

Fabrication and Characterizations of Mechanical Properties of Al-4.5%Cu/10TiC Composite by *In-Situ* Method

Anand Kumar^{*}, Manas Mohan Mahapatra, Pradeep Kumar Jha

Department of Mechanical and Industrial Engineering, Indian Institute of Technology, Roorkee, India
Email: ^{*}a7044dme@iitr.ernet.in, anand7044@gmail.com

Received July 5, 2012; revised August 7, 2012; accepted August 15, 2012

ABSTRACT

Addition of reinforcement such as TiC, SiC, Al₂O₃, TiO₂, TiN, etc. to Aluminium matrix for enhancing the mechanical properties has been a well established fact. *In-situ* method of reinforcement of the Aluminium matrix with ceramic phase like Titanium Carbide (TiC) is well preferred over the *Ex-situ* method. In the present investigation, Al-Cu alloy (series of 2014 Aluminium alloy) was used as matrix and reinforced with TiC using *In-situ* process. The Metal Matrix Composite (MMC) material, Al-4.5%Cu/10%TiC developed exhibited higher yield strength, ultimate strength and hardness as compared to Al-4.5%Cu alloy. Percentage increase in yield and ultimate tensile strengths were reported to be about 15% and 24% respectively whereas Vickers hardness increased by about 35%. The higher values in hardness indicated that the TiC particles contributed to the increase of hardness of matrix. Fractured surface of the tensile specimen of the composite material indicated presence of dimpled surface, indicating thereby a ductile type of fracture. During the fabrication of composite, reaction products such as Al₃Ti, Al₂Cu and Al₃C₄ were identified with various morphologies and sizes in metal matrix.

Keywords: *In-situ*; Metal Matrix Composites; TiC reinforcement; Mechanical Characterization

1. Introduction

Over the past few decades, researchers have emphasized on production of light and strong materials. Aluminium based metal matrix composites are the advanced materials with superior properties which are actively being sought for engineering applications. In recent years, Al based composite materials have gained significance in aerospace, automotive and structural applications due to their enhanced mechanical properties and good stability at high temperature. The necessary characteristics of advanced materials include high specific modulus, stiffness, strength, hardness, ductility, corrosion resistance, low heat expansion coefficient and so on [1].

The aim of designing the metal matrix composite materials is to combine the desirable properties of metal (high strength and ductility) and ceramics (high modulus and stiffness). In order to achieve the optimum mechanical properties, it is essential to achieve the uniform distribution of reinforcement within the matrix. Aluminium, Silicon, Copper, Titanium, Magnesium, and Nickel metals are widely used for preparation of metal matrix in composites materials where as monofilaments, whiskers,

fibers or particulate types are widely used as reinforcement phases in Metal Matrix Composites (MMCs). There are many processing techniques which have been developed for fabricating Metal Matrix Composites (MMCs). Such techniques are Powder Metallurgy (PM), liquid metal infiltration, compocasting, squeeze casting method, stir casting and spray decomposition method [2,3].

Among the fabrication techniques of MMC, stir casting (for particulate or discontinuous reinforced MMCs) is generally preferred. Its advantages lie in its simplicity, flexibility and applicability to large quantity of production. It is also attractive because of minimized final cost of the product. In the stir casting method, there are several factors that need considerable attention, including the difficulty of achieving a uniform distribution of the reinforcement. During these conventional processes, the major difficulties are improper wetting due to oxidation which exhibit interface binding between matrix and ceramic phases. Improved wetting must be achieved to obtain a good bond between the matrix and reinforcement. Other limitations are the distributions of reinforcement, interfacial reaction between the metal matrix and reinforcement, and control of volume fraction shape and size of reinforcement, often encountered during the fabrica-

^{*}Corresponding author.

tion of Metal Matrix Composites (MMCs). Therefore, all such factors can affect the expected mechanical properties of MMCs [4-8].

A new approach has been developed as *In-situ* processing, including SHS (Self-Propagating High Temperature Synthesis), reactive gas infiltration, liquid-solid or solid-gas-liquid reactions, XDTM, DIMOX, VLS, PRIMEXTM [9-11]. *In-situ* process involve a chemical reaction resulting in the formation of a very fine, thermodynamically stable, clean surfaces of ceramic reinforcement within a metal matrix. The other advantages of *In-situ* process are, isotropic in nature, strong chemical bonding between the matrix and reinforcement phase, the distribution of reinforcement particles within the metal matrix is homogeneous [12-14]. Therefore, the produced composite are of better mechanical properties as compared to monolithic materials.

Various types and sizes of reinforcements are used in matrix of Aluminium like SiC, TiC, Al₂O₃, B₄C, TiB₂, TiN, etc. Among these, TiC is a relatively new reinforcement in metal matrix composites and has good properties such as wettability, thermal stability and distribution in Aluminium metal matrix [15-17].

Present work deals with *In-situ* fabrication of Al-4.5% Cu metal matrix composite with 10 wt% TiC as reinforcement by *In-situ* process. The interfacial reaction between the elements in composite is very important, because the load is transferred at the interface and can affect the mechanical properties of composite. The produced MMC was evaluated in terms of microstructural characterization and mechanical properties testing such as hardness and tensile strength.

2. Experimental Procedure

The raw materials used for the present study for the preparation of the metal matrix was commercial available Aluminium ingot (97.95% pure) and Copper (99.98% pure). The chemical composition of commercial Aluminium ingot is shown in **Table 1**.

The setup used for the *In-situ* preparation of MMC through stir-casting route is shown in **Figure 1**. The high temperature electric furnace was used for preparation of Al-4.5%Cu matrix alloy and master alloy of Al-10%Ti.

In order to produce the composite, both alloys were melted in graphite crucible and stirred gently at 475 rpm with mechanical stirrer at 1150°C temperature with holding time of 90 minutes. Impurities from the Aluminium melt or dross was then removed from the surface of molten metal. The melts were degassed by using hexachloroethane (C₂Cl₆) at 750°C for removing dissolved hydrogen gas. The required amount of activated charcoal powder was weighed and then encapsulated in aluminium foil wrappings and added into the molten alloy. Mechanical stirring was carried out for 2 - 3 minutes. KF

and NaF were added as flux on the surface of liquid metal. The flux reacted with metal alloy and formed layer of slag that dissolved the oxide film from the liquid surface. Activated charcoal is assumed to react with Titanium and formed Titanium carbide (TiC) at 1150°C temperature with holding time of 30 minutes. TiC particles were reinforced into matrix materials by *In-situ* process. The resultant slurry was stirred for 1 - 2 min and finally poured into a metallic mould to get the solidified casting.

The Al-4.5%Cu/10TiC MMC samples for microscopic examinations were prepared by adopting standard metallographic procedure. Samples were polished using different size of SiC grit papers of 120, 220, 400, 600, 800, 1000, and 1200, followed by velvet cloth with aluminium paste. The Keller's reagent was used for etching with mixture of 0.5 ml HF, 0.75 ml HCl, 2.5 ml HNO₃ and balance amount of distilled water. The microstructures of the etched sample were examined using Scanning Electron Microscope (SEM) as shown in **Figure 2**. Compositional test of the sample were carried out using Energy-Dispersive X-ray spectroscopy (EDX) and phase analysis was done using X-Ray Diffraction technique (XRD).

3. Result and Discussions

3.1. Microstructural Analysis

Figure 2 Shows a typical SEM micrograph of cast *In-situ*

Table 1. Chemical compositions of commercial aluminium ingot.

Materials	Cu	Fe	Si	Ti	Mg	Mn	Cr	Ni	Al
Chemical Compositions	0.7	0.58	0.01	0.04	0.28	0.35	0.04	0.05	Balance

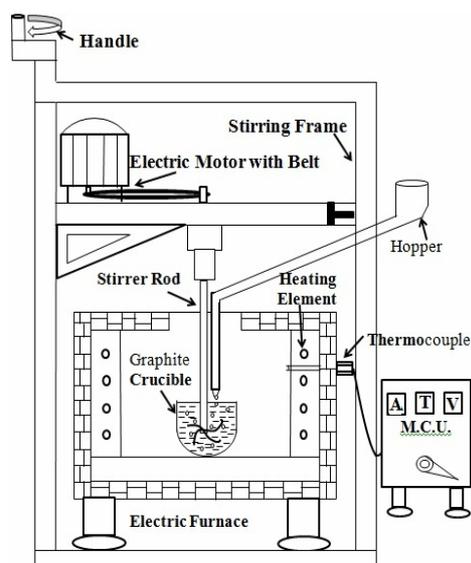


Figure 1. Schematic of experimental setup.

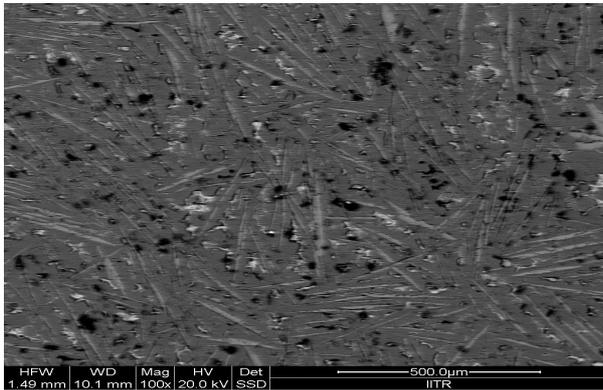


Figure 2. SEM micrograph of as cast composite.

Al-4.5%Cu-10%TiC composite. The presence of elements in metal matrix composite can be observed as peaks of Al, Cu, Ti and carbon by EDX spectrum shown in the **Figure 3**. The particles of TiC were formed by *In-situ* reaction method and were found to be located at Aluminum grain boundaries or partly inside the Aluminum grains. The microanalysis result exhibit the distribution of TiC as reasonably uniform, though several small clusters of particles are present along with some voids.

Figure 4 presents the XRD spectrums of the as cast Al-4.5%Cu/10%TiC composite. The dominant phases observed are Al_3Ti , Al_4C_3 , CuAl_2 and TiC in the cast composite. Among these phases, the binary compounds Al_3Ti and Al_4C_3 are mostly observed in first phase chemical reactions at lower temperature in *In-situ* process. The reason for synthesis of Al_3Ti may be due to excessive titanium reaction with aluminium. At higher temperature, these compound react to each other and form TiC particles in the composite [18]. The binary compound Al_4C_3 was formed due to excess of carbon present in molten aluminium. The compound Al_4C_3 is brittle in nature and unstable at higher temperatures. Decrease in retained carbon leads to increase in amount of TiC and reduction in amount of impurities along the grain boundaries. Consequently, both tensile and ductility improved in produced MMCs [19]. The phases identified have clearly been labeled in **Figure 5(a)**. The optimum molar ratio of Ti and C is 1:1.3 in composite. Under such condition these binary compound can be reduced to the produced composite. TiC particles form only when reaction temperature was more than 1000°C . TiC particles were spherical, needle or block in shape with smooth and clear surface [20-23]. **Figure 5(b)** shows that the size and shape of TiC particles formed were from $0.1\ \mu\text{m}$ to $0.8\ \mu\text{m}$ with spherical shape.

3.2. Mechanical Testing

In order to investigate the hardness of as-cast matrix materials and composites, the samples were tested by using

a standard Vickers hardness testing machine with a pyramid indenter of load 5 kg. The load was applied for 15 sec and average of five readings was taken for each specimen at different locations to circumvent the possible effects of particle segregation.

The average values of hardness of matrix material and composite were 61 and 94 respectively as shown in the **Table 2**. The TiC particles embedded with matrix materials affected 35.79% increase in the hardness.

The tensile specimens were prepared according to ASTM E-8 as shown in the **Figure 6**. Yield strength and ultimate strength of matrix and composite material was tested with universal testing machine. The strain rate was 0.01s^{-1} during testing of the specimens. For each testing condition, 3 specimens were subjected to tensile test and the average of three values was noted. The yield strength increases from 76 to 87 Mpa and ultimate strength increases from 118 to 147 Mpa after 10% TiC reinforcement in Al-4.5%Cu as shown in **Table 2**. The investigation of fracture surface morphology of matrix and composite material specimens was observed by Scanning Electron Microscope (SEM).

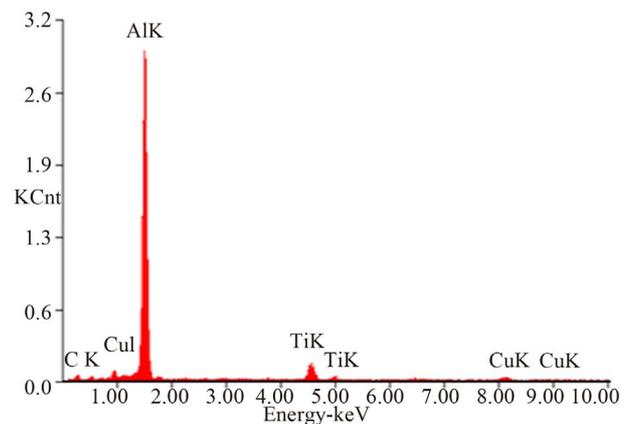


Figure 3. EDX results of Al-4.5%Cu/10%TiC.

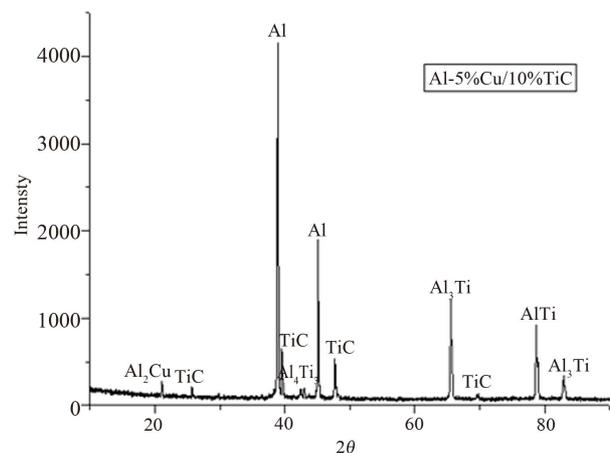


Figure 4. XRD pattern of composite material.

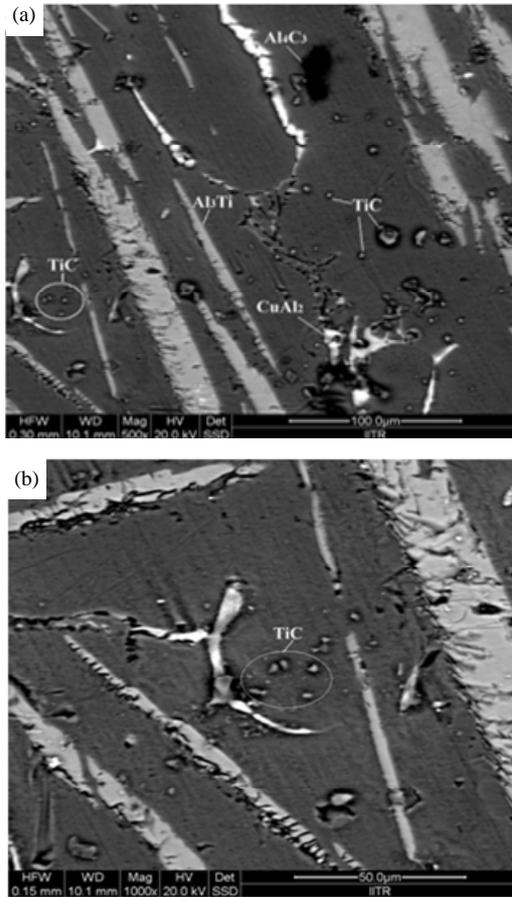


Figure 5. (a) SEM micrograph of the Al-4.5%Cu/10TiC; (b) SEM micrograph (at higher magnification) of Al-4.5%/10%TiC.

Table 2. Average Hardness and Tensile value of matrix and composite Materials.

Sl. No.	Materials	Vickers Hardness (Hv5)	Yield Stress (σ_y (Mpa))	Ultimate Stress (σ_u (Mpa))
1.	Al-4.5%Cu	61	76	118
2.	Al-4.5%Cu/10%TiC	94	87	147

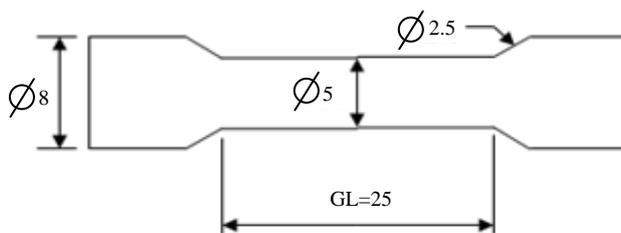


Figure 6. Schematic of tensile specimen (all dimension in mm).

3.3. Fractography

The fracture surface of dog-bone tensile specimens of matrix material and TiC reinforced material were ana-

lyzed under Scanning Electron Microscope (SEM) to determine the mode of failure of the material. Typically, all the specimens exhibited cup-and-cone failure mode which indicates a ductile fracture mode. The presence of dimples on fracture surface clearly indicates that necking had occurred prior to matrix fracture. The fractograph of Al-4.5% Cu matrix material shows a large deep dimple in fracture surface which exhibited ductile failure as shown in the **Figure 7(a)**. Al-4.5%Cu/10%TiC composite material observed small dimples on the fracture surface which also revealed the presence of fracture mode to be ductile one, as shown in **Figure 7(b)**. Some debonded particles are also seen on fracture surface which proves that relatively strong bonding is present in between the matrix material and reinforced particles. **Figure 7(c)** shows a higher magnified fractograph of Al-4.5%Cu/10%TiC composite material which observed crack propagation on fracture surfaces, though this propagation could no longer propagate and were arrested by reinforced particles. The transfer of loads form metal matrix to particles clearly understands to successfully synthesize hard TiC particles through *In-situ* processing.

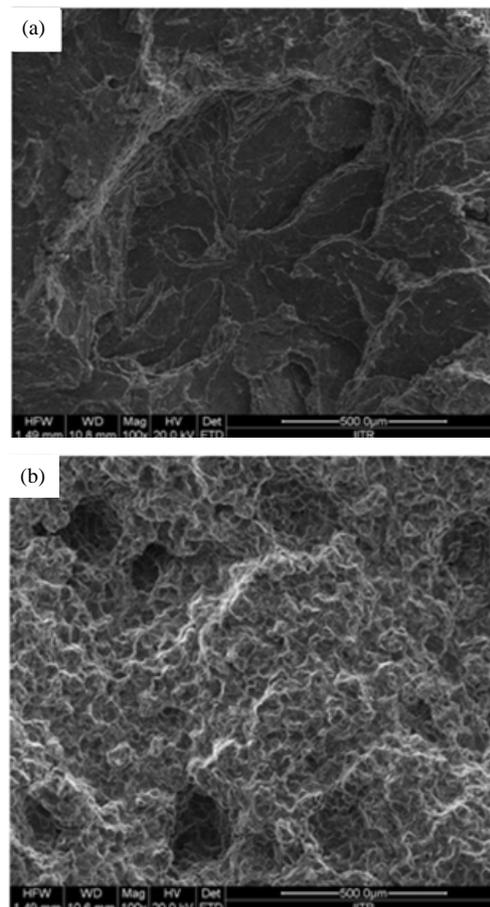


Figure 7(a) SEM micrograph of fractured specimen of Al-4.5%Cu; (b) SEM micrograph of fractured specimen of Al-4.5%Cu/10%TiC.

4. Conclusion

In-situ Al-4.5%Cu/10%TiC metal matrix composite was successfully fabricated using stir casting route. Ti was added in elemental form and activated charcoal powder was added in the melt which resulted into the formation of Titanium Carbide as reinforcement. As the reinforcement phase was formed inside the melt (*In-situ* process), the distribution of Titanium Carbide was seen to be more uniform. The yield, ultimate tensile strength and hardness were seen to increase by 12.64%, 19.72%, and 35.79% respectively in the composite material with TiC as reinforcement. The fracture surface of tensile sample shows small dimples indicates ductile fractures. Tensile strength, fracture surface and hardness of Al-4.5%Cu/10%TiC MMC fabricated in the present investigation indicated improvements of material properties.

5. Acknowledgements

The authors would like to thank the Department of Mechanical and Industrial Engineering, Indian Institute of Technology, Roorkee for providing the necessary facilities and fund for experiment and tests

REFERENCES

- [1] P. K. Rohtagi, "Metal Matrix Composites," *Journal of Defence Science*, Vol. 43, No. 4, 1993, pp. 323-349.
- [2] D. B. Miracle, "Metal Matrix Composites from Science to Technological Significance," *Composites Science and Technology*, Vol. 65, No. 15-16, 2005, pp. 2526-2540.
[doi:10.1016/j.compscitech.2005.05.027](https://doi.org/10.1016/j.compscitech.2005.05.027)
- [3] M. K. Surappa and P. K. Rohatgi, "Preparation and Properties of Cast Aluminium-Ceramic Particle Composites," *Journal of Materials Science*, Vol. 16, No. 4, 1981, pp. 883-993.
- [4] A. Sato and R. Mehrabian, "Aluminium Matrix Composites: Fabrication and Properties," *Metallurgical Transactions B*, Vol. 7, No. 3, 1976, pp. 443-451.
- [5] J. Hashim "The Production of Cast Metal Matrix Composite by a Modified Stir Casting Method," *Journal Teknologi*, Vol. 35, No. 35A, 2001, pp. 9-20.
- [6] S. M. L. Nai and M. Gupta, "Influence of Stirring Speed in the Synthesis of Al/SiC Based Functionally Gradient Materials," *Composite Structures*, Vol. 57, No. 1-4, 2002, pp. 227-233.
- [7] J. Hashim, L. Looney and M. S. J. Hashmi, "Metal Matrix Composites: Production by the Stir Casting Method," *Journal of Materials Processing Technology*, Vol. 92-93, 1999, pp 1-7.
- [8] S. P. Balasivanandha, L. Karunamoorthy, S. Kathiresan and B. Mohan. "Influence of Stirring Speed and Stirring Time on Distribution of Particles in Cast Metal Matrix Composite," *Journal of Materials Processing Technology*, Vol. 171, No. 2, 2006, pp. 268-273.
- [9] I. Gotman and M. J. Koczak, "Fabrication of Al Matrix *In-situ* Composites via Self Propagating Synthesis," *Materials Science and Engineering*, Vol. 187, No. A187, 1994, pp. 189-199.
- [10] S. Sheivani and M. F. Nazafabadi, "*In-situ* Fabrication of Al-TiC Metal Matrix Composites by Reactive Slag Process," *Material and Design*, Vol. 28, No. 8, 2007, pp. 2373-2378.
- [11] B. P. Khrishnan, M. K. Surappa and P. K. Rohatgi, "A Direct Method to Prepare Cast Aluminium Alloy-Graphite Particle Composite," *Journal of Material Science*, Vol. 16, No. 5, 1981, pp. 1209-1216.
- [12] L. Peijie, E. G. Kandalova and V. I. Nikitin, "*In-situ* Synthesis of Al-TiC in Aluminium Melt," *Materials Letters*, Vol. 59, No. 19-20, 2005, pp. 2545-2548.
- [13] T. V. Christy, N. Murugan and S. Kumar, "A Comparative Study on the Microstructures and Mechanical Properties of Al 6061 Alloy and the MMC Al6061/TiB₂/12," *Journal of Minerals and Materials Characterization and Engineering*, Vol. 9, No. 1, 2010, pp. 57-65.
- [14] C. Chunxiang, S. Yutian and M. Fanbin, "Review on Fabrication Method of *In-situ* Metal Matrix Composites," *Journal of Material Science and Technology*, Vol. 16, No. 6, 2000, pp. 619-626.
- [15] M. Singla, D. D. Dwivedi, L. Singh and V. Chawla, "Development of Aluminium Based Silicon Carbide Particulate Metal Matrix Composite," *Journal of Minerals and Materials Characterization and Engineering*, Vol. 8, No. 6, 2009, pp. 455-467.
- [16] S. B. Boppana and K. Chennakeshavalu, "Preparation of Al-5Ti Mater Alloys for the *In-situ* Processing of Al-TiC Metal Matrix Composites". *Journal of Minerals and Materials Characterization and Engineering*, Vol. 8, No. 7, 2009, pp. 563-568.
- [17] A. Albiter, C. A. Leon, R. A. L. Drew and E. Bedolla, "Microstructure and Heat-treatment Response of Al2024/TiC Composites," *Materials Science and Engineering: A*, Vol. 289, No. 1-2, 2000, pp. 109-115.
- [18] P. Sahoo and M. J. Koczak, "Microstructure-Property Relationships of *In-situ* Reacted TiC/Al-Cu Metal Matrix Composites," *Material Science Engineering*, Vol. 131, No. 1, 1991, pp. 69-76.
- [19] L. Lu, M. O. Lai and J. L. Yeo, "*In-situ* Synthesis of TiC Composite for Structural Application," *Composite Structure*, Vol. 47, No. 1, 1999, pp. 613-618.
- [20] K. B. Lee, H. S. Sim and H. Kwon, "Reaction Products of Al/TiC Composites Fabricated by the Pressureless Infiltration Technique," *Metallurgical and Materials Transactions A*, Vol. 36, No. 9, 2005, pp. 2517-2527.
[doi:10.1007/s11661-005-0125-0](https://doi.org/10.1007/s11661-005-0125-0)
- [21] R. F. Shyu and C. T. Ho, "*In-situ* Reacted Titanium Carbide Reinforced Aluminium Alloys Composite," *Journal of Material Processing Technology*, Vol. 171, No. 3, 2006, pp. 211-216.
- [22] B. Ranjit, "Synthesis of Al-TiC *In-situ* Composite: Effect of Processing Temperature and Ti:C Ratio," *Transaction of the Indian Institute of Metals*, Vol. 62, No. 4-5, 2009, pp. 391-395.

- [23] B. Yang, F. Wang and J. S. Zhang, "Microstructure and Characterization of *in Situ* Al/TiC and Al/TiC-20Si-5Fe-3Cu-1Mg Composite Prepared by Spray Deposition," *Acta Materialia*, Vol. 51, No. 17, 2003, pp. 4977-4989.