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Carbon Nanotube Aluminum Matrix Composites

by Brent J. Carey, Jerome T. Tzeng, and Shashi Karna

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14. ABSTRACT

The material demands of advanced weapons technologies have pushed the limits of available metals and alloys, emphasizing the need for next-generation lightweight, multi-functional materials. Carbon nanotubes (CNTs), whose tensile strength and thermal and electrical conductivity along their axis exceedingly surpass virtually all known materials, have been shown to provide marked increases in the mechanical strength and hardness of metal matrix composites (MMCs). Here, we explore the viability of a novel CNT MMC: a vertically aligned array (forest) of CNTs infiltrated with aluminum to achieve an anisotropic, continuous fiber composite. The application of a sputter-coated layer of aluminum on the CNTs resulted in the encapsulation of all exposed nanotubes for light deposition and the formation of a ~1-µm-thick composite layer for longer deposition, a promising result which strongly suggests an affinity between the two materials. This observation highlights the potential for non-destructively impregnating the CNT forests with aluminum, a technique demonstrated previously with a polymer matrix. Additionally, preliminary electrical testing and further aluminum deposition revealed novel behavior, which shows promise for utilization of these nanocomposites for other applications such as catalysis and as compliant, highly conductive electrodes in nanoscale devices. Upon refinement of the infiltration procedure, these composites should significantly improve upon already-existing materials for applications where greater structural integrity, thermal diffusivity, and high electrical conductance are desired.

15. SUBJECT TERMS

carbon nanotube, composite, metal matrix

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Contents

List of Figures

1. Introduction/Background

Since their discovery in the early 1990's (*1*), researchers have strived to exploit the remarkable mechanical, thermal, and electrical properties of carbon nanotubes (CNTs) in a number of diverse applications ranging from nanoscale transistors (*2*) and interconnects (*3*), supercapacitors (*4*), artificial muscle fibers (*5*), and even as axles in molecular vehicles (*6*). While a number of these innovations are still years or perhaps even decades from wide-scale implementation, recent advances in nanotube synthesis (*7*) have afforded the opportunity for the inclusion of CNTs to improve upon current technologies.

Sliding electrical contacts are essential in devices such as electrical motors, though their utility extends to a litany of other applications where electrical conduction between moving parts is necessary. In an effort to improve upon the carbon composite and filamentous metal brushes currently used as sliding contacts in high-performance applications, it has recently been shown that due to the excellent electrical conductance along their long axis, "forests" of CNTs grown via chemical vapor deposition (CVD) can serve as compliant nanoscale brushes which show almost an order-of-magnitude lower contact resistance as compared to these traditional technologies (*8*). Additionally, the excellent thermal conductance along the CNTs can allow these forests to serve as a heat sink that can draw thermal energy away from a sliding interface, a property exploited previously to utilize these structures as highly efficient cooling fins for modern electronics (*9*). A downside, however, is the susceptibility of these arrays to plastically deform under even modest stress. As such, a reinforcing matrix is necessary for these nanoscale bristles to be practical as robust sliding contacts.

By the way of composite materials, the contribution CNTs offer to mechanical strength has been seen predominantly with the reinforcement of glassy polymer matrices such as epoxies, which see notable improvement for even modest concentrations of nanotubes mixed into the matrix. For these composites, CNTs have been successful as both the sole reinforcing phase (*10*), as well as a supplement to carbon fibers (*11*). However, an optimal configuration for reinforcement in the development of strong composites is fibers that are evenly-spaced and which span the entirety of the composite. The ability to induce such an orientation with carbon nanotubes has proven to be particularly challenging due to their extremely small size, high aspect ratio, and affinity to cling to each other. Recently, it has been shown that the inter-tube spaces between these CVD-grown forests of CNTs can be readily infiltrated by a poly(dimethylsiloxane) monomer and cured *in situ* (figure 1) (*12*). The resultant continuously-reinforced composite offers a three-fold improvement as compared to randomly-aligned composites with identical CNT loading, a property owed to the unrivaled tensile strength inherent to carbon nanotubes (*13*). For a composite which has optimal conductivity, however, a conducting matrix would be necessary.

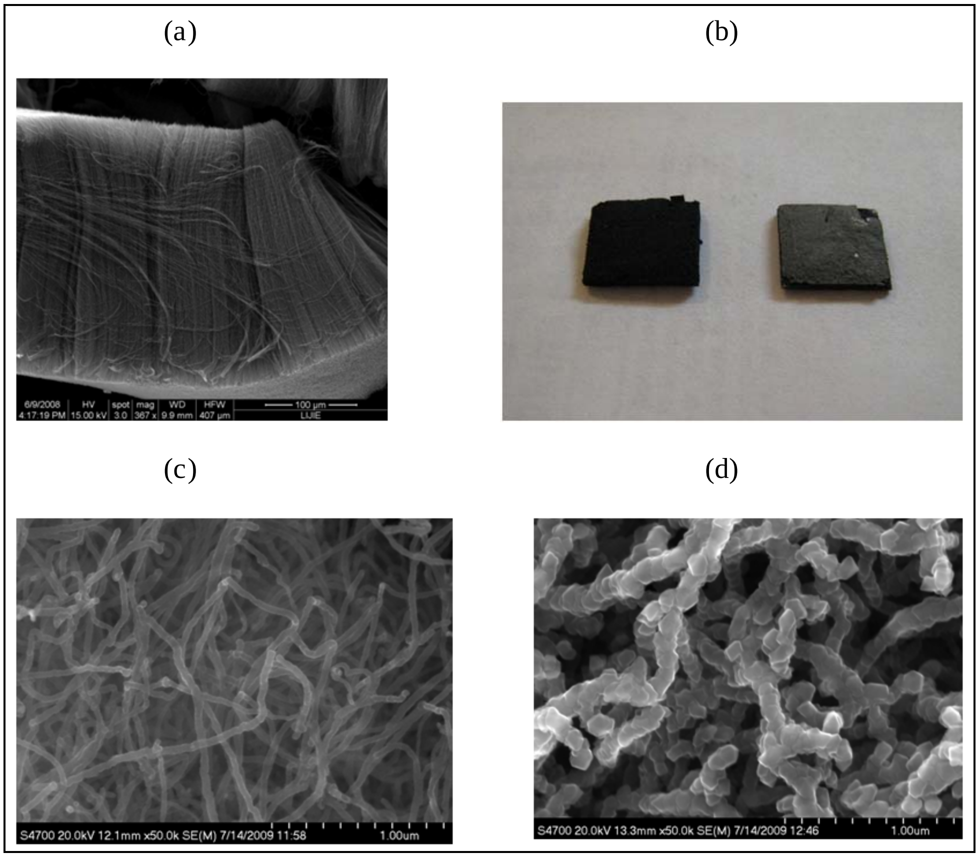

Figure 1. Characterizing the interaction between carbon nanotubes and aluminum.

Figure 1 shows: (a) the “forests” of CNTs synthesized via chemical vapor deposition (CVD) can be grown to macroscopic lengths on the order of millimeters and (b) the optical difference between the pristine and aluminum-coated forest. (c) This SEM image shows that the top of this CNT forest exists as somewhat of a tangled nest and that the CNTs measure between ~30–80 nm in diameter. (d) It can be seen that after sputter coating 1 µm of aluminum on the CNTs, they are sheathed in ~25–30 nm of aluminum. This strongly suggests good interaction (wetting) and is promising for the investigation of impregnation with aluminum to create a continuous CNT metal matrix composite. Additionally, the morphology of the coating is not smooth like the surface of the CNTs. The distinct edges indicate the nucleation of metal crystals on the CNTs, further confirming the mobility of the sputtered aluminum.

The concept of a metal matrix composite (MMC) isn’t new, though the processing challenges as compared to their polymer matrix analogs has impeded their advancement somewhat thus far. Even for MMCs reinforced by microscopic fibers, it is nontrivial to implement anything other than basic architectures. This problem is compounded for nanomaterials such as CNTs, where homogeneous dispersion (not to mention any form of arrangement or alignment) remains a very challenging problem. While little information exists about how the electrical and thermal behavior is altered as a result of the presence of the CNTs, a number of studies using various forms of forced dispersion such as hot extrusion (*14*) and ball mixing (*15*) have shown CNTs to be effective as structural reinforcement in a MMC. Conversely, while the level of homogeneity and the integrity of the nanoparticles after this type of processing is contested, nanoscale engineering techniques have resulted in truly homogenous dispersion providing up to a 78% increase in hardness with just 5 weight-percent CNTs dispersed in aluminum (*16*); unfortunately, the practicality of methods such as this is are suspect due to the relative expense and limited scalability.

There exist a number of U.S. Army applications which would benefit significantly from such a multifunctional CNT-reinforced MMC if an efficient and scalable process can be devised. In this work, we strive to implement these remarkable molecules as constituents in an aluminum MMC to impart mechanical, thermal, and electrical improvement over the pure metal. To this end, we have explored a method to produce an anisotropic, vertically aligned CNT MMC which could serve as a highly conducting, robust sliding contact. Through infiltration of a metal matrix into a forest of CNTs, the homogeneity is assured and the potential of damaging the CNTs during production, a significant concern of most CNT/metal matrix processing techniques, should be eliminated. Furthermore, the CVD method of nanotube synthesis is inexpensive and highly scalable, offering the potential for affordable application to both current and future technologies.

2. Experiment/Calculations

The production of the aligned, polymer matrix nanotube composites is possible due to the incredible wetting between the CNT surface and the PDMS polymer chains (~0° wetting angle) (*17*). This property allows for the spontaneous infiltration and replacement of air space with the polymer matrix. A similar affinity is not known to exist between CNTs and aluminum, where the wetting angle between these two materials is reported to be a discouraging 130°–140°, making infiltration unlikely (*18*). However, in this work by Ci et al. (*18*), they also report that the loosely-bound amorphous/pyrolized carbon coating on the CNTs grown by chemical vapor deposition (CVD) will react with aluminum at high temperatures to form aluminum carbide (Al_4C_3), a byproduct which is typically unfavorable due to its brittleness. In this case, however, the 50°–70° contact angle between Al and Al_4C_3 should significantly increase the probability of infiltration by improving the surface wetting, and also will enhance interfacial load transfer for the resultant composite (*19*).

To test the viability of this infiltration, the exposed ends of the CNTs were coated with increasingly-thicker layers of aluminum via sputter coating, after which the wetting behavior was visually analyzed. CVD-grown, vertically aligned multi-walled carbon nanotubes (figure 1) provided by Rice University (Houston, TX) served as the infrastructure for this continuously-reinforced MMC. The aluminum was sputtered at thicknesses of approximately 1, 5, and 10 µm using a 99.99% pure aluminum target ~4.5 in away from the samples at 120 W and under a continuous flow of argon at 1 mTorr. To gauge the viability of infiltrating further via heat treatment, previously-coated samples were subjected to 700 °C in 10^{-5} mTorr for 2 h. To observe the deposited layers and any morphological changes due to the heat treatment, the samples were imaged using a Hitachi S-4700 field emission scanning electron microscope at 20 kV in secondary electron mode.

To characterize the electrical properties of these aluminum-coated forests, two-probe conductance measurements were conducted on the samples using a 50-µm-diameter platinum pin as the source through the through-thickness direction of the forests to a brass substrate as the drain. A voltage of ±2 V was applied, and the subsequent current was observed as a function of the applied voltage. Voltages over 2 V began to degrade the samples, which caused the aluminum to coalesce and agglomerate the CNTs in the process.

3. Results and Discussion

3.1 Impregnated Composite Viability

It can be seen in the scanning electron microscopy (SEM) images in figure 1d that the ~1 µm of sputtered aluminum on both the tips and the sidewalls of the aligned CNTs resulted in a ~25–30 nm coating on each individual CNT. This is clearest in figure 2a and b, which show a view orthogonal to the deposited surface and into a small void in the coated surface, respectively; here, two observations can be made: (1) the single CNT protruding from the surface is surrounded by the coating, and (2) the deposition penetrated the surface layer of CNTs and coated the CNTs underneath the top surface. These results indicate that the deposition didn't simply occur in the "line-of-sight" from the target to the specimens, as is typical of sputter coating, but instead that the aluminum was mobile during deposition and coated the CNTs as it penetrated the forest. This suggests that the aluminum has an affinity for the CNTs (perhaps via the formation of Al_4C_3), which makes impregnation through infiltration a distinct possibility.

Another interesting observation is the fact that the coating isn't smooth like the surface of the CNTs it surrounds. In figure 1d this is clearly seen, and there appears to be distinct edges to the raised areas of the coating, hinting at the formation of aluminum crystals on the surface of the CNTs. After heat treatment at 700 °C for 2 h in vacuum, the morphology of the coating appears to have smoothed and lost its faceted edges (figure 2c). Closer inspection reveals that the surface has become nanoscopically rough (figure 2d), which is presumably due to the molten aluminum breaking up the oxidized coating which formed when the specimen was removed from the chamber after sputtering.

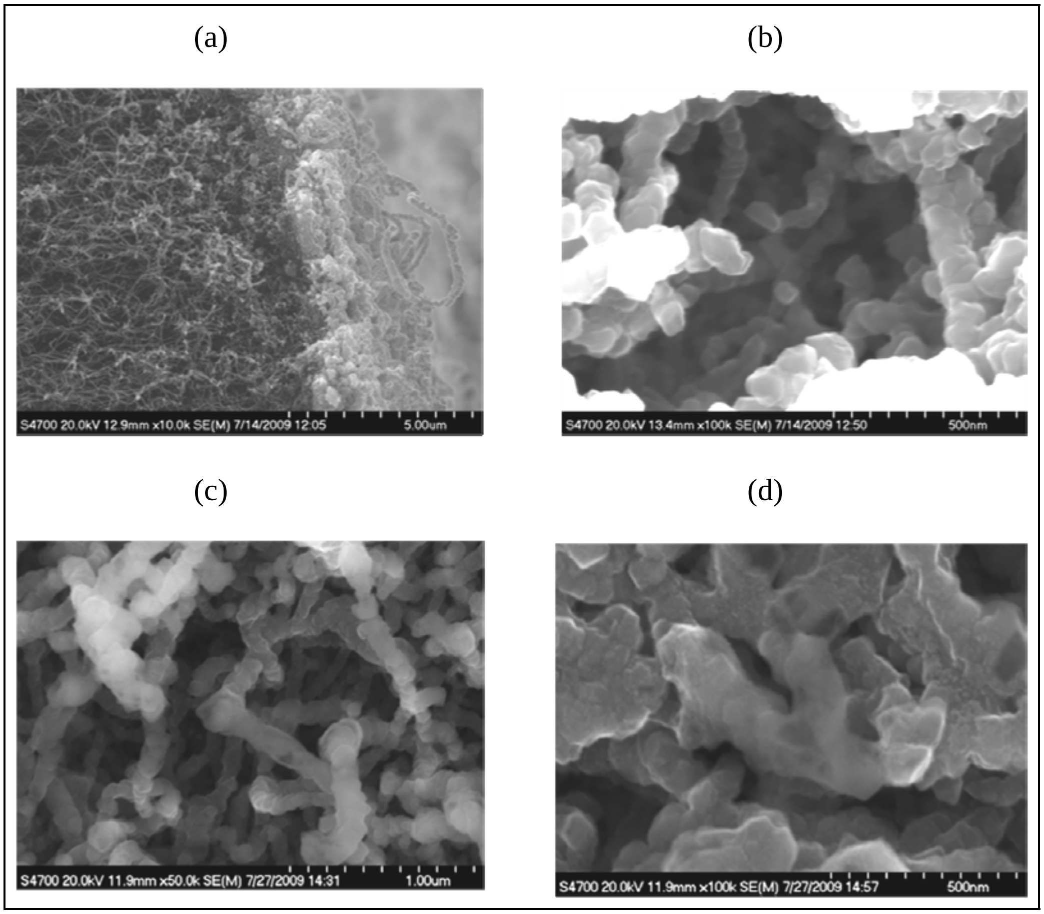

Figure 2. Surface mobility of aluminum during sputtering, and the subsequent effects of heat treatment.

To gauge the viability of sputtering as a means to impregnate a forest of CNTs, the amount of deposited aluminum was increased, and thicknesses of 5 and 10 μm were applied. A piece of the 10-μm-coated forest of CNTs was laid down to image the top surface of the forest. It can be clearly seen in figure 3a that there exists a transitional region where the CNTs on the top of the forest have been coated. Closer inspection (figure 3b) reveals that in this region, there is full impregnation of the sputtered aluminum, a layer ~1 μm thick. The CNTs below this region to a depth of a few microns have a coating of aluminum as predicted by the "diffusing" of the aluminum through the forest, but as aluminum was continually deposited it began to coalesce and saturate the uppermost part of the forest. This observation confirms the possibility of achieving a fully-impregnated carbon-nanotube-reinforced aluminum matrix composite.

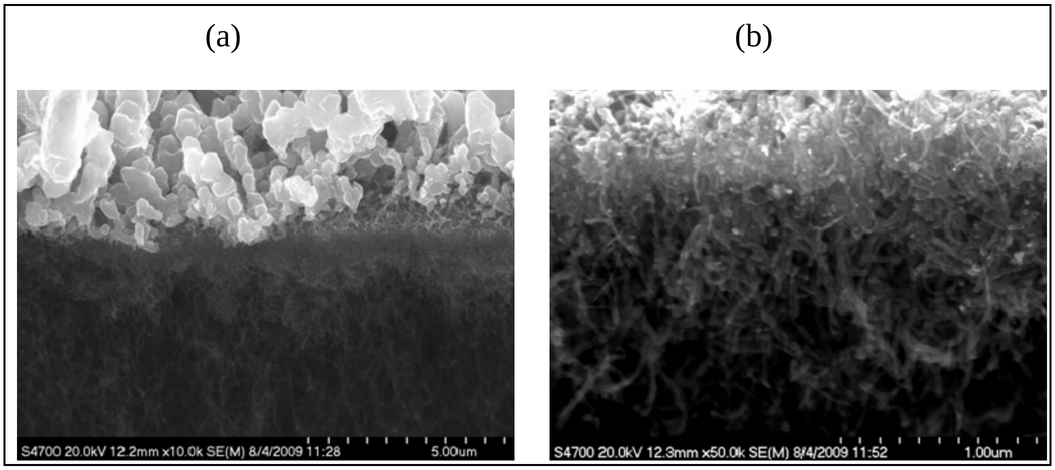

Figure 3. Confirming the viability of a fully-impregnated nanotube-reinforced aluminum matrix composite.

3.2 Crystal Growth

The surface of these coated forests offers additional insight into the deposition. For the 5-μm deposition (figure 4a and b), it can be seen that the CNTs are fully covered by the aluminum coating and are no longer visible. Curiously, the coated surface is not smooth, but is instead covered by ~500-nm crystals with very well-defined edges and facets which have nucleated off of the surface of the CNTs. This effect is amplified for the 10-μm deposition (figure 4c and d), where the crystals have grown in size to ~1 μm and have begun to coalesce. For this sample, we also note the existence of a cuboctahedron (upper left corner of figure 4d) and other related highly symmetric polyhedric crystals. The growth of such crystals (particularly using CNTs as the nucleating agent) has not been reported for aluminum, but the structures observed agree with geometries corresponding with face-centered cubic packing, the crystal structure of aluminum. The last sputtered coating was 10 μm, deposited on a sample which had previously received 1 μm of deposition (figure 4e and f). It can be seen that this surface is much smoother due to the inability of the newly-sputtered aluminum to penetrate the oxidized-aluminum-coated CNT forest.

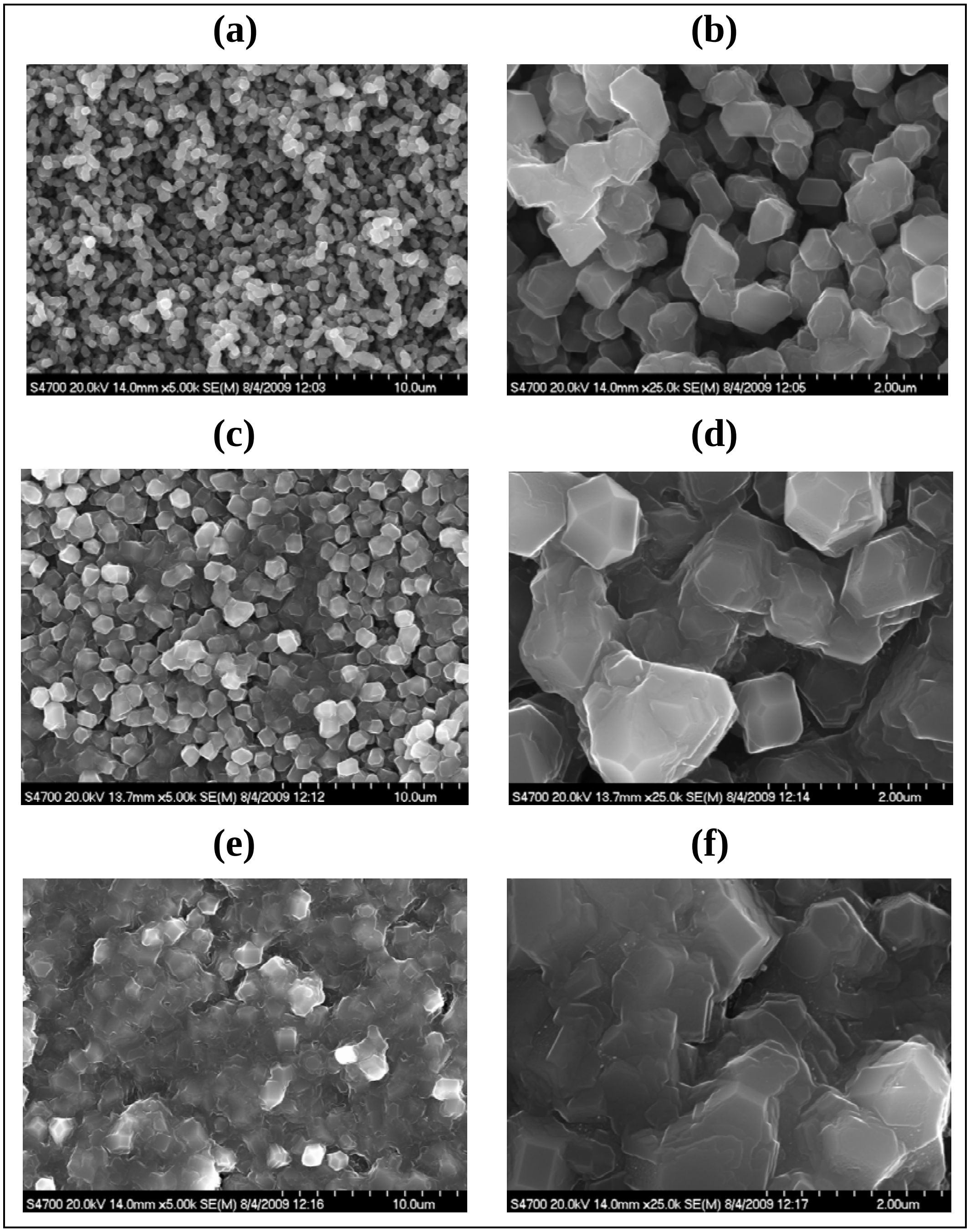

Figure 4. The effects of further aluminum deposition.

Figure 4 shows: (a, b) When scaled up to 5 µm of deposited aluminum, the CNTs are no longer visible as the surface is coated fully. On top of the CNTs, well-defined crystals of aluminum have grown, having nucleated from the surface of the CNTs. (c, d) For 10 µm of deposition, the crystals have developed very regular polyhedric shapes as they've grown in size and have begun to coalesce. (e, f) The deposition of 10 µm on top of a surface which had previously received 1 µm of deposition shows a faceted but much more even surface coating, likely due to the inability of the newly sputtered aluminum to penetrate the oxidized-aluminum-coated CNT network.

3.3 Electrical Characterization of Coated Forests

The measurement of current vs. voltage (I-V) for the pure CNT forests (figure 5a) yielded a predictable result: the forests of CNTs behave as a metal, displaying a linear I-V relationship. The behavior of the 1-µm-coated forest, however, was not as conventional, where two distinct responses were observed. Predominantly, a non-linear response was observed (figure 5b), which is indicative of a heterojunction between two dissimilar metals. Occasionally though, linear behavior was observed until a specific voltage (typically 1.6–2 V), where a quick drop in current would occur, quickly followed by a recovery of the linear behavior (figure 5c). This phenomenon is referred to as negative differential resistance (NDR), and is typically observed when there is a transition from classical conductance to electrical tunneling. This behavior has not yet been observed in CNT systems, and if this effect can be isolated, it has implications in the development of electrodes for nanoscale electronic devices.

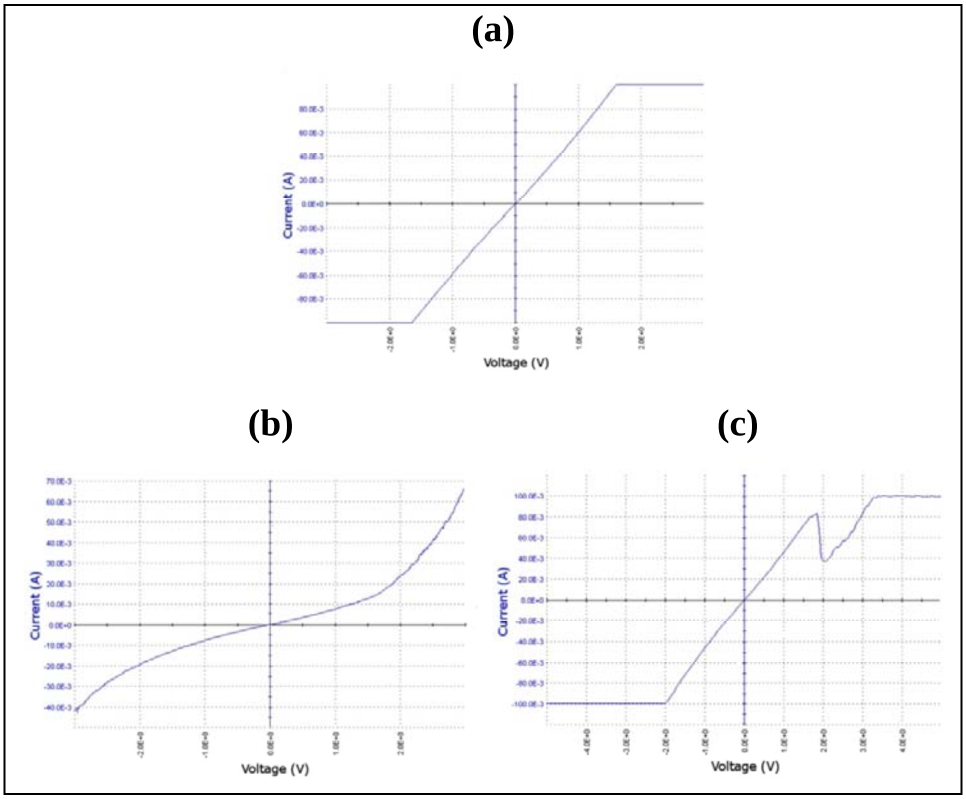

Figure 5. Electrical characterization of aluminum-coated forests.

4. Summary and Conclusions

With advanced weaponry requiring improved material performance in a lighter, less obtrusive package, engineering at a smaller scale becomes necessary. This proves to be no trivial task, and it becomes necessary to exploit self-assembly and nanoscale dispersion to achieve the homogeneity and alignment which provides the desired material properties. For a multifunctional composite, carbon nanotubes are ideal due to the remarkable blend of strength and conductivity.

In this work, we have proposed a method of producing a CNT-reinforced composite which should yield significant improvement in strength and thermal and electrical conductivity over traditional metals and alloys. The successful wetting initiated by this sputtering indicates the viability of the above infiltration mechanism, and through this deposition we’ve shown that aluminum can fully impregnate a forest of CNTs for a depth of approximately 1 μm. Further optimization of this process is necessary, and recent advances in CNT synthesis techniques should allow for scalability and implementation in both existing and future weapons technologies.

In addition to this result, two new phenomena were observed which have implications in unrelated fields and applications. The observance of the growth of nanoscale aluminum crystals could prove beneficial in the development of improved catalytic surfaces, while the existence of negative differential resistance, if isolated, shows promise for utilization in nanoscale electronic devices.

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