

## Submerged Friction Stir Weld of Polyethylene Sheets

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**ABSTRACT:** The objective of this research work is to join polyethylene sheets using submerged friction stir welding by varying rotation speeds and traverse speeds. The effects of process parameters on macrostructure, microstructure, and mechanical property are investigated. The result indicates that the tensile strength increases at first and then decreases with the increase of rotation speed and traverse speed. The maximum tensile strength value of underwater welded joint is 12.3 MPa which is higher than normal weld joint. The microstructure of joint is investigated by using metallurgic microscope and laser scanning confocal microscope. The major microdefect of the interface is crack and air bubble. The result of differential scanning calorimetry which is used to measure the crystalline content of materials indirectly shows the crystalline content of parent material, heat affected zone, thermomechanically affected zone, and weld nugget approximate are 54.5%, 54.0% 51.1%, and 48.2%, respectively. The chief reasons of decrease in the tensile strength are formation of crack and air bubble and the decrease of crystalline content. The analysis of small angle X-ray scattering indicates that the long period of weld regions decreases comparing parent material. © 2014 Wiley Periodicals, Inc. *J. Appl. Polym. Sci.* **2014**, *131*, 41059.

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### INTRODUCTION

Connection process is the key step in manufacturing plastic parts, especially the structural components. The main joining methods for thermoplastics are mechanical bond, adhesion, and welding.<sup>1–4</sup> The advantages of the mechanical bond are convenient disassembly and recycling of connection parts, but the major disadvantage is the stress concentration in the holes.<sup>1</sup> Adhesion is the intermolecular interaction at polymer–polymer interfaces with low stress concentration.<sup>5,6</sup> It can bond the immiscible polymer, but the joint may degenerate due to the influence of heat and environment. Welding is an effective joint technique, which can eliminate these above disadvantages.<sup>7</sup> Meanwhile, it can save a large amount of materials and reduce the weight of engineering structures significantly compared with the mechanical bond. Simultaneously, the processing and assembly process can be simplified. The common weld methods for thermoplastics are hot gas weld,<sup>4</sup> vibration weld,<sup>8</sup> laser weld,<sup>9</sup> ultrasonic weld,<sup>10</sup> friction weld,<sup>11</sup> and so on.

Friction stir welding (FSW) was invented at The Welding Institute (TWI) in 1991 as a novel solid-state, hot-shear joining technique that had been proven to be applicable to the joining of various materials.<sup>12,13</sup> The initial research about FSW focused on aluminum alloys. With the development of the FSW, it had been

successfully applied to weld Mg, Cu, Ti, steel, and composites.<sup>12,14–19</sup> However, the focus of the FSW was transferred gradually from metallic materials to thermoplastics. Unlike metal FSW, the FSW of thermoplastics could not be considered as a solid-state process because the molten materials were involved in weld regions.<sup>20</sup> So far the researches on thermoplastics FSW were polyethylene, polypropylene, polycarbonate, nylon, and acrylonitrile butadiene styrene (ABS). Bozkurt optimized FSW process parameters by Taguchi's L9 orthogonal array to achieve maximum tensile strength in polyethylene sheets. Their results showed that the tool speed contributed 73.85% to the overall welding parameters.<sup>2</sup> Arici and Saeedy studied the effects of double passes on FSW of polyethylene and increased the strength of the joints in this way.<sup>21–23</sup> Pirizadeh used a newly designed tool to friction stir weld ABS sheets. The results showed that pin shape had the greatest effect on tensile strength of welded parts.<sup>24</sup> Bagheri studied the effects of process parameters on mechanical properties of friction stir welded ABS sheets. He reported that welding at a high level rotational speed and a lower level of travel speed increased the weld quality and weld tensile strength.<sup>25</sup> Kiss used a novel *K* factor (rotation speed, the feed rate, and the tool diameter) to investigate the effect of welding parameters on the heat-affected zone (HAZ) and the mechanical properties of friction stir welded poly(ethylene-terephthalate-glycol). The test showed that the

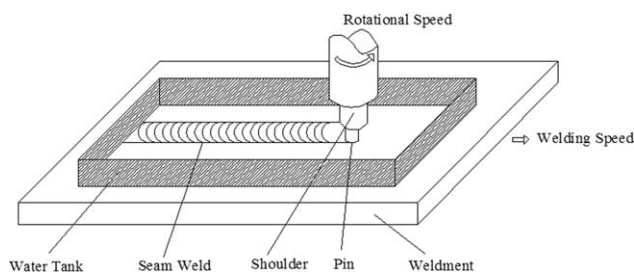


Figure 1. Schematic figure of submerged FSW.

joint quality was related to the width of the HAZ, and it depended on the welding parameters and the tool.<sup>26</sup> Panneerselvam investigated the effect of tool forces and joint defects during FSW of polypropylene plate. The results showed that the threaded pin profile tool produce lower force, and triangular, grooved and square pin profile produced defect free welds.<sup>27</sup> The potential use of FSW in thermoplastics is extremely large, mainly due to its higher assembly rates and lower assembly costs.<sup>1</sup>

The fundamental of thermoplastic welding is that the molten macromolecule mutually diffused and produced van der Waals' forces under the welding pressure and temperature, which pulled the two work-pieces together. So the heat and force are the critical factors for getting high-quality weld joints in the process of thermoplastic welding. Polymeric macromolecular chains are mutually restrictive and congealed before weld. When the heating temperature reaches glass transition point ( $T_g$ ) due to frictional heat by shoulder and work-piece in FSW, the macromolecular chains turn looser and minor macromolecular chains begin to move.<sup>28</sup> With the generation of frictional heat, the temperature of the interface gets over melting ranges. When the macromolecular chains get enough heat which overcome energy barrier, the chains become free, and they are allowed to diffuse into the opposite specimen at the weld interface. Finally, the whole chains in the weld regions deviate from their original position and the mass diffusions take place.<sup>7,28</sup> The properties of the joints are affected by molecular orientation, chain architecture, chemical dissimilarity, crystallization, and so on.<sup>29,30</sup>

Polyethylene is a semicrystalline material,<sup>22</sup> which has been successfully used to gas and water pipelines because of its excellent properties such as lower weight and corrosion resistance.<sup>31</sup> In the process of normal FSW for polyethylene, the rapid cooling and nonuniform temperature distribution of weld region led to the decrease of strength even welding failure.<sup>32</sup> To ameliorate the weld temperature distribution and achieve higher weld strength, the submerged FSW of polyethylene was conducted in this experiment. Figure 1 shows the process of submerged FSW. In FSW process, a rotating tool with a special shoulder and pin was slowly inserted into the weld joint until the shoulder con-

Table I. The Partial Physical Properties of the Polyethylene Sheet

Properties	Density (g/cm <sup>3</sup> )	Melting point (°C)	Glass transition temperature (°C)
Data	0.941-0.960	130-137	-120-125

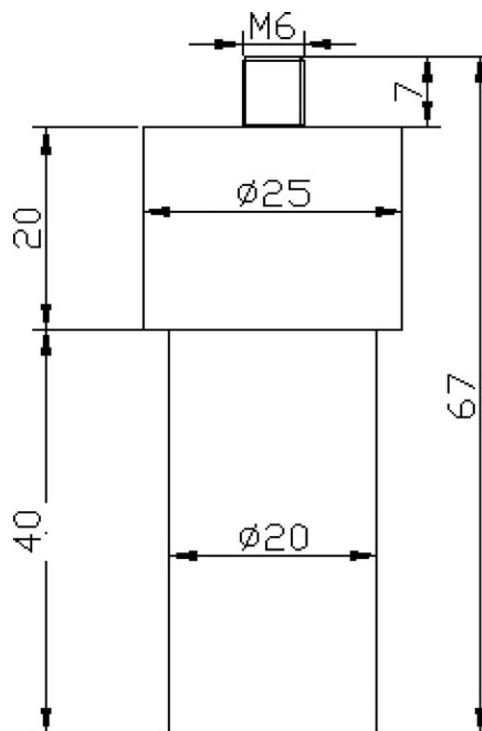


Figure 2. Schematic sketch of FSW tool.

tacted with the work-piece surface. The thermal energy which resulted from contact friction between the shoulder and the work-piece around the pin led to the formation of spiral plastic layer around the tool. The plastic layer moved from the front of the pin to the back, and the materials positioned over a tail end of the rotating tool cooled and formed welding seam, finally the two plates were joined.

The researches about submerged friction stir weld 7050 aluminum alloy showed that the mechanical properties of the underwater weld was higher than that in air.<sup>33</sup> So far, there are only few reports about thermoplastics submerged FSW. In this work, the experimental investigations on submerged FSW of polyethylene were conducted. The effects of the process parameters such as rotational rate and traverse speed on the weld strength, macrostructure, and microstructure of the weldment were

Table II. Weld Parameters and Levels

Experiment number	Rotation speed (r/min)	Traverse speed (mm/min)
1	1200	30
2	1800	
3	2400	
4	1200	45
5	1800	
6	2400	
7	1200	60
8	1800	
9	2400	

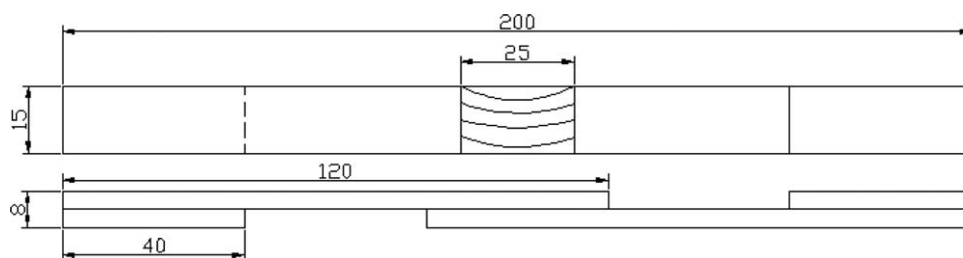


Figure 3. Configuration of tensile shear specimen (dimensions in mm).

investigated. As a comparative task, the FSW in air (hereafter called normal FSW) under the same parameters was performed.

## EXPERIMENTAL

The samples in this experiment include two 200 mm  $\times$  200 mm  $\times$  4 mm (length, width, depth) high density polyethylene (HDPE) sheets. The partial physical properties of this material are shown in Table I. The joints are produced on modified milling machines. The rotating tool used in this experiment has a threaded cylindrical pin. Figure 2 shows the size of the tool. The shoulder diameter of the tool which is made from H13 steel is 25 mm, the pin diameter of the tool is 6 mm, and the length of the pin is 7 mm. To ameliorate the friction heat and mixed ability, the pin of the tool is treated. The joints are produced in overlap configuration.

In this study, single-pass submerged FSW is performed at different parameters. The parameters and their levels used in this study are listed in Table II. The rotation speed and traverse speed include three different levels. The plunge depth of 0.4 mm and the attack angle of  $0^\circ$  are constant. The pin is plunged into the upper HDPE sheet until the shoulder touches the surface of the upper HDPE sheet. The rotating tool stays 1 s under the rotation condition before moving forward along the joint line. The temperature of the HDPE sheet below the shoulder increases due to the friction heating when the rotating tool advances. The localized heating softens the HDPE materials around the stirring pin. The combined action of the tool rota-

tion and translation gives rise to the HDPE materials that move from the front of the stirring pin to the back of the stirring pin. As a result of this process, two HDPE sheets are joined.

The weld tensile tests are carried out by CMT 5105 SANS microcomputer control electronic universal tensile testing machine (Shenzhen SANS Metering Technology), according to the GB/T 1040-92 standard, and the experimental results are recorded. The tensile specimens are prepared according to the dimension shown in Figure 3. The strength is obtained by averaging strengths of five individual specimens, which are welded with the same parameters.

To study the microstructure of the weld, thin slices (30  $\mu\text{m}$ ) are cut from the welded specimens using a LEICA RM2235 model rotary type microtome (Leica Microsystems Nussloch GmbH). These thin slices are investigated using OLYMPUS FV1000 laser scanning confocal microscope (LSCM) (Olympus Optical, Olympus Corporation) and XJP-300 metallurgic microscope (Nanjing Jiangnan Novel Optics), and the photographs of the cross-sections are obtained.

The thermal behavior of the materials is analyzed by STA 409 PC differential scanning calorimetry (DSC) (NETZSCH Grinding & Dispersing). Specimens (12–13 mg) are cut from different zones of weld. The heating rate of all specimens is  $10^\circ\text{C}/\text{min}$  at  $20^\circ\text{C}$  to  $170^\circ\text{C}$ . The experiment is conducted with  $\text{N}_2$  as a protective atmosphere and the flow rate of  $\text{N}_2$  was 30 mL/min.

Small angle X-ray scattering (SAXS) is used for the study of the orientation and long period of the samples. The examinations are performed using a Nano STAR SAXS (Cu  $K\alpha$ ,  $U = 40$  kV,  $I = 600$   $\mu\text{A}$ , Bruker AXS Company, Germany).

## RESULTS AND DISCUSSION

### The Tensile Tests on the Welds

In the process of FSW, the relations between total heat and parameters can be explained by<sup>12</sup>

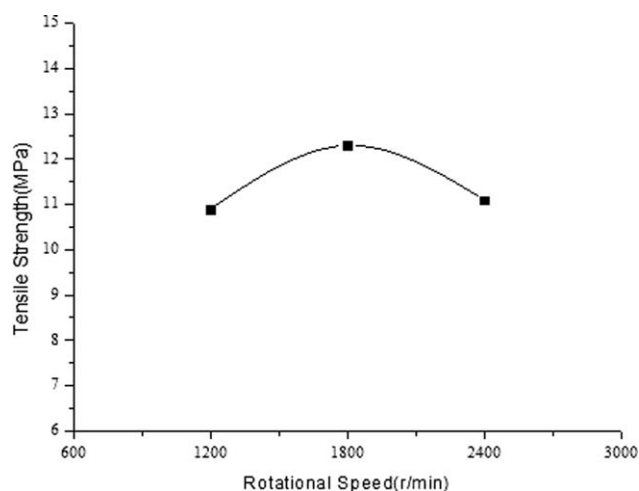


Figure 4. Effect of the rotation speed on the tensile strength at a constant traverse speed of 45 mm/min.

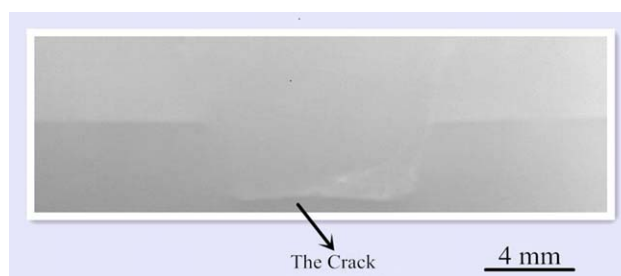
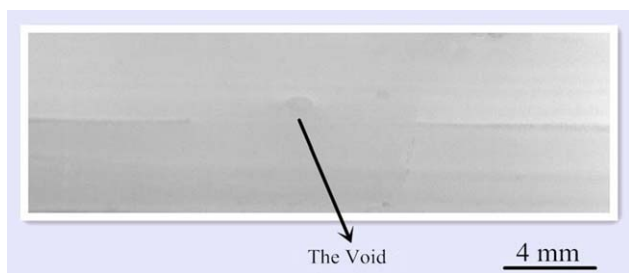


Figure 5. The crack due to insufficient heat input. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

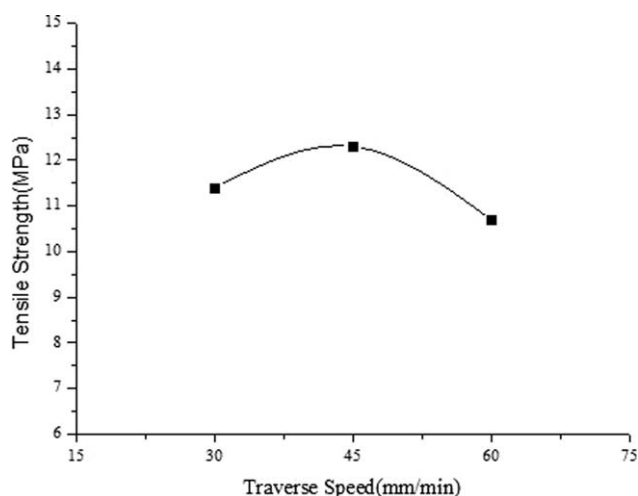


**Figure 6.** The void due to overmuch heat input. [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://wileyonlinelibrary.com).]

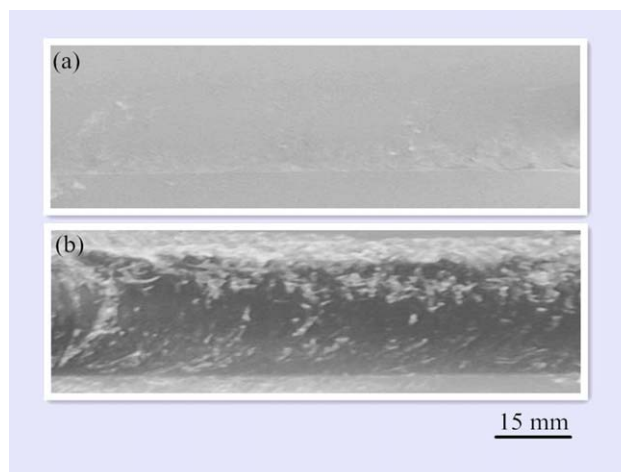
$$Q = \frac{2}{3} \pi \left( \delta \frac{\sigma}{\sqrt{3}} + (1 - \delta) \mu p \right) \omega \left( (R_1^3 - R_2^3)(1 + \tan \alpha) + R_2^3 + \frac{2}{3} R_2^2 H \right) \quad (1)$$

where  $Q$  is the total heat generation (W),  $\sigma$  is the yield strength (Pa),  $\delta$  is the contact state variable,  $\mu$  is the friction coefficient,  $p$  is the contact pressure (Pa),  $\omega$  is the tool angular rotation rate (rad/s),  $R_1$  is the tool shoulder radius (m),  $R_2$  is the tool probe radius (m),  $\alpha$  is the tool shoulder cone angle ( $^\circ$ ), and  $H$  is the tool probe height (m). As can be seen, when other parameters are determined, the total heat generation is related to the rotation speed.

Figure 4 shows the effect of rotation speeds on the tensile strength of the sample by FSW, and the traverse speed is 45 mm/min. It was revealed from Figure 4 that the tensile strength of the sample increased with the increase in rotational speed from 1200 to 1800 r/min. However, when the rotational speed further increased from 1800 to 2400 r/min, the tensile strength decreased. In the FSW of thermoplastics, whether the joint could succeed depended on how much the heat would input.<sup>25</sup> When the rotational speed was relatively low, small friction heat was produced, which led to the interface temperatures that could not reach the melting points of the polyethylene. Thus the materials in the weld nugget (WN) were not fully mixed, which led to a relatively low tensile strength. The crack which was caused by insufficient mix was shown in Figure 5.



**Figure 7.** Effect of traverse speed on the tensile strength at a constant rotation speed of 1800 mm/min.

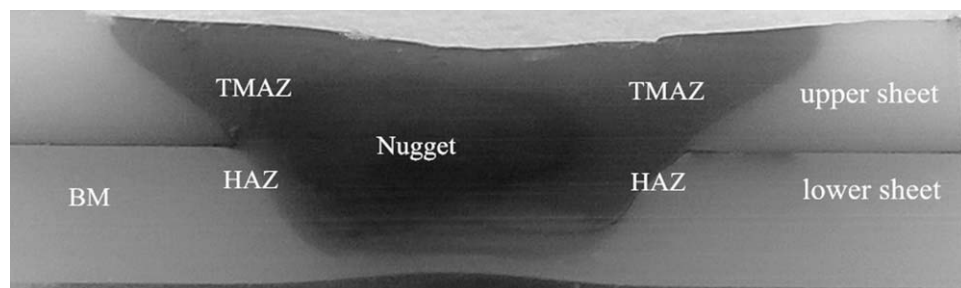


**Figure 8.** The weld surface of different welding surrounding: (a) under water (b) in air. [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://wileyonlinelibrary.com).]

With the increasing of rotational speed, the interface temperatures reached the melting points of the polyethylene, the melted polyethylene materials were stirred and squeezed, and macromolecule diffused in the whole weld surface. In this case, the maximal value of the strength was obtained. However, excessive rotational speed generated too much heating, which caused severe deformation to materials, and the temperature of interface would be too high, thus the WN regions easily formed the void, even might decompose. For that reason, the strength decreased.<sup>34</sup> Figure 6 shows the void which was caused by high heat input.

Figure 7 shows the effect of the traverse speed on the strength of the joints, and the rotation speed is 1800 r/min. It could be observed that the strength increased with the increase in traverse speed from 30 mm/min to 45 mm/min. On further increasing the traverse speed from 45 mm/min to 60 mm/min, the strength would be decreased. In the FSW of polymer, the main factor to improve the strength was sufficient flow and mix of the molten materials. The sufficient flow of the molten polymer caused larger diffusion at the interface and obtained higher weld strength.<sup>4</sup> When the traverse speeds were too low, the whole friction heat input was too high in the interface of the tool and work-piece, and it would produced extra friction heat which led to the polymer degrade,<sup>34</sup> which decreased the strength of the joints. However, at a too high traverse speed, the tool did not have enough time to soften the polymer materials and the ability of plastic softening materials filling in the cavity which caused by the walk of pin became weak. It easily formed tunnel groove in the weld seam. These factors caused the materials could not sufficient mix, thus the strength of the joints decreased. The maximal tensile strength of submerged friction stir welded polyethylene was 12.3 MPa under the process parameters of 1800 r/min, 45 mm/min.

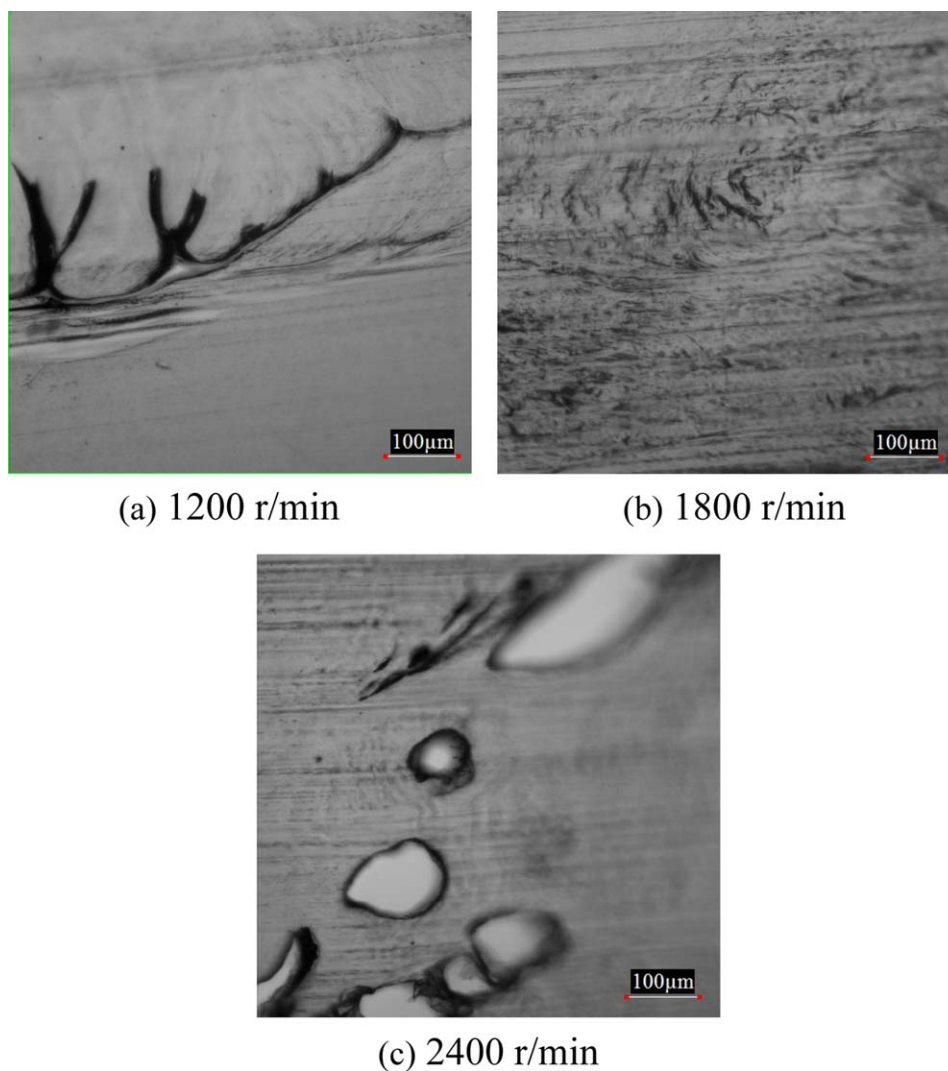
A contrast experiment was used to further study the influence of the welding surrounding on the tensile strength of the joints. The whole welding process was conducted in air, and the process parameters were 1800 r/min and 45 mm/min. The tensile



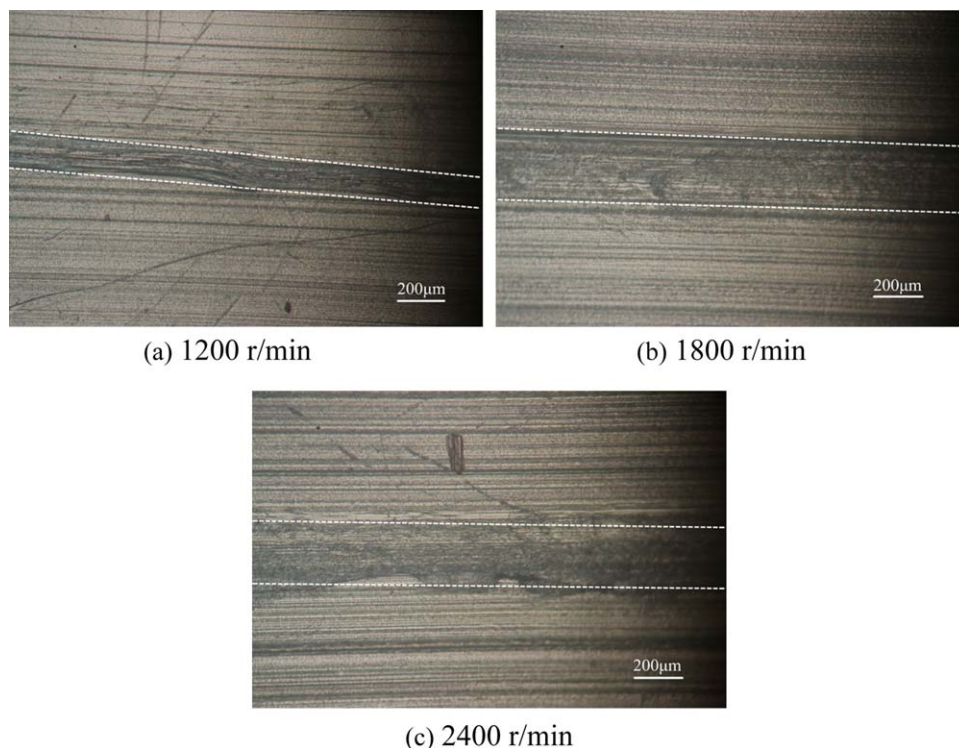
**Figure 9.** Macrograph of cross-sections perpendicular to tool traverse direction of submerged friction stir welded polyethylene.

strength only was 9.6 MPa (78% percent of the strength of value under the water). In the process of submerged friction stir welded, the existence of water could not only affect the heating rate and the peak temperature but also effectively control the thermal cycle of the weak part, thus increased the strength of the joints.<sup>29</sup> The weld surface of different welding surrounding

was shown in Figure 8. As can be seen from Figure 8, comparing to the sample under the water, the surface of normal joint material were highly affected by thermal conductivity of the material.<sup>35</sup> Polymer materials had low heat conductivity and short time of solidification, which led to high temperature



**Figure 10.** LSCM section photos of interfaces of welded specimens at different rotational speeds, and the traverse speed is 45 mm/min. [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://wileyonlinelibrary.com).]



**Figure 11.** The optical micrographs of cross-sections at different rotational speeds, and the traverse speed is 45 mm/min. [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://wileyonlinelibrary.com).]

gradient. In the process of normal FSW polyethylene, the low heat conductivity made overmuch heat concentrate WN and led to asymmetrical temperature distribution of weld region.<sup>20</sup> The rotating shoulder threw away the soft material from the weld region.<sup>34</sup> So the weld region became irregular, and the thickness of the upper sheet became thin. These factors ultimately led to the decrease of tensile strength of FSW polyethylene sheets.

#### Macrostructures and Microstructures

FSW was a complex process, and during the FSW of polymer, the materials undergo heat and cooling, plastic deformation and flow. A typical macrograph of the FSW joints was shown in Figure 9. There were four distinct zones: base material (BM), HAZ, thermomechanically affected zone (TMAZ), and WN. The WN and TMAZ undergo plastic deformation and frictional heating. However, the deformation strain of TMAZ was not sufficient. The HAZ which lied between the TMAZ and WN only was affected by heat.

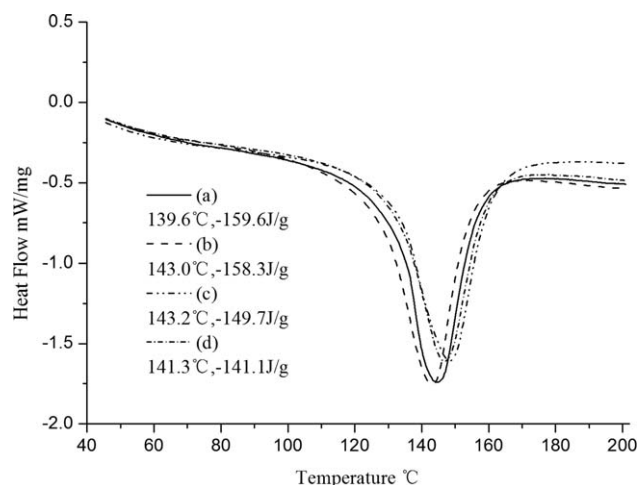
The LSCM section photos of welded specimens with different rotation rates were shown in Figure 10. Figure 10(a) shows the LSCM section photos of welded specimens, welded at 1200 r/min, and 45 mm/min. When the heat input was insufficient, the softened polyethylene could not sufficient fusion, which led to the presence of nonwelded areas. So the weld would not form a homogenous structure and the tensile strength had been weakened accordingly. Figure 10(b) shows the LSCM section photos of welded specimens, welded at 1800 r/min and 45 mm/min. A homogeneous structure could be observed in the image. There were no visible defects. The materials were evenly distributed, which was the reason for achieving a maximum tensile

strength under these process parameters. Figure 10(c) shows the LSCM section photos of welded specimens, welded at 2400 r/min, and 45 mm/min. The interfaces would form air bubble due to overmuch heat and stirring, and there was no enough time to discharge air bubbles due to the rapid cooling of polyethylene.

In the FSW of thermoplastics, the HAZ was special area. During tensile tests, all the fractures occurred in HAZ. The mixing of the two polymers could be assessed by examining cross-sections of the HAZ. Figure 11 shows the optical micrographs of HAZ, welded in different rotation speeds, and the traverse speed was 45 mm/min. It showed that the thickness of the HAZ in the weld region increased with the rotational rate increasing from 1200 r/min to 1800 r/min. However, when the rotational rate was further increased from 1800 r/min to 2400 r/min, the thickness of the HAZ would decrease. At the low rotation speed, the friction heat was insufficient and the materials were not fully mixed. There was not enough heat for melt polymer macromolecular to relax fully and diffuse. Thus the width of the interface of the HAZ was only 150  $\mu\text{m}$ . But when the rotational speed was overtop, the materials were excessive fusion even degeneration, which caused the width of the HAZ decrease to 260  $\mu\text{m}$ . In the optimal process of FSW, the width of the interface was 300  $\mu\text{m}$ . This change had the similar trend basically in joint strength. So it had reasons to believe that the joint strength was related to the width of the HAZ.

#### DSC

DSC was conveniently used to analyze the mechanical thermal behavior and quantify the energy of the structure-sensitive



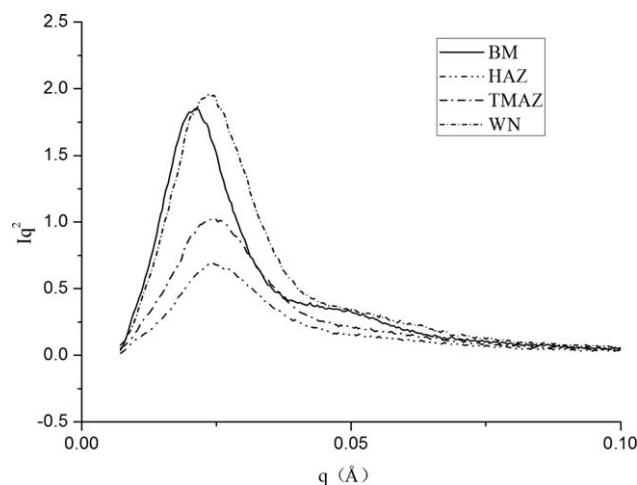
**Figure 12.** DSC curves of polyethylene in different conditions: (a) The BM; (b) The HAZ; (c) The TMAZ; (d) WN.

melting process.<sup>27</sup> The melting enthalpy ( $\Delta H$ ) was that the crystallization absorbed heat due to partial melting, which was got by measured melting peak area in the DSC curve, and it could be used to measure the degree of polymer crystallization. Polymer melting enthalpy was directly proportional to the degree of crystallinity. The degree of crystallinity was quantified by<sup>9</sup>

$$W_c = \frac{\Delta H_m}{\Delta H_m^0} \times 100\% \quad (2)$$

where  $\Delta H_m$  was the melting enthalpy of the sample, and the  $\Delta H_m^0$  was the melting enthalpy of perfectly crystalline polyethylene, of which the value was 293 J/g.<sup>18</sup>

The DSC curves of different weld zones were showed in Figure 12. The crystalline content of different conditions of BM, HAZ, TMAZ, and WN approximate were 54.5%, 54.0%, 51.1%, and 48.2% according to the formula (2).



**Figure 13.** SAXS curves, shown as  $Iq^2$  versus  $q$  plots, in the different regions.

It could draw the conclusion that the crystalline content of the weld regions decreased comparing the parent material. The crystalline content of WN decreased further compared to HAZ and TMAZ. Crystallization could be formed at a low force and slow cooling rate.<sup>23</sup> The FSW process was fast welding process, which caused a high cooling rate, thus decreased the crystalline content of weld regions. In the structure of the polymer, crystalline parts could improve the polymer density, hardness, and tensile strength but reduced the impact strength. So the decrease of the crystalline content was a factor of the decrease of the tensile strength.

### SAXS

SAXS was suitable for the study of the sub-microstructure of the polymer. The  $Iq^2$  versus  $q$  curves was presented in Figure 13 for the different weld regions. The existence of scattering peak indicated that the samples exhibited regular nanostructure. The relations between long period ( $L$ ) and the scattering vector ( $q$ ) of the polymer could be obtained in terms of equation<sup>36</sup>:

$$L = 2\pi/q \quad (3)$$

The  $q$  values which correspond to the scattering peak of different regions of parent material, HAZ, TMAZ, and WN were 0.02135, 0.0242, 0.02456 and 0.02385, respectively. Thus the long period ( $L$ ) of weld regions was decreased relative to parent material. In the process of FSW, the random order of polymer molecular chain was enhanced, and the crystal area was decreased, thus the long period was decreased.

### CONCLUSIONS

In this article, submerged FSW was used to weld the polyethylene sheets. Tensile strength of friction stir weld polyethylene sheets was studied. The main conclusions are summarized as follows:

- i. The optimum welding parameters are the tool rotation speed of 1800 r/min and the tool traverse speed of 45 mm/min. The maximum tensile strength value was 12.3 MPa, which was significantly higher than that of normal joint. It appeared that the tensile strength depend on the rotation speed and traverse speed.
- ii. The results of DSC showed that the crystalline content of joints decreased.
- iii. The microstructure of the joint was affected by the amount of heating and the materials flow and mixing.
- iv. During tensile tests, all the fractures occurred in HAZ. The formation of crack and air bubble and the decrease of the crystalline content were major reasons of the decrease of the tensile strength.
- v. The analysis of SAXS indicated that the long period of weld regions was decreased relative to parent material.

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