Iron nanoparticle surface treatment of carbon nanotubes to increase fatigue strength of steel composites

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ABSTRACT
A simple, scalable, and reproducible method is used to decorate carbon nanotubes (CNTs) with metal nanoparticles (NPs). Decorating CNTs with iron NPs prior to forming steel-based metal matrix composites (MMCs) improves the high-cycle fatigue (HCF) strength of the MMC by more than 350% vs. plain 1084 steel and more than 100% vs. an MMC with untreated CNTs. An inverse correlation between CNT loading and tensile strength was observed. The CNT surface treatment uses an organic microenvironment surrounding suspended CNTs and emulsion chemistry to treat individual CNTs or small bundles. This process can be adapted to work with other metal NPs or CNT types. This work demonstrates the potential for metal-treated CNTs to further improve the HCF strength of MMCs and paves a pathway toward additional CNT-reinforced composites.

GRAPHICAL ABSTRACT

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Introduction
Since first being reported in 1991 [1], carbon nanotubes (CNTs) have been of great interest to the scientific community for their incredible mechanical, electrical, and thermal properties [2]. There are two major forms of CNTs, single-walled and multi-walled, where single-walled CNTs (SWNTs) consist of a single plane of sp²-bonded carbon atoms rolled to form a tubular structure and multi-walled CNTs (MWNTs) consist of multiple planes in a concentric
tube arrangement. The sp² bonding arrangement of each carbon atom in CNTs offer extremely high strength and unique elastic properties. The presence of imperfect, sp³ bonds in MWNTs diminishes these properties and further, only the outermost wall of the MWNT is directly involved in carrying mechanical load [3,4]. Density functional theory models predict maximum tensile strengths for SWNTs as high as 110 GPa, nearly 50 times greater than specialty steels, and Young’s Modulus around 1 TPa [5–8]. Because of their extremely low density, CNTs have a predicted specific strength that far exceeds other metallic, ceramic, or organic materials.

Researchers have long sought to harness the beneficial properties of CNTs in pure-CNT structures and composite materials. Metal matrix composites (MMCs) consist of ceramic or organic particles or fibers dispersed in a metallic binder. Common additives include ceramics, such as tungsten carbide and silicon nitride, or organic materials, such as carbon fibers [9]. Compared to these additive materials though, CNTs have the highest specific strength to offer the most significant increase in composite strength while lowering the overall density of the composite. A comparison of additive material specific strengths is shown in Figure 1, indicating the potential benefits for CNTs as an additive material in MMCs.

In applications experiencing repetitive applied stress below the ultimate tensile strength, fatigue strength is a more critical parameter to the life expectancy of the material. A simple model for the fatigue behavior of fiber reinforced materials was developed by Drucker et al. in 1966 [11]. Their model indicated that fiber reinforcement in perfectly plastic materials has little effect on the stress-strain response. However, in work-hardened materials, such as metals, the fiber reinforcement would block slip between planes and ultimately lead to a significant increase in the fatigue strength. This behavior was confirmed later by other researchers using ceramic particles and metal matrices [12,13].

Many MMCs have been investigated for their potential in high fatigue environments [14,15]. These composites are typically aluminum or titanium matrices with ceramic reinforcing particles or fibers [14,15]. Work by Chawla et al. showed increases in fatigue strength with decreasing particle size and higher loading [16]. There has also been an abundance of research into the benefits of CNTs addition to the fatigue life polymer [17–20]. While simple models have shown the potential for CNTs to improve MMC fatigue strength [21], there has been limited research demonstrating their potential to improve the fatigue strength of MMCs. Empirical measurements of CNTs integrated into the metal matrix have shown the ability to improve both axial and bending fatigue strengths in the high cycle fatigue regime [22,23]. These improvements were attributed to the additives ability to stabilize slip planes thereby limiting deformation and preventing cracking. There are two major issues preventing the full potential of CNTs in reinforcing a MMC: (1) poor CNT dispersion and (2) poor adhesion between the CNT and surrounding matrix [24]. Evenly dispersing CNTs in MMCs is a challenge due to the high van der Waals forces present between CNTs that tends to cause them to aggregate [25–27]. Aggregated CNTs in a MMC then lead to an increase in void spaces that can induce dislocations and lead to increased slip. Improving the fatigue life of MMCs requires reducing or preventing slip from occurring. To combat these effects, there are a few methods commonly used to improve the dispersion and bonding of CNTs in an MMC: mechanical alloying, surface functionalization, and chemical treatments [24]. Each method has benefits and detriments that affect the overall performance of the resulting MMC. This paper presents an alternative approach to improving the performance of CNT-based MMCs by distributing metal nanoparticles on the surface of the CNT prior to dispersion in metal matrix.

Metal-coated CNTs have been investigated for their ability to improve composite strength, electrical, and thermal properties and compared to uncoated CNTs. By metal coating the CNT prior to composite formation, there is the potential to improve matrix/CNT interfacial interactions. Work
by Nie et al. showed increased thermal conductivity in Cu composites with the incorporation of tungsten-coated CNTs whereas there was no improvement in thermal conductivity on incorporating uncoated CNTs [28]. Traditionally, coating CNTs is performed via vapor phase processes, such as chemical vapor deposition or atomic layer deposition; however, these processes have limited throughput or potential for scalability.

Large-batch solution-based coating of CNTs requires suspending individual or small bundles to inhibit aggregation. Most common solvents do not provide sufficient solvation forces to suspend individual CNTs [29]. Therefore, CNTs are often dispersed in aqueous suspensions with the aid of surfactants [30]. Most aqueous dispersions use sodium dodecyl sulfate (SDS), sodium dodecylbenzene sulfonate (SDBS), and sodium cholate, although other surfactants have been investigated [31–34]. After suspending the CNTs in water, immiscible organic solvents can then be added to swell the hydrophobic core of surfactant micelles surrounding carbon nanotubes to yield an emulsion-like environment surrounding the CNTs [35]. Recent work has shown that mixing surfactant-suspended CNTs with an oil phase creates an emulsion-like phase surrounding the nanotubes as the solvent localizes in the oleophilic center of the surfactant micelle [35–37]. By dissolving reactive metal salts in the oil phase, it is possible to perform interfacial reactions to reduce the metal, resulting in NPs or uniform coatings on the CNT surface [37].

This technique has been shown to be useful for coating CNTs with nylon shells that could be dried and re-dispersed in aqueous solutions without problematic aggregation. In this work, a highly-scalable, solution-based reaction to metal-coat or metal-decorate suspended CNTs is presented together with its effect on the high cycle fatigue (HCF) behavior of CNT reinforced steel composites.

Materials and methods

The metal coating process is shown in Figure 2. Aqueous, suspended SWNTs were purchased from Nanolab, Inc. (Waltham, MA). The suspension consisted of 1.87 g/L SWNT (1.5 nm diameter, 1–5 \( \mu \)m length) with 100 g/L SDBS. A representation of a single CNT suspended in a micelle is shown in Figure 2(a). Iron acetylacetonate (Sigma-Aldrich, 97%) (\( \text{Fe(C}_5\text{H}_7\text{O}_2\text{)}_3 \); Fe(acac)) was dissolved in chloroform (Sigma Aldrich, Anhydrous, \( \geq 99\% \)) at 53 g/L. Equal volumes of the aqueous CNT suspension and chloroform mixture were mixed to swell the oleophilic region of the surfactant micelle, as shown in Figure 2(b). The mixture was then allowed to settle for 24 h and phase separated into the less-dense swelled CNT suspension and the excess chloroform, which was removed using a pipette. After separation, aqueous hydrazine (\( \text{N}_2\text{H}_4 \); Sigma Aldrich, 35% in \( \text{H}_2\text{O} \)) was added to the CNT suspension in a 7:1 molar ratio (hydrazine: Fe(acac)), as shown in Figure 2(c). The hydrazine reduced the Fe(acac) to metallic Fe at the micelle
interface, forming Fe NPs on the surface of the CNT as shown in Figure 2(d). After the Fe(acac) was completely reduced, an equal volume of methanol was added to the suspension to strip the surfactant and collapse the micelle. This step left the coated CNTs in an aqueous solution, as shown Figure 2(e). The CNTs were then collected via centrifugation (Hermle Z300, Germany). The suspension was portioned into 50 mL centrifuge tubes and spun at 4000 rpm for 60 min. The supernatant was decanted and the wet CNTs were rinsed with 2:1 volume ratio of methanol to wet CNTs. The centrifugation was repeated, the supernatant again removed and the wet CNTs were collected and dried at 100°C. The dried CNT sediment was broken up using an agate mortar and pestle to leave a fine FeCNT powder.

Individual FeCNT MMC sample portions were prepared by adding FeCNT powder to powdered, tough high carbon 1084 steel (USA Knifemaker, course mesh) at 0.1 vol%, 0.5 vol%, or 1.0 vol% by volume, based on an accepted value of 1.4 g/cm³ for SWNTs [38]. The powders were then mixed using a Thinky ARE-310 dual axis orbital mixer. Spark plasma sintering (SPS) was performed by California Nanotechnologies (Cerritos, CA) to rapidly consolidate the powders into solid samples. The SPS was performed at 80 MPa with a 200°C/ min ramp to 800°C, which was then held constant for 15 min. California Nanotechnologies then machined the sintered solids using electrical discharge machining to form dog bone shapes according to the respective ASTM standard for each tensile strength and high-cycle fatigue strength testing. Element Materials Technology performed tensile testing according to ASTM E8 for tensile strength and ASTM E466 for high cycle fatigue testing. Element Materials Technology performed tensile testing according to ASTM E8 for tensile strength and ASTM E466 for high cycle fatigue testing. The SPS was performed at 80 MPa with a 200°C/min ramp to 800°C, which was then held constant for 15 min. California Nanotechnologies then machined the sintered solids using electrical discharge machining to form dog bone shapes according to the respective ASTM standard for each tensile strength and high-cycle fatigue strength testing. Element Materials Technology performed tensile testing according to ASTM E8 for tensile strength and ASTM E466 for high cycle fatigue testing. Tensile tests were performed at a constant 1 mm/min cross head rate. Fatigue testing was also performed by Element Materials Technology at 30 Hz with stress limited set points of 214 MPa and -107 MPa for a total stress range of 321 MPa. A five million cycle limit was set for the fatigue testing. The number of samples tested at each condition is shown in Table 1. Metal-coated CNT samples were analyzed using scanning electron microscopy (AMRAY 1845) and with EDX (E2V Scientific Instruments silicon drift detector 874-8381A, 20 keV excitation, 60 s integration). Additional analysis was performed by Eurofins Materials Science (Sunnyvale, CA) via transmission electron microscopy (TEM, FEI Tecnai TF-20, 200 kV bright-field and high-angle annular dark field) with built in energy dispersive X-ray spectroscopy (EDX, FEI Talos with Bruker ChemiSTEM, 200 kV excitation) investigate the coating.

Results and discussion

Representative EDX spectra of a bundle of CNTs before and after coating are shown in Figure 3. The spectra were normalized to the C peak (0.28 keV) and show negligible Fe (6.4 keV, 7.06 keV) in the powder prior to coating but a distinct Ni peak (7.47 keV), indicating that Ni is a remnant from CNT synthesis. The Al (1.49 keV) and S (2.31 keV) peaks are from the SEM stub and residual surfactant, respectively. Elemental analysis, shown in Table 2, indicated that Fe content of the powders increased from 1 to 11.9 at% while Ni stayed constant at 2.2–2.4 at%. An elemental map of coated CNTs is shown in Figure 4. The map shows a strong presence of Fe distributed throughout the CNT bundle along with oxygen, which is likely due to oxidation of the Fe particles.
To further explore how the Fe reduction interacts with the CNTs, we performed TEM analysis on smaller CNTs bundles after Fe decoration. This analysis showed localized Fe NPs decorating the CNTs at inconsistent intervals, as shown in Figure 5. The Fe particles appear to be 10–30 nm across with spacing from 40–100 nm between particles. Particle edges are irregular but the particles appear to form to the curvature of the CNT. It further appears that there is a large bulk of particles agglomerated in the area between the CNTs, although at this time it is not possible to determine if they formed during the coating process or the drying process.

Testing with virgin 1084 steel powder, processed in the same manner as the FeCNT treated parts, showed an average fatigue to failure of $7.2 \times 10^4$ cycles, with a low $9 \times 10^3$ standard deviation and no samples reaching the $5 \times 10^7$ cycle runout limit, or

Figure 4. (A) TEM image of a bundle of FeCNTs and EDX elemental maps for (B) C, (C) O, (D) Fe, and (E) Ni. The O signal is a result of Fe oxidation while Ni corresponds to remaining catalyst particles trapped in the CNTs.

Figure 5. TEM images of CNT bundles (tubular shapes) with Fe decoration on the surfaces (bright spots).
maximum number of cycles before testing was stopped. Integrating FeCNTs into the 1084 steel led to as much as a 376% improvement in the average fatigue life. Samples containing 0.1 vol%, 0.5 vol%, and 1.0 vol% FeCNT had $3.1 \times 10^5$, $2.4 \times 10^5$, and $3.4 \times 10^5$ cycles to failure, respectively. The results are shown in Figure 6. The samples produced using uncoated CNTs in the composite had lower axial fatigue life, which was commensurate with the plain 1084 steel. These samples had cycles to failure of $8.3 \times 10^4$ and $1.5 \times 10^5$ for 0.1 vol% and 0.5 vol%, respectively. The 0.1 vol% CNT addition only had one sample not reaching failure (runout) conditions and thus we cannot draw significant insight from this singular result; though the data point is provided in the interest of full disclosure. While the untreated CNT loaded samples show an improvement from the plain 1084 steel, there is a drop-off from the FeCNT composite.

The cycles to failure did not appear to show a specific trend with regard to the loading of FeCNTs in 1084 steel and a significant standard deviation was associated with FeCNT composites. The 0.1 vol%, 0.5 vol% and 1.0 vol% composites showed standard deviations of $1.8 \times 10^5$, $1.2 \times 10^5$, and $1.8 \times 10^5$, respectively, which is approximately a 50% deviation. This error is extremely high, although we believe it to be a function of inconsistent FeCNT distribution within the composite. Further evidence of the potential for FeCNT distribution to drive inconsistent fatigue life is seen by the fact that more than 40% of all samples reached the $5 \times 10^7$ cycle runout condition, as shown in Figure 7. This suggests that some of these MMCs have well-dispersed CNTs and provide exceptional performance. The increased number of samples to reach the runout condition at 0.10 vol% CNT (no Fe) is likely an artifact from the limited amount of testing which was able to be performed at these conditions. We feel that if a larger number of samples were to be tested, the number of samples to reach runout conditions at 0.10 vol% CNT (no Fe) would fall more closely in line with the other materials which showed a negligible effect with respect to CNT loading.

It is possible that the samples which reached the runout condition have improved CNT dispersion leading to an increase in dislocation pinning. In many cases, particulate reinforcement in MMCs refines the grain structure surrounding the reinforcement and reduces plastic deformation...
during strain events; thereby increasing the composite fatigue strength in comparison to the monophasic metal [39]. There is evidence that many fatigue failures initiate at dislocation and/or stress accumulation sites within the grain structure of the material [39-41]. Under cyclic loading and unloading, these dislocations and stress accumulations form the basis for crack initiation and propagation [41,42]. When integrating a reinforcing additive into a metal, the risk of vacancies around the reinforcing material increases and the applied stress further exacerbates the dislocation formation at these sites [43]. Balancing these dislocation risks with the improved grain stabilization seen in reinforced material is critical to maximizing the fatigue life of the composite. These results indicate that the NP decoration on the CNT surface measurably improves the fatigue strength of the FeCNT composite in comparison to the untreated CNT composite.

Ultimate tensile strength (UTS) and Young’s modulus (YM) were also tested at 0.1 vol%, 0.5 vol%, and 1.0 vol% FeCNT, along with sintered virgin 1084 steel. Samples were prepped under the same conditions and milled into the necessary dog bone shape via electron discharge machining. Samples of sintered 1084 steel without any FeCNT or CNT addition showed a UTS of 508 MPa, approximately 23% lower than the literature UTS literature value of 820 MPa. The sintered plain steel also showed a YM of 167 GPa, just 16% lower than the accepted 200 GPa. Adding 0.1 vol%, 0.5 vol%, and 1.0 vol% FeCNTs to the steel matrix reduced the UTS of the steel by approximately 0%, 27%, and 56% respectively. The YM was reduced by 9%, 19%, and 20%, respectively. The results of the tensile testing are shown in Figure 8. The resulting decrease in UTS is contradictory to similar analysis of CNT reinforced MMCs which have shown up to 30% increases in UTS [44-46], though the cause of the observed decrease is not understood at this time. There is a similar large deviation in tensile strength results as seen in the fatigue results which may be attributed to inconsistent distribution of CNTs throughout the samples.

**Conclusions**

Integrating CNTs into MMCs is one method of producing high fatigue life composites. However, traditionally processed CNTs face issues with aggregation and poor interfacial bonding with the metal matrix that inhibits their maximum reinforcing efficiency. In this paper, we have demonstrated a method based on decorating CNT surfaces with metallic NPs by reducing a metal salt in a well-controlled water-solvent interface around surfactant-suspended CNTs. These FeCNTs can then be mixed with metal powders and sintered to form MMCs. Axial fatigue life testing showed up to a 376% improvement for composites containing 1.0 vol% FeCNTs vs 150% improvement from the addition of untreated CNTs. Adding FeCNTs caused a linear reduction in the tensile strength of the composite up to 66% at 1.0 vol% FeCNTs. A significant amount of variability was found in the fatigue and tensile strength data likely due to inconsistent mixing of the CNTs in the composite. The variability is further demonstrated by the number of samples (>40% of samples tested) that reached the runout condition. Ultimately, this work shows the potential to further improve the high-cycle fatigue strength of CNT-reinforced MMCs by decorating the surface with metallic NPs. This decoration technique can be extended to other metals by varying the metal salt and reductant.

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**Disclosure statement**

The authors declare no competing interests in relationship to this work.

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**Anthony Villa-Garcia** was an undergraduate researcher working with the University of Florida and Mainstream Engineering on a joint project for the application of coating carbon nanotubes with metals, specifically single walled nanotubes (SWNT). He is best known for having conducted iterative, experimental processing changes to successfully coat SWNTs with copper, which was confirmed with Electron Microscopy (S/TEM). Currently he works on product innovation and formulation...
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