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Microstructural and mechanical behavior of unique aluminum matrix composites reinforced with pre-synthesized Al/Cu core-shell particulates

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Abstract

In this study, Al matrix composites (AMCs) reinforced with Al/Cu core-shell particles were synthesized by hot pressing. The Al/Cu particulate reinforcements were pre-synthesized by galvanic replacement. The effect of the volume fraction of reinforcement on the microstructure and mechanical behavior of the composites was investigated by scanning electron microscopy (SEM), X-ray diffraction (XRD), micro-hardness and compression tests. The strength of the composite becomes 2.25 times the matrix by using 20 vol.% core-shell reinforcements containing only 6 vol.% Cu. Further, with increasing volume fraction of Al/Cu reinforcement, the degree of in-situ transformation of Cu into intermetallic also increases. Consequently, a specially tailored microstructure can be obtained by controlling the volume fraction of the reinforcement and its interfacial reaction with the matrix, which can improve the mechanical strength of composites without considerably compromising on fracture strain. The findings of this work provide stronger implications for the industry to fabricate viable engineering structures.

Key words: core-shell powder, galvanic replacement plating, interfacial reaction, hot-pressing, composite

1. Introduction

Particulate reinforced aluminum matrix composites (PRAMCs) have great prospects for high-tech applications in transport, chemical, and aerospace industries because of their low density coupled with extraordinary mechanical properties and corrosion resistance [1–5]. The PRAMCs can be divided into two categories. In the first category, hard particles (ceramic: Al_2O_3, B_4C, SiC, CNTs, TiB_2, etc., and intermetallic: $AlFe_3$, AlFeCu, etc.) are embedded in the aluminum matrix as reinforcement to achieve extraordinary strength [6–17]. However, with the increase in reinforcement volume fraction, the ductility of the composites significantly decreases due to the brittle nature of reinforcement and weak interfacial bonding between reinforcement and matrix [18–20]. Researchers have reported the second category of PRAMCs, reinforced with pure metallic elements (Ni, Ti, Cu, and Fe, etc.) to obtain ductility with high strength [21–24]. However, in these composites, interfacial reactions result in the formation of solid solutions or brittle aluminides. These aluminides cannot be avoided even by using high-tech techniques such as selective laser melting, microwave sintering, and spark plasma sintering, etc., wherein the reinforcement/matrix reaction time is very short [25–28].

To take advantage of these intermetallic phases, researchers have developed core-shell reinforcements using these metallic particulates in AMCs to increase strength without considerably reducing ductility. Although brittle intermetallic shells in these compos-

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T a b l e 1. Copper deposition bath composition and process parameters

Chemical required	Concentration (mol L^{-1})
Copper Sulfate ($CuSO_4 \cdot 5H_2O$)	0.10
Ethylene diamine tetraacetic acid, disodium (EDTA-2Na.$2H_2O$)	0.2
Copper Chloride ($CuCl_2$)	0.005
Boric Acid (H_3BO_3)	0.5
Process Parameters	
pH	9.0
Temperature	55 °C
Time	15 min

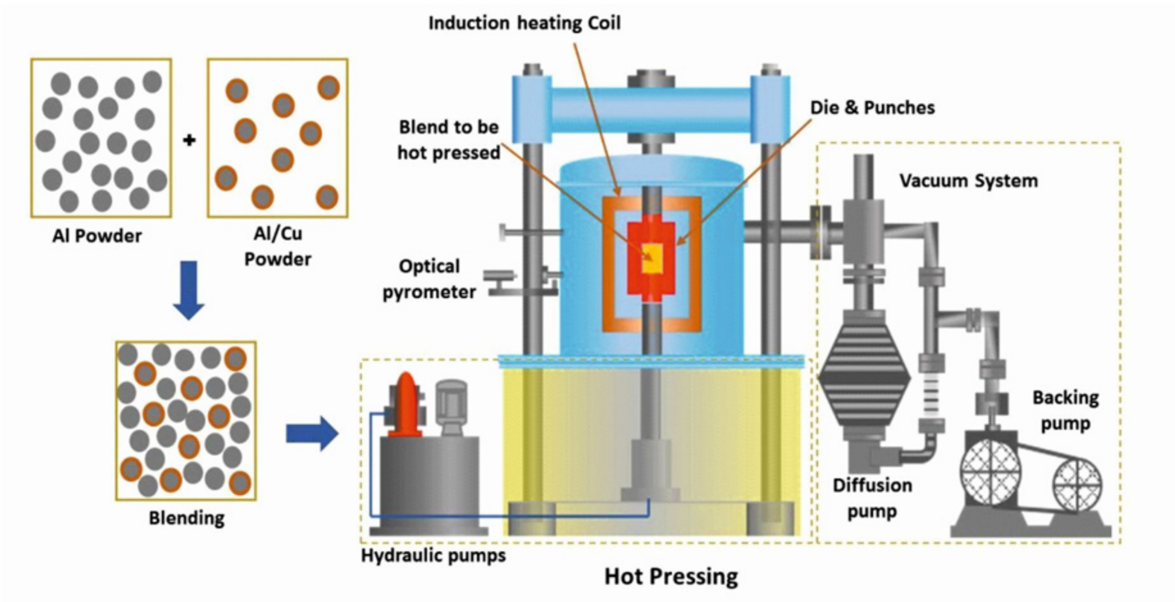

Fig. 1. Process flow for the synthesis of hot-pressed composites.

ites may lead to the formation of cracks during deformation, however, their inner ductile core can impede crack growth [21, 29, 30]. Thus core-shell structures provide a new idea to overcome the strength ductility trade-off in AMCs. Among metallic particulates, copper is an attractive choice because of its ability to produce strengthening through the solid solution as well as secondary phase(s) formation in aluminum under normal equilibrium conditions. The aluminum-copper composites, prepared via the powder metallurgy route, find several applications in aircraft structures, hardware, truck wheel rivets, and screw-machine products, etc. [31]. However, limited literature is available on this system and processing routes.

Considering the abovementioned advantages of copper-based core-shell intermetallic, pre-synthesized Al/Cu core-shell composite powder was used as reinforcement in this study. This powder was synthesized by plating Cu on Al particles via the galvanic replacement method using lab-optimized parameters [32]. The ambition of the current study was to explore the effect of reinforcement volume fraction on the mechanical behavior of AMCs with novel microstructure containing Al/Cu core-shell particles.

2. Materials and methods

Initially, the copper shell was deposited on aluminum powder particles (purity > 99.5 %, average grain size D_{50}: 10 µm) by the galvanic replacement method. In this process, 2.0 g of aluminum powder was etched for 120 s in a 50 ml pretreatment solution (45 ml distilled water and 5 ml of 33 % ammonia solution) at room temperature. Afterward, the suspension was directly poured into a pre-heated copper deposition bath [32]. The bath composition, along with process parameters used for copper deposition, is summarized in Table 1. After deposition, Al/Cu core-shell powder was filtered, rinsed with hot distilled water,

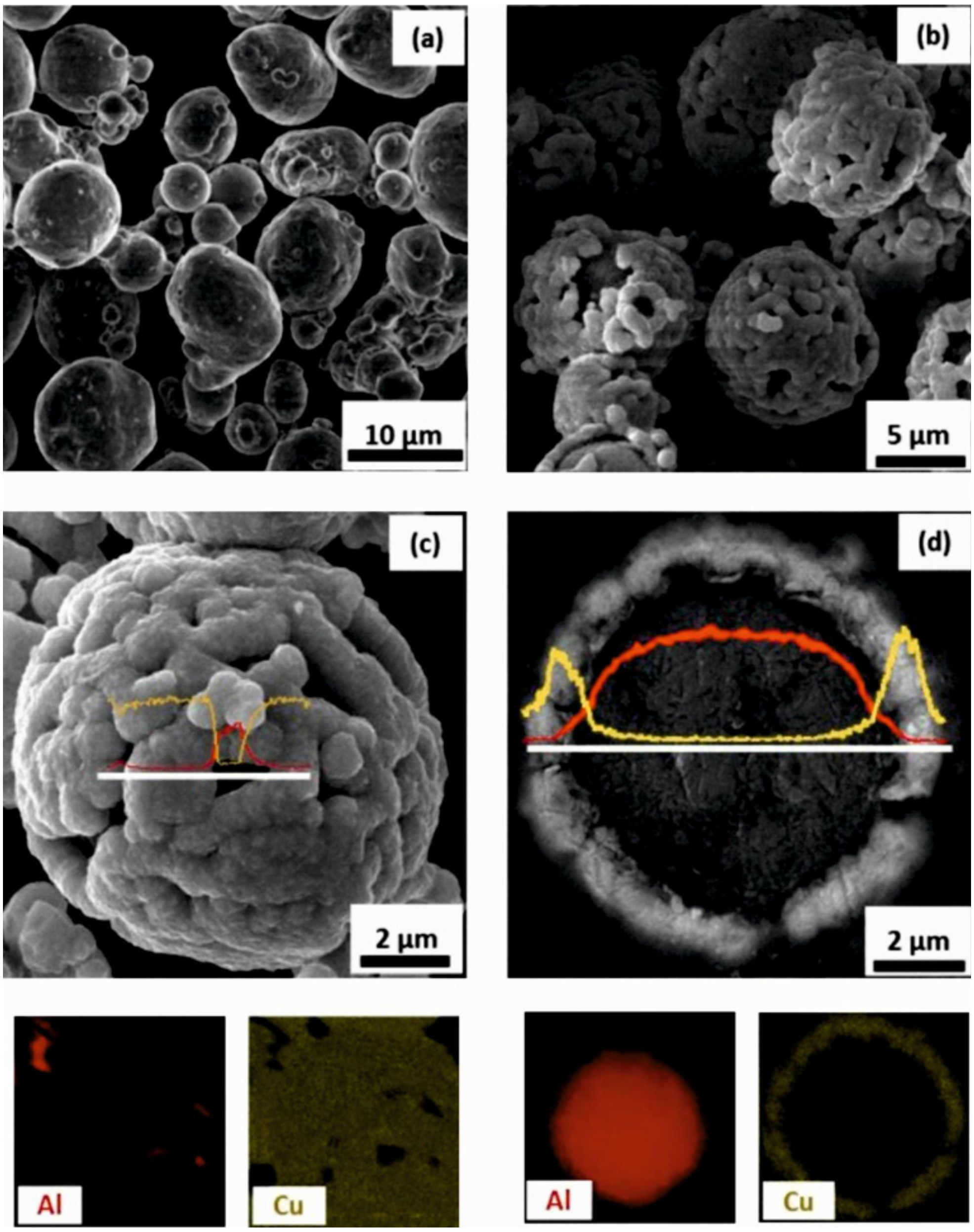

Fig. 2. SEM/ EDX results of: (a) aluminum, (b) pre-synthesized Al/Cu core-shell powder, (c) EDX surface area mapping with line scan of single grain, and (d) its cross-section.

and dried for 8 hours at 80 ℃. The density of this synthesized powder was measured with a helium gas pycnometer (AccuPyc-II 1340), which was later used for calculating the volume fraction of the reinforcement in AMCs. Afterward, aluminum matrix (consisting of the same aluminum powder used for deposition) and Al/Cu reinforcement (with volume fraction of 5, 10, and 20 vol.%) powders were blended using a powder mixer, rotated at 60 rpm for four hours to achieve homogeneous distribution. The powder blends were hot-pressed uniaxially at 640 MPa and 460 ℃ in a die (ø 10 mm) under an argon atmosphere for 20 min. The process flow is shown in Fig. 1. From each compacted cylinder (ø 10 mm, 10 mm height), three compression test specimens (ø 3 mm, 6 mm height) were machined by using electrode discharge machining (wire cut). The room temperature compression tests were accomplished, using HOYTOM HM-D 50 KN machine at a constant strain rate ($10^{-4}\,s^{-1}$), according to ASTM E9-09. The Vickers micro-hardness (Tinius Olsen FH 14, China) of the composites was measured at a load of 300 g for 15 s. Phase identification in Al/Cu core-shell powders and hot-pressed composites was carried out by XRD (Philips PW 1050 Bragg-Brentano diffractometer) having Cu Kα radiation (0.154 nm) source. The microstructural changes were characterized by SEM (Tescan MAIA3 FE-SEM) equipped with an energy dispersive X-ray spectrometer (EDX).

3. Results and discussion

3.1. Characterization of pre-synthesized core-shell Al/Cu powder

After pretreatment (etching), the smooth surface of aluminum particles became rough as reported in our previous work [33]. These roughened particles were immersed in the deposition bath for a particular time. The particle surface was covered by a uniformly dense and adherent layer of nano-sized copper grains. This copper layer (0.8–0.9 μm) was verified by surface area mapping and line scanning of the individual particle and its cross-section using an EDX (Figs. 2c, d). The Al/Cu composite powder was further characterized by XRD (Fig. 3a). The XRD results show that the visible characteristic peaks only belong to FCC copper and FCC aluminum structure in synthesized Al/Cu core-shell powder. Moreover, the measured density (5.5 $g\,cm^{-3}$) was used to calculate the deposited copper content (44.7 wt.%) on aluminum particles and, later, the volume fraction of reinforcement in the hot-pressed composites. Applying Scherrer's formula on the copper peak (Fig. 3a) gives the average crystallite size to be ∼ 25 nm.

3.2. Characterization of phases and microstructure in the composites

The XRD patterns of the hot-pressed AMCs reinforced with 5, 10, and 20 vol. % of Al/Cu core-shell powder (named 5 Cu, 10 Cu, and 20 Cu, respectively) are shown in Fig. 3b. Different studies show that five intermetallic phases (Al_2Cu, AlCu, Al_3Cu_4, Al_2Cu_3, and Al_9Cu_4) are possible in an Al and Cu diffusion couple [32, 34]. However, formation energies and growth constants (thermodynamics and kinetics) of the phases decide their formation [35, 36]. Figure 3b represents that all three hot-pressed composites have only two intermetallic phases (i.e., Al_2Cu and Al_9Cu_4) in addition to elemental Al and Cu. The additional intermetallics that emerge in the composites during hot pressing are due to in-situ interfacial reactions between aluminum and copper. The presence of Al_2Cu and Al_4Cu_9 phases is in good agreement with formerly reported work [36]. The diffraction patterns indicate that the peak intensities of Al_2Cu and Al_4Cu_9 increase, while the height of Al diffraction peaks (at 2θ angles values 38.4°, 44.8°, 65.1°, 78.3°, 82.3°, 99.4°, 111.8°, and 115.6°) decreases with increasing Al/Cu reinforcement volume fraction from 5 to 20 %. Cu has a lower atomic diffusion flux in Al as compared to Al in Cu [37–39]. Therefore, the formation of an Al-rich intermetallic phase (Al_2Cu) is anticipated due to the higher diffusional flux of Al atoms into the Cu shell. Hence, the prominent diffraction peaks in the XRD pattern are associated with the Al-rich Al_2Cu phase when compared to those of the Cu-rich Al_4Cu_9 phase [39]. From the relative intensities of the diffraction peaks of different phases, the quantity of these phases was estimated (as shown in Fig. 3c). It is quite clear in Fig. 3c that, with increasing reinforcement volume fraction, the amount of unreacted Cu, Al_2Cu, and Al_4Cu_9 phases increases.

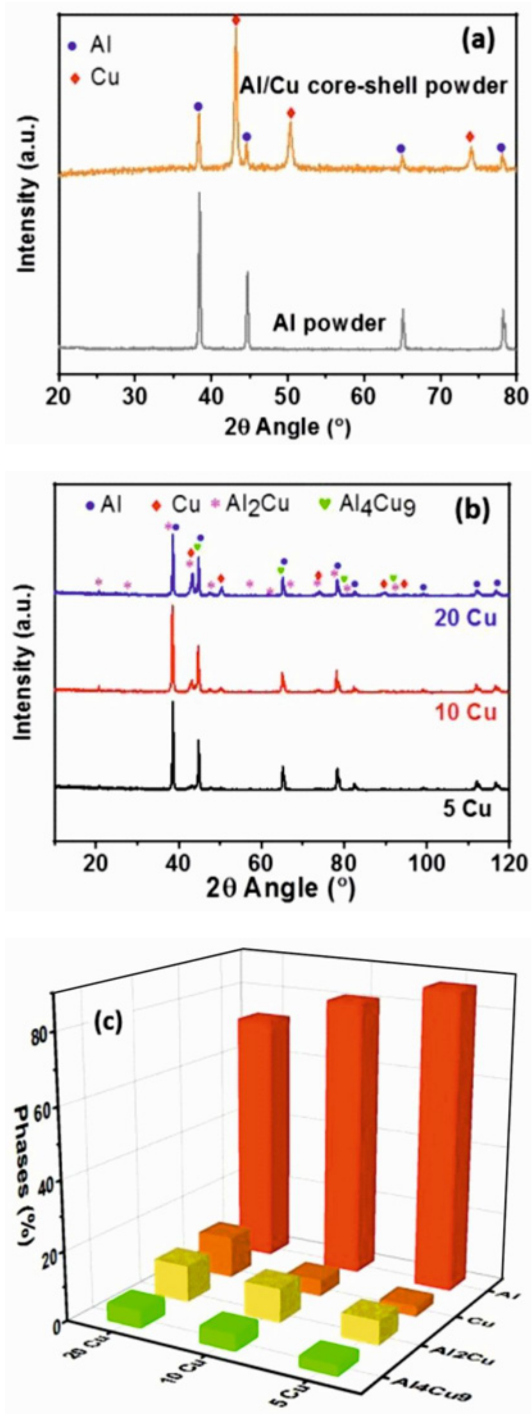

Fig. 3. XRD results of: (a) pre-synthesized Al/Cu core-shell powder, (b) hot-pressed composites, and (c) phase quantification.

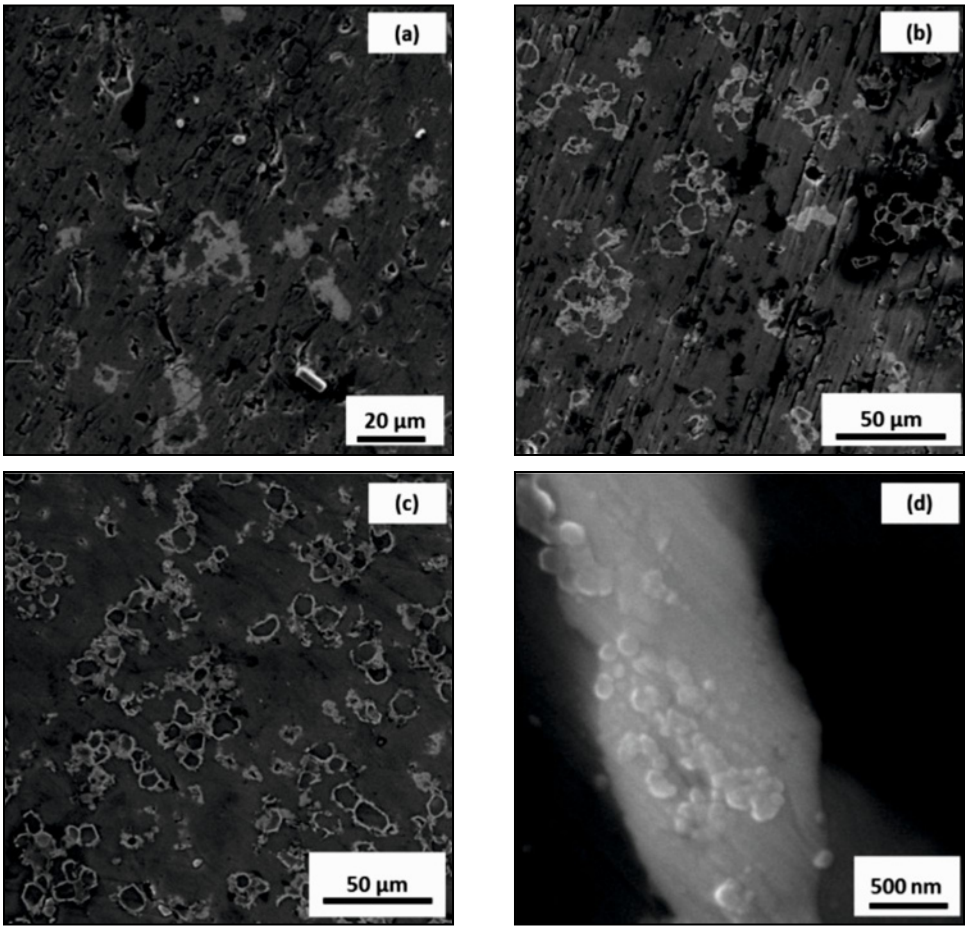

Fig. 4. SEM micrographs of composites reinforced with Al/Cu core-shell particles: (a) 5 vol.%, (b) 10 vol.%, (c) 20 vol.%, and (d) higher magnification image of (c).

Figure 4 shows SEM micrographs of all hot-pressed composites. The micrographs show the distribution of small agglomerates of Al/Cu core-shell reinforcement particles in the matrix. The contrast in micrographs denotes the chemical contrast, i.e., the lighter elements appear in dark contrast, like aluminum in the matrix and the core of reinforcing particle, while bright contrast represents the heavier elements, such as copper or copper-containing phases around the Al core. It is worth mentioning that intermetallic phase formation around the Cu shell is non-uniform, i.e., some portions of the Cu shell react more proactively with the aluminum matrix and form new phases. This is possibly due to the presence of higher diffusion paths available in the hot-pressed microstructure. Overall, the interfacial reaction does not lead to the complete transformation of the Cu shell into an intermetallic, and its shape remains intact. The higher magnification SEM micrograph (Fig. 4d) shows good bonding of Al-Cu due to diffusion during hot-pressing.

3.3. Physical and mechanical behavior of the composites

Figure 5a shows the variation in the density of the composite with increasing the volume fraction of reinforcement (Al/Cu). The initial and final densities were measured by the rule of mixture and pycnometer, respectively. The density increases from 2.79 to $3.17\,g\,cm^{-3}$ with increasing the reinforcement fraction from 5 to 20 vol.% due to the replacement of a lighter aluminum matrix with denser reinforcing particulates. Moreover, the relative density (densification) of the synthesized composites decreases from 98.34 to 97.10 % with the addition of reinforcement. The minimum relative density (97.10 %) was observed in the composite containing 20 vol.% of reinforcement. This decline in densification with increasing the fraction of reinforcement can be attributed to (1) increment of the fraction of oxide layer on aluminum or reinforcing particulates, which may hinder the sintering ability in solid-state, (2) the diffusion coefficient of Al in Cu is greater than Cu in Al, which may result in porosity formation at interfaces due to Kirkendall's

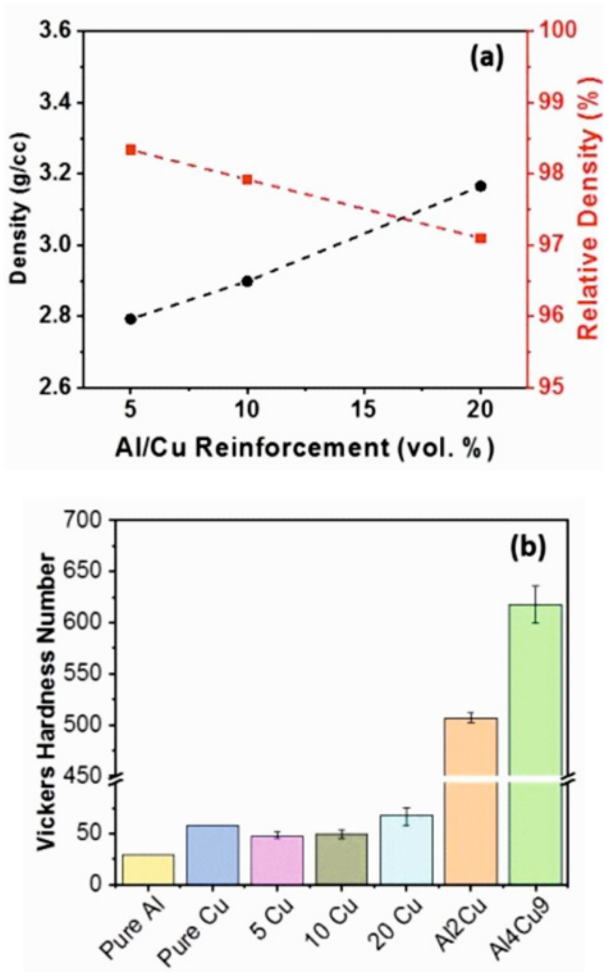

Fig. 5. Effect of Al/Cu reinforcement on: (a) density, relative density, and (b) the micro-hardness of composites.

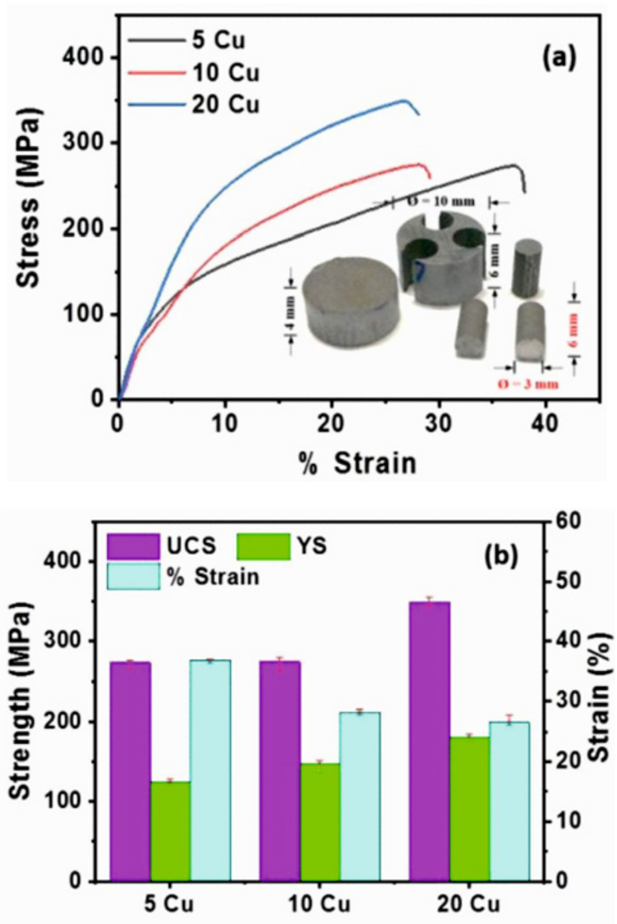

Fig. 6. Compressive stress-strain curves of: (a) composites reinforced with 5, 10, and 20 vol.% of pre-synthesized Al/Cu core-shell powder with sample cutting scheme given inset and (b) summary of compression tests results.

effect, (3) volumetric changes due to the formation of intermetallic phases, (4) higher fraction of harder phase (copper network) decreases the compressibility of powder blend, and (5) increase in the probability of agglomeration of reinforcing particles [40].

The mechanical behavior of the hot-pressed composite was assessed by micro-hardness and compression tests. The Vickers micro-hardness results show that with an increasing fraction of Al/Cu particulates, the micro-hardness of composites has significantly improved from 47.4 to 67.8 $HV_{0.3}$ (Fig. 5b). For comparison, the hardness values of Al, Cu (Al_2Cu and Al_4Cu_9) are also indicated in Fig. 5b [36, 41]. Hardness is an ability of a material to resist localized deformation. Thus, harder reinforcing particles provide an obstacle to dislocations during plastic deformation [42, 43]. This increasing trend in hardness by increasing volume fraction of reinforcement is due to: (1) the replacement of a softer matrix with harder reinforcing particulates, (2) the formation of hard intermetallic phases (Al_2Cu and Al_4Cu_9) during hot pressing as evident in XRD (Fig. 3).

The room temperature compression test results of the composites are presented in Figs. 6a,b. The results show that the compressive behavior of hot-pressed aluminum matrix composites is significantly influenced by increasing the fraction of Al/Cu core-shell reinforcing particulates. By increasing reinforcement content from 5 to 20 vol.%, the compressive strength of the composites increases from 274 to 349 MPa, and the fracture strain decreases from 37 to 27 %. The improvement in compressive strength of the composites can be attributed to (1) high volume fraction of reinforcement, i.e., high load-bearing capacity of reinforcing particles, (2) high volume fraction of intermetallic phase formation due to in-situ interfacial reaction, (3) large number of anticipated dislocations formation in the proximity of reinforcing shells, (5) intermetallic formation around the ductile core for suppression of crack nucleation, and (6) decreasing average matrix ligament size in between reinforcement particles [41].

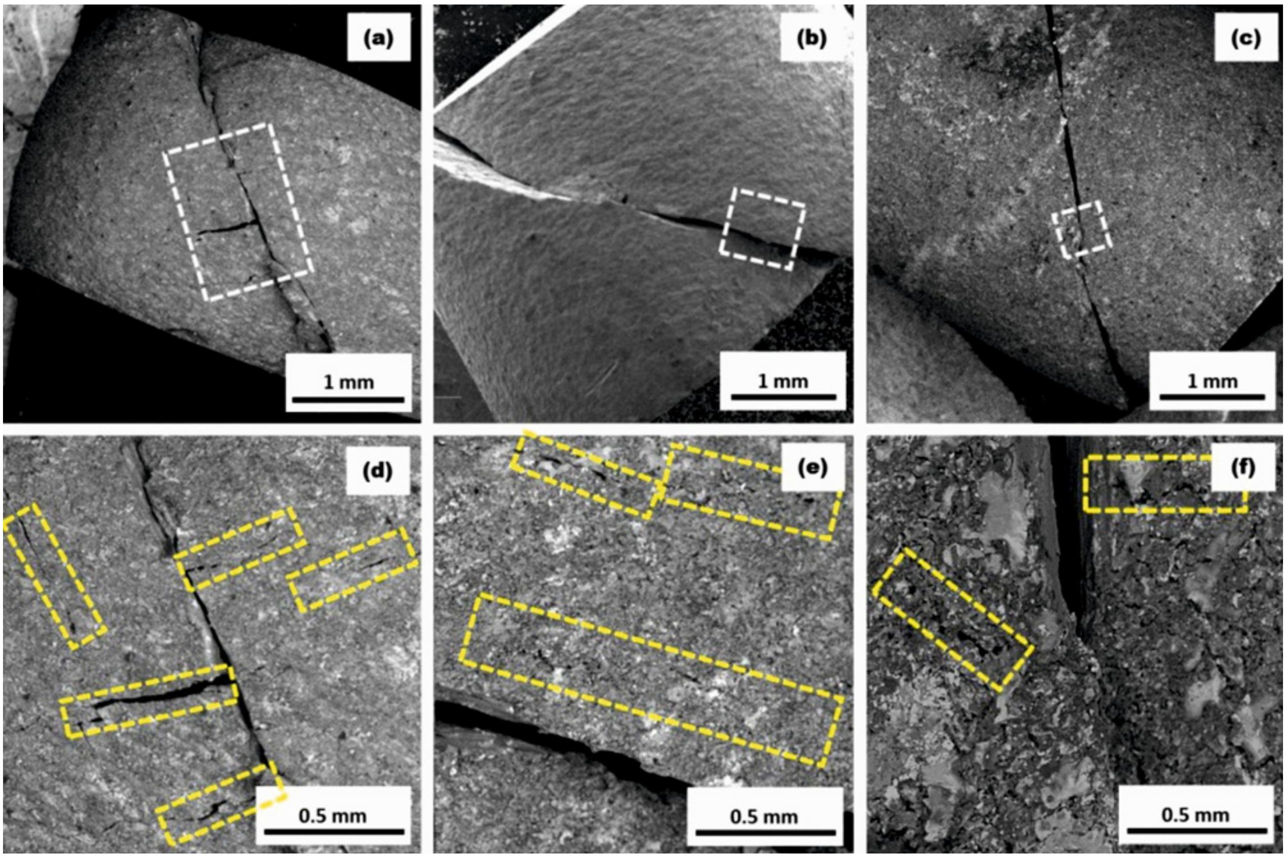

Fig. 7. SEM micrographs of lateral side of fractured composite samples containing: (a) 5, (b) 10, (c) 20 vol.% of pre-synthesized Al/Cu core-shell reinforcement particles, (d, e, f) magnified images of white dotted rectangular areas in (a, b, c), respectively.

However, the reduction in fracture strain with increasing volume fraction of reinforcement is due to the presence of a high fraction of brittle intermetallic phases in the composites accompanied by low densification.

Since strong interfacial bonding between matrix and reinforcement provides an obstacle for crack initiation during barreling, therefore, high compressive strength can be considered as an indication of good bonding among the composite constituents. The compressive strength and fracture strain of hot-pressed pure Al were reported to be 155 MPa and 46 %, respectively [44]. The results of this study validate the positive effects of the core-shell reinforcement on the mechanical behavior of AMCs due to the modification in its microstructure. Superior interfacial bonding in the composites facilitates efficient load transfer from matrix to reinforcements.

Further, the lateral sides of the fractured samples, after the compression test, were analyzed using SEM (Figs. 7a–f). From these images, it can be observed that along the main shear crack, a few parallel fine shear bands and branched cracks are visible (as highlighted by yellow rectangles). The parallel shear bands/cracks provide accommodation for large plasticity in the composites. The composite, containing 5 vol.% of Al/Cu core-shell particles, has maximum fractured strain. Therefore, much clearer and wider shear bands are visible in the composite, parallel to the main crack (Fig. 7d), in comparison to the other composites synthesized in this study. The branching of cracks and deflection of crack paths are responsible for toughness in the other composites.

4. Summary and conclusions

In the current study, aluminum-based matrix composites (AMCs) reinforced with different volume fractions of pre-synthesized Al/Cu core-shell powder particles were successfully prepared by hot pressing. The summary and conclusions of this study are as follows:

– Copper coating (< 1 μm thickness) was deposited on Al particles by galvanic replacement method to synthesize Al/Cu core-shell powder.

– The XRD phase analysis of hot-pressed composites with Al/Cu reinforcements revealed the formation of Al_2Cu and Al_4Cu_9 phases due to interfacial reaction.

– The degree of interfacial reaction increases with increasing volume fraction of Al/Cu reinforcement.

– The relative density of the composites decreased with increasing the degree of interfacial reaction, which resulted in a higher volume of intermetallic phases.

– The compressive strength of the prepared composites was increased up to 349 MPa with a strain of 27 %.

– The findings of this work provide strong implications for making viable engineering structures using AMCs reinforced with Al/Cu core-shell powder.

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