# Chapter 18 Fabrication and Corrosion Behaviour of Aluminium Hybrid and Non-hybrid MMCs Reinforced with B<sub>4</sub>C and Gr Additions by Powder Metallurgy **Technique**



#### K. Sunil Ratna Kumar, Ch. Ratnam, Ch. Ramakrishna and Ch. Lakshmi Poornima

Abstract In this tentative study, aluminium metal matrix composites (AMMCs) and aluminium metal matrix hybrid composites (AMMHCs) were fabricated by using powder metallurgy method. In this work, boron carbide  $(B_4C)$  and graphite (Gr) are the two types of reinforcements used in the fabrication of the composites, and these are added to the base aluminium alloy Al2024 used as matrix. The used reinforcements, in which one is a ceramic harder material boron carbide  $(B_4C)$ added in AMMCs and the other element is a softer material Graphite (Gr) is added in AMMHCs along with same  $B_4C$ . In the initial stage, various weight-based aluminium metal matrix composites (AMMCs) are fabricated by choosing Al 2024 as the matrix and are reinforced with the hard ceramic material which is boron carbide (3, 6, 9, 12 and 15%). In the lateral stage, aluminium metal matrix hybrid composites (AMMHCs) are also being fabricated by using same aluminium used as matrix and are reinforced with fixed percentage of soft solid lubricant Gr (3%) along with varying percentages of  $B_4C$  (3, 6, 9, 12 and 15%). All these composites are fabricated by using powder metallurgy technique; FESEM and microstructural analysis shows the presence and uniform distribution of reinforced particles in aluminium alloy. Corrosion test was carried out as per ASTM 59 standards for a definite time interval on all the fabricated specimens by using potential dynamic polarization technique in aerated 3.5% NaCl solutions with PH adjusted to 10 by

Department of Mechanical Engineering, Sir C R R College of Engineering, Vatlur, Eluru 534007, Andhra Pradesh, India e-mail: [ksratnakumar@gmail.com](mailto:ksratnakumar@gmail.com)

Ch.Ramakrishna Ch.Lakshmi Poornima

Andhra University College of Engineering, Andhra University, Visakhapatnam 530003, Andhra Pradesh, India

K. S. Ratna Kumar  $(\boxtimes)$   $\cdot$  Ch. Ratnam

Ch.Ratnam e-mail: [chratnam@gmail.com](mailto:chratnam@gmail.com)

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adding potassium hydroxide. After the test, corrosion behaviour of all the samples is analysed by Tafel extrapolation technique. The surface morphology of the specimen before and after corrosion was studied using the FESEM and microstructure images.

**Keywords** Hybrid composite  $\cdot$  Self-lubricating  $\cdot$  Powder metallurgy  $\cdot$  Sintering  $\cdot$  Consolidation  $\cdot$  Green pellets  $\cdot$  Ball milling  $\cdot$  Corrosion  $\cdot$  Corrosion rate  $\cdot$  Tafel  $\cdot$  FESEM  $\cdot$  EDS

## 18.1 Introduction

Aluminium metal matrix composites are extensively used in various applications due to its improved properties with low density. These materials can be used in aircraft, automotive, construction, packing, electronics and military industries. The type of reinforcements like metals, ceramic and oxide particles can be used in aluminium alloy, depending upon their applications. These materials are the encouraging materials to give better properties with low density and also cheaper than the other materials like Mg and Ti. In this present work, Al2024 is selected on the basis of usage of material in aerospace and automobile applications, which will lead to high strength-to-weight ratio and also having good mechanical properties.  $B_4C$  is used as one of the reinforcements, which is having high hardness value as compared to the other materials. The graphite is used as another best reinforcing material, having less density, and can be used as solid lubricant. The major usage of graphite can be seen in the composites to reduce the wear in between the contacting elements. Different methods like stir casting, liquid infiltration, squeeze casting, diffusion bonding and powder metallurgy technique can be used for the fabrication of composites; among all these methods, powder metallurgy (PM) technique [\[1](#page-12-0)–[6](#page-12-0), [10,](#page-13-0) [13](#page-13-0), [14\]](#page-13-0) is selected for the fabrication of the composites. The PM technique can be used for mass production and uniform distribution of particles with less wastage of materials. The hardness of the composite increases with the increase in  $B_4C$ content, and then, it is very difficult for machining process. To overcome this effect, small amount of Gr is used for the fabrication of composites. The monocomposites with single reinforcement restrict some of their properties; hence, adding more number of reinforcements called as hybrid composites [\[5](#page-12-0)–[18](#page-13-0)] improves their properties. The corrosion behaviour of various metal matrix composites [[14](#page-13-0)–[21\]](#page-13-0) is explained by many researchers. Hanji et al. [\[15](#page-13-0)] studied the corrosion behaviour of heat-treated hybrid metal matrix composites reinforced with  $B_4C$  and graphite particles, and the results revealed that the corrosion resistance of hybrid composite was the least since there is a formation of aluminium carbides which leads to galvanic and inter-granular corrosion. Muthazhagan et al. [\[16](#page-13-0)] fabricated successfully hybrid metal matrix composites by powder metallurgy route and concluded that the corrosion rate increased with the increase in  $B_4C$  and Gr in aluminium due to the localized attack on the grain boundaries. Ravindranath et al. [[17](#page-13-0)] explained the degradation of hybrid metal matrix composites reinforced with boron carbide and graphite due to pit corrosion. The corrosion resistance of  $B_4C$  reinforced composite is higher due to the presence of carbide, the least corrosion resistance for hybrid composites and the corrosion resistance is maximum for the base aluminium alloy. Bihari et al. [\[18](#page-13-0)] examined the corrosion behaviour of  $A17075/A1<sub>2</sub>O<sub>3</sub>/graphite$ hybrid composite in 3.5% sodium chloride solution. The corrosion behaviour of Al7075 and Al7075/Al<sub>2</sub>O<sub>3</sub>/Gr MMCs was tested by weight loss, potentiodynamic polarization and electrochemical impedance method. An increase in the percentage of  $A_1O_3$  particles reduces the potential, corrosion current density and corrosion rate. Singh et al. [\[19\]](#page-13-0) investigated the influence of SiC particle addition on the corrosion behaviour of Al2014-Cu alloy in 3.5% NaCl solution; the addition of SiC content to the base alloy decreases their corrosion resistance. The increase of SiC is likely to increase the cathodic sites in composite, and it leads to start the galvanic effect which results in the decrease in corrosion resistance.

#### 18.2 Materials and Methods

Table 18.1 Elemental composition of Al 2024

#### 18.2.1 Matrix and Reinforcement Materials

The matrix material used in this work is Al2024 alloy of grain size 50  $\mu$ m, and it is received from Parshwamani Metals, Mumbai. The elemental compositions of this composite are given in Table 18.1. The major contribution in this composite is copper, and it is used in aerospace and automobile applications. These alloys have good strength-to-weight ratio and corrosive properties. Two types of reinforcements are used to fabricate the required mono- and hybrid metal matrix composites. In the initial stage,  $B_4C$  of grain size 100  $\mu$ m is added to the aluminium alloy to obtain aluminium metal matrix composite. In the lateral stage, fixed amount of Gr (3%) of



Material	Average size (µm)	Density $(g/cm3)$
Al 2024	50	2.71
$B_4C$	100	2.52
Gr	100	2.09

Table 18.2 Particle size and density of various elements



Fig. 18.1 SEM images of received, a Al 2024, b B<sub>4</sub>C, c graphite

particle size 100  $\mu$ m by varying B<sub>4</sub>C (0, 3, 6, 9, 12, 15%) is added to the aluminium alloy to fabricate hybrid metal matrix composites. Graphite is used widely in industrial application because of its self-lubricating and dry lubricating properties. The particle size of used materials is given in Table 18.2, and SEM pictures of received powders are shown in Fig. 18.1. The required quantity of composite powders (Al-3%B<sub>4</sub>C, Al-6%B<sub>4</sub>C, Al-9%B<sub>4</sub>C, Al-12%B<sub>4</sub>C and Al-15% B<sub>4</sub>C) and (Al-3%Gr-3%B4C, Al-3%Gr-6%B4C, Al-3%Gr-9%B4C, Al-3%Gr-12%B4C and Al-3%Gr-15%B4C) is weighed in a simple balance of an accuracy 0.0001 g and is taken in a tungsten carbide bowl of planetary ball mill for proper mixing. The SEM images of received individual powders are shown in Fig. 18.1.

## 18.2.2 Ball Milling

Powder metallurgy technique is adopted for the fabrication of all the composites. In the primary stage, various weight-based measured composite powders (Al-3%B4C, Al-6%B<sub>4</sub>C, Al-9%B<sub>4</sub>C, Al-12%B<sub>4</sub>C and Al-15% B<sub>4</sub>C) are well mixed in the

Fig. 18.2. RETSCH 100 ball milling machine



Fig. 18.3 FESEM for microstructure analysis



planetary ball mill and taken out from the bowl of the mill. In the lateral stage, aluminium metal matrix hybrid composites (Al-3%Gr-3%B4C, Al-3%Gr-6%B4C, Al-3%Gr-9%B<sub>4</sub>C, Al-3%Gr-12%B<sub>4</sub>C and Al-3%Gr-15%B<sub>4</sub>C) are well mixed in the ball mill itself. For the fabrication of all these composites, mechanical milling and alloying (RETSCH 100 Ball Mill Germany make) as shown in Fig. 18.2 is used for proper mixing of composite powders. Tungsten carbide balls of 10 mm diameter are added to these powders during mixing with ball-to-powder ratio of 10:1 [\[13](#page-13-0), [14\]](#page-13-0), and this milling process is carried out for about 30 min selected from the literature. FESEM analysis is conducted on mixed powders by using FESEM machine (Fig. 18.3) to know the elemental composition in each blend as shown in Fig. [18.4](#page-5-0).

## 18.2.3 Compaction of Mixed Powders

Compression process is carried out on each blend by using hydraulic compression testing machine as shown in Fig. [18.6.](#page-6-0) For each composition, five numbers of

<span id="page-5-0"></span>

Fig. 18.4 FESEM micrographs of produced powder mixtures after ball milling

<span id="page-6-0"></span>Fig. 18.5 Hardened steel die with plunger



Fig. 18.6 Compression testing machine



Fig. 18.7 Muffle furnace for sintering



samples are considered for different analyses. The mixed compositions are consolidated in a hardened steel die along with a punch as shown in Fig. 18.5. These powder mixtures are compressed up to a pressure of 400 Mpa [[13,](#page-13-0) [14\]](#page-13-0); maintaining same pressure for about 5 s, then the pellets are ejected from the die. After compaction, the green pellets having 15 mm diameter and 25 mm length are obtained from the die. For each run, the die is filled with approximately same quantity of Fig. 18.8 Specimens after sintering



well-mixed powders. Silicon spray was sprayed into the die before the powder is put into the dye, so as to provide proper lubrication between the die walls and the powder. Then, the punch was placed over the filled powder and gradual pressure was applied over the powder.

# 18.2.4 Sintering

The sintering process is used to convert the green pellets into the required specimens. The grain growth and grain bonding of the green pellets are taken place after the sintering process. The specimens are placed in a muffle furnace as shown in Fig. [18.7](#page-6-0); the temperature is increased up to 600  $^{\circ}$ C which is the bonding temperature [[13,](#page-13-0) [14](#page-13-0)] and is maintained at this temperature for about 3 h by following 10 °C per min as the increase rate of the temperature. The sintering process is carried out under an inert atmosphere (Organ gas) to avoid oxidization during the process. The cooling is carried out in the furnace itself by following 5 °C per minute to reach room temperature. The final samples after the sintering process are shown in Fig. 18.8.

## 18.3 Microscopic Examination

Microscope is used for microscopic examination. Aluminium, boron carbide and graphite are detected in the analysis. The fabricated specimens of sintered preforms are examined in metallurgical microscopic, before going to conduct the examination, the fabricated samples are prepared by using standard hand polishing with 240, 600, 800 and 1000-grit silicon carbide papers. The etch–polish–etch procedure by using Keller's etchant is used to attain mirror-like surface. The images obtained from microscope show the uniform distribution of particles throughout the surface of the specimen without any agglomerations.

# 18.4 Corrosion Test

#### 18.4.1 Electrochemical Analysis

#### 18.4.1.1 Preparation of Test Solution

The test solution of 3.5 wt% NaCl is prepared by dissolving 35 g of NaCl powder in 1 L distilled water to obtain 0.6 M solution, in which chlorine is the active pitting agent during corrosion test [[16,](#page-13-0) 22]. All the experiments are conducted in aerated 3.5% NaCl solution with PH adjusted to 10 by adding potassium hydroxide.

#### 18.4.1.2 Corrosion Procedure

The corrosion phenomenon can be found by using several methods in which weight loss and spray test analysis are qualitative. In this experimental work, electrochemical analysis technique is used to get reproducible quantitative data. To study the corrosion behaviour of fabricated aluminium samples, software-based PAR electrochemical weld tester system is used to carry out potential dynamic polarization tests. The basic electrochemical system with electrochemical flat cell used in this work is shown in Fig. [18.9](#page-9-0). A standard calomel electrode SCE and carbon electrode are used as reference and auxiliary electrodes, respectively. All the experiments are conducted in aerated 3.5% NaCl solutions with PH adjusted to 10 by adding potassium hydroxide. The potential scan is carried out at  $0.166$  mvs<sup>-1</sup> with the initial potential of  $-0.25$  V (OC) SCE to the final pitting potential. The exposed area for these specimens is  $1 \text{ cm}^2$ . Polarization curve is plot between potential (mv) and current density (mA/cm<sup>2</sup>) which is having anodic and cathodic branches. Potential at which the anodic and cathodic branches are met is taken as corrosion potential  $(E_{\text{corr}})$ ; the typical polarization curves are shown in Figs. [18.11](#page-11-0) and [18.12.](#page-11-0) Specimens exhibiting relatively more positive corrosion potential (or less negative potentials) are considered to have better corrosion resistance. The corrosion test is conducted by potential dynamic polarization tests as explained in the previous procedure; then, the same corrosion test is conducted on all the samples to know the corrosion behaviour. The pits and cracks are observed in every specimen after the corrosion test. According to the ASTM G 59, potentiodynamic anodic polarization technique is carried out on the fabricated composite samples to know the corrosion behaviour of tested samples. The values of  $E_{\text{corr}}$ ,  $E_{\text{pit}}$  and corrosion current are calculated for all the test samples. The microstructure is visualized with optical microscope, and the micrograph is captured before the corrosion and FESEM analysis is conducted after the corrosion test.

The optical microscopic and FESEM images shown in Fig. [18.10](#page-10-0) describe the corrosion process taken over all the obtained samples before and after corrosion. The obtained images, number of pits and cracks formed over the surface of all the specimens after the corrosion test. Comparing these images, it has been observed

<span id="page-9-0"></span>

Fig. 18.9 Schematic representation of flat cell

that the pits and cracks formed over the surface of hybrid composites are more than the monocomposites and base aluminium alloy. The cracks and pits formed over the surface of the base aluminium alloy are less as compared with the other composites; this is because of the inter-granular corrosion which is the localized corrosion attack surrounding the  $B_4C$  particles in monocomposites, whereas in hybrid composites, the corrosion is more due to the presence of graphite which leads to galvanic corrosion by forming a primary corrosion mechanism [\[17](#page-13-0)].

The obtained potentiodynamic polarization curve of potential versus current is known as Tafel plots (Figs. [18.11](#page-11-0) and [18.12](#page-11-0)). Different regions like the active region, passive region, beginning of film removal and passive region can be known from the Tafel plot. These are also known as anodic and cathodic polarization curves. With the help of these graphs, the values of corrosion current  $(i_{\text{corr}})$ , corrosion potential ( $E_{\text{corr}}$ ) and pitting potential ( $E_{\text{pit}}$ ) are given in Table [18.3.](#page-12-0)

From the above table, it is clear that the  $i_{corr}$  values decreases for both mono- and hybrid composites but the values of monocomposites are little bit smaller than the hybrid composites having same composition. Similar trend is observed for  $E_{\rm corr}$ values, but the values are in an increased manner. For better corrosion resistance,  $i_{\text{corr}}$  values should be maximum and  $E_{\text{corr}}$  values should be minimum. The corrosion resistance decreases from the base metal to  $B_4C$ -reinforced composite. The corrosion resistance further decreases upon addition of graphite.

#### 18.5 Conclusions

The aluminium metal matrix mono- and hybrid composites are fabricated successfully by powder metallurgy technique. Microstructure and FESEM analysis shows the uniform distribution of the reinforcement in aluminium alloy. The electrochemical cell analysis by potentiodynamic anodic polarization technique is

<span id="page-10-0"></span>

S.N $\overline{O}$	<b>COMPOSI</b> <b>TION</b>	<b>OPTICAL</b> <b>MICROSTRUCTURE</b> BEFORE CORROSION	<b>FESEM IMAGES AFTER</b> <b>CORROSION</b>	
$\mathbf{1}$	Al2024 Alloy			
$\overline{2}$	Al 2024- 3%B4c			
3	Al 2024-15 %B4c			
$\overline{4}$	Al 2024- 3% Gr- 3%B4c			
5	Al 2024- 3% Gr-12 %B4c			
6	Al 2024- 3% Gr- 15%B4c			

Fig. 18.10 Images of specimens before and after corrosion

conducted on the fabricated composites. The corrosion is found from Tafel graphs. The results of the samples show that the corrosion resistance of the base aluminium alloy is least. The corrosion resistance of the monocomposite is higher due to the presence of carbide particles and the localized corrosion attack surrounding the B4C particles. The hybrid composite of  $B_4C$  and Gr is least corrosive resistant among the three; the reason for this is the presence of graphite content, so the corrosion resistance decreased due to soft nature of graphite.

<span id="page-11-0"></span>

Fig. 18.11 Polarization plots for individual composite specimens



Composition	$i_{\text{corr}}$ (mA/cm <sup>2</sup> )	$E_{\text{corr}}$ (mV)	$E_{\text{nit}}$ (mV)
Al 2024 alloy	0.389	$-726.3$	$-558$
Al 2024-3% Gr	0.352	$-718.55$	$-566$
Al2024+3% $B_4C$	0.204	$-686.82$	$-570$
Al2024+6% B <sub>4</sub> C	0.179	$-673.64$	$-562$
Al2024+9% $B_4C$	0.163	$-688.9$	$-595$
Al2024+12% $B_4C$	0.154	$-690.66$	$-600$
Al2024+15% $B_4C$	0.145	$-683.58$	$-626$
Al2024+3%Gr +3% B <sub>4</sub> C	0.192	$-667.23$	$-594$
Al2024+3%Gr +6% B <sub>4</sub> C	0.156	$-671.33$	$-580$
Al2024+3%Gr +9% B <sub>4</sub> C	0.146	$-699.2$	$-609$
Al2024+3%Gr +12%B <sub>4</sub> C	0.137	$-686.66$	$-598$
Al2024+3%Gr +15% B <sub>4</sub> C	0.120	$-680.5$	$-637$

<span id="page-12-0"></span>**Table 18.3**  $I_{\text{corr}}$ ,  $E_{\text{corr}}$ ,  $E_{\text{pit}}$  and corrosion values of the specimen in 3.5 wt% NaCl corrosion media

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