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A Review on the Influence of Processing Methods on Corrosion Rates of Mg-CNT Nanocomposites

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Abstract

Several studies have reported the corrosion rates of magnesium carbon nanotube nanocomposites, but their corrosion behavior is still not well understood. Adding carbon nanotubes (CNTs) to magnesium (Mg) matrices almost always results in an increase in mechanical properties, e.g., increased elastic modulus, hardness, ultimate tensile strength, and yield strength. However, this increase in mechanical properties usually comes at the expense of compromised corrosion resistance. Galvanic interactions between the CNTs and the Mg matrix are the usual culprits of nanocomposite corrosion. It is desired to study the corrosion behavior of these materials further to create a nanocomposite that is less susceptible to corrosion from the start, i.e., the fabrication method. In the present review, four processing methods (Disintegrated Melt Deposition, Friction Stir Processing, Powder Metallurgy, and Ball Milling) which were used to successfully synthesize Mg-CNT nanocomposites and test their corrosion properties are discussed. Attempts are made to correlate processing methods to corresponding corrosion rates. It was found that the corrosion rates extracted from each reviewed study may not be readily comparable, and looking into nanocomposite coatings and CNT vol.% or wt.% optimization may be the best way to proceed. The findings of this investigation can be used as a starting point for the creation of a Mg-CNT nanocomposite which is less inherently susceptible to corrosion as this could take the "potential" out of the many potential applications of these novel materials.

Keywords: Magnesium nanocomposite; corrosion; carbon nanotube; processing.

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Introduction – Why Magnesium? Why CNTs?

In the last twenty years, metal matrix composites (MMCs) have attracted numerous researchers and manufacturers worldwide, owing to their promising mechanical properties suitable for a large number of engineering applications, especially those in automotive and aerospace industries [1, 2]. Since weight reduction in the transportation sector is more critical than ever before, magnesium matrix composites are gaining more popularity since they are considered to be a lightweight, energy-saving family of metal matrix composites.

Magnesium has several propitious merits warranting this attention: it is the lightest structural metal with a density of 1.74 kg/m³ at 20 $^{\circ}$ C [3]; magnesium is the sixth most abundant element in the earth's crust, representing 2.7% of the earth's crust [4]; magnesium has excellent castability, high damping capacity, and it is biocompatible – which gives way to several medical applications [5]. Despite these merits, magnesium suffers from a hexagonal close-packed (HCP) structure which makes it brittle (due to limited slip systems), and it is prone to corrosion due to its low position on the electromotive force (EMF) series at -2.36 V [6].

Recent developments in nano-reinforcement technology are helping to strengthen magnesium and make monolithic Mg less susceptible to creep and corrosion. Specifically, this brief overview paper explores the effects that carbon nanotubes (CNTs) have on the corrosion rates of magnesium matrices and attempts to find any evidence to suggest that, of the fabrication methods examined, a certain nanocomposite fabrication method may produce a Mg-CNT nanocomposite which is inherently less susceptible to corrosion.

Ever since CNTs were discovered by Iijima in 1991 [7], material scientists have been attempting to incorporate the strength and unparalleled thermal and electrical properties of these carbonaceous tubes into monolithic metals to create composite materials with the best properties of both the tubes and the matrix. Table 1 highlights some of the most remarkable properties of CNTs.

Table 1. Noteworthy properties of carbon nanotubes

From a mechanical property perspective, adding CNTs can largely benefit the resulting Mg nanocomposite. For example, Goh et al. [17] achieved a simultaneous increase in 0.2% yield strength (YS), ultimate tensile strength (UTS), and ductility for Mg-CNT nanocomposites with 0.3 and 1.3 wt% carbon nanotubes. They report that 0.2% YS increases from 126 \pm 7 MPa (99.9% Mg) to 140 \pm 2 MPa (Mg – 1.3 wt.% CNT), UTS increases from 192 \pm 5 MPa (99.9% Mg) to 210 \pm 4 MPa (Mg – 1.3 wt.% CNT), and ductility increases from 8.0 \pm 1.6% (99.9% Mg) to 13.5 \pm 2.7% (Mg – 1.3 wt.% CNT). Similarly, Yang et al. [18] report an increase from 35.3 to 38.6 GPa in Young's Modulus when going from pure Mg to a Mg nanocomposite with 2 wt.% CNTs. Similar studies [19-21] report that Mg-CNT– and Mg alloy – nanocomposites outperform monolithic magnesium and magnesium alloys in terms of elastic modulus, thermal expansion coefficient, electrical and thermal conductivity, creep resistance, and other properties.

Of special importance, creep resistance is largely desired when attempting to design a particular service life for a part. It has been shown that adding a relatively small volume fraction of CNTs to magnesium effectively improves the creep resistance of the nanocomposite relative to pure magnesium. For example, our group conducted a study [22] on the creep behavior of Mg reinforced with 0.25, 0.5, and 0.75 vol.% CNTs against pure Mg. The best creep resistance belongs to the Mg matrices with 0.25-0.5 vol.% CNTs as the creep rates and displacement were lowest for these nanocomposites, attributed primarily to the CNTs acting as effective obstacles to dislocation motion. Kainer et al. [23] report that other Mg reinforcements can result in composite materials whose creep rates are 10-100 times lower than that of their monolithic alloys. They additionally state that these creep rates are competitive with that of some steels. It would be ideal to obtain an optimized volume fraction of CNTs (or other reinforcement) which yields the best creep resistance for a particular set of service conditions. However, creep resistance is just one of many response variables to consider when it comes to tailoring materials for practical applications.

Creep resistance, corrosion resistance, weight, ultimate tensile strength, yield strength, elastic modulus, among many others, are just a few of the most critical response variables one ought to consider when truly optimizing material for a particular engineering application. Again, it would be ideal to perform a rigorous engineering design of experiment (DOE) to obtain a statistically valid, defensible, and reliable conclusion - in this case, the exact composition of the Mg-CNT nanocomposite appropriate to a given set of service conditions. A DOE would readily reveal interactions among the factors, i.e., how adding CNTs may benefit one desired property and seriously impair another (tradeoffs in properties). Such a DOE would be an excellent study for future work and to the best of the author's knowledge, no such DOE has been performed to date.

This paper simply seeks to investigate the available responses of CNTs in Mg on one response variable, corrosion resistance, to begin to shed some light on creating fine-tuned Mg-CNT nanocomposites. It is suspected that the nanocomposite fabrication method may be a significant factor in the corrosion response, simply by the nature of these processing methods. These methods are discussed in greater detail in the following sections and already published studies are discussed to start to answer whether or not the fabrication method factor is significant. It is the authors' genuine hopes that the findings of this paper can drive nanocomposite fabrication forward, inspire future work toward using DOE to create optimum Mg-CNT nanocomposites, and ultimately get a better understanding of how these novel materials corrode.

Corrosion of Magnesium and its Nanocomposites

It is widely known that pure magnesium does not have appreciable corrosion resistance given the fact that it is commonly used as a sacrificial anode in water heaters, outboard motors in boats, and several other underwater applications where cathodic protection is crucial. Many studies [24-26] have shown that adding CNTs to magnesium only exacerbates the corrosion and causes the resulting nanocomposite to deteriorate faster. The increased corrosion is typically attributed to galvanic effects between the cathodic tubes and the anodic Mg matrix.

Magnesium has a fairly sensitive corrosion response, even when it comes to tracing impurities of nobler metals. Ghali et al. [27] report that even ppm concentrations of nobler elements like Fe, Ni, or Cu can accelerate the corrosion rate of the Mg matrix. Makar and Kruger [28] quantify how dramatically the corrosion rates increase with these particular impurities. Figure 1 is reproduced from their work to illustrate this point. They also suggest staying below the tolerance limits of Fe, Cu, and Ni of 170, 1000, and 5 ppm, respectively, in Mg to avoid the rapid corrosion.

Magnesium can be used in engineering applications, and it has not been taken seriously until recently on account of many reasons including, but not limited to (i) poor alloy design (e.g., using Mg with high concentrations of impurities like Fe, Cu, Ni, and or inclusions of melting salts), (ii) improper component design, or (iii) the appropriate surface protection for the particular application was not

considered at all (absent), or it was wrong, or inadequate altogether. It is no wonder why sourcing, surface considerations, and thorough consideration of the application itself are so important when it comes to creating a corrosion-resistant Mg alloy or composite.

Figure 1. Effect of several metal impurities and alloying elements on the corrosion rate of Mg alloys in 3 wt.% NaCl. Reproduced from Makar and Kruger [28].

Another important consideration for Mg nanocomposites regarding corrosion rate is the reinforcement phase itself. It turns out that nonconducting reinforcements are generally a better choice when trying to minimize the mentioned galvanic corrosion attack. However, some of the nonconducting reinforcements which may benefit the nanocomposite corrosion-wise may be toxic (e.g., most rare earth), warranting even more caution when selecting magnesium nanocomposite reinforcements, especially for implants [23, 29]. This is just one example begging the exploration of what options are available to minimize corrosion of Mg-CNT nanocomposites.

Typical advice that materials scientists give to avoid this inherent corrosion includes optimizing the volume or weight fraction of the nanoparticles, the use of surface coatings (sometimes coating the CNTs themselves), using surfactants that hinder powders from agglomerating or experimenting with other particle dispersion techniques, and processing with the purest Mg or Mg alloys available [30]. This guidance is effective and useful, however, there are additional aspects to consider.

Perhaps the optimization should begin by selecting a manufacturing process that inhibits agglomerations and creates less corrosion-prone materials from the start. For example, corrosion rates of Mg-CNT nanocomposites created via disintegrated melt deposition (DMD) do differ from those produced by friction stir processing (FSP), and every other processing method. One method may produce more oxidation-resistant materials since the oxide formation is almost completely inevitable in some of the processing steps involved in these methods.

 A fair amount of studies [24-26, 31-32] have reported corrosion rate responses, yet no study to date has attempted to compare and correlate Mg-CNT nanocomposite fabrication methods to corrosion rate. Hence, we arrive at the aim of this study. Four techniques that have been used to successfully synthesize Mg-CNT nanocomposites – and further study the corrosion properties of the nanocomposite – are thoroughly discussed. These techniques are 1) Disintegrated Melt Deposition (DMD), 2) Friction Stir Processing (FSP), 3) Powder Metallurgy (PM), and Semi Powder Metallurgy and 4) Mechanical Ball Milling.

1. Disintegrated Melt Deposition (DMD)

Disintegrated melt deposition is a stir-casting derived process. DMD is also considered to be a spraying/liquid metallurgy process. A composite slurry is first formed by creating a molten metal (Mg) and stirring (with an impeller) at a predetermined velocity and time to incorporate and distribute particles into the molten mix (usually with some kind of vibratory feeder) – which is the first step of a stir-casting process. DMD additionally processes this slurry by passing it through the pouring nozzle of a crucible and this is melt is disintegrated by an inert gas jet, e.g., argon, at a superheated temperature. Finally, this slurry is deposited on a metal substrate. The final product is an ingot that can then be hot extruded to the desired shape [33]. Figure 2 shows a schematic taken from Jayalakshmi et al. [34], depicting the DMD technique.

Figure 2. Schematic of the disintegrated melt deposition (DMD) technique taken from Jayalakshmi et al. [34].

The DMD technique is commonly employed to fabricate Mg-nanocomposites because this method results in fewer oxides in the final material, unlike stir casting. However, an issue with this processing method is that the superheated temperatures, e.g., 750°C, can damage the CNTs, creating voids and breakages in particle-matrix interfaces which are perfect for oxides and any kind of available compounds to fill and create a perfect place for pitting and other forms of corrosion to occur.

Aung et al. [24], synthesized a set of Mg-CNT nanocomposites with 0.3 and 1.3 wt.% CNTs via the DMD technique and used immersion tests in a 3.5 wt.% NaCl solution to study the corrosion behavior. Specifically, they used evolved hydrogen gas measurements, weight loss measurements, and potentiodynamic polarization measurements. They reported higher corrosion rates for the Mg nanocomposite with 1.3 wt.% CNTs. Table 1 shows their reported results in the form of corrosion potential (E_{corr}) , and corrosion current density (i_{corr}) .

Table 1. Corrosion quantities reproduced from Aung et al. study [24] demonstrate the corrosion rate increase. E_{corr} and i_{corr} values for the Mg-CNT nanocomposites in 3.5 wt.% NaCl solution.

Sample	$E_{\rm corr}$ (V vs. Ag/AgCl)	i_{corr} (mA cm ²)
Mg Mg-0.3 CNT Mg-1.3 CNT	-1.59 -1.57 -1.50	0.027 0.056 0.572

It is apparent from Table 1 that a drastic increase in the corrosion rate is observed when CNT weight percent increases from 0.3% to 1.3%. These authors also state that micro-galvanic corrosion is the predominant corrosion mechanism in the nanocomposite.

2. Friction Stirred Processing (FSP)

Friction stirred processing is a solid-state processing method, it is a derivative process of friction stir welding, and it is an effective way to form fine-grained microstructures at the surface region of metallic materials. First, a groove (or sometimes a series of holes) is created on the surface of the piece. Secondly, the groove (or series of holes) is filled with the desired amount of particles and then a rotating tool traps the particles within the material. Lastly, a pin tool, through a stirring process, distributes the particles throughout the bulk of the metallic substrate [20]. Figure 3 depicts these steps and is reproduced from the work of Jayalakshmi et al. [34]. The friction between the rotating tool and

the specimen generates heat, which softens the material, and results in plastic deformation and grain refinement [35]. However, temperatures attained during FSP are lower than the melting point of the materials to avoid porosity issues and interfacial reactions [36-37].

Figure 3. The steps of the friction stir process (FSP) taken from Jayalakshmi et al. [34]. FSP steps are as follows: (a) a groove is created on the surface of the matrix material, (b-c) a rotating tool closes the groove with the nanoparticles of the prescribed inside the region of interest, and (d) the rotating tool is traversed across the workpiece to fully close the groove, distribute the nanoparticles, and completes the FSP.

The largest challenge of the FSP method is achieving uniform dispersion of the reinforcement particles throughout the surface finish of the final material. The method described above typically encounters slipping and splashing of nanoparticles during the FSP process, so it is very common to see pre-manufactured grooves on the matrix surface filled with particles that can then be capped with a pin-less tool (preventing the splashing). Then, a tool with a pin performs multiple passes back and

forth on the groove or holes to achieve a more uniform distribution of the particles without the splashing or slipping issue [37]. Recent efforts are aimed at attempting to use FSP to fabricate bulk nanocomposites via the FSP technique.

Saikrishna et al. [25] produced multi-walled CNT-nanocomposites with the pin-less FSP method to avoid the slipping/splashing issue described earlier. They tested pure Mg, friction stir processed Mg (FSPed Mg), and a FSPed Mg-MWCNT nanocomposite. They report that the MWCNTs enhanced hardness and decreased corrosion resistance. Similar to the DMD study, potentiodynamic polarization results using a 3.5 wt.% NaCl solution was used to draw corrosion conclusions. Table 2 shows the corrosion rates obtained for each sample of this study in mils per year (mpy), or thousandths of an inch per year (distinctly different than mm/year). Notice how the FSPed Mg exhibits a much slower corrosion rate than the non-FSPed Mg sample.

Table 2. Corrosion quantities reproduced from Saikrishna et al. study [25] demonstrate the corrosion rate increase. The authors report electrochemical parameters and calculated the corrosion rate of the samples.

Sample	i_{corr} (μ A cm ²)	E_{corr} (V _{SCE})	Corrosion Rate (CR) (mpy)
Mg	40.49	-1.50	36.41
FSPed Mg	35.23	-1.62	31.70
FSPed MWCNT/Mg	41.93	-1.47	37.73

Saikrishna et al. [25] also note that the FSPed samples were more refined on the surface of the material, while the layers underneath were quite agglomerated. The "stir zone" was 2.5 mm deep into the material, and this is the refined surface to which Sakrishna et al. are referring. This highlights the issue of even dispersion of particles encountered with using the FSP method - the layers underneath the "stir zone" tend to have agglomerations. They attribute the increased corrosion to galvanic interactions between the MWCNTs and the Mg matrix.

3. Powder Metallurgy (PM) and Semi Powder Metallurgy

Powder metallurgy is considered to be a solid-state manufacturing method. There are three essential steps to this manufacturing process: (1) blending evenly the metallic powders with the desired amount of reinforcement, (2) compaction of the mix to form green compacts, and (3) sintering to form a more dense material which can then be further processed (e.g., isostatic pressing) to become even less porous for the final nanocomposite. Figure 4 shows a simplified schematic of this process reproduced from Mondal [38] for the general manufacture of a metal matrix nanocomposite.

Figure 4. A basic flow diagram of the steps involved in the powder metallurgy process adapted from Mondal [38].

Turan et al. [26] studied magnesium alloy AZ91 reinforced with MWCNTs, graphene nanoplatelets (GNPs), and fullerene (C_{60}) , produced by semi-powder metallurgy. What makes their experimental procedure semi-PM rather than traditional PM is that they ultrasonicated their carbonaceous reinforcements for 2 hours in ethanol to break van der Waals forces before blending them with the matrix material, and consequently a vacuum distillation was carried out to remove this ethanol. It is this extra ultrasonication step is what makes this a semi PM process. Their corrosion findings are tabulated in Table 3. It is easily seen from the corrosion rates of any AZ91 sample reinforced with any carbonaceous phase that experienced a higher corrosion rate than pure AZ91.

Sample	OC potential (V)	$i_{corr} (\mu A)$	Corrosion Rate (mm/year)
AZ91	-1.472	104.655	1.31
$AZ91 - 0.5$ MWCNT	-1.491	388.431	4.92
$AZ91 - 0.5$ GNPs	-1.449	326.902	4.13
$AZ91 - 0.5$ C60	-1.506	212.137	2.68

Table 3. Corrosion quantities of interest reproduced by Turan et al. [26] for all AZ91 samples.

It is apparent from Table 3 that pure AZ91 exhibits the best corrosion resistance of all materials tested, and it had no carbonaceous reinforcements. Turan et al. [26] attribute the MWCNT composite having the highest corrosion rate to the strong galvanic couple formation formed between the tubes and the matrix.

A relatively older study by Endo et al. [32] from 2008 has added controversy to the corrosion realm of Mg-CNT nanocomposites. Their study involved Mg-MWCNT nanocomposites with 1 and 5 wt.% MWCNTs which were fabricated via powder metallurgy followed by vacuum hot-press and extrusion. Contrary to the findings of all of the previous studies discussed, Endo et al. [32] concluded that the addition of MWCNTs resulted in an improvement in corrosion resistance for the Mg matrix. They report that the MWCNTs keep the oxide layers from detaching from the material, thus slowing the rate of corrosion, leading to an improvement of corrosion resistivity. Several papers [24, 39-40] are dubious of these findings and the anticorrosive conclusions of Endo et al. remain controversial.

4. Mechanical Ball Milling

Mechanical ball milling can be considered a sub-categorical process of powder metallurgy. Despite this, it is worth considering the method in isolation when it comes to examining the rates of corrosion. In this process, a high-energy ball mill is used to repeatedly cold weld, fracture, and re-weld powder particles. This practice is particularly useful for metal matrix composites using ceramic particulates because the process is effective at breaking up ceramic clusters, which ultimately results in a composite with more uniformly distributed reinforcement particles. After the milling step itself,

the process ends in a similar manner described in PM processing, e.g., hot extrusion. Figure 5 depicts the ball milling process and is reproduced from the work of Sivasankaran et al. [41].

Figure 5. (a) High-energy planetary ball mill and **(b)** schematic of the milling process to fabricate nanocomposite powders taken from Sivasankaran et al. [41].

Mindivan et al. [42] successfully synthesized Mg-CNT nanocomposites via mechanical ball milling followed by hot extrusion. They found that the corrosion rate increased as they increased CNT weight percent. Figure 6 shows the results of their study. Notice how the corrosion rate becomes drastically higher when the CNT weight percentage reaches 4 wt.%.

Figure 6. Corrosion rate as a function of CNT content from Mindivan et al. [42] study.

Similar to what a majority of the previously mentioned studies have attributed the corrosion increase to, Mindivan et al. [42] also attribute this outcome to galvanic cell formation between the Mg matrix and CNTs. All studies examined here, except for Endo et al. [32], agree that CNTs appear to hurt the corrosion resistance of magnesium.

The corrosion rates may not be completely comparable considering the differences in processing if Mg or Mg alloy was used as the matrix, and some of the CNTs were single-walled and some were multi-walled. The processing methods appear to be fairly inconclusive at this point –given that all resulting Mg-CNT nanocomposites have been more prone to corrode (again except Endo et al. [32]). Perhaps a better strategy is to look into various surface treatments for nanocomposites when changing fabrication methods is not feasible.

Surface Treatments – Nanocomposite Coatings

Investigating various coatings and other surface treatments for magnesium nanocomposites may be a more viable option in certain scenarios where an elaborate processing method is too expensive or simply not practical. A comparison of coating cost and processing cost still warrants conduction if one or the other must be chosen to prevent corrosion for a given application. Note that it may not be worth the time to coat a nanocomposite if fabrication did not begin with a highly pure Mg matrix simply because Mg is very susceptible to corrosion and the material will gradually corrode away underneath the chosen coating naturally.

Arrabal et al. [43] evaluated the viability of using a silicate plasma electrolytic oxidation coating for Mg and some of its alloys and nanocomposites. They deemed the coating to be effective for some of the alloys and nanocomposites. In their particular study, SiC particles were used. These are ceramic particles, but it is possible that this coating could apply to magnesium nanocomposites with carbonaceous nano-reinforcements too, e.g., CNTs.

Coating the nanocomposites also comes with its issues which cannot be overlooked. For example, any purity underneath the coating surface will eventually eat away at the uncoated surface underneath. Hence, coatings are not always the answer and one ought to do a thorough consideration of the surface and choose cautiously if a coating must be used.

Nezamdoust et al. [44] investigated a more creative approach to coating. Rather than coating the surface of a Mg-CNT nanocomposite, their group first coated the CNTs themselves before incorporating them into a Mg matrix. In this study, MWCNTs were embedded with phenyltrimethoxysilane (PTMS) sol-gel coating at various concentrations to improve the corrosion resistance of the resulting nanocomposite formed by these coated CNTs in a Mg alloy (AM60B) matrix. They found that coating the CNTs did improve the corrosion resistance of the overall nanocomposite. This is because they recorded smaller corrosion rates and also a greater wetting angle $(\sim 87^\circ)$ to 94.7°), implying that the nanocomposite is more hydrophobic and more likely to resist corrosion by the intrusion of water. Coating CNTs in gels before making them into nanocomposites is a relatively new approach, but it appears to have promising potential for creating a more corrosion-resistant Mgnanocomposite.

Summary

In summary, adding CNTs to a Mg matrix or a Mg alloy seems to benefit the mechanical properties of the resulting nanocomposite, but this benefit almost always comes at the expense of losing corrosion resistance. Many of the studies discussed here attributed this corrosion rate increase to galvanic interactions between the carbonaceous tubes and the magnesium matrix.

However, one exceptional study by Endo et al. [32] reported that adding MWCNTs to Mg results in a nanocomposite which is more resistant to corrosion, but several papers and studies dispute this work and consider it a fluke. At least one study from each fabrication method discussed has demonstrated that the interaction of Mg and CNTs adversely affects the overall nanocomposite, resulting in galvanic corrosion. However, galvanic cell formation is not the only form of corrosion observed. The DMD method, and liquid metallurgy methods in general, tend to damage CNTs, and damaged CNTs result in a particle-matrix interface with poor integrity and many voids – an ideal spot for preferential pitting and crevice corrosion to occur. Several mechanisms may be at hand, but galvanic appears to be predominant.

Moreover, corrosion rates obtained from each reviewed study may not be readily comparable. To this end, it may be worthwhile to look further into coatings for a Mg-CNT nanocomposite, or special gel coatings of the CNTs before adding them to a Mg matrix, if CNT fraction optimization with respect to a particular processing method is not a viable option. A DOE investigation is recommended for future work with nanocomposite fabrication methods being emphasized as a factor of particular interest for further investigation of the corrosion quandary explored in this paper since the preliminary comparison discussed here is inconclusive.

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