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A Comparative Study of Microwave Welding Using Multiwalled Carbon Nanotubes and Silicon Carbide Nanowhiskers as Microwave Susceptors

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ABSTRACT

Recently, microwave welding has arisen as an advanced joining method due to its versatility and rapid heating capabilities. Among others, microwave susceptors play a crucial role in microwave welding, as different classes of microwave susceptors have distinct microwave heating mechanisms. In this work, polypropylene (PP) was utilized as a thermoplastic substrate and two types of microwaves susceptors, namely multiwalled carbon nanotubes (MWCNTs) and silicon carbide nanowhiskers (SiC NWs), were studied for microwave welding. The susceptor was first dispersed in acetone to form susceptor suspension. Next, the susceptor suspension was deposited onto the targeted area on substrate and paired with another bare PP substrate. The paired sample was then exposed to 800 W microwave radiation in a microwave oven. Afterward, the welded joint was evaluated using a tensile test and scanning electron microscopy to determine its joint strength and cross-section microstructure. The results showed that the joint strength increased as the heating duration increased. The welded joint formed using MWCNTs achieved a maximum strength of 2.26 MPa when 10 s was used, while the SiC NWs-formed welded joint achieved a maximum strength of 2.25 MPa at 15 s. This difference in duration in forming a complete welded joint can be attributed to the higher microwave heating rates and thermal conductivity of MWCNTs. However, increasing the heating duration to 20 s caused severe deformation at the welded joint and resulted in low joint strength. Overall, this study highlights the significance of understanding the microwave heating mechanism of different susceptors and provides essential insight into the selection of a microwave susceptor for microwave welding.

Keywords: *Susceptor, Microwave Welding, Carbon Nanotube, Silicon Carbide, Nanocomposite*

1. INTRODUCTION

In modern daily life, electromagnetic radiation applications, especially microwave radiation such as radar systems and wireless telecommunication, have become indispensable. Essentially, microwave radiation refers to electromagnetic waves that fall within the wavelength range of 1 mm to 1 m [1]. With the global concern over the fossil fuel crisis, microwave heating has emerged as an alternative heating method for household appliances, including microwave ovens. Unlike conventional heating, microwave heating is not limited by the heat transfer from the external heating sources. Instead, it heats materials volumetrically through intermolecular interaction, leading to rapid and efficient heating. The reverse heating profile is another notable feature of microwave heating. In conventional heating, the external heating source heats the material from the outside to the center. However, microwave heating works the opposite way, heating the material inside out [2]. This unique heating process minimized the temperature gradient issues, resulting in time and power savings.

Microwave welding is a joining method that involves microwave heating and a material with exceptional microwave-absorbing abilities, known as a microwave susceptor [3]. Aside from the advantages associated with microwave heating, microwave welding also presents benefits with its versatility for different joining geometries and simplicity of disassembly [4]. For most of the cases, like ceramic and polymer, a microwave susceptor is necessary at the joining interface for microwave welding, as these materials are microwave transparent and do not associate with the microwave radiation directly. During the microwave welding process, the microwave energy absorbed by the microwave susceptor is transferred to the neighboring material until it attains the softening point of the material, allowing the joining interface to coalescence as a one-piece product upon cooling and solidification [5].

But different classes of microwave susceptors have different microwave absorbing mechanisms. According to Amini *et al*., microwave heating involves three distinct mechanisms contributing to the process, including conduction loss, dielectric loss, and magnetic loss [6]. Conduction loss, also known as joule heating, is primarily found in materials with excellent electrical conductivity, such as metals and carbon-based materials. This class of susceptor has an abundance of delocalized electrons, and the heat is produced due to the energy dissipation caused by the electrical resistance and collision of electrons with the lattice structure of the materials [7]. On the contrary, dielectric loss is prominent in materials with polar properties, such as silicon carbide (SiC). This type of heating mechanism occurs when the dipoles encounter friction during their reorientation in accordance with the alternating microwave field [8]. Besides, the process of reorientation leads to a change in dipole moment, which in turn results in dipolar polarization [9]. Dielectric heating is caused by a combination of frictional loss and dipolar polarization. In contrast, magnetic loss is generally pertained to magnetic materials such as ferrous material due to several factors, including hysteresis loss that is resulted from the expansion and contraction domains [8] and eddy current loss that is resulted from the change of the induced magnetic field [7].
Owing to the different heating mechanisms associated with different classes of microwave susceptors, it is believed that the choice of microwave susceptor has significantly influenced the mechanical and microstructure properties of the microwave-welded joint. Moreover, nano-sized susceptors are expected to have better microwave-absorbing ability compared to micro-sized susceptors due to their high specific surface area and their additional interfacial polarization [10]. To date, no study has compared the microstructure and mechanical properties of the microwave-welded joint formed by two different susceptors. Therefore, in this study, multiwalled carbon nanotubes (MWCNTs) and silicon carbide nanowhiskers (SiC NWs) were selected as the two different classes of microwave susceptors, in which MWCNTs is the susceptor that works based on conduction loss while SiC NWs works based on dielectric loss. These two susceptors were employed in microwave welding of polypropylene (PP), and their respective effects on the mechanical properties and microstructure of the resulted welded joint were studied and reported in this study.

2. MATERIALS AND METHODS

2.1. Materials

Polypropylene (PP) pellets (Lotte Chemical Titan (M) Sdn Bhd, Malaysia) were used to fabricate thermoplastic substrate. Multiwalled carbon nanotube powder (MWCNTs, purity >93%, diameter 10-40 nm, Fibermax Composites, Greece) and silicon carbide nanowhiskers (SiC NWs, purity >99%, diameter 100-250 nm, Nanostructured & Amorphous Materials Inc., USA) were utilized as susceptors in this study. Acetone (HmbG Chemical, Germany) was used as a dispersing agent. All materials and chemicals were used without further modification or purification.

2.2. Fabrication of PP Substrate

To fabricate PP substrate, compression molding was employed using a hydraulic thermal press machine (GoTech, Taiwan). First, the thermal press machine was heated to 180 °C, roughly 20 °C above the melting point of PP. Next, the PP pellets were fed into the mold cavity, which was sandwiched by two flat metal plates. Then, the mold with PP pellets was pre-heated at the heating compartment for 6 min, followed by isothermal compression for 3 mins. Following that, the mold was moved to the cooling compartment for another 3 mins. After the cooling process, the PP sheet was removed from the mold and cut into the dimension of the thermoplastic substrate according to ASTM D3163. Based on ASTM D3163, the PP substrate was fabricated into 25.4 mm x 101.6 mm x 1.6 mm. Besides, a targeted area of 25.4 mm x 12.7 mm was marked on the PP substrate.

2.3. Preparation of Susceptor Suspension

Susceptor suspension was prepared by dispersing the susceptor powder (MWCNTs and SiC NWs) into acetone. Prior to dispersing the MWCNTs into acetone, a PMMA stock solution was prepared by dissolving 0.75 wt% PMMA pellets in 100 mL acetone. The purpose of preparing this PMMA solution was to assist the adhesion of MWCNTs on the PP substrates. After that, 8.0 wt% of MWCNTs powder was added into 10 mL of PMMA solution and subjected to magnetic stirring for 15 min. The homogenous MWCNTs suspension was then put into ultrasonic bath for 60 min, to avoid agglomeration. To prepare SiC NWs suspension, MWCNTs was replaced by SiC NWs and all the steps were repeated.

2.4. Suspension Deposition on PP Substrates

Before microwave welding, the drop casting method was utilized to deposit the susceptor suspension onto the marked targeted area on PP substrate. In this study, 0.2 ml of sonicated susceptor suspension was dropped on the targeted area using a syringe, followed by drying the substrate on a 70 °C hot plate for 15 min. After that, a bare PP substrate was put on top of the susceptor deposited PP to form a PP/susceptor/PP paired sample.

2.5. Microwave Welding

In this research, an 800 W household microwave oven (2.45 GHz, Sharp, R213CST) was utilized. Throughout the experiment, the microwave power was set at high mode, which is also the maximum output power of the microwave oven. For microwave welding, clamping pressure is necessary to provide close contact between the PP substrate and force the occurrence of chain entanglement; hence, two microwave transparent glass slides were utilized to apply pressure on the targeted area.

Additionally, for ensuring the stability of the paired sample during microwave welding process, two PP tabs were positioned at the upper and lower side of the paired sample, as shown in Figure 1. The clamped sample was then sent into the microwave oven and subjected to microwave irradiation. The heating duration studied in this study is ranging from 5 s to 20 s, with an interval of 5 s.

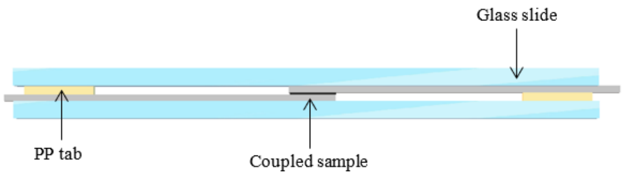

Figure 1 Setup of clamped paired sample in that are ready for microwave welding

2.6. Microwave Absorption Performance

To determine the ability to serve as microwave susceptor, the dielectric properties of both MWCNTs and SiC NWs were measured using vector network analyzer (VNA, Agilent Technologies E5071C E Series, Malaysia) supported with 8570E software. The studied frequency is limited to 2-18 GHz. Before the testing, the MWCNTs and SiC NWs were pressed into 3 mm pellets via powder metallurgy method. Besides, three types of calibrations, including air, short and water were carried out to avoid systematic error. After calibrations, a VNA connected probe was placed in contact with the susceptor pellets. Five susceptor pellets were tested and the dielectric properties were calculated based on their average value. In addition to dielectric properties, the reflection loss, R_L was computed using transmission line theory as shown in Equation (2 and (**2** [11].

$$R_L = 20 \log |(Z_{in} - Z_0)/(Z_{in} + Z_0)| \quad (1)$$

$$Z_{in} = Z_0(\mu_r/\varepsilon_r)^{1/2}\tanh[j(2\pi fd/c)(\mu_r\varepsilon_r)^{1/2}] \quad (2)$$

where Z_{in} stands for the input impedance, Z_0 stands for the vacuum impedance, μ_r stands for the relative permeability of susceptor, ε_r stands for the relative permittivity of the susceptor, f stands for the frequency of electromagnetic wave, d stands for the thickness of susceptor and c stands for the speed of light.

2.7. Tensile Test

The joint strength of the microwave-welded joint was determined using a universal testing machine (UTM, Instron 5569, Instron Malaysia) according to ASTM D3163 with a 1.27 mm·min^{-1} testing rate. Similarly, two PP tabs were attached at the upper and lower sides of the welded sample to ensure the tensile stress acted on the welded joint uniformly and avoid bending moments during the tensile test being conducted. Three samples were tested for each category, and the average value is calculated and recorded.

2.8. Scanning Electron Microscopy (SEM)

The tensile fracture surface was observed using scanning electron microscope (SEM, JEOL JSM-6460LA, JEOL Ltd, Japan). Because the welded sample is non-conductive, a fine layer of platinum was deposited on the fracture surface before SEM was carried out to avoid charging issues.

3. RESULTS AND DISCUSSION

To serve as an outstanding susceptor, the dielectric properties and its microwave absorption performance are important. Figure 2 shows the dielectric permittivity and microwave absorption performance of MWCNTs and SiC NWs. In general, the real part of permittivity, termed the dielectric constant, reflects a material's capacity for storing electric energy. Conversely, the imaginary part of permittivity, also termed the loss factor, reflects a material's capacity for dissipating electric energy. Figure 2(a) and (b) show that SiC NWs have a larger dielectric constant and loss factor than MWCNTs. Furthermore, SiC NWs exhibit the lowest reflection loss of -10.41 dB at 5.84 GHz, whereas MWCNTs only reached -7.75 dB at 8.24 GHz. This finding suggested that SiC NWs have a stronger microwave absorption performance in low microwave frequencies when compared to MWCNTs, and both susceptors convert more than 85% of incoming microwave energy into heat. From these results, it can be concluded that both MWCNTs and SiC NWs are excellent susceptors and suited for microwave welding.

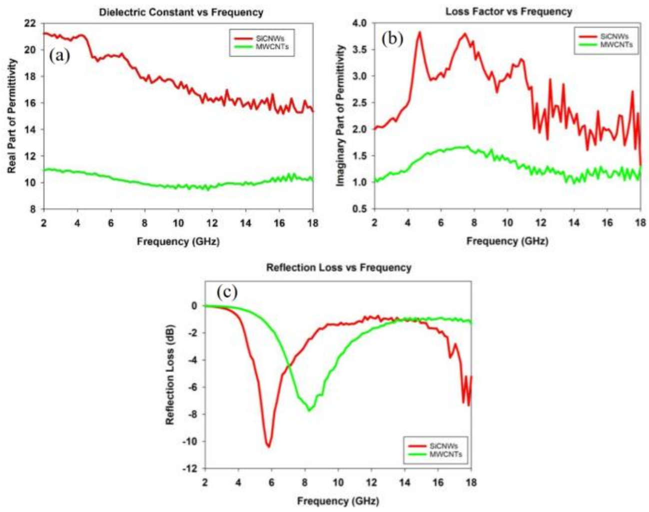

Figure 2 Comparison of (a) dielectric constant, (b) loss factor and (c) reflection loss between MWCNTs and SiC NWs

Upon confirm their microwave absorption performance, MWCNTs and SiC NWs were employed as susceptors for microwave welding of PP. The joint strength of the resulted welded joint was determined using single lap shear test according to ASTM D3163 and the targeted area was kept constant at 322.58 mm^2. Figure 4 shows the shear strength of the welded joint formed using MWCNTs and SiC NWs as susceptor. The shear strength of welded joint formed using MWCNTs increased from 1.11 MPa to a maximum of 2.26 MPa as the heating duration was extended from 5 s to 10 s. It is believed that this increase in joint strength is due to the sufficient energy absorption and heat released from the MWCNTs when the heating duration was extended to 10 s, which melted the PP substrates more effectively and the heat was transfer to a greater depth of the adjacent PP substrate, resulted in a stronger welded joint. As compared to study reported by previous study, a maximum joint strength of 5.85 MPa was achieved when microwave welding was performed using high-density polyethylene (HDPE) substrate[12]. This difference in joint strength is due to the different joint design and different thermoplastic substrate was used. They use HDPE as the thermoplastic substrate and PP was utilized in this work, the different chemical structure and density of the thermoplastic substrate might result in the different joint strengths. Moreover, in their experimental design, the overlap area was fixed at 100 mm^2, while in this study, the overlap area was fixed at 322.58 mm^2, which is more than tripled. Equation (3) shows the general formula of shear strength, σ_s.

$$\sigma_s = L/A \tag{3}$$

where L is the load taken to break the sample, in N and A is the overlap area, in mm^2. Based on this formula, due to the larger overlap area designed in this study, the joint strength is lower even a similar load taken to break the sample.

However, the joint strength of the welded joint started to decline as the heating duration was prolonged to 15 s and 20 s. This decrease in joint strength is likely due to the excessive microwave energy that was absorbed by the MWCNTs during extended heating duration, leading to excessive heat dissipation to the neighboring PP substrate and damaging the welded sample.On the other side, the welded joint formed using SiC NWs achieved its maximum joint strength of 2.25 MPa at a heating duration of 15 s. Even though the microwave absorption performance of SiC NWs is higher than MWCNTs in low frequency, due to the significantly lower thermal conductivity of SiC NWs (490 W/m.K.) than MWCNTs (3000 W/m.K.), the heats released from the susceptor cannot be transferred to the adjacent PP substrate effectively, resulting in the low heating rate in SiC NWs [13, 14]. For a fixed heating duration, such as 10 s, the heat dissipated by SiC NWs may not be sufficient to completely melt the targeted area, resulting in only partially welded joints with low joint strength [15]. Yet, for the heating duration of 10 s, the high heating rate of MWCNTs allows them to absorb energy and melt the entire targeted area of PP substrates. Therefore, an entirely welded joint with comparable joint strength was achieved in a shorter duration.

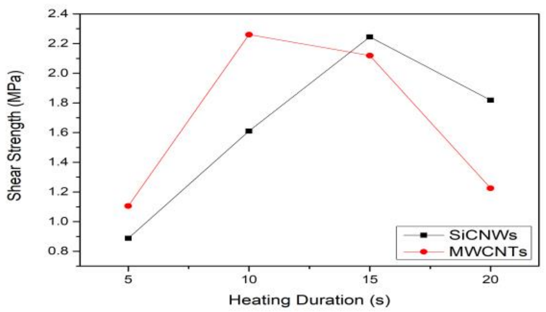

Figure 3 Shear strength of welded joint formed by different susceptor as a function of heating duration

Due to the higher heating rate and thermal conductivity of MWCNTs, a small burn mark is noticed at the paired substrate. The formation of burn marks might be due to the occurrence of fire flashes and sparks during prolonged exposure of MWCNTs to microwave heating. Consequently, the paired PP substrates are damaged and result in a slightly lower shear strength compared to a 10 s welded joint. For the welded joint created using SiC NWs, there are no fire flashes and sparks during microwave welding, and therefore, the paired PP substrates did not experience any damage. After testing, both welded joints failed at the edge of the welded joint. This type of failure can be explained as the welded joint is stronger than the PP substrates due to the creation of nanocomposite at the welded joint [16]. During microwave welding, the susceptor (both MWCNTs and SiC NWs) absorbed microwave energy and created a molten PP layer once the heat released attained the softening point of PP. Due to the presence of clamping pressure, the susceptors were forced to embed into the created molten layer, forming a MWCNTs-filled PP nanocomposite and SiC NWs-filled PP nanocomposite at the welded joint upon solidification. This development of nanocomposite is believed to contribute to the high joint strength and failure at the edge of welded joints instead of welded joints.

For microwave heating duration of 20 s, both welded samples experienced severe deterioration. The welded joint formed using MWCNTs exhibits a large burn mark at the paired PP substrate, and the PP substrate is slightly deformed and thinned. When a tensile test was performed for this welded joint, the welded joint fractured at the center instead of the edge. On the other hand, a void was noticed at the welded joint formed using SiC NWs due to overheating. The crack initiates from the void (which is also the weakening point) and breaks at the center of the welded joint. As discussed in Figure 2, the welded joint experienced severe deformation due to the excessive heat released from MWCNTs and SiC NWs, resulting in extremely low shear strength.

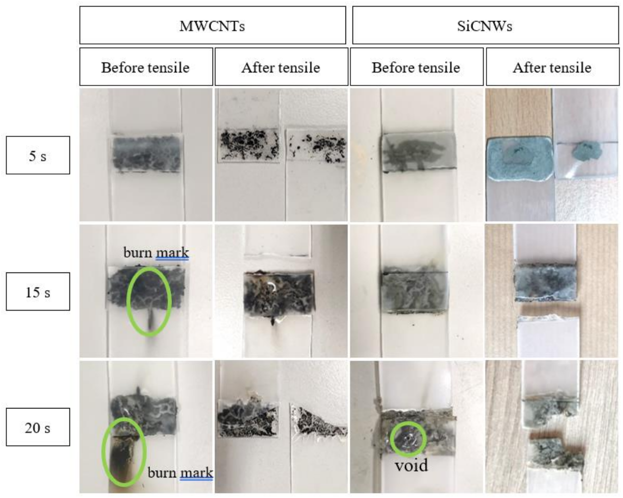

Figure 4 Snapshot of the welded sample before and after testing

Figure 5(a) and (b) shows the cross-sectional fracture surface of 5 s welded joint formed using MWCNTs and SiC NWs as susceptors at magnification 1000x, respectively. A similar observation was obtained for both welded joints, where a mixture of smooth and rough fracture surfaces was noticed. The smooth surface is believed to be the unwelded area, while the rough surface is the welded area. Furthermore, it is also observed that the MWCNTs tend to agglomerate at the joining interface, while SiC NWs are sparsely distributed on the fracture surface. This might be due to the smaller particle size of MWCNTs compared to SiC NWs, as mentioned in Section 2.1. Smaller particles may exhibit higher surface energy and cohesiveness, making MWCNTs more prone to agglomeration and non-uniform distribution compared to SiC NWs [17, 18]. This agglomeration is also believed to be the key reason for the lower joint strength of welded joint formed using MWCNTs than welded joint formed using SiC NWs at heating duration of 20 s. The cross-section fracture surface of welded joint formed at 15 s using MWCNTs and SiC NWs are presented in Figure 5(c) and (d) respectively. Both figures show rough and protruded susceptor from the PP surface. Additionally, there are no smooth surface and free-standing susceptor at the fracture surface, indicating that 15 s is long enough to fully melt the PP matrix and allow the complete embedment of MWCNTs and SiC NWs into the molten PP, resulting in MWCNTs-filled PP nanocomposite and SiC NWs-filled PP nanocomposite at the welded joint. This observation is in good consistency with Figure 4, which stated that the 15 s welded joint is stronger than the neat PP substrates due to the formation of nanocomposite at the welded joint.

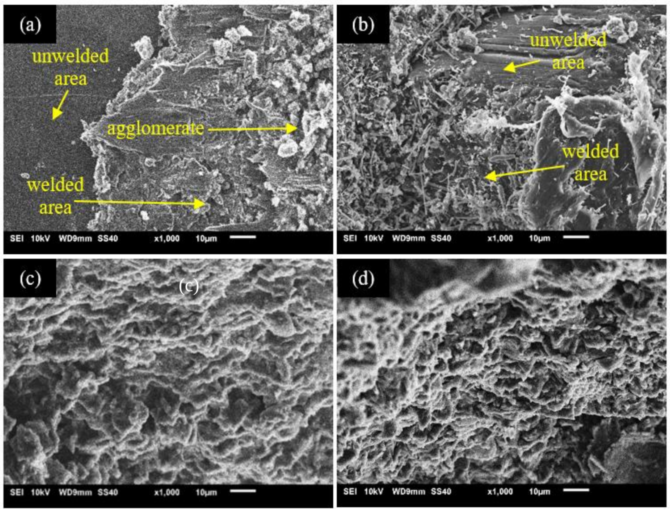

Figure 5 SEM images of (a, b) 5 s and (c, d) 15 s welded joint formed by MWCNTs and SiC NWs at 1000x magnification.

4. CONCLUSION

Based on the dielectric properties and microwave absorption performance, both MWCNTs and SiC NWs can be employed as microwave susceptors for microwave welding due to their high dielectric constant, loss factor, as well as reflection loss. Because of their different heating mechanisms, they exhibit different heating rates, which result in different properties at the microwave-welded joint. Generally, the joint strength of the welded samples increases as the heating duration increases. The increment in joint strength is attributed to the higher amount of energy absorbed by the susceptor, which subsequently melts a large depth of the PP substrates, allowing the embedment of the susceptor and the formation of MWCNT-filled PP and SiC NWs-filled PP nanocomposite at the joining interface. However, the welded sample experiences severe damage when the heating duration is prolonged to 20 s, which results in low joint strength. Besides, for welded joints formed using MWCNTs, a maximum joint strength of 2.26 MPa was obtained at 10 s heating duration. Meanwhile, welded joints formed using SiC NWs achieved a comparable joint strength at a 15-s heating duration. This indicates that the microwave heating rate of MWCNTs is higher than that of SiC NWs, resulting in complete joint strength in a shorter duration. This study's findings provide valuable insight into the potential advantages and limitations of using different types of microwave susceptors for microwave welding applications.

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