Study on the Development of a Quaternary Layer of B_4C-TiB_2-Al_3Ti-Al in AA7050 / B_4C Ex-Situ Composite and Influence of Heat Treatment on Mechanical and Wear Properties

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ABSTRACT

In the current investigation, AA7050-B_4C composite developed through flux assisted (K_2TiF_6 flux) stir casting method. The FESEM microstructure studies confirm the uniform distribution of the reinforcements. The EDAX analysis confirms the formation of the intermetallic phase of TiB_2 and Al_3Ti across the interface of B_4C particles and the aluminium matrix. The developed composite was heat treatment as per T-6 specifications, and a comparative study has been done on microstructures, mechanical properties and wear properties for as-cast composite(ACC) and Heat-treated composite (HTC). The effect of heat treatment on the microstructure and bond mechanism shows that the B_4C particles are fairly incorporated in the matrix. Ti decomposes and forms quaternary layers of B_4C-TiB_2-Al_3Ti-Al across B_4C particles. These layers not only prevent the decomposition of B_4C particles into the matrix, also acts as the effective load transfer layers. Consequently, there is an enhancement in tensile strength by 16.4%, hardness by 15.9%, compressive strength by 14.8%, and impact strength by 10.9%. This may be due to the interface strengthening and precipitation hardening mechanism. There is a significant reduction in wear rate and coefficient of friction after heat treatment; this may be due to the improvement in hardness and compressive strength of the composite.

Keywords: *AA7050/B_4C composite; wear; stir casting; K_2TiF_6 flux; ACC; HTC*

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Introduction

7XXX aluminium alloys are known for their high strength to weight ratio, excellent fatigue strength and appreciable corrosion resistance. These superior properties have made this material suitable for structural components of aircraft and automobiles [1-2]. Nevertheless, these alloys are week in wear resistance that restricts its application in many potential fields. Metal Matrix Composites (MMC) is the new generation engineered materials that possess the properties of matrix material and ceramic reinforcements. This combination resulted in unique mechanical properties such as high specific strength, elastic modulus, toughness and wear resistance [3]. Composite materials are highly demanded materials for automobiles and aerospace. The liquid metallurgical techniques of developing composite have some significant advantages such as simplicity, economical and manufacturing of intricate components [4]. Boron carbide (B_4C) is one of the sturdy materials (3700 HV), exhibits the high degree of chemical stability, efficient thermal properties, low density (2.52 g cm-3) and high elastic modulus of 427 GPa [5,6]. Besides these remarkable properties, B_4C lacks its wettability with aluminium below 1100 °C. Several efforts were put forward by many researchers to improve the wettability of B_4C by adding the same reacting agents such as K_2TiF_6, KCl-KF, Mg, Zr, and Ti into the molten matrix. The addition of K_2TiF_6 has got dual benefits; Ti reacts exothermally with B_4C and makes a strong interfacial bond between B_4C particles and the aluminium matrix [7,8]. The formations of a robust interfacial bond improve the mechanical and wear properties of the composite [9]. Popularly, wear is a phenomenon of progressive damage to the surfaces in terms of material loss or movement of material or by developing cracks on the surfaces caused by relative motion. In the phenomenon of wear, the combined stress of compression and shear occurs in the contact region resulting in plastic deformation [10, 13]. Therefore, the hardness of the materials plays a vital role in wear resistance. Since ceramic particles are hard materials, hence the ceramic reinforced composite have better wear resistance than unreinforced alloys [14]. Mahapatra et al. [15] reported that normal load is the dominant factor for material loss and sliding distance was a significant factor influencing coefficient of friction (COF). Similarly, Ranjith et al. [16] reported that for AA7050 alloy/B_4C_p/SiC_p hybrid composite wear rate increases with an increase in load and decreases with an increase in sliding speed.

Although, there are numerous research on the Al/B_4C composite, but the development of Al7050/B_4C composite, particularly effect of heat treatment on microstructure mechanical properties and wear properties have not been addressed yet. Therefore, the prime objective of this paper is to develop AA7050/B_4C composite though stir casting method and study the effect of heat treatment on the microstructure, mechanical and wear properties of the composite.

Materials and Methods

Al 7050 alloy (Al-5.8Zn-2.4-Cu-1.9 Mg) used as matrix material chemical composition of the alloy is shown in Table 1 and B_4C particles with mesh size F120 having an average particle size of 106 µm chosen as the reinforcement material. The chemical composition of the B_4C particles listed in Table 2. The presence of B_4C and its size confirmed from SEM and Point EDAX analysis of the particle shown in Figure 1. About 1.3 kg of the wrought Al 7050 alloy melted in an induction furnace as shown in Figure 2. Approximately 7.5 (wt %) of B_4C particles and the exactly similar amount of flux K_2TiF_6 were preheateds eparately at 400 °C for 2 hours in a muffle furnace and then mixed togather. This mixture was introduced into the molten matrix in a free flow manner and continually stirred at 350 rpm for 5 minutes. Hexachloroethane (degasser) was used for degassing the composite melt. Then the complete mixture was poured into the preheated permanent mould at 350 ºC and allowed the melt to cool slowly by insulating the mould with glass wool. The as-cast composite was then subjected to solutionising heat treatment at 480 °C to obtain a single-phase supersaturated solid solution followed by cold water quenching. Further, artificial ageing was performed at 120 ˚C for 24 hours to have controlled the decomposition of the supersaturated solid solution to form finely dispersed precipitates [17].

Table 1: Chemical Composition of Al-scrap alloy

Elements	Zn	Mg	Cu	Si	Fe	Cr	Mn	Al
% (by weight)	5.88	1.9	2.4	0.031	1.095	0.006	0.022	Balance

Table 2: Chemical Composition of B_4C

Elements	B	C	Others	B_4C
wt %	76	17	--	95

Tensile and compression test samples prepared as per ASTM E8/E8M and ASTM E9 standards shown in Figure 3(A) and Figure 3(B). Tensile and compression test carried on a universal testing machine with a crosshead speed of 0.5 mm/min. The impact test carried out on Zwick Roell impact tester machine as per ASTM E23-12c standard the test sample is shown in Figure 3(C). Three sets of test samples of tensile, compression and impact test were prepared for both ACC and HTC and the average value of each set is considered for analysis of the result.

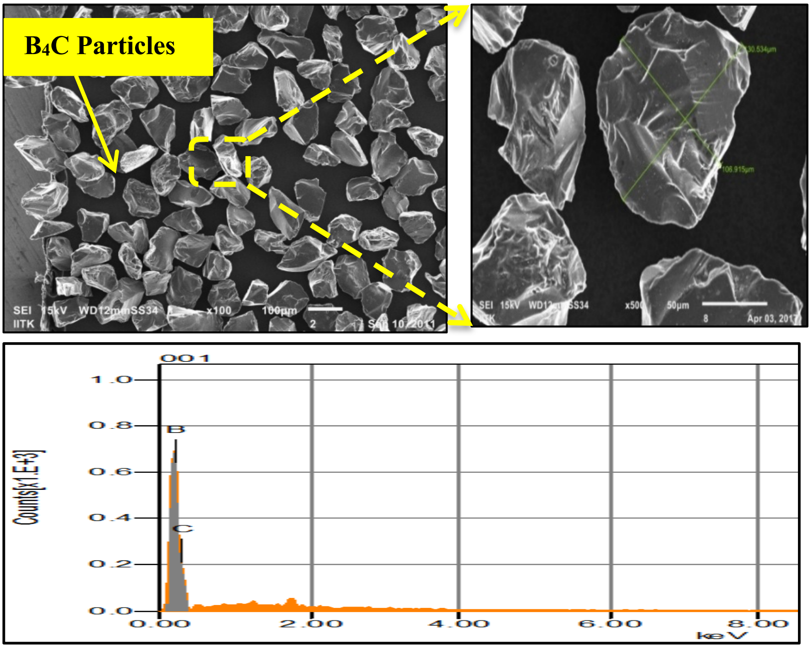


Figure 1: Boron carbide particles

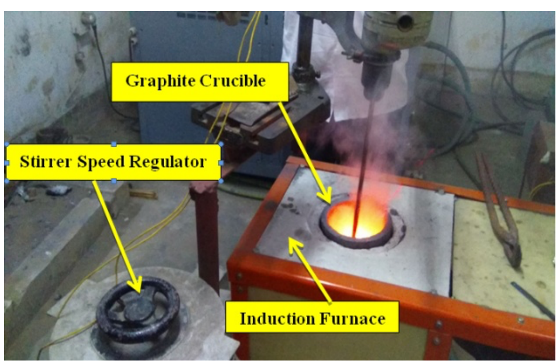


Figure 2: Stir Casting Setup

The hardness of the composite in was measured by Vickers hardness tester as per ASTM E 384, with the applied normal load of 2.5 kgf with a diamond pyramid indenter. Five measurements conducted and the average of these reading considered for reporting the hardness. For microscopic analysis, composite samples were polished and etched by Keller's reagent.

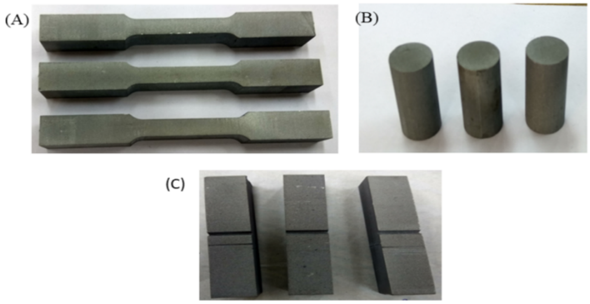
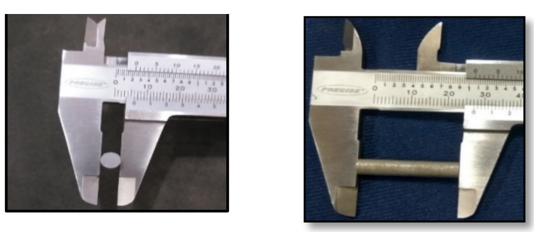


Figure 3: (A) Tensile specimen, (B) Compressive specimen, and (C) Impact specimen

Dry sliding wear tests of the composite were performed on DUCOM TR-20LE Pin-on-Disc tribometer as per ASTM G99 standard.The process parameters opted for the present experiment were normal loads and sliding speed which varies at levels of 10 N, 20 N and 30 N and of 0.5 m/s and 1 m/s respectively. Constant parameters for the wear test were sliding distance 1000 m, room temperature 23 °C and relative humidity of 60 %. The samples for wear test were cylindrical pin of diameter 6 mm and length 27 mm (Figure 4). Three tests conducted for each set of experiment. The test samples thoroughly cleaned with acetone before the test. The responses of the present analysis were wear rate and coefficients of friction (COF). Wear rate evaluated as weight loss of the pin per unit sliding distance. The weight loss of the pin during the wear test measured with a digital weighing machine of accuracy 0.001 g. COF evaluated as a ratio of frictional force to normal load.



Results and Discussions

Microstructure

Figure 5(A) and Figure 5(B) shows the microstructure of ACC and HTC. The microstructures reveal that there isa fair distribution of B_4C in the matrix with less patch of porocity. After heat treatment, the incorporation and distribution of B_4C particles in the matrix enhance (Figure 5B). As can be seen from Figure 5A that the B_4C particles surrounded by Tiaggomarations and B_4C particles clustering also appeared on the matrix surface. Furthermore, after heat treatment it found that the B_4C particles are well incorporated as Ti decomposes and form a clear reaction layer of Ti-bearing intermetallic enveloping the B_4C particles and Al matrix.

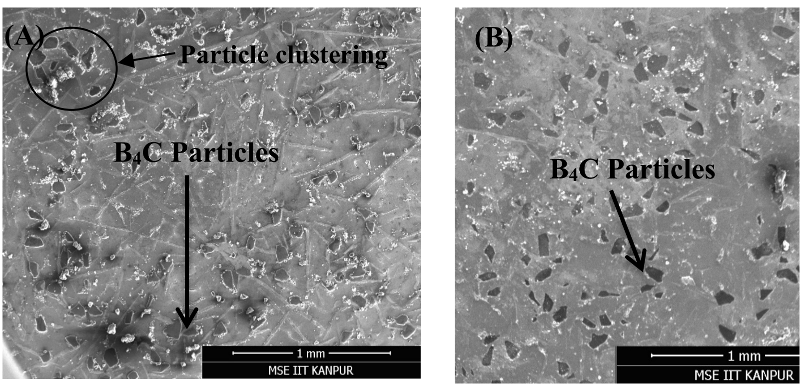


Figure 5: (A) Microstructure of ACC, and (B) Microstructure of HTC

It also seems that the application of K_2TiF_6 flux enhances the interface between B_4C and Al matrixes. These interfaces formed due to the exothermic reaction between Ti and B_4C. Ti has good affinity towards Boron carbide in the presence of molten Al and react exothermally with Boron [7,14]. Due to exothermic reaction, there evolves the enormous amount of heat at the vicinity of B_4C particle, results in local melting of B_4C and form TiB_x, also Ti react with Al and forms Al_3Ti intermetallic compound. Therefore, a quaternary layer of B_4C-TiB_2-Al_3Ti-α-Al is formed as evidence of proper incorporation of B_4C particles in the Al matrix. For the present study FESEM-EDAX line scan as shown in Figure 6 done at the interface, the line starts from α-Al to B_4C particle crossing recation layer. The line scan across the reaction layer indicates that the aluminium interact with Ti and forms Al_3Ti intermettalic compounds and the Ti line interact with Boron and form a thin layer of TiB_x intermettalic compounds after Al_3Ti. Therefore, the reaction layer comprises of a quaternary layerof B_4C-TiB_2-Al_3Ti-Al at Al-B_4C interface [11,12]. Uniform distribution and proper incorporation of B_4C particles in the matrix, enhances the load-bearing capacity of the composite after heat treatment [22]. The similar discussion made by Kennedy et al. [18]. Therefore, the mechanical strength and wear properties of the heat-treated composite improves.

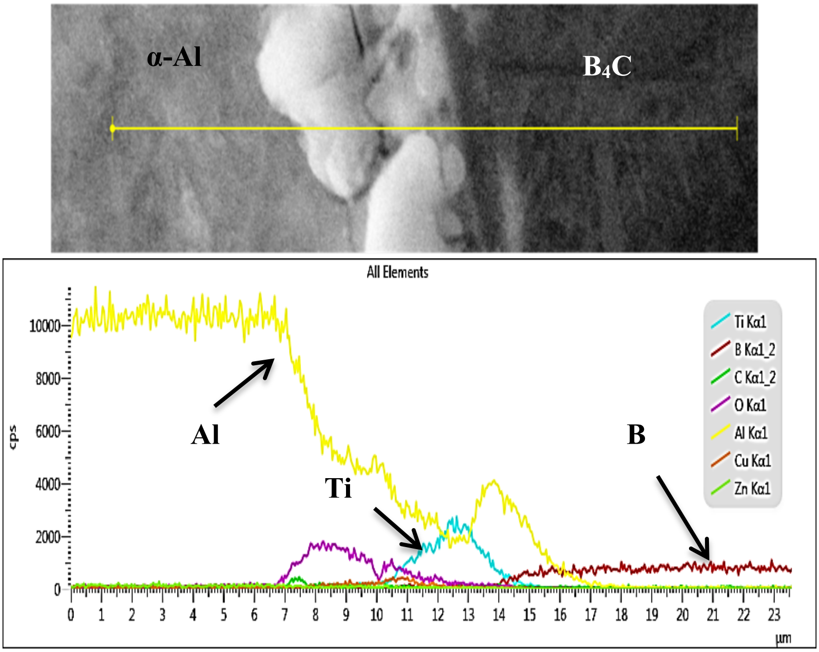


Figure 6: Reaction mechanism at particle matrix interface

Mechanical properties

Table 3 and Figure 7 shows the comparative study of mechanical properties of ACC and HTC. The result shows that there is a significant improvement of about 16.4% in the ultimate tensile strength in HTC over ACC. This improvement could be attributed to the precipitation hardening of the alloy matrix. Notably, the presences of titanium enhance the heterogeneous nucleation of α-Al grains, and thereby strengthen the grain boundaries by hindering the dislocation movements [14].

It is well known that the B_4C particles possess high inherent hardness. These particles, when dispersed into the soft α-Al matrix, enhance the hardness of the composite.

Table 3. Mechanical properties of the composite

AA7050/ B_4C Composite	Hardness (BHN)	Std. Dev.	Tensile Strength (N/mm^2)	Std. Dev.	Compressive Strength (N/mm^2)	Std. Dev.	Impact Energy (KJ/m^2)	Std. Dev.
ACC	132.1	12.53	230.66	15.04	374.21	12.47	24.17	1.69
HTC	157.7	8.04	275.33	13.50	439.52	14.35	27.16	1.42

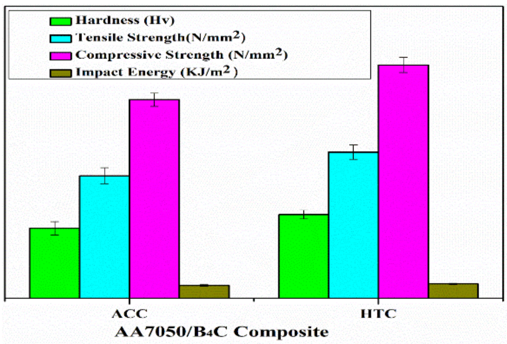


Figure 7: Mechanical properties of ACC and HTC

Furthermore, as discussed above the formation of interface layers contributes to the increase in the interface strength between α-Al matrix and B_4C. Also, the heat treatment further enhances the hardness of composite by 15.9% due to precipitation hardening mechanism. Collectively, the strengthening mentioned above mechanisms contributes to the improvement in the hardness of the composite through the Orowan mechanism [3,4]. Similarly, improvement in compressive strength and impact energy of the composite recorded as 14.8% and 10.9% respectively after heat treatment.

Wear properties of the composite

The pin on disc dry sliding wear tests were conducted on the ACC and HTC separately with the process parameters such as applied load and sliding speed. Each experiment was repeated for three times and the average of three has been taken for analysis. The test result reveals the improvement in the wear resistance in HTC than ACC. Wear is surface damage due to the combined effect of shear and compressive stress. Since due to the precipitation hardening effect, hardness and compressive strength of the composite increased after heat treatment. Therefore, the wear rate of HTC is lower than the ACC [3,20]. The load versus wear rate plots shown in the Figure 8 and Figure 9 indicate that the wear rate of ACC is higher than that of HTC for all loads and speeds investigated. Additionally, it is also anticipated that the strain hardening and dynamic ageing phenomena prevail during the dry sliding which contributes to the increase in the wear resistance of the composite.

Further, observations on the effects of load and sliding speed of the composite on the coefficient of friction (COF) have been presented in Figure 8 and Figure 9. From the plots, it can be found that the COF increases with the increase in the load until 20 N and decreases significantly on the further increase of load. This could be attributed to the change the modes of wear from two-body abrasion to three-body abrasion as the wear debris interfere between the sliding surfaces [21]. Such a trend is seen in both ACC and HTC conditions for sliding speeds of 0.5 m/s and 1 m/s. Moreover, the COF of HTC is less than ACC. This may be attributed to the uniform dispersion and excellent interface of B_4C particles with the matrix. B_4C particles are expected to provide adequate dislocation pile-up which results in the strain hardening of the composites, which significantly contributes to improving the wear resistance of the composite [22].

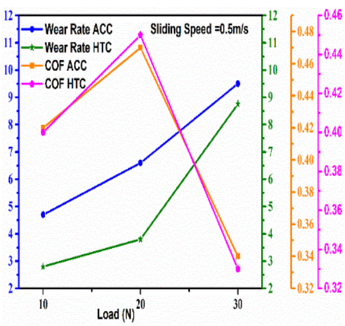


Figure 8: Wear rate and COF for ACC and HTC at sliding speed of 0.5 m/s

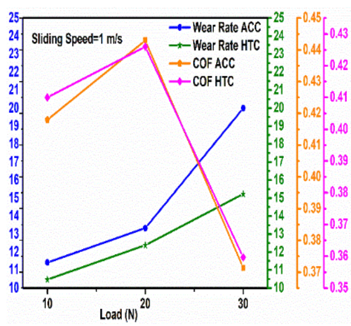


Figure 9: Wear rate and COF for ACC and HTC at sliding speed of 1 m/s

Conclusions

From the experimental evidence,the following conclusions have been drawn:

1. AA7050-B_4C composite developed successfully by flux assisted stir cast method, and the microstructure reveals the uniform distribution of B_4C particles into the matrix.
2. The EDAX analysis confirms the formation of a quaternary layer of B_4C-TiB_2-Al_3Ti-Al across the B_4C particles. These layers enhance the interface strength between the matrix and reinforcement.

3. Heat treatment enhanced the tensile strength, hardness, compressive strength and impact strength by 16.4%, 15.9%, 14.8% and 10.9% respectively through precipitation hardening mechanism.
4. After heat treatment wear properties of the composite improved, wear rate and coefficient of friction of HTC is less than ACC for all set of loads and sliding speed.

References

[1] J.C.Williams and E.A. Starke Jr., "Progress in structural materials for aerospace systems." Acta Materialia 51(19), 5775-5799 (2003).

[2] G.Shaand A Cerezo. "Early-stage precipitation in Al–Zn–Mg–Cu alloy (7050)," Acta Materialia 52(15), 4503-4516 (2004).

[3] Lashgari, H. R., Zangeneh, S., Shahmir, H., Saghafi, M., and Emamy, M., "Heat treatment effect on the microstructure, tensile properties and dry sliding wear behavior of A356–10% B_4C cast composites,' Materials and Design 31(9), 4414-4422 (2010).

[4] B.V. Ramnath, C. Elanchezhian, M. Jaivignesh, S. Rajesh, C. Parswajinan, A.S. Ghias, "Evaluation of mechanical properties of aluminiumalloy–alumina–boron carbide metal matrix composites," Materials and Design 58, 332-338 (2014).

[5] V.P. Mahesh, P.S. Nair, T.P. Rajan, B.C. Pai, R.C. Hubli, "Processing of surface-treated boron carbide-reinforced aluminium matrix composites by liquid–metal stir-casting technique," Journal of composite materials 45(23), 2371-2378 (2011).

[6] A. K.Suri, C.Subramanian, J. K.Sonber, and T. C. Murthy, "Synthesis and consolidation of boron carbide: a review," International Materials Reviews 55 (1), 4-40 (2010).

[7] K.Kalaiselvan, N. Murugan, and S Parameswaran, "Production and characterization of AA6061–B_4C stir cast composite," Materials & Design 32(7), 4004-4009 (2011).

[8] Q.Hu, Z.Haidong, and F. Li., "Effects of manufacturing processes on microstructure and properties of Al/A356–B_4C composites," Materials and Manufacturing Processes 31(10), 1292-1300 (2016).

[9] F.Toptan, A.Kilicarslan, A.Karaaslan, M.Cigdem and I.Kerti, "Processing and microstructural characterisation of AA 1070 and AA 6063 matrix B_4C_p reinforced composites," Materials and Design, 31, 87-91 (2010).

[10] A.Mazahery and M.O. Shabani, "Influence of the hard coated B_4C particulates on wear resistance of Al–Cu alloys," Composites Part B: Engineering, 43(3), 1302-1308 (2012).

[11] Z.Zhang, K.Fortin, A.Charette and X.G.Chen, "Effect of titanium on microstructure and fluidity of Al–B_4C composites," Journal of materials science 46(9), 3176-3185 (2011).

[12] Z.Zhang, X.G. Chen, and A.Charette, "Particle distribution and interfacial reactions of Al–7%Si–10%B_4C die casting composite," Journal of materials science 42(17), 7354-7362 (2007).

[13] Y.Sahin and V. Kilicli, "Abrasive wear behaviour of SiC_p/Al alloy composite in comparison with ausferritic ductile iron," Wear 271(11-12), 2766-2774 (2011).

[14] S. M. Kumar, V.Chakraborty, S,Subramanya and B. S. Murty, "Tensile and wear behaviour of in situ Al–7Si/TiB_2 particulate composites," Wear 265(1-2), 134-142 (2008).

[15] B.S.Yigezu, M.M. Mahapatra and P.K.Jha, "On modeling the abrasive wear characteristics of in situ Al–12% Si/TiC composites," Materials and Design 50, 277-284 (2013).

[16] R.Ranjith, P.K.Giridharan, J.Devaraj and V.Bharath, "Influence of titanium-coated (B4Cp+ SiCp) particles on sulphide stress corrosion and wear behaviour of AA7050 hybrid composites (for MLG link)", Journal of the Australian Ceramic Society 53(2), 1017-1025 (2017).

[17] D. Das, P Mishra, A Chaubey and S Singh, "Fabrication process optimization for improved mechanical properties of Al 7075/SiCp metal matrix composites", Management Science Letters 6(4), 297-308 (2016).

[18] A. R.Kennedy and S. M. Wyatt, "The effect of processing on the mechanical properties and interfacial strength of aluminium/TiC MMCs," Composites Science and Technology 60(2), 307-314 (2000).

[19] Liu, Z. W., Q. Han, and J. G. Li, "Formation of small blocky Al 3 Ti particles via direct reaction between solid Ti powders and liquid Al," Metallurgical and Materials Transactions A 43(12), 4460-4463 (2012)

[20] R.N.Rao, S.Das, D.P. Mondal, and G. Dixit, "Effect of heat treatment on the sliding wear behaviour of aluminium alloy (Al–Zn–Mg) hard particle composite," Tribology international 43(1-2), 330-339 (2010).

[21] M.Uthayakumar, S.Aravindan, and K.Rajkumar,"Wear performance of Al–SiC–B_4Chybrid composites under dry sliding conditions," Materials and Design 47, 456-464 (2013).

[22] J.M. Mistry, and P.P. Gohil, "Experimental investigations on wear and friction behaviour of Si_3N_4p reinforced heat-treated aluminium matrix composites produced using electromagnetic stir casting process," Composites Part B: Engineering 161, 190-204 (2019).