

Microwave Joining of Polymers

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Abstract: *Some products have a very complex structure to be molded as a single piece; this creates a hindrance for the manufacturers to let them mold single component products using polymers. Thus, in manufacturing of many plastic-based products into final products, assembly of subcomponents plays an important role. Presently, the most efficient joining method for plastics is welding. Compared to conventional heating techniques, microwave heating has many advantages due to the ability to heat the material directly through interaction of electromagnetic radiation. Therefore, microwaves can be seen as a direct means of forming and welding since it can rapidly melt thermoplastics in a highly localised way. In this study, considering multiwalled carbon nanotubes (MWCNTs) as a good microwave absorber, it is used as a susceptor for the microwave welding of high-density polyethylene (HDPE) and further results are analysed to carry out in-depth study of microwave welding. The dispersion created by ethanol and multiwalled carbon nanotubes in an ultrasonic bath, is dropped on a targeted area on the dumbbell-shaped sample and then dried in an oven for 30 min at 45°C for further microwave irradiation of 800W in a domestic microwave oven. Then with the help of tensile testing, the strength of the weld is tested and scanning electron microscopy (SEM) is used to analyse the cross section of the welded joint. In the study of the effect of MWCNT concentration and microwave heating duration, it is found that as the heating duration increases from 2 s to 8 s, the joint strength increases but decreased when the duration is further increased to 10 s. When duration of 10 s is used, the SEM images captured void formation at the joint interface which resulted in lowering of joint strength. The effect of the MWCNT concentration in the dispersion shows increased joint strength when the concentration is increased from 0.25 wt% to 0.75 wt% but decreased in the presence of 1.00 wt% concentration.*

Keywords: Microwave Welding, Polymers, HDPE, MWCNT, SEM

1. Introduction

Thermoplastics have a wide range of applications. High-density polyethylene (HDPE) is a thermoplastic which is used in automobiles, dental implants, orthopedic implants, and medical devices due to their ability to take a good finishing, good corrosion resistance, and excellent strength to weight ratio. However, the structures of most medical devices and final products are far too complex to be molded as a single piece so the manufacturers' goal of molding single component products from plastics is oftenly not fulfilled. Mechanical joining, adhesive bonding, and welding are the plastic joining techniques which are practised for the assembly of plastic components into final products.

Fasteners like screws, bolts, and nuts are used in mechanical joining. It is easy to manipulate but stress concentration easily appears at the bonding region which decreases joint reliability resulting in the increase of weight, thus deteriorating the design of lightweight [1]. Adhesive bonding is relatively mature. It involves the use of adhesive between components, in which the adhesive transmits load through the joint. But adhesive bonding needs a long process cycle and properties of adhesive joints such as impact resistance, fatigue resistance, humidity resistance are insufficient [1] and the mechanical properties of adhesively bonded joints are dependent on environmental factors.

Till date, welding is known to be the most efficient method of joining for plastics. It involves melting of plastics at the joint interface followed by the intermolecular diffusion and chain entanglements for the formation of joints. [2] However, not all polymers can be welded. Thermosets and thermoplastics are the two categories of polymers. In case of thermosets, during the processing and curing, irreversible cross-linking reaction occurs, and, therefore, thermosets cannot be reshaped by the application of heat. So joining of thermosets can only be done by mechanical joining and adhesive bonding. On the other hand, thermoplastics are

well known for their weldability because of their ability to be softened, melted, and remolded by reheating. Thermoplastics can be welded upon application of heat and pressure. [3]

Welding process can be categorized based on the heating method, namely external heating and internal heating. External heating methods such as hot tool, hot gas, and extrusion rely on convection and/or conduction to heat the weld surface. Welding by internal electromagnetic heating is a relatively new method that relies on the absorption and conversion of electromagnetic radiations such as microwave and laser into heat. Laser welding has been used for the welding of thermoplastic dental implants, orthopedic implants, and medical devices because it is a clean, precise, and near error-free joining process. However, laser welding requires extremely expensive equipment and high capital investment which incurs high cost to the final products.

Microwaves are known to have a wide range of applications in the heating and processing of polymer based materials [4]. Microwave heating consumes up to 100 times less energy than conventional heating [5] due to its volumetricity and occurs at the molecular level. Therefore during composite production it has the advantage of greater energy savings and reduced processing times. Additionally, regardless of geometries, microwaves offer uniform and complete heating of parts [7]. This is extremely beneficial since thick, and nonuniform parts are not very suitable for autoclave curing as the undesirable thermal gradients make it difficult to ensure uniform and complete curing.

Microwaves have a potential use for welding or bonding in polymer materials. Usually they are joined using mechanical fasteners or adhesives, but these methods require extensive surface preparation and can result in stress fields localized in the machined areas required for the placement of fasteners. Therefore, Polymer welding can be seen as an economical and effective bonding alternative. Till date, induction, lasers,

friction stir, hot plate, ultrasonic are some techniques in use. Since simultaneous irradiation is necessary for complex 3D structures, microwave welding can be considered advantageous over other forms. Since a lot of polymers have poor microwave absorption capabilities, the use of microwave adsorbent additives is required known as susceptors. Materials with high dielectric loss factors require susceptors while the ones with low-to-medium dielectric loss factors do not require them [8]. Since Carbon nanotubes (CNTs) absorb the energy and convert it into heat due to electronic losses within the CNT structure, they are considered as an ideal microwave absorbing material [9]. Appropriate dispersion is required to reach the percolation since microwave absorption is a bulk process; this minimizes the thermal gradient in the composite. CNT facilitates local heating of the interface of the surfaces to be welded. Microwave heating occurs only at the interface to have lower power consumption as heating of the entire material is not required. CNTs can also provide exceptional strength to the weld joint through interface bridging. Many studies have proved the exceptional capability of CNTs to efficiently weld polymers [10]. It is clear that new developments in the field of microwave welding of composite materials can bring advancements in additive manufacturing.

2. Literature Review

Microwave is electromagnetic radiation that has a wavelength range of 1 mm to 1 m and frequency range of 300 MHz to 300 GHz and. A susceptor is an electromagnetic absorbent material; a layer of susceptor is required in microwave welding. It is placed between the parts that are to be joined. [11] The susceptor material melts and heats the surrounding polymer via conduction when it gets heated by absorbing microwave energy [12]. Materials having polar groups in their molecular structure absorb microwave energy readily. Polyaniline (PANI) is the most common implant material. Some thermoplastics containing polar groups in their molecular structure such as nylon, polyvinyl chloride and acrylonitrile butadiene styrene get welded without an additional absorbing layer. Since this is a relatively new polymer welding technique a very few studies have been done on microwave welding of thermoplastics till date. Wu and Benatar used PANI at the weld line to develop a microwave joining method (butt joint) for HDPE. [13] They observed that the strength was influenced by the thickness of the molten layer and stronger bonds are a result of greater thickness. The weld has a tensile strength equal to that of the HDPE bulk strength (25 MPa) at the optimal welding conditions. Staicovici et al. carried out the study of the welding and disassembling of HDPE butt joints with various PANI concentrations at the weld line and discovered that under the right conditions the tensile strength of the joints could be similar to that of the bulk material strength of HDPE. [14] But, the PANI concentration that resulted in effective disassembly gave a maximum joint strength of only 80% of the bulk material. Those PANI concentrations did not disassemble effectively and achieved joint strengths equal to that of bulk material. [11]

Other than various polar materials, with the help of many carbon-based materials such as graphite and carbon

nanotubes (CNTs), several attempts have been made for the welding of thermoplastics. Due to their excellent relative permittivity and conductivity, carbon materials show high microwave absorbing properties. [15] Recently, Sun et al. used graphite powder as a susceptor and successfully welded polypropylene (PP). [16] They observed that when graphite powder was used after subjecting to ball-milling (90 μm), the joint strength of the welded joint is higher compared to those without subjecting to ball-milling (250–850 μm). They reported that small sized graphite powder could easily absorb microwave energy due to their high surface-to-volume ratio.

Carbon Nanotubes (CNTs) are nanoscale tubular allotropes of carbon having high length to diameter ratio. In single-walled CNTs and multiwalled CNTs (MWCNTs), they consist of single or multiple concentric rolled graphene layers. CNTs have excellent mechanical properties and high aspect ratios. They have large interfacial areas that render them promising reinforcements by improving the mechanical properties of polymer matrix composites. Kalakonda et al. prepared MWCNTs/polyurethane nanocomposites by backfilling preformed hydrogels and aerogels of individually dispersed MWCNTs (MWCNTs-Baytubes) and thermoplastic polyurethane. [17] In the study, they started by preparing a porous network form by solution fabrication method with MWCNTs in aerogel-and hydrogel-forms concentration for prevention of agglomeration. Then they soaked the porous network in a 1–6 wt% polymer solution for 5–10 h for facilitating polymer infiltration into the nanotube network. Further they annealed the composites under vacuum for 12 h at 150°C, evaporated the solvent, and then by hot-pressing method at 130°C for 15 min removed all voids. They found a 200-fold improvement in tensile modulus over pristine polymer when 19 wt% MWCNT loading was used. This indicates that mechanical properties of polymer matrix composites improve when MWCNTs are used. But the process involved several complex steps and took about 18 h for preparation of nanocomposites. Recently, for the welding of thermoplastics, CNTs/polylactic acid composite coating was used. The CNTs are a good microwave absorber due to the presence of delocalized sp^2 π electron. [18] Sweeney et al. found that using intense localized heating of CNTs/PLA coating by microwave irradiation for about 60 s, the welding of 3D-printed thermoplastic is possible. [19] The weld fracture strength showed an improvement upto 275%. In a study, Sun et al. used a pre-prepared CNTs-PLA solder system for microwave welding of PP. [20] They observed that the formation of CNT-filled PP nanocomposite at the joint resulted in high bonding strength for welded PP joints. All these studies show that using CNTs-filled composites coating as a susceptor for microwave welding provides fast joining of thermoplastic with a welded joint of excellent strength. However, before they can be used as microwave susceptors, several more steps are required for the preparation of CNTs/PLA solder system or CNTs-filled composite coating. Besides, in the form of bulk solid the microwave susceptors such as CNTs-filled composite coating cannot be used for the welding of structures having a complex geometrical configuration.

Presently, no study has been done till date on the use of only CNTs as the susceptor for microwave welding of thermoplastic. CNTs filler composite coating does not require any preparation when CNTs alone are used as susceptors, therefore the microwave welding process becomes less time-consuming by being achieved within 1 min. Also, for structures with complex geometrical configuration, CNTs can be easily used as susceptors. MWCNTs-polymer composites can have various advanced applications with the involvement of microwave welding. Jang et al reported such application, detection of the freezing temperature and deicing by self-heating with the help of smart coating materials.

3. Objective

Welding of thermoplastics has a wide range of applications in our daily occurrence. Till date, on an industrial scale it is practiced using techniques based on ultrasonics, infra-red heating vibration, resistive implantation etc. Since microwave engineering has seen adequate development recently only and due to the lack of presence of suitable microwave absorbers, the area of microwave welding is largely neglected. In this study, we see the use of MWCNTs as a susceptor for the microwave welding of HDPE. The thickness of the molten layer at the joint is affected by the microwave heating duration and the amount of susceptor used. Therefore, in this study we will report the effect of microwave heating duration on the tensile strength and microstructure of the cross section of the welded joint and the effect of MWCNT concentration in dispersion.

Materials Used

In a typical experiment, HDPE (high-density polyethylene) pellets, MWCNTs (multiwalled carbon nanotube), and a small amount of ethanol are used without any additional purification.

4. Methodology

HDPE pellets are used to produce dumbbell-like shaped samples with the help of a hot press machine and ASTM D638 Type IV dumbbell cutter. Then the samples are cut into the shape as shown in Figure 1. Then MWCNTs is mixed with ethanol and distilled water (weight ratio 1: 1) in four mass fractions, which are respectively 0.25, 0.50, 0.75, and 1.00 wt% with the help of an ultrasonic mixing bath, for a time duration of 1 h.

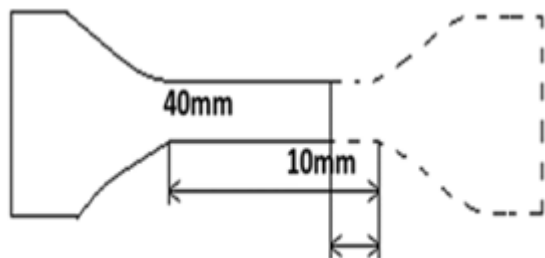


Figure 1. Sectioned dumbbell-shaped sample

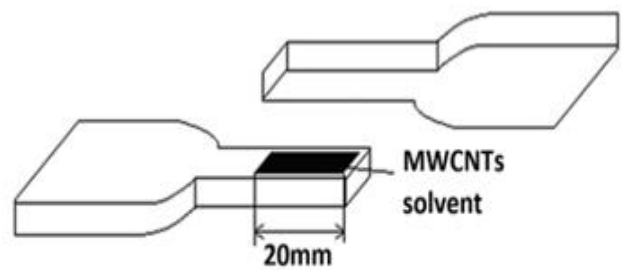


Figure 2. HDPE dumbbell shaped sample with MWCNTs coating between their targeted area.

A targeted area of 1 cm^2 is marked on the HDPE sample. Then, about 0.15 ml of MWCNTs containing dispersion is placed on the targeted area and dried by keeping in an oven at 45°C for 30 minutes. Then another sample is placed on top of the sample with MWCNTs coating (Figure 2) and the sample is exposed to microwave heating.

For microwave heating the sample, it is placed in an 800 W domestic microwave oven. To study the effect of duration of heating, microwave heating time of 2, 4, 6, 8, and 10 s and dispersion with 0.50 wt% MWCNT concentrations is used. To study the effect of concentration of MWCNTs in dispersion, microwave heating duration of 8 s is used and the MWCNT concentration in dispersion is varied from 0.25 wt % to 1.00 wt%. A glass slide is used for exerting pressure on the sample and the cutoff parts are used for balancing the sample, as shown in Figure 3.

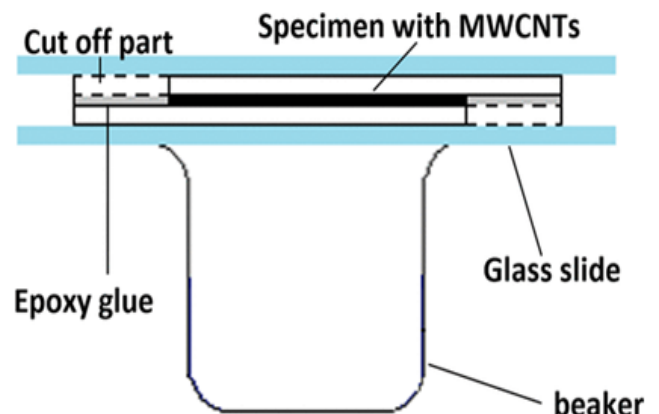


Figure 3: Schematic diagram of setup for microwave heating in a microwave oven

Characterizations

A scanning electron microscope (SEM) is used to examine the cross section of the samples. For obtaining the cross section sample of the welded joint, cryofracture method is used. For about 1000 s, the sample is put into 500 ml of liquid nitrogen. Immediately after removing the sample from liquid nitrogen cutting at the welded joint is done. Before subjecting the sample for examination using SEM, a coating of platinum is done using a sputter coater.

A universal testing machine (UTM) is used for studying the mechanical properties of samples. The crosshead speed of UTM is set to 50 mm/min. The cutoff parts of dumbbell shape are attached to the samples with the help of epoxy glue so that the sample remains straight and balanced when

it is gripped to the UTM machine. Then the sample is pulled until it reaches the fracture point. The UTM setup of the sample is shown in Figure 4.

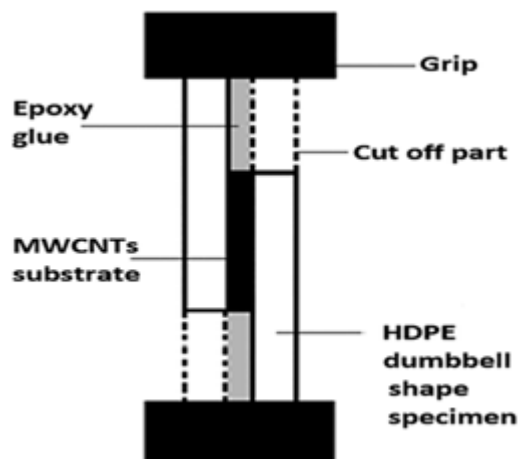


Figure 4: Schematic diagram of gripping of sample in UTM machine. UTM: universal testing machine.

5. Results and Discussions

Effect of microwave heating duration

Figure 5 shows tensile strength comparison of the samples when subjected to different microwave heating duration, it is clearly visible from this that on increasing heating duration from 2 s to 8 s, the tensile strength increases. The samples subjected to 2 and 4 s have a tensile strength comparatively lower than that of pure HDPE. At 8s, the maximum tensile strength of 7.5 MPa is observed. When 10 s is used, the tensile strength decreases. When the heating duration is increased from 2 s to 8 s, MWCNTs absorb sufficient energy from the microwave, forcing the surrounding HDPE to melt, which subsequently improves joint strength in the welded joint. A void defect may form at the joint due to prolonged microwave heating and subsequently result in lower joint strength when the heating duration is increased to 10 s. Wu et al. observed a similar result, in their study of using MWCNT/PP composite in the welding of PP substrates. When the heating duration is increased from 30 s to 50 s, the strength increases. Wang et al. mentions in his study that when the heating duration is increased from 5 s to 10 s, the peeling strength increases of polycarbonate (PC) /MWCNT

due to intercalation of MWCNTs by microwave heating into PC surface. The samples subjected to 6 and 8 s have a tensile strength slightly higher than that of pure HDPE. CNTs have a tensile strength higher than that of HDPE. In this study, due to the addition of CNTs as susceptors formation of CNTs-filled HDPE nanocomposite is observed at the welded joint which enhances mechanical properties more than that of pure HDPE. Therefore, when subjected to 6 and 8 s, the samples have higher tensile strength than pure HDPE.

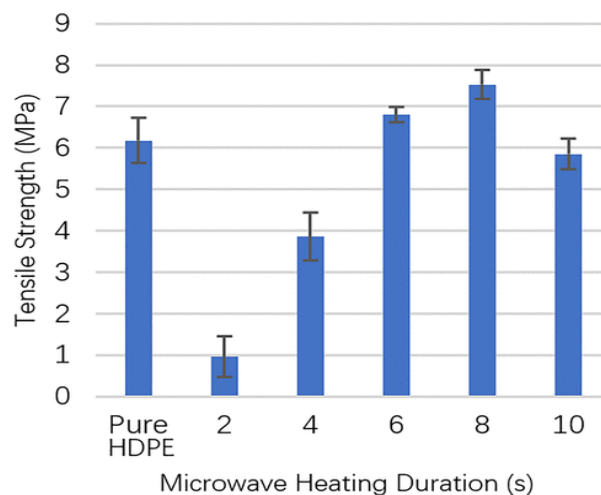


Figure 5: Tensile strength of samples subjected to different microwave heating duration.

The photograph of tensile tests of samples when subjected to different microwave heating duration is shown in Figure 6. The sample in Figure 6 (a) shows that the sample when subjected to 2 s of heating duration detached during the tensile test, this shows that the welded joint is not strong enough to hold the welded dumbbell-shaped sample together, resulting in a low tensile strength. Figure 6 (b) shows that the sample experienced necking before failing when subjected to 8 s of heating duration, this failure occurred at the point right next to the welded joint. In Figure 6 (c), the sample fails at the welded joint when subjected to 10 s heating duration. The presence of voids in the welded joint is clearly visible in the figure.

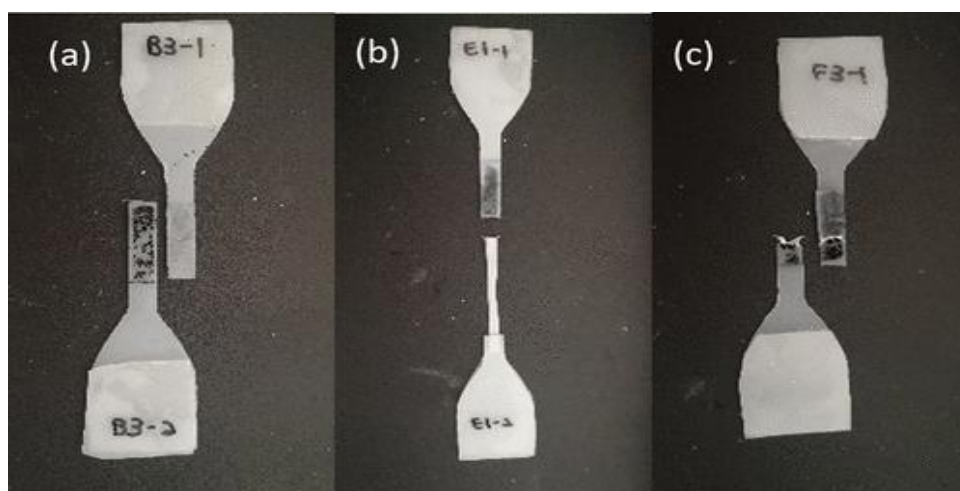


Figure 6: Photograph of tensile test sample of HDPE subjected to heating duration of (a) 2 s, (b) 8 s, and (c) 10 s.

In Figure 7, the SEM (scanning electron microscope) images of the cross section of the welded joint of samples are shown which are subjected to microwave heating of 8 and 10 s. The presence of welded joints is shown in Figure 7 (a), marked by a yellow circle. Figure 7 (b) is the magnified version of the welded joint area; it shows the formation of MWCNTs-filled HDPE composite at the welded joint. MWCNTs-filled HDPE composite is formed at the welded joint due to good microwave absorbing properties of MWCNTs. MWCNTs

absorb the microwave energy and get heated up when the sample is exposed to microwave heating. The surrounding HDPE gets melted from the heat from MWCNTs, allowing the MWCNTs to enter into the melted HDPE. The molten MWCNTs containing HDPE solidifies into MWCNTs-filled HDPE composite at the welded joint when the microwave oven is turned off.

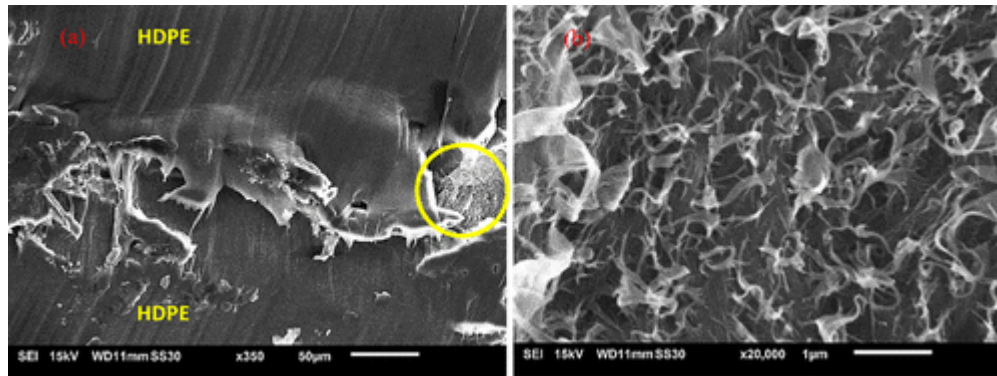


Figure 7: SEM image of cross section of welded joint for sample that was subjected to microwave heating of 8 s: (a) $\times 50$ and (b) $\times 20,000$.

The SEM image of the cross section of the welded joint of a sample which is exposed to 10 s of microwave heating is shown in Figure 8. Due to the prolonged microwave heating of 10 s, a void is observed at the welded joint, which results in the thermal degradation of HDPE. Several volatile or other decomposition products may be formed by thermal degradation of the HDPE which might cause voids at the weld interface. For 10 s samples due to the poor weld quality, reduction of tensile strength takes place due to the presence of void, as shown in Figure 5.

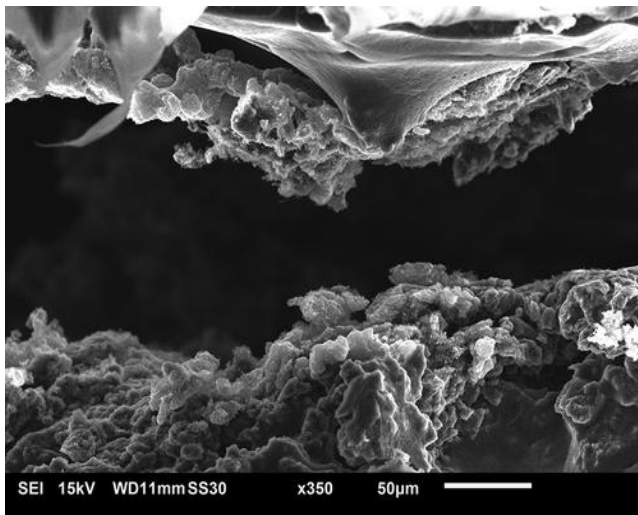


Figure 8: SEM image of cross section of welded joint for sample that was subjected to microwave heating of 10 s.

Effect of concentration of MWCNTs in dispersion

The comparison of tensile strength for the samples that are prepared by dispersion with different MWCNT concentrations is shown in Figure 9. It can be noted that when MWCNT concentration in dispersion is increased from 0.25 wt% to 0.75 wt%, tensile strength of the samples increased with 8MPa being the highest tensile strength when

0.75 wt% MWCNTs is used. However, when concentration of MWCNTs in dispersion was further increased to 1.00 wt% sample, a drop in tensile strength is observed. It is believed that the higher filler loading in the welded joint led to higher joint strength when MWCNT concentration in dispersion increases from 0.25 wt% to 0.75 wt%. But, on further increase to 1.00 wt% in MWCNT concentration in dispersion, formation of inhomogeneous-welded joint or welded joint with defects occurs as the amount of MWCNTs might be too high, which further causes stress concentration.

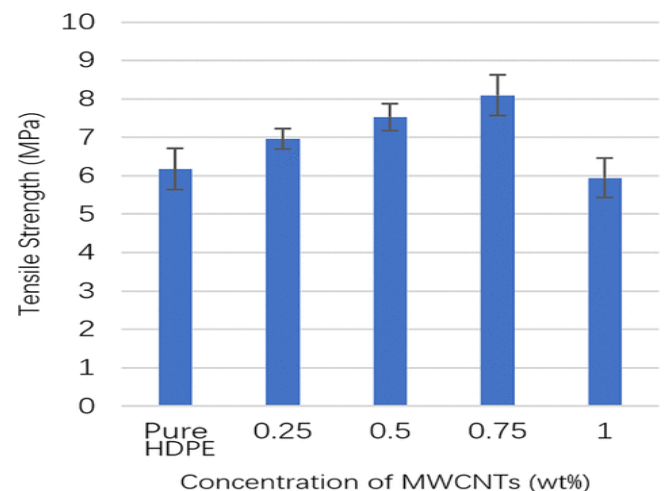


Figure 9: Tensile strength of samples prepared using dispersion with different concentrations of MWCNTs.

Figure 10 is the comparison between the SEM image of the sample prepared using dispersion with MWCNT concentration of 0.50 and 0.75 wt%. The presence of protrusion of MWCNTs from the HDPE surface is shown in both the images which indicate the formation of MWCNTs-filled HDPE composite at the welded joint. In Figure 10 (b), it is also noted that the sample prepared using dispersion with 0.75 wt% indicates significantly higher MWCNTs

protrusions compared to the 0.50 wt% sample. The MWCNTs-filled HDPE composite formation with higher

filler loading at the welded joint results in higher tensile strength for 0.75 wt% sample.

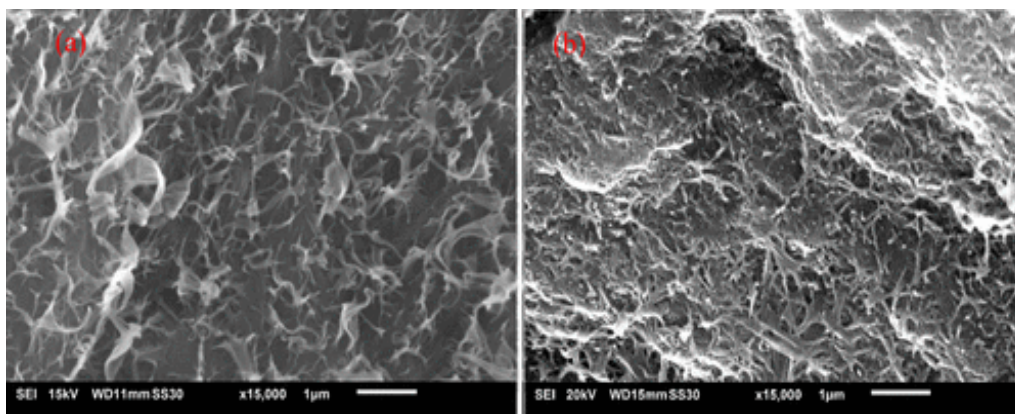


Figure 10: SEM image of cross section of welded joint for sample that was prepared using dispersion with concentration of MWCNTs: (a) 0.50 wt% and (b) 0.75 wt%.

The SEM images of the cross sectional area of the welded joint is shown in Figure 11 for sample which is prepared using dispersion with 1.0 wt% MWCNT concentration. In Figure 11 (a), the presence of void is observed at the welded joint between the MWCNTs-filled composite. Figure 11 (a) is the magnified version of the area denoted by the yellow circle in Figure 11 (b) which shows that the surface is

packed with free-standing MWCNTs. These penetrated into only one side of the HDPE plate. This shows that a thick layer of MWCNTs, which penetrated to only one side of the HDPE plate, is formed as the amount of MWCNTs at the welded joint is too high. This results in a weak-welded joint with lowered tensile strength as MWCNTs-filled composite layer cannot effectively join the two HDPE interfaces.

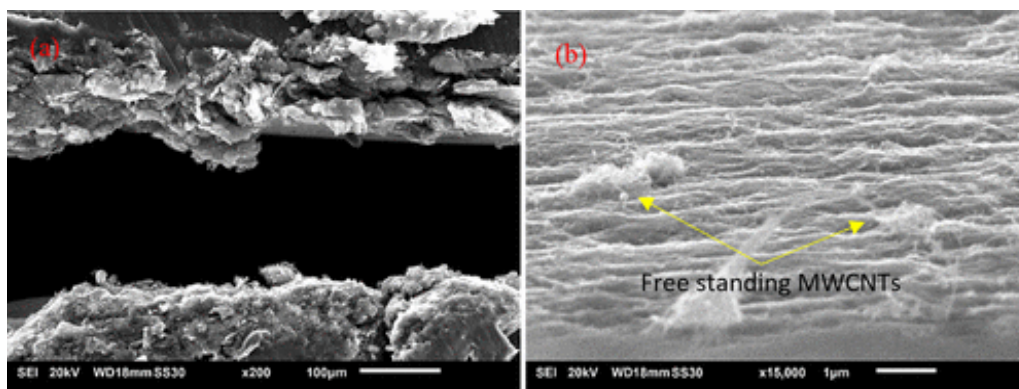


Figure 11: SEM image of cross section of welded joint for sample that was prepared using dispersion with 1.0 wt% concentration of MWCNTs: (a) $\times 200$ and (b) $\times 15,000$.

6. Conclusions

In conclusion, it has been shown that MWCNTs can be used as a susceptor for the microwave welding of HDPE. In the experiment of effect of microwave heating duration, the heating duration was varied from 2 s to 10 s and dispersion of MWCNTs with a concentration of 0.50 wt% was used. When the heating duration was increased from 2 s to 8 s, the tensile strength of the welded samples increased while decreased when it was further increased to 10 s. When 10 s is used, due to the formation of void at the welded joint a reduction in tensile strength takes place. In the effect of concentration of MWCNTs in dispersion experimental study, the concentration of MWCNTs in dispersion varied from 0.25 wt% to 1.00 wt% and 8 s of microwave heating duration. It was reported that for the welded samples, the tensile strength increased when the MWCNT concentration increased to 0.75 wt% and decreased when it was further increased to 1.00 wt%. This lowering of the tensile strength is due to MWCNTs penetration on one side of the HDPE

plate. This results in a weak-welded joint due to ineffective joining of the two HDPE interfaces.

The technology of microwave welding is currently at a very early stage of development. It would definitely have broader industrial applications as we unfold a better understanding of the underlying principles. Microwave joining techniques for various materials is a continuous development, where each discovered technique might differ substantially in design, allowing improvements for better bond quality.

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