreement with the results obtained.

**Figure 8** Fractured surface morphology of, (a) Al7075 alloy (b) Al7075 + 3 vol.%$MoS_2$ (c) Al7075 + 3 vol.%$MoS_2$ + 3 vol.%$ZrB_2$ composite




## 4 Conclusions

Following points can be concluded from the present work:

1 Self-lubricating Al7075/$MoS_2$/$ZrB_2$ hybrid composite is successfully synthesised, and characterised by microstructure and mechanical investigation.

2 Ex-situ $MoS_2$ and in-situ formed $ZrB_2$ particles in the aluminium matrix were identified by XRD analysis using JCPDS software.

3 Optical microscopy reveals refined grain structure of aluminium alloy matrix which occurs due to $ZrB_2$ and $MoS_2$ particles. Average grain size reduced from 143 μm to 55 μm.

4 SEM and TEM reveals rod and hexagonal morphology of $MoS_2$ and $ZrB_2$ particles along with good interfacial bonding with the matrix. $ZrB_2$ particles were observed in the size range of 55 nm to 2.5 μm.

5 $MoS_2$-based composite exhibits reduced hardness and strength over the base alloy, however an increase in ductility is observed due to soft and self-lubricating behaviour of $MoS_2$ particles.

6 Hardness and strength of hybrid composite enhanced by 20% and 18.5% respectively over unreinforced alloy.

7 In-situ formed $ZrB_2$ particles enhances hardness and strength with good amount of ductility in composite due to increased dislocation density, good interfacial bonding and grain refining behaviour of $ZrB_2$ particles.

8 Al7075/$MoS_2$/$ZrB_2$ hybrid composite shows excellent mechanical properties because of the addition of soft $MoS_2$ and hard $ZrB_2$ reinforcement particles.

9 Hybrid composite shows increased ductility along with good strength and hardness, which is an important outcome of the study.

10 Present study widens the scope of Al7075 alloy for manufacturing of many industrial components due to enhanced mechanical properties.

## Acknowledgements

Authors gratefully acknowledge the lab facilities at IIT Kanpur and IIT (BHU), Varanasi for completing present research work.

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