Review Article

Influence of sintering methods on the mechanical properties of aluminium nanocomposites reinforced with carbonaceous compounds: A review

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\textbf{A B S T R A C T}

This paper succinctly reviews from the corpus of literature the reinforcement of aluminium with carbonaceous nanocompounds (CNTs, GNFs, graphenes etc.), with particular emphasis on the strength and ductility of the resulting composites based on the utilized fabrication routes. Owing to the unique intrinsic properties of these carbonaceous nanocompounds, they have been widely reported as ideal reinforcement materials for aluminium and its compounds. Significant amount of work has been published on the use of solid-state sintering as a novel route for the incorporation of these nanocompounds in aluminium matrices. This paper therefore aims to review some relevant aspects of the fabrication processes of these aluminium based composites such as (i) effects of the sintering routes and parameters on the resulting properties of the composites; (ii) the effect of dispersion techniques on the resulting properties of the composites; (iii) the strength-ductility trade-off in the reinforced composites; and (iv) the intermetallics formed between the carbonaceous nanocompounds and aluminium.

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1. Introduction

Recent technological demands require a wide (and sometimes conflicting) range of properties unachievable in single traditional materials. This birthed the evolution of reinforcing traditional materials with various elements such that the resulting composite material would benefit from the synergistic effect of both the reinforcement and the matrix. The rudimentary principles of composite materials dates back in time, from vintage buildings with straw reinforced clay to present day structures using reinforced concrete [1]. This makes it possible for the different constituents to work in synergy in such a complementary manner as to subdue the limitations of each constituent [2]. Aluminium and its alloys
have been extensively investigated owing to their light weight [3], high specific strength [4], good corrosion resistance [5], good thermal conductivity [6], low electrical resistivity [7], high damping capacity [8] and low cost [9]. These unique properties make them ideal choices for automotive, aerospace and marine industries [10]. However, despite these excellent properties, the low strength of aluminium restricts its use especially for structural applications [11].

To overcome the strength limitation of aluminium and its alloys, the addition of silicon carbide [12], alumina [13,14], aluminium nitride [15], tungsten carbide [16], silicon nitride [17], titanium carbide [18], silica [19], boron carbide [20,21], titanium dioxide [22], graphite [23–25], zirconium boride [26], tantalum carbide [27] have been reported. Superior properties of these materials such as refractoriness, high hardness, high compressive strength and wear resistance, make them appropriate reinforcements in aluminium matrices [28–30]. The high cost of some of these ceramics, especially in developed countries, led to the incorporation of industrial and agro waste [31] such as flyash, coconut ash, bagasse ash, rice husk ash [32–34], that have shown huge promise as alternative reinforcements. Though they seem to possess inferior properties compared to the reinforcements offered by conventional ceramics [35,36] they have been successfully used as hybrid reinforcements alongside conventional ceramics.

2. Carbonaceous compounds

Recent advances in nanotechnology have emerged with numerous novel nanomaterials possessing extraordinary properties. Owing to the success of carbon nanotubes (CNTs) additions in polymer and ceramic matrices [37], CNTs and other carbon allotropes have been regarded as ideal reinforcements for aluminium matrices and recent research on this has grown enormously. Efforts have been largely focused on investigating their contribution to the enhancement of the mechanical performance of the composites. The excellent strength, wear properties and thermal conductivities of CNTs, graphene nanoplatelets, graphene oxides, graphene nanofibers and graphene nanosheets are obvious reasons for the incorporation of these nanomaterials in different matrices [38,39].

Carbon based reinforcements are ideal reinforcement candidates due to the remarkable combination of their properties. Carbon reinforcements present morphologically and dimensionally in different reinforcing phases – carbon nanotubes (CNT) (1D), graphene (Gr) (2D), and carbon fibre (3D) [40]. These graphitic structured materials have recently been among the more widely researched materials [41], due to their exceptional mechanical [42], thermal, [43–45], electrical [46] and tribological behaviours [41]. Their versatility extends to multidisciplinary applications like energy conversions, super capacitors and photocatalysis [47]. Moreover, improved manufacturing techniques have made these materials more economically friendly [48].

With Young’s modulus of ~1 TPa, tensile strength of ~130 GPa and thermal conductivity of ~3000 W/M/K [49], these carbon allotropes make excellent reinforcements in composites [50]. Furthermore, these materials have the added advantage of undergoing plastic deformation under severe bending stress conditions without failing prematurely [51].

Discovery of exceptional materials are, however, not sufficient in itself, as appropriate processing routes also have to be developed to incorporate these ‘super’ reinforcements in various matrices. Fabrication processes may be mostly categorized into solid and liquid state processes [52]. Some solid state fabrication methods include: diffusion bonding [53], electroplating [54], spray deposition [55], immersion plating [56], chemical vapour deposition [57], physical vapour deposition [58]. While some of the liquid state processes are: stir casting [59] squeeze casting [60], compo-casting [61], pressureless infiltration [62], ultrasound assisted solidification [63], vortex process [64], sol-gel synthesis [65], laser deposition [66] and powder metallurgy [67].

3. Powder metallurgy (PM)

Powder metallurgy can be described simply as the manufacturing of components from powder materials. The process starts with powder mixing and or milling, followed by compaction and sintering of the compacted powders to achieve the least possible porosities or the highest possible density. PM through solid-state sintering is particularly suitable for composites fabrication due to its flexibility to synthesize a wide variety of compositions and to produce intricate shapes with near net accuracy [68]. This simplicity places it clearly ahead of many other manufacturing processes with more complicated processes. In literature, a considerable amount of work has been devoted to producing aluminium matrix composites (AMCs) by using powder metallurgy [41,69]. In addition to the low temperatures employed, the solid-state mixing techniques lend themselves easily to the incorporation of these nano reinforcements in metal matrices [3]. Moreover, the interaction between matrix and reinforcements can be avoided because of lower processing temperatures usually associated with PM methods [70]. PM produces a better interface and more uniform distribution of reinforcement materials within the matrix, leading to improved mechanical properties [9]. It also simplifies the production of complex engineering components [71] and provides excellent control over microstructure, including size, morphology and volume fraction of matrix and reinforcement [72,73]. PM routes have proven their mettle as they offer several advantages over traditional casting processes, hence they have been the preferred route for most researchers especially for the incorporation of these carbon allotropes [3,74–76].

3.1. Sintering techniques

In simple terms, sintering can be described as the application of heat and pressure. The major focus during sintering processes is to achieve full or maximum densification, hence sintering parameters like temperature, pressure and holding time are varied till this is achieved. Extensive efforts have been carried out on the development of sintering methods for full densification of bulk composites. Since the pores in the material significantly affect the mechanical properties, the reduction of porosity can and will improve the performances
of the resulting composites [77]. Owing to variations on the sintering techniques available, differing properties are subsequently imparted in the resulting bulk composites.

3.1.1. Conventional sintering
This is the simplest and most traditional method for producing metal matrix CNT composite compacts. Conventional sintering was one of the earliest techniques utilized to synthesize metal composites, but limited success was achieved due to the poor densities of the resulting structures. In cases where nanomaterials like CNTs are incorporated into the metal matrices, post-sintering operations like hot pressing or extrusion of the product might be necessary to enhance densification. In the conventional heating method, slow heating rates and long dwell times during a sintering cycle usually result in exaggerated grain growth phenomenon [9]. In this method, the specimen is heated using heating elements like silicon rods and the heat is then transferred to the specimen either by conduction, convection or radiation. This poses the risk of non-uniform heating and creation of thermal gradients, which in turn results in internal stresses in the specimen. This is because part of the volumetric heat energy is lost by dissipation from surfaces, leaving the centre of the specimen slightly hotter than the surfaces [78].

In Gao et al. [79] graphene oxide and aluminium powders were sintered at 600 °C for 1 h under a pressure of 25 MPa to prepare Gr/Al composites. At 0.3 wt% CNT, the ultimate tensile strength (UTS) of the composite reaches the maximum, which is about 30% higher than that of pure Al prepared with the same process. The remarkable strengthening was attributed to the homogeneous dispersion of graphene in Al matrix. The enhanced tensile properties of the Gr/Al composites were explained by the efficient load transfer of graphene in Al matrix. During electrostatic self-assembly, the large sizes of the graphene are protected, so the contact areas between graphene and Al matrix were enhanced, resulting in the increase of load transfer sites.

3.1.2. Microwave sintering
Microwave sintering with volumetric heating mechanism offers a number of advantages over conventional sintering. These advantages include enhanced diffusion process, high heating rates, shorter processing times, and improvement of microstructures. Microwave sintering utilizes only a fraction of the time required in conventional sintering. In microwave heating, the rate of grain growth is reduced, due to a decrease in sintering time. Therefore, samples sintered in a microwave process obtain a uniform microstructure with smaller grains and consequently, a higher mechanical strength as compared to conventional sintering [80].

Initially there were misconceptions that metals reflect microwaves, resulting in sparks, and this constrained the use of microwave sintering to ceramics and their composites [78]. However, it was shown by Walkiewicz et al. [81] that the sparking phenomena applied to only bulk metals. In powder form, metals tend to absorb microwaves and can thus be heated significantly [82].

Saheb [83] compared microwave sintering to spark plasma sintering (SPS) of aluminium alloys Al 2124 and 6061 at similar sintering temperatures and holding times. Microstructures obtained from the SPSed samples showed that increase of sintering temperature from 450 to 500 °C improved the densification, and dramatically reduced the size and the number of pores. Higher sintering temperatures to 550 °C did not show any further improvement on densification. The complete densification by total elimination of pores however, was not achieved in the microwave sintered alloys as achieved in the SPSed samples as can be seen from Figs. 1 and 2. Apparently, obtaining similar results using microwave energy even under similar processing conditions was not possible, due to the differing mechanisms of sintering. For spark plasma and microwave sintering, increasing the sintering temperature from 400 to 450 °C increases the microhardness from 56.81 to 66.31 HV and 40.23 to 45.75 HV, respectively, while a further increase of temperature to 500 °C increases the microhardness to 70.16 HV for spark plasma sintering and decreases the microhardness to 32.27 HV for microwave sintering. The same trend was observed for the Al 2124. The increase of microhardness with the increase of sintering temperature followed the same trend as the increase of density. This was due to the elimination of pores during sintering and possibly precipitation of fine particles [84]. The observed decrease in microhardness with further increase in sintering temperature to 500 °C for the microwave sintering was however believed to be as a result of grain growth of the α-aluminium phase.

3.1.3. Hot pressing (HP) technique
In hot pressing, powder mixtures or the pressed compact are subjected to high temperature while being pressed in a die. The applied temperature and pressure result in easy deformation through creep and material transfer, and these help in achieving high densities. The heating could be done by electrical resistance heating, induction heating, or radiation. The holding time is critical since grain coarsening would occur during long heating periods. A hot press can also be used for near net shape manufacturing of components by low-pressure forging. Hot pressing has the advantage of producing high density composites due to the concurrent action of temperature and pressure. To prevent oxidation of the composites, the operation has to be carried out in vacuum and this can be expensive due to the need for insulation at high temperatures. The time required during hot pressing to attain useful densities is approximately 1 h in most studies [85,86].
Kim et al. [87] compared the hot pressing method to the SPS method in aluminium composites with CNT contents of 1, 3 and 5 wt%. Results show that the SPS method produced a greater hardness than the HP method. It was suggested that the composites fabricated with the HP method were exposed to a high temperature for a longer duration of dwell time that grain growth occurred. In contrast, the SPS method did not produce composites with particle growth because of the rapid temperature rise and fast sintering by self-generation of heat [88]. Maximum stress was also found to be greater in the SPSed samples as compared to the HPed composites. Maximum stress indicates the stress point at which crack initiates. In terms of the amount of wear, composites fabricated by the SPS method also exhibited improved resistance to wear than the composites fabricated by the HP method.

Long sintering times are most likely to stimulate thermally activated diffusion-controlled grain growth. If occurring, this phenomenon adversely affects the mechanical properties and renders the achievement of superior characteristics impossible [89]. On the other hand, high sintering temperatures can cause thermal damage of the CNTs and thus promote undesirable interfacial reaction between the Al matrix and the CNTs [90]. Houasaer et al. [91] in his work comparing the hot press (HP) technique with the spark plasma sintering (SPS), showed that there was interfacial reaction between the aluminium matrix and the CNTs during the HP method which was not observed in the SPS method. The authors explained that the duration of heating in the sintering process plays a major role in interfacial reactions and growth of intermetallics between the matrix and reinforcement. Hence, the longer sintering duration of the HP method facilitated the interfacial reactions which was discouraged in the SPS method due to the shorter sintering duration.

3.1.4. Hot isostatic pressing (HIP)
In case of HIP, the pressure is applied uniformly from all directions by using a gaseous or molten salt medium. This leads to uniform densification in all directions and ensures isotropic properties.

Yan et al. [92] combined graphene nanoflakes (GNFs) in aluminium alloy containing 1.5% Cu and 3.9% Mg elements. Hot isostatic pressing (HIP) was carried out at 480 °C and 110 MPa for 2 h. Afterward, the 40 mm diameter billet was preheated at 450 °C for 1 h in a stainless steel mould and then hot extruded into 12 mm diameter rods. Results show that with the addition of only 0.5 wt% GNFs, the yield strength was increased by nearly 50%, from 214 to 319 MPa and the tensile strength increased from 373 to 467 MPa. More remarkable was the fact that this increase in strength was achieved without causing a loss of ductility in the resulting composite. These results demonstrate that graphene nanoflakes (GNFs) have a great potential as an ideal reinforcement for aluminium matrix composites. They attributed the excellent ductility properties to the multiple wrinkled structures of GNFs.

3.1.5. Spark plasma sintering
Spark plasma sintering (SPS), also known as electric field assisted sintering (EFAS), plasma assisted sintering (PAS), and pulsed electric current sintering (PECS), is a variation of hot pressing in which the heat source is a pulsed DC current that is passed through the die or the powders (depending on whether the powder is electrically conducting) during consolidation. Spark discharges at the particle interfaces are believed to produce rapid heating, which enhances the sintering rate. The heating rates are quite high compared to hot pressing and can be up to 1000K s⁻¹. Efficient densification of powder can be achieved in this process through spark impact pressure, joule heating, and electrical field diffusion [93,94]. This method is, generally, suitable for consolidation of nano powders, without allowing sufficient time for grain growth. Compared to conventional sintering and hot pressing, SPS is definitely a promising method for obtaining high-density MM-Carbon compound composites. The short sintering time is also favourable in ensuring minimal or no reaction between CNT and metal matrices. Spark plasma sintering over the past decade has been extensively used in consolidating a wide range of materials including metallic alloy powders. This novel, non-conventional sintering method has proven to be
Advantageous offering full densification of materials owing to high heating rates, low sintering temperatures, and short sintering cycles over conventional sintering methods [1].

There is no general consensus on what mechanisms are activated during the sintering of initial powders in SPS, but some researchers have striven to propose theories based on their experimental results or assumptions. It is predicted that three mechanisms are active: (i) activation by pulsed current, (ii) resistance sintering (heating through electrical current passage) and (iii) pressure application [95–97]. However, overheating on the particles surface, which leads to localized melting in particle contacts, has been the popularly believed mechanism [98]. SPS features energy saving and low power consumption in comparison to the conventional sintering techniques. It is also reported that this process can lead to near full densification of bulk materials [99,100]. In this method, the starting powders are poured into a graphite mould and the sintering process is performed simultaneously with applied external pressure and electrical current [89,101] (Fig. 3).

However, high costs and limited sample geometries are the main disadvantages of SPS and HP [102]. Therefore, there is a need to upscale these processes for large scale production of various geometries having satisfactory performance at affordable prices [103].

Bisht et al. [104] successfully synthesized Al-GNP (graphene nanoplatelets) composite, with 21.4% improvement in strength and hardness, by spark plasma sintering route (SPS). They observed that SPS yielded high relative density of the structures, without affecting the intrinsic structure of GNPs [105]. Authors recorded relative density of 99.8% in the Al-1 wt% composite which yielded the best mechanical properties. Dense and clean interfaces between GNP and Al were obtained, without interfacial reaction product. Authors attributed this to the short sintering time availed by the SPS technique. Table 1 concisely recaps the advantages and disadvantages of the various sintering techniques discussed.

3.2. Effect of sintering temperature

Guo et al. [90] investigated the effect of SPS sintering temperature on the mechanical properties of AMCs using two different sintering temperatures (590 °C and 630 °C) and CNT content (0.75 and 1.0 wt%). Results showed that the composite which contained 0.75% CNTs and sintered at 590 °C had the lesser yield strength (YS), ultimate tensile strength (UTS) and elongation (EL). No matter the content of the CNTs, the mechanical properties of the composites were significantly improved by increasing the sintering temperature from 590 to 630 °C, including the strength and ductility. Thus indicating that the SPS sintering temperature plays a significant role in enhancing the mechanical properties of the composites. Higher sintering temperature contributes to the creation of the electric field generated among the particles and excites high-temperature plasmas under the action of pulse current, which facilitates a cleaning effect on the surface of particles, leading to the elimination of the barriers of atom diffusion. With externally applied pressure, the positive effects generated by higher temperature can accelerate the rearrangement
of particles and lead to a higher density. The composite with low content of the CNTs indicates less Al-CNTs interface and Al atom diffusion barriers, so Al atoms can diffuse readily and fill the interstices between the adjacent particles during the sintering process. Clustered CNTs act as a diffusion barrier during sintering process [106], and consequently reducing the sinterability of the powders (Fig. 4).

Saheb et al. [83] observed that, at constant sintering time of 20 min, the sintering temperature had an important influence on the densification of samples, due to the fact that sintering is a thermally activated process controlled mainly by diffusion [84]. Therefore, the higher the sintering temperature, the higher the diffusion rate, and consequently the less the pores in the bulk composite. The yield strength (YS), ultimate tensile strength (UTS) and elongation of the CNT/Al composite sintered at 800 K (527 °C) were measured to be 127 MPa, 186 MPa and 11.2%, respectively. As temperature is increased to 850 and 900 K respectively, both strength and ductility of CNT/Al composites improved progressively. YS, UTS and elongation of the composite sintered at 900 K (627 °C) were respectively 150 MPa, 212 MPa and 20.4%, translating to 18, 14 and 82% improvements respectively in comparison with those of the composite sintered at 800 K (527 °C).

3.3. Post sintering operations

Post deformation of sintered compacts can further enhance the density of the bulk composites [107,108]. This approach has been used mainly for Al-based composites. Hot extrusion, equal channel angular extrusion (ECAE), and hot/cold rolling have been widely employed as deformation techniques. Hot extrusion is the most commonly used process and, perhaps, the most preferred [109] for deformation processing of metal matrix (MM) – CNT composites [85,92]. In hot extrusion, the sintered or pressed compact is heated to the required temperature and then forced through a die typically conical in shape maintained at the high temperature. Inside the die, the compact undergoes a gradual reduction in area in the conical portion due to the application of shear stresses. The ratio of the area of the material at the entry to the exit is known as the extrusion ratio. Yan et al. [92] employed hot extrusion to achieve GNF alignment in the Al matrix. The stresses employed to force the compact through the die also helped in straightening and aligning the nanoparticle reinforcement, thus yielding a well-aligned composite. ECAE is a recently developed technique, similar to hot extrusion in which a material is extruded through a die with an extrusion ratio equal to 1. However, in this case, the material has to pass through a bent region. A lot of shear deformation is induced in the material. There is considerable grain refinement induced, due to the thermo-mechanical treatment of this process. Hot rolling on the other hand, is believed to improve the properties of the composites by enhancing densification and improving the dispersion of CNTs by breaking CNT clusters.

### Table 1 – Summary of the merits and demerits of the different sintering methods.

<table>
<thead>
<tr>
<th>Sintering methods</th>
<th>Method description</th>
<th>Merits</th>
<th>Demerits</th>
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<tbody>
<tr>
<td>Conventional sintering</td>
<td>Heating is through conduction, convection or radiation. Slow heating rates and long dwell times.</td>
<td>Simplicity of operation</td>
<td>Poor density of bulk compacts, coarse grains due to grain growth. Non-uniform heating resulting in thermal gradients across the composite.</td>
</tr>
<tr>
<td>Microwave sintering</td>
<td>Volumetric heating mechanism</td>
<td>Higher heating rates and shorter sintering times as compared to conventional sintering, leading to better mechanical properties</td>
<td>Poor densification as compared to novel sintering techniques like spark plasma sintering</td>
</tr>
<tr>
<td>Hot press sintering</td>
<td>Heating is done by electrical resistance, induction or radiation</td>
<td>Simultaneous application of heat and pressure leading to better densification</td>
<td>Long dwell times which poses the risk of grain coarsening and interfacial reactions which may be undesirable</td>
</tr>
<tr>
<td>Hot isostatic pressing</td>
<td>Simultaneous application of heat and uniform pressure from all directions</td>
<td>Uniform densification of the composite resulting in isotropic properties</td>
<td>High cost</td>
</tr>
<tr>
<td>Spark plasma sintering</td>
<td>Active mechanisms: activation by pulsed current, resistance heating and applied pressure</td>
<td>Highest heating rates and lowest sintering times. Grain growth is prevented resulting in highly dense, fine grained structures yielding excellent mechanical properties.</td>
<td></td>
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</table>
For example, Chen et al. [110] used hot extrusion as a post-sintering measure to enhance the bonding strength between the Al powder particles and the dispersion uniformity of the CNTs in the Al matrix. The traditional trade-off tendency between strength and ductility in metal based composites was evaded in Al/CNTs composites owing to concurrent improvement of Al–Al grains and CNT–Al interfacial bonding by hot extrusion. Zare et al. [107,108], applied equal channel angular pressing (ECAP) as the post-sintering processing to consolidate the fabricated materials. The well-densified composites with only 2 vol% CNTs after eight ECAP passes exhibited an approximate 30% increase in the yield strength compared to the pure Al samples. In general, ECAP process has two channels with the same cross-sectional area. Samples are usually circular or square according to the dimensions of the channels. After applying the appropriate lubricants, the sample is placed in the inlet channel and extruded through the outlet channel using a punch under pressure. The deformation mode in the ECAP process is a simple shear mode.

Rashad et al. [111] did not observe significant difference in experimental densities before and after extrusion, which they attributed to the low extrusion ratio (5:1). Though not much significant difference was observed in the densities, the hardness values increased with increase in GNP weight contents in aluminium matrix. The increased hardness of composite materials could be due to the presence of reinforcement particles possessing high strength, which offered high constraint during indentations. Furthermore, the hardness of pure aluminium and its composites increased after the extrusion process. This confirmed that the extrusion process played a vital role by minimizing the voids and cavities in the sintered materials, and this led to an increase in mechanical properties of composites. Expectedly, a reduction of voids and cavities ought to lead to an increased densification of the composites, which was not observed here.

In particular, it was found that a combination of SPS and hot-extrusion [110,112,113], was effective in fabricating high-performance composites because of the high density, fine grains and good CNT alignment [114].

In some rare cases, some researchers have observed difficulty in obtaining strong bonding strength between the aluminium particles and completely eliminating residual pores due to short diffusion times. Hence, it may sometime be necessary to strengthen the interfacial bonding and enhance the densification by post deformation [90]. Guo et al. [90] followed up with hot rolling process with S passes after spark plasma sintering to enhance densification and hardness as can be seen in Fig. 5. Results showed that hot rolling can further densify the composites sintered at 630 °C, since the residual pores were almost eliminated after the hot rolling process. It can be seen that the hot rolling can significantly improve the density of all the fabricated composites and the difference in density among the three composites is significantly decreased, due to the reduction of residual pores by the plastic deformation during hot rolling (Fig. 5).

By combination of SPS and hot-extrusion, authors have achieved high densities over 99% [110]. A large extrusion ratio of 37 was applied, which was helpful to improve the bonding conditions. This is because a large extrusion ratio brings a higher degree of material deformation. Larger deformation leads to better physical contact between grain boundaries and between CNT and Al, which is beneficial to improve the bonding conditions and mechanical properties of CNT/Al composites. The results suggest that high-energy consolidation conditions may be favourable in achieving optimal composite structures and excellent mechanical performances of CNT-reinforced MMCs by powder metallurgy.

4. Effects of dispersion methods

To successfully achieve the improvement in metal composites reinforced with these carbonaceous compounds, uniform dispersion is undoubtedly the singular most important requirement in AMCs. Different dispersion methods lead to differing degrees of dispersion even with the same combination of matrix and reinforcements. Hence, reports from literature show differing results even with similar reinforcement and matrix combinations. Ahmad et al. [86] and Bocanegra-Bernala et al. [102] both incorporated CNTs in an Al₂O₃ matrix but achieved different results. While Ahmad et al. [86] reported improvement in fracture toughness of 94% over monolithic Al₂O₃, Bocanegra-Bernala et al. [102] reported a 40% decrease in his study. Bartolucci et al. [115] also reported a decrease in mechanical properties with additions of 1 wt% of GNPs and CNTs respectively. These may be attributed to the differing processing routes, hence the intrinsic attributes
of the reinforcements are not sufficient in determining the resulting mechanical properties of AMCs as processing routes obviously play a crucial role as well.

The final performance of a metal matrix composite depend upon three key factors consisting the matrix, the reinforcement, and the matrix/reinforcement interface [68] with the interface strongly dependent on the ‘dispersibility’ of the reinforcement in the matrix. In the past, the role of the matrix was only associated with holding reinforcements securely. It is important to understand the roles of the matrix, the properties of the reinforcement and the ensuing interaction between the matrix and the reinforcement. The interface or the region between the matrix and the reinforcement actually plays a key role in stress transfer between the matrix and the reinforcement. If the bonding between the two is weak, which can occur due to poor wettability issues or lack of interaction in-between (and this is consistent with carbon allotrope additions and metal matrices), the final composite will have poor mechanical properties [2].

The dispersion of CNTs in the powder mixture is so crucial because subsequent processing stages like compaction and sintering will not improve the dispersion. Outstanding CNT clusters remaining in the powder mixture will ultimately still be present in the final component [1].

Achieving uniform dispersion of these carbonaceous compounds in the metal matrix has been the major challenge for researchers till date. Few researchers have been able to successfully disperse these allotropes owing to their high aspect ratio and strong van der Waals forces that keep them entangled in clusters. To solve this agglomeration problem, traditional and novel techniques such as nano-scale dispersion [116], high energy ball milling [117], in situ grown method [118], flame PM [119], solution coating [110], molecular level mixing [120], and cryomilling [121] have all been developed in the past decade.

4.2. Nano scale dispersion

Noguchi et al. [116] developed the Nano scale Dispersion (NSD) route for improvement of CNT dispersion. A sevenfold increase in compressive yield strength was reported with the addition of just 1.6 vol% CNTs. This remarkable strength was attributed to the uniform dispersion of the nanotubes using the NSD process. Though this process was quite complicated, the results were well worth it. The authors attribute the remarkable improvement to the novel dispersion method, which improved the wettability between the aluminium and CNTs.

4.3. Flake powder metallurgy

Jiang et al. [119] explored the flake powder metallurgy (flake PM) route in order to overcome the challenge of non-uniform distribution of carbon nanotubes (CNTs) in CNT/Al composites. This route solved the poor wettability issue between CNTs and aluminium powders by using slurry blending. By changing spherical Al powders to nanoflakes and surface modifying them with a polyvinyl alcohol hydrosol, flake PM produced homogeneously distributed CNTs on the aluminium. This route also preserved the structural integrity of the CNTs as they were protected from impact forces of milling balls as compared to ball milling processes. Hence, a composite with high tensile strength of 435 MPa and good combination of properties was produced and authors attribute these to the minimally damaged and uniformly distributed CNTs in the aluminium matrix.

5. Effect of reinforcement content

At higher sintering temperatures, the diffusion between the matrix and reinforcement particle is easier, resulting in better sinterability of materials [90,111]. However, Bisht et al. [104] reported an optimum volume fraction of 1.0 wt% GNPs above which the mechanical properties were not enhanced, irrespective of sintering temperature owing to less sinterability of the composites due to the difficulty in dispersing higher volume fraction of GNPs.

Gao et al. [79] observed a limiting volume fraction of graphene in his study at 0.3 wt% which yielded UTS of ~110 MPa. This was about 30% higher than that of unrein-
forced Al ~86 MPa. Results showed that in the 0.5 wt% Gr/Al composite, a lot of graphene was located at the boundaries of the Al grains, making cracks to form preferentially in graphene and extend to the Al matrix during tension, thus decreasing the tensile properties of the Gr/Al composite. A shift in the fracture mechanism was also observed from a typical dimpled fracture surface for pure Al to flat and not so dimpled surface as the graphene content increased, as shown in Fig. 6.

Both Clegg [124] and Wang et al. [125] explained that with a little CNT content, the hardness of the composite increases because the CNTs fill the microvoids of the aluminium particles. However, with addition of high CNT, the excess CNTs that remain after filling the microvoids form agglomerates with the aluminium particles. This conglomeration interrupts the sintering and causes defects. The effects of this could be as bad as porosity effects [109,126]. A point is therefore reached where, as the CNT content increases, irrespective of the intrinsic strengthening properties of the reinforcement, the composite strength begins to decrease as shown in Fig. 7.

Esawi et al. [3] investigated the mechanical properties of CNT reinforced Al composites. They observed that dispersion of CNT became progressively difficult above 2 vol% addition. Mechanical properties were observed to improve significantly with CNT addition with the most significant obtained at 2% CNT addition (tensile strength of ~260 MPa). Higher additions did not yield further enhancements as expected. This is owing to the agglomeration of CNTs at such high additions [121].

Guo et al. [90] observed an agglomeration of CNTs with increasing content from 0.75 to 1.0 wt% and this led to a reduction in the mechanical properties of the AMCs. This shows that the agglomeration of the CNTs in the 1.0CNTs-630 composite weakens the bonding between the Al powder particles and thus decreases the ductility of the AMCs as seen in Fig. 5.

Rashad et al. [111] observed an enhancement in the UTS and failure strain of aluminium composites with 0.25 wt% additions. But in contrast, the effect of higher additions of 0.5 and 1.0 wt% was detrimental to the mechanical properties. Both tensile strength and failure strain decreased with the increase in GNP additions. This is due both to overlapping of GNP with each other and to strong pi-pi attractions between graphene sheets.

6. The ductility/strength trade-off

The improvements in the strength properties of aluminium composites seem to be achieved at the expense of ductility and fracture toughness of the reinforced AMCs. Numerous reports exist in literature of enhanced tensile strength achieved by the reinforcement of aluminium with CNT accompanied by a reduction in ductility of AMCs [11,79,119,122,128]. Despite intense investigations on AMCs, achieving a balanced combination of strength and toughness has been a daunting task for researchers till date, as there seem to be a trade-off between the two very essential properties [110,129]. This is obviously a hindrance to their practical applications as these are crucial requirements since ductility is needed for formability while adequate fracture toughness is required for the prevention of catastrophic brittle failures in service. In addition to these essential properties, other key requirements to achieving balanced mechanical properties of AMCs are hinged on uniform dispersion of the CNTs in the matrix and good interfacial bonding between the reinforcement and the matrix [109,110].

It has been reported that these carbon-based nanomaterials could be incorporated into brittle materials to generate toughened composites that would be suitable for advanced engineering applications [99]. This strength-ductility trade-off has been severally reported in these composites. Saheb et al. [83] displayed this trade-off phenomenon. In their work, with the elongation of pure Al increasing from 21.1 to 24.1% came the reduction of UTS from 172 to 166 MPa as sintering temperature is increased from 800 to 900 K (527–627 °C). The trade-off
tendency in pure metals is due to the fact that both strength and ductility are determined by one factor—the level of difficulty in dislocation movement [130].

He et al. [118] fabricated high strength CNT/Al composites using the in situ grown CNT method. Nevertheless, while the strength increased up to more than 200 MPa (216–398 MPa), the ductility decreased significantly with fracture elongation of 0.7–4.7% as against 8.9 % elongation for the unreinforced Al composite. Najimi and Shahverdi [128] using a two stage milling method, achieved a remarkable tensile strength of 538 MPa with only 1.5 wt% CNT addition but with a ductility of ~0.05.

Aggressive dispersion methods like high energy ball milling tend to yield high strength values but are usually accompanied by very little ductility owing to the severe damage of the nano-reinforcements. Morisi and Esawi [131] reported that prolonged ball milling improved the disentanglement and dispersion of CNTs, but also compromised their structures.

6.1. Two stage milling

To overcome this dilemma, Xu et al. [129] employed a two stage milling process, precisely an 8 h low energy ball milling at 135 rpm followed by a 1 h high energy ball milling process at 270 rpm. Results showed that with 1.5 wt% CNT reinforcement, the AMCs exhibited a yield strength of 326 MPa, an ultimate tensile strength of 376 MPa and a total elongation of 12.4%. The enhanced properties could be explained by the dispersion method, which utilized shake powder mechanism, and then a short term high speed ball milling (HSBM), to obtain cold welded particles with good CNT/Al bonding, without too much structural damage to CNTs. Owing to this balance between the uniform dispersion, interfacial bonding and structural integrity of CNTs, the desired balance between strength and ductility was achieved.

6.2. Dual matrix approach

Salama et al. [132] experimented with a dual matrix approach. Typical single matrix (SM) was produced via dispersing CNTs in a pure aluminium matrix using HEBM of 400 rpm for 2h. Then the novel dual matrix (DM) was obtained by embedding the pre-processed SM powders into a secondary matrix of pure aluminium using HEBM for 30 and 60 min, while keeping the total CNT content strictly to 1 wt% and 2.5 wt% for both matrices. Results show surprisingly that secondary milling is associated with higher ductility as the dual matrices with secondary milling showed higher ductility values as compared to single matrices though accompanied by a loss in strength. This, the authors [132] attributed to the fact that secondary milling resulted in a stronger bond between the two phases due to the continuous impact of the milling media, which led to an efficient load transfer. The best combination of properties was achieved in the DM with 50% mixed powder ratio, with tensile strength of 298.3 MPa and 11.6% ductility and 348.3 MPa and 7.15% for both 1 and 2.5 wt% respectively. Increased strength was attributed to the work hardening effect associated with the milling process.

6.3. Boundary modification

Chen et al. [110] focused on improving the boundary conditions between aluminium and CNTs to obtain a good balance of strength and ductility by varying sintering temperatures. He observed that higher sintering temperatures enhanced the CNT-Al interface, promoting good interfacial bonding, thus yielding better mechanical properties. The CNT-Al interface in the composite sintered at 800 K (527 °C) becomes indistinct in the composite sintered at 900 K (627 °C), reasonably resulting from enhanced diffusions of atoms near the interface. For the specimens sintered at 800 K (527 °C), debonding phenomenon of local micro cracks produced at grain boundaries are suggestive of insufficient grain boundary conditions. As sintering temperature is increased to 900 K (627 °C) however, the fracture surface exhibits a ductile failure mode with numerous dimples and plastically deformed grain boundaries (GBs), suggesting good bonding conditions of Al matrix with the CNT reinforcement.

6.4. In situ grown method

This is a method in which CNTs are directly ‘grown’ on aluminium powder particles using a catalyst such as cobalt or nickel while the aluminium particles act as substrate. This direct growth of CNTs is able to facilitate the uniform dispersion of undamaged CNTs within the aluminium matrix, leading to a significant enhancement of mechanical properties. One of the major challenges of this method, however, is the cultivation or growth of high quality CNTs. In their work, Tang et al. [133] shows that by a careful control of parameters especially synthesis temperature, high quality CNTs with good crystallinity can be grown.

Yang et al. [122] explored an in situ chemical vapour deposition method where CNT was grown on Al using Cobalt as a catalyst to synthesize 2.5 wt% CNT. The Al/CNT were then dispersed using short term high energy ball milling at 500 rpm to improve interfacial bonding between CNT and Al. They obtained UTS of 334 MPa with ductility of 17.9%. This significant increase can be attributed to the homogenously dispersed CNTs as a result of the growth of CNTs on the aluminium particles. This led to efficient stress transfer during tensile deformation, thereby improving the strength of the composite [134] (Fig. 8).

6.5. Cryogenic milling

Cryogenic milling (or cryomilling) is a mechanical mixing process whereby the starting powders are milled at extremely low temperatures using liquid nitrogen. At these low temperatures, usually in the range of (~100 K, that is, ~−173 °C) recovery and recrystallization processes become exceptionally slow while the dispersion of reinforcements in the matrix occurs. As a result, composites sintered with the admixed powders processed by this method have considerably high strength [135].

He et al. [121] utilized a cryogenic milling process to disperse varying compositions of CNTs in Al 2009 matrix. CNTs/2009 powders were cryogenically milled under liquid
nitrogen using ball-to-powder weight ratio of 39:1, milling time of 2 h and rotation speed of 180 rpm respectively. They achieved a good balance of high strength with yield strength of 443.3 MPa and elongation of 10.2%, proving that CNTs could be dispersed in such a short time under cryogenic conditions. This balance of strength and ductility was attributed to the excellent dispersion and relatively low damage of the CNTs during cryogenic milling. CNTs effectively transferred stress to the matrix and reflected cracks under loading. Fractographic images revealed CNT pull-out and bridging with a dimpled fracture surface. The highlights of some of the main dispersion methods discussed can be seen in (Table 2).

Table 2 – Summary of main dispersion methods.

<table>
<thead>
<tr>
<th>Dispersion method</th>
<th>Method description</th>
<th>Advantages</th>
<th>Disadvantages</th>
<th>Results</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ball milling [126]</td>
<td>Low energy ball milling (LEBM) leads to partial declustering of CNT agglomerates. High energy ball milling (HEBM) is effective in detangling a higher proportion of CNTs from their clusters</td>
<td>LEBM preserves the structural integrity of the CNTs. HEBM is more effective in dispersing the CNTs</td>
<td>Difficult to achieve uniform dispersion with LEBM only. Damage of CNT unique structures which is detrimental to mechanical properties of resulting composites</td>
<td>UTS: 429 MPa Elongation: 0.035</td>
</tr>
<tr>
<td>Nanoscale dispersion [114]</td>
<td>Wettability between the aluminium matrix and CNT reinforcement is enhanced</td>
<td>Good interfacial bonding leading to remarkable increase in mechanical properties with little CNT content</td>
<td>Very complicated procedure</td>
<td>Compressive strength: 280 MPa Elongation: 15%</td>
</tr>
<tr>
<td>Flake powder metallurgy [117]</td>
<td>Poor wettability is improved by conversion of aluminium to thin flakes and slurry blending</td>
<td>Interfacial bonding is enhanced and CNT integrity is preserved</td>
<td>Slightly complicated</td>
<td>UTS: 435 MPa Elongation: 6%</td>
</tr>
<tr>
<td>Two stage milling [127]</td>
<td>Prolonged LEBM at low speed followed by a short term HEBM at high speed</td>
<td>Good combination of strength and ductility due to good dispersion and good interfacial bonding</td>
<td>Careful selection of parameters during HEBM is important to prevent CNT damage</td>
<td>UTS: 376 MPa Elongation: 12.4%</td>
</tr>
<tr>
<td>Dual matrix approach [130]</td>
<td>Comprises of embedding pre-processed CNT-Al admixed powders into pure Al matrix using HEBM</td>
<td>Good combination of properties</td>
<td>Two stages of HEBM which may lead to higher CNT damage</td>
<td>UTS: 348.3 MPa Elongation: 7.15%</td>
</tr>
<tr>
<td>In situ grown method [120]</td>
<td>CNTs are grown on Al powder particles using Co or Ni as catalyst</td>
<td>High CNT content can be incorporated into Al matrix. Clustering and agglomerations are significantly reduced or eliminated</td>
<td>High quality CNTs are seldom obtained</td>
<td>UTS: 334 MPa Elongation: 17.9%</td>
</tr>
<tr>
<td>Cryogenic milling or cryomilling [119]</td>
<td>Milling at cryogenic temperatures under liquid nitrogen</td>
<td>Prolonged milling is not required</td>
<td>Due to shorter milling times, CNT structure is preserved</td>
<td>UTS: 560 MPa Elongation: 10.2%</td>
</tr>
</tbody>
</table>

7. Intermetallics of aluminium formed during sintering

It has been established from literature that intermetallics are formed due to damage of the carbon allotropes during milling or sintering of Al-based composites. Structural damage (during milling) and/or thermal damage (during sintering) seem to trigger interfacial reactions which lead to the formation of the Al₄C₃ intermetallic phase [74]. It has been documented that sites of structural disorders, defects in the graphitic planes, amorphous carbon layers, nanodefects, and open tube
ends of MWCNTs, selectively favour the production of this intermetallic phase [136,137]. Usually, highly stable defect-free graphitic planes of the carbon nanotube or graphene tend not to react with aluminium to form aluminium carbide even at very high temperatures. What has not been established, however, is the actual role of these intermetallics in the mechanical behaviour and properties of the resulting composites. In Zhang et al. [85], it was observed that some of the GNPs were embedded in the Al matrix, but others reacted with Al and formed Al4C3 phase. Bastwros et al. [41], Wu et al. [138], Esawi et al. [3] also speculated the same effect of aluminium carbide on the composites. Ci et al. [137], in line with this, observed Al4C3 at the open ends of nanotubes and the amorphous surface layers at annealing temperatures as low as 500 °C. It is reported that such carbides could enhance the Al–CNT bonding and hinder CNT pull-out. Therefore, their presence, in this regard, would not be considered detrimental from the mechanical point of view. Gao et al. [79], Bartolucci et al. [115], Hassan et al. [28] and Liu et al. [139], however, seem to have a contrary view as they believe that this interfacial reaction product has a negative strengthening effect on the resulting composite. They observed carbide formation between defective graphene nanoplatelets and aluminium due to the employed processing route via thermal exfoliation of graphite oxide. Previous results that have reported the presence of Al4C3 in the composite utilized processing temperatures that were above 500 °C showing that thermal damage can trigger the formation of this carbide phase. Dispersion methods like ultrasonication and low energy ball milling tend to prevent mechanical damage of CNTs while SPS seem to prevent thermal damage of the CNT due to the short sintering times. However, there have been reported cases in literature where Al4C3 detection was observed after SPS. This is mostly attributed to the mechanical damage of the carbon allotropes in the course of its dispersion, especially during high energy ball milling. Hence the formation of this intermetallic phase is largely sensitive to processing routes. Ju et al. [105] observed that the formation of Al4C3 could be detrimental to the composites owing to its brittleness but Kwon et al. [140] suggest that if this brittle phase is homogeneously dispersed in the composite, it could lead to higher strengthening effects.

Houasaer et al. [91] compared HP and SPS for the fabrication of the CNT/Al composites. Al4C3 was found to be present in the hot pressed samples but there was no evidence of formation of same in the SPSed samples, proving that the sintering method and particularly the duration of sintering plays a major role in the initiation and growth of the Al4C3 particles. It was observed that the duration of the sintering stage in SPS was six times shorter than that of the HP process and as such, too short to encourage the formation and growth of Al4C3. SPS technique proved to be an effective route for controlling the CNT–Al interface while completing the sintering process.

As a novel and rapid powder consolidation process, SPS offers several advantages over the conventional sintering process such as much faster heating and cooling rates [141]. Such characteristics can avoid damage of the delicate structures of these nanocompounds and possible adverse chemical interfacial reactions, thus obtaining a fine grain sized metal matrix. Kwon et al. [142] showed that Al4C3 produced during the fabrication of MWCNT/Al composites led to an increase of the UTS from 85 to 194 MPa owing to the improved wettability between the matrix and reinforcement. In addition to this, the Al4C3–Al interface is said to possess superior strength in comparison to the Al–CNT interface [44]. On the contrary, other researchers claimed that Al4C3 was very brittle and thermodynamically unstable, and thus its formation at the interface would lower the strength of MWCNT/Al composites [143]. Li et al. [144] reported that the UTS of 1.0 vol% MWCNT/Al composite decreased by 24.4% after forming an Al4C3 layer between the MWCNTs and the Al matrix.

Park et al. [145] observed that the Al4C3 reaction on the surface of the CNTs improves the wettability between the Al matrix and CNTs [121,146]. Consequently, the formation of Al4C3 at the interface between Al and the CNTs improves interfacial bonding and facilitates effective load transfer, thereby improving mechanical properties of the composite. The role of this intermetallic on the interface is a critical issue as the interfacial strength is a determining factor of the composite performance [147]. The extraordinary properties of these carbon allotropes would be inconsequential if the applied load on the composites cannot be successfully transferred from the aluminium matrix to the reinforcements due to a weakened interface [148,149].

In Najimi et al. [128] it was further validated that the CNT being not fully transformed into Al4C3, allows the strong interior portion of the CNTs coupled with the formation of Al4C3 at the interface between Al and the CNTs for effective load transfer, improving mechanical properties of the composite [150]. The generated aluminium carbide during the sintering process was implanted into the aluminium matrix in the extrusion process. Such implanted aluminium carbides would adhere to the Al matrix and the CNTs more strongly [140].

Zhou et al. [151] clarified in their study that the yield strength (YS) increased with increase in the Al4C3 amount, reaching to a maximum value of 116.4 MPa at 11% Al4C3. That is, formation of a small quantity of Al4C3 (~11%) produced at the MWCNT-Al interface could be effective to improve the load transfer in the MWCNT/Al composites [152]. They produced Al4C3 nanostructures at the end of the MWCNTs, incorporated in the Al matrix, by appropriate heat treatment. The stress contrast seen around the Al4C3 in the HRTEM image revealed the evidence of a trace of friction, which may lead to the improvement of the anchor effect from the Al matrix. This anchor effect of Al4C3 may restrict local interfacial slipping and increase the resistance of the Al matrix to deformation. They concluded that the formation of Al4C3 could effectively enhance the load transfer in MWCNT/Al composites as this stress contrast is a proof of strong interfacial bonding [151]Fig. 9.

The control of the size, amount and geometry of interfacial Al4C3 compounds reacted from CNTs and Al matrix seem to be crucial to clarifying the role of this intermetallic compound [153]. The Al4C3 precipitate is identified to either occur as individual nanoparticles formed from partially reacted CNTs with Al [142] or nanorod crystals evolved from completely reacted CNTs [152,154]. Chen et al. [114] showed a preference for the nanoparticles by showing that at medium temperatures
(800–875 K), that is (527–602 °C), the quantity of Al₄C₃ nanoparticles increased at the interface between partially reacted CNTs and Al matrix. This had a positive effect on the interface strength and load transfer efficiency as both increased significantly compared to the low temperature sintered composites, proving that this intermetallic in nanoparticle dimensions can improve the mechanical properties. At high temperatures of 900 K (627 °C), however, some mono-crystal Al₄C₃ phases with rod shapes were formed in Al matrix, and these resulted in decreased strengthening effect.

Guo et al. [155] corroborated this by extensively evaluating the size effect of Al₄C₃ intermetallic by its precipitation during heat treatment. It was observed that the higher the heat treatment temperature, the larger the size of the precipitated Al₄C₃. This led to the deterioration of both strength and ductility of the composite for two reasons. First, the production of high quantity and large sized Al₄C₃ consumed a high quantity of CNTs available for load bearing functions. Secondly, the inherent brittleness of large sized Al₄C₃ significantly reduces the efficacy of the load transfer mechanism. It was thus concluded that few smaller sized Al₄C₃ are beneficial for the enhancement of the mechanical properties of the composites, while a high amount of larger sized Al₄C₃ would be detrimental to the composite properties.

8. Conclusion

The significance of effective processing routes to facilitate the incorporation of these carbonaceous compounds ‘super’ reinforcements into AMCs, to bring about the desired properties, has been established. Among the discussed sintering techniques, SPS has emerged as a novel route owing to the short sintering times which effectively prevents grain growth and preserves the integrity of the delicate structures of the carbon allotrope reinforcements. Other sintering techniques that favour grain growth tend to produce composites with inferior properties to that produced by SPS. However, there is a need to develop this process economically to aid commercialization of SPSed composites.

Achieving homogenous dispersion of these carbonaceous compounds in aluminium matrices has remained a major challenge for researchers till date. Traditional and sophisticated methods have been explored with varying degrees of success. To effectively exploit the full potential of these carbon allotropes, the challenge to homogenously disperse undamaged nanostructures have to be fully overcome. Moreover, more fabrication methods yielding composites with good combination of both high strength and good ductility are
required to mitigate the strength-ductility trade-off usually observed in reinforced composites. Also, effective, economical and uncomplicated methods of dispersion have to be developed and adapted for large scale production.

More work is required in clarifying the actual role of the intermetallic compound Al₄C₃ in the carbon allotrope reinforced composites. It has been a major controversial subject within the research community. Three schools of thought have been established. Some observe that it is detrimental and has negative strengthening effects. Some others document that it has positive strengthening effects. While some yet believe that it is beneficial in small amounts but detrimental in large amounts. That is, the role of the intermetallic might be size, amount and geometry dependent.

Conflict of interest

Authors declare no conflict of interest.

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