



Aligned carbon nanotube coating on polyethylene surface formed by microwave radiation

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ABSTRACT

Multifunctional carbon nanotube (CNT) architectures have been created on polyethylene (PE) surface by a microwave welding process. The continuous and aligned CNT films drawn from super-aligned CNT arrays can significantly absorb microwave energy and act as a network of nanosized thermal sources to locally melt the PE substrate beneath, leading to polymer wrapping around individual nanotubes. Uniform and highly conductive CNT/PE nanocomposite layer was formed without undermining the original alignment of the CNTs. CNT patterns have also been precisely fabricated on PE samples. The PE/CNT/PE bonds showed high interfacial strengths, which were affected by the duration of microwave radiation. With ultra-low content of CNTs introduced as antistatic agents, the dissipation of surface charges on PE substrate has been tremendously improved.

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1. Introduction

The response of carbon nanotubes (CNTs) to microwave radiation has been actively explored in recent years. It was reported that CNTs displayed strong microwave absorption with intense heat release and fast temperature increase [1,2]. Energy can be transferred from microwave field to CNTs via dipole polarization induced by alternating electric field, as well as mechanical vibration caused by phonon–phonon interaction [3]. Although the theory of CNT–microwave interaction remains incompletely understood, the observed super-heating phenomena have been widely explored for many applications. The extreme temperature reached during this heating process is high enough to induce the reconstruction of the CNT walls, which can be used to reduce the defect density of CNTs. Fibers spun from the microwave annealed CNTs showed increased modulus, tensile strength, and electrical conductivity [4]. The microwave induced heating of CNTs can reduce the reaction time for chemical functionalization of CNTs compared with conventional thermal methods [5–7]. CNTs can interact with microwave efficiently when dispersed in a polymer matrix and dramatically enhance the microwave absorbance of the polymer [8]. Microwave radiation and the subsequent heating of CNTs can also be used to rapidly cure CNT/ceramic composites [9]. Furthermore, there is a great deal of interest in decorating various nanoparticles onto CNTs with microwave radiation. For example, Pt nanoparticles were decorated onto CNT nanowebs and buckypa-

pers by microwave assisted thermal reaction in solution [10]. Very recently, metal and metal oxide nanoparticles were formed on CNTs by a solvent-free microwave heating method [11]. The solvent-free microwave heating technique can play an important role in fabricating CNT/polymer composites. However, this rapid microwave processing method has not been fully explored, as the heating process is too vigorous to be explicitly controlled in air, which limits the manipulation of macroscale CNT structures. So far, only a few attempts have been successfully made, such as to embed CNT paste onto polymer substrates and to weld polymer stacks by CNT films by microwave radiation [12–14].

In this work, we demonstrate a rapid approach to introduce continuous CNT network onto polymeric substrates with well-bonded interfaces. This is the first case of modifying polymer surfaces with continuous CNT structures by the advanced microwave welding technique. The microwave welding process can be precisely controlled to form durable CNT patterns on polymer surfaces. The microstructure and the electrical conductivity of the CNT/polymer nanocomposites were characterized and their applications as antistatic coatings were explored. The interfacial strengths of the CNT/polymer bonds were further investigated.

2. Materials and methods

2.1. Preparation of CNT films

CNT films were drawn from super-aligned CNT (SACNT) arrays with height of around 300 μm , which were synthesized on a 4 in. silicon wafer in a low-pressure chemical vapor deposition (LPCVD)

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system by using Fe film as the catalyst and acetylene as the precursor [15–17]. Strong van der Waals forces among CNTs in the super-aligned array ensure the end to end joining of CNTs during the pulling process to form continuous and aligned CNT films. These free-standing CNT films are extremely lightweight, transparent, and highly conductive. For a typical single-layer CNT film, the mass per unit area is $1.5 \mu\text{g}/\text{cm}^2$, the optical transmittance is 78%, and the sheet resistance is $700\text{--}1000 \Omega$ along the CNT alignment, which is distinguished from randomly oriented CNT films [18,19]. A CO_2 laser beam was scanned on the surface of the SACNT array to etch two parallel lines at a desired distance. CNTs at the two parallel etching lines were destroyed after the laser scanning and thus could not be drawn out, which assured a uniform width of the resulting CNT film [20].

2.2. Fabrication of CNT/polyethylene composites

Polyethylene (PE) substrates with a low melting point around 110°C and dimensions of $50 \text{ mm} \times 50 \text{ mm} \times 3 \text{ mm}$ were used in the experiments. Multiple layers of the continuous CNT films with controlled width of 50 mm were stacked on top of a PE substrate. Several drops of ethanol were spread and uniformly wet the CNTs film onto the PE surface. During evaporation of ethanol, the surface tension of ethanol compressed the CNT films, which ensured good contact between the CNT films and the PE substrate. A microwave oven operating at 2.45 GHz and 750 W was used as the energy source. PE material can be considered as transparent to 2.45 GHz electromagnetic waves, which works as an ideal matrix system for the study of the selective microwave heating of CNTs. The PE samples with adhered CNT films were microwave radiated for $5\text{--}150 \text{ s}$ to create uniform CNT coatings on the PE substrate. In order to study the effect of CNT morphology on the rapid heat release during the microwave welding process, some CNT bundles were peeled off from the SACNT arrays, ultrasonically dispersed in ethanol for 5 h , deposited onto PE substrates, and then microwave radiated for the same duration.

PE/CNTs/PE bonds were formed by the microwave welding method. Two layers of CNT films were stacked between a pair of PE strips, with dimensions of 50 mm in length, 9 mm in width, and 3 mm in thickness. The whole assembly was pressed together and the interface area is $9 \text{ mm} \times 3 \text{ mm}$. The duration of microwave radiation ranged from 30 s to 90 s and PE/CNTs/PE bonds were formed by the microwave induced heating of the CNT films. For comparison, a pair of PE strips was pressed together without introduction of any CNT films and microwave radiated for the same duration.

2.3. Characterization of CNT/PE composites

The microstructures of the CNT/PE nanocomposites were observed using optical stereomicroscopy, scanning electron microscopy (SEM: Sirion 200, FEI, with acceleration voltage of 10 kV) and transmission electron microscopy (TEM: Tecnai G2 F20 S-TWIN, FEI, with acceleration voltage of 200 kV).

The interfacial strengths of the PE/CNTs/PE bonds were measured using an Instron 5848 microtester under a tensile test. The whole bonded PE/CNTs/PE strip was clamped by fixtures at both ends and pulled apart with a constant cross head speed of $0.1 \text{ mm}/\text{min}$ and a gauge length of 68 mm . After tensile testing, the fracture surfaces of the PE/CNTs/PE bonds were observed using SEM and the failure mechanism was analyzed.

Surface resistances of the PE samples welded with different layers of CNT films were measured in directions both parallel and perpendicular to the CNT alignment ($R_{//}$ and R_{\perp}) using a Keithley 2410 source meter. Because the anisotropic conductance cannot be characterized by a 4-point probe method, the surface resistances here were measured by a 2-point method by attaching two copper elec-

trodes at the two parallel edges of the samples. The durability of the PE samples welded with one layer of CNT film was further investigated by an anti-scratch test. A needle with a tip area of around 0.25 mm^2 was pressed against the CNT/PE composite surface under a force of 0.7 N . The needle scratched the composite surface along the direction perpendicular to the CNT alignment at a speed of $2 \text{ mm}/\text{s}$. The variation of $R_{//}$ during the anti-scratch test was measured. The anti-scratch property of a PE substrate adhered with one layer of CNT film without any microwave treatment was also studied for comparison.

A comparative study on the antistatic performance for the PE samples welded with CNT films and the bare PE sample was carried out. All the samples have the same dimension of $30 \times 30 \times 3 \text{ mm}^3$ and their surfaces were scrubbed with a piece of leather. The electrostatic charges remaining on the sample surface were characterized by a surface DC voltmeter (Model SVM2 AlphaLab Inc.), which can accurately detect surface potential voltage on materials without making any physical contact. An electrostatic charge monitor probe was placed at a distance of 8 mm from the sample surface, and the probe body was driven to a potential V corresponding to the surface charge density of the sample by an internal electronic circuit. This measurement does not affect the distribution of the surface charge and the surface charge density Q/A (C/cm^2) can be calculated from the displayed potential V (kV) by the following equations:

$$Q/A = V \cdot 3.6 \times 10^{-11} \cdot \frac{f}{f-1}$$

$$f = \sqrt{1 + (D/2L)^2}$$

where D is the distance from the probe to the sample surface, and L is the width of the sample. For each sample, the measurement was performed for five times, and the average value of the surface charge density was taken.

3. Results and discussion

3.1. Rapid microwave welding process

During microwave radiation, the as-stacked CNT films absorbed energy from the microwave field and released large amount of heat. The heat generated by individual CNTs was rapidly dissipated into the environment and transferred along the continuous CNT network because of the high thermal conductivity of CNTs [21]. Due to the relatively low thermal boundary resistance between CNTs and polymers [22], the temperature difference between the CNT films and the PE substrate was not significant, so that the temperature of the PE surface increased rapidly, leading to localized melting of the PE surface. As the CNTs are sparsely aligned within the continuous CNT films [18], the molten PE material can fill into the spaces within the porous CNT layers and wrap around individual CNTs to minimize the surface potential energy. Owing to the short processing time and the small amount of CNT film per unit area ($1.5 \mu\text{g}/\text{cm}^2$), the total amount of heat generated from CNTs was limited. As a result, the entire PE matrix except for the PE–CNT interface remained at room temperature, so that the overall structure of the bulk material was unchanged during this process. The PE matrix served as a cooling reservoir to keep the temperature of the CNT film below 700°C , which protects the CNTs from oxidation in air.

The continuous and aligned CNT film has a uniform thickness of about 100 nm , which is within the skin depth of microwave to graphite (about $30 \mu\text{m}$). Therefore the entire CNT network can efficiently absorb microwave energy and rapidly release heat to form CNT/PE composites within 10 s . In contrast, the CNT bundles taken from the SACNT arrays were still strongly entangled and aggregated together even after long time of sonification. These CNT agglomera-

tions (200 μm in diameter) on PE samples resulted in small effective surface area to absorb microwave energy. Within 10 s of microwave radiation, the amount of heat released from CNTs was insufficient to melt the PE surface, not to mention to form any CNT/PE composites. After extended microwave radiation for as long as 150 s, at only a few spots, overheated CNTs caused polymer degradation, accompanied with fire flashes and smoke. This is mainly because the uneven microwave field cannot heat the entire sample surface at a same speed and the CNT agglomerations cannot form a continuous CNT network for quick heat dissipation across the entire surface. As a result, uniform temperature distribution of the sample surface cannot be achieved, which may cause overheating at certain areas and degrade the polymer matrix beneath. These results suggest that the morphology of CNTs plays an important role in the formation of uniform CNT/polymer nanocomposites by microwave radiation. Dispersed CNTs are not suitable to make uniform CNT/PE nanocomposite coating by the microwave welding process. Instead, using the continuous and aligned CNT films can overcome the above-mentioned problems and successfully produce uniform CNT/PE nanocomposite coating on PE surfaces.

3.2. Microstructures of CNT/PE nanocomposites

The SEM image of a PE sample adhered with two layers of CNT films before microwave radiation is shown in Fig. 1a. Good alignment of CNTs can be observed and the diameter of the CNTs ranges from 20 nm to 50 nm. After microwave radiation for 15 s, the CNT films were uniformly embedded onto the PE substrate. The continuous CNT network was preserved and CNTs kept their original alignment, as shown in the SEM image in Fig. 1b. The diameter of CNTs on the PE surface increased to a range from 50 nm to 150 nm, which suggests significant PE coating onto CNTs.

The microwave welding method can be further controlled to coat CNT films on selected area of a PE substrate. By using pre-designed masks, aligned CNT structures can be welded onto certain areas of the PE surfaces with a single step. The temperature at the selected areas on the PE substrate beneath the mask was lower than other areas that directly contact with the CNT films, and PE material at these selected areas did not melt. Meanwhile, the part of the PE surface with direct contact to CNT films was successfully welded and infused into the CNT layers, creating desirable patterns with very sharp boundaries, as shown in the optical graph with a pattern of “THU” in Fig. 1b. These refined CNT architectures can be potentially used in applications such as flexible circuits and heating arrays.

TEM images in Fig. 2 clearly show that a large amount of molten polymer wetted CNTs, resulting in increased diameters compared to the original CNTs. The inter-tube spaces were also filled with molten polymer. Most CNTs wrapped by polymers were shaped

into CNT–PE nanowires, which protected the CNT films from environmental damage.

3.3. Interfacial strength of CNT/PE nanocomposites

Using the microwave welding approach, two PE strips were bonded together by sandwiching CNT films in the middle. Microwave radiation induced heating of the CNT film, subsequently the locally melted PE material intercalated into the CNT layer and filled the inter-tube spaces, and PE/CNT/PE bonds can be successfully made after cooling down. Uniform bonding region was obtained and CNTs were well distributed at the PE/CNT interface made by microwave welding for 75 s, as can be observed from a cross-sectional SEM image of a PE/CNTs/PE bond (Fig. 3a). The thickness of the bonding zone was about 1 μm , which composed of layers of PE and CNTs. After tensile testing, the PE-wrapped CNTs were pulled out from the PE matrix with bases embedded in the substrate, as observed from the fracture surface of the PE/CNTs/PE bond (Fig. 3b), indicating that the CNTs intercalating across the interface contributed to the load transfer and enhanced the strength of the PE/CNTs/PE bond. By contrast, two equivalent PE strips pressed together without using any CNT films exhibited no temperature increase at the interface after minutes of microwave radiation, and no bond could be formed.

The bond strength of the PE/CNTs/PE bonds was 3.78 MPa for the sample after microwave radiation for 30 s. The bond strengths increased with the duration of microwave radiation (Fig. 4). After microwave radiation for 75 s, the bond strength increased to 9.66 MPa, comparable to the tensile strength of a pure PE strip at 15.17 MPa. Excessive microwave radiation for longer than 90 s caused overheating of the PE matrix and resulted in severe degradation and deformation of the PE sample. Therefore it is essential to control the duration of microwave radiation to optimize the bond strength.

3.4. Electrical conductivity and durability of the CNT/PE composite coating

The surface resistances ($R_{//}$ and R_{\perp}) of PE samples welded with different layers of CNT films by microwave radiation are shown in Fig. 5a, which are compared with those of PE samples adhered with same layers of CNT films by ethanol without any microwave treatment. Surface resistances decreased with increasing layers of CNT films for all the samples. The PE substrates welded with CNT films exhibited higher surface resistances than the PE substrates adhered with CNT films, because a certain percentage of the electrical pathways in the PE substrates welded with CNT films were intercepted by polymer wrapping onto CNTs. It is worthy to mention that, the values of $R_{//}$ of the nanocomposite layer are always less than $10^3 \Omega$, while the values of R_{\perp} can be tuned in a wide range

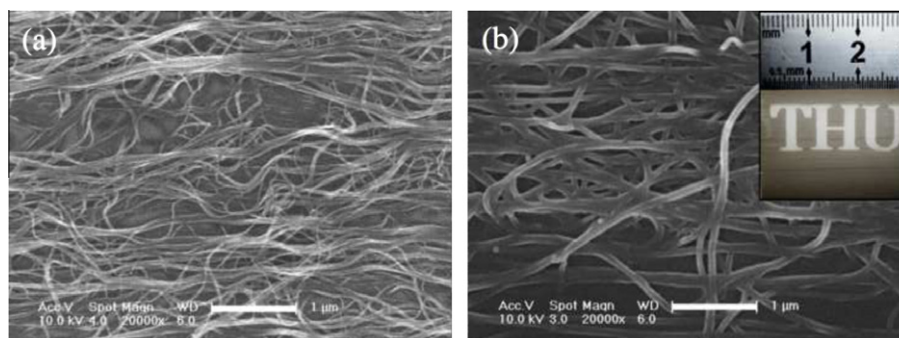


Fig. 1. (a) An SEM image of a PE substrate adhered with two layers of CNT films before microwave welding; (b) An SEM image of a PE substrate welded with two layers of CNT films by microwave radiation for 15 s and an optical micrograph of a CNT pattern of “THU” welded on a PE substrate.

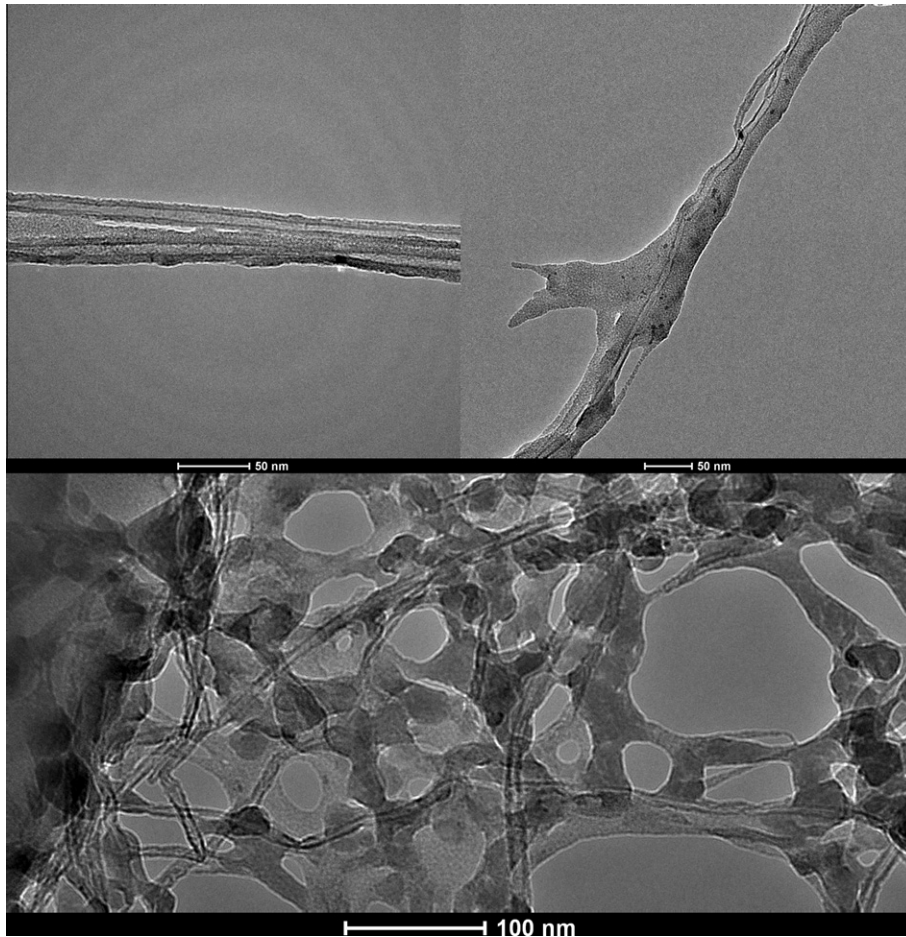


Fig. 2. TEM images showing that polymer wetted and wrapped around individual CNTs and filled the inter-tube spaces after microwave radiation. The conductive CNT network was preserved by polymer wrapping.

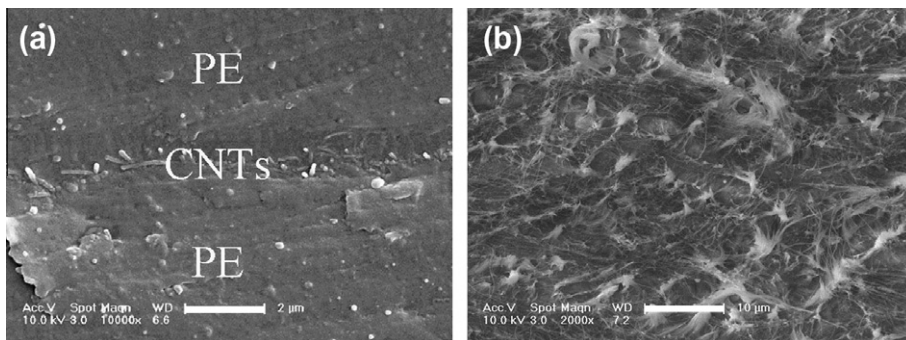


Fig. 3. SEM images of (a) cross section and (b) fracture surface of a PE/CNTs/PE bond made by microwave welding for 75 s.

from $10^3 \Omega$ to $10^5 \Omega$, which serves as an anisotropic conductive surface. The surface resistance of the CNT/PE nanocomposite layer is referred to the value of $R_{//}$ in the following context, because cross-stacked CNT films can be easily welded onto PE surfaces to achieve low resistances in both directions.

The durability of a PE sample welded with one layer of CNT film by microwave radiation was investigated by an anti-scratch test. The variation of the surface resistance during the anti-scratch test is shown in Fig. 5b. Initially the surface resistance is about 700Ω . After repeated scratches for 50 times, the surface resistance varied from 700Ω to 765Ω with an increase less than 10%. In contrast, one layer of CNT film adhered on PE substrate by ethanol treatment without microwave radiation was easily removed after just one scratch, leaving the bare PE surface insulated (surface resistance

greater than $10^{11} \Omega$). These results suggest that good bonding between CNT films and the PE substrate can be formed by the microwave welding method. PE substrates welded with CNT films can be used as durable and conductive surfaces for various applications such as flexible circuits and touch screens.

3.5. Antistatic abilities

The amounts of electrostatic charges left on PE surfaces with and without welded CNT films after being scrubbed by a piece of leather are illustrated in Fig. 6a. For a bare PE surface without any CNT coating, the surface charge density as high as 2.05 nC/cm^2 was generated. With the introduction of CNT films as anti-static coatings, the surface resistances of the nanocomposites were

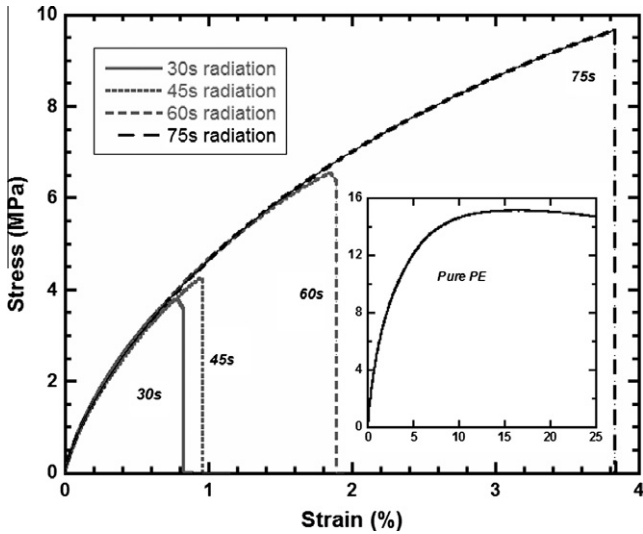


Fig. 4. Tensile stress–strain curves of PE/CNTs/PE bonds made by microwave welding for various durations.

reduced to $10^3 \Omega$, which is much lower than the required value of dissipative materials (10^9 – $10^4 \Omega$). With one layer of CNT film as the antistatic coating, the remaining surface charge density was 0.10 nC/cm^2 , corresponding to 5% of the value on the bare PE surface. The antistatic abilities of the nanocomposite layer can be further improved by increasing the layers of CNT films. On the PE surface welded with 5 layers of CNT films, the surface charge density was reduced to 0.02 nC/cm^2 , corresponding to only 1% of the value on the bare PE surface. As reported by dispersing 3 wt.% of CNTs in polypropylene, the electrostatic charge density was reduced to 10% of the value on pure polypropylene samples [23]. In our experiments, the effect of continuous CNT films on reducing surface charge density is more significant, which can reduce the surface charge density to as low as 1% compared to the value on pure polymer surface.

Both carbon nanofibers (CNFs) and CNTs can be used as conductive additives in polymer matrix to make lightweight antistatic coatings due to their high aspect ratio and high electrical conductivity. However, it is very difficult to uniformly disperse CNFs or CNTs into polymer matrix, which may result in entanglement and agglomeration. Furthermore, most of the CNFs or CNTs in the matrix do not have any effects on the antistatic abilities, because only the conductive network on the surface contributes to the

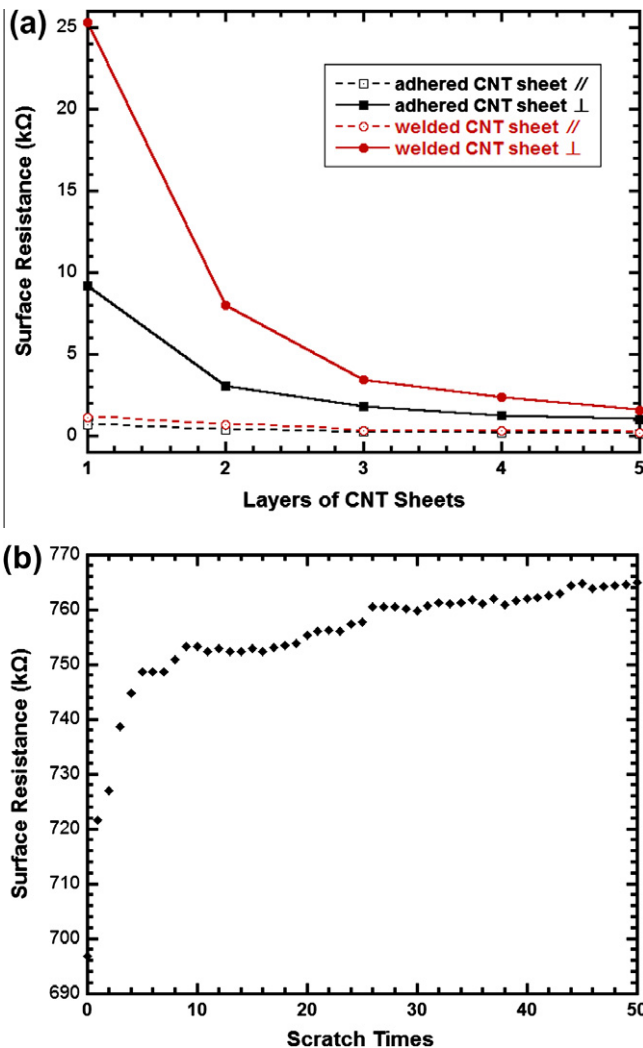


Fig. 5. (a) Parallel and perpendicular surface resistances of PE samples welded and adhered with different layers of CNT films. (b) Surface resistance of a PE substrate welded with one layer of CNT film during an anti-scratch test.

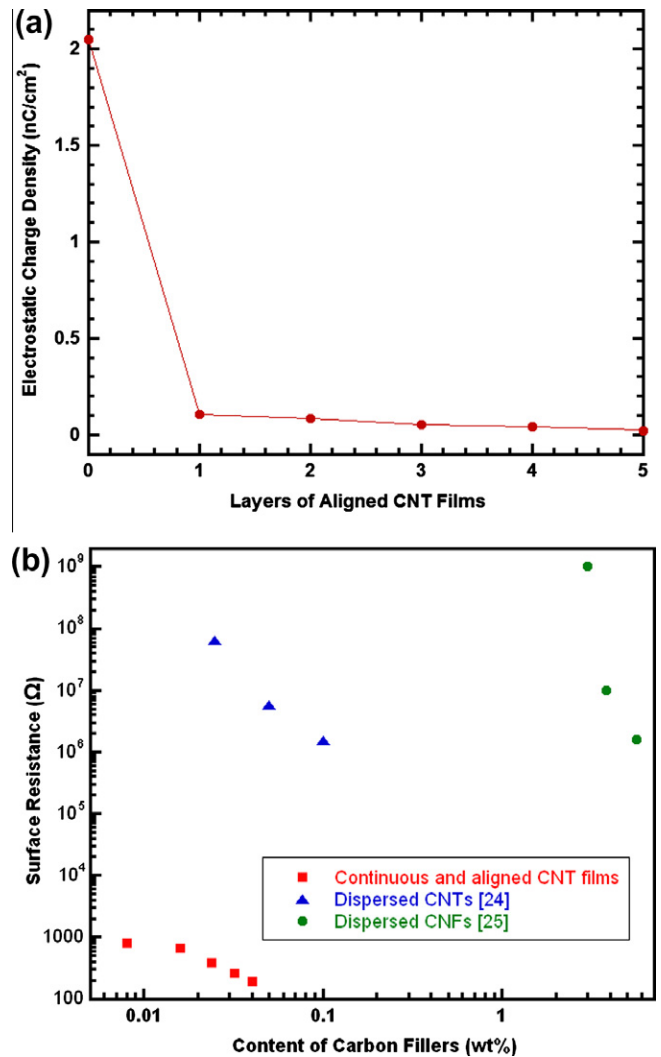


Fig. 6. (a) Surface charge density on PE surfaces welded with different layers of CNT films coating. (b) Comparison of the surface resistances of polymer substrates using different kinds of carbon fillers.

dissipation of electrostatic charge. It was reported that 0.025 wt.% of CNTs [24] and 3 wt.% of CNFs [25] were respectively dispersed into epoxy matrix to form antistatic coatings with thickness of around 200 μm and surface resistance less than $10^9 \Omega$. In this work, up to five layers of continuous and aligned CNT films were welded onto PE substrate as the antistatic coating. Assuming that the PE substrate is also 200 μm thick, then each layer of CNT film (with a density as low as $1.5 \mu\text{g}/\text{cm}^2$) equals to 0.008 wt.% addition of CNTs to the PE substrate. As shown in Fig. 6b, using the continuous and aligned CNT films, the antistatic CNT/PE nanocomposite coatings were formed with much lower surface resistance ($10^3 \Omega$) and less content of CNTs (10^{-3} wt.% of CNTs), which is several orders of magnitude lower than results in the previously reported work with dispersed CNTs and CNFs (10^6 – $10^9 \Omega$). In cases that CNT films are welded onto PE substrates thicker than 200 μm , the calculated weight fraction of CNTs should be even smaller. Therefore, using the continuous and aligned CNT film is a more efficient way to form a conductive network on polymer surfaces in antistatic applications.

From the aspect of polymer processing, conventional melting and molding processes are both energy and time consuming. Microwave welding process is preferable because microwave directly couples energy with the selective material and induces localized heating. The welding process operates at room temperature and only takes a short period (10 s) to form a nanocomposite layer, thus saves a great amount of energy. The as-produced conductive and durable nanocomposite coatings can be created on non-planar surfaces performing as antistatic shielding of gasoline pipes, fuel containers, and airplane wings, etc.

4. Conclusions

In summary, we report the formation of a uniform CNT coating onto PE substrates using an advanced microwave welding method. Upon microwave radiation, the continuous and aligned CNT films released heat rapidly and melt the PE substrate beneath in seconds. The molten PE material then filled into the spaces within CNT layers and wrapped around individual CNTs. The CNT network kept its original morphology after being welded onto the PE substrate. The CNT/PE nanocomposite coating exhibited high electrical conductivity and excellent antistatic performance with a very small amount of CNT ($1.5 \mu\text{g}/\text{cm}^2$). The CNT/PE interfacial bonds were very strong and the CNT/PE nanocomposite coating showed good durability. The combination of the superior structures of the aligned CNT films and the advantages of microwave welding technique make fabrication of such nanocomposites interesting in scientific studies and practical in engineering applications.

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