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Effect of Adding Al₂O₃ on The Macrostructure of Friction Stir Welding Polypropylene Sheet

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Abstract

In this study, an experimental investigation had been carried out to determine the effect of alumina (Al₂O₃) addition in joining Polypropylene. Polypropylene is a thermoplastic material used mostly as a non-metallic material. One of the joining methods that can be applied in Polypropylene is Friction Stir Welding (FSW). The use of Al₂O₃ as a filler was to modify the Polypropylene matrix's properties to improve the joint's quality. The Al₂O₃ powder with 99.9% purity was inserted along the Polypropylene plates in the groove. This research analyzed the role of adding Al₂O₃ and tool rotation speed concerning the joint's quality. The experiment was performed under different values of tool speed rotation (204 rpm, 356 rpm, 620 rpm, and 1140 rpm) and the presence or absence of the addition of alumina powder. Then, the joint's quality was visually observed by optical macroscopy at the top and cross-section view. From macroscopic observations, adding alumina could make the visual of the joint look better and result in minimum defects than the joint without alumina addition. Instead, the increasing tool speed rotation helped the distribution of alumina during the welding process.

1 Introduction

Polypropylene is a thermoplastic-type polymer formed from long molecules [1]. Polypropylene is becoming the most widely used thermoplastic polymer as a non-metallic raw material in various industrial sectors. The automotive industry, aviation, and medical equipment use Polypropylene due to its high strength, anti-corrosion, more economical price than metal, and good manufacturability [2]. The manufacturing process of joining polymers is often carried out with adhesives. However, this method is ineffective because it visually causes a joining with poor mechanical properties [3-5]. Various studies have been conducted to improve the mechanical properties or the visual of polymer joints. One of the joining methods that can be used on polymers is friction welding, known as Friction Stir Welding (FSW), which was first introduced in 1991 by Wayne Thomas at The Welding Institute (TWI) [6]. FSW is a type of solid state welding where the process of joining two plates is carried out in a solid state (without melting the metal) by utilizing the heat generated from the rubbing of tool rotation against the material's surface [7].

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FSW has the advantage of being environmentally friendly because it does not require a protective gas and does not produce sparks or smoke. Instead, this joining method is also energy efficient, and in terms of cost, this method is more affordable [8]. The selection of FSW welding parameters, such as tool speed rotation, transverse speed, and tool geometry, needs to be adjusted to the material properties to produce a good joint. The rotation of the welding tool determines the adequacy of the heat input that enters during the welding process [9]. During the joining process, the friction between the tool and workpiece causes a decrease in the mechanical properties of the weld joint [2]. Several studies were carried out to improve the weld joint properties by adding reinforcing powder as an alloying material in welded joints [10]. In polymer materials, reinforcement can be done with nanoparticles as an alloy [11]. Alloys used as reinforcement are Cu, Si, Zn, Mg, Mn, and ceramics materials such as TiB2, ZrB₂, TiC, SiC, B₄C, Al₂O₃, and AIN [10,12]. For example, adding CNTs as reinforcement in Polypropylene [13], Polycarbonate (PC)/ Al₂O₃ [14], ABS/ SiO₂ [15], PMMA/ Al₂O₃ [16].

One of the ceramic nanoparticles used as reinforcement to modify the mechanical properties of polymers is Al_2O_3 [16]. This chemical compound has good dielectric properties and thermal conductivity and has high strength and resistance to high temperatures [17]. In previous studies, research has been conducted on the tensile strength and hardness of Al_2O_3 -reinforced Polypropylene joints as reinforcement using the FSW method, which is equipped with additional heating to help soften the material [2]. Until now, there is still minimum information about the reinforcement carried out on polymer materials using the FSW method. Therefore, this study will discuss the effect of mechanical properties such as tensile strength and hardness by varying the rotational speed during Polypropylene's friction stir welding process with the addition of Al_2O_3 reinforcing powder.

2 Experimental Methods

2.1 Materials

The material used in this research was a Polypropylene (PP) sheet with a 6 mm thickness produced by Rochling with mechanical properties shown in Table 1. An alumina (Al₂O₃) powder with an average diameter of 50 nm and 99.9% purity was used as a nano-filler with mechanical properties shown in Table 2.

| Description | Value | Unit |
|-----------------------|-------|--------------------|
| Density | 1 | gr/cm ³ |
| Tensile strength | 33 | MPa |
| Modulus of elasticity | 1450 | N/mm ² |
| Hardness | 75 | N/mm ² |
| Impact Charpy | 1.26 | J/cm |

Table 1. Mechanical properties of PP [18]

| Table 2. | Mechanical | properties | of $Al_2O_3[2]$ |
|----------|------------|------------|-----------------|

| Description | Value | Unit |
|-----------------------|-------|--------------------|
| Density | 3.86 | gr/cm ³ |
| Tensile strength | 416 | MPa |
| Modulus of elasticity | 380 | N/mm ² |
| Hardness | 1440 | kg/mm ² |

2.2 Methods

These PP sheets were cut into $100 \times 250 \text{ mm}^2$ plates and trimmed by a conventional milling machine. As shown in Figure 1, a minor rectangular groove with a depth of 1 mm and a width of 2 mm was made along the specimen length for each plate to place the alumina powders. The design of the groove was made

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closed to restrict the powder from expelling out during the FSW process. After inserting the alumina powder into the groove, a cylindrical tool with dimension, as shown in Figure 2, made of American Iron and Steel Institute (AISI) 4140 used to stir and join the plate. The FSW process was done using a 57-3C conventional milling machine modified with FSW tool attachment, such as a backing plate and clamping. The travel speed of the welding was 7.3 mm/min, and various tool rotations (204 rpm, 356 rpm, 620 rpm, 1140 rpm). The tool tilt angle was 2° with 0.1 mm plunge depth. During the welding process, the surface temperature of the workpiece was measured by a non–contact thermos gun. Welded workpieces (coupon test) were cut into several parts according to the tests carried out by a Computer Numerical Control (CNC) machine.



Figure 1. Workpiece: (a) Plate dimension, and (b) Groove dimension



Figure 2. Tool dimension

3 Results and Discussion

This study will discuss the visual of friction welding joints by taking macro photos. The macro photos were taken by digital camera and optical microscope from both, the top and cross-section view to observe

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the flaws in this FSW joint of Polypropylene. Flaw formation when FSW had been identified to occur because of either imbalance in material flow or geometric factors associated with the position of the tool concerning the joint, the temperature data becoming the support data for this study. The PP joint with the addition of alumina compared with the PP joint without alumina. The welding process was successfully carried out using a cylindrical pin with several kinds of tool speed rotation (204 rpm, 356 rpm, 620 rpm, 1140 rpm) to determine the best joint quality from four different rotating speeds at 7.3 mm/minute tool travel speed. The effect of tool speed rotation was to generate heat during the welding process and optimize the stirring process carried out by the pin and shoulder.



Figure 3. Graphic welding temperature

Several researchers had explored temperature measurement during FSW and different methods applied to predict the weld temperature. In this study, to measure the temperature during the FSW process, a non-contact thermos gun was used. The temperature was measured along the joint every 2.5 cm for four tool speed rotations. It is shown in Figure 3. It can be seen that the welding temperature for the four different rotational speeds had the same thing that tended to increase for every 2.5 cm of measurement. But at 1140 rpm tool speed rotation, the temperature increased sharply in the last 5 cm. The increased temperature occurred because the melted material thrown from the welding zone was not entirely stored on the retreating side but was stirred again below the shoulder, increasing tool pressure on the material, and causing more friction and shear. The parameter that affects the temperature of the FSW is tool speed rotation, tool travel speed, plunge depth, the backing plate material, and the welding plate's thickness [19]. The optimum welding temperature is relative to the material and not a specific temperature that can be broadly applied across all materials [20]. The heating process is one of many factors for a successful FSW process. However, those parameters must be optimized to control the welding temperature [20]. The temperature changes occurred due to the different process parameters in this hardness investigation. Usually, the temperature profiles on the retreating side are slightly lower than those on the advancing side [3]. Similar conditions occurred in the FSW process's hardness profiles of Polypropylene material.

The next focus will be the macro photos of the welding results, as shown in Figures 4 and 5. From macro photos, several defects can be identified found in welding joints, both on the surface and cross-section, to find the effect of the parameters of rotation speed and the addition of alumina on visual and macroscopic joint quality from Polypropylene plates.

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Figure 4. Welding visual top view: (a) Without $Al_2O_3 - 204$ rpm, (b) Without $Al_2O_3 - 356$ rpm, (c) Without $Al_2O_3 - 620$ rpm, (d) Without $Al_2O_3 - 1140$ rpm, (e) With Adding $Al_2O_3 - 204$ rpm, (f) With Adding $Al_2O_3 - 356$ rpm, (g) With Adding $Al_2O_3 - 620$ rpm, and (h) With Adding $Al_2O_3 - 1140$ rpm

Polypropylene peeling out of the plates formed a small crystal from the parent material found in all welding joints in Figures 4 and 5. The cause of defects was the rubbing of the tool that occurred over the plates during the welding process. The rubbing of the tool's shoulder on the top surface removed the molten material in the form of tiny crystal particles. Instead, the tool rubbing during welding produced accumulated material along the weld zone on the retreating side. The accumulated material could form a defect called a flash on a big scale. The different results of the weld seen from the top view are shown in Figure 4. From Figure 4 (a)-(d), we can find a flash in almost all welds. Flash is a typical weld surface defect on FSW caused by excessive heating during the welding process. It is formed by the soft material near the shoulder edges that flows from the weld zone and accumulates at the retreating side during the FSW [21]. Figure 4 (a)-(d) and Figure 4 (e)-(h) proved that the higher the tool speed rotation, the bigger the flash defect.

The joint resulting from high-speed tool rotation speed and low tool travel speed will generate high temperature. At this point, the molten material flows out of the stir zone when the tool travels along the joint, forming like flower structures edging around the stir zone shown in Figure 4 (d) and (h). The flash defect found in Figure 4 (f)-(h) resulted from welding with alumina as an addition. However, the defect resulted on a small scale (accumulated material) if compared with the welds without alumina addition, as shown in Figure 4 (a)-(c). On a small scale, this defect did not deteriorate the joint quality. This reduction

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in the flash's severity was helped by adding alumina powder with a high melting point to prevent the Polypropylene from molten more.

Another surface defect that can be found in this research was the groove defect shown in Figure 4 (a) and Figure 4 (e). The groove defect appeared from the wormhole that extended until the weld surface. It was caused by shoulder contact with the surface material. The lack of shoulder plunge would make minimum contact between the shoulder and the weld region, resulting in less pressure during welding [22]. Moreover, groove defects will appear if the tool speed rotation is too low because insufficient heat input is not enough to melt the material. More heat will result in the bond of the material formed not uniform along the joint interface. Therefore, the defects appear in the form of grooves because the material is not bound well with the advancing side. Not adequately bonded material will be stored on the retreating side, resulting in poor contact with the base material [22]. This study proved that groove defects appeared in weld joints with low tool speed rotation. Instead of that, the insufficient heat input could also be formed the surface looks to have a rough texture shown in Figure 4 (a) and Figure 4 (e)-(f). It occurred when the shoulder moved along the joint, but at low temperature, so it will leave feed marks. This defect might have an impact on mechanical properties like fatigue cycle life.

The following surface defect observed visually was surface galling. These types occurred due to FSW under too hot a processing condition. It was manifested by material particles that settled loosely on the surface of the weld seam or protrude from the weld surface, as shown in Figure 4 (b)-(c). The excessive heat generated from 356 and 620 rpm tool speed rotation could cause the FSW process's softening beyond the tool shoulder's boundary. It can be prevented by regulating parameters affecting the heat generation rate, such as tool speed rotation or travel [22]. This defect did not occur in Figure 4 (g)-(h), where alumina powders were inserted as fillers. This might be due to alumina powder having a higher heat resistance than Polypropylene, so mixing PP and alumina in the weld zone forms materials such as composites in the weld zone, which form Polypropylene with better properties. This can prevent the Polypropylene from melting more because its heat resistance has increased. But in Figure 4 (g), the voids found at a few surface joint points. This defect occurred because of lousy stirring during the welding process. The bad stirring caused molten material to form by rubbing the tool unevenly distributed [6]. Also, the heat input must be increased to mix the Polypropylene and alumina.

The comparison of Figure 4 (e), Figure 4 (f), Figure 4 (g), and Figure 4 (h) was also observed. The result of weld joint from high tool speed rotation results in a good distribution of alumina along the weld zone. It was because the Polypropylene alumina was perfectly molten at a high temperature. When the tool moved along the joint, the high temperature melted the mixed material and caused a decrease in the surface. So, not only the pin but also the shoulder stirs the material [23]. In low rotation, the alumina was only distributed along the stirring zone because the zone pressed by the shoulder did not melt, unlike at the high tool speed rotation. However, adding alumina that can be observed macroscopically was visually better when compared to welds with the specimen without alumina addition.

From Figure 5 below, the cross-section view of the weld zone can be observed. Figure 5 (a) and Figure 5 (b) formed a convex shape in the weld region along the line of the joint. Also, in Figure 5 (a)-(c), the nugget area created a boundary with the parent material. It may be caused by low tool speed rotation during the FSW process [6]. It was because the higher tool speed rotation resulted in better bonding between the parent material and the weld nugget. At tool speed rotation 620 rpm, shown in Figures 5 (c) and (g), they looked more suitable than the other. It might cause the heat input to be enough to melt the material during the welding process.

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Figure 5. Cross section of the weld: (a) Without $Al_2O_3 - 204$ rpm, (b) Without $Al_2O_3 - 356$ rpm, (c) Without $Al_2O_3 - 620$ rpm, (d) Without $Al_2O_3 - 1140$ rpm, (e) With Adding $Al_2O_3 - 204$ rpm, (f) With Adding $Al_2O_3 - 356$ rpm, (g) With Adding $Al_2O_3 - 620$ rpm, and (h) With Adding $Al_2O_3 - 1140$ rpm

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The welding results at a rotational tool speed of 204 rpm in Figure 5 (a) showed a concave shape in the weld area. This welding process also formed small particles on the surface. Small particles formed are common in the FSW welding process, resulting from shoulder friction with the workpiece surface [24]. In addition, low tool speed rotation also affected the formation of these tiny particles because the temperature generated during the welding process was not high. As a result, the shoulder cannot soften the material, so the moving process in welding did not take place optimally. This part of the welded joint was also clearly visible as the boundary between the weld area and the Polypropylene material. The inhomogeneous joint results from using a lower tool rotational speed so that an inhomogeneous distribution of molten material occurs in the weld area. This inhomogeneous connection can cause a decrease in mechanical properties because the weld area and the parent material have a less strong bond [6].

In comparison, welding results with adding Al_2O_3 showed the boundary between the weld area and the parent material that looked vaguer. However, some voids and tunnels were formed at the root of the weld joint due to the lack of heat input to soften and mix Polypropylene and Al_2O_3 powder. The addition of Al_2O_3 caused no lack of penetration defects, as shown in Figure 5 (a). The nature of Al_2O_3 , which had a better thermal conductivity value than PP, made it easier to conduct heat. Then, the heat received during the welding process will be more easily transmitted to the root connection by Al_2O_3 particles.

The results of the following study that will be evaluated were studies with the tool speed rotation parameter of 356 rpm, as shown in Figures 5 (b) and (f). The result of welding without adding Al₂O₃ showed a convex shape seen on the upper surface of Figure 5 (b). The convex shape was the side of the surface galling defect formed along the joint. Meanwhile, in the result of splicing without the addition of Al₂O₃ shown in Figure 5 (f), the convex shape did not appear because there was no surface galling defect. Then, the PP welded joint's root part looked like the plate interface could be more perfectly joined. This defect is known as lack of penetration. This defect has many causes, such as a tool probe that is too short, a plunge depth that is too low, or a different plate thickness [22]. The research that has been carried out analyzed that this defect arose due to the heat generated from the rotating speed of the tool not reaching the root of the connection in the PP joint weld, as shown in Figure 5 (b). However, with the addition of Al₂O₃, the lack of penetration defect was not formed because the Al₂O₃ particles helped to transmit heat to the root connection. In addition, Figure 5 (b) shows the boundary between the weld area and the parent material, which was more apparent when compared to the welding results given the addition of Al₂O₃ as a filler, as shown in Figure 5 (f). This because the Al₂O₃ particles helped the heating process by spreading the heat to the boundary between the weld area and the parent material. So that the heat received at the boundary was more optimal and the bond formed becomes stronger. However, defective voids in the PP/ Al₂O₃ joint were formed as a result of the low rotational speed of the tool to mix PP with Al₂O₃. Al₂O₃, which was not mixed with PP thoroughly, will interfere with the moving process, forming defect voids.

The results of macro observations with a rotation speed of 620 rpm, as shown in Figures 5 (c) and (g), showed that the resulting welds were smooth with fewer defects. Because at this rotational speed, the resulting temperature was sufficient to soften the material so that the stirring and joining process can occur optimally. The welding results shown in Figure 5 (c) still showed the boundary between the weld zone and the parent material, which indicated that the bond between the two parts could be better. However, the tool rotation speed results of 620 rpm were better than the previous parameters. Meanwhile, the bond between the weld area and the parent material also looked better when welding with Al₂O₃. The Al₂O₃ particles helped transmit heat to the weld area and the parent material. So that the heat received at the boundary was more optimal when compared to the connection without the addition of Al₂O₃. The phenomenon of void defects appeared in the welding results without adding Al₂O₃ in Figure 5 (c) due to the high rotational speed of the tool. The increase in tool rotation speed also increased the friction heat during the welding process, causing the material to become too soft. The too-soft material will cause more molten material to be thrown from the weld area and not fill the joint correctly so that voids are formed [23]. Observations of the macrostructure on the welding results using the 1140 rpm tool rotation speed parameter in Figures 5 (d) and (h) showed poor welding results. The weld surface looked concave due to material loss that occurred

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during the welding process. Severe surface subsidence occurred due to the high rotational speed of the tool, causing an increase in temperature in the welding process. This increase in temperature causes the welding temperature to approach the melting point of Polypropylene so that the material becomes too soft [24]. The too-soft material will be thrown out of the weld area during the joining process and form a flower-shaped flash, as seen in Figures 4 (d) and (h) along the retreating side. However, if observed more clearly, the PP/Al₂O₃ connection shown in Figure 5 (h) looked thicker than the PP connection in Figure 5 (d). This may be because Al₂O₃ particles, which were more heat resistant than PP, will help PP to retain heat. So that the incorporation of Al₂O₃ can reduce the material thrown out of the weld area due to too high heat, although it did not have a significant effect. However, it is sufficient to show that adding Al₂O₃ can improve the welding results of PP joints. This severe subsidence dramatically affected the mechanical properties of the joint. Thus, the workpiece at this parameter was not suitable for testing. The tunnel defect was seen at the root of the connection in Figure 5 (h). This welding defect was also caused by the material being too soft so that the soft material will stick to the pin (sticking). The sticking phenomenon will interfere with the moving process during the welding process, resulting in a weld joint that is not optimal [23].

4 Conclusions

The study has declared that Polypropylene plates can be joined using the FSW process. The current knowledge of FSW of thermoplastics has been presented, and an experimental investigation was carried out on PP sheets of 6 mm thickness. A cylindrical tool was used to stir materials during the welding process. The 2×1 mm groove was made on the side of each plate as a place for alumina powder to prevent the alumina powder from being expelled during the welding process was proven successful. The macrostructure investigated the effects of adding Al₂O₃ and the processing parameters, such as tool speed rotations (204, 356, 620, and 1140 rpm). From macroscopic observation, adding alumina results in better-quality weld joints than plates without alumina. So that the defects that arose as a result of the welding process could be minimized by the presence of alumina powder as reinforcement, the mechanical properties of filler powder (Al₂O₃), which had high heat resistance than the parent material (Polypropylene), helped to improve the properties of Polypropylene which had a lower melting point. Instead, the welding temperature was analyzed against the quality of the weld joint. The temperature during the welding process was affected by tool speed rotation: the higher the tool speed rotation, the higher the heat generated by the tool and shoulder friction. The welding temperature tended to rise over time. The high tool speed rotation that was used affected the distribution of alumina in the weld region. However, with the high temperature resulting from the high tool rotation, the defect occurred, such as the flash being more significant, causing material loss and creating a poor-quality joint.

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