A COMPARATIVE STUDY ON EX-SITU & IN-SITU FORMED METAL MATRIX COMPOSITES

An attempt has been made to synthesize the aluminium based ex-situ (Al-SiC) and in-situ (Al-TiB₂) formed metal matrix composites with varying weight percentage of reinforcement contents such as 4wt.%, 6wt.% and 8wt.%. Synthesized composites were subjected to a cold extrusion process followed by heat treatment according to the ASTM B 918-01 standards. The mechanical properties of in-situ composites were evaluated as per the ASTM guidelines and compared with ex-situ formed composites and base metal properties. Superior properties were noticed in the in-situ formed composites and the mechanical properties such as yield strength, Ultimate tensile strength (UTS) and Hardness for both ex-situ and in-situ composites were found to increase with increasing the reinforcement addition. Cold extruded Al-8 wt.% SiC composite properties such as hardness, yield strength and UTS are 87 Rb, 152 mPa, 216 mPa respectively. Whereas, for Al-8 wt.% TiB₂ composite, the corresponding properties are 94 Rb, 192 MPa, 293 MPa. The morphology of the composites is analysed by Optical and Scanning Electron Microscopic (SEM) whereas presence of reinforcement particles such SiC and TiB₂ along with intermetallic phases Mg₂Si and Al₅FeSi are confirmed by EDX, XRD and Element Mapping analyses.

Keywords: MMCs; Al-SiC; Al-TiB₂; Extrusion; Heat treatment; Mechanical properties; Microstructure

1. Introduction

Aluminium metal matrix composites (AMMCs) are widely used materials, due to the fact that they have the potential for current demands of pioneering engineering applications [1,2]. Because of its high specific strength, superior formability and corrosion resistance, aluminium and its alloys are widely employed in numerous industries such as aerospace, transportation, and a variety of structural applications [1,3]. Porosity, bigger grain size, particle clustering, poor bonding, poor wetting, and unfavourable interfacial reaction product are common issues of AMMCs manufactured using the classical ex-situ technique, in which externally prepared reinforcements are put into the matrix material [1-4]. The wettability of ceramic particulates is one of the major criteria to achieve good interfacial bonding between the matrix and reinforcement phases. Though most of the ceramic particles i.e., SiC, Al₂O₃ are not wetted by the aluminium melt and hence it is difficult to obtain uniform dispersion of reinforcement particles throughout the aluminium matrix. Contact angle is one of the signify factors for the wettability of the composite.

Wettability was enhanced with decreasing the contact angle between Al and ceramic reinforcements [5,6]. In other words, contact angles between ceramic particles (SiC, Al₂O₃, graphite) and molten aluminium are important factors for the wetting behaviour. Sarina et al. (2012) reported that the contact angles of Al/Al₂O₃, Al/SiC and Al/graphite at 700°C are calculated to be 97°, 79°, 92° (for vitreous graphite) and 126° (single- and poly-crystal graphite), respectively. At elevated temperature both SiC and Al₂O₃ are reacted with aluminium alloying elements, resulting in formation of MgAl₂O₄ and Al₅C₃ brittle intermetallic phases. These brittle intermetallic phases are severely affecting the properties of the composite. To overcome these difficulties, in-situ formed TiB₂ particles is one of the major solutions to counteract wettability associated problems [7,8]. As reported by Rhee et al. the contact angle of Al on hot-pressed TiB₂ was 55° at 98% theoretical density at 840°C. Storozhenko et al. [4] reported that the NiCrBSiC alloy had good wetting properties with TiB₂ (θ = 34°). Hence the wettability of Al/TiB₂ good as compared with Al/Al₂O₃, Al/SiC and Al/graphite MMCs. Low density, good thermal and electrical properties, corrosion resistance,
and good damping capacity make aluminium alloys one of pre-
dominant matrix materials for composite [6,7]. Fabrication of
AMMCs through in-situ technique comprises synthesizing suit-
able reinforcements while the process itself. This method offers
notable advantages over the traditional ex-situ method in terms
of its capacity to produce smaller grain sizes and maintaining
high thermodynamic stability [8,9]. Nowadays, many researchers
have fabricated and characterised the mechanical and tribological
properties of in-situ formed MMCs but, meagre work has been
reported so far related to effect of secondary working and heat
treatment of this composites. In spite of the fact that the stir
casting technique will yield cast metal with good mechanical
and tribological properties [10]. The process parameter must
be chosen in such a manner to achieve defect free, good quality
cast metal [5]. The material properties predominantly depend on
the quality of the cast metal [9,10]. In other words, synthesized material properties generally depend on the process parameters
such as reinforcement particles addition, stirring speed, stirring
time, holding time, secondary working and heat treatment [7-9].
Among these parameters, reinforcement addition, secondary
working and heat treatment play a vital role for deciding the
final properties of composites [10]. Reinforcement particle add-
ition has a significant role in deciding mechanical properties
[11-14]. The addition of reinforcing particles into the matrix
gives superior mechanical properties than base metal but beyond
a certain level, it may reduce the properties. It is due to the fact
that, when the addition of particles exceeds a certain volume or
weight percentage, clustering or agglomeration, porosity and
pin holes were formed in the cast metal resulting in defects and
hence materials undergo failure [15]. The intrinsic limitations of
the casting process limited the use of as-cast composites and they
are exposed to secondary working followed by the heat
treatment [16,17]. The morphology of the composite is generally
changed during secondary working because of the distinction
in the properties between the ceramic reinforcement and the
ductile matrix which offer the beneficial effect to improve the
final properties of the composites. In other words, the increase
in mechanical properties of secondary worked materials can be
attributed to the improvement of microstructural densification,
interface bonding and lowering of porosity [13,15]. Moreover,
selection of reinforcement is one of the key roles to decide the
composite properties [16]. The reinforcement particle size, shape,
and bonding strength are playing a vital role for deciding the
final properties composite. Ex-situ formed SiC particles have
an irregular shape and are larger in size, hence crack initiation
is faster than in fine in-situ formed TiB₂ particles. This is due to
the in-situ formed TiB₂ particles have micron in size and good
interfacial bonding strength [12,16]. Heat treatment of particulate
reinforced composite comprises solutionizing, quenching and
ageing. The heat-treatment is carried out not only to improve
mechanical properties, but also to remove induced stresses and
structural instability [16,17].

Chidambaran et al. [19] reported that 5wt.% TiB₂ particle
addition in the MMC gives better mechanical properties. Rajaravi
et al. [17] investigated the hardness and tensile strength properties
of Al-6wt.% TiB₂ composite and it was observed that a significant
improvement of hardness and tensile strength was achieved.
Many research workers in their earlier studies, the mechanical and
tribological characterization of in-situ formed Al-TiB₂ MMC with
less than 6wt.% of reinforcement addition and limited literatures
only are reported so far related to Al-TiB₂ MMC above 6wt.%. Hence,
in this investigation, an effort has been made to study the effect TiB₂ particles addition (4wt.% to 8wt.% with increment of
2wt.%) on mechanical properties of an aluminium based
MMCs and compared with Al-SiC (ex-situ) MMCs. Moreover,
this paper is a follow up work of the published paper [11].

2. Experimental work

The selected base metal for this investigation was wrought
Aluminium 6061 alloy. The weight fraction of the individual
alloying components contained in the base metal was estimated
using a vacuum spectrometer (ARL, model 3460). The spect-
raums were obtained by igniting sparkles at various locations
and estimating their compositions (TABLE 1).

The fabrication of ex-situ formed Al-SiC MMCs consist
of incorporation of Silicon Carbide particles (SiC) with sizes of
22 μm and weight percentages of 4wt%, 6wt%, and 8wt% into
the aluminium melt at a temperature of 720°C, whole melt was
carried out by the stir casting method. The melt temperature
was kept between 700 and 750°C. The current rating was kept
at 20Amps. SiC powder was pre-heated at 750°C for 2 hours in
a muffle furnace before being gradually added to molten aluminium.
Nitrogen gas was applied to the surface of the aluminium
melt at a rate of 4 litres per minute, and 1 percent Magnesium
was added and mechanically stirred. The mechanical stirrer
is made of mild steel, rotates at a constant speed of 300 rpm.
The temperature was then kept between 710 and 730°C. Within
the next 15 minutes, all of the SiC powder was completely
dropped into the melt, and the stirring process was continued for
an additional 10 minutes. After 25 minutes of total stirring, the
720°C melt was poured into the permanent mould and allowed
to solidify at room temperature. The permanent mould was also
preheated at 250°C for one hour before pouring to remove any
moisture that had adhered to the mould.

The initiating materials for the fabrication of in-situ formed
Al-TiB₂ MMCs are potassium hexafluoro titanate (K₂TiF₆),

<table>
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<th>Elements</th>
<th>Mg</th>
<th>Si</th>
<th>Mn</th>
<th>Fe</th>
<th>Cu</th>
<th>Zn</th>
<th>Ti</th>
<th>Cr</th>
<th>Al</th>
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<td>Composition (weight %)</td>
<td>0.85</td>
<td>0.69</td>
<td>0.067</td>
<td>0.214</td>
<td>0.221</td>
<td>0.013</td>
<td>0.025</td>
<td>0.077</td>
<td>Remaining</td>
</tr>
</tbody>
</table>

TABLE 1

Chemical composition of base metal (Al6061)
potassium tetrafluoroborate (KBF₄), and wrought aluminium 6061. These halide salts were mixed in Al-TiB₂ composites with stoichiometric compositions of 4wt. percent, 6wt. percent, and 8wt. percent TiB₂. The aluminium was melted in a graphite crucible. Under normal atmospheric conditions, an electrical resistance furnace was used. The (K₂TiF₆) and (KBF₄) salts were preheated at 250°C for 30 minutes before being manually incorporated into the liquid aluminium, which was kept at 820°C. The temperature was maintained at this level for 15 minutes to allow the in-situ TiB₂ particles to form in the matrix. To prevent atmospheric contamination, nitrogen gas was supplied through a fine copper tube. The dross was skimmed off the surface of the melt twice: once before adding salts and another just before pouring. The composite melt was cast in a sand mould to fabricate the cast ingot. As-Cast Al6061 alloy and synthesized composite ingots were machined into the required number of billets (18 mm diameter and 90 mm length) from each ingot. Under laboratory conditions, billets were cold extruded with an extrusion ratio of 1.2656.

The schematic image of an extrusion die is shown in Fig. 1. Fig. 2(a) depicts as cast and extruded samples. All as-cast and cold extruded composite samples were shaped into the required number of tensile and hardness specimens per the ASTM E8M-16 and ASTM E18-15 standards, respectively, and then subjected to heat treatment per the ASTM B 918-01 guideline. As shown in Fig. 3, the heat treatment processes involved were solutionizing, hot bath quenching, ageing, and furnace cooling. Solutionizing was carried out for one hour at 540°C. To avoid warping of specimens, generally quenching was carried out through hot water at 43°C. Immediately, artificial ageing was performed in a muffle furnace at a temperature of 171°C for 8 hours. Temperatures were maintained to within 2°C in both solutionizing and ageing, and quench delays were kept to within 10 seconds in all cases.

Microstructural investigations were studied using a light optical microscope (make: MEJI, Japan; Model: MIL 7100). The test samples were machined from as-cast and cold extruded composites. Specimens were polished with all sides to ensure the flatness. To ensure flatness, specimens were polished on all sides. The specimens for metallographic analyses were generally rough polished with higher-grade emery sheets before being fine polished with a series of water emery sheets. Final polish was carried out by a disc polishing machine with alumina powder. The specimens were etched according to the ASTM standard E407 with standard Keller’s reagent containing 190 ml of distilled water,
5 ml of HNO₃, 3 ml of HCl and 3 ml of HF. The chemical etchants were thoroughly washed in running water. Following that, the etched samples were placed in the optical microscope to spot the macrograph. SEM (Make: JEOL, JSM-6610LV) analysis was conducted in the Scanning Electron Microscopic laboratory, Department of Manufacturing Engineering, Annamalai University, Tamilnadu.) Energy-dispersive X-ray spectroscopy (EDX) analysis is primarily useful in understanding the main microstructure, fracture analysis, boundary studies, and elements in composites. XRD and Element Mapping analysis (XRD and Element Mapping analyses were conducted in PSGTECHS COE INDUTECH Laboratory, Center of Excellence for Industrial & Home Textiles, Promoted by Ministry of Textiles – Government of INDIA.) used to identify the phases present in the fabricated MMCs.

2.1. Limiting of weight percentage of reinforcement addition

Two trial tests have been conducted on Al/10wt.% TiB₂ MMC, mechanical and microstructural characterization was analysed. The tensile strength, Elongation at fracture and hardness of the first tail is 24 MPa, 9.2% and 45 R₉₀ respectively whereas the second trail is 23 MPa, 9.8% and 42 R₉₀ respectively which was very poor compared to base metal as shown in Fig. 4. It is because of the formation of Al₃Ti and AlB₂ intermetallic coupled with TiB₂ particles agglomeration in aluminium matrix as shown in Fig. 5(a & b). This reduced the load carrying capacity of the composite material and propagation of crack was accelerated. When the Al-10wt.% TiB₂ composite billet was subjected to a cold extrusion process, it could not withstand the extrusion load and broke into three pieces as shown in Fig. 3(b). Hence, the percentage weight fraction of TiB₂ addition is limited within the range of 4wt.% to 8wt.%. 

Fig. 4. stress strain curve for Al/10wt.% TiB₂ MMC

Fig. 5. SEM micrograph and EDX analysis of Al-10wt.% of TiB₂ MMC
3.1. Effect of cold extrusion on microstructure of Al-SiC MMCs (Ex-situ)

The microstructure of the cold extruded & heat-treated base metal and its Al-SiC MMCs are shown in Fig. 6. As shown in Fig. 7(b-d), the SiC particles are dispersed more homogeneously in the cold extruded composites after heat treatment. Clusters of SiC particles initially present in the as-cast condition (Fig. 6(b-d)) have completely disappeared [22]. This is because the shear stress applied during cold extrusion splits the SiC clusters and results in more homogeneous distribution. According to the Hall-Petch effect, after the cold extrusion process, aluminium grains have been reduced. The average grain size of the as-cast condition lies between 75-90 μm (Fig. 6) whereas in cold extruded condition 50-70 μm (Fig. 7). Hence, numerous aluminium grain boundaries were formed as shown in Fig. 7. These grain boundaries act as barriers to the dislocation motion hence, the mechanical properties were enhanced. As shown in Fig. 7, the presence of Mg2Si

Fig. 6. Optical Microstructure of as-cast base metal & Al-SiC MMCs. a) base metal, b) Al-4wt.% SiC MMC, c) Al-6wt.% SiC MMC and d) Al-8wt.% SiC MMC

Fig. 7. Optical microstructure of cold extruded base metal & Al-SiC MMCs. a) base metal, b) Al-4wt.% SiC MMC, c) Al-6wt.% SiC MMC and d) Al-8wt.% SiC MMC
and Al₂FeSi intermetallic phases can be seen in the base metal and its composites. These intermetallic phases are precipitated during ageing, contribute to the increase in strength of the base metal as well as composites [23]. Fig. 8(a-d) shows the SEM micrographs of base metal and its Al-SiC MMCs at cold extruded & heat-treated condition.

They reveal that the SiC particles are uniformly distributed in the aluminium matrix. The size of the SiC particles may be found smaller after extrusion and the same can be because of breakage of particles during extrusion. On the whole, the strength of the composites can be attributed to the uniformly distributed SiC particles along with Mg₂Si intermetallic phases.

Fig. 8. SEM micrographs of cold extruded base metal & Al-SiC MMCs. a) base metal, b) Al-4wt.% SiC MMC, c) Al-6wt.% SiC MMC and d) Al-8wt.% SiC MMC

Fig. 9. XRD results of cold extruded base metal & Al-SiC MMCs. a) base metal, b) Al-4wt.% SiC MMC, c) Al-6wt.% SiC MMC and d) Al-8wt.% SiC MMC
XRD studies performed on cold extruded & heat-treated Al-SiC MMCs and base metal (Fig. 9) indicate the presence of Al, SiC, Mg$_2$Si, and Al$_3$FeSi. During heat treatment, supersaturated solid solution of Mg and Si in aluminium matrix causes the formation of Mg$_2$Si intermetallic phase and sometimes reacts with Fe forming Al$_3$FeSi phase. The formation of these intermetallic phases will influence the enhancement of properties.

Moreover, the intensity of Mg$_2$Si peaks progressively decreases with increasing presence of hard ceramic reinforcement [24,25]. This is because, when the contents of reinforcements increase, it leads to increase in the number of particle interfaces and this affects precipitation process and hence, reduces the Mg$_2$Si intermetallic phases. Fig. 10 shows EDX analysis of Al-8 wt.% SiC MMC, which confirms the presence of aluminium, SiC and Mg$_2$Si intermetallic and its alloying elements viz. Si, Mg, Cr, Fe in the cold extruded and heat-treated composites. These reinforcements and intermetallic phases are confirmed by XRD analysis in the Fig. 9.

Similar trend was noticed in the element mapping analysis as shown in Fig. 11. For Al-8 wt.% SiC MMC the elemental compositions are Si-5.21%, Mg-6.52%, C-4.32%, Fe-2.01%, Cr-0.73%, and Al-81.21% and these values are very close to the EDX results. Hence, the presence of SiC particles along with Mg$_2$Si intermetallic phase are once again confirmed by this analysis.

Fig. 10. EDX results of cold extruded Al-8wt.% SiC MMC

Fig. 11. Element mapping analysis of cold extruded Al-8wt.% SiC MMC
3.2. Effect of cold extrusion on macro hardness of Al-SiC MMCs

TABLE 2 shows mechanical properties of cold extruded Al-SiC MMCs. Increases in the weight percentages of SiC content can result in a significant increase in hardness. The maximum improvement of hardness of cold extruded Al-8wt.% SiC MMC over base metal is 29.85%. It is concluded that, the enhancement of hardness is mainly attributed to the content and uniform dispersion of SiC particles, microstructural densification and precipitation of fine Mg2Si intermetallic phase. In general, when SiC particles are incorporated into a soft aluminium matrix, they resist dislocation movement, increasing the hardness of the composite. Extrusion process disburse clusters, reduces pores and improves the microstructural densification leading to increase in hardness of the composites [23,25]. Nevertheless, during heat treatment, formation of fine Mg2Si intermetallic phases will resist the dislocation motion. The above discussed phenomenon’s influences the increase in hardness of the cold extruded composites [16,17,26].

3.3. Effect of cold extrusion on tensile property of Al-SiC MMCs

The increase in tensile properties (Fig. 12) of composites is mainly governed by matrix and reinforcement material properties, as well as secondary processing. Cold extruded base metal properties such as 0.2 percent proof strength and UTS are 102 MPa and 175 MPa, respectively, whereas Al-8 wt. percent SiC composite properties are 152 MPa and 216 MPa, respectively. Cold extruded Al-8 wt. percent SiC composite improves proof strength and UTS by 0.2 percent over base metal by 49.01 percent and 23.42 percent, respectively.

The cold extruded Al-SiC MMCs have more 0.2% proof strength, UTS than that of base metal. This is because secondary working coupled with heat treatment will result in reduction of the pores and agglomeration present in the composites. Meanwhile, extrusion increases the fraction of aluminium grain boundaries [27]. Fine Mg2Si intermetallic phase gets precipitated during heat treatment, playing a dominant role in dictating the final properties of composites [28]. In other words, during extrusion, densification takes place and thus increases grain boundaries resulting in low deformation during tensile load. Furthermore, the coefficient of thermal expansion mismatch between matrix (23.5×10⁻⁶ K⁻¹) and SiC (4.7×10⁻⁶ K⁻¹) results in residual plastic strain between matrix and reinforcement, increasing composite strength [27,29]. The elongation to failure decreases gradually as the fraction of ceramic particles in the matrix increases. This is due to the brittle nature of the ceramic particles, which reduces the ductility of the matrix [17,29,30].

3.4. Fractography of cold extruded Al-SiC composite

Fig. 13, reveals brittle fracture of SiC particles. Meanwhile, the surrounding matrix exhibits ductile shear bonds and shallow dimples confirming features of ductile rupture.

The correlation between the interfacial bonding strength and particle strength is the critical phenomenon for deciding fracture mode of the composites during tensile test. Extrusion increases the strength of the particle-matrix interfacial bonding, allowing the applied tensile load to be effectively transferred to the reinforcing phases [26,27]. However, beyond a certain point, particle fracture occurs, as evidenced by the microstructure.

4.1. Effect of cold extrusion on microstructure of Al-TiB2 MMCs (In-situ)

The composite microstructure (Fig. 14) shows that TiB2 particles are uniformly distributed in the matrix, along with

<table>
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<th>Cold Extruded condition</th>
<th>Rockwell hardness (RN)</th>
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<th>Ultimate tensile strength (MPa)</th>
<th>Elongation in gauge length (%)</th>
</tr>
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<tr>
<td>2</td>
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<td>112</td>
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</tr>
<tr>
<td>3</td>
<td>Al-6 wt.% SiC</td>
<td></td>
<td>76</td>
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<td>191</td>
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</tr>
<tr>
<td>4</td>
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<td>87</td>
<td>152</td>
<td>216</td>
<td>24.6</td>
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</table>
Mg$_2$Si intermetallic phases. TiB$_2$ particle clusters are completely eliminated by the extrusion as compared to as cast condition (Fig. 15). Moreover, due to Hall-Petch effect the aluminium grains size is considerably reduced after extrusion. Extrusion followed by heat treatment reduces pores and clusters while increasing microstructural densification, as shown in Fig. 16. Hence, numerous numbers of TiB$_2$ particles are more uniformly distributed in the matrix as compared to that of as-cast conditions of the same. The lack of brittle intermetallic phase (Al$_3$Ti) and distribution of fine equally dispersed TiB$_2$ particles along with Mg$_2$Si precipitates significantly improves composite strength and hardness [22].

The presence of Aluminium, TiB$_2$, in-situ by-products such as Al$_3$Ti & AlB$_2$ and intermetallic phases like Mg$_2$Si, Al$_3$FeSi are confirmed by XRD analysis as shown in Fig. 17. Fig. 17(b-d) clearly indicates Mg$_2$Si peaks to decrease with increase in TiB$_2$ peaks. This phenomenon may find to have been discussed in section 3.1.

As shown in Fig. 18, EDX analysis of Al-8wt.% TiB$_2$ MMC, also confirms the presence of aluminium, TiB$_2$ and Mg$_2$Si intermetallic in the cold extruded and heat-treated composites.

Similar trend can be noticed in the element mapping analysis as shown in Fig. 19. For Al-8wt.% TiB$_2$ MMC, Si-2.84%, Mg-4.82%, B-8.96%, Ti-4.23%, Fe-1.92%, and Al-77.23% elemental compositions can be observed and found to be very close to the EDX results.

### 4.2. Effect of cold extrusion on macro hardness of Al-TiB$_2$ MMCs

TABLE 3 shows the change in hardness of the cold extruded Al-TiB$_2$ MMCs as a function of TiB$_2$ particle addition. The cold extruded composites can be found to have superior hardness than in base metal [31]. The maximum hardness improvement of cold extruded Al-8wt.% TiB$_2$ MMC over base metal is 40.29 percent. The increase in hardness can be attributed to in-situ formed sub-micron TiB$_2$ particles that are distributed evenly in the matrix (Orowan strengthening mechanism). In other words, the in-situ formed TiB$_2$ particles have higher hardness and stiffness than base metal, which may increase their resistance to plastic deformation [32]. Also, the reduction of aluminium grain size during extrusion, increases the density of the grain boundaries significantly and this could result in increase in hardness of the composites.
As explained by Hall-Petch, the relationship between hardness and grain size is follows: [33].

$$H = H_o + kd^{(-1/2)}$$  \hspace{2cm} (4.1)

Where, $H_o$ – equivalent to the friction stress, which is the intrinsic resistance of the crystal to plastic deformation, $d$ – grain size and $k$ – constant specific to each material. As per the above equation, it is clear that the reduction of grain size during the extrusion
process has a predominant role for increasing the hardness of the composites. The other important factors influencing the hardness of the composite are interfacial bonding strength, wetting angle and atomic nature of the interface [33,34].

4.3. Effect of cold extrusion on UTS of Al-TiB₂ MMCs

Engineering stress strain curves for cold extruded Al-TiB₂ MMCs are shown in Fig. 20. The tensile properties of Al-TiB₂ composites after cold extrusion are shown in TABLE 3. As can

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<th>S.no</th>
<th>Synthesized materials</th>
<th>Cold Extruded condition</th>
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<td>4</td>
<td>Al-8 wt.% TiB₂</td>
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be seen, the 0.2 percent proof strength and UTS of the composites increase as the TiB$_2$ particle concentration increases. Cold extruded base metal properties such as 0.2 percent proof strength and UTS are 102 MPa and 175 MPa, respectively, whereas Al-8 wt. percent TiB$_2$ composite properties are 192 MPa and 293 MPa.

The maximum improvements in 0.2 percent proof strength and UTS of Al-8 wt. percent TiB$_2$ composite after cold extrusion are 88.23 percent and 67.42 percent, respectively, over base metal. The strength of the cold extruded and heat-treated composite is primarily attributed to two distinct approaches. The first is defined as the effective load transmission capacity from matrix to reinforcement, in which bonding strength and casting quality play a significant role. In this case, an In-situ formed TiB$_2$ particle has good bonding strength and the extrusion process increases the bonding strength. The second approach consists of three distinct mechanisms: the first mechanism is concerned with the grain refinement of the Al matrix, which is accomplished by the addition of Reinforcement particles to the Al matrix (Hall-Petch effect). It is understood that grain boundaries act as obstacles to dislocation motion, resulting in an increase in yield stress. TiB$_2$ particles restrict crystal growth in the aluminium matrix phase and enhance the fraction of grain boundary area [4]. Second mechanism entails the Orowan strengthening effect of TiB$_2$ particles. According to this mechanism, a large volume fraction of fine and sub-micron TiB$_2$ particles dispersed uniformly in the aluminium matrix would act as obstacles to dislocation motion [35]. As a result, dislocation loops form around TiB$_2$ particles, enhancing the stress required for further deformation. The final one is the emergence of residual plastic strain between the matrix and

![Element mapping analysis of cold extruded Al-8wt.% TiB$_2$ MMCs](image)

![Engineering stress strain curves of cold extruded base metal & Al-TiB$_2$ MMCs](image)
reinforcement as a result of a thermal mismatch in the coefficient of thermal expansions, which causes an increase in dislocation density. Thus, in terms of strength increase, the aforementioned phenomena apply to the Al-TiB₂ composite [35-37]. When the elongation to failure is considered, a continuous decrease in ductility is observed as the amount of TiB₂ particles increases. Section 3.3 covered detail about why this is so.

4.4. Fractography of cold extruded Al-TiB₂ composite

As it is seen in Fig. 21, many fine and deep dimples appear on the fracture surface of cold extruded Al-8wt.% TiB₂ MMC. Moreover, it is also observed that some particles exist in some dimples, yet most of the dimples do not contain TiB₂ particles and thus it is decided that the failure of in situ composite is mainly caused by particle detachment. There is no evidence of particle fracture [38-40]. The particles and their surrounding regions are subjected to tensile load during the tensile test; the strongly embedded TiB₂ particles in the matrix are pulled out, resulting in the formation of dimples. As it is seen fig. 24, Most of the dimples are less than 5 μm in diameter and are mainly caused by in situ formed TiB₂ particles. As a result, it is concluded that the increment in mechanical properties is primarily due to TiB₂ particle pull outs. Because particle pull outs acquire more energy during deformation, they increase strength and toughness [41].

![Fractured surface of cold extruded & heat-treated Al-8wt.% TiB₂ MMC](image)

Fig. 21. Fractured surface of cold extruded & heat-treated Al-8wt.% TiB₂ MMC

5.1. Comparison of Mechanical properties of Ex-situ & In-situ formed MMCs

As observed in TABLE 4 & Fig. 22(a), In-situ formed Al-TiB₂ MMCs have significant improvements in hardness as compared to the hardness values of cold extruded Al-SiC MMCs of weight fractions of 4wt.%, 6wt.% and 8wt.% SiC. The hardness values of cold extruded Al-TiB₂ MMCs of corresponding weight fractions of TiB₂ are higher by, 5.71%, 7.89% and 8.04% respectively than Al-SiC MMCs. It is clear that the enhancements of hardness of in-situ composites over ex-situ composites are not more than 10%. It is because, during the hardness test, the localised pressure was induced directly beneath the indenter surface to endorse the majority of the load. As a result, the stress field directly beneath the indenter is widely compressive with the matrix when subjected to severe triaxial pressure.

Both the SiC and TiB₂ particles can withstand the compressive load effectively. The deformation caused by the indentation pressure is accommodated by the plastic flow of the matrix between the particles, and the particles are driven into the matrix material [40,46].

As a result, the hardness of the composites increases as the weight fraction of SiC particles increases. From the above discussion, both the SiC and TiB₂ particles are almost equally effective in resisting the indentation load during hardness test. Hence the difference is much less than other properties.

Variation of 0.2% proof strength corresponding to varying wt.% of reinforcement addition with different working conditions are shown in TABLE 4 & Fig. 22(b). It is concluded that, in-situ composites have a higher 0.2% proof strength. As compared to the 0.2% proof strength of cold extruded Al-SiC MMCs of weight fractions of 4wt.%, 6wt.% and 8wt.% SiC, the 0.2% proof strength of cold extruded Al-TiB₂ MMCs of corresponding weight fractions of TiB₂ are higher by, 13.39%, 23.80% and 26.31% respectively.

Two approaches can be used to increase the 0.2 percent proof strength of aluminum-based in-situ composites. First approach involves two phenomenons, one is based on the efficient load transfer capacity from matrix to reinforcement, in which bonding strength and casting quality have played a significant role and the second is based on the effect of TiB₂ particles on aluminium matrix yield strength. The second approach includes three distinct mechanisms: the first mechanism is matrix grain refinement, which is accomplished by incorporating TiB₂ par-

<table>
<thead>
<tr>
<th>S.no</th>
<th>Materials</th>
<th>Rockwell hardness (Rₐ)</th>
<th>0.2% Proof Strength (σ₀) in MPa</th>
<th>Ultimate tensile strength (σₘ) in MPa</th>
<th>Elongation in gauge length (%)</th>
</tr>
</thead>
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<tr>
<td></td>
<td></td>
<td>AI-SiC</td>
<td>AI-TiB₂</td>
<td>AI-SiC</td>
<td>AI-TiB₂</td>
</tr>
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<td>1</td>
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<td>67</td>
<td>102</td>
<td>102</td>
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<tr>
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<td>4wt.%</td>
<td>70</td>
<td>74</td>
<td>112</td>
<td>127</td>
</tr>
<tr>
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<td>6wt.%</td>
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<td>126</td>
<td>156</td>
</tr>
<tr>
<td>4</td>
<td>8wt.%</td>
<td>87</td>
<td>94</td>
<td>152</td>
<td>192</td>
</tr>
</tbody>
</table>

TABLE 4
ticles into the matrix material (Hall-Petch effect). The grain boundaries are believed to act as barriers to dislocation movement [42,43]. As a result, 0.2 proof strength is increased. TiB$_2$ particles limit the grain growth of the aluminium matrix phase and greatly increase the grain boundary. Furthermore, during cold extrusion, composites are subjected to serious compressive stress along the transverse direction, resulting in significantly reduced grain size and an increase in 0.2 percent proof strength [44,45].

According to the Second mechanism, a higher volume fraction of fine and sub-micron TiB$_2$ particles distributed uniformly in the aluminium matrix would act as obstacles to dislocation motion. As a result, dislocation loops form around TiB$_2$ particles, increasing the stress required for further deformation [42,46]. Third is the establishment of residual plastic strain between the matrix and reinforcement as a result of a thermal mismatch in thermal expansion coefficients, resulting in an increase in dislocation density [43-46]. The effect of heat treatment on 0.2% proof strength, is mainly attributed by the preeminent holding temperature (around 540°C at one hour) of the composite during the solutionizing period. It leads to an increase in dislocation density due to imbalance in coefficients of thermal expansion between the aluminium matrix (23.5×10$^{-6}$ K$^{-1}$) and the TiB$_2$ reinforcement (7×10$^{-6}$ K$^{-1}$) at the interface during water quenching. This effect is a major contributor to the increased strength of composites. Thus, in terms of strength increase, the aforementioned phenomena apply to the Al-TiB$_2$ composite. The Ultimate tensile strength of ex-situ & in-situ formed composites are shown TABLE 4 & Fig. 22(c). As compared to the UTS of as-cast Al-SiC MMCs of weight fractions of 4wt.%, 6wt.% and 8wt.% SiC, the UTS of as-cast Al-TiB$_2$ MMCs of corresponding weight fractions of TiB$_2$ are higher by, 21.73%, 26.70% and 35.64% respectively. In-situ formed MMCs have higher UTS than ex-situ MMCs. This is because TiB$_2$ is finer and more regular in shape than SiC. It is embedded into a soft molten aluminium, resulting in increased deformation resistance and, as a result, an improved UTS of the composite material. The modification and fineness of the reinforcement also play an important role in deciding composite material strength. Fine and clear interfacial bonding is achieved through the exothermic reaction that occurs during in-situ composite fabrication. Likewise, manufacturing technique and casting soundness strengthen the composite significantly [42-45]. The load transfer capability from matrix to reinforcement is enhanced by the reaction free interface. The strength of the composite increases as the matrix’s load transfer capability to the reinforcement increases [43]. It is noted that the homogeneity of TiB$_2$ particles, associated with a reaction-free interface between matrix and reinforcement, allows for effective load transfer from matrix to reinforcement. As a result, the strength of the composites improves.

Chidambaram et al. (2019) produced AA6061-5wt. %TiB$_2$ in-situ metal matrix composite subjected to equal channel angular pressing and reported that, the AA6061 alloy’s yield strength is 105 MPa, ultimate strength is 123 MPa, and elongation to failure is 27%. Rajaravi et al. (2019) Fabricated Aluminium based in-situ formed 6wt.% TiB$_2$ composites with different pouring temperatures such as 800°C, 820°C and 840°C and maximum obtained UTS was 165 MPa. In the present work remarkable improvement in UTS has been obtained 293 by the cold extruded composite. In general, the strength of an aluminium alloy is explained by the Hall-Petch hardening mechanism and the size of the -Al grain. Particle size in the aluminium matrix is demonstrated by Orowan mechanism and strengthening of the composite is explained through solid solution hardening of the composite. The ageing kinetics are improved by the amount of TiB$_2$ reinforcement particles [38,46].

The variations of elongation to failure of Ex-situ & In-situ formed MMCs are shown in TABLE 4 & Fig. 22(d). It is evident that the values of elongation to failure of in-situ formed composites have consistent increment over ex-situ formed MMCs. In general, in-situ formed composites retain their ductility better than ex-situ MMCs. This is due to the particle size, shape, and bonding strength of the TiB$_2$ particles. Whereas, SiC particles have an irregular shape and are larger in size, hence crack initiation is faster than in fine in-situ formed TiB$_2$ particles. During the tensile test, the particles and their surrounding regions are subjected to tensile load; the strongly incorporated TiB$_2$ particles in the matrix are pulled out, resulting in dimple formation. As it is seen Fig. 21, Most of the dimples are less than 5 μm in diameter and are mainly caused by in situ formed TiB$_2$ particles. As a result, it is concluded that the improvement in mechanical properties can be attributed primarily to TiB$_2$ particle pull outs. Furthermore, the pull-out particles absorb energy during deformation, increasing the ductility and toughness of the in-situ composites [40,42,46].

5. Conclusion

The effect of cold extrusion on microstructure and mechanical properties are concluded below:

- The microstructure revealed that the presence of Mg$_2$Si precipitation, the precipitation of Mg$_2$Si phase from the supersaturated solid solution of Mg and Si in the Aluminium matrix caused by the aging process. The presence of reinforcement particles such SiC and TiB$_2$ along with intermetallic phases Mg$_2$Si and Al$_5$FeSi are confirmed by EDX, XRD and Element Mapping analyses.
- Cold extruded Al-8 wt.% SiC composite properties such as hardness, 0.2% proof strength and UTS are 87 R$_b$, 152 MPa, 216 MPa respectively. Whereas, for Al-8 wt.% TiB$_2$ composite the corresponding properties are 94 R$_b$, 192 MPa, 293 MPa. The improvements of hardness, 0.2% proof strength and UTS of cold extruded Al-8 wt.% SiC composite over base metal are 29.85%, 49.01% and 23.42% respectively. Whereas, for Al-8 wt.% TiB$_2$ composite the corresponding improvements are 40.29%, 88.23% and 67.42%.
- As compared to Al-SiC composites, the maximum improvements of Al-8 wt.% TiB$_2$ composite properties such as hardness, 0.2% proof strength and ultimate tensile strength are 8.04%, 26.31% and 35.64% respectively.
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REFERENCES


