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Development and Comparative Study of Aluminium-Copper Metal Based Composite Through Different Routes

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Abstract

The aim of this study is to develop aluminium-copper composite through powder metallurgy technique. Powder metallurgy is an art or technology which can produce exact dimension of final roducts utilizing less energy. Copper oxide (CuO) was used as the source of copper, which was extracted through a reduction process using dextrose. The reaction was thoroughly examined and reported. Commercially available aluminium powder served as source of the aluminium. Aluminium- copper powder mixtures were prepaired where copper percentages (weight percentage) were varied from 0 to 20 percentage. Prepared powders were uniaxially pressed and sintered at 600^{0}C for one and half hours into controlled environment towards developing the composites. The sintered composites were subjected to various characterization and measurements, including apparent porosity, density and mechanical properties. Furthermore, developed composite materials underwent low-temperature aging treatment, afterwhich mechanical properties were evaluated. The effects of heat treatment on microstructural changes were investigated using optical microscopy and Field Emission Scanning Electron Microscope (FESEM). Results show a direct correlation between the increase in copper content and a systematic enhancement in hardness values. Aluminum, due to its lower density compared to copper, contributes to composites with a high strength-to-weight ratio while also enhancing stiffness and overall mechanical performance compared to pure sintered aluminum.

Keyword: Powder metallurgy, aluminium matrix, Copper reinforcement, reduction technique, surface morphology, density, porosity, hardness

INTRODUCTION

Several metal matrix composites have been developed in recent years, among which aluminum-based composites have prominent attention for various industrial applications [1–2]. These metal-based composites are specially designed to achieve an excellent strength-to-weight ratio along with satisfactory stiffness properties [2–3]. Although many studies have shown improvements in mechanical performance, limited flexibility remains a common challenge [3-6]. The plastic behavior of composites is closely related to the bonding matrix, which is significantly affected by the production and characteristics of the reinforcements [1, 7–8].

Copper oxide is an important precursor used in many chemical reactions to produce various products [2–4]. Several processes have been proposed for the complete reduction of CuO, leading to the formation of metallic copper, which

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is considered to the actual active phase in different applications [9–11].

Investigation of the reduction of CuO by hydrogen (H_2) under in situ conditions is therefore of primary importance. Research conducted ex situ may not accurately describe the true state of the copper phase under reducing environments [12–13].Copper is considered as an effective reinforcement material in metal-matrix composites. Combining aluminum as the matrix metal with copper reinforcement enables the fabrication of metal matrix composites through the powder metallurgical technique [13–15]. Powder metallurgy is an attractive processing technique for producing near-net-shape products [13, 15–16] and is widely employed in the development of engineering components and particulate-reinforced metal matrix composites [17–19]. The fundamental process in powder metallurgy technology involves the mixing and blending of powders, followed by the compaction of the mixture and subsequent sintering of the compacted powders to achieve densification [12–13, 15, 20].

Heat treatment is one of the most important methods for enhancing the mechanical properties of aluminum alloys, as it significantly influences the microstructure of the composites [13, 21]. Al-Cu alloys containing a copper content of up to approximately 5% are particularly suitable for heat treatment process, known as precipitation hardening [21–23]. Copper is one of the most significant alloying elements in aluminum due to its high solubility and potent strengthening effect [12–13, 15]. In precipitation hardening, or age hardening, Al-Cu alloys are subjected to aging at elevated temperatures [2, 10, 14]. However, various age-hardening behaviors have been observed in aluminum matrix composites fabricated through powder metallurgy. Scientific literature indicates that composite materials exhibit rapid hardening and reach peak hardness so earlier compared than the corresponding unreinforced alloys [15–16, 24, 25]. This behavior suggests accelerated aging characteristics facilitated by the reinforcement [17, 26, 27]. One of the significant challenges in the production and large-scale application of composite materials is the high cost of raw materials. However, scientists and researchers are actively addressing this issue by focusing on the development of composites utilizing reinforcement materials that are relatively low in cost [12–13, 15]. In addition, powder metallurgy techniques further help in reducing the overall fabrication cost [15, 26–28]. The aim of the present study is to investigate different aspects, including the reinforcement behavior associated with varying weight percentages of copper into aluminum matrix, the reduction process of copper oxide, its influence on mechanical properties, the age hardening behavior, and the evolution of surface morphologies.

EXPERIMENTAL METHODS

Reduction Process

High-purity copper oxide (CuO) powders supplied by Loba Chemie Pvt. Ltd. was used in this study. In this process, 1 gm of copper oxide was carefully mixed with 0.565 grams of dextrose obtained from SRL Pvt. Ltd. The powders were thoroughly blended before initiating the reaction at about 650°C in an inert atmosphere. The reduction reaction occurred within a timeframe of merely 12 to 15 minutes prior to the commencement of the experimental study.

The primary reactions that ensued were as follows:

Dextrose ($C_6H_{12}O_6$) undergoes thermal decomposition to yield carbon (C) and water vapor (H_2O), leading ultimately to the formation of carbon monoxide (CO) and hydrogen gas (H_2).

$$C_6H_{12}O_6 \Rightarrow 6C + 6H_2O \Rightarrow 6CO + 6H_2 \quad (1)$$

$$CuO + H_2 \Rightarrow Cu + H_2O \quad (2)$$

The copper oxide (CuO) engaged in a direct reduction reaction with hydrogen gas (H_2), yielding elemental copper (Cu) and water vapor (H_2O).

Through meticulous preparation and controlled conditions, the experimental setup induced these significant chemical transformations, elucidating the intricate interplay between copper oxide and dextrose under elevated temperatures under inert atmosphere.

Synthesis of Aluminium- Copper Composites

Method-I

In these experimental procedures, commercially available pure aluminum powder was used as the primary source of material. To attain uniformity, the aluminum powder was blended using mortar parcel for 2 to 3 hours. This thorough mixing ensured a homogeneous distribution of particles, laying the groundwork for uniform properties in the ensuing composite material. After blending aluminium, commercially available copper powders with varying weight percentages were incorporated. Each variation of copper powder underwent a careful mixing regimen, with one-hour intervals between each blending session to guarantee comprehensive homogenization. This systematic approach ensured the uniform dispersion of copper particles throughout the aluminium matrix, which is essential for achieving the desired characteristics in the final composite products. Once the powders were thoroughly mixed, the blended mixture was transferred into a cylindrical die chamber for compaction. Utilizing a compaction machine with a precisely calibrated setup, the powders were subjected to compression at a pressure of 4 ton for timing 3 to 4 minutes and finally considered for sintering at 600^0C for one and half hours. After sintering, samples were allowed to cool in the same furnace.

Method-II

In these experimental procedures, commercially available pure aluminum powder was considered as the fundamental material. These aluminum powders underwent a meticulous blending process in a mortar parcel, meticulously conducted to complete homogeneity. This step was critical to ensure uniform dispersion of particles throughout the entire mixture as well as getting consistent properties in the final composite products.

Following the blending process, copper powder was introduced into the mixture. This copper powder was obtained through a reduction process facilitated by dextrose material, ensuring its purity and desired characteristics. Careful incorporation of the reduced copper powder into the blend aluminum powder was followed by further blending in the mortar parcel to achieve thorough integration. This step was crucial as it introduces copper, which can impart specific properties such as enhanced strength or conductivity to the composite material.

Subsequently, the meticulously blended powders were transferred into a solid cylindrical die chamber. The purpose of this transfer was to prepare for the compaction process, which involves applying 4 ton pressure for timing 3 to 4 minutes to the powder material to form a solid shape. The choice of a cylindrical die chamber is deliberate, as it allows for uniform compaction and the production of cylindrical or rod-like shapes, depending on the desired final product. Finally, sintering was carried out at 600^0C for one and half hours for developing final composite products.

Aging

Developed all the sintered composites were subjected to heat treatment, followed by an aging process conducted at temperatures ranging between 190°C and 200°C. The aging duration was set to 4 hours to allow for sufficient time for the desired material transformations to occur.

During the heat treatment process, the samples were exposed to controlled temperatures within the specified range to induce structural changes and enhance certain material properties. Subsequently, the aging phase commenced, during which the samples were maintained at the predetermined temperature for the specified duration.

Evaluation of Properties

Each sample was polished and considered for properties evaluation. Physical properties like density, porosity and mechanical properties like hardness were evaluated and reported. The detailed procedures were described in earlier literature [13, 15].

Characterization

XRD

The atomic distribution and molecular structure are analyzed using X-Ray diffraction study. All the samples are taken in a trace amount and XRD test is executed over them using “Rigaku Ultima III analytical diffractiometer.” machine.

Optical Micrograph

Each developed composite was etched using Keller’s reagent (containing 2.5 ml of HNO_3, 1.5 ml of HCl, 1 ml of HF, and 95 ml of distilled water) and optical micrograph was taken at various magnifications using Leco Spectrum System 1000.

FESEM Study

Microstructure, morphology and chemical compositions are studied more accurately using Field Emission Scanning Electron Microscopy (FESEM) imaging method. Basically in this study surface morphology based on FESEM image was observed employing INSPECT F50 SEM.

RESULTS AND DISCUSSION

Reinforcement Composition with Heat Treatment

Figure 1 represents the composition of the composite material in which aluminium acts as the matrix and copper is used as reinforcement. Various combinations of aluminium- copper composites with different reinforcement percentages are considered. Result shows uniform diffusion of copper particulates within the aluminium matrix. Additionally, increasing the reinforcement percentage, significantly enhances the hardness of the composite materials after conducting heat treatment process.

XRD

In this series of experiments, the aluminium- copper composites underwent comprehensive phase characterization. Figure 2 shows the diffraction pattern of copper where copper is extracted from copper oxide. Figure 3 and 4 shows the diffraction pattern for aluminium- copper composites.

Phase analysis of conventionally sintered aluminium- copper composites (Figure 3) show a heightened level of crystallinity, which is evident from the sharp diffraction peaks.

Upon scrutiny of the XRD peaks (Figure 4), the presence of copper, derived from the reduction of copper oxide (CuO), is evident, alongside a minor occurrence of cuprite. The distinct, sharp peaks underscore the peak intensity of copper. Figure 3 depicts the discernible presence of both aluminum and copper in the XRD pattern, with copper originating from the reduction of copper oxide. The peak intensity of aluminium- copper further validates the authenticity of the material within these phases.

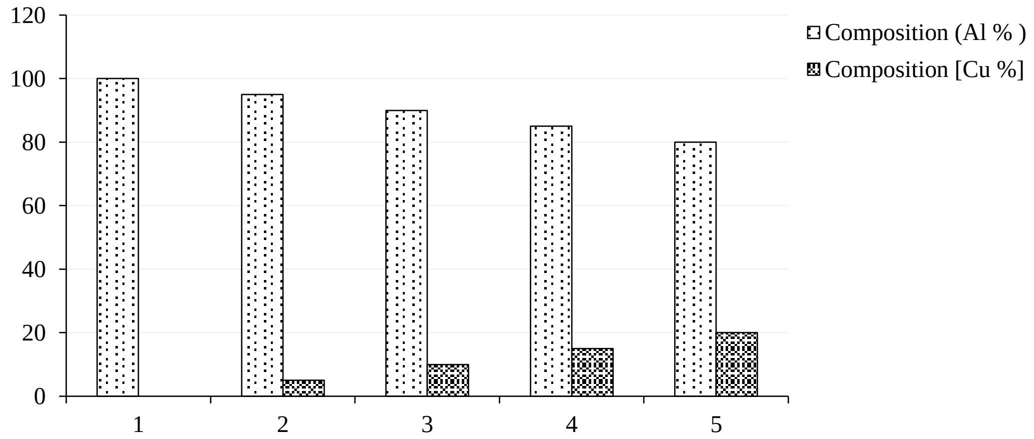

Figure 1. Effect of copper content after heat treatment.

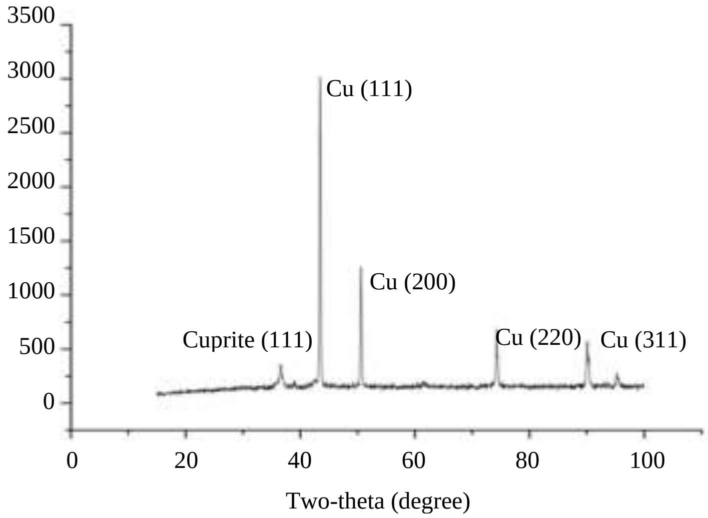

Figure 2. XRD plot of copper obtained from reduction of copper oxide.

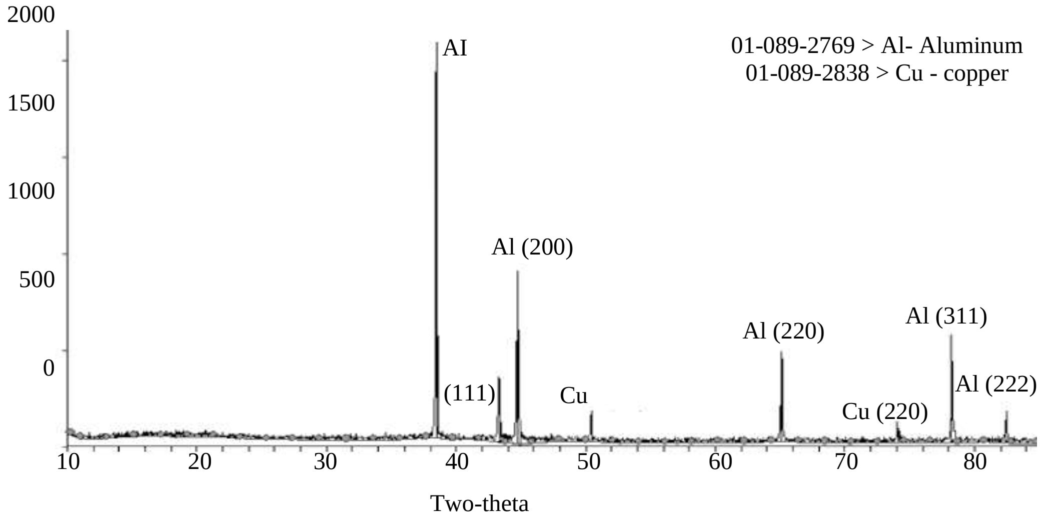

Figure 3. XRD plot of aluminium- copper composites (used commercial copper).

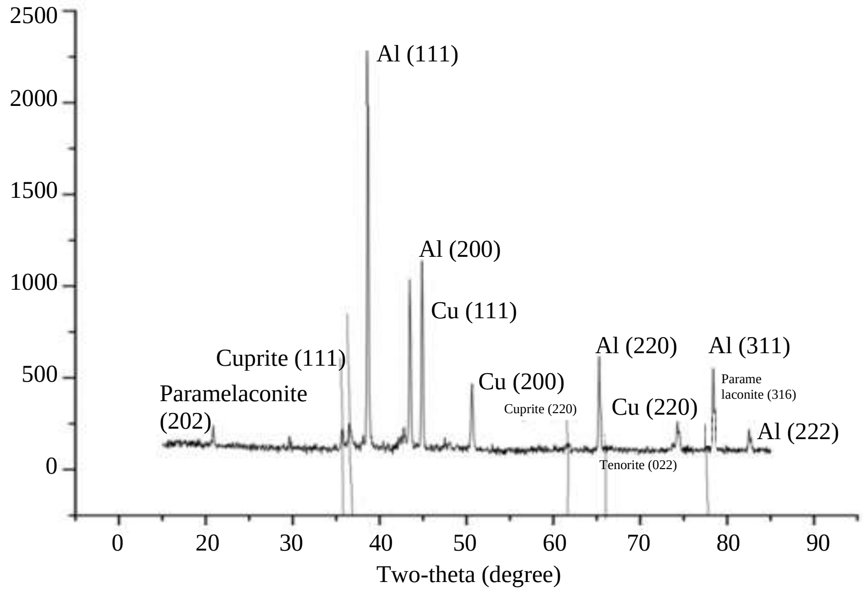

Figure 4. XRD plot of extracted copper- aluminium composites.

The XRD pattern of the pressed composite samples with different Cu contents, processed at 600°C with pure aluminum acting as the matrix material, is shown in Figure 3. The material exhibits distinct phases of aluminium and copper, both of which have exceptionally high diffraction intensities. The pure aluminum phases were found to have the highest intensity at a 2θ value of 38.482°, which corresponds to the (111) plane. There are also peaks at 44.736°, 65.106°, 78.250°, and 82.458° that are indicative of aluminum. The corresponding (hkl) planes for these peaks are (200), (220), (311), and (222), respectively. Copper, on the other hand, peaks at the highest intensity at a 2θ - value of 43.309°, which corresponds to the (111) plane. In the Al-Cu composites, there are additional copper-related peaks at 50.449° and 74.146°, corresponding to (hkl) planes of (200) and (220), respectively.

Similarly, at 600°C under inert atmosphere, the XRD pattern in Figure 2 shows the reduction of copper compounds from copper oxide. The (111) plane, with a 2θ value of 43.446°, exhibits the maximum copper intensity. Moreover, additional copper phases with equivalent (hkl) planes of (200), (220), and (311) are found for 2θ values of 50.586°, 74.245°, and 90.051°. Additionally, this XRD pattern shows evidence of Cuprite traces.

Likewise, pure aluminum and reduced copper were seen in Figure 4. XRD pattern at about 600°C under inert atmosphere. The graph of these XRD patterns shows that the (111) plane, or a 2θ value of 38.548°, is where pure aluminum exhibits the highest intensity. Other peaks with comparable planes of (200), (220), (311), and (222) are also detected at 44.802°, 65.191°, 78.304°, and 82.508°. Additionally, the XRD pattern shows that the (111) plane has the maximum intensity of copper with a 2θ value of 41.3°. A similar observation was made of further copper peaks at 50.537° and 74.198°, which correspond to planes (220) and (200), respectively. Additionally, from the XRD perspective, traces of paramelaconite, cuprite, and tenorite are detected, albeit with significantly lower intensities.

All the XRD analyses were conducted with the assistance of the JPCDS and MDI/JADE7 software and detailed results are noted in Table 1.

Surface Morphology Analysis

Optical Micrograph

Optical micrograph for aluminium-copper composites are shown in Figure 5 and 6 under same magnifications. The same composite materials after completion of the aging process, are shown in Figure 7 at same magnifications.

The material employs copper (Cu) as its reinforcement, exhibiting a distinct dark hue within these profiles. These conspicuous dark inclusions intricately map out the distribution of copper within the microstructure, while the gleaming white portions denote the aluminum matrix. Upon increasing the proportion of reinforcement from 0% to 20%, the microstructure undergoes a significant augmentation in the distribution of copper phases, accompanied by a notable surge in porosity levels. This evolution underscores the intricate interplay between reinforcement proportions and the resulting micro-structural characteristics, offering insights into the material's mechanical properties and performance.

Table 1. Copper particle size calculation.

Element	2θ (Degree)	θ in radian	Flex width	d - value	Particle size (Angstrom)
Cuprite (111)	36.55	0.3185	0.412	2.4564	3.36413
Copper (111)	43.45	0.37915	0.353	2.081	3.92643
Copper (200)	50.55	0.44115	0.353	1.8041	3.92646
Copper (220)	74.25	0.648	0.353	1.2762	3.92659
Copper (311)	90.05	0.786	0.412	1.0889	3.36439

Journal of Polymer & Composites
Volume 14, Special Issue 3
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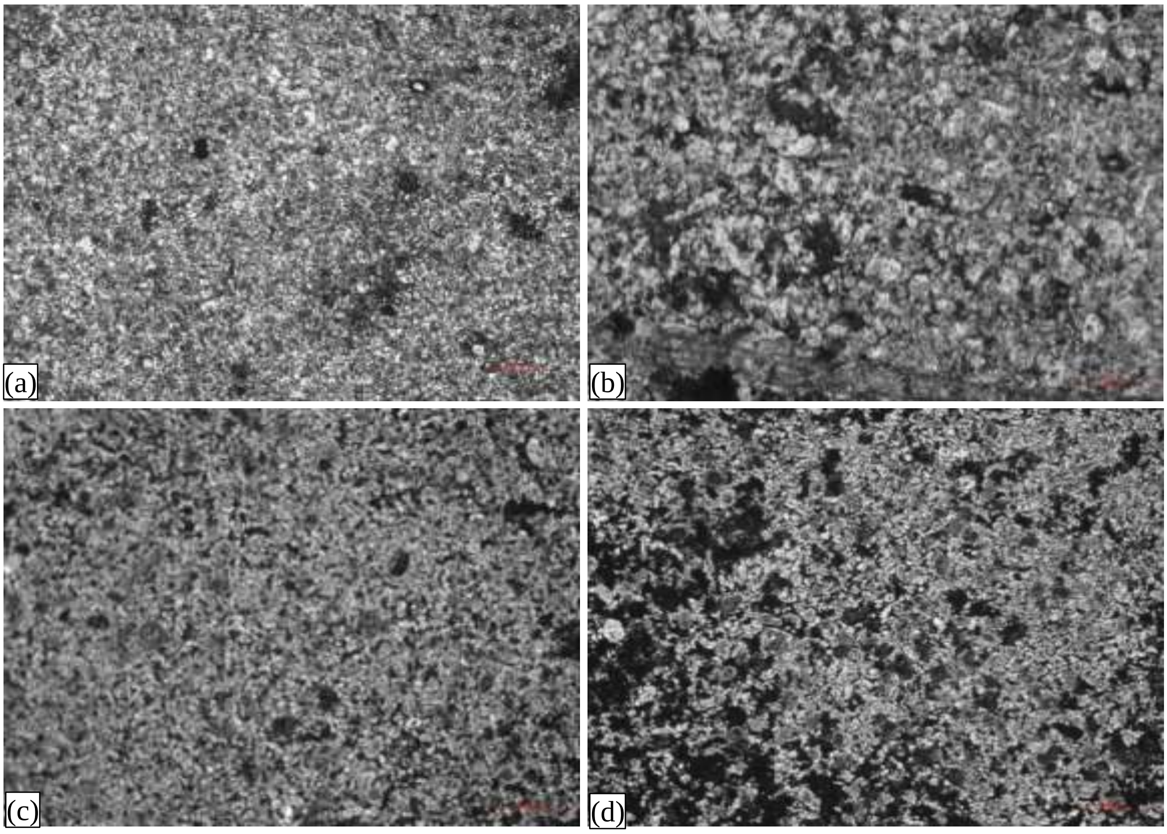

Figure 5. Aluminium- copper composites containing (a) 5%, (b) 10%, (c) 15% and (d) 20% copper.

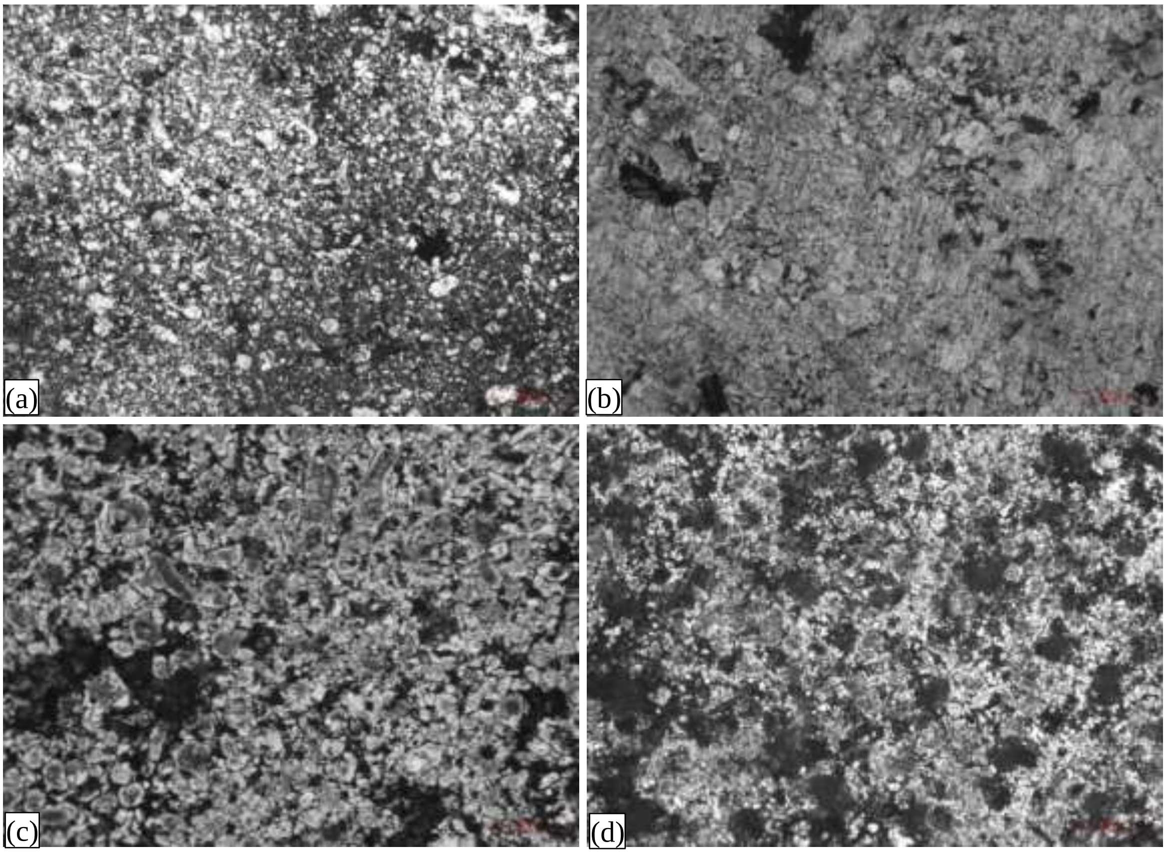

Figure 6. Extracted copper- aluminium composites containing (a) 5%, b)10%, (c) 15% and (d) 20% copper.

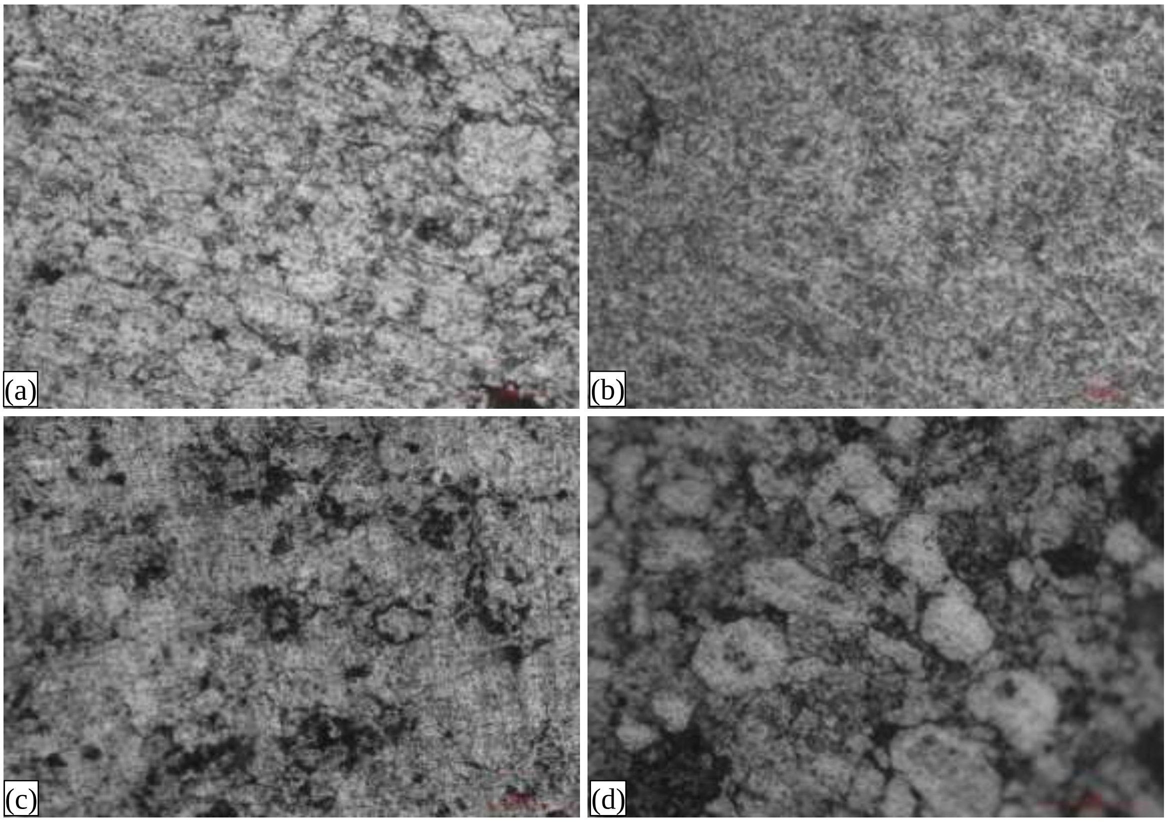

Figure 7. Aluminium- copper composites (after aging) containing (a) 5%, (b) 10%, (c) 15% and (d) 20% copper.

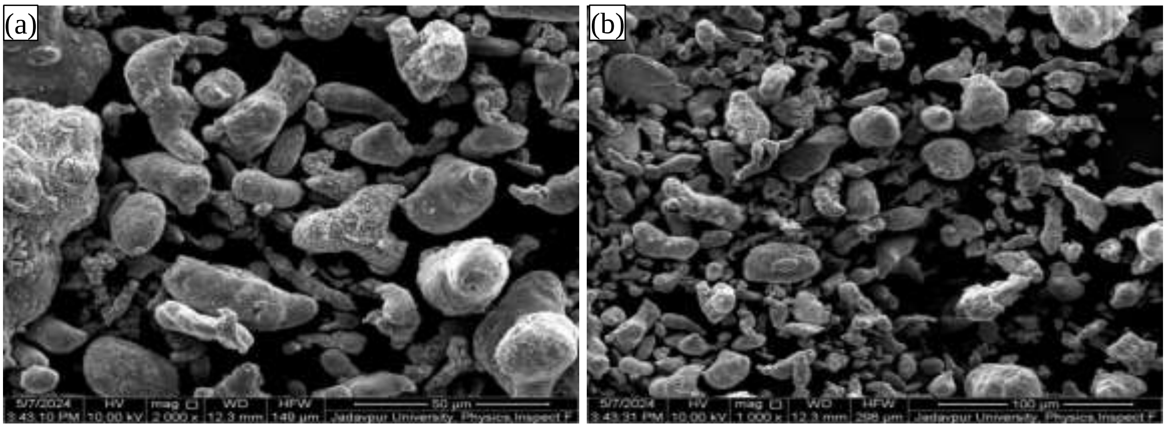

Figure 8. (a)–(b)Aluminium- copper composite at different magnifications under FESEM.

FESEM

The surface structure of the aluminum-copper composite under FESEM is shown in Figure 8. Figure 8 provides an overview of the entire structure of the aluminium- copper composite material, offering a comprehensive view of its intricate composition. In Figure 9, the copper powder is prominently displayed, showcasing the reduction of CuO to dextrose and highlighting the transformation process. Moving on to Figure 10, the cubic structure of these composite materials is clearly depicted, revealing the inherent structural integrity of the aluminium-5% copper composition.

Hardness

Aluminum-copper composite materials with varying weight percentages of copper were subjected to the Macro-Vickers hardness test and displayed in Table 2, Table 3 and Table 4. After examining the

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mocro-hardness, a distinct pattern became evident that hardness values were increased noticeably with reinforcements as shown in Figures 11–13.

Table 3. Micro-Vickers Hardness.

Composition (Aluminium % + Copper %)	Vickers Hardness Number (VHN)
Aluminium- 5% copper composite (95+5)	HV 65.83
Aluminium- 10% copper composite (90+10)	HV 72.43
Aluminium- 15% copper composite 85+15	HV 76.00
Aluminium- 20% copper composite (80+20)	HV 92.80

(b)

Table 4. Macro-Vickers Hardness (Method-II).

Composition (aluminium % + copper %)	Vickers hardness number (VHN) [before aged]	Vickers hardness number (VHN) [after aged]
Aluminium- 5% copper composite (95+5)	HV 30.62	HV 34.90
Aluminium- 10% copper composite (90+10)	HV 38.80	HV 52.20
Aluminium- 15% copper composite (85+15)	HV 41.71	HV 53.87
Aluminium- 20% copper composite (80+20)	HV 56.10	HV 55.28

Table 2. Macro -Vickers Hardness (Method-I).

Composition (aluminium % + copper %)	Vickers hardness number (VHN) [before aged]	Vickers hardness number (VHN) [After aged]
Aluminium- 5% copper composite (95+5)	HV 32.98	HV 34.86
Aluminium- 10% copper composite (90+10)	HV 46.05	HV 51.64
Aluminium- 15% copper composite (85+15)	HV 52.45	HV 53.39
Aluminium- 20% copper composite (80+20)	HV 59.52	HV 61.57

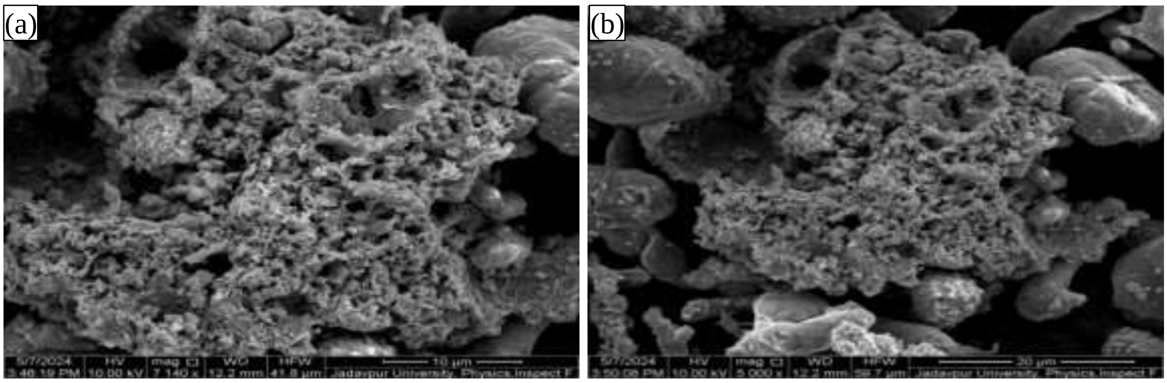

Figure 9. (a)–(b) Extracted copper structure at different magnifications under FESEM.

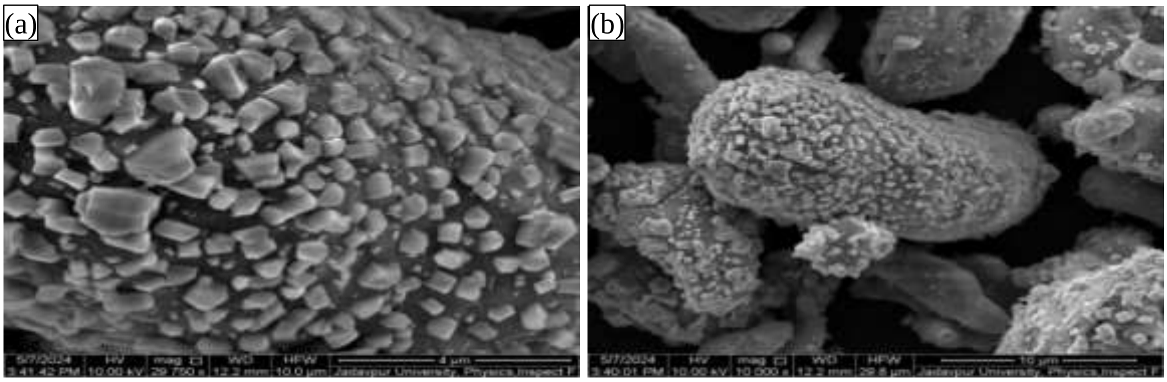

Figure 10. (a)–(b) Aluminium-copper composite (after aging) cubic structure at different magnifications under FESEM.

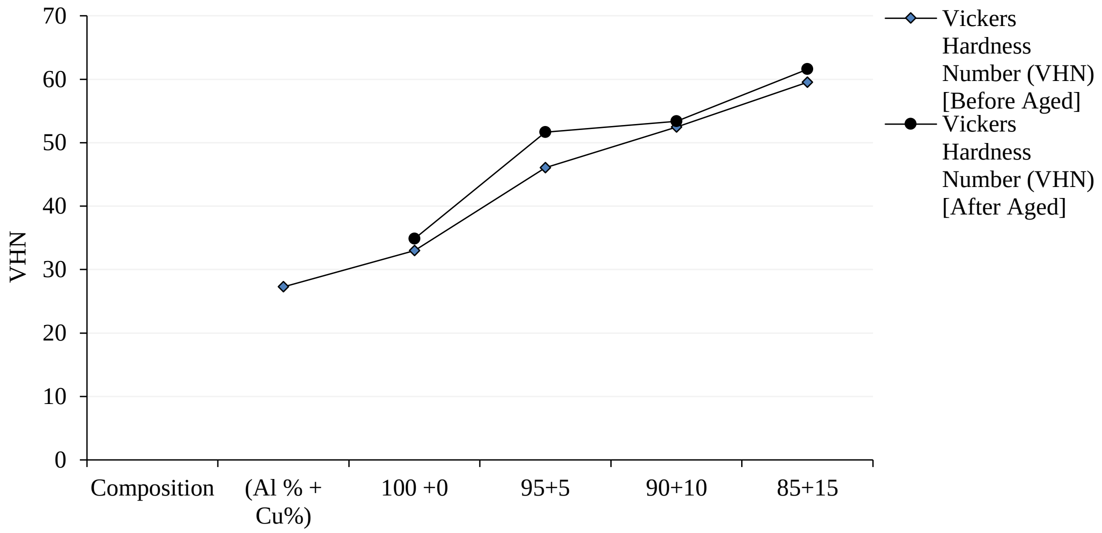

Figure 11. Macro-Vickers Hardness before and after aged samples plot (Method-I).

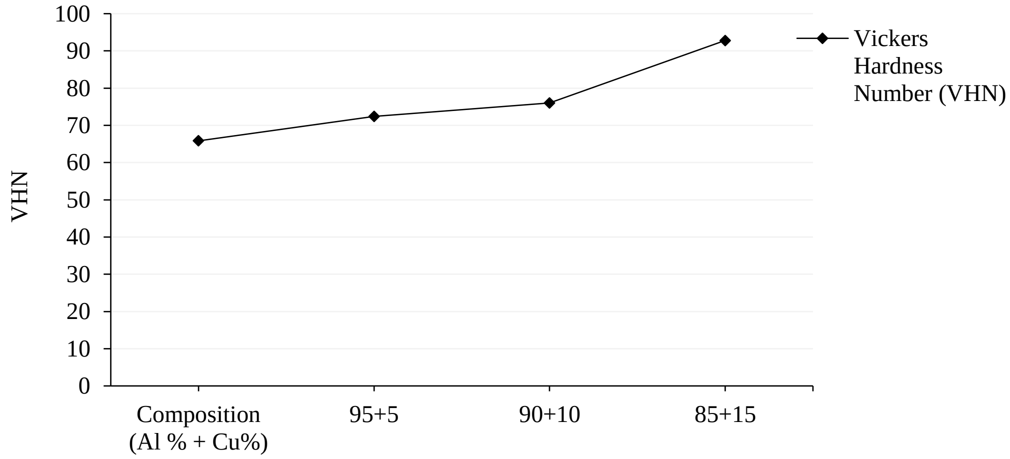

Figure 12. Vickers mocro-hardness for aluminium- copper composites.

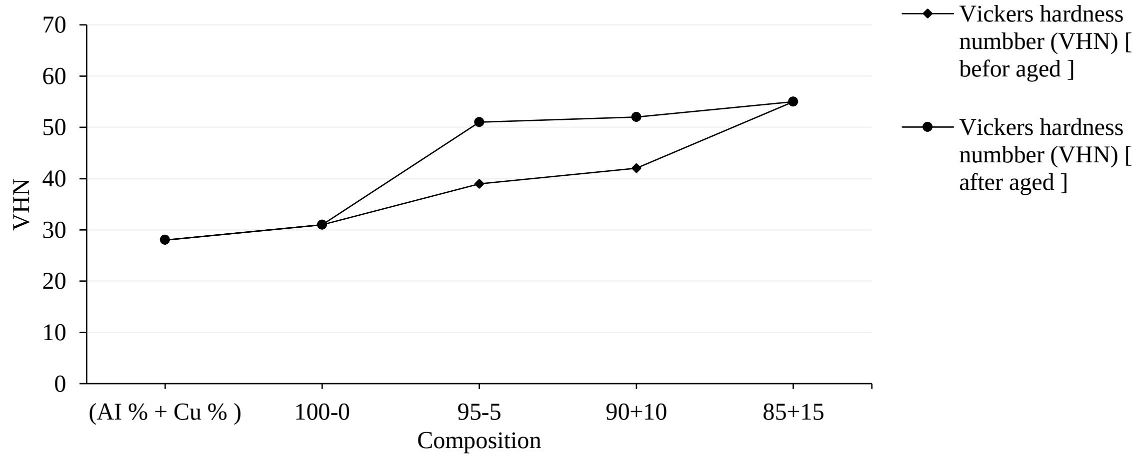

Figure 13. Vickers micro-hardness before and after aged samples (Method-II).

Upon completion of the aging process, the hardness values of all samples were once again measured using a macro-Vickers hardness tester machine. It was noted that as the percentages of the reinforcement were increased, there was a corresponding increase in all measured values.

Journal of Polymer & Composites
Volume 14, Special Issue 3
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Table 5. Various physical properties (fabricated through method-I).

Composition (Aluminium % + Copper %)	Density as per ROM Theory (gm/cm^3)	Compact Density (gm/cm^3)	Compact Porosity (%)	Sintered Density (gm/cm^3)	Sintered Porosity (%)
Pure Aluminium	2.700	2.200	2.72	2.360	2.52
Aluminium- 5% copper	3.013	2.602	2.78	2.891	2.56
Aluminium- 10% copper	3.326	2.801	3.74	3.102	2.72
Aluminium- 15% copper	3.639	3.204	3.96	3.404	3.86
Aluminium- 20% copper	3.952	3.503	4.62	3.703	3.94

Table 6. Various physical properties (fabricated through method-II).

Composition (Aluminium % + Copper %)	Compact Density (gm/cm^3)	Compact Porosity (%)	Sintered Density (gm/cm^3)	Sintered Porosity (%)
Pure Aluminium	2.200	2.72	2.36	2.52
Aluminium- 5% copper	2.390	8.6	2.93	3.41
Aluminium- 10% copper	2.502	9.16	2.707	8.95
Aluminium- 15% copper	2.710	11.68	2.915	9.87
Aluminium- 20% copper	3.009	14.77	3.201	10.77

Similarly, samples of aluminum-copper, where copper was obtained from reduction copper, were tested for hardness using the Macro-Vickers method at different weight percentages. The associated hardness values were displayed in the tables below. After analyzing the data, a clear pattern emerged, showing that in all sample compositions, hardness values rose markedly and rapidly as the quantity of reinforcement increased.

Upon completion of the aging process, the hardness values of all samples were once again measured using a macro-Vickers hardness tester machine. It was noted that as the percentages of the reinforcement were increased, there was a corresponding increase in all measured values.

Density and Apparent Porosity

Upon completion of the measurement process, the subsequent values have been listed in the Table 5. Through these experiments, it was seen that the reinforcement percentages, specifically the copper content, were systematically increased, and there was a corresponding slight rise observed in porosity values. Furthermore, density increased after the completion of blending, mixing, and compaction processes. However, upon the sintering of all samples, a notable shift occurred: density values experienced a further increase in post-sintering, while porosity values exhibited a slight decrease attributed to the sintering process.

Likewise, Table 5 and Table 6 also provide insights into the green and sintered density, as well as the porosity of all composite samples, subsequent to the measurement of these calculated values. It became evident that as the percentages of copper content increased, there was a corresponding rise observed in the values.

CONCLUSION

In this present study, a comprehensive investigation was conducted into the apparent density, porosity, microstructure, and hardness of aluminium- copper composite samples, varying the percentages of copper within the aluminium matrix. The development of the aluminium- copper composites were executed with precision across a spectrum of weight percentages, encompassing 5%, 10%, 15%, and 20% of copper content within the aluminium matrix. This was achieved adeptly at a temperature of 600°C using the powder metallurgy technique.

A meticulously planned investigation was undertaken to ascertain the reduction of copper oxide to dextrose within an inert atmosphere. This meticulous process involved the gradual introduction of nitrogen gas at an elevated temperature of approximately 650°C. Subsequently, the X-ray diffraction results were meticulously scrutinized to assess the state of the copper powder. A discernible enhancement in sintered density was observed with an incremental rise in sample composition. Conversely, the percentage porosity exhibited a notable surge, particularly with the rapid augmentation of copper reinforcement. An insightful comparison between the conventional technique and the reduction process highlighted discernible alterations in structural morphology, effectively demonstrated through optical microscopy.

The introduction of copper content was found to significantly augment the susceptibility of Vickers hardness. Remarkably, the highest hardness value was recorded in the aluminium- 20% copper composite sample. Additionally, in aged hardening on aluminium- copper composite samples, there was a rapid escalation in Vickers micro-hardness values.

Recognized for its lightweight properties, aluminum experiences a substantial enhancement in mechanical properties with the incorporation of copper. This enhancement translates into manifold advantages across diverse industries, notably in aerospace and automobile applications. These benefits extend to components such as leaf springs in suspension systems and pistons in select diesel engines, among myriad others.

AUTHOR CONTRIBUTIONS

The first author and the second author contributed equally to this work and share first authorship.

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