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Microwave Welding of Thermoplastic using Silicon Carbide Nanowhiskers as Susceptor: Effect of Heating Duration

Phey Yee Foong[a], Chun Hong Voon[a,*], Bee Ying Lim[b], Pei Leng Teh[b], Cheow Keat Yeoh[b], Mohd Afendi Rojan[c], Nor Azizah Parmin[a], Subash C. B. Gopinath[a,b], Foo Wah Low[d], Muhammad Kashif[e], Nor Azura Abdul Rahman[b], Sung Ting Sam[b] and Veeradasan Perumal[f,g]

[a]*Institute of Nano Electronic Engineering, Universiti Malaysia Perlis, Seriab, 01000, Kangar, Perlis, Malaysia.*
[b]*Faculty of Chemical Engineering & Technology, Universiti Malaysia Perlis, 02600, Arau, Perlis, Malaysia.*
[c]*Faculty of Mechanical Engineering & Technology, Universiti Malaysia Perlis, Pauh, 02600, Arau, Perlis, Malaysia.*
[d]*Department of Electrical & Electronic Engineering, Lee Kong Chian Faculty of Engineering & Science, Univerisiti Tunku Abdul Rahman, Bandar Sungai Long 43000 Kajang, Selangor, Malaysia.*
[e]*School of Electrical and Information Engineering, Tianjin University, 92 Weijin Road, Nankai District, Tianjin 300072, China.*
[f]*Centre of innovative Nanostructures and Nanodevices (COINN), Universiti Teknologi PETRONAS, 32610, Seri Iskandar, Perak Darul Ridzuan, Malaysia.*
[g]*Department of Mechanical Engineering, Univerisit Teknologi PETRONAS, 32610, Seri Iskandar, Perak Darul Ridzuan, Malaysia.*
∗ *Corresponding author. Tel.: +6017-9427832; e-mail: chvoon@unimap.edu.my*

ABSTRACT

Microwave welding is becoming more popular than conventional joining methods due to its advantages such as rapid and localised heating as well as applicable to components with complicated geometry. Previously reported susceptor, such as carbonaceous materials and conductive polymers, are toxic and the welding process involving these susceptors is time-consuming. Because of its exceptional microwave absorption and biocompatibility, silicon carbide nanowhiskers (SiCNWs) was employed as the microwave susceptor for microwave welding. Microwave welding in this study comprises of only three simple steps: SiCNWs suspension preparation, SiCNWs application and microwave heating. The weld strength of welded joint was then characterised using tensile test and energy dispersive x-ray spectroscopy equipped scanning electron microscopy (EDS-SEM) to study its mechanical properties and cross-section microstructure. The influence of microwave irradiation time was studied in this study, and it is found that the weld strength rose with the extension of microwave irradiation time, until a maximum weld strength of 1.61 MPa was achieved by 17 s welded joint. The development of SiCNWs reinforced PP nanocomposite welded joint layer is responsible for the enhanced weld strength. Prolonged heating duration may also result in flaws such as void formation at the welded joint, which subsequently lowered the weld strength to 0.60 MPa when the heating duration was extended to 20 s. In sum, a strengthen welded joint can be formed with rapid microwave heating under the proper control of heating duration.

Keywords: *Microwave Heating, Nanocomposite, Nanowhisker, Silicon Carbide, Welding*

1. INTRODUCTION

Microwave heating is a well-developed technique that had been utilised in various processing including food processing [1, 2], ceramic sintering [3] and material synthesis [4, 5]. Microwave welding is considered as a relatively new technology among microwave processing. Typically, microwave is an electromagnetic wave having wavelength between 1 mm and 1 m [6]. Besides, it is known that welding is a joining process that involves heat transfer and melting of joining interface. Therefore, the electromagnetic joining technique that uses microwave radiation to melt the joining interface is known as microwave welding. In fact, microwave welding has many advantages that cannot be offered by conventional welding methods such as localised and selective heating, high heating rates and applicable on complex geometry parts [7]. Furthermore, direct absorption of microwave energy by the materials induces volumetric heating within the material which also reduces the temperature gradient, heat affected zone, processing time and power consumption [8]. For these reasons, microwave welding attracts research attention and number of reported studies is increasing significantly.

In the past, microwave welding of ceramics and metals has been relatively mature and are widely reported [9-13]. Yet, microwave welding of thermoplastic is rarely reported due to the large penetration depth and microwave transparent properties of most thermoplastic [14]. In other words, microwave transmits through thermoplastic without any interaction. To overcome this issue, microwave susceptor, a material that is microwave susceptible and can be heated up by microwave irradiation is needed for microwave welding of thermoplastic. The microwave absorption performance of carbonaceous materials such as carbon nanotube (CNT) and graphite were widely studied. It is reported that the existence of sp^2 π electrons in carbon structure are responsible for the excellent microwave absorption as well as their heating mechanism [15]. For this reason, carbonaceous materials are suitable to serve as microwave susceptor for microwave welding. As an example, Sweeney et al. demonstrated welding of polylactic acid (PLA) 3D-printed thermoplastic using multiwalled carbon nanotube (MWCNT)/PLA composite coating as susceptor [16]. The joint strength of the microwave welded joint was then characterised using tear test and they stated that the joint strength of microwave welded joint was improved by 275 % when comparing to PLA sample fabricated by 3D printing. However, carbonaceous material is known for their toxicity and have side effect such as causing lung inflammation in human health [17].

Aside from carbonaceous materials, conductive polymers such as acrylonitrile butadiene-styrene (ABS), polypyrrole (PPy) and polyaniline (PANI) also show their potential as susceptor for microwave welding. On the basis of reported study, conductive polymers can absorb microwave after their structures were altered using acidic dopants with electron withdrawing group such as amine group, carboxyl group and hydroxyl group [18]. Wu et al. investigated microwave welding of high-density polyethylene (HDPE) using hydrochloric acid (HCl) doped PANI as susceptor [7]. Their findings revealed that the strength of welded joint is comparable to the neat HDPE when 50% PANI-1 mm gasket and 90 s were used as susceptor and heating duration, respectively. Besides, the microwave absorbing ability can also be controlled by varying the doping level, leading to the controllable heating rates of susceptor. Although this study shows that rapid heating and improved strength can be achieved by conductive polymer, but additional steps are needed for the preparation of conductive polymers before they can be used as susceptor, which is time consuming and incurs a higher cost.

As compared to conductive polymers, silicon carbide (SiC) stands out from them due to its intrinsically dielectric properties and dipolar polarisation when it is exposed to microwave irradiation. Besides, SiC also offers higher level of safety and biocompatibility than carbonaceous materials. For these reasons, SiC is a better candidate of susceptor for microwave welding. Recently, nanomaterials had become tremendously popular for their enhanced performance owing to their high aspect ratio. Liu et al., for example examined the microwave absorbing properties of two different sized SiC, namely ultra-fine SiC (diameter <1000 nm) and nanometre SiC (diameter < 100 nm) [19]. They show that both dielectric constant and loss factor of nanometre SiC are greater than ultra-fine SiC. Besides, nanometre SiC achieved a minimum reflection loss of -5.53 dB at 9.72 GHz, due to its high aspect ratio which is prone to multi-scattering and interfacial polarisation, leading to its better microwave absorbing capacity. In another study, Poyraz et al. also claimed that susceptor in nanosized can increase the miscibility through their active rough surface area during the formation welded joint [18].

With the combined benefits of microwave welding, nanomaterial and SiC, microwave welding of thermoplastic using SiC nanomaterial was investigated in this study. With the use of SiC nanomaterial alone as susceptor, no additional steps are required and thus, it is time saving. In this study, polypropylene (PP) and SiC nanowhiskers (SiCNWs) were used as thermoplastic substrates and susceptor respectively. Besides, Wu et al. claimed that the thickness of melted layer during microwave welding has great impact on the joint strength, wherein thicker melted layer results in higher joint strength. It is known that microwave irradiation time can significantly influence the heating rate and the amount of heat dissipated by susceptor and subsequently, the thickness of liquefied layer formed, and the nanocomposite welded joint layer. As a result, the strength of the welded joint is affected. Hence, the effect of microwave irradiation time on the weld strength and cross-sectional morphology of welded joint was investigated and reported.

2. MATERIAL AND METHODS

2.1. Materials

PP pellets (Lotte Chemical Titan (M) Sdn. Bhd., Malaysia), SiCNWs (Nanostructured & Amorphous Materials Inc.) and acetone (HmbG Chemical, Germany) were used without any further modification and purification.

2.2. Methodology

In this work, the PP substrate used was prepared in accordance with ASTM D3163. To fabricate PP substrates, PP pellets were first hot pressed into a 1.6 mm PP plate using a hydraulic heat press machine (GoTech, Taiwan), then sectioned the PP sheets using a customised cutter with the aid of a hand press. Then, a designated area was drawn on the PP substrate.

Figure 1 depicts the configuration and designated area of the PP substrate.

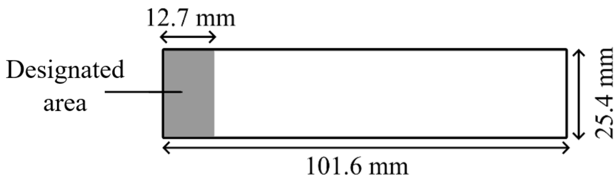

Figure 1. Configuration and designated area of PP substrate.

For the preparation of SiCNWs suspension 7 wt% SiCNWs powder was mixed with acetone using magnetic stirrer for 15 mins, followed by 1 h in an ultrasonic mixing bath (Delta DC200H, Qatar). After that, 0.2 ml of SiCNWs suspension was applied on the designated area. This was then allowed to dry on a 70 °C hot plate for 15 mins. The SiCNWs coated PP substrate was then covered with another plain PP substrate once the SiCNWs suspension was fully dried. The experimental configuration of the coupled sample that is ready for microwave is shown in Figure 2. A pair of microwave transparent glass slides were utilised to enable close contact and to apply pressure to the coupled sample. Besides, two PP tabs with 1.6 mm thickness were used to balance the coupled sample during microwave welding process. In this study, an 800 W household microwave oven (Sharp R213CST, 2.45 GHz, Malaysia) was employed to perform microwave welding and the effect of microwave irradiation time was studied and reported. The microwave irradiation time was varied from 5 s to 20 s while the amount of SiCNWs, microwave power and clamping pressure were remained constant.

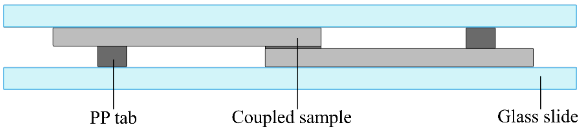

Figure 2. Schematic diagram of coupled sample configuration prepared for microwave welding in microwave oven.

2.3. Sample Characterisation

Scanning electron microscopy (SEM, Hitachi TM3000, Japan) and energy dispersive x-ray spectroscopy (EDS, Bruker Quantax, Germany) were used to characterise the welded joint's cross-section morphology. To avoid the breakage or detachment of welded sample during the cutting process, it was first molded in epoxy resin and allowed to cure prior to SEM examination. After that, the molded sample was cut and various grit of sandpapers (up to grit 1200) were used to grind the sample until a smooth surface was obtained. Prior to SEM analysis, a thin platinum coating layer was coated onto the ground sample using a sputter coater (JEOL JFC-1600 Auto Fine Coater, Japan). Figure 3 shows the epoxy moulded welded sample ready for SEM and EDS.

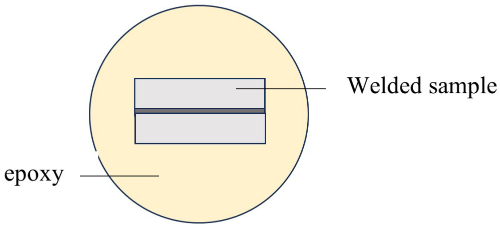

Figure 3. Sample preparation for SEM and EDS.

Mechanical properties such as the weld strength was examined using a universal testing machine (UTM, Instron 5569, Malaysia) according to ASTM D3163. Before testing, two PP tabs was affixed at the gripping section of welded sample to ensure sample alignment as well as evenness of applied force on the welded joint. The welded sample was then subjected to tensile test with a crosshead rate of 1.27 mm/min until failure. Figure 4 depicts the configuration of welded sample at the UTM machine.

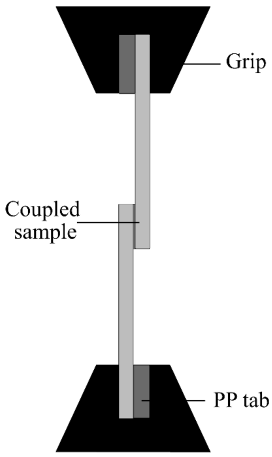

Figure 4. Setup of welded sample in UTM.

3. RESULTS AND DISCUSSION

Figure 5 compares the weld strength of welded joint versus microwave irradiation time. Based on Figure 5(a), the weld strength of welded joint rose as the microwave irradiation time extended from 5 s to 15 s, reaching a maximum weld strength of 1.12 MPa at 15 s welded joint. However, there is a reduction in weld strength as the microwave irradiation time further rose to 20 s. To be more concise, the welded joint was further examined in the range of 15 s to 20 s with a step of 1 s of heating duration, as illustrated in Figure 5(b). Apparently, it appears that when the microwave irradiation time rose to 17 s, the welded joint reached its peak weld strength of 1.61 MPa. After that, the weld strength drops to 0.98 MPa and 0.60 MPa as the microwave irradiation time further extended to 19 s and 20 s respectively.

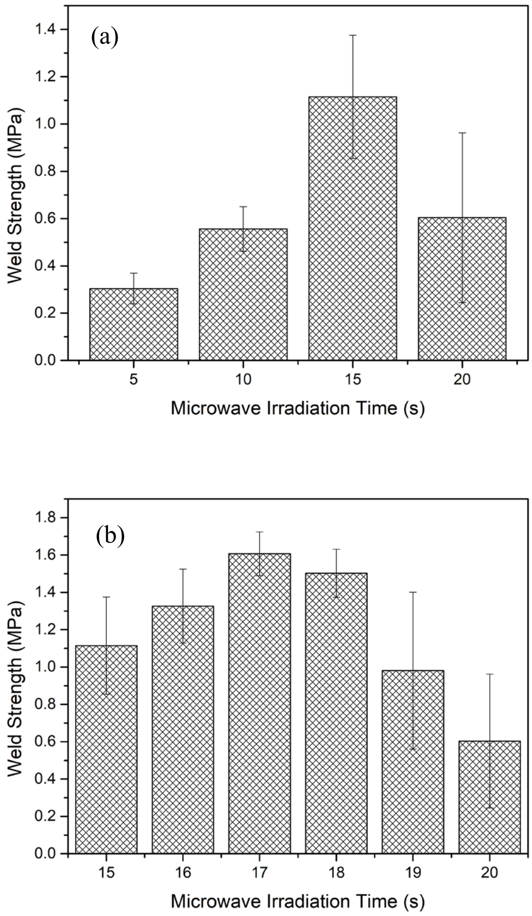

Figure 5. Weld strength of welded joint exposed to microwave irradiation time ranging from (a) 5s to 20 s and (b) 15 s to 20 s.

In fact, the quantity of microwave energy absorbed and heat emitted by SiCNWs is significantly affected by the microwave irradiation time. For instance, when the coupled sample was exposed to short microwave irradiation time such as 5 s, the energy absorbed and the heat emitted by SiCNWs is only enough to melt a small portion of PP's designated area and hence, resulted in an incomplete and weak welded joint. SiCNWs has more time to absorb the microwave energy as the microwave irradiation time is lengthened, leading to the increase in heat emitted by SiCNWs. Subsequently, the welded region grew larger until a fully welded joint was developed at 17 s, and thereby, improved weld strength was achieved for 17 s welded sample. Yet, prolonged microwave irradiation time over 17 s may cause formation of defects such as flash or voids on the PP substrates due to the excessive heats released by SiCNWs. As a result, a loss in weld strength was noticed for these welded joints. This finding is in line with several studies in which they showed that the weld strength of the welded joint increased with the increasing microwave irradiation time [20-23]. As an example, Wang et al. conducted polymer welding of polycarbonate plate using MWCNT powder as susceptor by microwave irradiation [24]. They examined the bonding strength of the welded joint using peeling test. Their result revealed that the peeling strength of 5 s welded joint was 10 N, and the strength increased significantly to 35 N when the heating time increased to 10 s. They also claimed that this enhancement of strength is resulted from the complex intercalation and friction of PC chain with the MWCNT.

Figure 6 depicts the photograph of welded sample after tensile test was conducted. Detachment was observed for the welded sample that was microwave irradiated for 5 s, as shown in Figure 6(a). This type of failure, known as adhesive failure, implies that the welded joint is insufficiently strong to keep both PP substrates in place when tensile force is applied, thus, results in low weld strength. Figure 6(b) depicts the welded sample after microwave irradiated for 17 s and it is observed that the designated area is fully welded. Besides, when tensile test is conducted, the welded sample fails at the border of welded joint. This type of failure, known as cohesive failure, denotes that the welded joint has a higher strength than the PP substrates. This observation is corroborated with the work that was demonstrated by Sun et al. in which they employed nanographite powder as susceptor for joining of PP by microwave heating [23]. They revealed that the formation of nanographite reinforced PP nanocomposite is the main contributor of the strengthened welded joint. Figure 6(c) illustrates the 20 s welded sample that has undergone tensile test. After being heated by microwave for a longer time of 20s, defects such as void was created at the welded joint and hence, the crack initiated from the voids and broke at the welded joint.

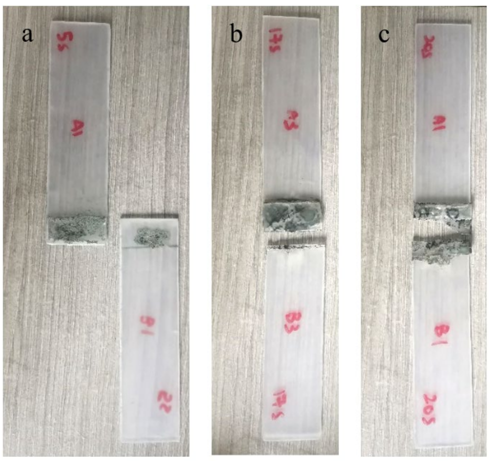

Figure 6. Condition of (a) 5 s, (b) 17 s and (c) 20 s welded joint after tensile test.

Figure 7 compares the SEM-EDS images of welded joints that were microwave irradiated for various microwave irradiation time. For 5 s microwave irradiated welded joint, the mean thickness of welded joint layer was measured to be 121.41 μm, as shown in Figure 7(a). Besides, it can be observed that the SiCNWs were not evenly distributed at the welded joint layer and the SiCNWs were only filled in the outer boundary of welded joint layer. Figure 8(a) further demonstrates that most of the SiCNWs remained concentrated at the middle of welded joint layer, which is the joining interface of PP substrate prior subjected to microwave heating, rather than being filled into the welded joint. This incomplete welded joint is attributed to the short microwave irradiation duration such as 5 s. The limited absorbed microwave energy is only sufficient to melt a thin layer of PP matrix and most of the SiCNWs were not able to incorporate into the thin melted layer, resulting in a low weld strength for 5 s welded joint.

In contrast, welded joint exposed to microwave irradiation time of 17 s in Figure 7(c) shows a thicker welded joint layer with a mean thickness of 147.69 μm. It is thought that 17 s is sufficient for SiCNWs to absorb adequate microwave radiation and heated up quickly, bringing the surrounding PP matrix into liquid state. Due to the higher amount of emitted heat, a melted layer with increasing thickness was formed and all the SiCNWs were filled into the melted layer, forming a SiCNWs reinforced PP nanocomposite welded joint layer with increasing thickness. This statement can be supported by Wu et al. in which they studied microwave welding of HDPE using PANI/HDPE composite gasket as susceptor. According to their findings, extending the heating time can result in thicker molten layer and development of composite welded joint, which consequently increased the welded joint's strength [7]. Besides, Figure 8(b) reveals that all SiCNWs were filled into the PP matrix without forming any gaps. The creation of SiCNWs/PP nanocomposite welded joint layer and the inclusion of SiCNWs throughout the welded joint layer are further confirmed in Figure 7(d). This SiCNWs/PP nanocomposite welded joint layer is believed to be the primary contributor to the highest strength of 17 s welded joint. In previous study, when super aligned CNT film was used as susceptor in Xie et al.'s experiment for the joining of polyethylene (PE), they discovered similar results.[22]. They stated that the inclusion of CNT at the PE interface and the formation of PE/CNT/PE sandwiched structure improved the welded joint's bonding strength. Figure 7(e) and (f) depicted the SEM and EDS images of 20 s welded joint, respectively. Although the mean thickness of welded joint layer rose to 209.98 μm when the sample was microwave irradiated for 20 s, voids were observed in the welded joint layer. This can be explained by the excessive heat that was emitted by SiCNWs after subjecting to 20 s microwave irradiation, which causes overheating and deterioration of the welded joint. As a result, emission of volatile organic compounds resulted in the development of voids in the welded joint. This observation is well-supported by the 20 s welded joint's reduced weld strength and the fracture condition illustrated in Figure 8(c).

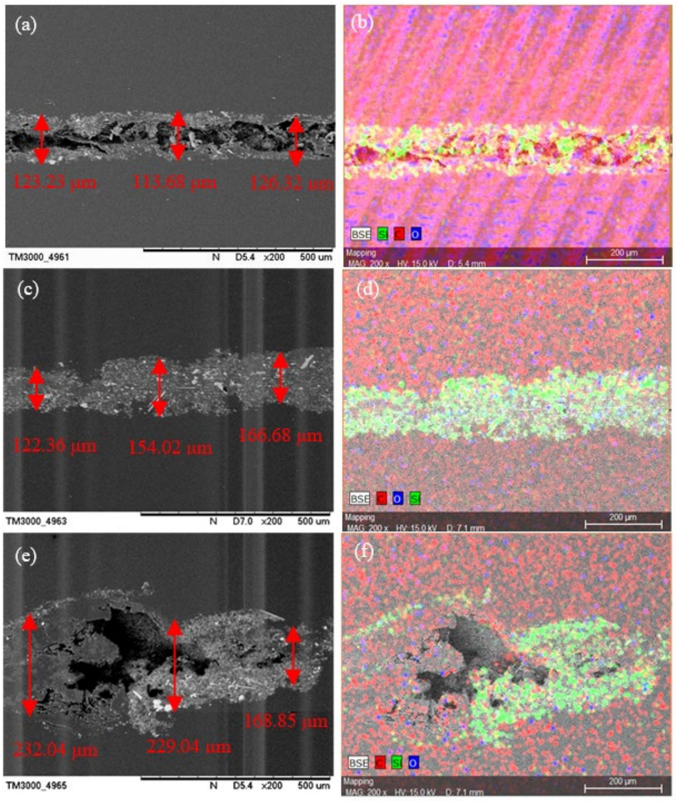

Figure 7. Cross-sectional morphology and EDS mapping of welded joint exposed to microwave irradiation time of (a, b) 5 s, (c, d) 17 s, and (e, f) 20 s.

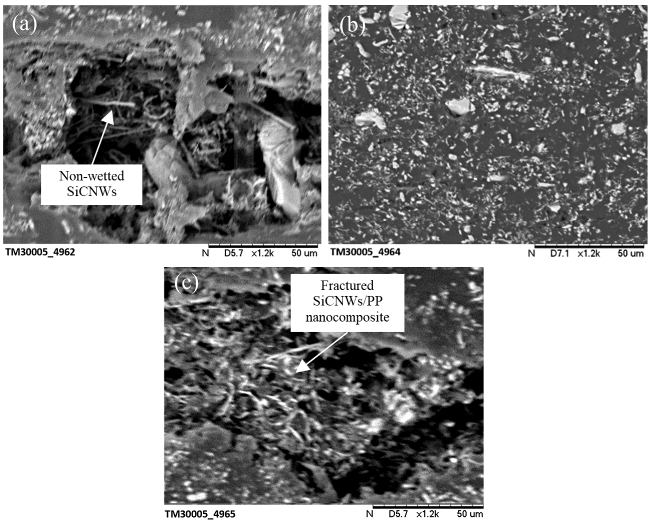

Figure 8. Cross-sectional morphology of (a) 5 s, (b) 17 s and (c) 20 s welded joint at magnification of 1200x.

4. CONCLUSION

To conclude, PP substrates were successfully welded by microwave irradiation using SiCNWs as susceptor. It was found that the increasing heating duration from 5 s to 17 s had increased the weld strength of the welded joint from 0.30 MPa to 1.61 MPa. With the increasing heating duration, SiCNWs were capable to absorb and released more heats to melt the PP substrate, thus resulted in melted layer with increasing thickness. A nanocomposite welded joint layer with incrementally thicker structure was formed under clamping pressure, which increased the weld strength of welded joint by thoroughly filling SiCNWs into the generated melted layer. However, the weld strength dropped to 0.60 MPa when the heating duration was extended to 20 s. The creation of voids at the welded joint by overheating is thought to be the root of the reduced weld strength. To summarise, microwave welding can produce a welded joint with enhanced strength when the heating duration is properly controlled.

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