Effect on corrosion behaviour of the surface of aluminium 4.5 Cu with bamboo leaf ash composites by laser treatment

Gadudasu Babu Rao 1, Praveen Kumar Bannaravuri 1 and Anil Kumar Birru 2

1 Mechanical Engineering Department, Karunya Institute of Technology and Science, Coimbatore, Tamil nadu-641114, India
2 Mechanical Engineering Department, National Institute of Technology Manipur, Imphal-795004, India

E-mail: banna.mech@gmail.com

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Abstract

Aluminium composites have various applications in engineering thanks to their greater strength with lightweight, formability and ductility. However, the properties of the Aluminium composites such as wear, corrosion, hardness, and fatigue resistance are critical at near-surface regions. In this present research work, the effect on the surface of Aluminium 4.5Cu with Bamboo Leaf Ash composite by laser surface melting was done, wherein the fabrication was done by stir-casting with bamboo leaf ash reinforced particles with 2 and 4 weight percentage. By using the laser machine, the composite’s surface was melted. Microstructure and corrosion behaviour of composites before surface melting and after laser surface melting was inspected and the outcomes were compared. It was perceived that, the diminishment of porosity in the grain refinement. The corrosion resistance of composites had improved remarkably by Laser Surface Melting. The Corrosion mechanism was analysed by observing the morphology of the corroded surface of composites. Henceforth the Laser Surface Melting will be applied appropriately to boost the surface properties of aluminium composites.

1. Introduction

Last few decades, there has been an increasing demand for higher-strength with lower-weight materials with excellent surface properties in the field of aerospace and automobile industries. There are an inclined interest and emphasis over Aluminium alloys and their corresponding metal matrix composites (MMCs). The Aluminium metal matrix composites (AMMCs) with hard ceramic particulates as reinforced like SiC, TiC, B4C, Al2O3, fly ash, Rice husk ash etc, have become demanded materials because of their lower weight, thermal expansion coefficient, high wear resistance, and excellent strength [1]. Studies claim an addition of reinforcements to aluminium makes a significant change in the hardness and corrosion behaviour [2]. In a wide-range, when aluminium composites are reinforced with hard particles, voids are formed round about the particles by reason of wettability of the particles while casting.

Voids and uneven distribution of particles are the surface defects of composites, which leads to minimizing applications in hard environments. The surface is melted by means of high-energy beams and solidification can minimize the imperfection and remarkably elevate the surface properties such as tribological and corrosion resistance [3, 4]. Bamboo Leaf Ash (BLA) squeezed from agro-waste and used as strengthening element in aluminium composites. BLA particles are considered as agro-waste in India and are available in large quantities. BLA particles that are used in MMCs, are of the economy and low density. The reinforcement of Aluminium done with the adding of BLA by the stir-casting method can minimize the density and overall cost of aluminium composites. Al-4.5 Cu B−1L−1A−1 composite was fabricated by stir casting method and perceived that mechanical properties of as-cast material had been improved reported by Praveen and Anil [5]. It was also observed the particles-agglomeration and porosity in their fabricated composite. Hence, in order to elude surface defects, a secondary improvised process was needed to be taken up. To boost the surface characteristics, few surface treatment technologies had been projected and examined. Laser surface melting is one of fascinated recent research interest in the surface treatment technologies. Reliant on the interaction topographies between
laser beam and composite materials surfaces, many laser technologies have been established such as laser heat-treatment, laser surface alloying (LSA), cladding and laser surface melting (LSM). Surface melted by LSM produced a refine microstructure that minimized the size and sites of the galvanic couples for pits nucleation [6]. LSM involves the application of short, high power pulses to melt the surface of the material, which then rapidly solidifies. The LSM can be used for local surface treatment as well as for treatment of large surfaces areas. Improvement of surface resistance to corrosion of aluminium composites can be achieved remarkably by laser treatment techniques which were used by the earlier researcher for aluminium alloys [7, 8]. In the LSM technique, special attention is paid to local corrosion improvement. Application of LSM to aluminium composites in the available literature is meagre.

Surface behaviour such as corrosion damage of Al-Fe alloy was noticed that less after laser treatment by Yb-fiber laser—2 kW (IPG YLR-2000S) functioning at 1.06 μm wavelength by Pariona et al [9]. Because of cyclic polarization a wide passive area was attained after laser treatment by ten times current reduction which confirms corrosion damage was diminished. Microstructure and the corrosion behaviour of a laser-treated surface of aluminium alloy were inspected by Serbinski et al [10]. The results derived indicated that laser treatment enhanced the corrosion resistance of the alloy remarkably in 0.01 m sulphuric acid solution. Man et al [11] observed erosion and the corrosion behaviour of a laser-treated surface of aluminium matrix. The results revealed that there was a reduction in pitting resistance that was acquired after the laser treatment process on the surface. Hu et al [12] have reported about corrosion properties of the surface of aluminium composite by YAG laser treatment. It was observed that the reinforcement of Al18B4O33 whisker and an intermetallic CuAl2 reduced on the surface after laser treatment, the formation fine grain structure with defect-free surface caused in the enhancement of resistance corrosion.

From earlier results, it understood that corrosion damage higher in the untreated samples, where, less in the laser-treated samples. It can be concluded that the LSM process may influence on surface properties. In this research work, the laser surface melting of composites of Al-4.5Cu with bamboo leaf ash reinforced particles was experimented by changing laser specific energy (LSE). Composites were made by varying the weight percentage of reinforcement particles BLA as 2 and 4. After treatment of laser surface melting, the surface integrity was examined and made a comparison with the base material and observed a convinced development.

2. Experimental details

Aluminium 4.5Cu was taken as matrix material and its chemical composition is depicted in table 1. Reinforcement particles of bamboo leaf ash (BLA) with an average size of 75 μm and 1.78 g cm⁻³ density were designated for the composite.

2.1. Manufacturing of composites

By using the stir-casting, the composites of Al-4.5Cu with BLA particles were fabricated. Here the Magnesium is taken in the form of the ingot in order to advance the wettability of reinforcement particles in the matrix alloy during the making of composites [13]. The fabrication methodology of composites is depicted in the flowchart as shown in figure 1. Tests such as microstructure and the hardness tests were conducted with the help of equipment and standardised tools.

An Aluminium 4.5Cu alloy, in ingot form was made into pieces and house in the crucible and then heated up to a temperature 800 °C in an induction electric resistance furnace in an argon atmosphere until the entire alloy is melted. The BLA particles were preheated for an hour at 350 °C to remove moistness and to stimulate the wettability with matrix. The BLA particles were incorporated at 0, 2 and 4 wt% into the matrix alloy by argon gas and in very casting nearly 0.1 wt% of Mg also added to stimulate the wetting of BLA reinforcement in the matrix alloy. After incorporated the BLA particles stirring was done with 600 rpm for 10 min, to improve the uniform spreading of BLA particles in the matrix alloy. The composite was fabricated with 0, 2 and 4 wt% BLA in the foundry and then, the microstructure and corrosion analysis were done by suitable equipment.

2.2. Surface melting by laser

The cast samples surface was melted by 2.5 kW CO₂ Laser machine, at a wavelength of 10.6 μm, whose schematic diagram is depicted in figure 2. The Laser parameters listed in table 2 are used for present research work and they

### Table 1. Chemical Composition of Al-4.5Cu alloy.

| Elements | Cu | Fe | Si | Mn | Zn | Ni | Mg | Mg | Pb | Sn | Ti | Al |
|----------|----|----|----|----|----|----|----|----|----|----|----|----|
| Weight percentage | 4.52 | 0.663 | 0.538 | 0.131 | 0.118 | 0.075 | 0.066 | 0.066 | 0.029 | 0.021 | 0.013 | Balance |
Figure 1. A flowchart for the making of composites.

Table 2. Parameter of the laser machine considered for LSM

| Sr. No. | Material (Al-4.5Cu matrix/ x wt% BLA) | Parameters of laser |
|---------|--------------------------------------|---------------------|
|         |                                      | P in kW  | D in mm | E in J mm⁻² |
| 1       | Matrix/0                             | 1.9      | 6.61    | 43          |
| 2       | Matrix/0                             | 1.9      | 7.56    | 38          |
| 3       | Matrix/0                             | 1.9      | 8.5     | 34          |
| 4       | Matrix/2                             | 1.9      | 6.61    | 43          |
| 5       | Matrix/2                             | 1.9      | 7.56    | 38          |
| 6       | Matrix/2                             | 1.9      | 8.5     | 34          |
| 7       | Matrix/4                             | 1.9      | 6.61    | 43          |
| 8       | Matrix/4                             | 1.9      | 7.56    | 38          |
| 9       | Matrix/4                             | 1.9      | 8.5     | 34          |

Figure 2. LSM Schematic diagram.
are optimized using below expression as laser specific energy (LSE), [33]

\[ E = \frac{P}{vd}, \]  

where \( P \) = laser power, \( v \) = scan speed constant (400 mm s\(^{-1}\) considered), and \( d \) = laser beam diameter.

2.3. Material characterization
An Optical Microscope is used for the metallographic inspection of the composites. The samples were machined to the required shapes and the surface is polished. Keller’s reagent (\( \text{H}_2\text{O} = 95 \text{ ml}, \text{HNO}_3 = 2.5 \text{ ml}, \text{HCl} = 1.5 \text{ ml}, \text{HF} = 1.0 \text{ ml} \)) was used as an etchant for microstructure examination [14]. An Optical microscope for microstructure analysis and an average grain size of the composites are measured at several places or locations on every specimen by a linear-intersect method with the help of ImageJ [15].

2.4. Hardness and corrosion measurement
Vickers hardness tester is used for hardness test and the average of 6 readings was taken on every sample by as per ASTM E384–11 and 100 gram the load. These tests were conducted before and after laser surface melting. Corrosion immersion test [16–18] was conducted for as-cast and LSM composites. Then the samples were mounted with phenol resin that resisted corrosion caused due to salt solutions. The portion of the surface to be tested for corrosion was located in the corrosive media (NaCl) for 200 h. The pH of the solution was noted before the corrosion test by pH meter. After the corrosion test, the weight loss because of corrosion damage on the surface of as-cast and LSM composites was measured and was analysed by SEM.

2.5. Electro-chemical test
The Electro-chemical test was carried out by using a three-electrode cell with Versa-Studio software (Ametek instruments India private limited, model: PMC-CHS08A) as shown in figure 3. Silver/Silver Chloride as a reference whereas platinum wire coiled used as counter electrodes. The Al-4.5Cu/BLA composite anode samples (before and after LSM) are used as working electrodes and were machined into cylindrical shapes with an exposed surface area of 10 mm\(^2\). The copper wire was attached to working electrodes and it was immersed in glass tubes. The prepared samples were coated using conducting sliver paint and epoxy resin except for one surface, which was exposed to corrosion. The exposed surfaces were ground with wetted 1000 grade SiC paper and then washed thoroughly with distilled water and cleaned with acetone prior to testing. The test environment was 5 wt% NaCl buffered to pH 8.15 using 0.5 M NaOH. Electrochemical Impedance Spectroscopy (EIS) test was carried out at Open Circuit Potential (OCP) at a scanned frequency from 100KHz to 100 mHz, with an alternating current (AC) perturbation of ± 10 mV to give the corresponding Nyquist and Bode plots. The attained results were inserted with an equal circuit with elements that were estimated to have physical meaning in the system.

3. Results and discussions
Microstructures analysis for the composite samples and mechanical tests conducted on them were discussed in the following sections.
3.1. Microstructure analysis
Before performing laser surface melting, optical micrographs of matrix alloy and aluminium composite reinforced with BLA at 2 and 4 wt% were depicted in figure 4. The α-Al and Cu eutectic phase and the larger grain size of matrix alloy were identified from the micrograph of the matrix as depicted in figure 4(a). Micrographs of BLA 2 and 4 wt% reinforced aluminium composites were disclosed that uniform dispersion of BLA particles are identified in the matrix alloy from figures 4(b) and (c). It was observed that there are many blow holes in all the micrographs due to air bubbles entrapped in the melt because vertex generated when stirring process performed. And also porosity was noticed in the matrix and BLA reinforced composites as shown in micrographs (figure 4). The particles agglomeration was identified in some places of composites as shown in figure 5. Along with the aluminium grain boundaries, the BLA particles were ensnared by converging dendrites in the intracellular regions and porosity observed as shown in figure 5. The particles agglomeration was identified in some places of composites as shown in figure 5. Along with the aluminium grain boundaries, the BLA particles were ensnared by converging dendrites in the intracellular regions and porosity observed as shown in figure 5. With hasty melting, the agglomerated area can be break and uniform distribution of particles may achieve with rapid solidification [19].

The micrograph of matrix alloy at laser specific energy of 34, 38 and 43 J mm$^{-2}$ correspondingly as shown in figures 6(a) to (c). It is noticed that some grain refinement and porosity of the matrix at LSE of 34 J mm$^{-2}$. With LSE of 38 J mm$^{-2}$, it is achieved the fine grain refinement and porosity minimized on the surface of the matrix as depicted in figure 6(b). Surface with out defect was detected because of apposite melting and higher solidification rate can be achieved at LSE of 38 J mm$^{-2}$. But at higher LSE of 43 J mm$^{-2}$, several micro-pores and cracks were observed on the surface of the matrix as shown in figure 6(c), the reason for this is overheating. It was perceived that there was fine grain refinement after re-melting of surface by laser when compared with the matrix alloy premelting.

The optical images of aluminium composites at 2 and 4 wt% of BLA after LSM at 38 J mm$^{-2}$ of LSE as depicted in figures 7(a) and (b) respectively. The interface between the matrix and BLA particles was seen clearly, which confirmed the particles spread in the matrix alloy uniformly. Remarkable grain refinement was viewed in the 2 and 4 wt% of BLA reinforced composite as shown in figures 7(a) and (b). Better surface properties can be
achieved with refinement grain size of the material. It was observed the smallest grain size at 4 wt% of BLA reinforced and micro-cracks are not detected as shown in figure 7(b).

The surface of aluminium composite melted by laser beam gain the better microstructure and eludes the agglomeration of BLA particles by dispersing uniformly as shown in figure 8. Mean linear intercept method is used for the calculation of average grain size. Figure 9 displays an average grain size of the as-cast material and LSM material at LSE of 38 J mm$^{-2}$. The remarkable reduction notified in the average grain size after LSM when compared with as-cast materials, this because of rapid solidification arise after LSM. Similar outcomes were also observed after LSM on the surface of Al–1.5 wt% Fe alloy by using a high-power Yb-fiber laser (IPG YLR-2000S) by Pariona et al[20], furnished with an ytterbium single emitter semiconductor diode.
3.2. Micro-hardness
The density and micro-hardness of matrix and composites before LSM are portrayed in figure 10. It was observed that the density of the composites reduced by the adding of reinforcement since the density of BLA is lower than the matrix alloy as depicted in figure 10(a). And also found that the hardness was improved by the adding of BLA in the composite as shown in figure 10(b). The enhancement in hardness due to reinforcement of particles which acts as load-bearing elements through the interface between matrix and particles. Saravanan et al [21] processed aluminium alloy-based composites with addition of Rice Husk Ash (RHA) by stir-casting method, it was reported that hardness was enhanced with the addition of RHA particles and some porosity had existed.

The micro-hardness of the matrix and BLA reinforced composites material shown in figure 11 after LSM at LSE of 34, 38 and 43 J mm$^{-2}$ respectively. The hardness was enhanced notably in all the cases of material after LSM when compared with as-cast material. For Al-4.5Cu/4BLA composite after LSM, the maximum hardness was noticed at LSE 38 J mm$^{-2}$ due to the presence of the fine grain structure of composite and uniform dispersal of reinforcement particles in the composite. As distribution of particles increased uniformly throughout the surface of composite, there is an enrichment in load bearing capacity, thereby an enhancement of hardness.

An improvement of micro-hardness after LSM is achieved due to the surface-temperature being lower than the melting-temperature of reinforcement (BLA), the hard particles remained in solid phase during the laser melting process. The melting-temperature of reinforcement is significantly higher than the melting temperature

![Figure 9. An average grain size of matrix and laser surface melting composite at laser specific energy of 38 J mm$^{-2}$.](image)

![Figure 10. Experimental density (a) and micro-hardness (b) of as-cast material.](image)
of the matrix material. Although, coefficient of thermal expansion (CTE) of reinforcement and matrix alloy are different. The variances in CTE generates strain fields around reinforcement particles due to the rapid solidification process. The dislocations movement is obstructed in the composite by strain fields when the load is applied \[22-24\]. Grain size is the greatest substantial feature to improve the hardness. At laser specific energy of 38 \(\text{J mm}^{-2}\), the highest micro-hardness was observed due to the finest grain size which was formed. Grabowski and Moskal \[25\] fabricated AlSi12/SiC\(_p\) composite by means of stir casting method and carried out LSM; they also observed an enhancement of hardness due to LSM.

3.3. Salt solutions corrosion analysis

The assessment of corrosion attack on the surface of matrix alloy and the composite was described after corrosion immersion test in alkaline (salt) solution (5% NaCl) by percentage weight loss method. The pH value of solution is 9.08 for salt solution. The corrosion weight loss values of as-cast matrix and composites were caused by corrosion damages in salt solution is shown in figure 12. The as-cast material weight loss in salt environment was 0.37% for matrix, 0.28 % for 2 wt% BLA and 0.24 % for 4 wt% BLA of Al-4.5Cu composites. It is clear from figure 12 that corrosion weight loss is more for the all as-cast materials in salt environment.

![Figure 11](image1.png)

Figure 11. An Effect of LSE on Micro-hardness.

![Figure 12](image2.png)

Figure 12. The percentage of weight loss due to corrosion for as-cast and LSM material.
Composites have higher corrosion resistance than as-cast matrix material. Moreover, with the increase in the wt% of BLA, the corrosion resistance increases. The corrosion weight loss values of LSM matrix and composites at LSE of 38 J mm$^{-2}$ are also shown in figure 12. The fine grain structure and highest hardness value observed at LSE of 38 J mm$^{-2}$ due to this reason corrosion study was conducted. The material weight loss in salt environment was 0.28% for matrix alloy, 0.21% for 2 wt% BLA and 0.18% for 4 wt% BLA Al-4.5Cu composites. Overall, corrosion resistance improved after LSM due to elimination of porosity and fine grain structure.

Figures 13 to 14 show the surface morphology of as-cast and LSM matrix alloy and composite after alkaline corrosion immersion tests, carried out for 200 h. In the as-cast matrix material, corrosion was in the form of uniformly distributed holes called pits as shown in figure 13 (a) in a salt environment. These are not observed after LSM (figure 13 (b)).

The corroded surface morphologies of as-cast and LSM Al-4.5Cu/4BLA composite after salt corrosion immersion tests are shown in figure 14. The corrosion damage was higher in the as-cast composite and micro-cracks appeared (figure 14(a)); lesser corrosion damage was detected in the composite after LSM (figure 14(b)). Pit size in as-cast surface of the material is significantly higher than that in the LSM composite. In the composite, boundaries of reinforcement particles remain uncorroded (figure 14(b)). LSM improved corrosion resistance. Similarly, Yue et al and Yilbas et al [26, 27] noticed that the corrosion resistance of Aluminium composite and AA7075 aluminium alloy after LSM got improved.

3.4. Open circuit potential decay
The plots of open circuit potential (OCP) decay for the Al-4.5Cu/4BLA composite before and after LSM as shown in figure 15. The OCP was monitored for two hours to ensure the stability of the system before carrying
out the electrochemical tests. Significant fluctuations were observed for the first 4000 s, but later which the composites became relatively stable drifting within 0.05 V. As-cast Al-4.5Cu/4BLA and LSM Al-4.5Cu/4BLA had OCP values of $-0.63$ and $-0.64$ V measured versus Ag/AgCl reference electrode. The OCP values are purely reliant on the chemical composition of composites are being tested.

3.5. Effects of immersion time on the EIS behaviour of the composite

From the EIS test, very useful information on the electrochemical reactions was noticed at the surface of aluminium composites. Figure 16 shows the Nyquist plots for as-cast and LSM composites. The Versa-Studio software was used to fit the equal circuit diagram shown in figure 17 and acquire values for the circuit elements. In the equivalent circuit the constant phase element (CPE) was used as depicted in figure 17, in place of a pure capacitor as it represents sound the corrosion phenomenon that was taking place. $R_s$ signify the solution resistance. The impedance of CPE is represented as:

$$ Z(j\omega) = \frac{1}{\omega^n Y_0} $$

(2)
where \( Y_0 \) denote the CPE constant, \( j \) signifies the imaginary unit, and \( n \) the CPE power and angular frequency. The EIS parameters attained from suitable the equivalent circuit are shown in table 3. Rct and CPE signify the charge transfer resistance and constant phase element. The Rct value is considered to be equivalent to the polarization resistance \( R_p \). The EIS diagrams (from Nyquist plots) for the Al-4.5Cu/BLA composite before and after LSM is characterised by a onetime constant \([28]\). The Nyquist plots show clearly for composite, there was a noteworthy decrease in the Rct values throughout the period of immersion. In the LSM composite, the Rct values were normally higher signifying an enhancement in corrosion resistance \([29, 30]\). These increment results in Rct value be accredited to a likely thickening of the passive film on the composite surfaces. There were no visible signs of pitting on the LSM composite surface confirmed by the absence of any inductive loops on the Nyquist diagrams. These results were in line with Idusuyi \( et \ al \)[31] and Alaneme \( et \ al \)[32].

It can be concluded that the incorporation of hard (BLA) particles into the matrix alloy, the corrosion resistance get enhancement by the physical properties of reinforcement. LSM composite was identified as the better corrosion resistance composite due to uniform distribution of reinforcement particles in comparison with as-cast composite. Increasing the weight fraction of the reinforcement particles can improve the corrosion resistance of the LSM composite.

### 4. Conclusions

Here are some of the conclusions that were drawn for Al-4.5Cu alloy with 2 and 4 wt% of BLA as reinforcement material fabricated by means of stir casting method and surface of stir casted material was re-melted by laser:

1. The reinforcement particles are uniformly distributed in the composite fabricated by means of stir casting method and it is observed there are some particles agglomeration.

2. There is a decrement in the density by adding reinforcement particles in the composite whereas porosity increases.

3. There is an improvement in the surface properties of composites by eliminating porosity by refining the grain particles and uniform distribution by means of LSM. There is a significant improvement in the hardness of composite after LSM.

4. The corrosion behaviour of as-cast and LSM material was observed in alkaline (salt) environment by weight loss method. The corrosion damage was higher in the as-cast material when compared to LSM material.

#### Table 3. EIS parameters for Al-4.5Cu/BLA composites.

| Material     | Day | \( R_{ct} \) (Ohm) \( \times 10^4 \) | CPE \( \times 10^{-5} \) | N \( \times 10^{-1} \) |
|--------------|-----|------------------------------------|--------------------------|---------------|
| As-cast Al-4.5Cu/4BLA | 0   | 1.110                             | 1.11                      | 6.23          |
|               | 1   | 3.420                             | 2.12                      | 6.10          |
|               | 2   | 1.230                             | 2.34                      | 6.00          |
| LSM Al-4.5Cu/4BLA | 0   | 3.920                             | 1.17                      | 6.46          |
|               | 1   | 3.650                             | 1.12                      | 6.55          |
|               | 2   | 2.980                             | 1.02                      | 7.32          |

Figure 17. Equivalent Circuit Diagram for Al-4.5Cu/4BLA composite.
5. Corrosion resistance was observed by Electro-Chemical test, and it was observed that the corrosion resistance was higher after LSM.

6. It is summarised that, after LSM, the surface properties of the composite material enhanced remarkably.

**ORCID iDs**

Gadudasu Babu Rao 🌐 https://orcid.org/0000-0002-0161-6515

Praveen Kumar Bannaravuri 🌐 https://orcid.org/0000-0002-4740-194X

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