Induction assisted friction stir welding (i-fsw): A Novel Hybrid process for joining of Thermoplastics

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Approval Sheet

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Dedicated to

My Parents

&

My Sisters

Contents

Abstract

In the present work an attempt was made to investigate the weldability of High density polyethylene (HDPE) sheets by a hybrid friction stir welding process called i-fsw (Induction Assisted Friction stir welding) where the friction stir tool during welding is heated by Induction and the temperature is precisely maintained through a temperature feedback control system. The mechanical behavior and weld microstructure of the joints were studied over a wide range of tool rotational speeds and tool pin temperatures. A narrow transition zone between the weld and base material without any defect ensures joints with a strength similar to the base material, better than previously reported results with the same material. A drop in hardness at the weld zone, for all the parameters, and a transition from brittle to ductile nature of the joints at higher tool pin temperatures were observed. The percentage crystallinity of the selected welded samples and base material from Differential Scanning Calorimetry (DSC) test confirmed the narrow transition zone width obtained at higher tool pin temperatures. The Infrared spectra from Fourier Transform Infrared Spectroscopy (FTIR) test showed the presence of aromatic functional group for the sample showing maximum joint efficiency and signifies the chemical change i-fsw brings at moderate and high heat input conditions.

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Chapter 1

Introduction

1.1 Background

Thermoplastics have extensive applications in aerospace and automotive industries due to their high strength to weight ratios and toughness. Among them polyethylene is the most produced and used plastic material due to its light weight, flexibility, ease of joining, long term durability, low cost and lack of corrosion [1], hence finds place in gas distribution pipeline applications.

Different grades of PE have been used as pipe materials, however High density polyethylene (HDPE) is the most preferred material for natural gas pipe manufacturing because it has high strength and a high modulus. For the last several years HDPE is being successfully used in geotechnical and civil engineering applications. In order to join PE materials various fusion joining techniques are being employed because of its high welding capability. All plastic welding techniques consist of three common stages: (a) Formation of a layer of molten material on the surfaces to be joined, (b) Bond formation by application of pressure, (c) The melt is allowed to cool and in this stage pressure should be maintained in order to prevent forming voids inside the weld zone.

1.2 Friction Stir Welding

Friction stir welding is a solid state welding method that was invented by Thomas W.M at The Welding Institute (TWI) in United Kingdom in 1991 [2]. Initially it was used for welding of aluminium and aluminium alloys and later extended to magnesium and titanium alloys. It is a solid state hot shear joining process in which a rotating tool with a shoulder and a pin traverses along the weld seam as shown in Fig. 1. The frictional heat from the tool shoulder and deformation heat from tool pin enable the material to soften without reaching the melting point. Severe plastic deformation and flow of this plasticized material occurs as the tool is translated along the welding direction. Material is transported from the leading to the trailing edge of the tool where it is forged to form a joint.

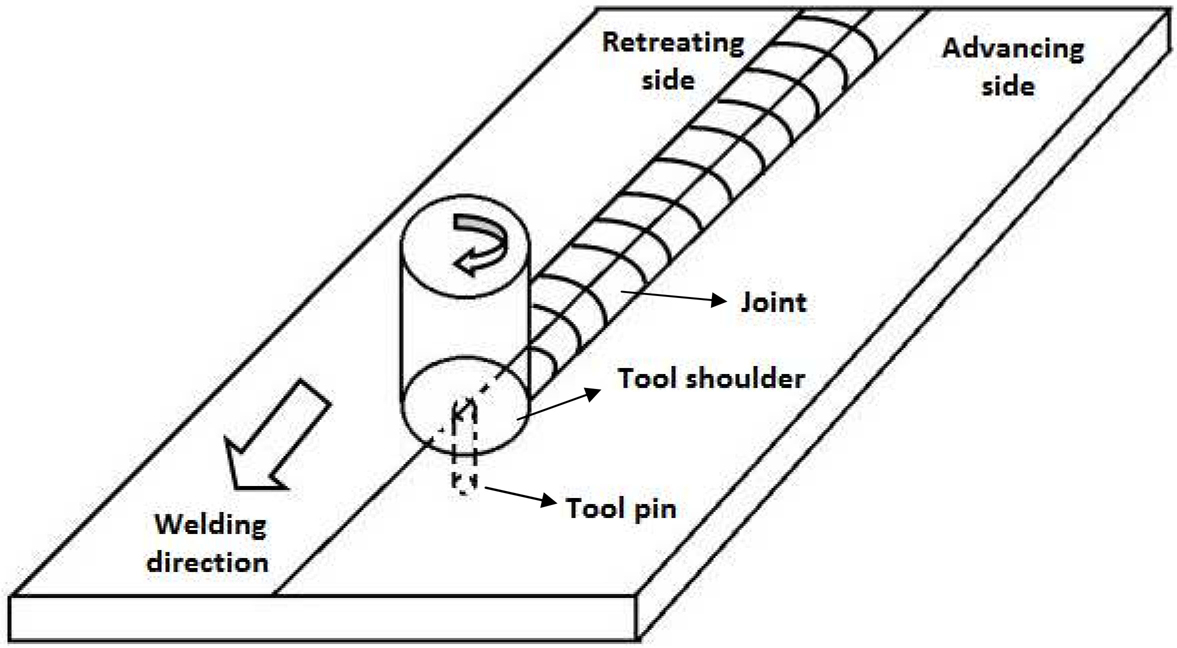


Fig. 1 Schematic of Friction Stir Welding process

1.3 Advantages of FSW

1) FSW is an environmentally friendly process as no cover gas or flux is required, is versatile and consumes less energy in comparision with conventional welding methods [3,4-6].
2) A milling machine and a tool are the only required equipment without any other welding machines.
3) Increased productivity and quality due to ease of automation and possibility of long continuous welds with an ability to weld almost all thermoplastics [7].

1.4 Limitations of FSW

Despite friction based welding such as friction stir welding or ultrasonic welding were very successful for metals, there are some limitations for plastic welding that are difficult to overcome such as [7,8]:

1) Low welding speed, low melting temperature, low hardness, relatively poor impact resistance and short solidification time.
2) Insufficient heat input due to very low thermal conductivity.
3) Non uniform weld bead and uneven polymer mixing [9]
4) Flash formation or ejection of melted thermoplastic from the weld region.

The FSW at present is primarily proved for producing linear welds and the process is different from that of metals because of differences in material structure and morphology. The tool in FSW of some hard metals sometimes need to be externally cooled whereas the same need to be externally heated in case of thermoplastics in order to avoid problems with conventional tools, such as squeezing of the polymer from the weld nugget.

If the conventional FSW process which is capable of curvilinear welding can be modified such that the tool temperature can be maintained over the entire length of the weld then the limitation of welding plastics by the existing methods like shoe heating method may not be best for curvilinear welds, preheating of tool may not perform lengthier welds because of dip in tool temperature and external heating of tool may lead to unnecessary heating of base material, can be eliminated.

The present work presents an attempt where the FSW tool is induction heated and precise temperature control is achieved through temperature feedback. This process is termed as Induction Assisted Friction Stir Welding process (i-fsw) and no publication was found on fsw of HDPE using this process. Tool rotational speed and tool pin temperature at the beginning of the process were considered as the variable parameters in order to study their effects on the Tensile strength, Micro structure and Micro hardness of the joints. Differential Scanning Calorimetry (DSC) examination of the Base material and five different cases of welded samples was performed for getting information about the crystalline nature of the joints (% crystallinity). Fourier Transform Infrared Spectroscopy (FTIR) technique was applied with the main objective of identifying any alteration in the HDPE composition ie composition of the welds performed at different parameters from the base material (case of no welding).

1.5 Document Organisation

Chapter 1 is an Introduction to this document containing a brief discussion of the purpose of this thesis. A brief understanding about the friction stir welding process is given.

Chapter 2 contains an in-depth review of literature published on friction stir welding of polymers.

Chapter 3 gives information about the Materials and Experimental procedure employed in this research. Procedures for welding, specimen preparation and testing are explained.

Chapter 4 presents the results obtained by butt welding HDPE plates using i-fsw process. It gives the relationship between the microstructure of the polymer within the weld zone and the mechanical properties of the welded joint.

Chapter 2

Literature Review

In this chapter the existing literatures on polymer welding are illustrated focusing mainly on friction based welding techniques.

Polymers can be welded by three methods that depend on the heat generation technique:

Methods based on a) Heat conduction b) Heat Radiation c) Mechanical Friction. In heat conduction based techniques such as Hot plate welding, Hot gas welding, Extrusion welding, the surfaces to be welded are electrically heated and then they are pressed together by applying pressure to form a strong joint. In welding methods based on radiation such as Laser welding, Induction or High Frequency welding, Infrared welding the material absorbs the electromagnetic radiations there by rising its temperature. In the case of friction welding methods such as Spin welding, Linear Vibration welding, Ultrasonic welding the heat is generated the surface friction between the surfaces. Friction Stir Welding is a new member in this family of welding for joining soft metals and was initially used for aluminium alloys.

2.1 Welding with conventional FSW tool (Integrated pin and shoulder)

Bozkut [10] employed a conventional FSW tool with an 18mm diameter shoulder and 6mm diameter pin varying the tool rotation speed from 1500 to 3000rpm and travel speed from 45 to 115 mm/min in welding of High Density Polyethylene (HDPE). Temperature variations between 120°C and 165°C were measured and the author observed root cracks and voids in the welds which were responsible for the poor tensile properties.

Saeedy and Givi [11] investigated the effects of critical process parameters on FSW of polyethylene where the parameters ranged from 1000-1800 rpm, 12-20 mm/min travel speed and tool tilt angles of 1° and 2°. A weld strength of 75% of the base material was achieved for only one optimized set of welding parameters that showed the importance of process parameters on the strength of the weld.

Arici and Selele [12] and Arici and Sinmaz [13] performed double pass butt welding of PE using conventional FSW tool to avoid root defects at rotation speeds upto 1000rpm and traverse speeds upto 60 mm/min. They observed that depending on the amount of heat input, some material was expelled from the joining area during welding.

Arici and Selale [12] also investigated the influence of tool tilt angle on FSW of PE where they observed that the strength and thickness of the welds decreased with increase in tilt angle.

Panneerselvam and Lenin [14] used square, triangular, threaded and tapered pin profiles to analyse the influence of pin geometry on FSW of Polypropylene (PP) at rpm of 1500-2250 and traverse speed of 30-60 mm/min. They reported poor joining at the retreating side and the threaded pin profile gave the best welding results.

These others also used a left hand threaded tool pin profile operated at 1000rpm and 10 mm/min feed to analyse the influence of tool rotation direction on the quality of the weld for Nylon 6 Polyamide as the base material and found that the weld was free of defects with improved strength when the tool was rotated in the anti clock wise direction. The clock wise direction resulted in expulsion of material from the seam resulting in poor weld quality.

2.2 Preheating of tool and Preheating of Plates

Aydin [15] used Ultra High Molecular Weight Polyethylene (UHMW-PE) as the base material and preheated it to perform single pass butt welding. They reported that the surface morphology of the weld and global properties were improved by preheating.

Squeo and Quadrini [16] performed preheating of tool and work piece using conventional rotating tools in FSW of polyethylene claiming that the weld quality was improved but optimization of process parameters was needed to achieve optimum FSW conditions

2.3 FSW with Modified tools (Hot Shoe)

To avoid expulsion of melted polymer from the weld seam some researchers used a modified tool with a stationary shoulder called Shoe.

Nelson et al. [8] proposed the patented FSW tool with a hot shoe to overcome the problems with conventional tools like ejection of material by the rotating shoulder where the heat input is given by an electric heater connected with the shoe as shown in Fig. 2, thus retaining the molten material in the weld region and providing forging pressure for thermoplastic consolidation. Joint efficiency of atleast 75% of the base material was achieved.

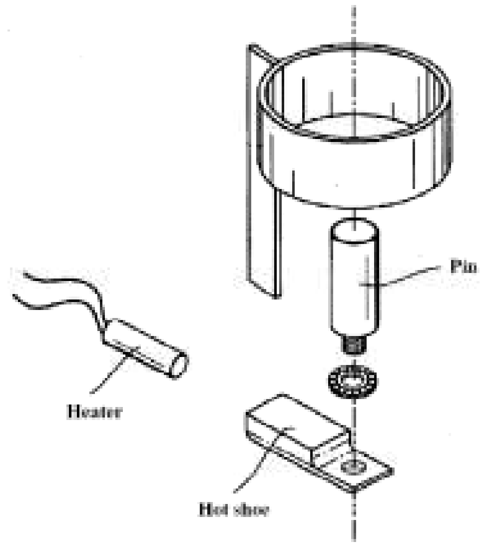


Fig. 2 Friction stir welding tool with a heated shoe [8]

Mostafapour and Azarsa [17] varied the rpm from 1000-1600, travel speed from 10-40mm/min and used shoulder temperatures 80°C, 110°C and 140°C to weld HDPE using a heated aluminium shoulder and reported defects such as lack of bonding between the weld nugget and the base material, incomplete tool penetration as well as material degradation at high temperatures. When there was sufficient heat input to the weld seam these defects were reduced.

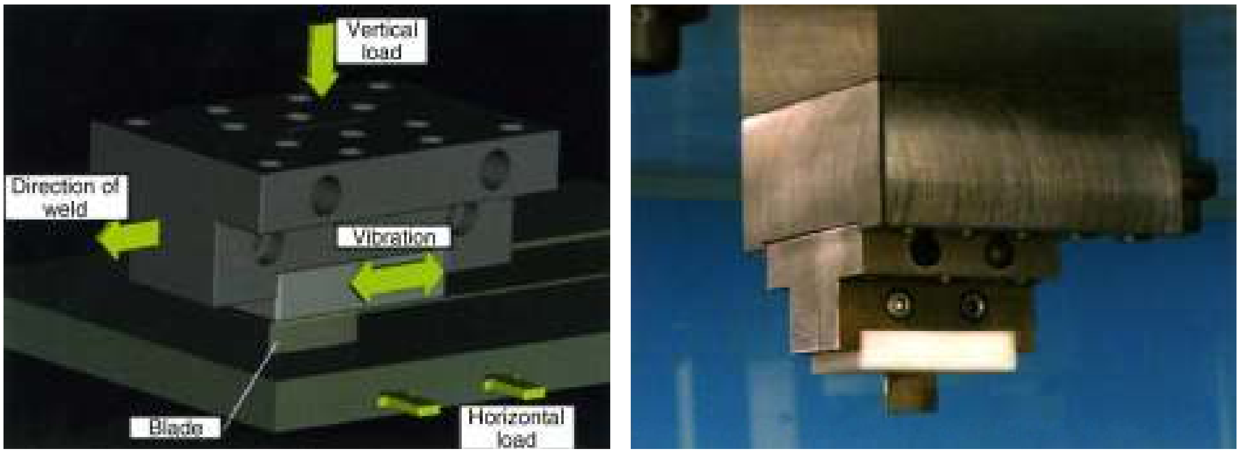
Rezgui et al. [18] used a stationary shoulder made of wood to weld HDPE at three different cylindrical threaded pins and used Taguchi method to optimize the weld parameters. They observed that the presence of discontinuities in the weld caused the specimens to fail for very small strain values.

Kiss and Czigany [19,20] used an non heated shoe to perform FSW for Polypropylene (PP) and Polyethylene terephthalate glycol (PETG) where spherulitic structures similar to the base material (B.M) were observed in the nugget due to the slow cooling rate of the weld, for PP welds. In case of PETG welds a sharp discontinuity between the weld and B.M were reported.

Bagheri et al. [21] used a heated shoe to weld ABS and observed inadequate material mixing and lack of joining between the seam and the base material at low rpm and burning of material at high rpm. The results were improved by heating the shoe.

2.4 Viblade Method

Viblade method belongs to the family of friction based methods of heat generation where an Oscillating blade and Shoulder move in the horizontal direction as shown in Fig. 3 and hence the heat reaches the root of the joint. It is used for welding thick plastics but can produce only linear welds due to the limitation of the blade tool [22].



a) b)

Fig. 3 Viblade welding process. a) Schematic outline of the Viblade process b) Viblade machine[22]

From the literature review it becomes clear that in order to avoid plastic material being expelled from the weld seam and obtain good quality welds, Stationary shoulder tools with a heating system need to be used but they cannot perform curvilinear welds. In the present work an attempt was made to weld HDPE plates using a tool that is induction heated, because of its advantages as there is no publication on fsw of HDPE using i-fsw process.

2.5 Objectives of present study

1. To demonstrate a new technique of friction stir welding called Induction Assisted friction stir welding (i-fsw) that can overcome the limitations of existing methods.
2. To study the effects of critical process parameters on the mechanical properties ie tensile strength and microhardness and identify the relation between the types of fracture and strength of the joints.
3. To evaluate the microstructure and understand the crystalline nature of the welded joints.

Chapter 3
Materials and Experimental Method

The present study is carried out on High density polyethylene thermoplastic as the raw material and a vertical milling machine is used to perform the FSW experiments. The following section gives details about the experimental set up, welding parameters and materials and tools used.

3.1 Experimental set up

The i-fsw process consists of an induction coil that encircles the FSW tool as shown in fig. 4. The Induction coil along with the induction power source and optical infrared temperature sensor are mounted on the machine head and move with the tool as shown in fig. 5.

3.2 Principle of Working

When an alternating electrical current is applied to the primary of a transformer, an alternating magnetic field is created. According to faradays law, if the secondary of the transformer is located within the magnetic field, an electric current will be induced. In fig. 4 the induction power source serves as the transformer primary and the part to be heated (work piece) becomes a short circuit secondary. When a metal part is placed within the induction coil and enters the magnetic field, circulating eddy currents are induced within the part. These eddy currents flow against the electrical resistivity of the metal, generating precise and localized heat without any direct contact between the part and the induction coil. This heating occurs with both magnetic and non-magnetic parts and is often referred to as, the Joule Effect.

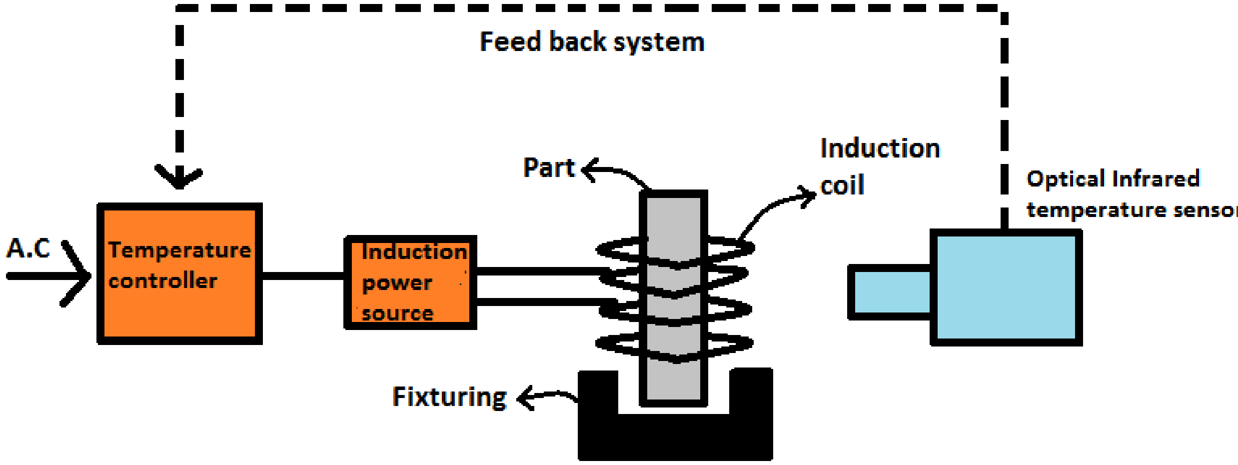


Fig. 4 Schematic of basic Induction heating setup

a) b)

Fig. 5 a) Induction power source with coil and Infrared temperature recorder b) Welding equipment installed on the milling machine.

In this way the FSW tool is consequently heated and the temperature sensor senses the temperature at the tip of the tool and sends signal to the temperature controller which is synchronized with the induction power source.

3.3 Material Specification and Tool Dimensions

Commercial 5mm thick high density polyethylene (HDPE) plates were cut into 170x85 mm size plates for FSW with mechanical properties given in table 1.

Table 1: Mechanical properties of HDPE

Youngs Modulus	Yield Strength	Break Stress	Strain at Break	Hardness
0.714 Gpa	19.92 Mpa	11.12 Mpa	117.23 %	4.85 HV

The tool used was made of H13 tool steel material with a hardness of 53HRC. The shoulder has a diameter of 10mm and the pin is taper threaded with a major and minor diameter of 6mm and 5mm respectively. The pin length is 3.83mm. The direction of tool rotation was anticlockwise to promote a downward flow of material, since the pin was right hand threaded.

3.4 Process Preparation

After preparing the HDPE plates as per the required dimensions they were held tightly together in the butt configuration in a simple fixture as shown in fig. 6.

Fig. 6 Fixture for butt joint configuration

The rotational speed of the tool, travel speed and tool pin temperature were considered as the process parameters and were chosen through pilot experiments conducted on bead-on-plate (BOP) welds. Three different rotational speeds, four different tool pin temperatures and a constant feed rate were selected as shown in table 2.

Table 2: Processing parameters

Welding Parameters (feed rate=50mm/min)	
Rotational speed(RPM)	Tool Pin temperature(˚C)
1000	40
2000	45
3000	50
	55

Once the processing parameters were set and the two pieces held tightly in the fixture a hole of dimater 7mm was made initially using a drill bit until a depth of 4.7mm on the butt line and then the tool was induction heated where the temperature of the pin was measured using the optical infrared temperature sensor. Once the pin reached the required temperature the rotating tool was plunged into the sheets 1.17mm shorter than the thickness of the workpiece in order to prevent outpouring of the melted material from the bottom. A dwell time of 15 seconds was given to let the tool warm up the material by friction and then it was traversed along the weld line. The heating of the tool by induction is continued throughout the weld line and once the tool reaches the end of the butt line it is lifted up together with the induction coil and then heating of the tool is stopped. After FSW the welds were allowed to cool in the fixture for about 15 minutes to avoid bending caused due to shrinkage of the material in the weld zone.

3.5 Measurement and Tests

3.5.1 Tensile test

For each welding condition (using a given set of process parameters) two welds were made at different points of time to bring into effect different environmental conditions and a dog bone shaped specimen was cut at the centre of the welded sample as per ASTM D-638 standard type 1 specimen shown in fig. 7 and tensile test was performed with the specimen without removing the flash in a Universal Tensile Testing Machine at a cross head speed of 5mm/min as shown in fig. 8.

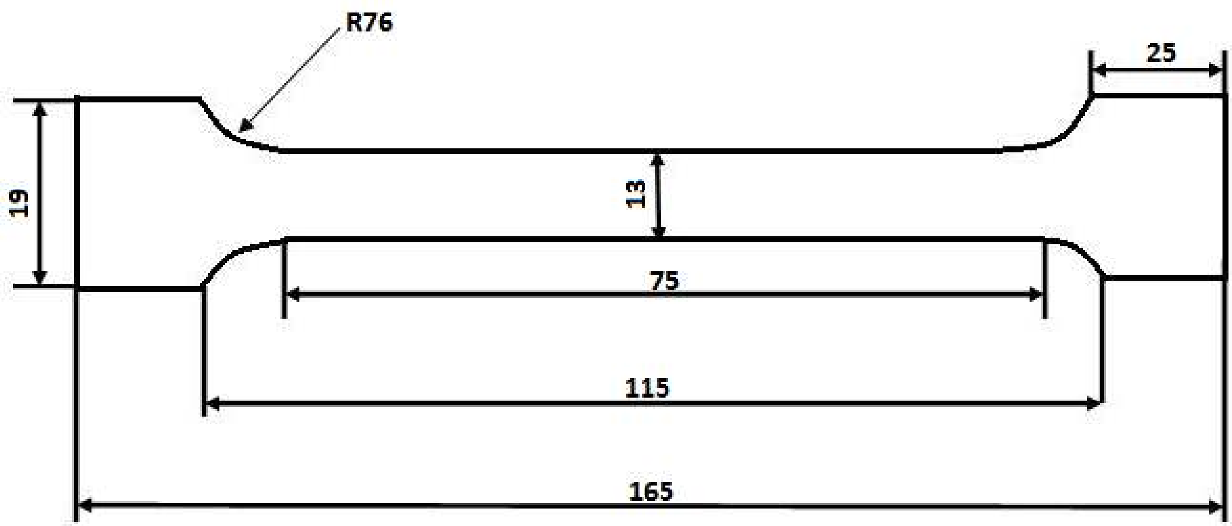


Fig. 7 Dimensions of dog bone shaped tensile specimen as per ASTM D-638 standard in mm

Fig. 8 Tensile testing of the Base material

3.5.2 Hardness Distribution

Vickers hardness test was conducted on the cross section of the welded specimens, each of size 40 × 20 × 5mm at distances 1.2mm and 2.4mm above the base. Samples were polished in a polishing machine until they were smooth and then indented with a load of 100g for a dwell time of 15 seconds. Before Indentation the surface was rubbed with a small amount of acetone to enable a clear identification of the diagonals of the indent.

3.5.3 Microstucture Examination

Microstructure images of the weld zone were examined using the optical microscope at 50X, 100X and 500X magnifications. Samples were initially polished with emery papers of grit sizes 180, 360, 600, 1200, 2000 and later were subjected to diamond polishing to get a smooth surface without any scratches. Once polished they were etched in 0.7% solution of $Kmno_4$ in a mixture of 35% volume of ortho phosphoric acid and 65% volume of sulphuric acid. The etching time was 2 hours followed by rinsing in a mixture of 2 parts by volume of sulphuric acid and 7 parts of water which were cooled to near freezing point, washed with hydrogen peroxide from the fridge followed by distilled water and acetone. After rinsing the samples, they were allowed to dry for about 5 minutes before being examined under the microscope.

3.5.4 Fourier Transform Infrared Spectroscopy (FTIR)

FTIR is a method to identify unknown materials, where the chemical bonds in the material interact with the infrared radiation and the amount of absorbed/transmitted radiation at different wave lengths gives information about the functional groups in the sample. The FTIR technique was applied with the main objective of identifying any alteration in the HDPE composition ie composition of the welds performed at different parameters from the base material (case of no welding).

3.5.5 Differential Scanning Calorimetry (DSC)

DSC is an analytical technique that measures the heat flow rate to or from a sample as it is subjected to a controlled temperature program in a controlled atmosphere. When specimens of semicrystalline thermoplastic pass through phase transitions such as melting points, the additional energy required to pass through such transitions are measured as endotherms. Melting point measurement and calorimetric studies of samples welded at 5 different parameters was performed on Differential Scanning Calorimeter. 10mg of each sample was taken from the center of the weld seam in a sealed aluminium sample holder with perforated cap and heated at a rate of 10°C/min from 40°C to 200°C. The crystallinity of a polymer can be calculated using the enthalpy of fusion (ΔH) and equation 1.

$$\% \text{ Crystallinity} = (\Delta H/\Delta H_C) \times 100 \qquad (1)$$

ΔH-Enthalpy of fusion for the sample

ΔH_C - Enthalpy of fusion for a 100% crystalline standard.

Melting point and enthalpy of fusion are listed in table 3.

Table 3: Physical properties of HDPE

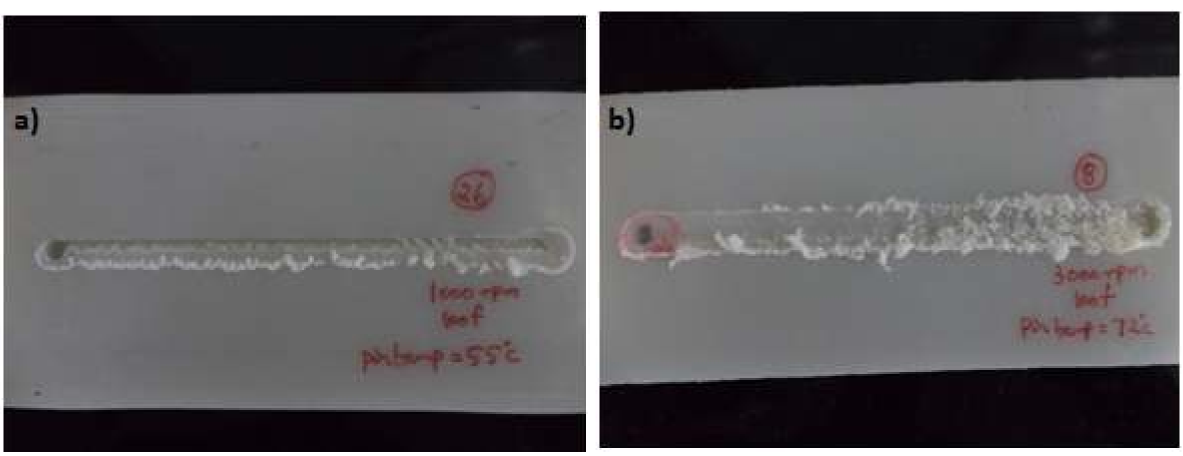
Density (g/cm^3)	Melting point (°C)	ΔH (J/g)	Crystallinity (%)
0.9705	144.4	107.7	36.39

Chapter 4

Results and Discussion

4.1 Pilot Experimentation

The input parameters shown in table 2 were chosen through pilot experiments conducted on bead-on-plate welds. The amount of flash and visual quality of BOP welds were observed. Welds were made at 1000, 2000 and 3000rpm at tool pin temperatures of 50°C, 55°C, 60°C, 65°C, 72°C and 80°C with feed rates of 50 and 100mm/min. Fig. 9 shows the effect of process parameters of single HDPE plates. From the observations of the BOP welds, keeping in mind the flash criteria and the quality of the weld bead it was decided not to perform the butt joints at tool pin temperatures more than 55°C and feed rates not more than 50mm/min. So in order to get the basic understanding of the effect of induction heating on HDPE, rotational speeds of 1000, 2000 and 3000rpm and tool pin temperatures varying from 55°C to the room temperature (case of no tool heating) at an interval of 5°C were chosen as the process parameters for performing the butt joints, keeping the feed rate constant at 50mm/min. The observations of pilot experiments is shown in table 4.



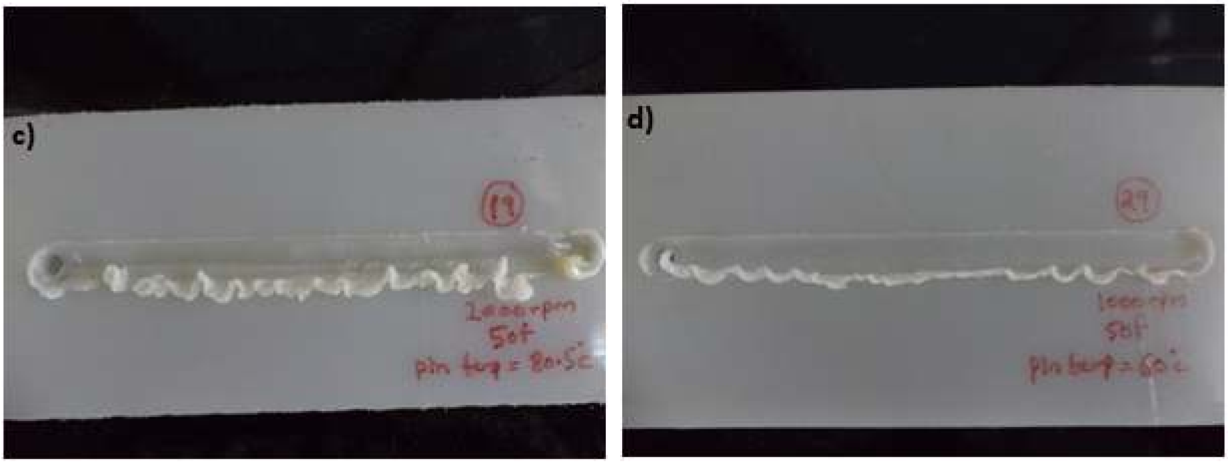


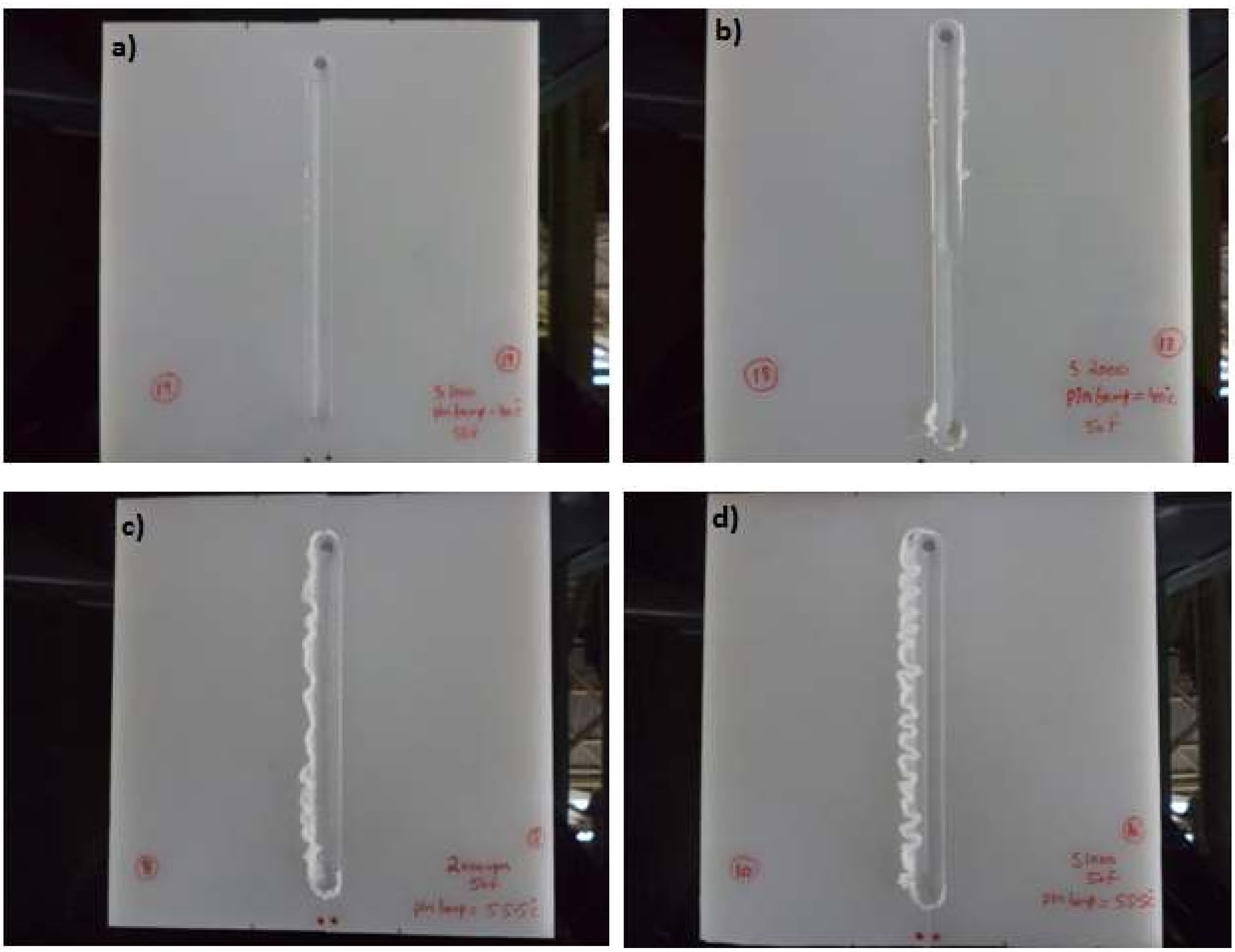
Fig. 9 Cases of a) Improper material mixing b) Poor quality of weld bead c) Burning appearance of plastic material d) Good material mixing with heavy flash appearance

Table 4: Observations made on single HDPE plates at different parameters

RPM	Feed rate (mm/min)	Tool pin Temperature(°C)	Observations
1000,2000,3000	100	55	Groove was observed throughout weld line
3000,2000	50,100	60	Material mixing was not proper and small slots were seen on the weld bead
1000,2000,3000	50	65	Material mixing was good but heavy flash was seen
1000,2000,3000	50	72,80	Slight yellow colour of the flash was seen which marked the onset of burning of the plastic
1000,2000,3000	100	72,80	Solid HDPE particles were deposited on the weld bead

4.2 Surface Views, Material Flow and Tensile Strength

The material flow, ie flash can be controlled by selecting proper process parameters. Fig. 10 shows the surface views of two HDPE plates joined by friction stir welding at different parameters indicating four representative cases of flash namely No flash (fig. 10a), Moderate flash (fig. 10b), Heavy flash (fig. 10c) and Very heavy flash (fig. 10d). It can be seen that changes in rotational speed and temperature greatly influence flash generation. For a change in temperature of 15°C for the same rotation speed no flash to very heavy flash condition is observed in fig. 10 (a) and fig. 10 (d). Similarly change in rotation speed at constant temperature brings a considerable change in flash generation. The flash criteria is important when the weld is seen from the aesthetic point of view but is not always decisive in terms of joint strength.



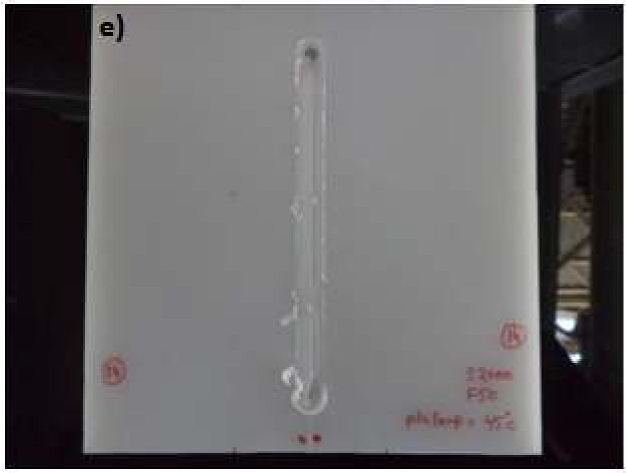


Fig. 10 Flash appearance in samples welded at different parameters

Fig. 10 (e) shows the surface view of the joint made at 2000rpm and a tool pin temperature of 45˚C that is having the maximum tensile strength compared with other parameters. Though heavy flash was seen for the joint made at 2000rpm and tool pin temperature of 55˚C, shown in fig. 10 (c), the strength was very close to that of the base material, indicating that the strength of the joint cannot be judged based on the flash criteria ie, it cannot be ensured that the welds showing considerable amount of flash, have a strength less than the welds with zero flash, which is the case shown in fig. 10 (a) that is having minimum tensile strength.

4.3 Effect of Rotational speed and Tool pin temperature on Tensile strength of the joints

Fig. 11 shows the surface plot of ultimate tensile strength with tool rotational speed and pin temperature which is convex shaped showing that the strength increased in a linear fashion from 1000 to 3000rpm for the welds performed without tool heating at room temperature while it increased from 1000 to 2000rpm at all tool pin temperatures, however a further increase in rpm caused the strength to decrease. It shows an interactive effect of tool rotational speed and pin temperature on the tensile strength of the joints. Similarly an increase in temperature from 35°C to 45°C resulted in an increase in strength, which came down at 50°C (showing the onset of flash) and later increased at 55°C in case of 1000rpm and 2000rpm but showed a slight decrease at 3000rpm.

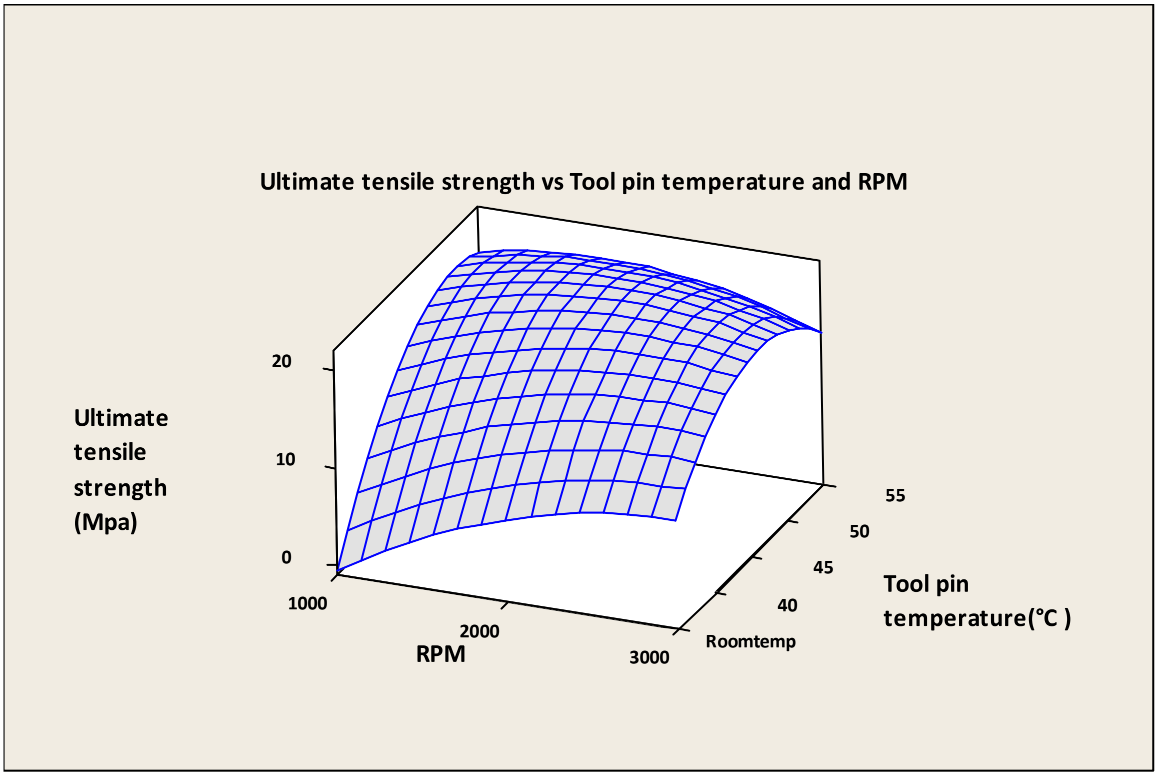


Fig. 11 Effect of tool pin temperature and RPM on the tensile strength

Though 1000rpm and tool pin temperature of 40°C resulted in minimum flash this combination also resulted in minimum strength. At low rotational speed and low tool pin temperature the heat generated is insufficient to soften and plasticize the polymer, resulting in improper fusion between the seam and the base material, as shown in Fig. 12. Stagnant material near the bottom of the seam on the retreating side of the joint indicates improper merging between the weld seam and the adjacent material.

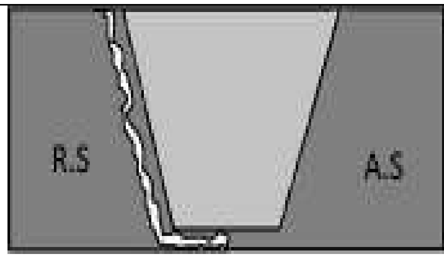
Fig. 12 Stagnant solid material near the bottom of the seam at the retreating side of the joint at 1000rpm and tool pin temperature of 40°C

Increase in rpm increases the temperature at the weld zone which is necessary for plastic deformation and thorough mixing of the plastic material. But higher rpm also results in heavy flash that reduces the thickness of the weld seam at the cross section. Thus a moderate rpm and temperature work optimal for i-fsw process in terms of ultimate tensile strength. At the high rotational speed of 3000rpm, the deformation and frictional heat cause excessive turbulence of material in the seam and hence the flow of softened material cannot be controlled, resulting in out flow of material. As a result the strength is reduced compared to that at 2000rpm where sufficient heat is generated at all the tool pin temperatures to soften the seam and properly fuse with the base material. In this case, the material rather than getting expelled is confined to a greater extent within the weld seam.

4.4 Fracture Analysis

Table 5 shows five different cases of fracture locations identified after conducting the tensile test. The welded samples fractured at different locations starting from outside the weld seam, at the retreating interface, in the retreating side of the weld seam, at the center of the weld seam, and in the advancing side of the weld seam.

Table 5: Types of Fracture Locations

<table>
<tr><th>Types of Fracture</th><th>Schematic</th><th>Photograph</th></tr>
<tr><td>a) Crack formed outside weld seam</td><td>
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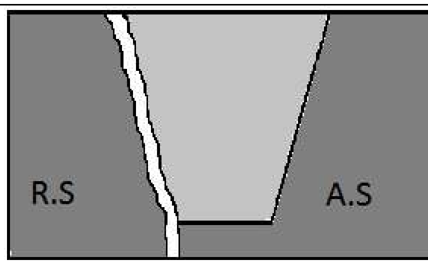
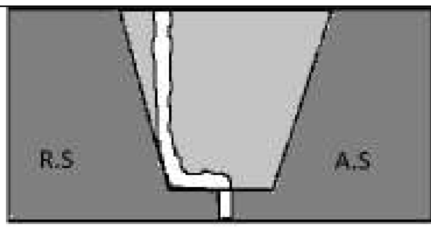
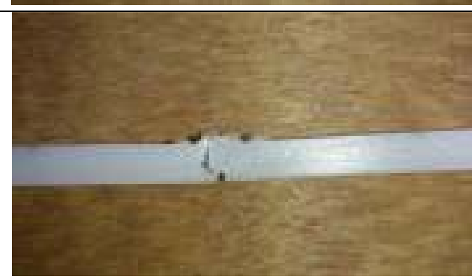
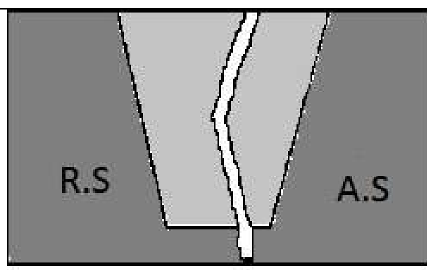
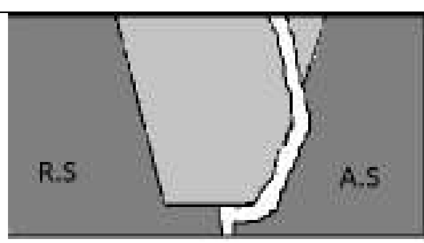
<tr><td>b) Crack formed at the retreating interface</td><td>
</td><td></td></tr>
<tr><td>c) Crack formed in retreating side of weld seam</td><td>
</td><td></td></tr>
<tr><td>d) Crack formed at the centre of the weld seam</td><td>
</td><td></td></tr>
<tr><td>e) Crack formed in the advancing side of weld seam</td><td>
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</table>

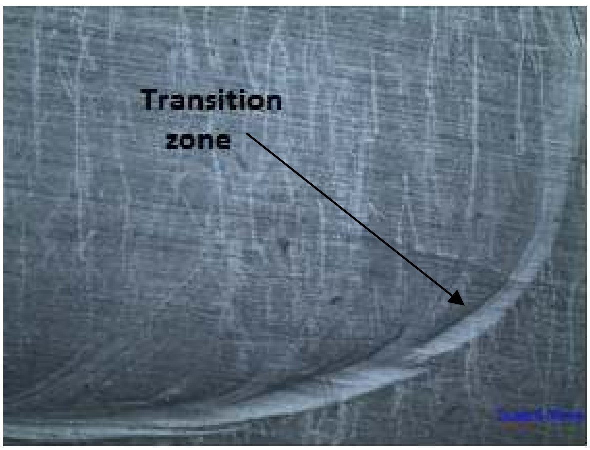
Table 6: Joint efficiency, fracture type and % elongation in friction stir welded samples

Sno	RPM	Tool Pin Temperature(˚C)	Ultimate Tensile Strength (Mpa)	Joint Efficiency(%)	Types of Fracture	Percentage Elongation (%)
1	1000	35 (room temperature)	0.55	2.87	Type c	6.96
2	1000	40	8.17	41.01	Type c	6.56
3	1000	45	15.81	79.37	Type c	15.56
4	1000	50	15.36	77.11	Type c	15.08
5	1000	55	18.76	94.18	Type d	21.69
6	2000	35 (room temperature)	4.5	23.62	Fractured at the root tip	1.37
7	2000	40	18.75	94.13	Type b	16.26
8	2000	45	20.78	104.32	Type a	20.17
9	2000	50	17.68	88.76	Type d	18.42
10	2000	55	19.66	98.69	Type c	18.05
11	3000	35 (room temperature)	9.48	49.82	Type e	2.59
12	3000	40	17.9	89.86	Type e	14.29
13	3000	45	19.69	98.85	Type d	13.49
14	3000	50	15.72	78.92	Type d	20.7
15	3000	55	15.51	77.86	Type e	23.55

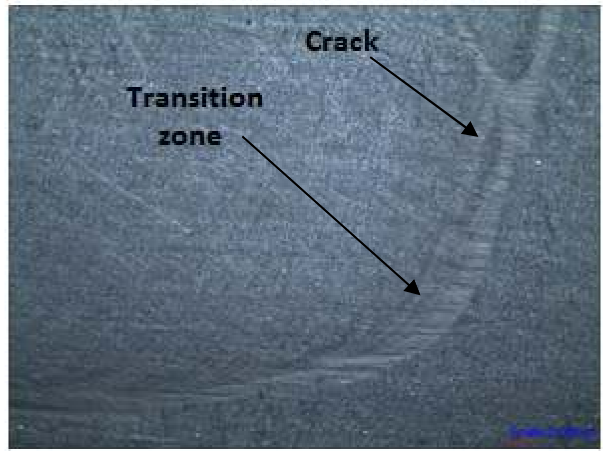
Table 6 shows the joint strength, types of fracture and % elongation of samples welded at different process parameters. The welds performed at 2000rpm and tool pin temperature of 55°C and 3000rpm and tool pin temperature of 45°C showed a joint strength of more than 98% which was very close to the base material and was not achieved earlier. The weld made at 2000rpm and 45°C pin temperature was found to have a joint efficiency of more than 100% which was a distinct case. From the above table it can be observed that the welds performed at low rpm and at all tool pin temperatures showed type c failure where all the welds failed in a brittle manner at the retreating side. These welds showed very low joint efficiency and percentage elongation on comparing with other welds. In the i-fsw welding process, the rpm which is mainly responsible for mixing of the plastic material (discussed in the later section) did not mix the plasticized material properly at low rotational speeds resulting in poor bonding between the weld seam and the base material that caused the welds to fail at the retreating side of the weld seam. When the crack formed at the interface of the weld seam and base material in the retreating side (type b) or at the center of the weld (type d) the joint efficiency was between 78-98 %. From table 5 the parameters at 2000rpm and pin temperatures of 40°C, 45˚C and 55˚C show a strength more than that at 50˚C because the parameter at 50˚C shows type d failure where the crack is in the middle of the seam indicating that the seam is weak in this portion. In the other parameters the crack is present outside the seam or just at the interface indicating that the seam is strong due to good material mixing and proper fusion with the base material. Comparing parameters at pin temperature 40˚C and rpm of 1000, 2000 and 3000 the position of the crack shifts from the retreating interface(just inside seam, type c) to the interface (type b) and then to the centre(type e) indicating the strong effect of rpm on the tensile strength of the joint.

4.5 Transition zone and comparision of DSC and FTIR results

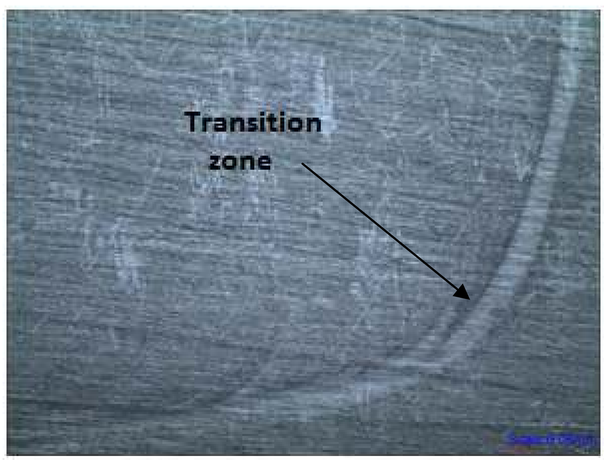
The fracture location and pattern are greatly influenced by shearing or flow developed by the outer perimeter of the tool. There exists some relationship between the microstructure of the polymer within the weld zone and the mechanical properties of the welded joint. Fig. 13 shows transition zone observed at the bottom of the tool pin in retreating side, between the seam and the base material. The width of the transition zone in case (a) is narrow compared to others. The case (a) represents the weld that was performed at 2000rpm and a tool pin temperature of 45°C and resulted in the maximum joint efficiency. This is in agreement with the findings of Kiss and Czigany [19] who observed that the strength of the FSW joint was close to that of the base material if the overall width of the transition zone is small and the less complex its morphology is. The width of the transition zone is controlled by cooling, molecular alignment/relaxation and crystallization, where crystallization plays the most important role. A wider zone can be observed in the case of the weld that was performed at 1000rpm and a tool pin temperature of 50°C, as shown in Fig. 13 (b). In this case the fracture occurred in the weld zone at the retreating side and the joint efficiency was quite low. In the absence of sufficient rotational speed, even if the temperature was increased to 50°C, proper fusion between the seam and the base material was not obtained. A crack at the weld and base material interface can be observed in Fig. 13 (b). When the rotation speed was increased to 3000 rpm the transition zone was uniform but wider and more clearly visible (Fig. 13 (c)) than the maximum joint efficiency condition of 2000 rpm (Fig. 13 (a)).



a) 2000rpm and pin temperature=45°C



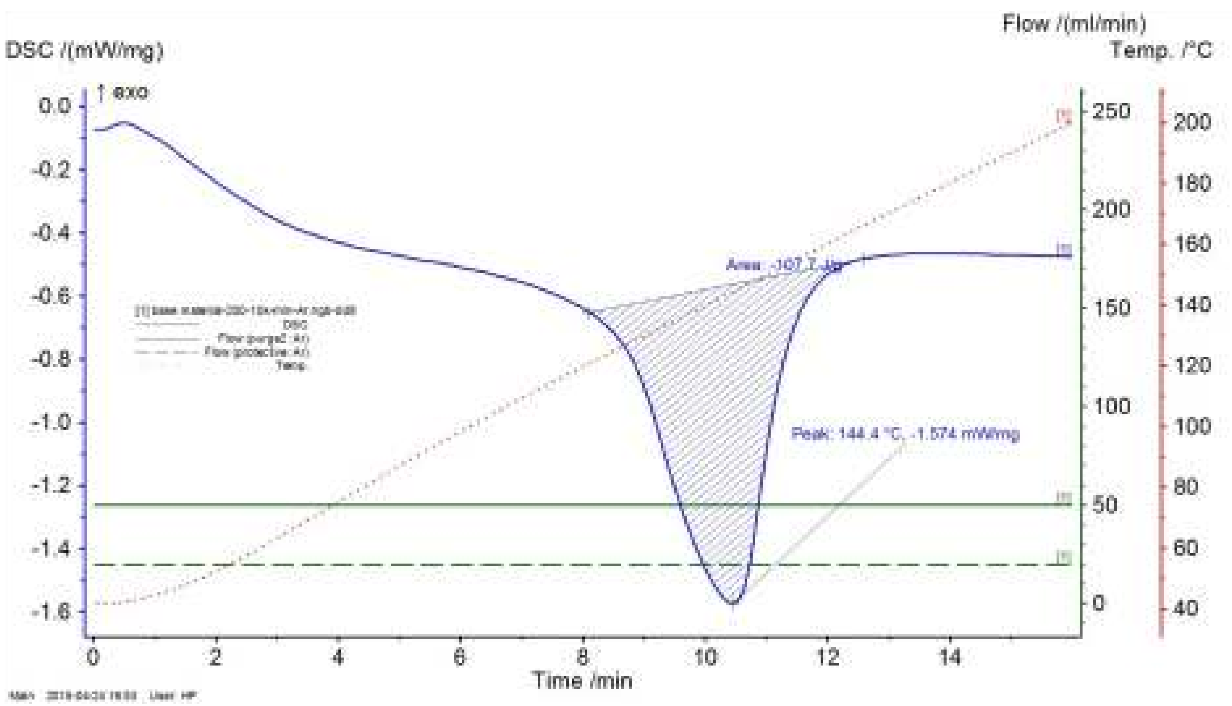
b) 1000rpm and pin temperature=50°C



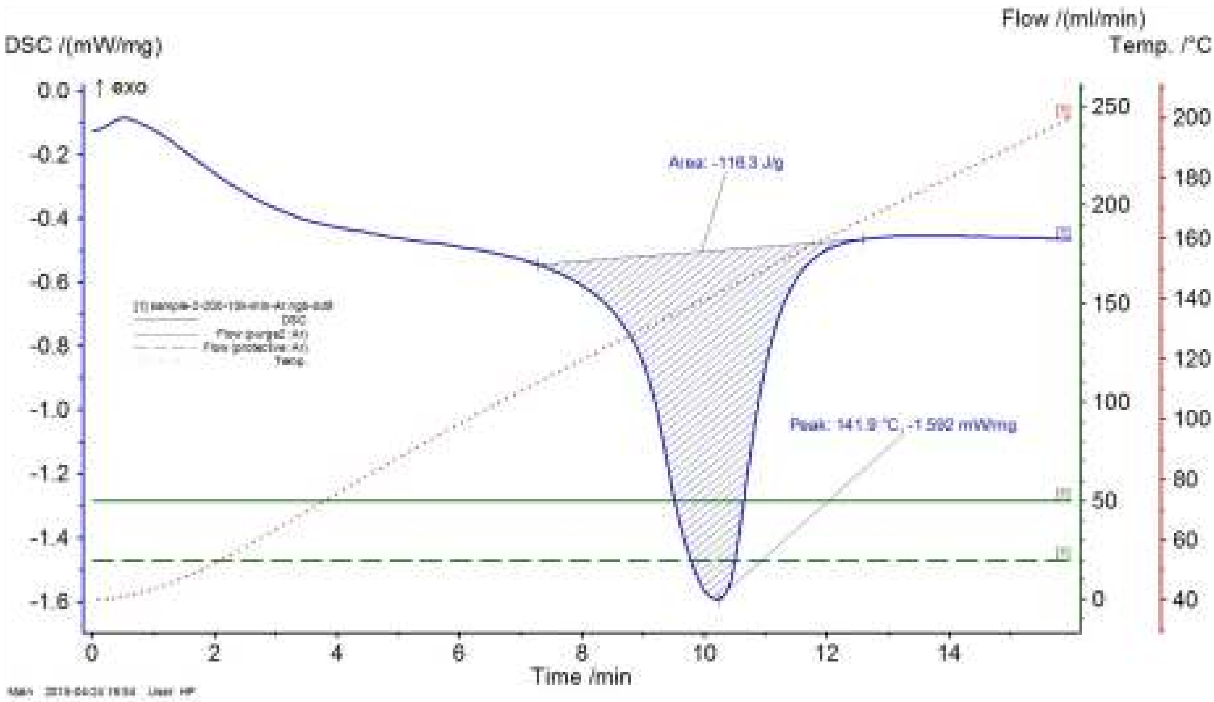
c) 3000rpm and pin temperature=45°C

Fig. 13 Transition zone at different welding conditions

The transition zone is due to skin-core structure formation in the weld. The central part of weld seem (core) cools slowly where as more intense heat withdrawal occurs at the base material and weld interface (i.e. skin). Thus, spherulitic crystallization could take place in the core section while supermolecular structure is formed in the skin. Moreover, temperature generated due to the rotation of the pin in FSW helps in crystallization because of longer molecular relaxation time [19]. As a result narrow transition zone width is obtained at higher tool pin temperature. The skin-core postulation was verified with the help of Differential Scanning Calorimetry (DSC) results. Fig. 14 shows the DSC thermograms for the base material and a sample welded at 3000rpm and 50°C tool pin temperature. The samples welded at higher tool rotation speed such as 2000 rpm showed 34% crystallinity which was close to base material (i.e. 36 %). With increase in rpm to 3000 crystallinity increased to 39 %. However for the sample at 1000rpm that showed wider transition zone, the crystallinity reduced to 13% shown in fig. 15, which may also be the reason for the fracture to originate from the weld seam at the retreating side.



a)



b)

Fig 14. DSC thermograms of a) Base material and b) Sample welded at 3000rpm and 50°C pin temperature

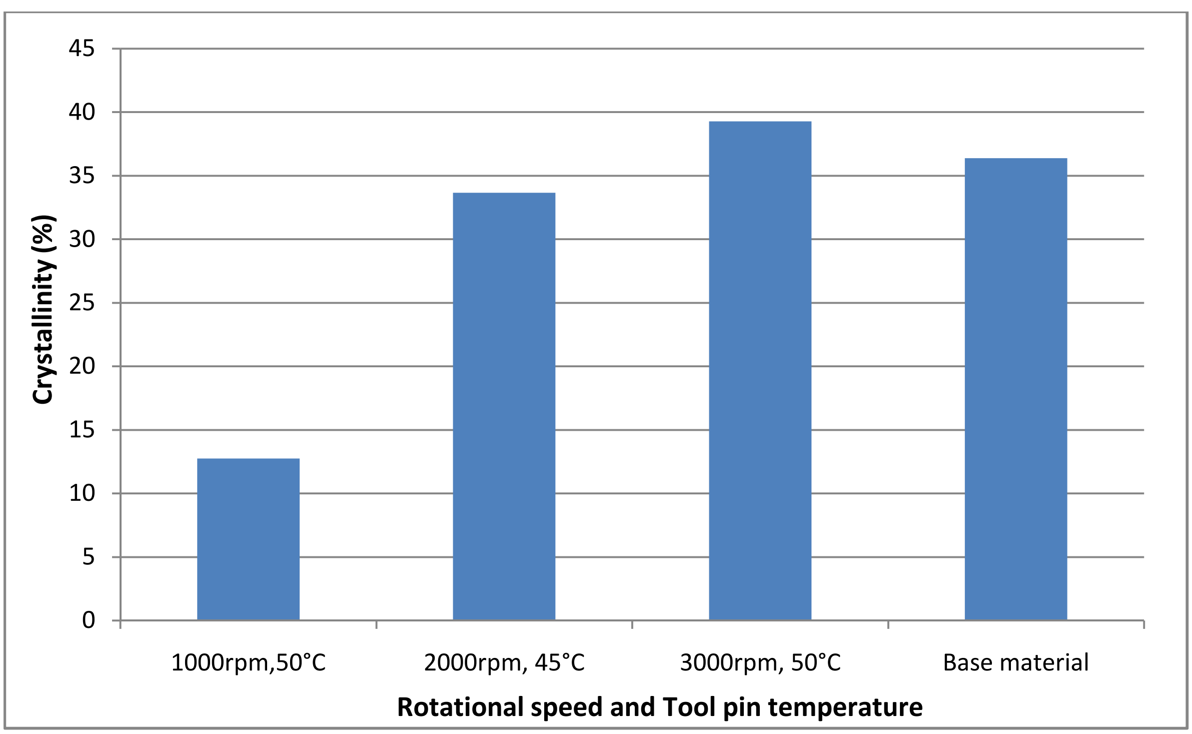


Fig. 15 Percentage crystallinity for base material and welds made at three different welding conditions

The DSC results were supported by FTIR results. Figs. 16 and 17 show the Infrared spectra (IR) of the base material and welds obtained at higher, moderate and lower heat input represented by 3000rpm and 50°C, 2000rpm and 45°C and 1000rpm and 40°C. The IR spectra of the weld made at 3000rpm and 50°C (case of higher heat input) resembles more with the base material. In these two cases similar bands (ie strong bands at 2912 and 2845 cm^{-1} which is due to asymmetric and symmetric stretching frequency of C-H group, a medium band at about 1465 cm^{-1} due to stretching frequency of the C-C bond and medium band at 721 cm^{-1}) are observed, which are not observed at medium and low heat input welds, shown in table 7. The IR spectra of the weld at 2000rpm and 45°C showed a strong bond (close to 800 cm^{-1}) similar to that observed in weld produced at 3000rpm and 50°C. This band indicates the presence of aromatic functional group and signifies the chemical change i-fsw brings at moderate and high heat input conditions. In case of 2000rpm weak bands in the range of 1665-1760 cm^{-1} are observed that is related with oxidation phenomenon. The oxidation range becomes stronger with low heat input weld. Moreover phenomenon of chain breaking (observed at bands of 900, 1177, 1368, 1375 and 1678 cm^{-1}) was observed, presented in table 8. Thus from the FTIR results it can be inferred that higher relaxation time due to high heat input conditions like 3000rpm and 50°C tool pin temperature, helps in preserving the internal structure of the welds, however the flash formation at this high rpm, lowers the strength to some extent as described in the section on surface views and material flow.

Table 7: Table indicating wave number, type of bond/vibration and functional group

Specimen	Wave number (cm^{-1})	Type of Bond/vibration	Type of Functional group
Base material	2912.81 (s), 2845.81 (s)	C-H stretching	Alkanes
	1465.43 (m)	C-C stretching	Aromatics
	722.39 (m)	C-H rocking	Alkanes
3000rpm and tool pin temperature=50°C (High heat input	2913.39 (s), 2846.06 (s),	C-H stretching	Alkanes
	1466.87 (m)	C-C stretching	Aromatics

condition)	721.43 (m)	C-H rocking	Alkanes
2000rpm and tool pin temperature=45°C (Medium heat input condition)	799.68 (s)	C-H "oop"	Aromatics
1000rpm and tool pin temperature=40°C (Low heat input condition)	551.1 (s)	C-Cl stretching	Alkyl halides
	670.36 (m)	C-Cl stretching	Alkyl halides

note: (s), (m) indicate strong and medium intensities of infrared radiation

Table 8: Phenomenon of chain breaking, chain branching, cross link and oxidation

	Types of Phenomenon			
Specimen	Chain breaking	Chain branching	Cross links	Oxidation
Base material	✓	✗	✗	✗
3000rpm and 50°C pin temperature	✗	✗	✗	✗
2000rpm and 45°C pin temperature	✗	✗	✗	✓
1000rpm and 40°C pin temperature	✓	✓	✓	✓
3000rpm and 40°C pin temperature	✓	✓	✓	✓
1000rpm and 50°C pin temperature	✗	✗	✗	✗

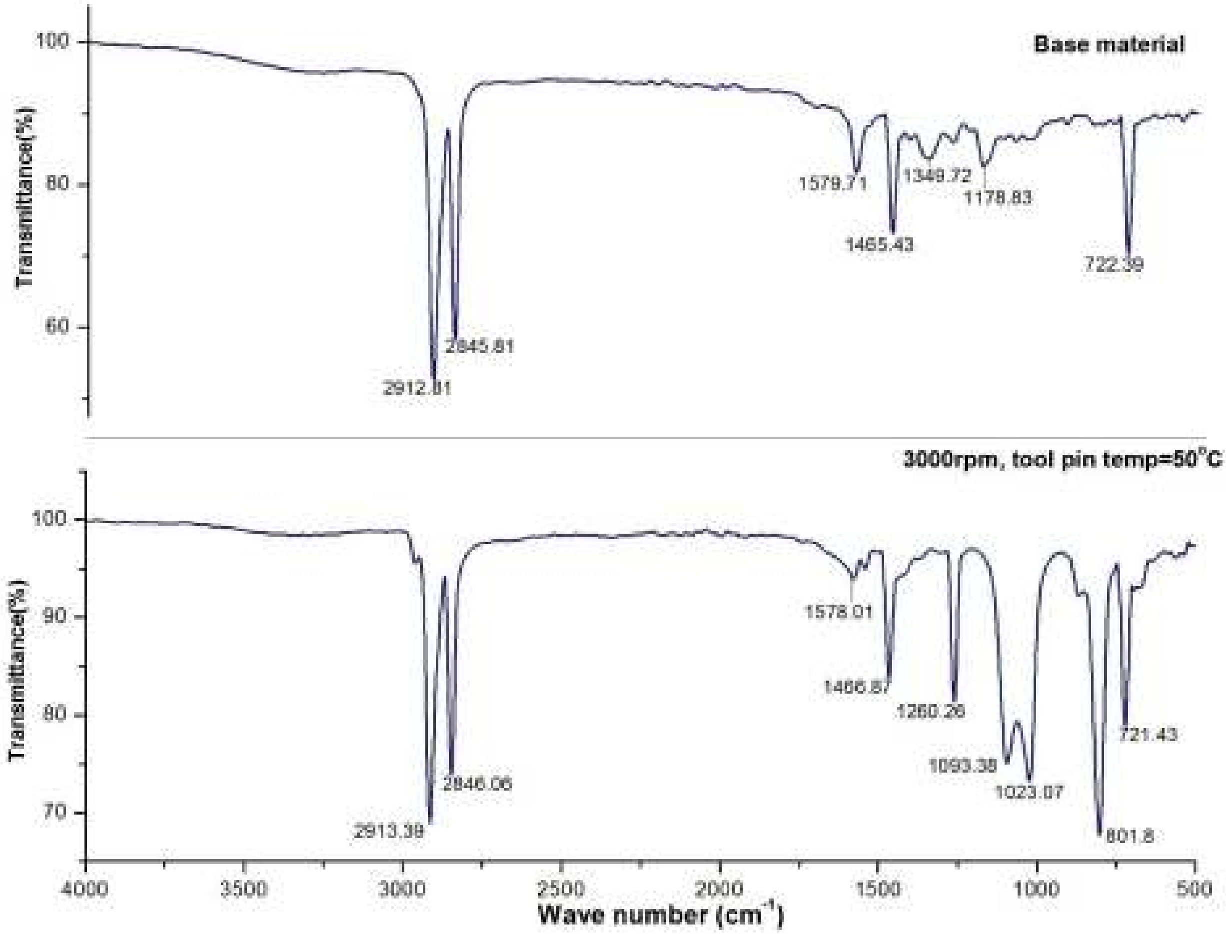


Fig. 16 FTIR results of the Base material and the weld performed at 3000rpm and tool pin temperature of 50°C

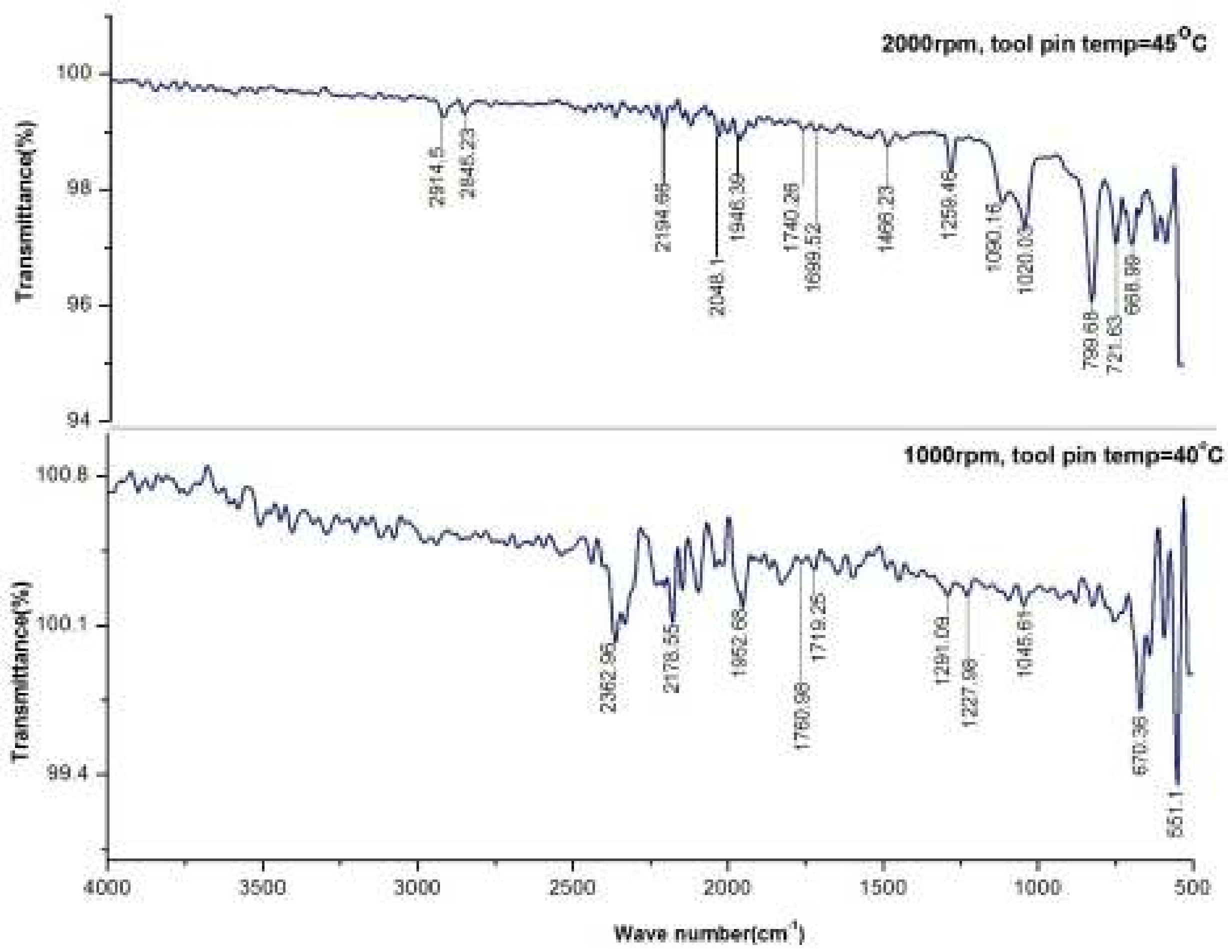


Fig. 17 FTIR results of the welds performed at 2000rpm and tool pin temperature of 45°C and 1000rpm and tool pin temperature of 40°C

4.6 Stress-Strain curves and their relation with fracture locations

The fracture locations and stress strain curves obtained for low tool pin temperature welds, is shown in Fig. 18 that indicate that all the joints failed in a brittle manner where there was a sudden drop in the strength after reaching the ultimate tensile strength. In all the three cases at different rotation speeds the frictional heat generated was not sufficient to have a good fusion between weld and base material. From the fracture locations seen along with the stress-strain curves in Fig. 18, the weakest part of the joint was seen at the interface zone (between the weld seam and the base material) due to the difference in internal structure between the interface and the weld seam [12].

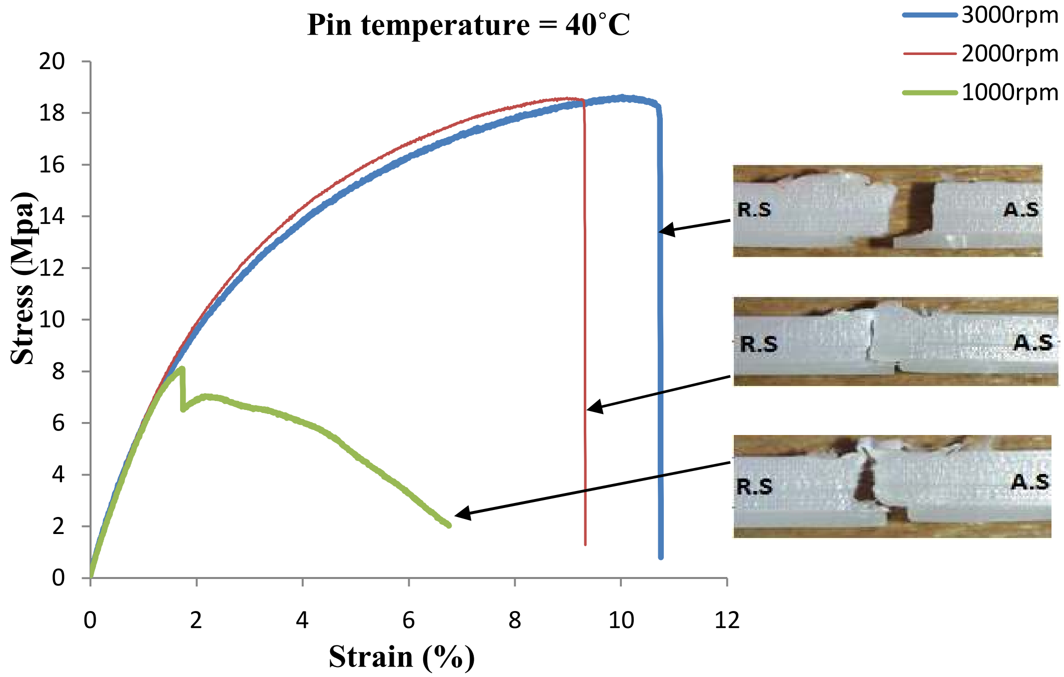


Fig. 18 Stress-strain curves and fracture locations at tool pin temperature of 40˚C

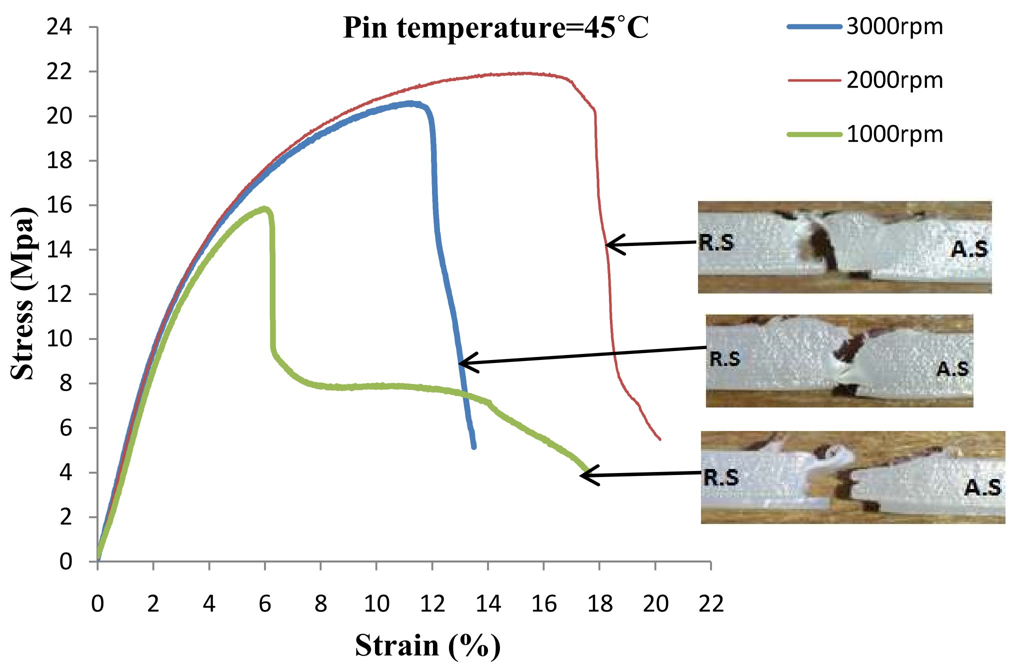


Fig. 19 Stress-strain curves and fracture locations at tool pin temperature of 45°C

When temperature was increased to 45°C, improvement in tensile strength and resilience was observed, as shown in Fig. 19. The ultimate tensile strength and resilience was at a maximum at

2000 rpm and the fracture was well outside the weld seam unlike the case of the joint made at 3000rpm where the fracture was at the center of the weld seam. The cross section images of the weld at 3000 rpm at the top, middle and bottom portions of the weld zone are shown in Fig. 20, that reveal the presence of defects, such as cavities and cracks, which reduced its strength. In the case of a joint made at 2000rpm, very few or negligible defects were seen. One of the reasons may be the fact that the rotational speed of the tool is not greatly responsible for heat generation; rather it is more responsible for the mixing of the material in the weld seam. Hence at 2000rpm the mixing of the material is good and the material was confined within the weld zone as compared to that at 3000rpm where, due to excessive turbulence, the flow became uncontrolled, reducing the strength of the joint.

(a)

(b)

(c)

Fig. 20 Defects in the weld joint made at 3000rpm and tool pin temperature of 45°C, a) Top region, large pore b) Middle region, small pores c) Bottom region, appearance of crack.

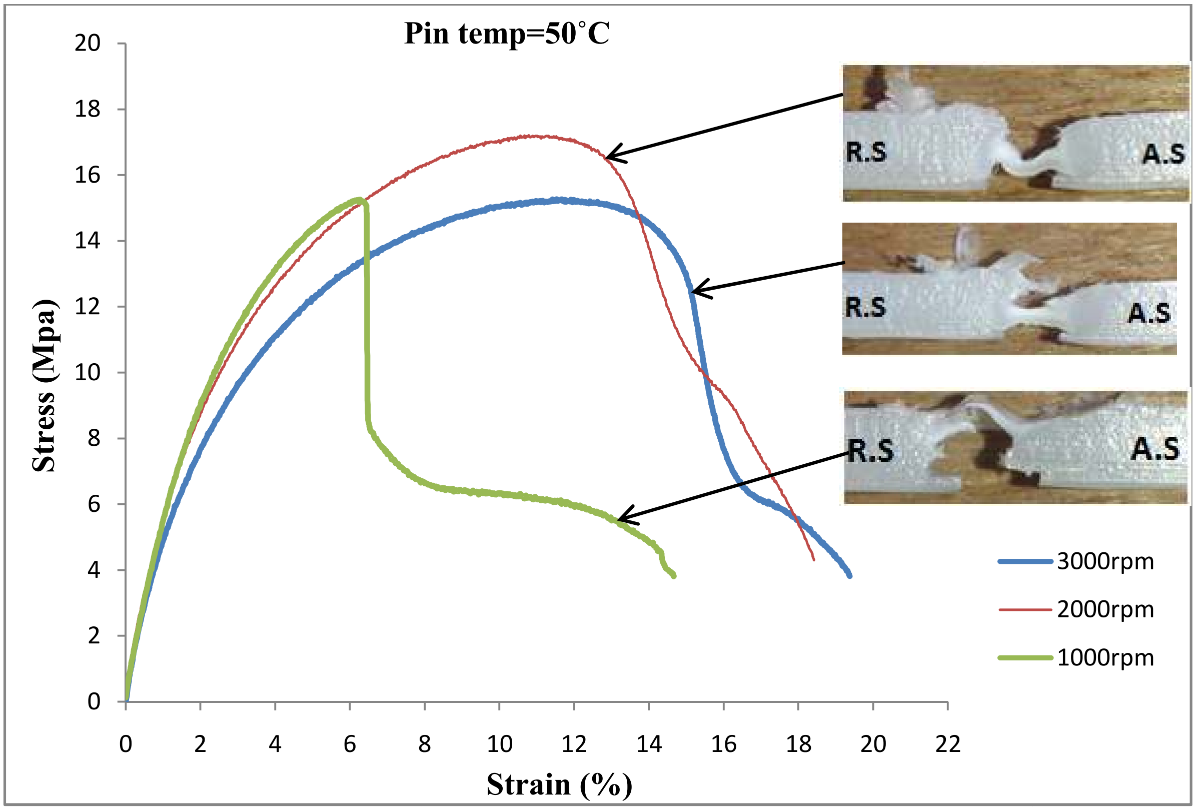


Fig. 21 Stress-strain curves and fracture locations at tool pin temperature of 50°C

The strength of the joints was reduced in all the parameters at a tool pin temperature of 50°C, shown in Fig. 21 compared to Fig. 19, since this temperature marked the onset of flash at the retreating side of the welds that reduced the strength, whereas in the former case material retention within the seam contributed to an increase in joint strength. There is a sudden reduction in strength after reaching the optimum strength in all joints in Fig. 19, which is not the case in Fig. 21, indicating that the strength is mainly governed by the ductility of the joints. At a tool pin temperature of 55°C the elongation (in percentage) of all the joints increased as shown in Fig. 22 compared to Fig. 21 and a fibrous structure was seen at the fracture locations. Compared to a tool pin temperature of 50°C, joint strength increased at 55°C for cases of 1000 and 2000rpm along with an increase in the amount of flash. The reason being that width of transition zone between seam and the base was narrow that governed strength of the joints, as discussed earlier. In all the four Figs. 18,19,21,22 the strength of the welds performed at

2000rpm, were well above the remaining curves indicating that this rpm had a strong effect on the mechanical properties (ie. joint strength).

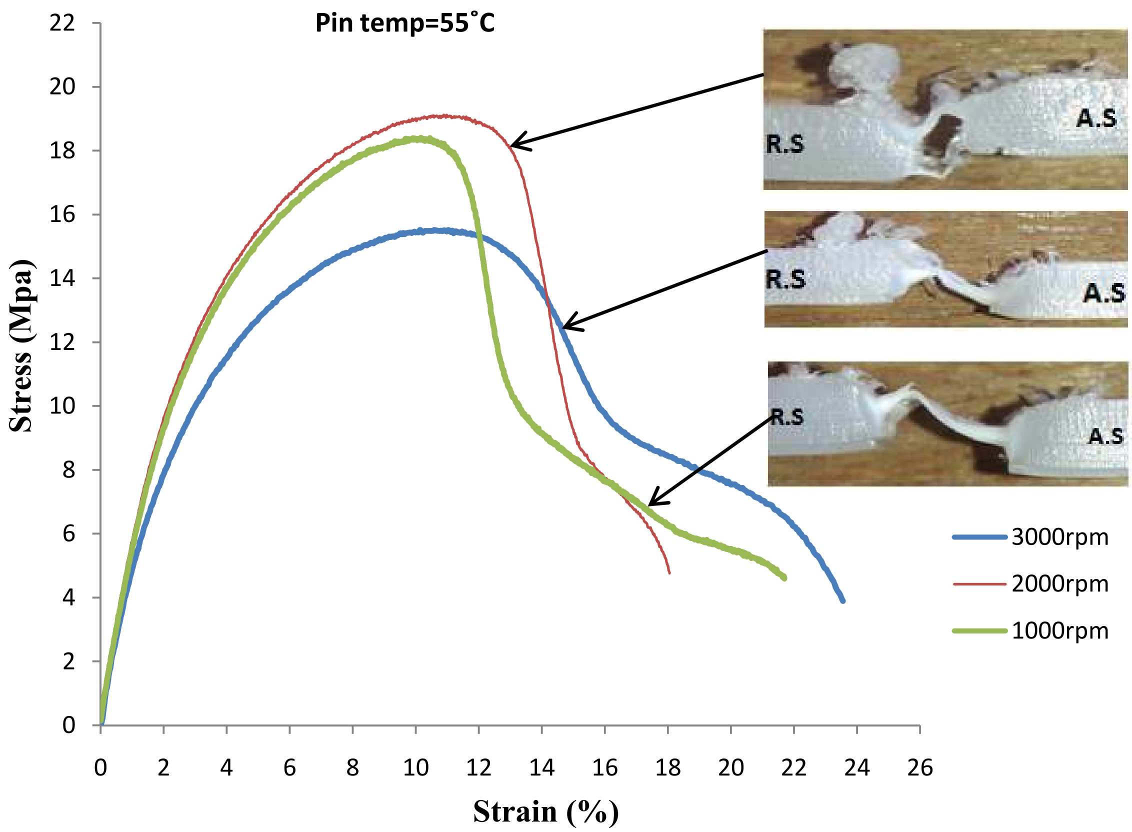


Fig. 22 Stress-strain curves and fracture locations at tool pin temperature of 55°C

4.7 Mechanism of material flow

Fig. 23 Representation of deformation zones in the transverse section of a tensile-tested sample.

The mechanism of material flow and weld formation is quite different in polymers compared to metals. Initially when the tool pin comes in contact with the work piece, plasticized material is formed which is surrounded by the bottom of the shoulder, cooler base material adjacent to the pin and the backing plate at the bottom. When linear motion is given to the tool, material from the leading edge is progressively plasticized and flows from the retreating side to the advancing side and vice versa. During this transfer, if the resistance to the flow of the plasticized material between the tool and the base material is high, material will flow out of the weld cavity which mainly happens on the retreating side, since the peripheral velocity of the tool is opposite to the traverse direction. At the advancing side material coming from the retreating side is extruded against the side wall base material where the pressure generated by the flow of material is sufficient to consolidate it giving rise to an almost continuous joint interface as shown in Fig. 23. The zone I indicates retreating side deformation zone and II indicates advancing side deformation zone.

In i-FSW, at high tool pin temperatures, the shoulder driven material is squeezed out rather than entering into zone II which happens in the case of metals [23] and it is mainly the pin-driven flow volume that contributes to joint formation. In metals the welds result from the coalescence of the shoulder and pin-driven material flow, that happens at low tool pin temperatures in the case of i-FSW process. At the advancing side, where the peripheral velocity of the pin and traverse of the tool have the same direction, a polymer element spends a longer time and is more influenced by the velocity field. Hence the grain structure is more refined and the resultant hardening is higher than on the retreating side. This is discused in detail in the following section on hardness of the welded joints.

4.8 Hardness Results

Micro Vickers hardness results were analysed measured at 2.4mm above the bottom of the specimens. Figures 24, 25 and 26 show the hardness distribution at the cross section of the joints welded with different process parameters. The weld zone (-5mm to +5mm) showed lower hardness value compared to the base material. The drop in hardness is due to softening of material caused by the heated tool. From Figs. 24 and 26, the drop in hardness at the stir zone or pin influence zone, which is approximately -2mm to +2mm from the center of the seam, is greater at a tool pin temperature of 55°C compared to that at 40°C, showing a decrease in hardness with increase in tool pin temperature.

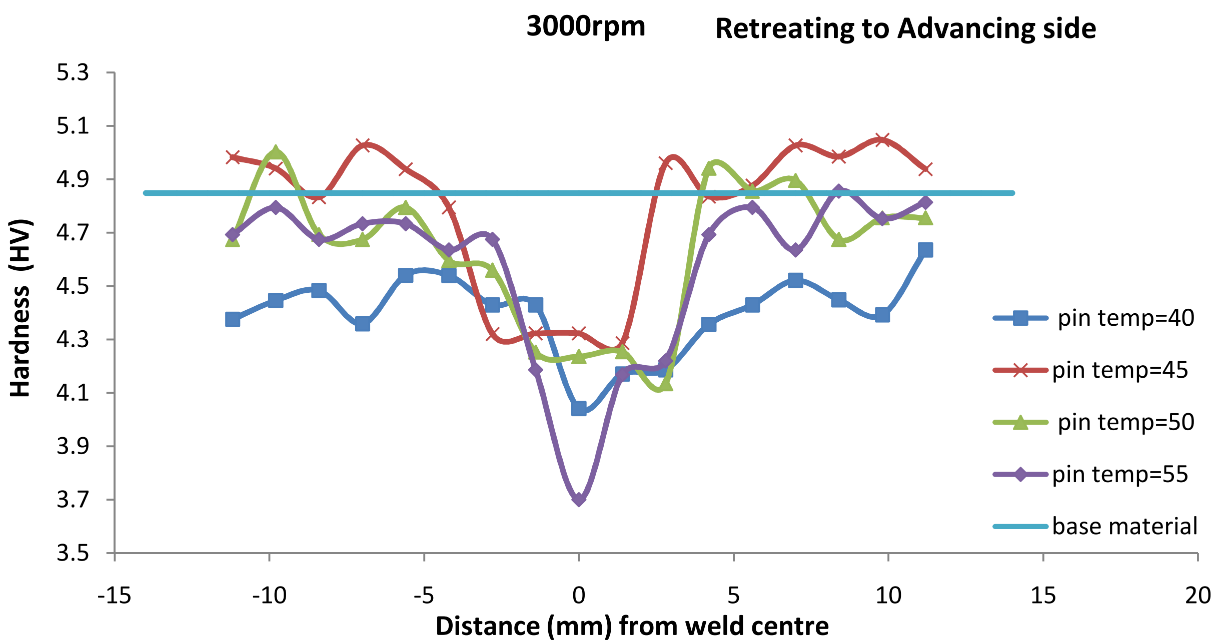


Fig. 24 Hardness plot at 3000rpm and different tool pin temperatures

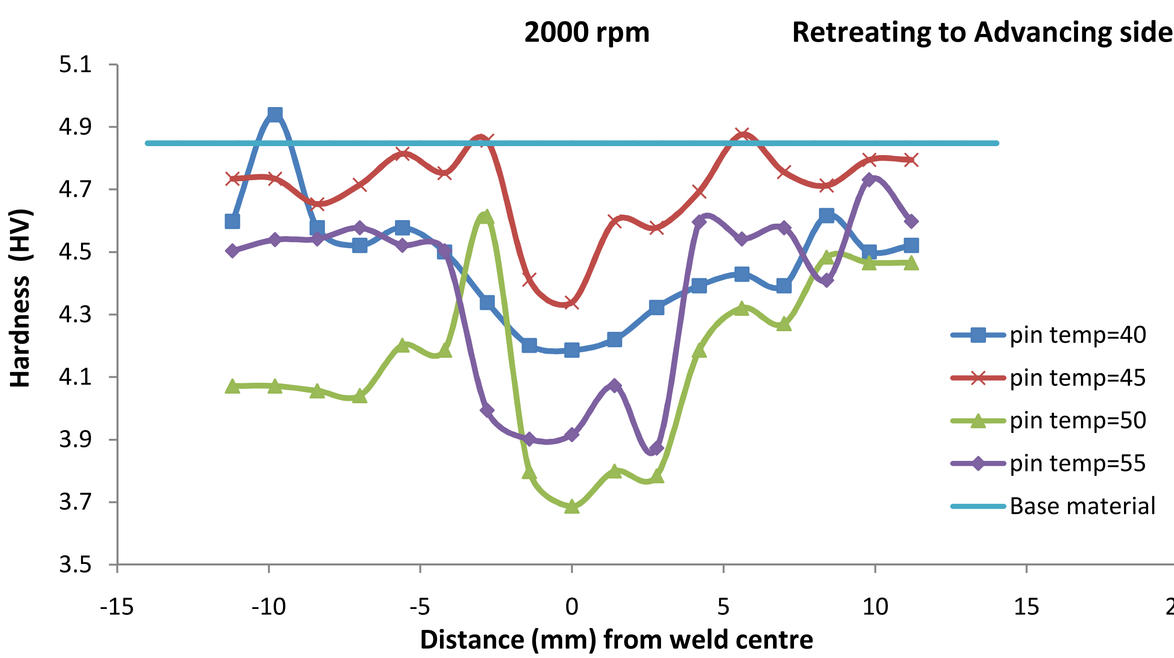


Fig. 25 Hardness plot at 2000rpm and different tool pin temperatures

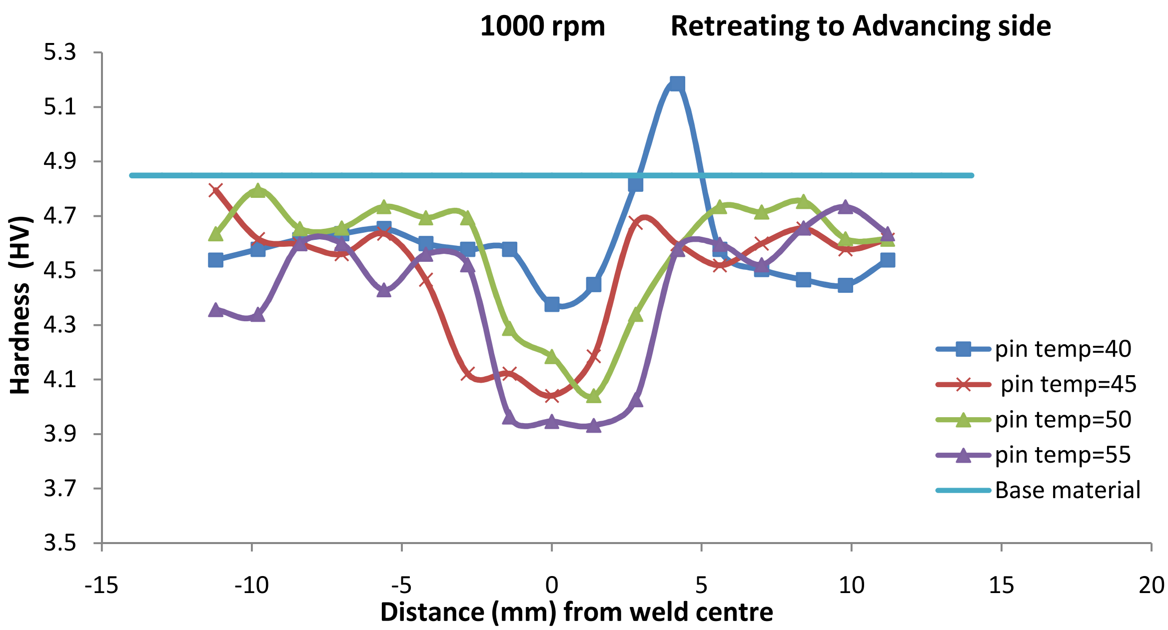


Fig. 26 Hardness plot at 1000rpm and different tool pin temperatures

The observed hardness pattern is mainly due to the ductile nature of the joints, as evidenced from the fracture locations. Hardness reduction ratio at the weld center [24] as shown in Eq. (1) is a good indicator in understanding the behavior of the weld joint.

$$Hardness\ reduction\ ratio\ at\ weld\ center(\%) = \frac{(Basematerial,HV - Weldcenter,HV)}{Basematerial,HV} \qquad (1)$$

Comparing Figs. 27 and 28, the hardness reduction ratio showed the same tendency with the joint efficiency; that is with a decrease in the hardness reduction ratio the joint efficiency increased at tool pin temperatures of 40°C and 45°C. This effect was not seen at higher tool pin temperatures of 50°C and 55°C for the cases of 1000rpm and 2000rpm. At these temperatures, fibers were observed at the fracture locations and the strength was mainly governed by the ductility of the joints. The joint produced at 1000rpm and 40°C was an exception, since in this case the fusion of seam and base material at the retreating side was improper (shown in Fig. 12) and the joint failed in a brittle manner. The minimum hardness reduction ratio, other than the above mentioned case was seen for the weld performed at 2000rpm and a tool pin temperature of 45°C which showed the highest value of joint efficiency among other welds, also shown in Fig. 28.

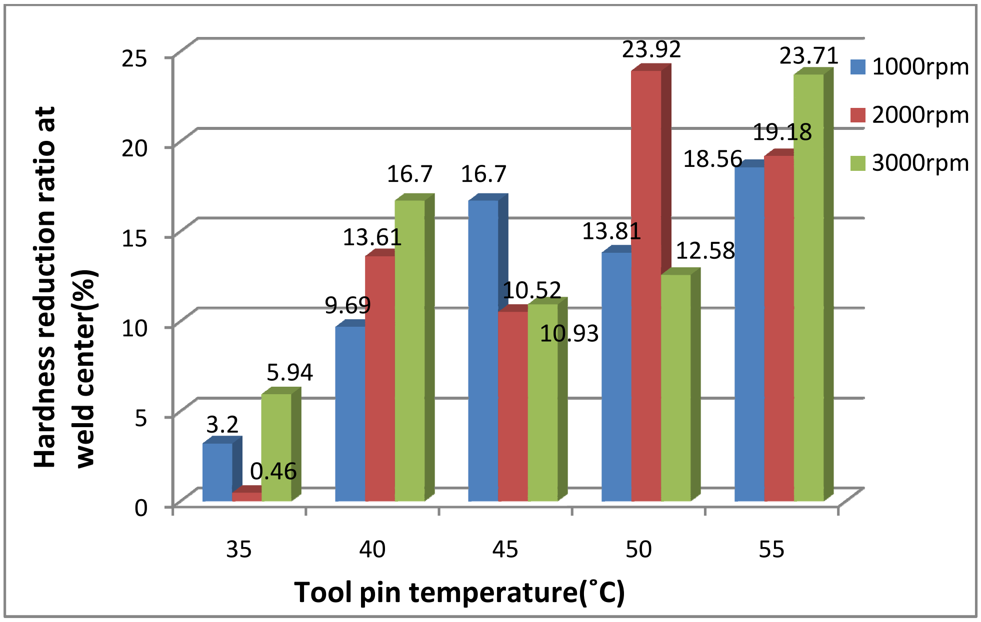


Fig. 27 Plot of Hardness reduction ratio at weld center for different parameters

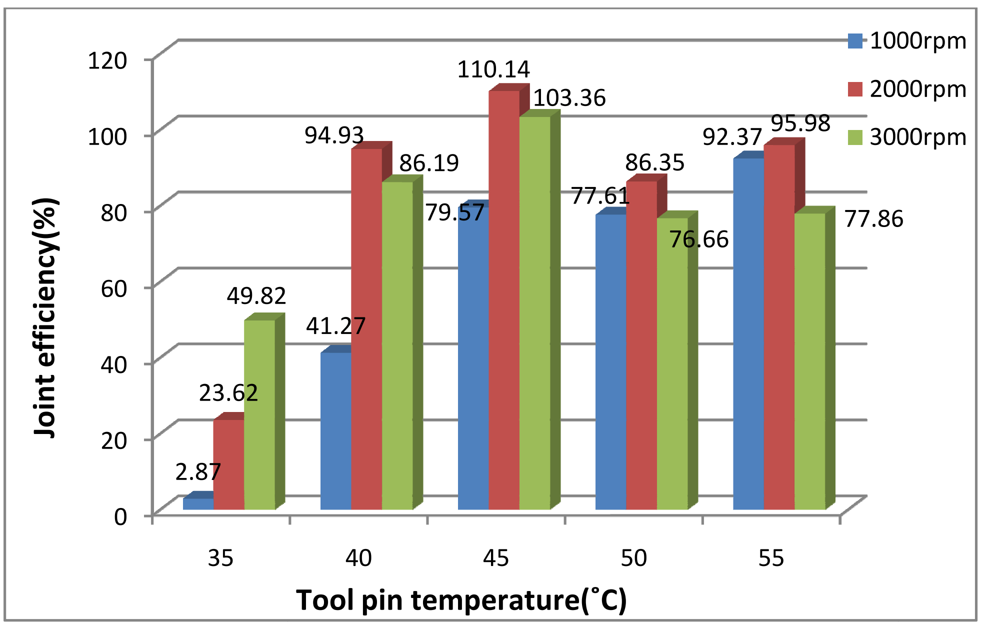


Fig. 28 Plot of joint efficiency for different parameters

It is evident from fig. 28 that the joint efficiency for the welds performed at room temperature (without tool heating) is quite low compared to the welds where the tool was induction heated.

Moreover without tool heating, joint efficiency at 3000rpm is higher indicating that frictional heat generated at higher rpm helps in good fusion between the seam and base material. Thus when the tool is induction heated, even at low rpm, little improvement in joint efficiency is obtained beyond a tool pin temperature of 45°C.

Chapter 5

Conclusions

The present work presents an attempt where the FSW tool is induction heated and precise temperature control is achieved through temperature feedback. The investigation also seeks to assess the efficacy of the proposed process through examination of joint efficiency, weld microstructure, type of fracture and weld hardness so that a better insight of the FSW of thermoplastics may be developed. FTIR and DSC tests were conducted to understand the structural and chemical changes respectively.

5.1 Conclusions

1. A new technique of joining thermoplastic using friction stir welding, namely Induction Assisted Friction Stir Welding (i-FSW) is introduced wherein an induction-heated tool welds plates of High density polyethylene in a butt configuration and few cases with zero/minimum amount of flash were reported.
2. Induction heating of tool enabled the plastic material to soften in a short time and to be easily stirred. As the tool pin temperature increases, the hardness at the stir zone decreases due to the ductile nature of the joints and softening at the stir zone. At high tool pin temperatures the strength of the joint is governed by the ductile behavior of the material. Turbulence of the material, caused by the stirring action of the tool, was the main factor that governed the strength of the welds at low tool temperature or high rotational speed conditions.
3. At low rotational speed and low tool pin temperature the heat generated was insufficient to soften and plasticize the polymer, resulting in improper fusion between the seam and the base material.
4. The optimum conditions for the maximum strength of the joints were a tool pin temperature of 45°C and a rotational speed of 2000rpm. Microstructural examination revealed that the strength of the friction stir welded joint will be close to that of the base material if the overall width of the transition zone is small with less complex morphology. Such welds also exhibit structural and chemical similarity with the base material evidenced from the DSC and FTIR results.

5. In i-FSW process, at high tool pin temperatures, the shoulder driven material is squeezed out rather than entering into advancing side deformation zone which happens in the case of metals and it is mainly the pin-driven flow volume that contributes to joint formation.
6. It is postulated that the effect of i-FSW on the crystallization mechanism could be an important factor, determining the mechanical performance of the joints, which merits further investigation in future studies.

5.2 Future Scope

1. The effect of tool tilt angle, that has a strong influence on retainment of plastic material within the weld zone, can be studied on i-fsw process.
2. The effect of Joint strength and Micro hardness of the joints can be studied further with tool rotational speeds higher than 3000rpm and feed rates less than 50mm/min.

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