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13. SUPPLEMENTARY NOTES Due to Patent Dispute, PI was dismissed from IPYCyT. Final report prepared by PM					
14. ABSTRACT The project objectives are to fabricate novel composites with polymers and arrays of aligned carbon nanotubes for developing enhanced heat transfer devices. Since carbon nanotubes could exhibit extremely high thermal conductivity, aligned nanotubes embedded in polymeric matrices may be a way to generate materials that are able to remove heat, especially in radars, electronic components, and energy and power transformers. This work was focused on the production of aligned nanotube and different arrays of the latter. In particular, different types of doped (or functionalized) nanotubes such as N-doped, Si-doped, O _x H _y -doped, N _x P _y -doped, etc were used. Subsequently, these arrays embedded in polymer matrices and their thermal properties of these nanotube composites were to be tested.					
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FINAL REPORT

Polymer Composites using Aligned Carbon Nanotubes for Efficient Heat Transfer

**Contract No.
FA9550-09-1-0026**

Program Manager

James Fillerup, PE

Southern Office of Aerospace Research and Development
Air Force Office of Scientific Research
Air Force Research Laboratory
U.S. Embassy – Santiago
Santiago, Chile

e-mail: james.fillerup@afosr.af.mil

Ph: +562 330 3237 / 3139

Principal Investigator

Prof. Mauricio Terrones

Advanced Materials Department
IPICyT
Camino a la Presa San José 2055; Lomas 4a seccion
78216 San Luis Potosi, SLP
México

e-mail: mterrones@ipicyt.edu.mx

Tel: +52 (444) 834-2000, ext: 7237

Fax: +52 (444) 834-2010

web-page: <http://materials.ipicyt.edu.mx/english/>

Summary of the Research

The project objectives are to fabricate novel composites with polymers and arrays of aligned carbon nanotubes for developing enhanced heat transfer devices. Since carbon nanotubes could exhibit extremely high thermal conductivity, aligned nanotubes embedded in polymeric matrices may be a way to generate materials that are able to remove heat, especially in radars, electronic components, and energy and power transformers. This work was focused on the production of aligned nanotube and different arrays of the latter. In particular, different types of doped (or functionalized) nanotubes such as N-doped, Si-doped, O_xH_y -doped, N_xP_y -doped, etc were used. Subsequently, these arrays embedded in polymer matrices and their thermal properties of these nanotube composites were to be tested. Due to a patent dispute, the PI, Prof Terrones was dismissed from the IPICYT and the work was not successfully completed. A summary of the project status is provided below.

Objectives: *The specific goal of this project is to evaluate the thermal properties of polymer-nanotube composites using arrays of doped and undoped nanotubes. Different types of doped nanotubes planned to be tested doped. Variables to be explored include tube diameter, type of array, nanotube length, type of dopant and concentration of dopants within the tubes. We believe the thermal properties of the composites will depend strongly on these variables. The proposed studies contain the following specific aims:*

Aim 1. Patterning of Substrates using electrochemical etching techniques and Nanolithography on Silicon

Aim 2. Production and Synthesis of different aligned nanotube arrays with different dopants

Aim 3. Characterization of the Nanotubes produced.

Aim 4. Embedding by infiltration of different polymers in the nanotube arrays

Aim 5. Thermal Properties Evaluation for the different composites

Hypothesis: *The nanotube dopant composition, length and diameter appear to be important in the thermal transport. However, these properties have not been tested to the best of our knowledge. We believe that dopants (such as N, P, Si, OH) within carbon nanotubes will have a strong influence in the thermal properties. Therefore, polymer composites using arrays of aligned nanotubes with different dimensions and dopant stoichiometries will result in enhanced heat dissipators, useful in various applications for the Aviation and Missile Research, Development and Engineering Center (AMRDEC) and the Air Force Research Lab (AFRL) Materials and Manufacturing Directorate.*

Introduction:

The thermal conductivity of carbon nanotubes (dominated by phonons), along the tube axis, may be one of the highest ever when compared to other materials. It is important to note that the thermal conductivity of diamond and graphite (in-plane) is extremely high. In this context, Ruoff and Lorents were the first to discuss this possibility [1]. In the case of graphite, phonons dominate the specific heat above 20 K [2], whereas in SWNTs and MWNTs the phonon contribution governs at all temperatures [3]. Yi et al. [4] found that thermal conductivity k varies linearly with temperature from 4 to 300 K in MWNTs. Due to the large diameter of the tubes, it is expected that they behave as 2D graphite, and indeed k increases following T^2 , behaviour similar to that of graphite ($T^{2.3}$). These authors also realized that the thermal conductivity of MWNTs at room temperature is comparable to that observed in non-crystalline carbon fibres, due to the small crystal size in the graphitic domains [4]. McEuen's group was the first to determine the thermal conductivity of individual MWNTs, which is higher than that of graphite (3000 W K⁻¹) at room temperature, and two orders of magnitude higher than values obtained for bulk MWNT mats [5]. Here, the phonon mean free path is 500 nm, and the temperature dependence of the thermal

conductivity exhibits a 'peak' at 320 K due to the onset of Umklapp phonon scattering [5]. The thermoelectric power shows an expected linear T dependence, which was totally absent in previous bulk nanotube studies. Smalley's group performed thermal conductivity measurements on bulk samples of SWNTs [6]. The results show a different temperature dependence of k , thus implying the presence of smaller crystalline sizes within the graphitic domains when compared to MWNTs. Hone, et al. [3] measured the thermal conductivity of nanotubes and found that mats of randomly oriented SWNTs exhibited values of ca. $35 \text{ W m}^{-1} \text{ K}^{-1}$, whereas aligned SWNTs exhibit thermal conductivities $>200 \text{ W m}^{-1} \text{ K}^{-1}$.

Novel properties of carbon nanotubes continue to be observed, such as the ignition of SWNTs when exposed to a standard photographic flash [7]. In these experiments, it is believed that the ignition and burning occurs when local increases in temperature are sufficient to initiate the oxidation of the carbon, which propagates as more heat is released by this exothermic reaction. Since the thermal conductivity of nanotubes along the tube axes is very high, the heat pulse generated by the absorption of flashlight will initially be confined to the tubes within a bundle, especially along their axes. The high energy densities, necessary for ignition, are easily attained when the bundles are separated, surrounded by oxygen, and the heat wave is locally confined in the nanotube structures. However, further studies on MWNTs and SWNTs produced using different synthetic techniques need to be carried out in the near future in order to fully elucidate the mechanisms governing their thermal properties.

In this context, the fabrication of aligned nanotube arrays in polymeric matrices could be a way to evaluate further the thermal properties of nanotubes. It is believed that due to their high thermal conductivity of nanotubes, the composite could behave as heat dissipators.

It is important to note that our group was the pioneer in the synthesis of N-doped nanotubes and has work on their applications more than 10 years ago. In addition, we are able to produce different types of novel doped tubes containing different dopants such as P, Si, OH, etc. We have also been successful in using electrochemical techniques to create various patterns of aligned nanotubes arrays that could be used in designing heat dissipation devices. In addition, we have been able to grow 5mm long aligned nanotube arrays of specific diameters that could need to be tested for thermal applications. It is possible that the dopants could increase the phonons that could aid thermal conduction.

Detailed Program

1. PRODUCTION OF ALIGNED NANOTUBE ARRAYS (to be carried out at IPICYT)

1.1 Patterning of Substrates using electrochemical etching techniques and Nanolithography on Silicon

We will use electrochemical HF-etching on Si substrates to generate patterns of SiO_x on the Si substrate, where nanotubes could grow selectively. We have noted that during etching, the creation of H_2 bubbles could be controlled by changing the anodization current and the HF solution. The combination of the current and the way the electrolyte flows on the substrate results in the formation of SiO_x -rich micropatterns with different shapes and sizes (Fig. 1). These micropatterns are responsible for producing fascinating morphologies of aligned CN_x MWNTs arrays after a chemical vapor deposition (CVD) process (see details on CVD process below).

1.2 Bulk production of CN_x nanotubes for generating polymer composites

We will use the CVD processes to produce CN_x tubes of different lengths and diameters using the Si substrates treated with the techniques mentioned in point 1.1. In particular, we will use an ultrasonic sprayer system (Figs. 2 & 3). We will thermolyze ferrocene-benzylamine solutions at 850°C under an Ar atmosphere. The material produced planned to be then used for impregnating the nanotube arrays with different polymers to produce composites that could be tested for thermal transport applications.

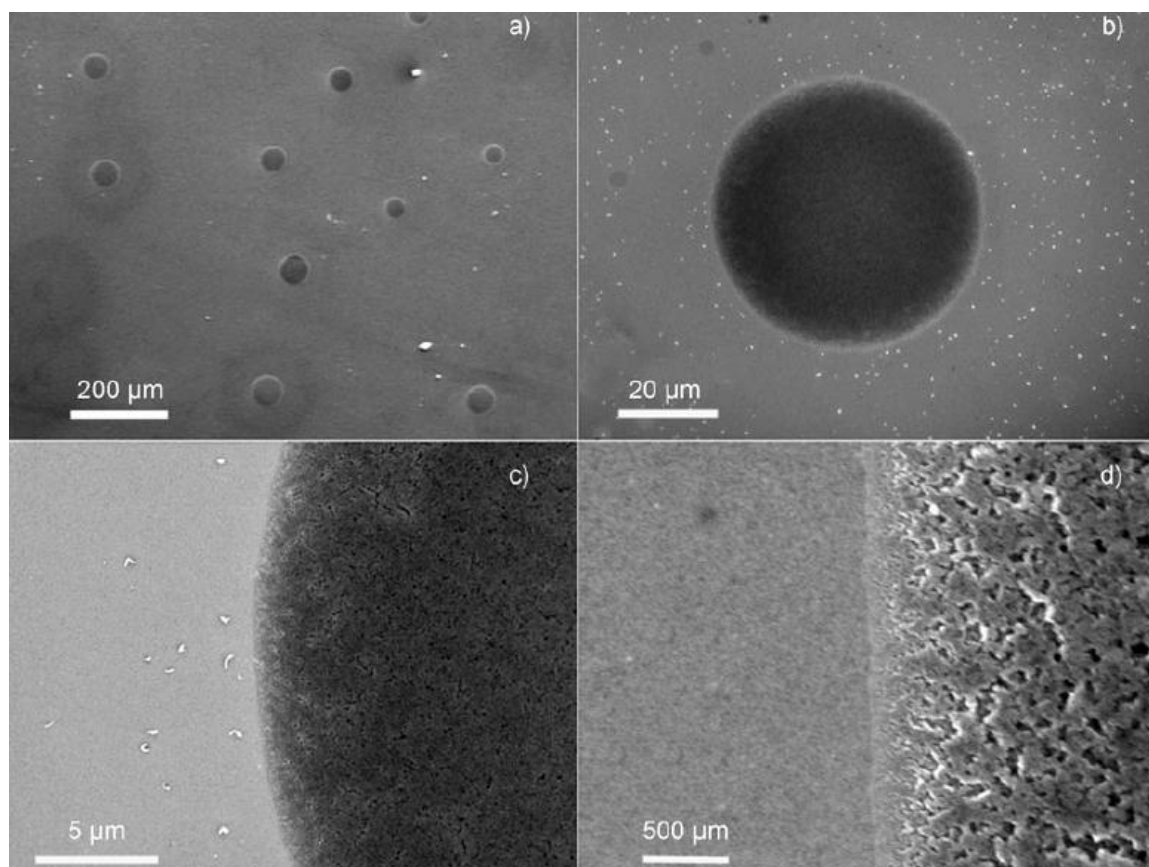


Fig. 1 SEM images of circular micropatterns formed on the etched porous silicon surface by using a current density of 4 mAcm^{-2} for 300 s, with the electrolyte circulating through the peristaltic pump and striking the Si wafer directly. a) Moderate-resolution image showing the micropatterns with diameters of approximately 40–60 μm , separated by 200–300 μm ; b) salt crystallites are absent from the center of the circular (darker region) microdot, but relatively tiny nanoparticles are distributed on the outside surface (brighter dots); c) and d) higher magnification images of edges from the circular micropatterns.

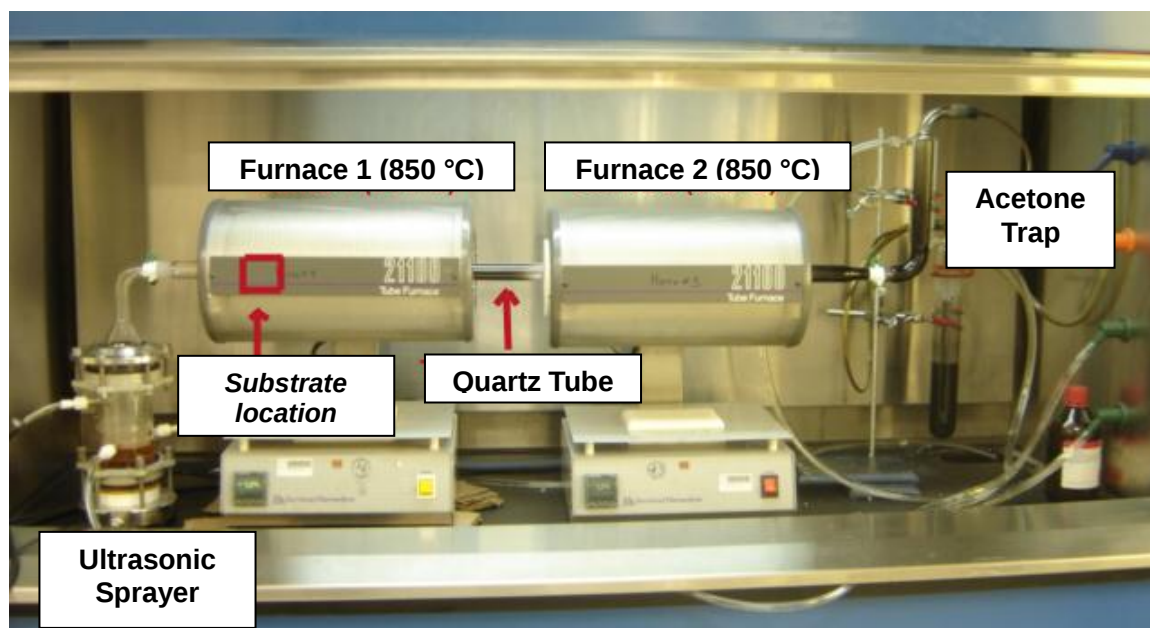


Fig. 2 Ultrasonic sprayer and a two-furnace system that planned to be used to produce the aligned nanotube arrays on etched Si and SiO_x substrates.

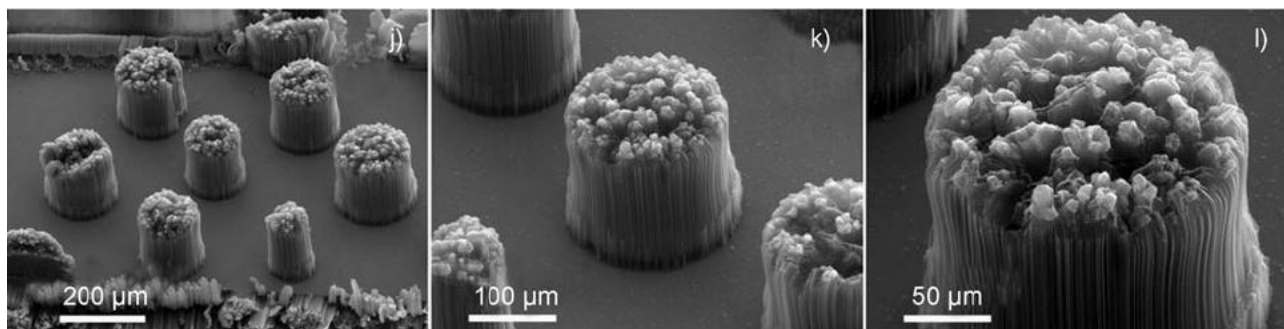


Fig. 3 SEM images of nanotube microarrays grown with CN_x nanotubes by a specific HF-etched porous silicon substrate in conjunction with the CVD process.

1.3 Production of CSi_y , CP_y and CN_xP_y Nanotubes by CVD methods

We also intend to produce new families of doped CNTs with silicon and nitrogen-phosphorous using the CVD approach indicated above. In particular, we will work with various compounds containing P such as silane, $P(C_6H_5)_3$ (Fig. 4). We will change the temperature conditions in order to find the best conditions to dope CNTs with phosphorous and silicon. These arrays of doped tubes planned to be then impregnated with polymers and their thermal properties determined. The performance of these tube composites planned to be compared with that of pure carbon nanotubes.

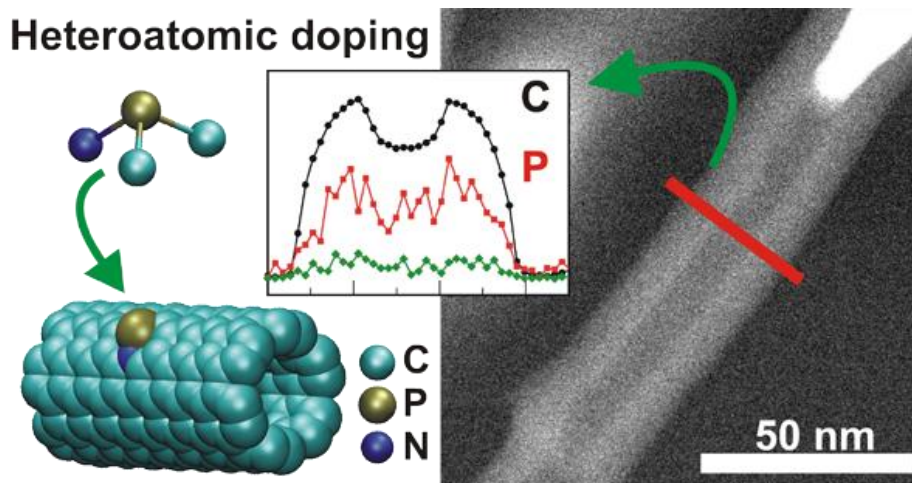


Fig. 4 Left, molecular model of a carbon nanotube doped P and N; Right High angular-annular dark field images of a PN-doped carbon nanotube, the red line represents the elemental linescan performed, which shows the presence of P and C (center panel).

1.4 Production of CO_xH_y nanotubes

We will use the same ultrasonic sprayer but in this case with solutions of ferrocene ($FeCp_2$) and different concentrations of ethanol (Et-OH). We have recently demonstrated that ethanol is able to produce extremely long nanotubes that are more reactive when compared to standard CNTs. For example, when adding 2.5% by wt. of ethanol in a ferrocene-toluene solution containing 5% by wt. of ferrocene, it is possible to obtain the longest MWNTs (from 1 - 10 mm) that exhibit high degree of crystallinity (Fig. 5). These chemical reactivity of the tubes has been confirmed using thermogravimetric analyses (TGA), Fourier Transform IR (FTIR) and Raman spectroscopy.

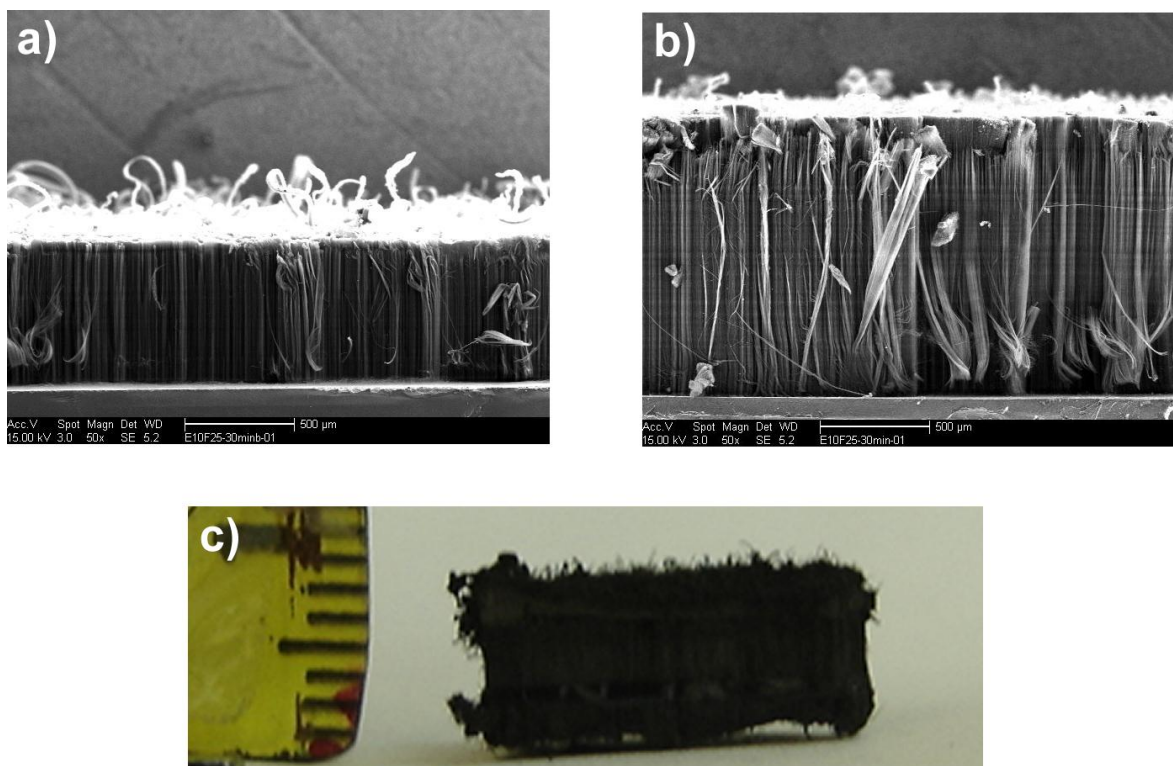


Fig. 5 Tall MWCNT forests grown with 1% by wt. of EtOH and 2.5% by wt. of FeCp₂ in toluene. With different synthesis times a) 0.5 hours (640 μm), b) 1 hour (1270 μm), c) 3 hours (~ 5000 μm)

2. CHARACTERIZATION OF NANOTUBE ARRAYS (to be carried out at IPICYT)

2.1 SEM characterization. The bulk morphology of the tubes, cells and hybrid structures planned to be studied by SEM. We will use the field emission SEM microscope Philips XL 30 (Fig. 6).

2.2 High Resolution Transmission Electron Microscope (HRTEM): In order to reveal the structure of the doped and functionalized tubes (as produced and processed under different acid treatments and temperatures) and to resolve the internal structure of surface structure we will use transmission electron microscopy. We will also study the cross-sections of the cells and study how the nanotube were ingested by the cells and identify the places and where the tubes are located within cells. This technique planned to be carried out using a Philips Tecnai F30 operating at 300 kV located at IPICYT, equipped with a Gatan image filter (GIF) filter for EELS analysis, CCD camera and STEM.

2.3 Elemental mapping and electron energy loss spectroscopy (EELS). Inelastically scattered electrons from the specimen planned to be monitored so as to provide information about the chemical, crystallographic and electronic structure of a particular specimen within very narrow limits ($\leq 3\text{nm}$). It is thus possible to establish both their stoichiometry and hybridisation state(s) as well as obtaining an elemental mapping at the nanometer scale. Elemental mapping and EELS characterization planned to be performed using the Philips Tecnai F30, equipped with a Gatan image filter (GIF) for EELS.

2.4 Energy Dispersive X-ray (EDX) analysis and mappings. The technique enables us to detect impurities in samples (dopant concentrations, functional groups deposited on the surface) and the composition of various types of nanoparticles. Equipment: Philips Tecnai F30 and Philips SEM XL30 (Fig. 6).

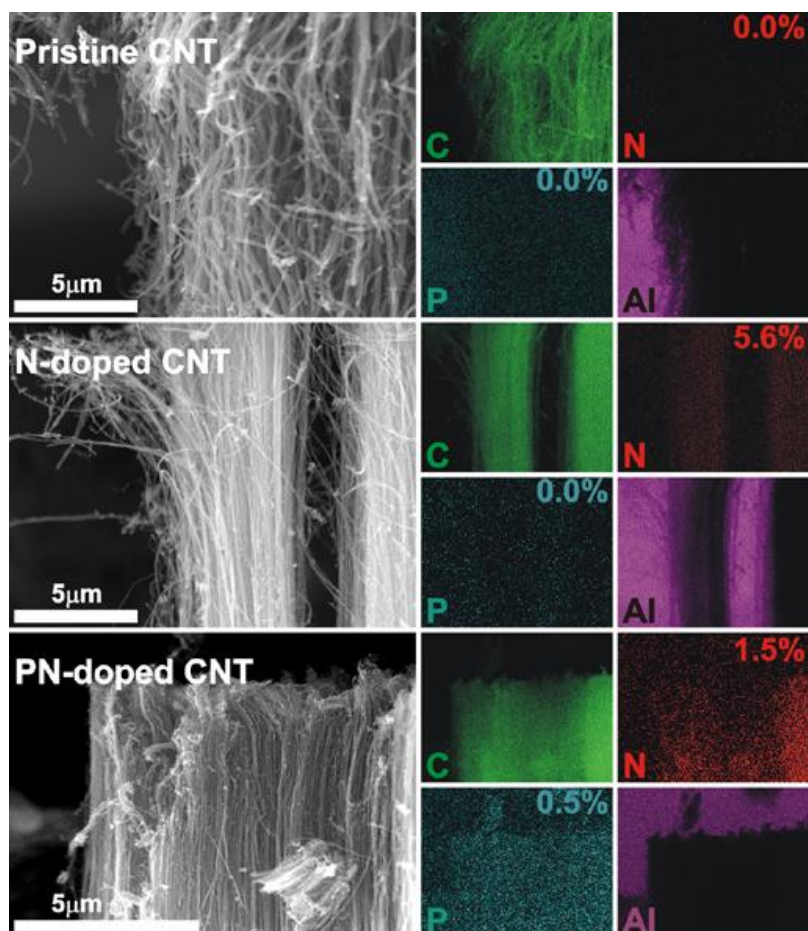


Fig. 6 EDX elemental mappings for three different types of nanotubes: Pristine pure carbon nanotubes (CNTs) obtained by thermolazing toluene ($C_6H_5-CH_3$) and Ferrocene at 850 °C (see top frame); N-doped CNTs described in the materials and methods section (see middle frame), and PN-doped CNTs described in the materials and methods section (see bottom frame). Samples were mounted on standard aluminum pins in order to have contrast for the carbon map. It can be clearly observed that the PN-sample contain both phosphorus and nitrogen in their structure, thus confirming the successful synthesis of PN heteroatomic doping. The other samples did not show heteroatomic doping.

2.5 X-ray powder Diffraction -IPICYT, for determining crystalline phases and average dimensions of bulk nanomaterials and composites.

2.6 Electron Diffraction (ED)- IPICYT. This technique is a powerful tool for determining the fine structure of doped and functionalized nanotubes (e.g. graphite stacking, interlayer spacing, morphology, etc.). Electron diffraction micrographs planned to be recorded using the nanodiffraction mode in the Philips Tecnai F30.

2.7 X-ray photoelectron spectroscopy (XPS). We will use this approach to characterize the surface of the doped and functionalized nanotubes. We will determine the binding energies for C, Si, N, O, Fe, P, B, and other elements and compounds. The signals obtained in the materials will also be compared with other results reported in the literature.

2.8 Atomic Force Microscopy (AFM). This technique is another commonly-used scanning probe microscope where the spatial variations of topographic surface. We will use this technique to detect the topographic morphology of the nanotubes and its arrays. The measurements planned to be carried out using an environmental AFM-MFM JEOL JSPM 5200.

2.9 Micro Raman Spectroscopy. We will carry out Raman measurements on the different types of

tubes and arrays of tubes in order to observe the changes in the vibrational modes for carbon. Due to the interaction of carbon with dopants (e.g. N, Si, O, P, etc.), we expect slight changes in the Raman signals for materials produced.

3. FABRICATION OF COMPOSITES BY IMPREGNATION POLYMERS (to be conducted at AFRL and AMRDEC)

3.1 Functionalization and Impregnation of Nanotube Arrays with Polymers.

AFRL will perform functionalization of the nanotube arrays specifically for compatibility with epoxy resins. A light infiltration of epoxy will also be performed to provide handling strength to the aligned array.

3.2 Fabrication of Composite Samples

AMRDEC will integrate the functionalized nanotube arrays into composite test specimens through filament winding and other composite fabrication techniques.

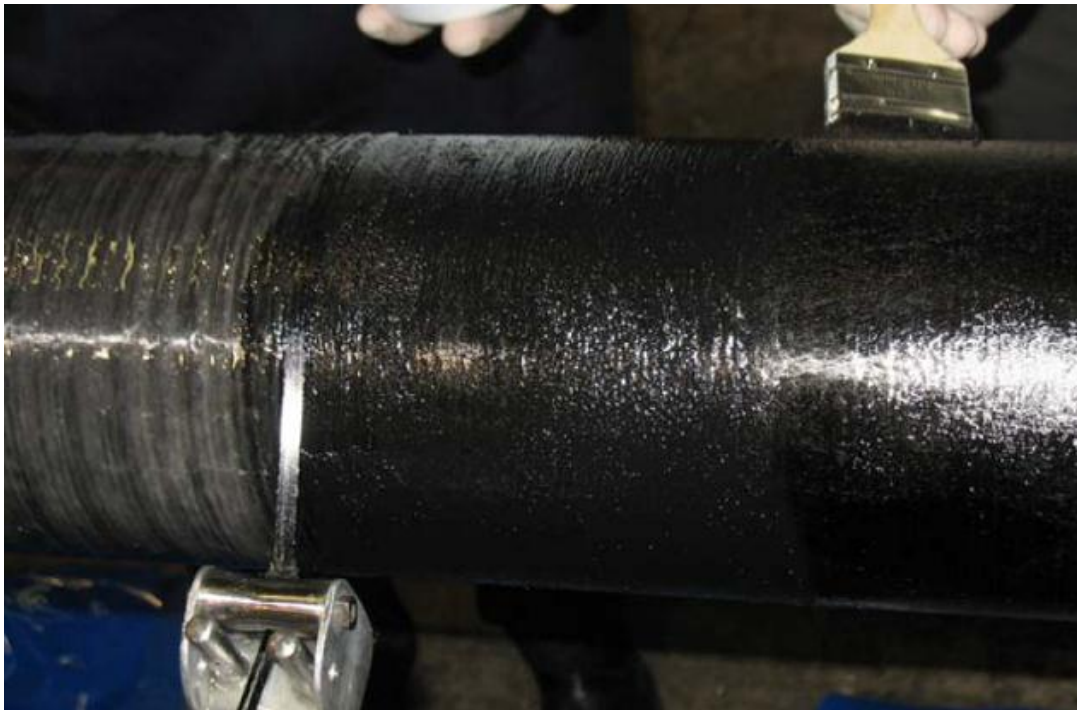


Fig. 7. Filament winding of carbon epoxy cylinder

4. THERMAL PROPERTY MEASUREMENTS (to be conducted at AMRDEC)

The thermal diffusivity of the nanotube arrays and the composites with embedded nanotube arrays planned to be performed by AFRL using a laser flash apparatus (LFA).

Thermal conductivity measurements will also be performed by AMRDEC on two inch diameter specimens using a Hunter quick link line 10-C thermal conductivity tester at Tuskegee University.

5. Deliverables.

At the end of the project investigators will deliver the initial technical report describing the thermal properties of polymer composites using different types of nanotubes (different dopants, lengths, diameters, crystallinity). Their possible applications for heat removal in radars, electronic devices, energy and power transformers planned to be evaluated. If applicable, we will submit this work for

publication in the peer-reviewed literature.

6. EXCHANGE OF RESEARCHERS AND STUDENTS (IPICYT, AFRL & AMSRD)

This multidisciplinary collaborative program is mainly concentrated on working synergistically between AFRL, AMSRD and IPICYT. It is therefore, extremely important to keep excellent communication among the three centers. One of the main objectives is also the training and exchange of researchers and students in order to learn novel techniques not available in their laboratories. In addition, researchers from the three Institutions could travel to the other laboratory in order to carry out the production, characterization of doped and functionalized nanotubes, so that all objectives of the project are fulfilled ([see work plan](#)).

The CNT synthesis planned to begin immediately and continue throughout the course of this effort. Throughout the project, the Terrones group will deliver different nanotube arrays to AFRL and AMRDEC. In particular, Dr. Ajit K. Roy (AFRL Materials and Manufacturing Directorate) will conduct the polymer impregnation experiments, and Dr. Keith Roberts (AMRDEC Propulsion and Structures) will carry out the thermal measurements and applications tests of the polymeric composite materials.

Relevance of the Project

The Aviation and Missile Research, Development and Engineering Center (AMRDEC) Propulsion and Structures Directorate and the Air Force Research Lab (AFRL) Materials and Manufacturing Directorate are interested in collaborating with the Instituto Potosino De Investigación Científica Y Tecnológica (IPICYT) to address the critical need for thermal management within composite structures. Radars, guidance electronics, directed energy and power transformers require mechanisms for removing heat. Tailored thermal conductivity is needed in composite materials as they replace traditional metal structures in missile, aviation, and aircraft applications. IPICYT has developed the technology to produce aligned carbon nanotube mats with properties that are optimized for thermal conductivity. Integration of these nanotube mats into composite structures could provide these structures with thermal property/conductivities that are similar to their metallic counterparts. Nanotubes of specific surface functionality can be implemented to make the nanotube surface compatible to the host material, for example polymers, ceramics or metallic materials.

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BUDGET REQUIRED FOR IPICYT

<i>Concept</i>	<i>Price</i>
	<i>USD</i>
Student Salary	11,250.00
Two Furnaces	5,000.00
TRAVEL	2,000.00
CONSUMABLES (12 months)	14,000.00
OVERHEAD	6,450.00
TOTAL REQUIRED	38,700.00

WORKPLAN

SCHEMATIC DIAGRAM

	Month 1-2	Month 3-4	Month 5-6	Month 7-8	Month 9-10	Month 11-12
METHODS						
PRODCUTION OF NANOTUBE ARRAYS USING ULTRASONIC SPRAY PYROLYSIS	Production of CNx nanotube arrays on SiOx substrates and functionalized nanotbes with EtOH by pyrolysing ferrocene, benzylamine and ethanol, using one ultrasonic sprayer. (IPICYT).	Bulk Production of CNxPy nanotube arrays. Change of conditions to produce CNx nanotubes of different lengths, diameters and crystallinity (IPICYT). Send samples to AFRL & AMRDEC.	Tests for producing CSix, CNxPy nanotubes using solutions of ferrocene and P- or Si- containing compounds. Continue with bulk production of CNx (IPICYT). Send samples to AFRL & AMRDEC	Production of CNx nanotubes and EtOH doped nanotubes of diameters above 100nm and lengths of ca. 30 microns. Continue producing CSi, CPy and CNxPy nanotubes. Production of CNx tubes of narrow diameters (<15nm OD) and longer than 300 microns (IPICYT).	Bulk production of CSix, CPy and CNxPy nanotube arrays for composite fabrication. (IPICYT). Produce the tubes with optimized thermal performance (IPCIYT).	
ETCHING OF SILICON SUBSTRATES	Production of Etched substrates of Si wafers using electrochemical etching and HF, different currents and conditions (IPICYT).	Production of Etched substrates using different electrochemical conditions and nanolithography techniques (IPICYT). Send samples to AFRL & AMRDEC			Production of doped nanotubes the etched substrates (IPICYT). Send samples to AFRL & AMRDEC.	
COMPOSITE FABRICATION	Impregnation of polymer A in the nanotube arrays of CNx and EtOH nanotubes (AFRL).	Impregnation of polymer B in the nanotube arrays of CNx and EtOH nanotubes (AFRL & AMRDEC). Send samples to IPICYT for electron microscopy characterization.	Impregnation of polymer C in the nanotube arrays of CSI, CPxNy nanotubes (AFRL). Send samples to IPICYT for electron microscopy characterization		Impregnation of the best performance polymer in the nanotube arrays using the best type of nanotubes (AFRL). Tests of the composites in prototypes (AMRDEC)	
MICROSCOPY CHARACTERIZATION	SEM, XRD and HRTEM studies on the CNx nanotubes produced by (IPICYT). Send results to AFRL and .AMRDEC	HRTEM, ED, EDX and EELS and elemental mapping on the tubes produced (IPICYT).	SEM, HRTEM and EELS studies and mappings of doped nanotubes (IPICYT)	Detailed AFM studies of nanotbe arrays and composites using the doped tubes (IPICYT).	SEM, HRTEM and EELS studies of doped nanotube arrays (IPICYT). Characterization of doped tubes with using prototypes (AMRDEC)	
THERMAL AND OTHER CHARACTERIZATION	Thermal characterization of the composites (AMRDEC). XPS, Raman Spectroscopy and AFM studies of doped nanotubes produced by CVD (IPICYT)		Thermal characterization of the composites (AMRDEC). Raman Spectroscopy, XPS and AFM studies of doped and functionalized nanotubes (IPICYT).			
EXCHANGE PROGRAM	First Meeting by the principal investigators to discuss research details and video-conferencing strategy	First exchange of a PhD student between both institutions. Short Visits researchers	Send one PhD students to IPICYT for conducting experiments and characterization.	Summer courses on Nanotubes and Thermal Transport in Nanomaterials and Nanocomposites. Workshop in San Luis Potosí on progress and challenges. Meeting of Principal investigators for evaluation and progress of the multidisciplinary project. Writing of the results for publication or patent application.		

