

ASSEMBLY AND CHARACTERIZATION OF NANOELECTROMECHANICAL SYSTEMS

NSF Summer Undergraduate Fellowship in Sensor Technologies
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ABSTRACT

Nanoelectromechanical systems, or NEMS, are characterized by minute dimensions that are relevant for the function of the devices. Using varied methods, many studies have sought to integrate nanostructures with functional circuitry. Some studies have focused on alternative methods that rely on the assembly of nanometer scale particles such as an electric-field assembly technique. The research reported here focused on the acquisition and development of a new experimental system that has enabled the control of individual nanostructures onto their predefined sites on the silicon chips. An electric-field assisted assembly technique was used to manipulate dielectric particles suspended in a medium between two electrodes 1 to 3 microns apart, defined lithographically on an SiO₂ substrate. Assembly experiments were first conducted in which a few drops of the medium were dispensed onto the sample and a 30 V AC signal fed at 1 kHz for varying lengths of time to the silicon chip that contains a series of prefabricated buried inter-digitated electrodes.

Keywords: nanoelectromechanical, electric-field assembly

1. INTRODUCTION

Nanoelectromechanical systems, or NEMS, are characterized by minute dimensions that are relevant for the function of the devices. Nanometer-scale science and technology provide control over materials and systems at the level of individual atoms and molecules. Feature sizes may range from hundreds to only a few nanometers. However, such small dimensions results in new physical properties, many of which may dominate how the devices operate.

An increased interest in molecular-scale electronics has led to developments in fabricating extremely complex circuits. This nanoelectromechanical systems revolution will profoundly affect many industries by providing control over applications such as sensors, medical diagnostics, displays, and even data storage. Sensors operating at such capacities are highly beneficial. This decrease in sizes will reduce cost, power consumption, lower force constraints and increase resonant frequency.

Many studies using a variety of methods have sought the same eventual goal of integrating nanostructures with functional circuitry. Such methods rely on the assembly of nanometer-scale particles by using innovative chemical and electrical-field assisted

assembly techniques to manipulate the placement of nanostructures. The successful placement of these nanostructures will assist in the development of multifunctional circuits.

Non-uniform electric fields have been used to manipulate dielectric particles that are suspended in a liquid solution [1]. Dielectrophoresis has been used in recent studies to direct individual rod-shaped particles of nanometer-scale in an electro-optical cell [2]. Studies have also been conducted on the manipulation of carbon nanotubes between electrodes on a glass substrate [3, 4].

Electrofluidic assembly enables collaborative activities aimed at the electromechanical studies of nanostructures as varied as resonant biosensors and magnetic sensors. Most significantly, however, this development serves as a stepping-stone in reaching the goal of integrating these nanostructures with functional circuitry. The ability to manipulate the nanostructures represents potential and realistic possibilities for the further development of integrated nanosystems that will assist in the deployment of nanosensors in multifunctional silicon chips and of nanostructures with engineered responses to several environmental conditions such as magnetic, electric, and electromagnetic fields.

The research reported here focuses on the acquisition and development of a new experimental system that has enabled the control of individual nanostructures onto their predefined sites on the silicon chips.

2. EXPERIMENTAL APPROACH

2.1 Electrofluidic Assembly Setup

The development, characterization, and integration of nanosystems require their placement onto pre-established sites. An electric-field assisted assembly technique was used to manipulate dielectric particles suspended in a medium between two electrodes 1 to 3 microns apart, defined lithographically on an SiO₂ substrate. In this technique, an assembly field is induced by buried electrodes. Charge separation along the structure causes its alignment and assembly along the field. A pair of passive top surface pads laterally defines the field, thereby, enabling lateral control of the assembly. Assembly is accomplished through the use of non-uniform alternating electric fields between an electrode pair. These electric fields have been used to manipulate dielectric particles suspended in liquid media. A charge separation and assembly field causes the alignment of the nanostructure. An AC field is induced in isolated pads by buried electrodes. In this application, the nanostructures are precisely oriented between two lithographically defined electrodes.

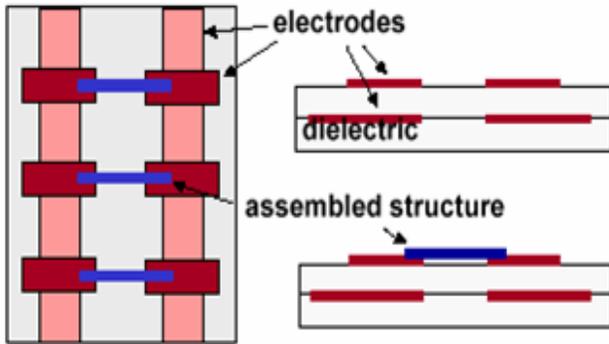


Figure 1: Top- and cross-sectional views of the electrode structures used in the field assisted assembly experiments. The buried electrodes induce an AC field in the isolated pads.

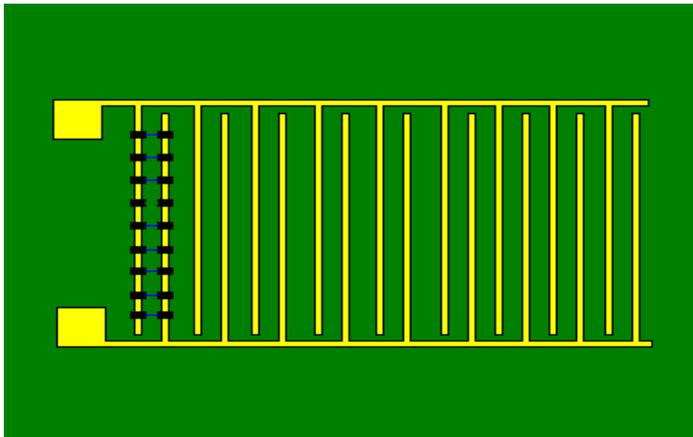


Figure 2: The metal electrodes consisted of an inter-digitated finger pattern.

Nanostructures consisted of rhodium rods and carbon nanotubes. The rhodium rods and the silicon chips used in this work were fabricated by Pennsylvania State University through nontemplated electroplating into a porous membrane. The carbon nanotubes used in this study were fabricated by Dr. Jack Fisher of the University of Pennsylvania.

Assembly experiments with the rhodium rods were conducted by feeding the assembly signal using probes from a probing station. The probing station was used to feed a 1 kHz and 30 V AC signal to the silicon chip, which contains a series of prefabricated buried inter-digitated electrodes (see Figure 2). A function generator and amplifying circuit provided the assembly signal. The oscilloscope was used simply to monitor the signal being applied to the chip (see Figure 3).

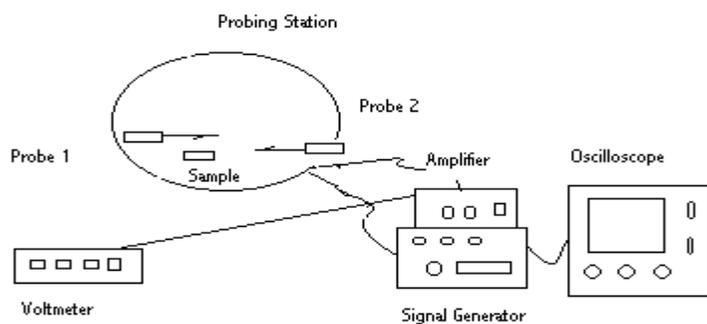


Figure 3: Experimental Setup

Assembly experiments were first conducted by dispensing a few drops of the medium onto the samples. The solution consists of rhodium rods 5 microns in length suspended in isopropyl alcohol. The concentration of the isopropyl alcohol is 20 $\mu\text{g/ml}$. The rhodium rods were aligned by applying the AC signal of 30 V at a frequency of 1 kHz for a period of 3, 6, and 9 minutes. Normally, the solution would evaporate completely within this time, so careful consideration should be taken when applying the voltage. Although, alignment was accomplished, several rhodium rods were distributed randomly along the electrode fingers.

Assembly experiments with the carbon nanotubes were conducted with the same parameters and experimental setup used for the assembly of rhodium rods. The chip used for the carbon nanotube assembly also contains a series of prefabricated buried interdigitated electrodes (see Figure 2). A function generator and amplifying circuit provided the assembly signal. The oscilloscope was used simply to monitor the signal being applied to the chip (see Figure 3).

The mass of the carbon nanotubes is significantly less than that of the rhodium rods. Assembly experiments were first conducted by placing the bottled solution of nanotubes and toluene into a sonicating bath for 10 minutes in order to evenly disperse the nanotubes throughout the medium. Assembly was then conducted by dispensing a few drops of the medium onto the samples. The solution consists of carbon nanotubes 500 nm in length suspended in toluene. The concentration of the toluene is 20 $\mu\text{g/ml}$. The carbon nanotubes were aligned by applying the same AC signal of 30 V at a frequency of 1 kHz. However, the voltage was applied for 6 minutes. Unlike the isopropyl medium, this solution did not evaporate completely within the 6 minutes.

2.2 Characterization Techniques

ATOMIC FORCE MICROSCOPY (AFM)

The atomic force microscope is one type of scanned-proximity probe microscope. These work by measuring local properties such as height, optical absorption, or magnetism with a probe or tip which is placed closely to the sample. The minuscule separation between the probe and sample makes it possible to take measurements over a small area. To acquire an image the microscope raster scans the probe over the sample while measuring the local property in question. The resulting image consists of many rows or lines of information placed one above the other. Unlike traditional microscopes, scanned-probe systems do not use lenses; therefore, the size of the probe rather than diffraction effects generally limits their resolution [5].

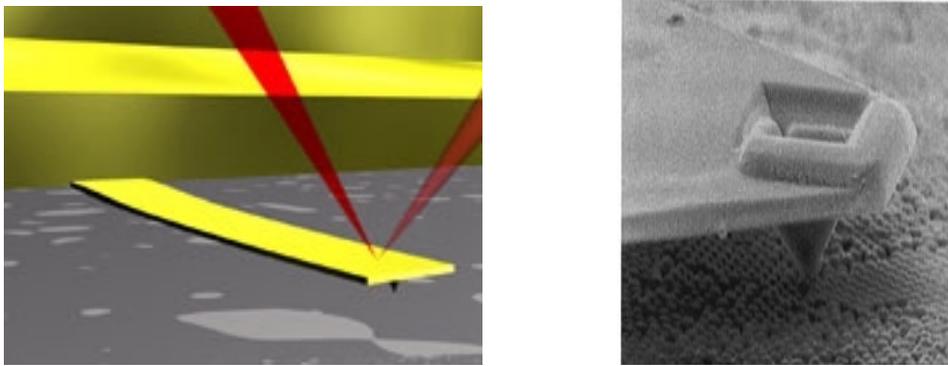


Figure 4: Atomic Force Microscopy and the optical lever: (left) a yellow cantilever touching a sample in gray; (right) Scanning Electron Microscope image of an AFM tip used to probe the structure of a sample surface.

In the non-contact mode, which consists of distances greater than 10\AA between the tip and the sample surface, Van der Waals, electrostatic, magnetic, or capillary forces produce images of topography. However, in the contact mode, ionic repulsion forces take the leading role.

The AFM mainly operates by measuring attractive or repulsive forces at the atomic level. The atomic forces are measured between a sharp probing tip and the sample. In a contact mode, repulsive forces are measured. Images are taken by scanning the sample relative to the probing tip and measuring the vertical deflection of the cantilever as a function of lateral position. In a non-contact mode, the AFM derives topographic images from measurements of attractive forces. This experiment required a non-contact mode AFM, which provides a means for measuring sample topography with little or no contact between the tip and the sample. So in this experiment, the samples of silicon chips were not contaminated through contact with the tip.

In the non-contact AFM, Van der Waals forces acting between the tip and the sample are detected, and topographic images are constructed by scanning the tip above the surface. Unfortunately, the attractive forces from the sample are substantially weaker and therefore more difficult to measure than the forces used in the contact mode, for two reasons: the small force values in the non-contact mode, and the greater stiffness of the cantilevers used. Therefore the tip must be given a small oscillation so that AC detection methods can be used to detect the small forces between the tip and the sample by measuring the change in amplitude, phase, or frequency of the oscillating cantilever in response to force gradients from the sample.

In this mode, the system vibrates a stiff cantilever near its resonant frequency (typically from 100 to 400 kHz) with amplitudes of a few tens of angstroms. As the probing tip comes near the sample surface, it detects changes in the resonant frequency or vibration amplitude. The resonant frequency of a cantilever varies as the square root of its spring constant:

Additionally, the spring constant of the cantilever varies with the force gradient experienced by the cantilever. The derivative of the force versus distance curve is the force gradient, as depicted in Figure 5. The force gradient changes with tip-to-sample separation. Therefore, the changes in the resonance of the cantilever can be used to measure changes in the force gradient, thereby reflecting changes in the distance between the tip and sample.

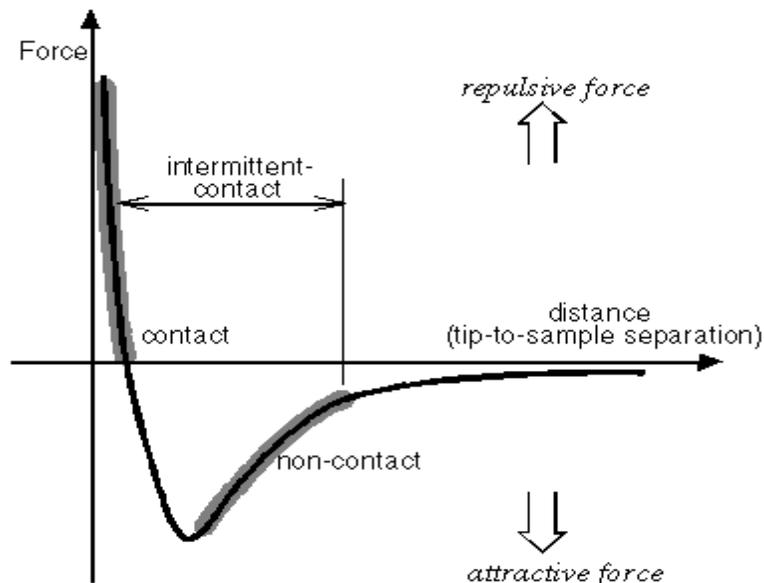


Figure 5

In the non-contact AFM mode, the system monitors and keeps constant the resonant frequency of the cantilever with the assistance of a feedback system that moves the scanner up and down. The average tip-to-sample distance is kept constant by keeping the resonant frequency constant. The motion of the scanner is used to generate the data set.

SCANNING ELECTRON MICROSCOPY

A scanning electron microscope accelerates an electron beam from the cathode and focuses it with a series of lenses onto the specimen by a spot whose diameter may be as small as 1 nm. Through the use of deflection coils, this spot is scanned over the specimen surface in a television-type raster, causing secondary electrons (in conjunction with other particles) to be emitted from the surface where the beam impinges. These electrons are detected, and the resulting signal is amplified and used to modulate the intensity of a cathode ray tube display that is scanned in tandem with the spot on the specimen. Therefore, a one-to-one correspondence exists between points on the specimen and on the cathode ray tube display, with the intensity of any point on the display representing the signal from the corresponding point on the specimen. Magnification may range from 100 times to over 100,000 times, given the ratio of the raster sizes [6].

3. RESULTS

3.1 Rhodium

This experiment indicates that the alignment of the rhodium structures between the electrode pairs is due to the electric fields. These forces direct the structure between the electrode pairs, a region of high field strength. The assembly field is induced by the buried electrodes. The charge separation along the structure causes the nanostructures to polarize in the electric field. Since the dielectric medium is less polarizable than the nanostructure, the rhodium nanostructures will undergo a dielectrophoretic force. This force produces the movement in the direction of the increasing field strength. When the rhodium nanostructures approach the electrode pairs, the strength of the electric field between the nanostructure's ends and the electrodes increases proportionally to the inverse of the distance from the electrodes. However, the spacing between the electrode pairs must be approximately equal to the length of the nanostructures. The combination of the charge separation along the structure and the assembly field causes the alignment of the structures (see Figure 6).

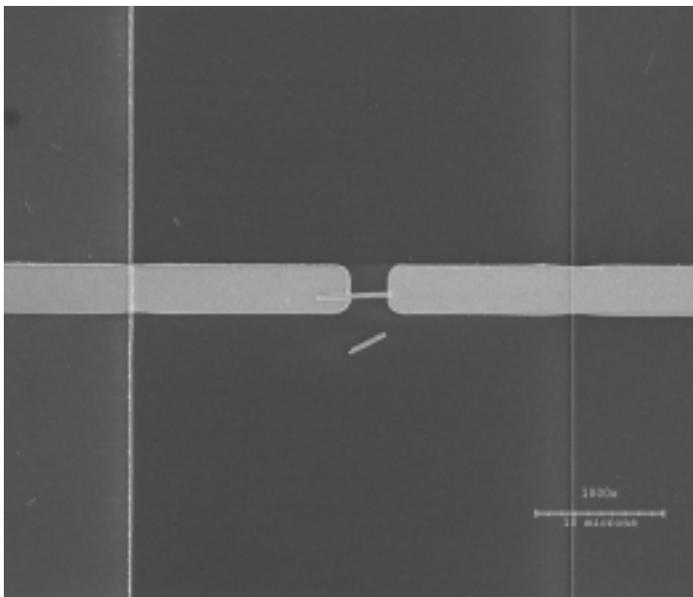


Figure 6: Scanning Electron Microscopy image of 5 μm long, 100nm diameter Rh nanostructures aligned by applying a 30V AC voltage at 1 kHz frequency to the structure with hand-held probes.

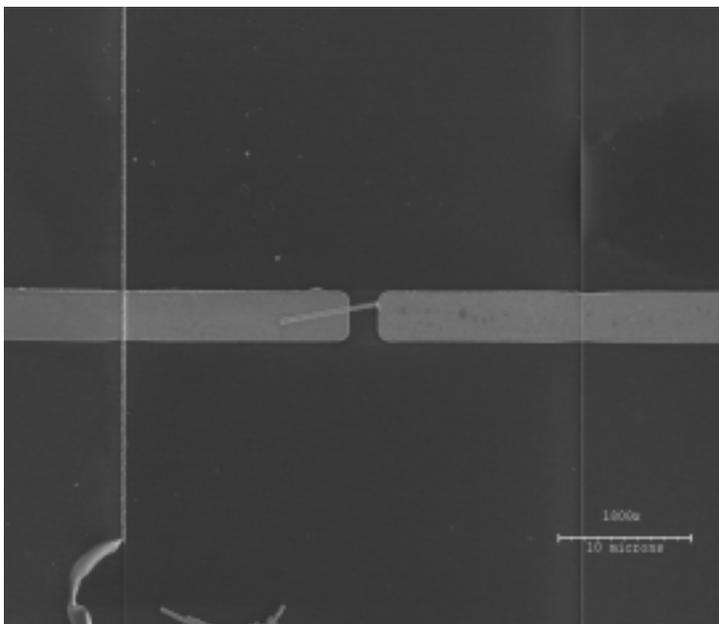


Figure 7: Scanning Electron Microscopy image of 5 μm long, 100 nm diameter Rh nanostructures aligned by applying a 30V AC signal at 1 kHz frequency to the structure with hand-held probes.

All through the assembly process, the electric field strength varies. The random placement of the rhodium nanostructures can be accounted for by considering these variations as a function of position along the electrode pairs. Preceding the application of the AC signal, the electric field strength at each position along the electrodes is the same. Given that the electric field is identical, the probability that an individual nanostructure will assemble is the same for all nanostructures in the medium. However, after assembly, the electric field strength is reduced. With the reduction in field strength, additional

nanostructures are prevented from aligning on the same electrode. This will result in the random assembly along the buried electrodes (see Figure 8).

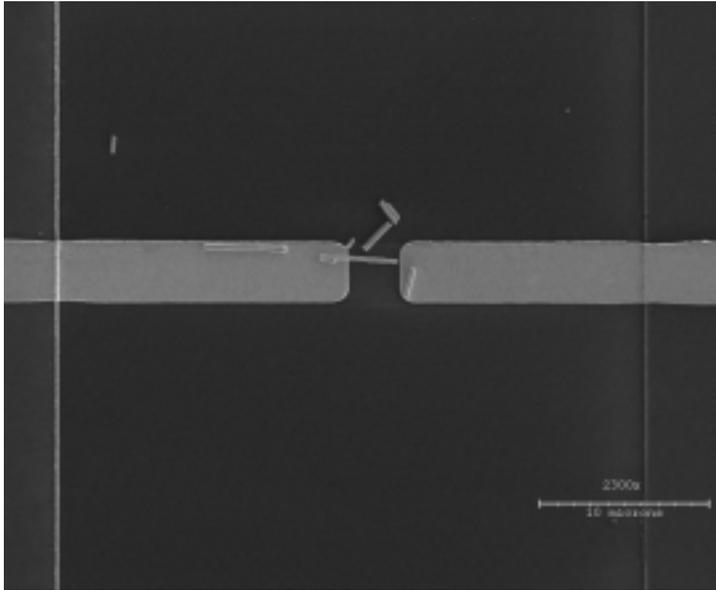


Figure 8: Scanning Electron Microscopy image of Rh nanostructures. This image indicates the random placement of nanostructures due to the variety of the electric field strength as a function of position along the electrode pairs.

This study showed a direct relationship between the voltage and the duration of the electric forces exerted on the nanostructures. At a constant frequency and constant voltage, the amount of nanostructures aligned between the electrode pairs is related to the length of time the nanostructures were exposed to the AC signal. For this study, as the duration of the electric forces was increased, the nanostructures were exposed to the force for longer periods, resulting in an increase in the number of nanostructures that were aligned.

Chip #1

Three trials, each lasting 3 minutes.

<u>Attempt # 1</u>	<u>Attempt #2</u>	<u>Attempt #3</u>
After 3 minutes:	After 6 minutes:	After 9 minutes:
4 sites assembled	8 sites assembled	32 sites assembled

Once the nanostructure assembles between the electrode pairs (Figures 7 and 8), the electric field between them is eliminated, thus acting as a short in the circuit. Essentially, the electric field between neighboring electrode pairs is reduced, which prevents any additional nanostructures from aligning anywhere on the electrode.

3.2 Carbon Nanotubes

Electric-field assembly has been used to position the rhodium nanostructures from their liquid medium onto electrically isolated electrode pairs. The assembly process was also conducted with carbon nanotubes, operating with the same principle of alignment. Assembly between the electrode pairs is due to the electric fields. These forces direct the carbon nanotubes between the electrode pairs, which is a region of high field strength. The assembly field is induced by the buried electrodes. The charge separation along the structure also causes the carbon nanotubes to polarize in the electric field. The movement of the carbon nanotube is a result of dielectrophoretic forces, which occur in the direction of the increasing field strength. When the carbon nanotubes approach the electrode pairs, the strength of the electric field between the nanotubes' ends and the electrodes also increases proportionally to the inverse of the distance from the electrodes. However, the spacing between the electrode pairs was significantly greater than the length of the carbon nanotubes. Consequently, assembly of the carbon nanotubes was not as successful.

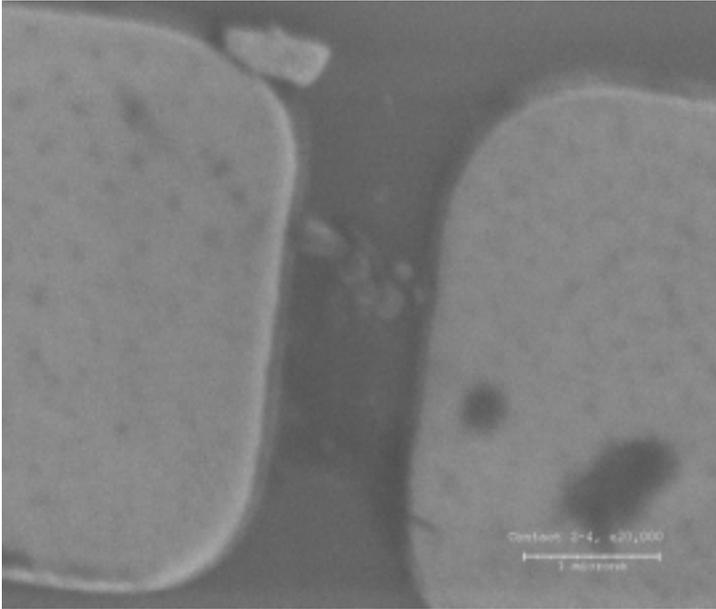


Figure 9: Scanning Electron Microscopy image magnified 20,000x of 500 nm long, 1nm diameter carbon nanotube bundles aligned by applying a 30V AC signal at 1 kHz frequency to the structure with hand-held probes.

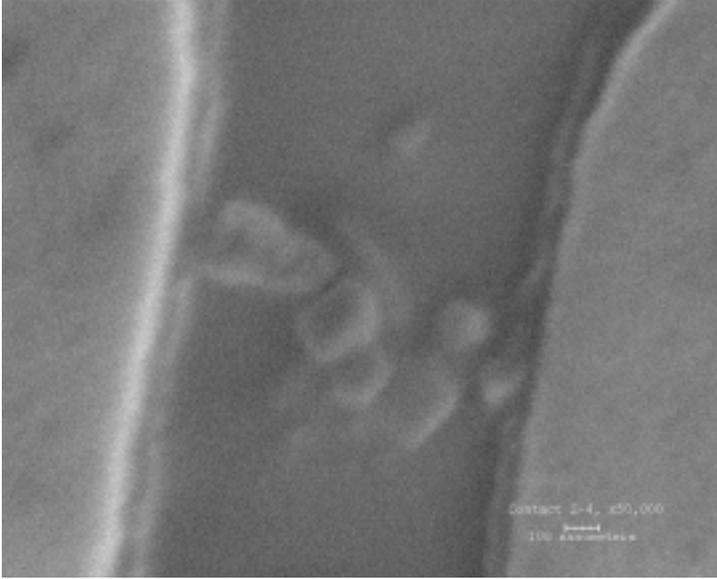


Figure 10: Scanning Electron Microscopy image of figure 1 magnified 50,000x of 500 nm long, 1nm diameter carbon nanotube bundles aligned by applying a 30V AC signal at 1 kHz frequency to the structure with

Since the carbon nanotubes are so minute in size it was difficult to obtain an unambiguous image. As the magnification increased the image became more distorted. With the use of AFM, images were less skewed (Figure 11).

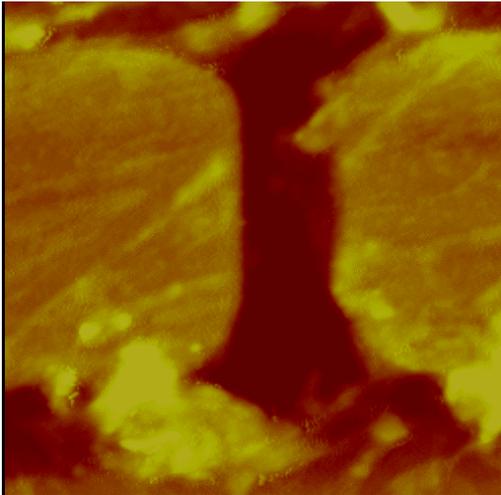


Figure 11: Atomic Force Microscopy image of figure 10. Image depicts carbon nanotube bundles between the two electrodes.

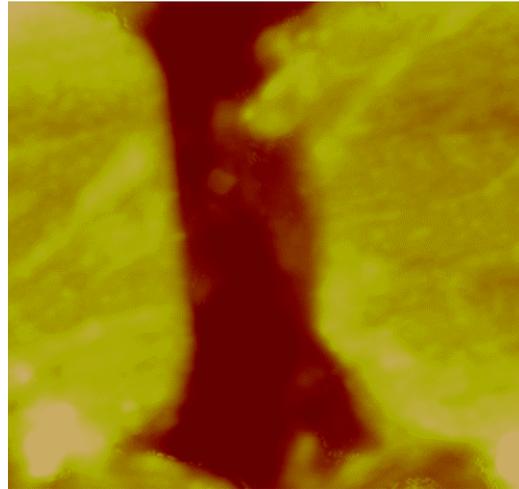


Figure 12: Closer image of figure 11. Image depicts carbon nanotube bundles between electrode pairs.

6. CONCLUSIONS

Electric-field assembly techniques were used to align the rhodium nanostructures between the electrode pairs. The research indicates that the alignment forces are results of polarization of the nanostructures in the electric field. Additionally, this research indicates a correlation between the numbers of successfully assembled sites and the length of time the nanostructures were exposed to the voltage. Assembly of the carbon nanotubes was not as successful, because of difference in lengths. Since the electric-field strength between the electrodes and nanotubes increases proportionally to the inverse of the distance from the electrodes; it is imperative that the nanostructures be about equal in length to the spacing between the electrode pairs. Also, the nanotubes may not have been properly dispersed.

7. RECOMMENDATIONS

Absence of optical magnification also prevented the observation of the assembly. It is recommended that for future work a more enhanced microscope be used. It is necessary to be able to observe the assembly as it takes place. Unfortunately, this was not possible with the microscope used for this research.

8. ACKNOWLEDGMENTS

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