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The Effect of Sintering Duration on Metal Matrix Composite Reinforced with CNT Fabricated by Powder Metallurgy: A Review

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ABSTRACT

Metallic alloys have broad applicability due to their characteristics, but there are still opportunities to improve on it or remove their constituent flaws that comes from their pure metallic form. Lightweight metals such as magnesium and aluminum have excellent strength-to-weight ratio, but they suffer from issues such as the tendency to ignite at high temperatures, porosity, and oxidation even when it is alloyed, which can be a safety risk when used in aircrafts or automobiles. Hence methods to overcome it include fabricating metal matrix composites (MMC) from metals such as aluminum, magnesium, titanium, and copper with carbon nanotubes (CNT) as a reinforcing material using powder metallurgy. Fabrication of such materials requires identification of important parameters to ensure the product has ideal characteristics. Thus, this study aims to identify and determine how the parameter of sintering duration in powder metallurgy and various factors of CNT, such as its concentration in the matrix, affects the mechanical properties of MMCs. This review is to encourage future breakthroughs and research in MMCs and powder metallurgy, as well as the usage of CNT in MMCs for any applicable fields that rely on such materials as the data obtained in this study can assist in the synthesis of MMCs.

Keywords: Metal Matrix Composite; Sintering Duration; CNT; Powder Metallurgy.

INTRODUCTION

Since the stone age, humankind has evolved their usage of workable materials for use in construction and creating tools. The genesis and subsequent development of such products has been a subject of interest which formed the field of material sciences. Therefore, discovering and researching on the behavior of materials under certain conditions and finding methods to improve or modify its properties has always been a primary goal mainly due to the potential to overcome various challenges or provide breakthroughs to an otherwise impossible obstacles due to flaws in a materials' property. Composites are one of the materials that is covered in the realm of material sciences and are defined as a solid material formed by combining two or more separate substances; usually of different physical or chemical properties, to create an entirely new material with superior properties compared to their individual constituents [1]. According to Shaffer *et al.* [2], one of the earliest techniques of composite material used in building construction, has been dated to at least 6000 years ago in terms of its usage. Whereas a primitive sculpture was discovered in the Czech Republic that was made of clay with animal bone meal and its creation was estimated to be around 29,000 years ago [3]. Both have shown that humanity has known the concept of using a separate material to reinforce another even in prehistoric ages.

With the advent of the 20th century, research in composite materials has seen leaps and bounds to meet the demands of rapid industrial expansion, scientific advancements, and military systems. The specific field of metal matrix composite (MMC) has been an interest during that period, but it was not until the 1980s when it received greater attention which boosted its development and research [4]. MMC is referred to as a specific class of composite material consisting of a metallic matrix and a secondary material that is dispersed in it as a form of reinforcement. Metallic matrix are usually lightweight metals such as aluminum, copper, magnesium, or titanium, whereas the reinforcing material, which can be in the form of particulate or fiber, can either be ceramic or metallic. While MMCs have superior properties in certain aspects, it is still plagued by issues such as high manufacturing costs which can be a deterrent for commercial use.

As stated previously, metals that are often used in MMCs are magnesium, titanium, aluminum, and copper primarily due to their relative density and lightweight trait [5]. Prior to the development of MMCs, metal alloys such as magnesium had large-scale usage in the late 1930s to the 1940s for manufacturing of military aircrafts for World War II, likely due to their high strength-to-weight ratio, low density, and good damping behavior [6-7]. Titanium alloys however have high specific strength and remarkable corrosion resistance which appeals to its usage in aerospace, automotive, and biomedicine [8]. The presence of extraordinary properties in metallic alloys also applies to copper alloys and aluminum alloys, broadening their usage in various industries and fields. An example being that copper alloys have high electrical and thermal conductivity whereas aluminum alloys have similar characteristics to magnesium alloys being lightweight with high strength levels [9-10]. Regardless of their outstanding properties however, metallic alloys are still plagued by many issues that affect and limit their applications. Example being magnesium alloys having low plasticity and corrosion resistance as well as the tendency to ignite under specific conditions [11–13]. This brings us to metal matrix composites, which allows the reinforcement of the properties of metallic alloys using particulates or fibers to overcome such issues. The focus of this review is specifically on the metal matrix composites of four different metals, which are magnesium, aluminum, titanium, and copper reinforced by carbon nanotubes (CNT) and their properties after undergoing a specific powder metallurgy method which is called solid state sintering.

Solid state sintering is referred to as a method of forming a solid mass from materials; usually metals and ceramics, using pressure or heat below their melting point [14]. This process is commonly used in manufacturing steel, or in producing alloys, plastics, and complex components where conventional metallurgy involving the melting of the substance is considered inefficient due to their high melting point. The materials used in sintering begin in the form of a powder, which is then compressed together while being heated to a temperature below melting point to initialize the fusion of the powders' atom to bond together into a single piece of solid [15].

Characterization of Mg, Al, Ti, and Cu ALLOYS

Before characterizing the alloys of magnesium, aluminum, titanium, and copper, the properties of the pure metals must be known first as shown in Table 1.

Table 1: The properties of pure metals magnesium, aluminium, titanium, and copper

Metal	Atomic Number	Number of electrons	Density (g/cm^3)	Properties
Magnesium	12	12	1.738	High biocompatibility Good recyclability Low strength Low corrosion resistance High flammability
Aluminium	13	13	2.7	Lightweight metal Better corrosion resistance Does not ignite
Titanium	22	22	4.506	Lightweight metal High melting point (1668 °C) Corrosion resistant High ductility Low thermal and electrical conductivity
Copper	29	29	8.96	High ductility Low hardness Excellent electrical conductivity

Notes: Taken from [13, 16-18]

To improve the properties of the metals stated above, alloying is one of the ways of doing so and it displayed better mechanical properties compared to the pure counterpart with better ductility and strength. Song *et al.* [13] has reviewed several types of magnesium alloy, which are cast magnesium alloys, wrought magnesium alloys, and functional magnesium alloys. Each of these has their own improved characteristics such corrosion resistance and superior fatigue property. Despite the alloying of magnesium however, it still retains some of its pure constituent flaws such as the tendency to combust at a certain temperature threshold, which still limits their application especially in aeronautics where the usage of the material poses a risk towards fire on aircrafts [11]. This pattern is also applicable to aluminum alloys, in which according to Altıparmak. *et al.* [19] it is great material for aerospace components due to their remarkable strength-to-weight, stiffness-to-weight, and machinability but still suffers from defects due to manufacturing factors and the properties of the alloy itself such as cracking, porosity, and oxidation of the alloys, and is further supported in research by Leirmo *et al.* [20] and Zhu *et al.* [21]. Therefore, the properties of metallic alloys still have many improvements to be made when it is compared to conventional materials such as steel. Fabrication steps in sintering have significant influence towards characteristic improvements as it is more focused on the microstructures of the alloys itself to overcome or reduce any pre-existing flaws.

The current applications of metallic alloys are metallic alloys are dependent on the characteristics of the pure metal themselves, example being magnesium and titanium being suitable for biomedicine as implants due to their biocompatibility, followed by automobiles and aerospace where lightweight materials are in high demand.

Characteristics of Carbon Nanotubes

Carbon is a chemical element with an atomic number of 6 and has 6 electrons with an electron configuration of $1s^2 2s^2 2p^2$. Due to carbon's valence electrons, it can form different bonds with other carbon atoms which results in several types of carbon allotropes such as diamond and graphite. Carbon nanotubes consist of graphitic carbon or graphene, which is one of the allotropes of carbon with a hexagonal or "honeycomb" crystal system due to the sp2 hybridization, in the form of a cylinder [22]. It has extraordinary characteristics such as high thermal conductivity and tensile strength, as well as chemical stability and electric conductivity [23]. These properties however are highly dependable on the nanotube's structure itself, such as its chirality, diameter, presence of impurities or defects, and whether it is single-walled or multi-walled [24]. Figure 1 shows a diagram model of graphene, graphite, single-walled carbon nanotube, and multi-walled carbon nanotube.

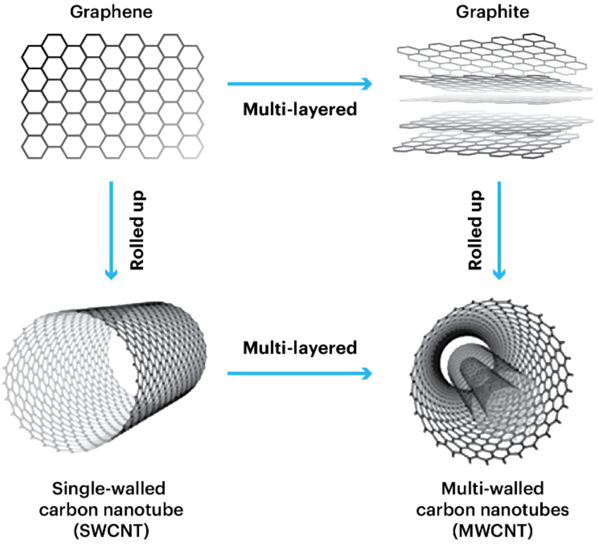

Figure 1: Diagram model of graphene, graphite, single-walled carbon nanotube, and multi-walled carbon nanotube [25].

In manufacturing and synthesizing CNT, multiple methods can be used which are Chemical Vapor Deposition (CVD), Arc Discharge, and Laser Ablation [24]. The methods have their own advantages and disadvantages since each of them produces different yields, types (Single-walled or multi-walled) of CNT, and possible defects.

Due to the properties of carbon nanotubes, it makes for an ideal material and thus serves multitude of purposes and applications in real life ranging from aeronautics to biomedical. CNTs are also a viable material for composite reinforcement in MMCs, where it can contribute its properties towards the metal matrix in many ways. The dispersion of CNT reinforcement in metal composites have proven to enhance their mechanical properties such as their tensile strength and hardness, expanding the usability and application of CNT-reinforced MMCs into realms of engineering, energy storage and electronics [26]. This indicates that CNT is a remarkable material when it is used stand-alone or as a complementary reinforcement for other materials.

Metal Matrix Composites

Metal matrix composites, also known by its abbreviation MMC, is a specific type of composite material where the primary material or "matrix" are metal based and is reinforced by a secondary material or phase that can either be ceramic or metallic in a particulate, continuous or discontinuous fibers form. MMCs are characterized to have superior properties compared to their constituent components such as improvements in tensile strength, hardness, and working temperature [27]. Evidently these properties make MMCs ideal alternatives to conventional materials for engineering purposes such as aeronautics [28]. MMCs can be manufactured, fabricated, and processed in different ways, with each method resulting in different characteristics in the product of the composites even when the composition remains the same. Solid-phase processing and liquid-phase processing are the most used method to manufacture MMCs, with the former being the easiest way to achieve uniform dispersion of reinforcement material in the matrix while the latter being generally cost-effective [28-29]. Other manufacturing methods include two-phase processing and layering [30-31].

Typically, the metal matrix used in fabrication of MMCs are alloys of lightweight metals such as magnesium alloys, aluminum alloys, copper alloys, and titanium alloys. Mg alloys, Al alloys, and Ti alloys are the mainly used materials as a metal matrix because of their high strength-to-weight ratio and low density, Mg based MMCs are becoming the more preferred choice due to it being two-thirds the density of aluminum and one-third the density of titanium [27]. Cu alloys however are far denser than the three metals stated previously; with the density of copper being 8.94 g/cm^3, which limits their usage to thermal and electrical applications due to its pre-existing trait of being an excellent thermal and electrical conductor [26].

From research conducted by Abazari *et al.* [32], a composite consisting of magnesium (Mg), zinc (Zn), manganese (Mn), oxygen (O), and carbon nanotubes (CNT) were fabricated using semi-powder metallurgy to form a Mg-alloy composite called ZM31/MgO-CNTs composite. The composite underwent testing in terms of mechanical, corrosion, and biological properties, with the results indicating that the composite obtained improvements in its uniaxial compressive strength and microhardness, less considerable influence on the corrosion resistance by CNTs, and good biocompatibility.

Another example of Mg-alloy composite research is by Akinwekomi *et al.* [33], where a Mg alloy called AZ61, and CNT are combined to fabricate a CNT-reinforced AZ61 composite foam via rapid microwave sintering. The composite foam displayed improvements in terms of compressive strength and energy absorption, and simultaneously showing that rapid microwave sintering is an efficient sintering method due to its ability to achieve similar results as conventional powder metallurgy while reducing overall sintering duration.

Sintering duration is shown to influence the properties of composites according to Bahaj *et al.* [34], wherein an aluminum matrix composite $Al\text{-}A_{l9}Co_2\text{-}Al_{13}Co_4$ undergo sintering for 4, 8, 24, 48, and 72 hours at different cobalt concentrations, and is followed by a hardness test. The results showed that at lower cobalt concentrations which is Al-0.5%Co and Al-1%Co, the microhardness of alloys increases with sintering time, but the opposite occurs for aluminum alloys with higher cobalt concentrations. Al-3%Co displays a decrease in hardness after 48 hours of sintering, whereas Al-5%Co displays a decrease in hardness after 24 hours of sintering. Therefore, alloys with large compositions are likely to generate pores during the sintering process, which affects its hardness.

In Cu-CNT composites however, the sintering duration to achieve the optimal improvements towards its properties decreases with the increase in diameter of CNT but with constant length, except for its hardness, thermal conductivity, and relative density which remains unchanged regardless of the sintering duration, concentration of CNT and the size of CNT [35].

Fabricating MMCs in general has shown enhancements towards characteristics of the metal matrix as stated by Malaki *et al.* [36], ranging from tensile strength to compressive strength depending on their reinforcement content.

Effects of Sintering Duration on the Characterization of MMCs

In powder metallurgy, there are different parameters that can influence the characteristics of MMC, in this section the sintering duration or holding time are the focus. Other factors such as sintering temperature or sintering pressure are also considered as it has some influence.

Based on research by [37], multiple aluminum MMC samples were prepared using powder metallurgy method with different processing parameters which are compaction pressure (100, 110, 120, and 130 MPa), sintering temperature (300, 400, 500, and 600 °C), and sintering time (120, 180, 240, and 300 minutes). Based on the general linear model they have obtained; they have identified the ideal processing parameters from the samples, with sintering duration being the second most crucial factor in influencing the mechanical properties during the process of sintering in powder metallurgy. It is also noted that high sintering time (300 minutes) at low sintering temperatures (300 °C) results in poor compression strength at 139.96 MPa in the sample compared to high sintering time (300 min) at high sintering temperature (600 °C) which results in a compressive strength of 279.36 MPa, and the highest compressive strength obtained is 290.57 MPa for a sintering duration of 180 minutes at sintering temperature of 600 °C.

Meanwhile Nayak *et al.* [38] concluded that aluminum composite with the composition of $Al\text{-}SiC\text{-}Al_2O_3$ received lesser effect in contribution to the compressive strength of the composite material with the increase of sintering duration but greater densification during analysis when the sintering duration was increased from 30 minutes to 90 minutes.

Chakrapani & Suryakumari [39] have stated that for their research on aluminum matrix composites, homogenous distribution of particles with fewer interfacial reactions are obtained by minimizing the duration of sintering when manufactured using powder metallurgy method, where in two samples that is sintered from 30 to 90 minutes, the elongation of aluminum composite does not drastically increase beyond 45 minutes for a sample sintered at 600 °C. Interfacial reactions are known to influence the properties of MMCs in different ways, both positively and negatively.

According to Bahaj *et al.* [34], sintering duration does impact the mechanical properties of an aluminum composite (Al-Al_9Co_2-$Al_{13}Co_4$), with a peak hardness value of 153.89 ± 4 HV at 72 hours of sintering. However, there is a gradual reduction in hardness value as the concentration of cobalt increases even at the same parameter of sintering duration (4, 8, 24, and 72 hours). This is attributed to the increase in porosity formation due to Kirkendall effect, which negatively impacts the mechanical properties of the composite.

Furthermore, Vignesh Babu *et al.* [35] have stated that in CNT-reinforced copper composites, the sintering duration at 60, 75, and 90 minutes does not have a significant impact towards the composites' relative density, electrical and thermal conductivity, and hardness at a specific concentration and diameter of CNT in the sample. It is even noted that the highest sintering duration at 90 minutes seemed to lower electrical and thermal conductivity of the samples compared to when the sample is sintered at 75 minutes.

For Mg-based composites manufactured using the method of spark plasma sintering done by Ali *et al.* [40], they used the Taguchi method to determine the parameters of sintering temperature (450, 500, and 550 °C), sintering duration (5, 10, and 15 min), and sintering pressure (30, 40, and 50 MPa) to determine its influence on the microhardness, microhardness, and density of the composite. It is determined from their studies that an increase in sintering time results in greater grain growth, which leads to reduced microhardness and microhardness. Hence for spark plasma sintering method, the ideal sintering time is 5 to 10 minutes.

Kumar & Pandey [41] have also fabricated Mg-based composites via microwave sintering, and at sintering duration of 10, 25, 40, and 55 minutes. Based on their results, the density and ultimate compressive strength of the composite was observed to be increasing up until the 40 minutes mark, however at 55 minutes mark it then decreases due to the increasing porosity.

On the analysis of Ti-based composites, Kumar & Bharti [42] have stated that an increase in sintering duration results in higher density due to internal diffusion and compression, but any further in sintering time results in reduced density of the metal mainly due to thermal expansion and from their research the ideal duration is 5 to 6 hours for Ti-based alloys or composites from a range of 1 to 8 hours.

From this, it can be stated that sintering duration does have a positive impact on MMCs but only up to a certain point before other factors or parameters such as sintering temperature and the concentration or size of reinforcing material begins to have greater influence on the mechanical properties. Furthermore, as the sintering duration increases, it is more likely that the microstructures of composites begin to be impacted in a way that it will negatively affect the mechanical characteristics of the MMC and therefore there is limit on how long the composite should undergo sintering to obtain the optimal results. It should also be noted that other factors such as concentration of reinforcement phase or sintering temperature do have an effect as to how long should the composite be sintered. In general, the optimal sintering duration can be determined to be somewhere around the average values of the sintering duration, such as 10 minutes for Ali *et al*. [40] and 32.5 minutes for Kumar & Pandey [41]. Table 2 shows the summarization of the sintering duration discussed.

Table 2: Sintering Duration of MMCs

MMCs	Sintering Duration (min)	Ref No.
Al based alloys	180	[37]
Cu based alloys	75	[35]
Mg based alloys	5 to 10	[40]
Ti based alloys	300-360	[42]

Characterization of CNT-Reinforced MMCs

As stated previously, MMCs are shown to have exceptional properties due to varying reasons and factors, one of them being the reinforcing material of the composites themselves. In this section, CNT-reinforced MMCs are the focus to determine how the presence of CNT, its concentration, and other factors relevant to its reinforcement of MMC can affect the properties of the composites. CNTs that are distributed in metal matrix composites are usually in the form of whiskers or short fibers, as shown in Figure 2.

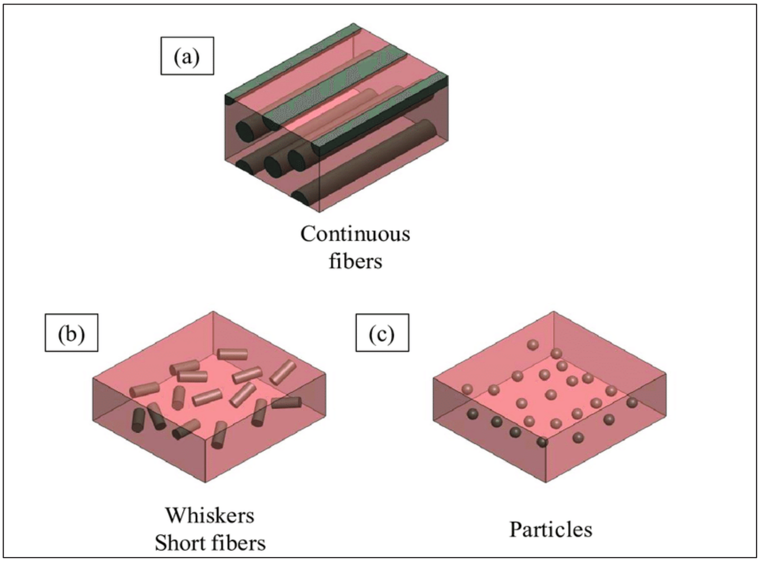

Figure 2: Diagram of Metal Matrix Composites with different types of reinforcement phase [43].

In Mg-based composites, many articles and journals have covered the specific MMC being reinforced with CNT. In a research paper presented by Handayani *et al.* [44] they prepared multiple samples of Mg matrix composites with differing concentrations of MWCNT, ranging from 0.1 wt.% to 0.5 wt.% and determined that the increasing concentrations of MWCNT resulted in the increase in porosity and pore size of the composite, with the optimal hardness value being obtained at a concentration of 0.3 wt.%. Say *et al.* [45] have also conducted similar research, but with AZ series Mg alloy which also consists of Al and Zn, wherein different CNT concentrations (0.1 to 0.5 wt.%) were used to produce the composite, with results showing an increase in CNT concentration in the composite causing an increase in compressive strength and porosity but a decrease in yield strength and ductility when compared to the unreinforced samples, it also noted that unreinforced samples alongside samples with lower CNT concentration (0.1 wt.% and 0.2 wt.%) had better resistance to corrosion compared to samples with higher CNT concentration. Furthermore, Upadhyay *et al.*[22] has reviewed several types of Mg-CNT composites with differing manufacturing methods and concluded that mechanical properties of the CNT-reinforced Mg composites differ with methods used to fabricate it likely due to the distribution of CNT in the matrix, and the increase in wt.% of CNTs are a factor in the decrease of corrosion resistance of the composite.

For Al-based composites, Jagannatham *et al.* [46] stated that Al matrix composites have increased toughness at lower CNT content (less than 2 vol.%) compared to higher CNT content (more than 3 vol.%) and that the tensile strength to yield strength ratio decreases when the amount of CNT increases in the composite, which indicates greater brittleness. Meanwhile Khanna *et al.* [47] mentioned similar statements based on their studies, where CNTs does enhance the mechanical properties of the composites such as the ultimate tensile strength, compressive strength, Young's modulus and so on, but after an additional amounts of the reinforcement in terms of weight percentage, there is a decrease in the same properties of the composite due to the formation of agglomerates in the boundaries and the matrix since CNTs have strong Van der Waal forces between each other.

Whereas for Ti-based composites, in a review conducted by Okoro *et al.* [48] it was stated that the addition of CNT into Ti-based matrix ranging from 0.4 wt.% to 1.0 wt.% displayed improved mechanical properties, example being that 1.0 wt.% of CNT in the composite amounts to an improvement of 80.7% in its ultimate tensile strength and 143.6% in yield strength when compared to pure Ti, but it comes at a cost of ductility being reduced. Similar to other metals, a drop in tensile properties of the composites were observed at higher weight percentage of CNTs due to the same aforementioned reasons. Sharma *et al.* [49] have presented in their research paper that the yield strength and tensile strength of their CNT reinforced Ti composite sample increases as the volume fraction of CNT increases, ranging from 0 vol.% to 20 vol.%, and that the rate of increase for elastic and shear moduli begins to decrease at higher values of volume fraction.

For Cu-based composites, it is mentioned by Singh *et al.* [26] that the mechanical characteristics of Cu-CNT composites are dependent on numerous factors including its concentration, dispersion, and orientation inside the Cu matrix, example being a Cu matrix that is reinforced with super aligned CNT shows an increase to ultimate tensile strength and yield strength of 90% to 100% and 45% to 50% respectively, and is due to the alignment in direction by the CNTs. Furthermore, optimal processing parameters and conditions help in obtaining greater enhancements towards the composites likely due to better dispersion of CNT in the matrix. For similar reasons as previously mentioned metal composites, it is also mentioned that higher CNT concentrations beyond certain point (>1.0 vol.%) in Cu-based composites can lead to the decrease in electrical and thermal conductivity in the same study.

Based on all the reviews of the different types of metal used in CNT reinforced MMCs, they share the same results that CNT reinforcements in metallic based composites are beneficial to their mechanical properties, however there is a limit to how much CNT can be added into the composite before it becomes more of a detrimental to the properties due to how CNTs tend to agglomerate in the matrix of the metal. This can be resolved by using better processing conditions or methods to allow better distribution of the reinforcement. Therefore, it is ideal to determine the correct amounts of CNT that can be used as a reinforcement to obtain the optimal mechanical properties of the MMCs, which is somewhere between 0.1 to 0.4 wt.% of CNT but it is dependent on the metal matrix used; which may have some influence on the behavior of the CNT itself in the matrix, and the usage case of the composites as certain characteristics are lost while others gained enhancements, with one example being the Ti-based composite fabricated by Okoro *et al.* [48] it gained improvements towards ultimate tensile strength and yield strength but loss in ductility at higher concentrations of CNT. Table 3 provides an overview of the optimum concentrations of CNT for each metal matrix composite discussed.

Table 3: Optimum concentrations of CNT for metal matrix composites.

MMC	Optimum concentrations of CNT	Ref. No.
Mg based alloys	0.3 wt.% 0.1 wt.% to 0.2 wt.%	[44] [45]
Al based alloys	<0.2 vol.%	[46]
Ti based alloys	1.0 wt.% 5 vol.% to 15 vol.%	[48] [49]
Cu based alloys	<1.0 vol.%	[26]

CONCLUSION

In conclusion, this literature review has covered the effects of sintering duration on MMCs and the presence of CNT as the reinforcement material of the composite when fabricated by powder metallurgy, which can be used to determine the optimum conditions when fabricating a metal matrix composite reinforced with CNT by the method. The influence of sintering duration during the manufacturing process of MMCs are notably positive towards the mechanical properties, however it is only up to a certain amount of time since greater duration would cause significant grain growth and porosity, which negatively impacts the properties of the composite. Whereas for CNT as reinforcement material, it shows an overall improvement towards the MMCs, but greater concentration of the reinforcement in the matrix often results in the formation of agglomerates due to the strong Van Der Waals forces between the CNTs themselves, causing unwanted decrease in the mechanical properties in the MMCs. There are also other influences to be considered such as sintering temperature or sintering pressure as the optimal duration can vary depending on the changes of the variables.

Therefore, fabricating a CNT reinforced MMC via powder metallurgy requires a specific condition to obtain the ideal characteristics regardless of the type of metal matrix since sintering duration has considerable influence alongside the concentration of CNTs. In general, the ideal CNT-reinforced MMC fabricated by powder metallurgy has at most 0.4 wt.% of CNT content with a sintering duration around their average value of a set amount of sintering time. Further research should be conducted on related topics to this review to determine the most optimum achievable conditions and specifications for MMC fabrication.

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