# CARBON NANOTUBE-BASED CONDUCTIVE FILMS FOR WELDING OF THERMOPLASTIC POLYMERS

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### **ABSTRACT**

Multi-walled carbon nanotube (MWCNT)-based materials, either buckypapers or nanocomposite films were fabricated and used as novel heating elements for welding of thermoplastic polymers. The heating efficiency of the fabricated films to weld polyethylene coupons was assessed using two different welding approaches: microwave welding (MWW) and induction welding (IW). MWCNTbuckypapers were fabricated by a vacuum filtration of nanotubes solution, followed by acidic treatment to remove the impurities and the filter membranes. Nanocomposite films of different thicknesses were fabricated using pre-mixed MWCNTs/polyethylene nanocomposite pellets under compression in a hot press. The buckypapers and the nanocomposite films were placed between the two thermoplastic coupons to be welded to form single lap shear specimens. The welding efficiency of both types of films was then evaluated by the variation of the films' thickness, their electrical conductivity and the welding time. The welding quality was assessed though mechanical testing of the welded coupons and optical observation of the welded interface after mechanical fracture. The mechanical strength of the joints was investigated in a lap shear mechanical testing. In general, a higher film's thickness, a higher conductivity and a longer welding time resulted in a welding with better quality and higher mechanical strength. The electrical conductivity of the films was of high importance with the IW process where the relatively lower values of the nanocomposite films led to an unsuccessful welding. The present work offers a new perspective for joining of thermoplastic polymers and pushes the boundaries towards high quality welding of thermoplastic composites using nanomaterials.

## 1 INTRODUCTION

Thermoplastic polymers and their associated composites are being increasingly used in the aerospace and auto industries. Through their ability to be melted and processed several times, thermoplastic polymers offer the possibility to be joined by welding (as opposed to mechanical fastening or adhesive binding) [1]. Polymer welding is a mature efficient technology through which the mechanical strength of the welded joint can reach up to the values of the bulk of the adherents. Several processes such as resistance welding, induction welding (IW), microwave welding (MWW) and ultrasonic welding have been developed for joining of thermoplastic-based materials [2, 3]. The principal of all these techniques is an efficient production of heat at the interface of the two components to be welded. The heat is generally provided by an external energy source which heats up an implant, called heating element, placed between the two parts. Upon the heating, the temperature of the polymer located in the vicinity of the heating element increases until it reaches a desired value.

Thus, a welded joint is achieved upon cooling under the application of pressure when consolidation takes place [4].

Different types of heating elements such as carbon fiber prepreg ply [5] and stainless steel mesh [2] have been used for welding of thermoplastic polymers and composites. Temperature non-uniformity and technical difficulties associated with the electrical connection in case of carbon fiber prepreg ply pushed researchers to seek innovative heating elements [5]. The stainless steel meshes currently used as heating elements in the industry do not always present good adhesion properties with the surrounding polymer. Therefore, further progress in advanced materials is still required to develop new types of heating elements to achieve high welding quality.

Nanomaterials, specifically carbon nanotubes (CNTs), are being used in various technological applications and serve as structural [6], sensing [7, 8] and conductive elements [9]. Recently, conductive films of CNTs have shown a high potential to be used in a wide variety of optoelectronic devices, micro electromechanical systems (MEMS) [7, 10, 11], heat exchangers [12] and electrical heaters [13]. A heater element having a layer of aligned CNTs has been developed and shown high potential for high performance electrical heaters [12, 14]. Similar to conventional heating materials such as nichrome and kanthal which are commonly used in heat generating appliance, the CNT-based heater element generates heat via Joule heating. However, those materials are limited to geometries such as strips or wires because of their insufficient electrical resistivity [12]. The CNT-based heating elements could be flexible enough so that they could be fixed to any substrate having complex geometry. In addition, CNTs are much lighter than other heating materials, thus they could offer huge benefits where weight is of importance, such as in aerospace applications [12]. As heating elements, CNTs can be used for welding of thermoplastic polymers and their associated composites. The flexibility of CNTs films would be ideal for welding geometrically complex parts.

Induction welding is a very promising technique to join thermoplastic polymers and composite components [1]. In the induction welding process, an electrically-conductive component, most often a stainless steel mesh, is used as heating element. A coil in which a high-frequency alternating electrical current is produced is placed in proximity to the mesh. The alternating electrical current produces an alternating magnetic field of the same frequency which in turn produces eddy currents in the heating element. The heating element and surrounding polymer heat up until the polymer reaches a desired welding temperature. The current is then stopped and the two polymer parts are welded upon cooling and application of pressure [1, 4]. Alternatively, if the adherents to be welded are good electrical-conductors, no heating element is necessary at the weld interface as heat is generated directly in the adherents. When adherents of low electrical conductivity are used, the electrical properties and the geometry of the CNTs could be tailored to be used as conductive heating elements. To this end, a layer of CNTs or CNTs films made of high aspect ratio CNTs should be good candidates to provide high heating rates and reach a desired temperature in a reasonable heating time.

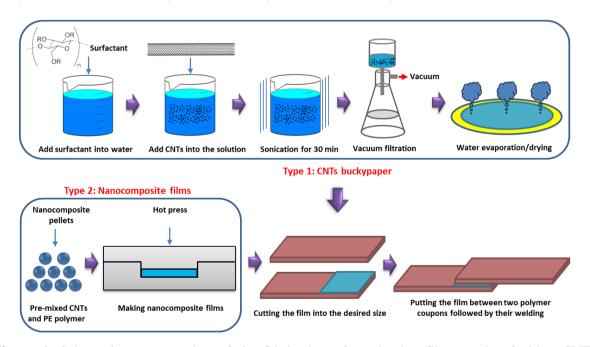
Microwave welding could also make use of CNTs to generate heat at the weld interface. It is found that a large amount of heat is released upon the interaction between CNTs and microwaves and that very high temperatures, as high as 2000°C, can be reached [15]. The concept of the heat production by microwave irradiation is that the material of interest absorbs electromagnetic energy and then dissipates it through the release of heat. Despite the extensive use of microwave irradiation to produce heat in different fields, for instance, sintering of ceramics, the MWW is still under development. MWW offers several benefits such as very short processing times (seconds), low cost and is suitable for a wide variety of materials [16].

Here, we present the use of multi-walled carbon nanotube (MWCNT) buckypapers and nanocomposite films consisting of MWCNTs and polyethylene polymer as new types of heating elements for polymer joining. The welding performance of the two heating elements is evaluated by welding polyethylene coupons to form single lap shear specimens using the IW and MWW techniques. The overall goal of this work is to find a preliminary perception about the main parameters influencing the welding ability of the nanotube-based heating elements, their benefits and constraints in the two welding processes used. The processing parameters studied here are the films' thickness, their electrical conductivity and the necessary welding time to obtain a high quality welded interface.

### 2 EXPERIMENTAL DETAILS

## 2.1 Fabrication of CNT-based heating elements

CNT buckypapers: MWCNTs produced by catalytic CVD method were purchased from Nanocyl (NC 7000) and used as received. The diameter and length of the MWCNTs were 10-20 nm and 5-30 um, respectively. The nanomaterials were observed by field emission scanning electron microscopy using a Jeol JSM-7600TFE (FESEM, 5 kV) microscope and also by transmission electron microscopy (TEM) using a Jeol JEM-2100F (FEG-TEM, 200 kV) microscope. MWCNTs were well dispersed in a solution of water and a surfactant by sonication and centrifugation as shown in Figure 1. First, 20 mg MWCNTs were added to 20 ml 1% carboxymethyl cellulose (CMC) solution. The nanotube suspension was then sonicated in an ultrasonication bath (Cole-Parmer) for 30 min and then centrifuged in a centrifugal machine at 3500 RPM for 30 min. The supernatant was finally collected and stored in a 50 CC centrifugal vial. The buckypapers were then fabricated by a commonly used vacuum filtration method in which nanotubes solution are filtered through a microporous membrane (here, 400 µm pore size). After the filtration, the dispersion was let dry at room temperature. The CMC surfactant which may act as insulating materials inside the film was dissolved and removed by immersing the buckypaper into 4M HNO3 for 12 hours. The film was then washed with deionized water by rinsing for 4 h. The films having lower thickness (50 and 100 µm thick) were collected over one of the two polymer coupons and dried. The thicker buckypapers (500-700 µm-thick) were relatively robust and easy to handle after they dried.



**Figure 1.** Schematic representation of the fabrication of conductive films made of either CNTs buckypaper (type 1) or CNTs/polymer nanocomposites (type 2) as heating elements for welding of thermoplastic coupons in a single lap shear fashion.

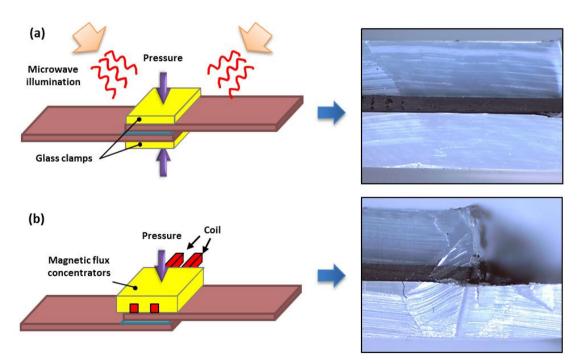
Nanocomposite films: the second type of heating element fabricated here was nanocomposites consisting of MWCNTs and polyethylene (PE) thermoplastic polymer. This pre-mixed concentrate in form of pellets was purchased from PLASTICYL<sup>TM</sup> and used to make nanocomposite films as received. The nanocomposite is composed of polyethylene loaded with 15 wt.% of MWCNTs (Nanocyl-NC7000). The nanocomposite films of different thickness were fabricated by compression molding in a hot press (Type 2 in Figure 1). A desired amount of the nanocomposite pellets were loaded between two stainless steel rectangular plates (stacked in the center). Two Teflon films were placed to separate the plates and nanocomposite pellets for easy removal of the compressed films. The processing parameters were adjusted after a few trials so that a uniform and smooth film with a proper

thickness was fabricated. A processing temperature of 140 °C was set while the pressure and the holding time varied between 2-4 bar and 10-20 min, respectively. The plates were taken out of the mold and cooled down to room temperature. The compressed sample was then cut into several rectangular specimens to dimensions of 12.7 mm × 25.4 mm using a knife cutter.

# 2.2 IW and MWW of the polymer coupons

The welding (heating) performance of the two heating elements was evaluated using two welding approaches: Microwave and induction welding methods, as schematically represented in Figure 2. The fabricated heating elements, either CNT buckypapers or the nanocomposite films, were placed between two thermoplastic coupons to be welded (Figure 1 and Figure 2). The samples were assembled in a lap shear configuration according to the ASTM D3163-01standard. Prior to the welding, the electrical conductivity of the heating elements was measured using a two-point probe electrical measurement apparatus, Keithley 4200 semiconductor parametric analyzer (MM2000 probe station). Two flat metallic tabs were fixed at the two ends of the heating elements and served as electrodes in order to minimize the contact resistance. The coupons made of high density polyethylene (HDPE) were cut from a large sheet to dimensions of 101.6 mm  $\times$  25.4 mm using a water-cooled diamond saw.

A kitchen-used microwave oven with controlled power level was used to provide microwave irradiation in MWW process. Figure 2a shows a scheme of the process and a representative optical image of the welded specimens. The single lap shear specimens were sandwiched between two glass slides and fixed using three rubber bands. The rubber bands were also responsible for providing welding pressure. The glass slides were used in order to provide a uniform applied pressure over the whole joining area. At a fixed power level (500 W), welding time varied in order to obtain the best welding quality in terms of mechanical strength.



**Figure 2.** Schemes of the two welding processes and representative optical images of the welded parts: (a) MWW and (b) IW of two polymer coupons in a single lap shear configuration using the fabricated heating elements (here, PE/MWCNT nanocomposites).

Similar materials and configuration were used in the IW method as shown in Figure 2b. The IW set-up was composed of an induction heating device including a hairpin type induction copper coil, a cooling system and a power supply, a pneumatic device to apply pressure, and a welding platform and fixture. A temperature acquisition system (Graphtec) was used to observe the temperature increase as a

function of time. The distance between the two tubes of the coil was 6.35 mm (red rods in Figure 2b). The power supply (Ambrell Easy Heat machine) was a 10 kW device with a frequency ranging from 150 kHz to 450 kHz and maximum output current of 750 A. The current frequency was automatically adjusted by the power supply based on the heating materials and the coil's impedance. Different output current values up to 700 A were tested for a welding time of 90 sec. As it can be seen in Figure 2b, the specimens were located under the coil so that the welding area was centered with the coil. A magnetic flux concentrator (the part shown in yellow) was also used on top of the coil to increase the magnetic field intensity. As it can be found elsewhere [17], this geometry of magnetic flux concentrator led to the concentration of the coil's current density on the bottom of the coil, close to the heating element.

Lap shear mechanical tests were finally carried out on the welded coupons according to the ASTM D3163-01standard for the evaluation of the mechanical strength of the joints. The tests were conducted using a tensile machine (Instron) equipped with a data acquisition software. The tests were done at room temperature using a 5 kN load cell and with a crosshead speed of 1.3 mm/min. The load-displacement data was collected until a complete failure of the specimens occurred. The lap shear strength (LLS) was then calculated from the maximum tensile force divided by the welding area. The visual quality of welding in terms of polymer diffusion at the interfaces was evaluated under optical microscopy.

## 3 RESULTS AND DISCUSSION

Figure 3 shows typical SEM and TEM images of the MWCNTs used in this study. The MWCNTs are seen as entangled materials in the SEM image (Figure 3a). The ability to manipulate these entangled structures without damaging their aspect ratio (i.e., length/diameter) and their structural integrity (e.g., defects created during acidic treatment)) is crucial in order to be effectively used for the enhancement of the electrical conductivity [18]. According to the supplier as well as our results shown in Figure 3, the MWCNTs have been purified, although few impurities (i.e., dark particles in the TEM image) are also observed. These are likely to be some residual catalyst nanoparticles, which were not entirely digested during the possible purification step. The TEM image (Figure 3b) permits the direct measurement of the nanotube diameters in which the nanotubes exhibit a diameter in the 10-20 nm range and lengths reaching up to few tens of microns.

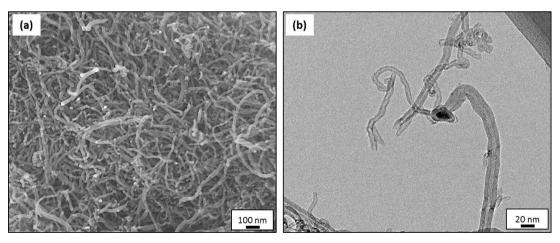
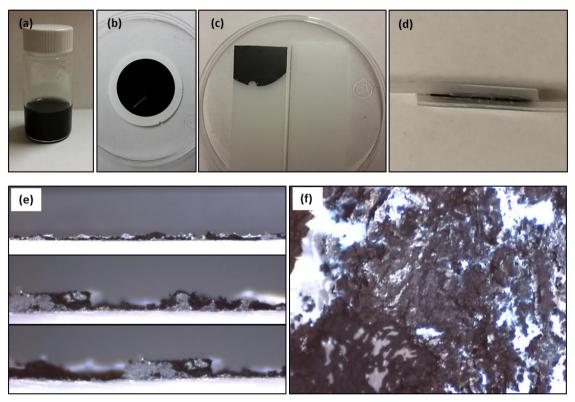


Figure 3. SEM (a) and TEM (b) micrographs of MWCNTs used in this study.

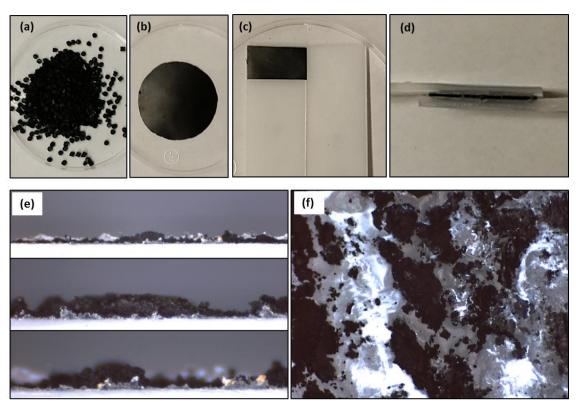
Figure 4 represents different aspects of the MWW of the polymer coupons using a CNTs buckypaper. Figure 4a shows the nanotube suspension after the sonication and centrifugation. The suspension was stable with almost no nanotube precipitation for several weeks. It indicates that the CMC surfactant was capable of individualizing the MWCNTs and stabilizing the suspension. It can be explained as the sonication of the nanotube suspension helped overcoming the van der Waals interaction between nanotubes and then the surfactant attached non-covalently to the surface of nanotubes. The mechanism of this type of interaction is based on the attachment of the hydrophobic

end of the surfactant to CNTs and the attraction of its hydrophilic end to water [19]. Figure 4b shows a representative image of the fabricated buckypapers using vacuum filtration. The buckypaper was then immersed in 4 M nitric acid and then rinsed in water before being used as a heating element for welding. It has been reported that the acidic treatment can efficiently remove the insulating surfactants from buckypapers and leads to the improvement of their conductivity by several orders. In the present work, the membrane was also dissolved in acid that helped easy buckypapers removal from the membrane. The remaining surfactant might be also removed by rinsing off in subsequent washing of the buckypapers. Figure 4c and 4d shows the specimen before and after microwave welding. Figure 4e and 4f illustrates the side-view and top-view optical images of the specimen welded area after mechanical rupture in the lap shear tensile testing. It is clearly seen that the polymer diffusion has occurred at the interface, although the welding interface is not uniform and the CNTs distorted possibly when the polymer melted and flowed.



**Figure 4.** MMW of PE coupons using CNT buckypapers: optical image of (a) nanotube suspension, (b) filtered buckypaper, (c) and (d) the specimen before and after the MWW, (e) side-view and (f) top-view optical images of the specimen welded area after mechanical rupture in tensile testing.

Figure 5 represents similar results as Figure 4 when the nanocomposite films were used as heating elements in the MWW. As shown in Figure 5, the nanocomposite pellets were compressed to make a film and then the film was cut into the desired dimension and used to weld two PE coupons. Similar to the case of buckypapers, the resin penetration is observed at the interface while the nanocomposite film is separated into parts. A few reasons might be responsible for this non-uniform welding. First, the welding pressure was applied using the rubber bands which might cause non-uniform pressure over the whole surface. This could be addressed in a future work by using plastic clamps. Second, the heat generation in microwave is not uniform, and the microwave irradiation mostly affects the edges, as it has been seen in the other welding processes such as induction and resistance welding. Third, several ignition were observed during the welding as a result of oxidation. This flammability issue can be later addressed by welding in a controlled no-oxygen atmosphere. Therefore, a uniform heat distribution can be expected and the heating elements are no longer flammable [20].



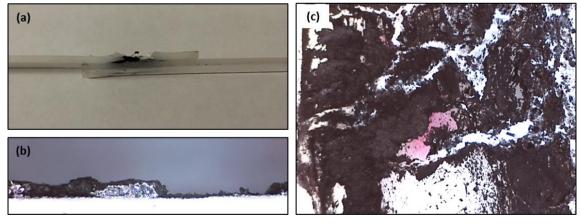
**Figure 5.** MMW of PE coupons using PE/MWCNT nanocomposites: optical image of (a) nanocomposite pellets, (b) a hot pressed film, (c) and (d) the specimen before and after the MWW, (e) side-view and (f) top-view optical images of the specimen welded area after mechanical rupture.

The capability of the two types of heating elements was tested in the IW method. Table 1 lists the performance of both types of heating elements in the two welding methods with detailed processing conditions. The IW of polymer coupons using the nanocomposite heating elements was unsuccessful under any processing conditions using the present induction welding setup. Almost no welding was seen with the variation of film's thickness, the distance from the coil, and the input current. Similar no-welding condition was observed for IW of the polymer coupons when the lower-thickness buckypapers (e.g., 50-100 µm-thick) were used. For all of these conditions, the temperature acquired at a close distance from the interface gradually increased and stopped at a maximum temperature of 90 °C. Actual induction welding using CNT buckypapers only occurred under a certain condition when a thick layer (700 µm-thick) at a close distance from the induction coil was used. Figure 6 shows the optical images of the welded specimen and also the side-view and top-view of the interface fractured under mechanical testing. Although the resin diffusion is observed in some regions (Figure 6b), the high thickness of the film limited a proper polymer penetration and thus an efficient welding, as it is seen in Figure 6c. The main reason for the unsuccessful IW is the low electrical conductivity of the two types of heating elements when compared to that of commonly-used stainless mesh (Resistance  $=\sim 10 \Omega$ ). The electrical conductivities of the different types of the films are compared in Table 1. The nanocomposite films and 50 µm-thick buckypaper are almost two orders more resistive than the stainless steel mesh and the 700 µm-thick buckypaper.

The MWW process is found to be more efficient than the IW method in this study since the welding either partially or entirely happened for both types of fabricated heating elements. The reason might be that the interaction between CNTs and microwaves can produce a large amount of heat that can helps reaching high temperatures [15]. According to the results shown in the table, a higher welding quality in MWW process can be achieved by the decrease of the films' thickness and the increase of the welding time. The occurrence of ignition can be then minimized by controlling those parameters as well as by protecting the specimen from oxygen. This may lead to achieve the best welding quality (resin diffusion and mechanical strength) using these nanotube-based materials.

Welding method	Sample	Welding condition	Welding quality	Resistance $(\Omega)$	LSS (MPa)
Polymer coupon	Neat PE polymer	-	-	Insulator	TS ~8.3
Induction welded	Buckypaper Thickness= ~50 μm	Current = 700A Time = 90 sec	No welding	2340	-
	Buckypaper Thickness= ~700 μm	Current = 700A Time = 90 sec	Partially welded	17.4	4.6
	PE-NC-15 wt.%MW Thickness= ~500 μm	Current = 700A Time = 90 sec	No welding	802.7	_
Microwave welded	Buckypaper Thickness= ~50 μm	Power = 500 W $Time = 10 sec$	Partially welded	2340	2.3
	Buckypaper Thickness= ~50 μm	Power = 500 W $Time = 20 sec$	Good welding	2340	5.4
	Buckypaper Thickness= ~100 μm	Power = 500 W Time = 10 sec	Partially welded	1042	2.8
	Buckypaper Thickness= ~100 μm	Power = 500 W Time = 20 sec	Partially welded	1042	5.1
	PE-NC-15 wt.%MW Thickness= ~250 μm	Power = 500 W Time = 30 sec	Partially welded	1532	4.4
	PE-NC-15 wt.%MW Thickness= ~250 μm	Power = 500 W Time = 60 sec	Good welding	1532	7.9
	PE-NC-15 wt.%MW Thickness= ~500 μm	Power = 500 W Time = 30 sec	Partially welded	802.7	3.7
	PE-NC-15 wt.%MW Thickness= ~500 μm	Power = 500 W Time = 60 sec	Partially welded	802.7	6.1

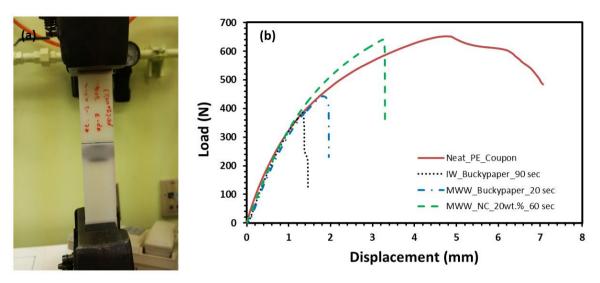
**Table 1:** Welding conditions and quality for the IW and MWW of the polymer coupons.



**Figure 6.** IW of PE coupons using the CNT buckypapers: optical images of (a) the specimen after the IW, (b) side-view and (c) top-view of the specimen welded area after mechanical rupture.

Lap shear mechanical tests were finally carried out on the welded coupons for the evaluation of the mechanical strength of the joints. Figure 7a shows a representative photo of the welded PE polymer coupons during the lap shear mechanical testing and Figure 7b illustrates the load-displacement curves of the selected specimens with the best mechanical strength values in each welding category. The results obtained for all the other specimens are also listed in the last column of Table 1. For all the welded specimens, a sudden fracture occurred. The maximum force value for the induction-welded coupons using the thick buckypapers was found to be ~380 N. This value increased to ~450 N and

650N for the microwave-welded specimens using the buckypaper and nanocomposite film, respectively. The highest value of lap shear strength (LSS) was achieved for the microwave welded specimens using the nanocomposite film having a thickness of ~250 µm and the welding time of 60 sec. The results are in good agreement with the quality of the interface discussed earlier using the optical microscopy results. According to Table 1, a longer MW irradiation time leads to higher heat generation, which allows melting a larger polymer area at the weld interface, resulting in better polymer diffusion and stronger bonding. However, the electrical conductivity should be controlled to avoid ignition and fire as a result of high conductivities.



**Figure 7.** (a) A representative photo of the welded PE polymer coupons during the lap shear mechanical testing using a 5 kN load cell and a displacement speed of 1.3 mm/min and (b) load-displacement curves of the selected different specimens.

# **CONCLUSIONS**

Two conductive nanotube-based films were fabricated and tested as new types of heating elements for welding of thermoplastic polymers using microwave and induction welding processes. The quality of welding was characterized under the optical microscopy and using mechanical testing in a single lap shear configuration. The MWW showed a higher efficient method compared to the IW using these materials due to high rate of heat generation as a results of microwaves and CNTs interaction. The mechanical strength of the microwave welded specimens revealed a high value of 7.9 MPa which is close to that of the polymer bulk. Future works will focus on finding the best welding conditions in terms of avoiding ignition and non-uniform welding interface as well as control the conductivity of the heating elements, making them suitable for each technique. The ultimate goal of this study is to tailor the properties of these heating elements to be used as heating elements for welding of high-performance thermoplastic composites currently used in aerospace structures. The development of this type of heating elements will address the issues existing with metallic meshes and make the repairs of composites less-expensive and environmentally friendly. This will be of great interest for the aerospace industry in terms of maintenance and cost reduction. The present work offers a new perspective for joining of thermoplastic polymers and composites.

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