

STUDY ON HARDNESS AND MICRO STRUCTURAL CHARACTERIZATION OF THE FRICTION STIR WELDED NYLON 6 PLATE

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ABSTRACT

Friction stir welding (FSW) is a solid-state method of joining thermoplastic materials. FSW process parameters such as tool rotation speed, welding speed, and tool pin profile etc play a major role in deciding the weld quality. In this study, an attempt has been made to understand the mechanism of FSW and the role of tool pin profile, rotation speed and welding speed in Nylon 6 plates. Experiments were performed at rotational speed of 600-1200 r/min, Welding speeds of 10-40 mm/min, and FSW tool pin profiles of Triangular, square, Threaded and Grooved pin profiles.

This has been done by understanding the material flow pattern in the weld regime. Optical microscopy was used to evaluate the microstructural characteristics and Rockwell hardness is observed in weld joints. Weld zone microstructure were investigated using different images of optical microscopy. The micro structure and Rockwell hardness of the welded region was created by Grooved pin profile with welding speed of 10 and 20 mm/min and rotation speed of 800 and 600 r/min identified as correct FSW parameters to avoid defects in Nylon 6 plates.

KEYWORDS: Feed, FSW, Hardness, Microstructure, Nylon 6, Pin Profile, Speed, Weld Zone

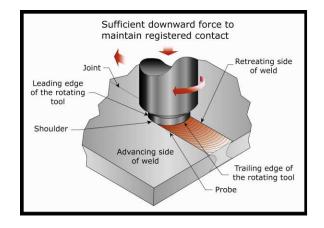
INTRODUCTION

FSW is a novel solid-state welding process for the joining of metallic alloys and composites; initially developed by the welding institute (TWI) in 1991. It is nowadays a well known welding techniques and it has been widely investigated for several alloys and polymers. It has the advantage of non-consuming fabrication of continuous linear welds. The most common form of weld joint configuration and is energy efficient, environmentally friendly and versatile [1].

This process is illustrated in fig.1.Won et al characterized and compared the microstructures of copper base metal with those of the residual FSW zones using optical metallography (OM) and transmission electron microscopy (TEM). Microhardness profiles through the weld zone and corresponding tensile strength test data were also correlated with these microstructures.

Hardness variation also existed in the stir zone from the upper to the lower region with thermal and mechanical conditions [2]. Vickers hardness tests were conducted on the welds to evaluate the hardness distribution in the thermalmechanical affected zone, heat affected zone and the base metal. The temperature on the Advancing Side (AS) was slightly higher than those on the retreating side (RS). But, there is no significant difference in hardness between the AS&RS [3]. Chowdhury et al explained the hardness of the stirred zone for 10 to 30mm/sec feed rate of 1000 to 2000rpm in the magnesium alloys [4].

Sauvage et al explained the hardness based only on composition of magnesium, silicon and silicon carbide. They had not explained thoroughly about the changes of hardness in the nugget, RS as well as AS for different compositions [5]. Negin's et al reviewed the current techniques used for direct bonding of polymers, with a focus on thermoplastics [6].





Most of the literature on FSW focuses on aluminum, magnesium, Steel, dissimilar material and their alloys; however, recently interest has grown in applying this technique to the joining of thermoplastic materials. Currently, there are few investigation related to thermoplastics in friction stir spot welding [7-9] and FSW process [10-12]. This paper addresses a report on the effects of process parameters such as rotational speed, welding speed and tool pin profile for nylon 6 plates were achieved using FSW. The effects of the process parameters on the weld region and hardness are measured and the microstructure of the weld zone is investigated.

Experimental Procedure

Friction stir welded joints of Nylon 6 plate, with a thickness of 10 mm and length and breath as 200x100mm were selected in the present study. FSW was carried out using CNC Vertical machining centre. The fixed non consumable mild steel pin tool with a nominal diameter of 6mm and shoulder diameter of 12 mm was used in the present investigation. In this study, four different tool pin profiles utilized to investigate the microstructure and hardness of welded region and AS as well as RS. The different FSW tool pin profiles are shown in the Figure 2. Design of experiments (DOE) is a powerful analysis tool for modeling and analyzing the influence of process variables over some specific response variables, which is an unknown function of these process variables. The most important stage in the DOE lies in the selection of the control factors. As many as possible should be included, so that it would be possible to identify non-significant variables at the earliest opportunity.

Taguchi defines the quality of a product, in terms of the loss imparted by the product to the society from the time the product shipped to the customer. Taguchi creates a standard orthogonal array to accommodate this requirement. Depending on the number of factors and their level, an orthogonal array is selected by the investigator. In this experimental work, L'16 orthogonal array was utilized for the design of experiments. The designed process parameters and their levels are explained in the table 1. Based on this process parameters, Sixteen different welding conditions were applied to process the samples. Welding speeds were varied from 10 to 40 mm/min, and rotational speeds were varied from 600 to 1200 rpm.

Samples for microstructual characterization were taken cross section of welded region (parallel to the rolling direction). All samples were cut approximately 20mm length. The samples were then manually ground and polishing in the polishing machine. The polished samples were etched in O-xylene acid. Microstructural image were taken by the optical microscopy at a magnification of 100x, 200x and 400x. Rockwell hardness testing machine was used for hardness tests where a load of 100 kgf and 30 second duration time and ¹/₄" ball indenter were used. The tests were carried out at different distances (every 5mm upto 15mm) from the centre of nugget.



Figure 2: Different Tool Pin Profile Selected in this Investigation

| Table 1: D | Designed | Process | Parameters | and | their | Levels |
|------------|----------|---------|------------|-----|-------|--------|
|------------|----------|---------|------------|-----|-------|--------|

| S.No | Process Parameters | Level 1 | Level 2 | Level 3 | Level 4 |
|------|---------------------------|---------|---------|----------|------------|
| 1 | Pin profile | Square | Grooved | Threaded | Triangular |
| 2 | Feed rate mm/min | 10 | 20 | 30 | 40 |
| 3 | Rotating speed r/min | 600 | 800 | 1000 | 1200 |

| Run | Pin Profile | Feed Rate in mm/min | Rotating Speed in Rpm | | |
|-----|-------------|------------------------|--------------------------|--|--|
| 1 | Square | 10 | 600 | | |
| 2 | Square | 20 | 800 | | |
| 3 | Square | 30 | 1000 | | |
| 4 | Square | 40 | 1200 | | |
| 5 | Grooved | 10 | 800 | | |
| 6 | Grooved | 20 | 600 | | |
| 7 | Grooved | 30 | 1200 | | |
| 8 | Grooved | 40 | 1000 | | |
| 9 | Threaded | 10 | 1000 | | |
| 10 | Threaded | 20 | 1200 | | |
| 11 | Threaded | 30 | 600 | | |
| 12 | Threaded | 40 | 800 | | |
| 13 | Triangular | 10 | 1200 | | |
| 14 | Triangular | 20 | 1000 | | |
| 15 | Triangular | 30 | 800 | | |
| 16 | Triangular | 40 | 600 | | |

RESULTS AND DISCUSSIONS

FSW Region and their Microstructural Images of Nylon 6 Plate

Joints were successfully produced within the following ranges: spindle rotational speed of 600-1200 r/min, welding speed of 10-40 mm/min, and four different pin profiles such as triangular, square, grooved with square, and threaded pin. Based on the L'16 orthogonal array, the Nylon 6 plates were welded for sixteen different welding process parameters through shuffling of rotational speed, welding speed and tool pin profile in FSW process. L'16 orthogonal array are illustrated in table 2.

For different process parameters, the welded joint fabricated and compared in this investigation. The microstructural studies were important to analyze the defects of the welded region. So, the joint interface's cross section was taken the microstructural images by using the optical microscopy for 100x, 200x, and 400x magnification. Whatever welded region appeared for different conditions and their microstructure, those were explained in figure 3-18.

In fusion welding of metal alloys, the defects like porosity, hot cracks etc deteriorates the weld quality and joint properties. Usually, friction stir welded joints are free from these defects since there is no melting takes place during welding and the metals are joined in the solid state itself due to the heat generated by the friction and flow of metal by stirring action. However, FSW joints are prone to other defects like pin holes, tunnel defects, piping defects, kissing bond, cracks, etc due to improper flow of metal and insufficient consolidation of metal in the FSW region [13]. But, when the welded region's cross section was evaluated in nylon 6 materials, some cracks, pin holes, porosity, cavity and etc appeared in the welded region.

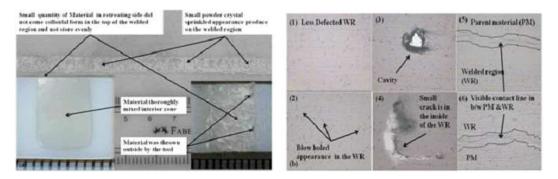


Figure 3: a & b Appearance and Microstructure of FSW Region for Square Pin at 600 r/min and 10 mm/min

The defects occurred in the welded region that was clearly explained in these figures (3-18) such as cavity, porosity, blow holes, unmolded materials, inclusions and etc. The contact line of the nugget material with AS as well as RS were visible for few designed process parameters. The microstructural images were taken in different places for different magnifications.

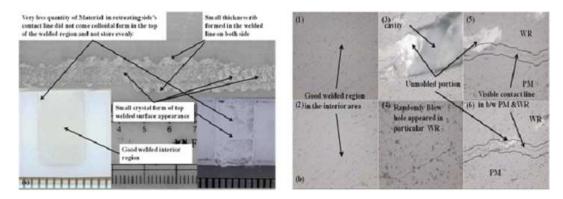


Figure 4: a & b Appearance and Microstructure of FSW Region for Square Pin at 800 r/min and 20 mm/min

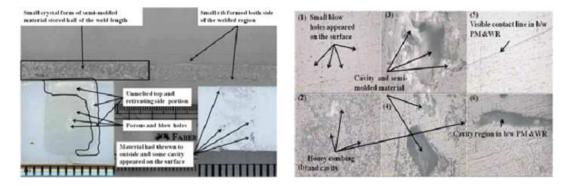
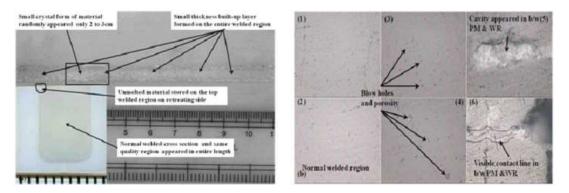


Figure 5: a & b Appearance and Microstructure of FSW Region for Square Pin at 1000 r/min and 30 mm/min

FSW Region and their Rockwell Hardness of Nylon 6 Plate

The hardness was measured across the weld at mid thickness region and 15mm from the joint interface for 5 mm interval using Rockwell hardness testing machine and the values are tabulated in table 3 and values are presented in figure 19. The hardness of the base material (nylon 6) is 93RH. The left side data mentioned the AS hardness and the right side data mentioned the RS hardness in the table 3 and figure 19.

Temperature is not the only factor for a successful FSW process. Other factors, such as welding speed, spindle rotational speed of the tool and tool pin profiles are all important factors for a successful FSW process. These factors influence the temperature distribution in the workpiece, and eventually, the control of the temperature becomes the key factor that affects the final properties of the weld [3]. The temperature changes were occurred due to the different process parameters that were focused in this hardness investigation. Normally, the temperature profiles on the AS are slightly higher than those on the RS. Those similar conditions occurred in hardness profiles of Nylon 6 material in FSW process.



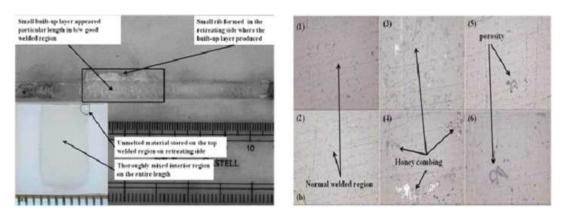


Figure 6: a & b Appearance and Microstructure of FSW Region for Square Pin at 1200 r/min and 40 mm/min

Figure 7: a & b Appearance and Microstructure of FSW Region for Grooved Pin at 800 r/min and 10 mm/min

Effects of FSW Tool Pin Profile on Nylon 6 Plate Joining

In this study, there are four different tool pin profiles such as triangular, square, threaded, and grooved pin profile were investigated. The figure 3-6 depicts the effects of the welded region and their microstructure for square pin profile in FSW process. Small quantity of material in RS did not come colloidal form in the top of the welded region and not stored evenly in the first condition as shown in the figure 3a. Small crystal form of material sprinkled appearance created on the joint interface. But, the interior material thoroughly mixed except the poor contact of the RS. That was focused in microstructure images as shown in the figure 3b. One or two cavities and two blow holes appeared in the welded region as shown in the figure3b. These similar conditions followed in the forth coming welded region. Small thickness rib formed in

the joint interface on both sides as shown in the figure 4a. The material had thrown to outside and some cavity appeared on the joint interface surface. Some visible porous were appeared in the welded region and the nugget region contact with the RS parent material was poor as shown in the figure 5a. Small crystal form of material randomly appeared only 2 to 3 centimeter on the middle of the joint interface (see figure. 6a). For square pin profile, the contact line of the nugget with RS was the major problem as shown in the figure 4b-6b.

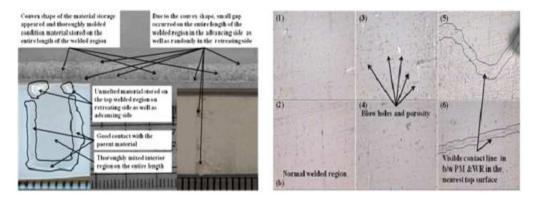


Figure 8: a & b Appearance and Microstructure of FSW Region for Grooved Pin at 600 r/min and 20 mm/min

The grooved tool pin profile for different process parameters were analyzed and the welded region with their cross section had explained in figure 7a-10a. The corresponding microstructure was analyzed together in figure 7b-10b. The small built-up layer appeared particular length in between good welded region for few centimeter and small rib formed in the RS where the built-up layer appeared. When the cross section was analyzed, the small unmolded material stored on the top of the RS as shown in the figure 7a. Apart from honey combing and porosity, any other defects did not occur in this nugget area as shown in the figure 7b. Due to the convex shape of the material consolidation on the nugget region, the small gap appeared in the AS even the material thoroughly mixed in interior section and good contact of the nugget region with both sides as shown in the figure 8a. In other two conditions, much problem or defects were not occurred except small built-up layer formation and gap appeared in the AS as shown in the figure 9a &10a. Cavity, inclusion, and porosity occurred in both condition's nugget area (see figure 9b & 10b).

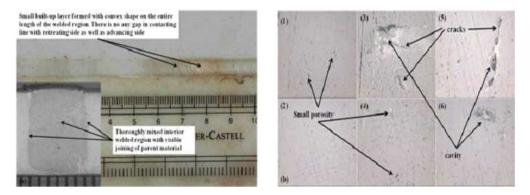


Figure 9: a & b Appearance & Microstructure of FSW Region for Grooved Pin at 1200 r/min and 30 mm/min

In the threaded tool profile, overheat created in the entire length of the welded region. So, the material consolidation of the joint interface was in convex shape for four different welding conditions as shown in the figure 11a-14a. Due to the convex shape of the welded region, the small visible gap formed in the contacting of AS as well as minute gap formed in the RS even the nugget material thoroughly mixed in the entire length. In some case, small rib formed on the AS (see figure 12a) and small crystal form of material appeared on the joint interface randomly (see figure 14a). The nugget

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had only few defects such as cavity, blow holes as shown in the figure 11b-13b except the last condition (Threaded pin at 800 r/min and 40 mm/min) of welded region (figure 14b). If the material was not effectively contacted with the parent material of both sides, more defects occurred in the contact line of nugget as shown in the figure 14a.

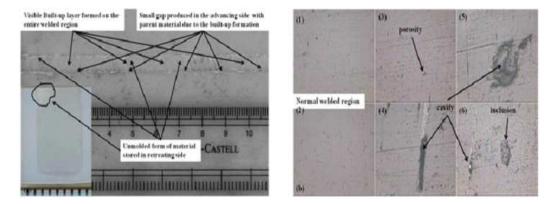


Figure 10: a & b Appearance & Microstructure of FSW Region for Grooved pin at 1000 r/min and 40 mm/min

The triangular pin was mostly created defective welded region in this investigation such as rib, blow holes as shown in the figure15a-18a. If the tool had not brought the material as colloidal form otherwise the tool created temperature was not enough to bring the material as colloidal form as shown the figure 17a & 18a., the semi-solid and unmolded crystal form of material appeared in/on the welded region and there was good contact with AS parent material as shown in the figure 18a. Mostly the triangular pin profile created defective welded region and number of defects were easily identified from empty eyes.

Effects of Spindle Speed on Nylon 6 Plate Welding by FSW

In this investigation, there are four different spindle speed analyzed such as 600, 800, 1000 and 1200 r/min. Except triangular pin profile (figure18a & 18b), other tool pin profile created good weld region for 600 r/min and also had only few defects. The grooved pin profile for 800 r/min spindle speed created good welded region even one or two porosity appeared in that nugget. The other tool pin profile for this spindle speed developed more defects in/on the joint interface and also contact line of the nugget with both sides.

For 1000 r/min spindle speed, triangular and square pin created poor weld region and the contact line of nugget in the both sides was also poor. The threaded pin produced better result compared with the grooved pin profile for 1000 r/min spindle speed as shown in the figure 10 & 11.

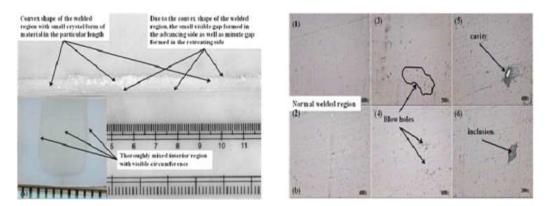


Figure 11: a & b Appearance & Microstructure of FSW Region for Threaded Pin at 1000 r/min and 10 mm/min

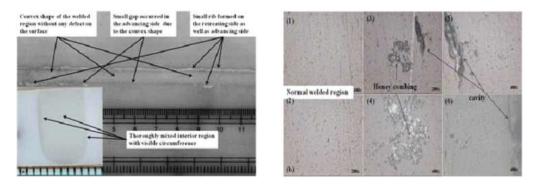


Figure 12: a & b Appearance & Microstructure of FSW Region for Threaded Pin at 1200 r/min and 20 mm/min

Square pin created contact line defects for 1200 r/min as shown in the figure 6. The other three pin profiles (Triangular, Threaded, and Grooved pin) produced normal welded region for this spindle speed. There is no much defects occurred in this condition as shown the figure 9, 12 and 15. When the spindle speed was evaluated, that did not directly involved to create the optimum welded region. The spindle speed depends on the tool pin profile and welding speed.

Effects of FSW Welding Speed on Nylon 6 Plate

The welding speed as 10 mm/min had produced good welded region without considering the tool pin profile and spindle speed in this investigation for Nylon 6 material as shown the figure 3, 7, 11, and 15. When we analyzed the microstructural images, only few porosity, blow holes and cavity appeared in the nugget area. Obviously, the square pin profile for all welding speed and spindle speed created major problem related to contact line with parent material and triangular pin created interior defects. For 20 mm/min welding speed, the triangular pin and square pin both were created defects. The grooved pin and threaded pin was produced good region except few porosity in the nugget area. The similar conditions followed for 30 mm/min welding speed. There is no much difference compared with 20mm/min with 30 mm/min welding speed except triangular pin profile. The small crystal form of material stored in/on the joint interface and some material had semi-molded conditions (see figure 17). The contact line of nugget area with AS was better compared with RS's parent material. The bottom nugget area material became as colloidal form even the top material appeared as semi-solid form as shown in the figure 17a. It had more defects as visible level and microstructural level as shown in the figure 17b. Square pin and grooved pin produced defects free nugget region except contact line of square pin profile. The threaded pin and triangular pin developed more defects for 40 mm/min welding speed. From this investigation, the good welded region is mostly based on the welding speed even the tool pin profile and spindle speed acted as the process parameters in nylon 6 plate for FSW. Obviously, 10mm/min and 20mm/min welding speed had produced defects free welded region as shown in the figures 3, 4, 7, 8, 11, 12, 15, and 16.

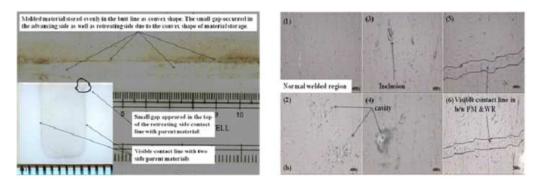


Figure 13: a & b Appearance & Microstructure of FSW Region for Threaded Pin at 600 r/min and 30 mm/min

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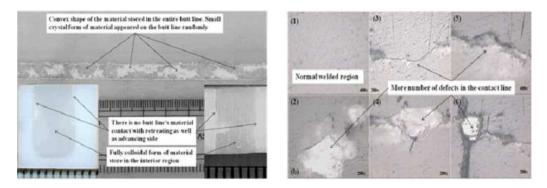


Figure 14: a & b Appearance & Microstructure of FSW Region for Threaded Pin at 800 r/min and 40 mm/min

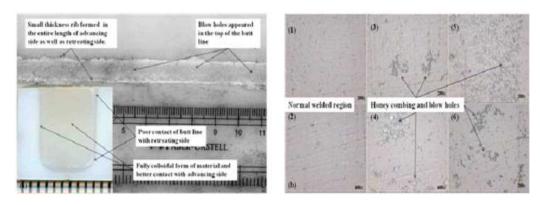


Figure 15: a & b Appearance & Microstructure of FSW Region for Triangular Pin at 1200 r/min and 10 mm/min

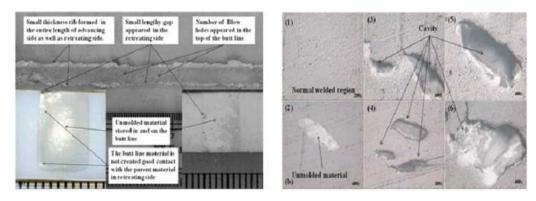


Figure 16: a & b Appearance & Microstructure of FSW Region for Triangular Pin at 1000 r/min and 20 mm/min

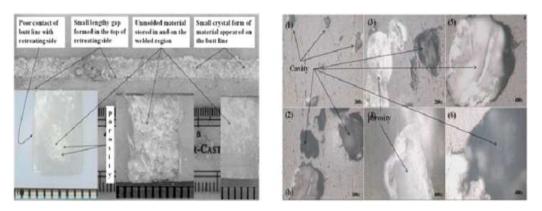


Figure 17: a & b Appearance and Microstructure of FSW Region for Triangular Pin at 800 r/min and 30 mm/min

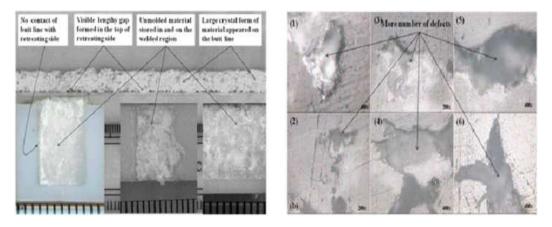


Figure 18: a & b Appearance & Microstructure of FSW Region for Triangular Pin at 600 r/min and 40 mm/min

| S.No | Advancing Side | | | Middle | Retreating Side | | |
|------|----------------|------|-----|--------|------------------------|------|------|
| | 15mm | 10mm | 5mm | Middle | 5mm | 10mm | 15mm |
| 1 | 92 | 92 | 87 | 82 | 86 | 92 | 92 |
| 2 | 92 | 92 | 88 | 83 | 87 | 92 | 92 |
| 3 | 92 | 92 | 90 | 84 | 89 | 92 | 92 |
| 4 | 92 | 92 | 89 | 83 | 88 | 92 | 92 |
| 5 | 92 | 92 | 88 | 84 | 88 | 92 | 92 |
| 6 | 92 | 92 | 91 | 85 | 90 | 92 | 92 |
| 7 | 92 | 92 | 89 | 84 | 87 | 92 | 92 |
| 8 | 92 | 92 | 90 | 80 | 88 | 92 | 92 |
| 9 | 92 | 92 | 92 | 82 | 88 | 92 | 92 |
| 10 | 92 | 92 | 89 | 84 | 89 | 92 | 92 |
| 11 | 92 | 92 | 88 | 84 | 88 | 92 | 92 |
| 12 | 92 | 92 | 90 | 81 | 88 | 92 | 92 |
| 13 | 92 | 92 | 89 | 84 | 89 | 92 | 92 |
| 14 | 92 | 92 | 90 | 73 | 88 | 92 | 92 |
| 15 | 92 | 92 | 88 | 69 | 86 | 92 | 92 |
| 16 | 92 | 92 | 89 | 66 | 85 | 92 | 92 |

Table 3: The Rockwell Hardness Distribution Middle, AS as well as RS for Each 5mm upto 15mm

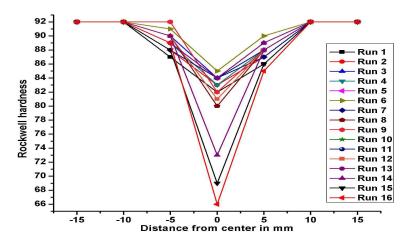


Figure 19: Effects of Different Process Parameters on Hardness

The hardness of base material has a range of 92 to 95RH. However, the hardness near weld zone shows variables value from 65 to 85 RH. The weld center has a slightly lower hardness than that of the base material. The hardness on the

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AS was slightly higher than those on RS. But, there is no much difference in hardness between the both sides as shown the figure 19. The left side of the middle mentioned AS and right side mentioned RS in this figure. The most of the nugget area's hardness had only 80 to 85RH except three nugget area. Triangular pin created very poor welded region with low hardness. The hardness difference was only occurred upto 5 mm from the joint interface on both sides.

CONCLUSIONS

In this investigation, an attempt was made to select proper tool pin profile, spindle rotational speed and welding speed to friction stir welded nylon 6 plate. From this investigation, the following important conclusions are derived:

- The welding speed acts as independent process parameter in nylon 6 plate for FSW process.
- In the Friction stir welding of Nylon 6 plate, Tool pin profile and spindle speed is mostly depended on the welding speed
- The grooved pin profile for different spindle speed and welding speed produced good welded region in Nylon 6 material.
- Due to the convex shape of nugget area profile, the threaded pin created small gap on the entire length in the both sides and poor contacting of nugget area with AS even the material become good colloidal form and stored evenly.
- More experiment's nugget area hardness are similar in this investigation except nugget area was created by the triangular pin profile. The hardness on the AS slightly higher than those on the RS.
- Nylon 6 plates were jointed with triangular and square pin profile at low welding speed and these tool produced more defects at the high welding speed.

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