Improving appearance and mechanical strength of aluminum-polypropylene/talc composite friction stir joint using a novel tool design

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Abstract

In this research, friction stir joining of aluminum-polypropylene/talc composite was investigated due to the numerous applications of aluminum-polymer joints in automotive and aerospace industry. A novel tool design, including a rotary/stationary holder, was used to improve the appearance and mechanical performance of friction stir lap joints by preventing the mixed molten polymer and aluminum particles from exiting the stirred zone. Effect of tool transverse speed on the joint microstructure and strength was investigated. Using the rotary/stationary holder, tensile-shear strength of the joints increased (by ~45 to ~220% at various transverse speeds). Since during the tensile-shear test all joints failed from the aluminum/stirred zone interface, joint strength was affected by the formation of anchor-like aluminum pieces in the composite substrate as the mechanical locks, reaction layer at the joint interface, and large voids near the aluminum/stirred zone interface. Joining at the optimum transverse speed led to the highest tensile-shear peak load of 315 N.

Keywords Friction stir joining · Aluminum · Polypropylene · Composite · Joint strength

1 Introduction

Production of lightweight structures is one of the highly effective solutions to the “CO2 emission” problem by reducing the amount of fossil fuel consumption. One way to produce lightweight structures is to replace some parts of the metallic structures with lightweight materials like polymers and polymer matrix composites [1–4]. This approach can improve the strength-to-weight ratio in the structure. However, fabrication of successful dissimilar metal-polymer joints has been a great challenge for researchers in the last few decades, mostly due to the high difference between their physical, chemical, and mechanical properties [5–7].

The most common metal-polymer joining processes are mechanical and chemical methods, using bolts, pins, and adhesives [8, 9]. However, mechanical joints can promote crack initiation in the material due to the presence of stress concentration sites. In adhesives, the joint strength depends highly on the surface energy of the base materials [10]. Therefore, other joining methods have been explored by researchers to make reliable metal-polymer joints such as laser joining [11–14], additive joining [15, 16], and friction stir joining (FSJ) [17–19]. FSJ, based on the friction stir welding concept, is claimed to include the advantages of both mechanical and chemical joining mechanisms for the metal-polymer joints. Compared to the other joining methods, FSJ also needs less surface preparation and provides shorter joining time, as well as low energy consumption and high economic advantages [20–23].

Various researchers have worked on the metal-polymer joints using FSJ and have found out that there are a few important factors determining the joint strength. The first and the most contributing factor to the joint strength is formation of mechanical locks. Mechanical locks in FSJ are mainly formed in the shape of anchor-like aluminum pieces penetrated into the polymeric substrate. Huang et al. [24, 25] in two separate researches investigated the lap joining of aluminum alloys to the polymer and polymer-based composite and found that the main joining mechanism was the formation of macro/microscopic mechanical locks. The macroscopic mechanical locks were anchor-like aluminum segments at the edge of the
stirred zone (SZ), which penetrated into the melted and re-solidified polymer. The strength of the joints was found to be directly proportional to the size of these anchors.

Another influencing factor on the metal-polymer friction stir joint strength is the nature and properties of the reaction layer formed at the aluminum and stirred zone interface. Indeed, in addition to the mechanical locks, chemical bonds can be formed at the aluminum/stirred zone interface. These bonds (mainly hydrogen bonds) are formed between the oxygen atoms from surface oxides and the hydrogen atoms from the polymer in the stirred zone, as well as between other atoms like aluminum and magnesium. The extent of chemical bonding area varied by the process parameters and the related heat input [1, 6, 26, 27].

One of the important challenges in the FSJ of metals to polymers is the unsuitable appearance of the joint which has not been regarded in the previous researches. This unsuitable joint appearance is caused by the protrusion of the melted polymer from the stirred zone and formation of tunnel-like voids along with the other defects [27, 28]. The appropriate joint appearance not only can result in the superior joint strength, but also may increase the joint industrial applications in which the joint appearance is of great significance.

Application of the polypropylene- and polypropylene-based composites has a growing trend in the automotive industry. For example, polypropylene/talc composite has common applications in the interior and exterior parts of cars [29, 30]. However, according to the best knowledge of the authors, no attempt has been made so far to join polypropylene/talc composite and aluminum alloys using friction stir processes. Consequently, this work studies the friction stir joining of polypropylene/talc composite to Al–Mg alloy. A novel rotary/stationary holder, which has not been used in the previous researches, is employed in order to improve the joint appearance and strength by preventing the mixture of molten polymer and aluminum particles from exiting the stirred zone. Additionally, effect of the tool transverse speed is evaluated on the joint performance.

### 2 Materials and methods

Al5052-H34 aluminum alloy sheets with the dimensions of 100 × 70 × 2 mm³ and polypropylene-20%talc composite sheets with the dimensions of 100 × 70 × 2.5 mm³ were used as the metallic and polymeric parts of the joints, respectively. PP-20%talc was produced by making a mixture of 75 wt% PP-Z30S, 20 wt% talc powder, and 5 wt% PP-graft-maleic anhydride (PP-G-MA), extruding and cutting the mixture into granules and injection molding of the granules into final sheets. Talc powder with an average size of 20 μm was added to PP as a reinforcement to improve the tensile strength of the polymer matrix and reduce the coefficient of thermal expansion [31, 32]. PP-G-MA was added to the mixture due to its compatibilizer properties, which can help form a more coherent interface between PP and talc, as well as help better dispersion of talc into the PP matrix [31]. Chemical composition and mechanical properties of Al5052-H34 aluminum alloy (measured by spark emission spectrometry and uniaxial tensile test, respectively) are presented in Table 1. Table 2 shows the chemical composition (of the pre-injected composite granules), mechanical properties (measured by uniaxial tensile test), and melting temperature (measured by differential scanning calorimetry) of PP-20%talc composite.

The tool used for FSJ was made of H13 steel with a hardness of ~52 HRC, composed of a shoulder with diameter of 16 mm and a triangular pin with 4.3-mm sides and 3-mm height. Schematics and macrograph of the tool are presented in Fig. 1a. Aluminum and polymer sheets were joined in the lap joint configuration by an overlap of 25 mm. Preliminary tests showed that while the composite sheet was placed on top and in contact with the tool shoulder, the amount of generated frictional heat was much lower. Additionally, the polymer base composite (with considerably lower heat conductivity compared to aluminum) was not able to conduct the generated heat between the tool shoulder and the composite to the adjacent regions and the bottom aluminum sheet. Therefore, aluminum was placed on the composite sheet so that the

| Table 1 Chemical composition and mechanical properties of Al5052 base sheet |
|---------------------------------|-----------------|-----------------|
| **Chemical composition (wt%)**  | **Mechanical properties** |
| Al5052-H34                      | Base            | Mechanical properties |
| Al     | Mg | Cr | Fe | Si | Mn | Tensile strength (MPa) | Elongation (%) |
| Base   | 2.02 | 0.18 | 0.33 | 0.15 | 0.04 | 266 | 15.4 |

| Table 2 Chemical composition, mechanical and physical properties of composite base sheet |
|---------------------------------|-----------------|-----------------|
| **Composition (wt%)** | **Mechanical properties** | **Physical properties** |
| PP-20%talc | PP | PP-g-MA | Talc | Tensile strength (MPa) | Elongation (%) | Melting temperature (°C) |
| 75 | 5 | 20 | 34 | 10 | 166 |
tool shoulder was in contact with the aluminum sheet (Fig. 1b). Some important parameters of FSJ such as the tool rotational speed, rotating direction, and tilt angle were determined using preliminary tests and were set to 600 rpm, counter clockwise, and 2°, respectively. Five levels of tool transverse speeds ($v = 50, 75, 100, 125, \text{and} 150 \text{ mm/min}$) were used to make the joints. A K-type thermocouple was also placed between the sheets at the edge of SZ in the middle of the joint, in order to determine the effect of welding parameters on the temperature variation during the process (Fig. 1c).

In order to improve the joint appearance, a novel rotary/stationary holder was designed and used (Fig. 2). The rotary part included a ball bearing with an inner diameter equal to the tool shoulder diameter (part “i” in Fig. 2a), so that the ball bearing would be in contact with the joining tool and rotate by it at the same rotational speed (i.e., 600 rpm in this research) during the joining process. The outer part of the
ball bearing (part “ii” in Fig. 2a) was welded to a plate (part “iii” in Fig. 2a) as the stationary part of holder. Finally, a smaller plate as wide as the tool shoulder diameter (part “iv” in Fig. 2b) was welded in other side of the first plate. This second (lower) plate was designed long enough to keep pressure on the SZ for a few seconds (at least 20 s) after passing the welding tool to ensure enough cooling (Fig. 3). Consequently, given the maximum tool transverse speed used in this work (i.e., 150 mm/min or 2.5 mm/s), the lower plate length was designed to be 50 mm. Using this design, while the rotary part rotated with the tool shoulder, the stationary part did not rotate with the tool. Thus, the stationary part could move with the tool along the joint seam and its bottom portion applied pressure on the stirred zone behind the tool in order to prevent the molten polymer and aluminum particles from exiting the SZ.

After joining, appearance of the joints was observed using stereomicroscope. For macro/microstructure investigation of the joints, transverse section of the joints was prepared using conventional metallographic techniques. Optical microscope and field emission-scanning electron microscope equipped with an energy-dispersive spectrometry (EDS) detector was used to study the joint interface, composition of the reaction layer, and fracture surface after tensile-shear test. An image analysis software was used to quantify the size aluminum anchors formed at the joint interface. Differential scanning calorimetry (DSC) analysis was also carried out on the base composite as well as the samples from the SZ at various transverse speeds in order to assess the thermal effects of joining process on the polymer base of composite.

Two samples from each joint were used for tensile-shear test, according to the ASTM D1002-5 standard (dimensions are presented in Fig. 1c). The tension rate was set to 0.5 mm/min.

### 3 Results and discussion

#### 3.1 Effect of rotary/stationary holder on joint appearance and strength

Figure 4 shows the macrograph from top view and the macrostructure of transverse section of the joints made with the conventional tool without the rotary/stationary holder at different transverse speeds. During the joining process, the frictional heat melted the polymer and simultaneous vertical pressure of the tool pushed the melt towards the SZ. The triangular pin tore large segments of aluminum and stirred them with the polymeric melt inside the SZ. The rotating pin also crushed the separated Al segments into smaller pieces and resulted in formation of an aluminum-polymer composite material in the SZ. Consequently, a joint was formed between the aluminum and composite sheets. FSJ process comes with some inherent issues, namely weld thinning, kissing bonds, and keyhole, as well as other possible flaws like tunnel, groove, and cavity defects [33]. However, no visible weld thinning and kissing bonds were observed in the joints macrostructure, mainly due to low tool pressure and high fluidity of the molten composite inside the SZ. Since keyhole at the end of joint line cannot be straightforwardly
prevented, it should be removed from the end of joint after completion. Formation of voids will be discussed in the following sections.

Although the tensile-shear strength of joints at various transverse speeds made by the conventional tool without rotary/stationary holder was relatively low (< 180 N), the most negative issue in these joints was the appearance. Unsuitable appearance of the joints was principally due to the protrusion of the mixture of composite melt and aluminum pieces from the SZ as a result of the tool pressure behind the moving tool. Figure 3a–c schematically show the joint made by the conventional FSJ tool and the joint cross- and longitudinal sections, as well as the flow of composite in SZ which leads to protrusion. This phenomenon led to formation of large voids in the SZ. From the mechanical strength point of view, formation of voids and discontinuities in the SZ which act as weak spots during the tensile-shear test may be responsible for low joint strength.

Figure 4 also shows that the amount of protrusion increased by reduction of the tool transverse speed. At lower transverse speeds, both heat input and joining time are higher. In other words, decreasing the tool transverse speed increases the frictional heat generation as well as the time needed for the tool to travel across the sample to complete
Fig. 4 Appearance (top view) and cross section of the joints made using the conventional tool without rotary/stationary holder at the transverse speeds of a 50 mm/min, b 75 mm/min, c 100 mm/min, d 125 mm/min, and e 150 mm/min.
the joint [3, 33]. Figure 5a gives variations in the temperature of a point in the middle of SZ (marked as thermocouple in Fig. 1) at different tool transverse speeds. During the FSJ process, the experienced peak temperature at all tool transverse speeds passed the melting point of the polypropylene (166 °C, measured by DSC analysis), resulting in melting and flow of the polypropylene in the SZ. Higher experienced temperatures at lower tool transverse speed further decreased the viscosity of the polypropylene melt which in turn facilitated polymer flow and led to more protrusion of the composite melt from the SZ. Moreover, longer joining time at lower tool transverse speeds promoted the protrusion.

Figure 5b shows the DSC analysis results of the base composite and samples from stirred zone at various tool transverse speeds. Polymers subjected to elevated temperatures may become degraded and decomposed. Change in the melting temperature (measured by DSC analysis) can show the extent of thermal damage. In fact, thermal damage usually means reduction in the length of polymer chains. In thermoplastics, this length reduction leads to a decrease in the melting temperature [34, 35]. As shown in Fig. 5b, melting temperature of the composite was slightly decreased after joining, an evidence for the thermal damage. Additionally, higher heat input (lower tool transverse speed) led to further decrease in the melting temperature (by ~5 °C at tool transverse speed of 50 mm/min). This can be justified by the higher peak temperature (Fig. 5a) and extent of thermal damage experienced by the composite inside SZ at lower transverse speeds.

In order to improve the joint appearance and strength, a rotary/stationary holder (Fig. 2) was used to apply pressure on the SZ just after passing the tool shoulder. As shown in Fig. 3d–f, this rotary/stationary holder imposed a forging force on the SZ, keeping the mixture of composite melt and aluminum pieces inside the SZ until solidification of the composite. Therefore, no composite melt protrusion occurred during the FSJ process. Indeed, the rotary/stationary holder promoted formation of a uniform and smooth weld profile and resulted in a satisfactory joint appearance (Fig. 6). Preventing composite melt protrusion also hindered formation of voids in the joint, since none of the stirred material was allowed to exit the SZ. As a result, joints made using the rotary/stationary holder showed superior load bearing capacity (by ~45 to ~220% at various tool transverse speeds), due to the improved integrity of the joints.

3.2 Effect of the tool transverse speed on joint macro/microstructure

Variation of the tool transverse speed had two effects on the joint profile and microstructure of the SZ:

(i) One of the important features observed in the cross-section of the joints in Fig. 7 was the anchor-like pieces of aluminum penetrated inside the composite substrate. These anchors were formed on both advancing side (AS) and retreating side (RS) of the joint. Aluminum anchors played a key role as the mechanical locks between the aluminum and polymer base materials. In fact, the load bearing capacity of the joints is mostly determined by the presence and the average size of these anchors at the joint cross-section. Table 3 presents the average cross-sectional area of the anchors (at both advancing and retreating sides of the joints) measured using an image analysis software. The anchor size increased with tool transverse speed from 50 to 100 mm/min. This may be related to the lower heat generation with enhancement of the tool transverse speed because of shorter friction time between the tool and the substrates. Lower heat reduces the thermomechanical effect and crushing impact imposed by the rotating pin in the SZ resulting in the formation of larger anchors. However, Figs. 6 and 7 and Table 3 show that the average size of the anchors did not increase.
Fig. 6 Appearance (top view) and cross-section of the joints made using the rotary/stationary holder at the tool transverse speeds of a 50 mm/min, b 75 mm/min, c 100 mm/min, d 125 mm/min, and e 150 mm/min.
significantly while transverse speed increased from 100 to 150 mm/min.

(ii) Increasing the tool transverse speed to higher than 100 mm/min (125 and 150 mm/min) led to the formation of voids near the aluminum base metal/SZ interface (Figs. 6 and 8). Voids in the stirred zone of the joints made by the rotary/stationary holder may be formed due to two phenomena: insufficient material flow and decomposition of the composite matrix.

**Table 3** Anchor size with the tool transverse speed

| Tool transverse speed (mm/min) | 50 | 75 | 100 | 125 | 150 |
|--------------------------------|----|----|-----|-----|-----|
| Anchor cross section area (mm²) | 0.024 | 0.041 | 0.077 | 0.080 | 0.081 |

**Fig. 7** Microstructure of joint and shape and size of anchors formed at aluminum/SZ interface at tool transverse speed of a 50 mm/min, b 100 mm/min, and c 150 mm/min

![Fig. 7](image1)

**Fig. 8** Aluminum/SZ interface at a 50 and b 150 mm/min transverse speeds and formation of voids at higher speeds

![Fig. 8](image2)
Since voids were formed in the joint with the two highest tool transverse speeds (i.e., 125 and 150 mm/min, equivalent to the lowest heat inputs), the reason for the void formation at the Al/SZ interface is insufficient material flow. In other words, voids are not caused by decomposition of the polypropylene, since joints with higher process heat input and temperature and consequently, higher risk of polymer decomposition, were free of voids.

Figure 9 presents map of the elemental distribution at the aluminum/stirred zone interface. On the aluminum surface, there was a narrow layer with higher concentration of oxygen, an evidence for presence of the aluminum (and/or magnesium) oxide. Covalent bonds or van der Waals attraction can be formed between this oxide layer and carbon and hydrogen from the polymer chains in the SZ which may enhance adhesion between them. Other researchers have also reported formation of Al₂O₃ at the aluminum/polymer interface and formation of hydrogen bonds between oxygen from the oxide layer and hydrogen from the polymer chains [23, 33]. Additionally, Meng et al. [36] reported that a porous oxide layer created intentionally on the aluminum surface before joining (for example using chemical or electrochemical treatments) was more effective in formation of secondary bonds. In other words, the melted composite may penetrate into the porous oxide layer and create the substantial bonds.

Figure 10 presents SEM image and linear EDS analysis of the interfacial layer at the aluminum/SZ interface. Linear EDS analysis again shows high concentration of oxygen atoms at the interfacial layer promoting chemical attraction as mentioned before. The SEM image, however, shows that some cracks were formed at the oxide layer as well as the oxide layer/aluminum interface. Presence of cracks is an indication of poor interfacial joining, and again emphasizes on the fact that mechanical locks are the main joining mechanism in the metal/polymer friction stir joints [36].

3.3 Fracture mode and mechanical performance of joint

Figure 11 shows the fracture location and the morphology of the fracture surface at various tool transverse speeds. In all of the joints, fracture occurred from the interface of the aluminum base sheet and stirred zone. Since the weakest point of the joint is the interface of the aluminum base sheet and stirred zone (which is only held together by the weak covalent and/or hydrogen bonds between the oxide layer and polymer in the stirred zone), the crack initiates and propagates from this location. The mechanical locks (anchors) at the interface are the primary load bearing parts, and the final fracture occurs when the crack propagates inside the anchors. From what was mentioned above, the fracture surfaces were composed of two distinct regions:

(i) a rather flat, smooth surface in which separation occurred from interface of the oxide layer and the stirred zone, and
(ii) A coarse, rough surface including the fractured anchors.

At the former region (flat surface), no indication of plastic deformation was observed. This may be related to the lack of strong bonds in this region. Indeed, weak covalent and/or hydrogen bonds are easily separated by the tensile-shear
force without plastic deformation in the materials. At the latter region, however, the tearing and fracture of the anchors occurred (Fig. 11). It is worth mentioning that in a lap joint configuration, there is always an intrinsic notch around the joint region at the base metals’ interface which acts as a stress concentration site and promotes crack growth and fracture of the joint with a relatively low elongation. This may be responsible for brittle fracture of the aluminum anchors. However, application of the rotary/stationary holder does not encourage the creation or propagation of cracks. On the contrary, as comparison of Fig. 4 with Fig. 6 shows, rotary/stationary holder improves the integrity of the joints and decrease formation of voids and crack by preventing the composite melt protrusion from the SZ (especially at low tool transverse speeds).

Figure 12 presents the peak load (maximum load bearing capacity) of the joints made using the conventional tool without the rotary/stationary holder and with the rotary/stationary holder at various tool transverse speeds. There are two factors contributing to the strength of aluminum-polymer joints made by friction stir processing:
• Mechanical locks: the first factor determining the joint strength is the size of aluminum anchors penetrated into the lower composite sheet. Size of the aluminum anchors was found to be increased with enhancement of the tool transverse speed (Fig. 6 and Table 3) leading to the stronger mechanical lock between the aluminum and composite sheets.

• Chemical bonds: the second factor is the covalent and hydrogen bonds and the integrity and continuity of the aluminum/SZ interface. Although the nature of the bonds is not affected by the process heat input, voids and discontinuities are formed at the aluminum/SZ interface (Figs. 6 and 8) when joints are made with high tool transverse speeds (125 and 150 mm/min).

Figure 12 shows that all the samples joined using the conventional tool without the rotary/stationary holder had considerably lower tensile-shear peak loads compared to the samples joined using the rotary/stationary holder. This may be attributed to the protrusion of the melted composite from the SZ and formation of voids and crack inside it. However, using the rotary/stationary holder led to stronger joints, since it effectively prevented formation of defects at the joint interface due to the composite melt protrusion.

Peak failure load of the samples joined using the rotary/stationary holder improved up to ~25% (from 255 to 315 N) as the tool transverse speed increased from 50 to 100 mm/min. Given two factors contributing to the strength of joints, this can be due to increase in the anchor size which strengthens the mechanical locks and increase the load bearing capacity of the joints (Fig. 6 and Table 3). However, by increasing the tool transverse speed to 125 and then 150 mm/min, the peak load decreased (from 315 to 283 N and from 283 to 188 N, respectively). This trend may be caused by the formation of voids and defects at the joint interface. In fact, while enhancement of the tool transverse speed from 100 to 150 mm/min did not lead to significant increase of the aluminum anchors size (Table 3), some defects were formed at the aluminum/SZ interface as mentioned in Sect. 3.2. These defects (i.e., large voids and discontinuities at the aluminum/SZ interface) act as crack initiation and stress concentration sites. Thus, negative effect of these defects led to deterioration of the load bearing capacity with enhancement of the tool transverse speed. Therefore, the tool transverse speed of 100 mm/min was considered the optimum transverse speed.

4 Conclusions

High mechanical strength and suitable appearance are two challenges in friction stir joining of aluminum alloys to polymers. In this research, an alternative tool was introduced for lap joining of Al 5052 to polypropylene/talc composite sheets using friction stir joining process. The main conclusions taken from this research are as follows:

• Protrusion of the mixed molten polymer and aluminum particles from the stirred zone behind the process tool adversely affected both the joints appearance and strength.

• Use of a novel rotary/stationary holder around the joining tool improved the appearance and tensile-shear strength of the joints by preventing the polymer melt and aluminum particles from exiting the stirred zone. Tensile-shear strength of the joints increased (by ~45 to ~220% at various transverse speeds) using the rotary/stationary holder.

• Three microstructural features influenced the joint strength: (i) formation of mechanical locks by penetration of the anchor-like aluminum pieces in the composite substrate, (ii) development of the oxide layer at the joint interface, and (iii) presence of large defects near the aluminum/stirred zone interface.

• The load bearing capacity of the joints first increased up to 315 N due to the formation of larger anchors, but then decreased with enhancement of the tool transverse speed (from 100 to 125 mm/min and from 125 to 150 mm/min) because of the formation of large voids at the aluminum/stirred zone interface.

• Interface of the aluminum base sheet and the stirred zone was the weakest point of the joint and all joints failed from this point during the tensile-shear test. Consequently, local strengthening of the aluminum/stirred zone interface, for example using the second pass of friction stir process, may improve the aluminum/composite joint strength.
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