Polymeric materials, despite being thermal insulators, are now being welded using different welding techniques. In the current work, the feasibility of the friction stir welding (FSW) process on 16-mm-thick Nylon 6 plates was studied. The effects of rotational speed on the weld quality were investigated by the temperature development, micromechanical properties, crystallization growth, and fracture analysis of the joints. Results showed the dependence of temperature and tensile values on rotation rates was insignificant. However, appearance of considerable defects at higher rotation rates, observed in visual and microscopic analysis, indicated that Nylon 6 is weldable only at lower rotation rates due to its low melt viscosity. Moreover, identical fracture locations during tensile tests revealed that the interface of weld zone on the retreating side was the weakest part of the joint. It can be attributed to the lack of bonding at the interface of the weld zone on retreating side and relatively low crystallinity in the retreating side region. Due to different rheological and physical properties of polymers than metals, the flow phenomenon in Nylon 6 was found to be different from that of metals, resulting in a distinct isolated pin plunged zone.

KEYWORDS
- Friction Stir Welding • Nylon 6 • Polymer • Material Flow • Threaded Pin
of the tool or bobbin tool may result in excess heat due to the dual friction on upper and lower surfaces of the workpieces, eventually increasing the flash. The preweld heating, performed by Aydin (Ref. 7) on 4-mm-thick ultra-high molecular weight (UHMW) polyethylene sheets, may not be suitable for polymers with low melt viscosity and can intensify the flash formation due to increasing heat.

The influence of pin shape, studied on PE sheets by Bagheri et al. (Ref. 10) and Ahmadi et al. (Ref. 9) made them conclude that the truncated cone pin has the highest tensile strength as compared to other pin shapes. However, due to large rheological and property differences among polymers, one pin profile cannot be assumed as optimum for all polymers. Another tool consisting of the “hot shoe” was investigated by Bagheri et al. (Ref. 10) on ABS sheets. The main reason to use this shoe was to heat the polymer through the shoulder during the welding process. Their results were comparable with the work carried out by Mendes et al. (Ref. 11), who used a stationary shoulder tool on the same material without external heating. However, squeezing out of the plasticized material below the shoulder, particularly in low melt viscosity polymers still remained a problem.

Inaniwa et al. (Ref. 12) and Panneerselvam et al. (Ref. 13) joined Nylon 6 sheets with thicknesses of 5 and 10 mm, respectively. In their studies, they eliminated the primary heat source by keeping a small opening between the shoulder and workpiece top surface. Comparing their approaches, it was found that Panneerselvam et al. (Ref. 13) joined Nylon 6 at a quite higher revolution pitch (ratio between welding speed and rotation rate) compared to Inaniwa et al. (Ref. 12) work. However, considering the Nylon 6 properties’ especially low melt viscosity behavior, it is believed that higher revolution pitch will produce enormous flash. Flash formation has been reported by Panneerselvam et al. (Ref. 13) as well. On the other hand, the gap between shoulder and workpiece will certainly lead to the formation of a crown above the weld zone.

Material flow, due to its direct relation with weld quality, has been thoroughly investigated on metals by various means. Lorrain et al. (Ref. 14) and Li et al. (Ref. 15) used foil insert technique, Edwards and Ramulu (Ref. 16) used powder as a tracer material, Seidel and Reynolds (Ref. 17) utilized the marker material insert technique, whereas Colligan (Ref. 18) inserted small steel balls in aluminum to study material flow. Colligan (Ref. 18) concluded that material moved behind the pin and deposited on the retreating side. In another study on aluminum 6061, Guerra et al. (Ref. 19) reported different flow on advancing side (AS) and retreating side (RS). Seidel and Reynolds (Ref. 17) observed that the majority of the material in the weld nugget simply moved around the pin and displaced behind the pin.

The material flow in polymeric materials has been studied on poly methyl methacrylate (PMMA) by Simões et al. (Ref. 20). They compared their flow study with the Arbegast (Ref. 21) flow model and observed that the pin-affected zone remained isolated and straight along the pin. Their results showed no cross flow from the weld zone to the base material. Similarly, a clear distinction between the shoulder-affected zone and pin-affected zone could be seen.

Current work involved studying the process on 16-mm-thick Nylon 6 plates by investigating the temperature development, micromechanical, and thermal properties of the joint. With the aim to reduce the flash formation, a small-diameter shoulder tool with right-hand threaded pin was used. Moreover, marker material insert technique was utilized to examine the flow phenomenon and stirring uniformity in the weld.

Materials and Method

In the present investigation, 180-mm-long weld passes were made on 16-mm-thick Nylon 6 (Polyamide-6) plates in butt joint configuration at room temperature. Bridgeport VMC 2216 CNC machine was utilized for FSW of specimens welded at a 0-deg tilt angle and FSW-TS-F16 FSW machine was used to prepare welds at a 3-deg tilt angle.

The FSW tool used in this study was machined from H13 tool steel rod — Fig. 1. The pin of the tool was made right-hand threaded for uniform stirring, while rotating in a clockwise direction (Ref. 13). The tool was heat treated before being used for welding and, therefore, its hardness was increased to 56 HRC from 24 HRC.

Rotational speed, due to its main contribution in the FSW process, has been studied (Ref. 22). It was there-
fore varied between 300 and 1000 rev/min. The other parameters, such as feed rate or welding speed, dwell time, and tilt angle, were kept constant and were selected based on previous studies and preliminary tests (Refs. 12, 13). Feed rate in polymers was usually kept low so that the material in front of the pin could get sufficient time to plasticize. Parameters used in this study are shown in Table 1. A tilt angle of 3 deg, reported as an optimum FSW angle for HDPE by Bozkurt (Ref. 22), was also used at the optimum rotation speed. The optimum rotation speed, used in the present work, was obtained from performed tests.

Furthermore, a K-type thermocouple was also placed at 1 mm below the pin tip to estimate the weld zone (WZ) temperature at each rotation speed — Fig. 2. In all experiments, a clockwise rotating tool was plunged into the workpiece with a 10-mm/min plunge rate to the depth equal to the pin length. All the welding operations were performed at room temperature.

Cross sections of weld specimens, perpendicular to welding direction, were made and were observed visually. The mechanical strength of the joints was analyzed by tensile testing on specimens that were obtained perpendicular to the welding direction. The tensile tests were performed in accordance to ASTM Standard D638-10 using a Zwick-Roell UTM machine at the crosshead speed of 1 mm/min. A schematic of the D638-10 specimen is shown in Fig. 3. Weld zone and fractured surfaces during tensile tests were analyzed by scanning electron microscope (SEM). In order to determine the crystalline content in the WZ, a differential scanning calorimeter (DSC) test was carried out using a Perkin-Elmer differential scanning calorimeter at a heating rate of 10°C/min. Furthermore, material flow during the welding process was studied by marker material insert technique at achieved optimum parameters. Subsequently, due to the marker material’s difference in color from the base material, its movement was visually analyzed by different sectioning of the specimens.

Results and Discussion

Morphological and Micrographic Analysis

In order to observe any visual defects, friction stir welded specimens were cross-sectioned perpendicular to the welding direction. Figure 4A–E shows the different morphologies of top surfaces of the welded specimens.

Although the flash formation can be observed in all specimens, Fig. 4A, B specimens show comparatively less flash formation. It is also noted that the flash in Fig. 4B is on retreating side (RS). For this reason, it is believed that the temperature on the RS is always higher than the advancing side (AS), which leads to the formation of flash on the RS (Ref. 23). Further increase in rotation speed to 500 rev/min resulted in

<table>
<thead>
<tr>
<th>Tilt Angle (Deg)</th>
<th>Feed Rate (mm/min)</th>
<th>Dwell Time (s)</th>
<th>Rotation Rate (rev/min)</th>
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</thead>
<tbody>
<tr>
<td>0</td>
<td>25</td>
<td>15</td>
<td>1000</td>
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<tr>
<td>0</td>
<td>25</td>
<td>15</td>
<td>500, 400, 300</td>
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<tr>
<td>3</td>
<td>25</td>
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![Fig. 4 — Cross sections of specimens welded at the following: A — 300 rev/min with 0-deg angle; B — 400 rev/min with 0-deg angle; C — 500 rev/min with 0-deg angle; D — 1000 rev/min with 0-deg angle; E — 300 rev/min with 3-deg angle.](image-url)
higher heat input, leading to excess flash in the form of bubbles, which appeared at some intervals — Fig. 4C. 300 rev/min with a 3-deg-angle specimen also showed bubble-like flash, which indicates that tilting of the angle at 3 deg generated high heat during the process — Fig. 4E. It may be due to the tilted tool trailing edge of the shoulder that applies higher compressive load in the surface of material increasing friction and resulting in higher heat generation.

The formation of a crown-like shape was also observed in all specimens except the 1000-rev/min specimen. Crown peak was less in 300 rev/min with 0-deg specimen angle compared to 400, 500, and 300 rev/min with 3-deg-angle specimens. A specimen welded at 1000 rev/min showed a different morphology — Fig. 4D. Excess semimolten Nylon 6 squeezed out of the WZ, and after swirling around the shoulder, appeared to have solidified on the shoulder. As a result, the WZ decreased in thickness, which would eventually reduce the tensile strength. It is believed the appearance of weld defects at higher rotation rates is due to overheating generation, which eases the plasticized polymer to flow out.

Low magnification SEM images of weld zones, taken on cross sections, are shown in Fig. 5. The specimen welded at 1000 rev/min was excluded from further results and discussion due to its poor weld quality. The weld in Fig. 5A, which was made with the lowest rotation speed of 300 rev/min with 0-deg angle, presented an excellent superficial appearance, unlike welds produced at higher rotation rates (Fig. 5B–D), which showed different defects, likely caused by excess heat input. However, a minor defect in the form of improper bonding was observed at the bottom of the RS interface. A specimen welded at 400 rev/min rotation rate showed various small porosities with improper bonding on the RS border line — Fig. 5B.

Likewise, increasing the rotational speed to 500 rev/min resulted in a major defect called tunnel defect with poor bonding on the RS border line — Fig. 5C. Considering the good weld appearance of 300 rev/min with a 0-deg-angle specimen, and in order to remove the minor weld defect at the bottom, it was investigated using a 3-deg tilt angle as well. However, the micrograph result showed a large cavity with a slight improper bond on the RS in Fig. 5D.

Moreover, weld zones were also analyzed at higher magnification. Micrographs shown in Fig. 6A–D are the higher magnifications of Fig. 5A–D, respectively. It is clear rotation speed has a significant effect on the microstructure of the welds. The 300 rev/min with 0-deg angle specimen in Fig. 6A showed uniform and perfect surface quality, whereas the specimens at higher rotation rates or a 3-deg tilt angle showed a relatively rough surface — Fig. 6B–D. It is assumed that fracture is easy to occur in rough surface compared to smooth surface, as it can provide stress concentration points.

From the above detailed description, it can be deduced that, due to low melt viscosity compared to other polymers, such as ABS (Ref. 11), PE (Ref. 22), PP (Ref. 24), and PMMA (Ref. 25), Nylon 6 is weldable only at lower rotation speeds.

**Tensile Test Results**

Figure 7 exhibits the peak stress values of tensile specimens, obtained from stress strain curves. The trend indicates the increase in tool rotation speed leads to a decrease in tensile strength. However, considering the visible aforementioned defects, Fig. 7 shows the difference in strength values is not that large. The tensile strength obtained at 500 rev/min is also comparable to that of the specimen at 400 rev/min, even though it comprises tunnel defect in it. Moreover, the strength of each specimen, in terms of value, was quite less than that of the base material. The highest tensile strength, obtained at 300 rev/min, 0-deg tilt angle, was 27.21 MPa, which corresponds only to about 32% of base material. At the same rotational rate when the angle is tilted to 3 deg, the strength decreased to its lowest value of 18.08 MPa.

Compared with Panneerselvam et al. (Ref. 13) and Inaniwa et al. (Ref. 12), who studied 10- and 5-mm-thick Nylon 6, respectively, almost similar results were found. Although Nylon 6 FSW results are lower in tensile strength compared to other polymers, such as PE (Ref. 4), ABS (Ref. 10), and polypropylene (PP) (Ref. 24), it is believed that this process, due to its certain advan-
tages over other techniques, can be suitable for this material.

Fracture Analysis

The fractured area in any place of the welded joint is a direct indication of the weakest part of that joint. In the present investigation it was noted that all specimens of each set of parameters exhibited identical fracture location, which is at the interface of the WZ on the retreating side (IW-RS). However, interface of the WZ on the advancing side (IW-AS) remained intact. One fractured specimen for each rotation rate is shown in Fig. 8. It is also important to note here the specimen welded at 500 rev/min (Fig. 8C) also showed fracture at the IW-RS, despite the fact it contains a tunnel defect. It indicates that the IW-RS is weaker than the tunnel defect.

Inaniwa et al. (Ref. 12) and N. Mendes et al. (Ref. 11) also observed the same fracture locations in their study on FSW of different polymers. This preferred fracture location in these specimens can be related to the formation of flash preferentially on the retreating side. In general, flash formation causes the lack of material, which ultimately results in cavities and blow-holes. In addition to it, other phenomena in the WZ make the RS weaker than the AS include higher temperature on the RS (Ref. 23), low shear velocity on the RS (Ref. 12), and difference in flow on both sides (Ref. 10). Scanning electron microscopy results of fractured specimens observed toward the WZ, shown in Fig. 9, exhibit the same fracture phenomenon in all specimens.

Temperature Analysis

Temperatures measured at 1 mm below the pin tip are shown graphically in Fig. 10. An increase in temperature with the increase of rotation speed is obvious and can be observed in the graph. Although the temperature differences at low rotation rates are not significant, noticeable differences in weld quality were observed. Cavities, tunnel defect, and smoke during the process were seen above 400 rev/min rotation speeds. A maximum temperature of 167°C at 1000 rev/min showed large amounts of flash formation with the emission of smoke. Smoke is a combination of different volatiles, majorly carbon dioxide (CO₂), water (H₂O), and ammonia (NH₃), evolved due to endothermic reaction during the FSW process. Evolution of volatiles is directly linked to the weight, suggesting a decrease in polymer weight (Ref. 26). Therefore, it is believed that at relatively higher rotation rates, a small decrease in weight also occurred.

Fig. 7 — Effect of rotational speed on tensile strength of Nylon 6 FSW specimens.

Analysis of Crystallinity

In order to analyze the postweld thermal conditions of the joint and to investigate the reason of identical fracture locations, the degree of crystallinity of different sections of the WZ was analyzed using DSC curves. The specimens, for this purpose, were taken from the base material (BM), weld center (WC), AS, and RS. The degree of crystallinity of any polymer has a direct relation to its mechanical and physical properties. Increase in the degree of crystallinity has shown increase in tensile strength, stiffness,
yield point, and hardness, but a reduction in impact strength (Refs. 28–30).

Figure 11 shows the DSC curves of a specially chosen 300 rev/min with 0-
deg-angle specimen, due to its comparatively good micro-mechanical results. The peak value of the curve gives the melting enthalpy ($\Delta H_m$) of the polymer, which is directly proportional to the crystallinity according to this formula (Ref. 31)

$$W_c = \frac{\Delta H_m}{\Delta H^{100\%}_m} \times 100\% \quad (1)$$

where $\Delta H_m$ is the enthalpy of fusion and $\Delta H^{100\%}_m$ is the enthalpy of fusion of 100% crystalline Nylon 6, of which the value is 230 J/g (Ref. 32).

Keeping in view Equation 1 and Fig. 11, it can be concluded the crystallinity of the specimen is reduced in the WZ compared to the base material. It reduced further in the RS and became lowest in the WC by making the AS the comparatively highest crystalline region in the WZ. However, low crystallinity along with some defects on the borderline of the RS made the RS the preferred fracture location. It is also important to mention here the reduction in crystallinity is basically linked to the rapid cooling of the material (Ref. 32). In this case, the cooling rate for the whole joint was the same, as postweld specimens were placed in an open environment at room temperature. Therefore, it is believed that it is mainly the temperature differences at different regions of the WZ that lead to the cooling difference and, therefore, the difference in crystallinity.

**Material Flow during Friction Stir Welding**

Material flow during the welding process was studied by employing a 1.5-mm-thin ABS sheet as a marker material. For this purpose, specimens were friction stir welded at optimum parameters of 300 rev/min rotation speed, 0-deg tilt angle, and 25-mm/min feed rate, selected based on previous results. The set of parameters is shown in Table 1. Postweld specimens were cut in different sections, such as longitudinal section (parallel to welding direction), vertical cross section (perpendicular to welding direction), and horizontal cross section, to visually analyze the displacement of marker material in x, y, and z directions. The sectioning scheme is also shown in Fig. 12.
Figure 13A shows the specimen before welding with marker material in the longitudinal direction (parallel to welding direction). After welding, vertical cross sections of the specimen were made to observe the flow in x-direction, which is perpendicular to the welding direction (WD). It can be clearly seen in Fig. 13B that thin marker material, when placed on the RS (I), spread all over the welding zone, equivalent to pin diameter. Similarly, in Fig. 13C, marker material placed on AS (II) stirred and spread in complete welding zone. This spreading indicates a uniform stirring, either marker material is on the AS or the RS. However, in both cross sections it can be observed that marker material at the top of the specimen is not well stirred and positioned toward the AS. It is believed this unstirred area is due to a small, unthreaded part of pin near the shoulder. Furthermore, it is also observed the depth of the WZ is equivalent to the pin plunged length. It shows there was no cross flow from the plunged area to the unplunged zone at the bottom of the pin. A similar phenomenon was observed on the adjacent right and left sides of the plunged area. This restriction of WZ within the plunged area is also mentioned by Simões and Rodrigues (Ref. 20) in their study of PMMA.

In order to observe the y-direction flow (parallel to WD) of the specimen, marker materials were placed transversely (perpendicular to WD) on the AS (I), weld interface (II), and RS (III), as shown in Fig. 14A. After welding, horizontal sections were prepared. It is clear from Fig. 14B–D marker material after stirring was displaced behind the pin. The maximum displacement measured in Fig. 14C, D was remarkably very long, 11 mm, whereas the diameter of the pin is 7.5 mm. However, distribution of marker material (shown in Fig. 14C, D) was uniform, but narrowing of marker material at the end was observed. It is believed the farthest narrow part is squeezed and extruded by the pin. This extrusion phenomenon is similar to Colligan’s (Ref. 18) material flow study on FSW of aluminum alloy, in which he considered the welding process due to stirring and extrusion. As the material of the AS in Fig. 14B is prone to flow on sides, it can be said the vacant sides at the end in Fig. 14C, D can be filled by material from the AS. Thereby, it covers a complete welding zone and leaves no defects.

In order to understand the complete flow during welding, flow in the z-direction was also observed. For this purpose, marker materials were placed at bottom, middle, and top of the AS and RS. It is shown in Fig. 15A, B, respectively. After welding, longitudinal sections of welded specimens were made to observe vertical movement. These are shown in Fig. 15C, D. It is clear from the sections that material at the bottom expanded up to the surfaces of both specimens. A similar case was observed for middle marker materials. Marker material at the top expelled out from the specimen and resulted in formation of flash. No difference in this upward movement of marker materials was found either on the AS or RS of specimens. A large, vertical movement of material during welding was also observed by Guerra et al. (Ref. 19), Li et al. (Ref. 15), and Seidel and Reynolds (Ref. 17) in their study on aluminum.

Conclusions

Systematic work was carried out on the friction stir welding of 16-mm-thick Nylon 6 plates using a threaded pin tool with a small-diameter shoulder. Based on the aforementioned results and discussion, the following conclusions can be made:
Nylon 6, due to its low melt viscosity, is weldable only at lower rotation rates. Therefore, a 300 rev/min rotation speed and 25-mm/min feed rate has given comparatively good weld results at 0-deg tool angle.

At higher rotation rates, squeezing out of excess plasticized material and defects formation in the weld zone were observed.

A small-diameter shoulder, on the other hand, reduced the amount of flash by reducing the primary heat.

The processing temperatures, measured 1 mm below the pin plunged zone, were quite below the thermal degradation temperature of Nylon 6 (350°C). Thus, it is assumed the Nylon 6 in the stirring zone did not undergo extreme thermal degradation, although minor reduction in molecular weight due to smoke evolution is believed to have occurred.

- The tensile strength of all joints was quite lower than that of the base material.
- As the result of microstructure observation in the weld zone regions, relatively smooth and uniform microstructure was observed at the optimum set of parameters.
- DSC results showed the crystallinity of the weld zone decreased compared to base material. Moreover, the retreating side compared to the advancing side was found to have low crystallinity.
- During tensile tests, all specimens fractured at the interface of the weld zone on the retreating side, and low crystalline content in the retreating side region.

- Material flow examination revealed large (more than pin diameter) backward displacement of plasticized material. However, overall a uniformly mixed distinct isolated pin plunged zone was found.

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