Surface Composite Fabrication of Carbon Steel via Friction Stir Processing

M.Tech. Dissertation

BY

Chandan Kumar
2K15/PIE/05

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Submitted in partial fulfillment of the requirements for the award of the degree

of

Master of Technology

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CERTIFICATE

I hereby certify that the work which is being presented in the M.Tech. Dissertation entitled **"Surface Composite Fabrication of Carbon Steel via Friction Stir Processing",** in partial fulfillment of the requirements for the award of the **Degree of Master of Technology in Production and Industrial Engineering** and submitted to the Department of Electrical Engineering of Delhi Technological University is an authentic record of my own work carried out under the supervision of **Dr. N.Yuvraj, Assistant Professor ME Department**.

The matter presented in this report has not been submitted by me for the award of any other **Degree/Diploma** elsewhere.

Chandan Kumar
2K15/PIE/05

Date: , 2017

Guided By:

Dr. N. Yuvraj
Assistant Professor
ME Dept., DTU

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Apart from the efforts of me, the success of any project depends largely on the encouragement and guidelines of many others. I take this opportunity to express my gratitude to the people who have been instrumental in the successful completion of my M. Tech thesis.

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I must give my high, respectful gratitude to my supervisor, Dr. N.Yuvraj for his guidance, supervision and help throughout this project. I have learned a lot throughout this course, with many challenging yet valuable experience.

Chandan Kumar

2K15/PIE/05

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ABSTRACT

Fabrication of metal matrix composites (MMCs) with improved mechanical properties and modified microstructures has attracted many attentions. One of the novel methods to produce MMCs is friction stir processing (FSP). FSP is a method of changing the properties of metal through intense, localized plastic deformation. This deformation is produced by inserting a non-consumable tool into the workpiece, and revolving the tool in a stirring motion as it is pushed laterally through the workpiece. Friction between the shoulder and work piece results in localized heating which raises the temperature of the material to the range where it is plastically deformed. During this process, severe plastic deformation occurs and due to thermal exposure of material, it results in a significant evolution in the local microstructure.

The present work aims the fabrication of a Steel/TiCanano composite by introduction fantasized TiC powder into the stir zone. The fabricated nano composite layer exhibited micro hardness value of ~ 320HV. This is greater than 235 HV of the friction stir processed layer without adding of TiC powder. In addition, a significant improvement in wear resistance of the nanocomposite layer was observed. The properties of the surface composite are attributed due to the uniform dispersion of hard TiC reinforcements in a matrix of Ultra fine dynamically recrystallized grains.

1. Introduction

1.1 Significance of Friction Stir Processing

Friction stir processing (FSP) is a grain refinement technique. This method comes from the FSW (Friction Stir Welding) technology which was developed by Wayne Thomas from the Welding Institute (TWI Ltd.) in Cambridge. The FSW method is a friction welding technique with the stirring of the metal, and is used to join metals in solid state as performed Thomas et.al(2000). In both the FSP and FSW methods same principle is used i.e. the heat generated as a result of friction between the tool and the material surface which heats up and plasticises the material. The tool is usually made up of high speed steel (HSS), cubic boron nitride (B_4C), tungsten carbide (TiC), etc. When the tool shank is put into rotation it sinks slowly into the joint area (in FSW technology) or into the material being modified (in FSP technology). At First Phase, only the front shank surface gets in contact with the material being modified, and later the side shank surface and the surface of the retaining collar. Because of Friction between the tool and the material being modified a number of thermodynamic processes occur like heating up and cooling down of the metals at different rates, plastic strains, and the flow of processed material around the tool as described by Kalemba et.al(2014). The heat plasticises the processed material below the melting point. The thermal effect accompanying structural changes, including the shape and the dimensions of the zone being modified, depend not only on the treatment parameters (RPM of the tool), but also on the construction and the dimensions of the tool. The friction between the shank of the tool and the surface of material constitutes approx 20% of the total heat generated during the process, and the rest is the heat released due to the friction between the surface of material and the front surface of the retaining collar as performed by Lacki et.al(2013)

Though FSP was initially engaged for micro structural refinement of aluminum and magnesium alloys. It is also very useful process for also fabricating composites. Mishra et.al(2003) fabricated the SiC/Al surface composites by FSP, and showed that SiC particles were very well distributed in the Al matrix, and good bonding with the Al matrix was generated. Friction stir processing is a technique for subsurface structural modification of different materials including not only alloys of aluminium but also steels and other alloys.

One of the route is using the FSP by introducing the reinforcing hard particles into the subsurface layers. Another one is using the FSP for refining the grain structure. It is a promising technique for obtaining hardened layers on the machine components and in this regard it may be analogous to the nano structuring burnishing where the subsurface of steel sample is hardened to the depth of several microns below the surface but such processing technique requires setting up the process parameter values to very high accuracy. In case of FSP, the thickness of a modifiedalayer is determined only by the strength and geometry of the FSP tool

Various approaches have beenatried in Friction Stir Processing to fabricate a defect free surface composite with the uniform distribution of reinforcements in the alloy. The FSP tool probes commonly used in surface composites processing are circular, square and triangular. A square probe produces more pulse per sec compared with a triangular probe, whereas no such pulsating action is observed when cylindrical, tapered or threaded probes are used as studied by Elangovan et.al(2015).

There are various conventional methods for fabricating composites such as powder metallurgy, plasma spraying, laser melt treatment, stir casting etc but these techniques lead to the deterioration of composite properties due to inter facial interaction between the reinforcement and the metal matrix as discussed by Mishra et.al(2003).

FSP is one of the solid state processing techniques which have proved its potential in fabrication of all variants of surface composites with little or no inter facial reaction with the reinforcement. FSP offers many advantages over conventional and also the newer techniques of material processing which include being a single step process, use of the simple and inexpensive tool, less processing time, use of existing and readily available machine tool technology, adaptability to robot use, suitability to automation, being energy efficient and environmental friendly. Though, the limitations of FSP are being reduced by intensive research and development, it still has few limitations like the rigid clamping of work pieces, backing plate requirement, and the keyhole tightening at the end of each pass.

The potential applications of the surface composites can be found in automobile, aerospace, marine and power generation industries.

2. Literature Review

Friction stir technology is a revolution in the field of welding. This innovative technique produces very fine grains in weld zones. If this could be used as a processing technique, it would replace the existing traditional, complex and expensive processing techniques especially for aluminum and other alloys. FSP can enhance super plasticity in the materials.

2.1 General idea of the friction stir technology

This section gives an insight into the innovative technology called friction stir technology. The action of rubbing two objects together causing friction to provide heat is one dating back many centuries as stated by Thomas et al (2001). The principles of this method now form the basis of many traditional and novel friction welding, surfacing, and processing techniques. The friction process is an efficient and controllable method of plasticizing a specific area on a material, and thus removing contaminants in preparation for welding, surfacing/cladding or extrusion. The process is environmentally friendly as it does not require consumables (filler wire, flux or gas) and produces no fumes. In friction welding, heat is produced by rubbing components together under load. Once the required temperature and material deformation are reached, the action is terminated and the load is maintained or increased to create a solid phase bond. Friction is ideal for welding dissimilar metals with very different melting temperatures and physical properties.

The schematic illustration of FSP operation is shown in Fig. 2.1. First of all, the no consumable cylindrical tool is rotated to a predetermined RPM. After that, the tip of the rotating tool is plunged into the workpiece which performs two primary functions: (1) Heating and (2) Deformation of workpiece material. The tool’s shape consists of a small diameter pin which is fully inserted into the metal, and a concentric, larger diameter shoulder which is intended to prevent upward displacement of the material at the surface of the workpiece. As the tool penetrates the surface, the rotating pin creates frictional and adiabatic heating. This combination of heating softens the material so that the tool can further penetrate the material. As the tool rotates, it induces a stirring action and material flow around the pin. The depth of penetration is controlled by the tool's shoulder and the length of the pin. Now, the shoulder makes contact with the material's surface. This expands the hot zone due to heating caused by the rotating tool shoulder. The shoulder prevents the upward flow of material caused by the stirring action of the pin results in forging action on the deforming. material. When the tool is fully inserted into the workpiece, it then traverses across the metal at a specific rate, IPM (inches per minute).

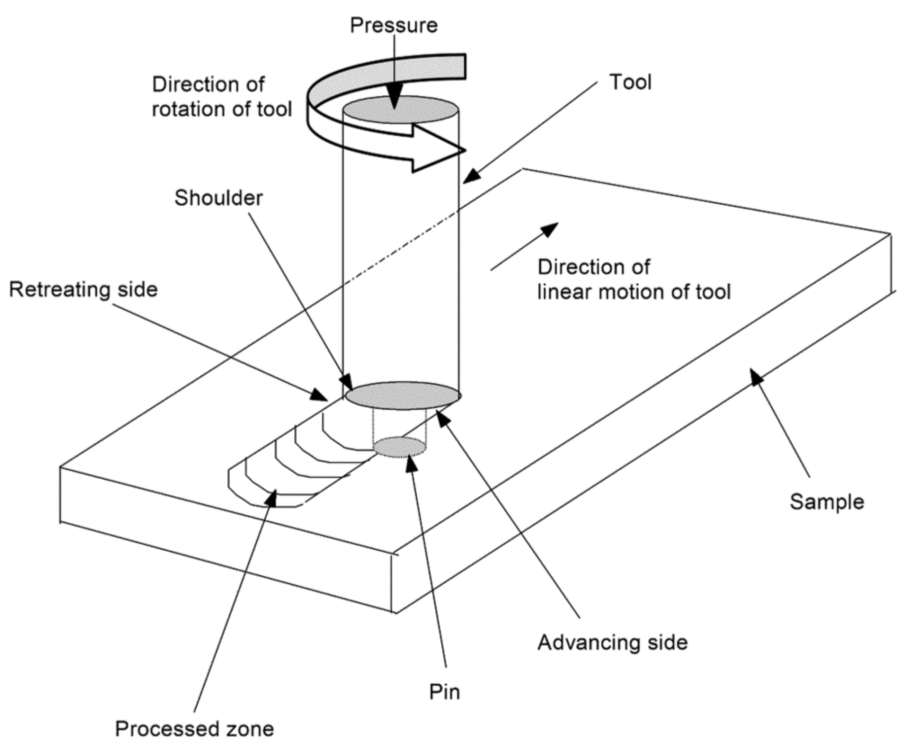

Figure 2.1 Schematic Illustration of Friction Stir Processing Technique

During FSP, the rotating tool provides a continual hot working action, plasticising the metal within the narrow region surrounding the pin, while transporting the metal from the leading face (advancing side) of the pin to its trailing edge (retreating side). It is important to note that the metal never melts. The peak temperatures achieved in this process are typically 80 to 90 percent of the melting temperature. After the tool passes by, the processed material cools and exhibits a refined and homogenized microstructure. Essentially, FSP is a thermomechanical metal working process that changes the local properties with minimal influence on the properties in the rest of the material .

2.1.1 FSP Process Parameters

FSP includes following types of process parameters which affect microstructural characteristics and mechanical properties of magnesium alloy. These are

- Tool Geometry: - In FSP, the tool performs two functions of localized heating and material flow. It includes tool pin shape (threaded cylindrical or square) and shoulder shape (concave or convex).
- Welding Parameter: - It includes tool rotational speed and tool traverse speed along the welding line. The tool rotation results in stirring and mixing of softened materials around the pin, and the tool translation moves them from the front to the back of the pin and finishes the weld.
- Tool tilt or tilt angle
- Tool penetration depth

2.1.2 Processed Region Terminology

It is also common to discuss the processed area in FSP with specific terminology. As shown in fig.2.2, three distinct zones are identified after FSP of aluminum alloy i.e., Nugget or Stirred Zone (SZ), Thermo Mechanically Affected Zone (TMAZ) and Heat

Affected Zone (HAZ). The microstructural changes in various zones have significant effect on post-weld mechanical properties

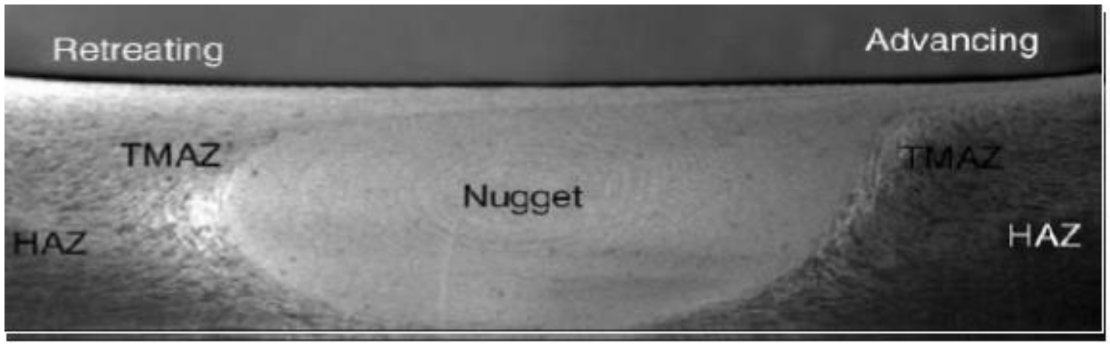

Figure 2.2 Various zones in the cross-section of FSP 7075Al-T651

Intense plastic deformation and frictional heating during FSW/FSP result in the generation of a recrystallised fine-grained microstructure within stirred zone. This region is usually referred to as nugget zone (or weld nugget) or dynamically recrystallized zone (DXZ). Unique to the FSW/FSP process is the creation of a transition zone—thermo-mechanically affected zone (TMAZ) between the parent material and the nugget zone, as shown in Fig.2.3. The TMAZ experiences both temperature and deformation during FSP. The TMAZ is characterized by a highly deformed structure. The parent metal elongated grains were deformed in an upward flowing pattern around the nugget zone. Beyond the TMAZ there is a heat-affected zone (HAZ). This zone experiences a thermal cycle but does not undergo any plastic deformation.

2.2 Material Properties and Designation

About steel.

The term steel is used for many different alloys of iron. These alloys vary both in the way they are made and in the proportions of the materials added to the iron. All steels, however, contain small amounts of carbon and manganese. In other words, it can be said that steel is a crystalline alloy of iron, carbon and several other elements, which hardens above its critical temperature. Like stated above, there do exist several types of steels which are (among others) plain carbon, stainless steel, alloyed steel and tool steel**.**

Plain carbon steel.

Carbon steel is by far the most widely used kind of steel. The properties of carbon steel depend primarily on the amount of carbon it contains. Most carbon steel has a carbon content of less than 1%. Carbon steel is made into a wide range of products, including structural beams, car bodies, kitchen appliances, and cans. In fact, there are 3 types of plain carbon steel and they are low carbon steel, medium carbon steel, high carbon steel, and as their names suggest all these types of plain carbon steel differs in the amount of carbon they contain. Indeed, it is good to precise that plain carbon steel is a type of steel having a maximum carbon content of 1.5% along with small percentages of silica, sulfur, phosphorus and manganese

General properties of plain carbon steel.

Generally, with an increase in the carbon content of 0.01 to 1.5% in the alloy, its strength and hardness increases but still such an increase beyond 1.5% causes an appreciable reduction in the ductility and malleability of the steel.

Low carbon steel or mild steel, containing carbon up to 0.25% responds to heat treatment as improvement in the ductility is concerned but has no effect in respect of its strength properties.

Medium carbon steels, having carbon content ranging from 0.25 to 0.70% improves in the machinability by heat treatment. It must also be noted that this steel is especially adaptable for machining or forging and where surface hardness is desirable.

High carbon steels, is steel-containing carbon in the range of 0.70 to 1.05% and is especially classed as high carbon steel. In the fully heat-treated condition, it is very hard and it will withstand high shear and wear and will thus be subjected to little deformation. Moreover, at maximum hardness, the steel is brittle and if some toughness is desired it must be obtained at the expense of hardness. Depth hardening ability (normally termed as hardenability) is poor, limiting the use of this steel. Furthermore, as it has been seen that hardness, brittleness, and ductility are very important properties as they determine mainly the way these different carbon content steels are used. Considering the micro structure of slowly cooled steel; for mild steel, for instance, with 0.2% carbon. Such steel consists of about 75% of pro-eutectoid ferrite that forms above the eutectoid temperature and about 25% of pearlite (pearlite and ferrite being microstructure components of steel). When the carbon content in the steel is increased, the amount of pearlite increases until we get the fully pearlitic structure of a composition of 0.8% carbon. Beyond 0.8%, high carbon steel contains pro-eutectoid cementite in addition to pearlite. However, in slowly cooled carbon steels, the overall hardness and ductility of the steel are determined by the relative proportions of the soft, ductile ferrite and the hard, brittle cementite. The cementite content increases with increasing carbon content, resulting in an increase of hardness and a decrease of ductility, as we go from low carbon to high carbon steels.

2.3 Surface Composite Fabrication Of Carbon Steel

Carbon steel is used as a structural material in industry and construction. However, the wear resistance of carbon steel is considered to be poor in certain applications. Dispersion of hard ceramic particles in the mild steel matrix can improve strength and wear resistance compared to those of the monolithic counterparts

Titanium carbide (TiC) is widely used in coatings due to its high hardness value, high strength, high rigidity, good wear resistance, low friction coefficient, high melting point and high chemical stability. Due to the mentioned features, TiC is extensively used as a reinforcing phase in metal matrix composite coatings, such as steel based composites. Iron and steels are relatively low-cost material and considered as a coating matrix with potential application prospect

In this study, TiC powder was used as a reinforcement material. The technique of FSP was employed for the production of steel/TiC composite surface layer on a mild steel work piece. The microstructure of the fabricated composite layers was characterized. In addition, mechanical assessment of the layers was carried out using hardness and pin-on-disk wear testing.

2.4 Wear and Wear Mechanism

Undesirable removal of material from operating solid surface is known as wear. Removal of material from operating solid surfaces by solid particles depends upon Load, Velocity, Environment, and Materials. Removal of material from operating solid surface by Fluid (liquid/gas) depends upon Velocity, pressure, Environment, and material.

As wear increases power losses increases, oil consumption increases, the rate of component replacement also increases. Ultimately, it reduces the efficiency of the system. Therefore, as far as possible wear should be minimized.

All wear process involves one or a combination of wear mechanisms including **abrasion**, **adhesion**, **fatigue**, and **oxidation** or other **tribo-chemical** actions.

Abrasive wear (wear by abrasion) is the most frequently encountered wear mechanism in the industry.

Mechanism of the abrasive wear-The mechanism of material removal in abrasive wear is basically the same as machining and grinding during a manufacturing process.At the onset of wear, the hard asperities or particles penetrate into the softer surface under the normal contact pressure. When a tangential motion is imposed, the materials in the softer surface is removed by combined effects of 'micro-ploughing', 'micro-cutting' and 'micro-cracking'. As a result, the worn surface is generally characterized by grooves and scratches .The wear debris often has a form of micro-cutting chips.

Adhesive wear -Engineering surface is never perfectly flat. The surface of a most highly polished engineering component show irregularities or asperities. When two such surfaces are brought into contact, the real contact actually occurs only at some high asperities which are a small fraction, e.g. 1/100 of the apparent contacting area.

Oxidation Wear- Metal surface is normally covered with a layer of oxide, which could prevent metal-to-metal contact, and thus avoid the formation of adhesion and reduce the tendency of adhesive wear. In this connection, the oxide is a favorable factor in reducing wear rate of metallic materials.

2.5 Literature Study

In this section, various investigations regarding Friction Stir Processing (FSP) of aluminum alloys, copper alloys, and steel are presented. Also, the friction-stir processing (FSP) technique applied to the development of surface composites of alloys reinforced with titanium carbide.

A basic understanding of the evolution of microstructure in the dynamically recrystallized region of FS material and relation with the deformation process variables of strain, strain rate, temperature and process parameters is very essential. This section would also give an insight into such studies.

ALUMINIUM

Mishra et.al(2001) extended the FSW innovation to process Al 7075 and Al 5083 in order to render them superplastic. They observed that the grains obtained were recrystallized, equiaxed and homogeneous with average grain sizes <5µm. They had high angles of misorientation ranging from 20 degrees to 60 degrees. They had also performed high-temperature tensile testing in order to understand the superplastic behavior of FSP aluminum sheets. Metal matrix composites reinforced with ceramics exhibit high strength, high elastic modulus and improved resistance to wear, creep and fatigue compared to unreinforced metals.

Mishra et al.(2003) experimented and proved that surface composites could be fabricated by friction stir processing. Al-SiC surface composites with different volume fractions of particles were successfully fabricated. The thickness of the surface composite layer ranged from 50 to 200µm. The SiC particles were uniformly distributed in the aluminum matrix. The surface composites have excellent bonding with the aluminum alloy substrate. The micro hardness of the surface composite reinforced with 27 volume % SiC of 0.7 µm average particle size was ~173 HV, almost double of the 5083Al alloy substrate (85 HV). The solid-state processing and very fine microstructure that results are also desirable for high-performance surface composites.

Thomas et al. (1996) presented a review of friction technologies for stainless steel, aluminum, and stainless steel to aluminum, which is receiving widespread interest. Friction hydro pillar processing, friction stir welding (FSW), friction plunge welding are some of these unique techniques. They observed that this technology made possible the welding of unweldable aluminum alloys and stainless steel feasible. Using this technology sheets up to 75mm thickness can also be easily welded.

Peel et.al (2003) reported the results of microstructural, mechanical property and residual stress investigations of four AA5083 FS welds produced under varying conditions. It was found that the weld properties were dominated by the thermal input (thermal excursion) rather than the mechanical deformation by the tool, resulting in a >30 mm wide zone of equiaxed grains around the weld line. Increasing the traverse speed and hence reducing the heat input narrowed the weld zone. It is observed that the recrystallization resulting in the weld zone had considerably lower hardness and yield strength than the parent AA5083. During tensile testing, almost all the plastic flow occurred within the recrystallized weld zone and the synchrotron residual stress analysis indicated that the weld zone is in tension in both the longitudinal and transverse directions. The peak longitudinal stresses increased as the traverse speed increases. This increase is probably due to steeper thermal gradients during welding and the reduced time for stress relaxation to occur.

The evolution of the fine-grained structure in friction-stir processed aluminum has been studied by Rhodes et.al. (2003) using a rotating-tool plunge and extract technique. In these experiments, the rotating tool introduced severe deformation in the starting grain structure, including severe deformation of the pre-existing sub-grains. Extreme surface cooling was used to freeze in the starting structure. Heat generated by the rotating tool was indicated as a function of the rotation speed and the external cooling rate. At slower cooling rates and/or faster tool rotation speeds, recrystallization of the deformed aluminum was observed to occur. The initial sizes of the newly recrystallized grains were in the order of 25–100 nm, considerably smaller than the pre-existing sub-grains in the starting condition. Subsequent experiments revealed that the newly recrystallized grains grow to a size (2–5μm) equivalent to that found in friction-stir processed aluminum, after heating 1–4 min at 350–450 °C. It is postulated that the 2–5 μm grains found in friction-stir welded and friction-stir processed aluminum alloys arose as the result of nucleation and growth within a heavily deformed structure and not from the rotation of pre-existing subgrains.

Sato et.al (2004) applied FSW to an accumulative roll-bonded (ARBed) Al alloy 1100. FSW resulted in the reproduction of fine grains in the stir zone and a small growth of the ultrafine grains of the ARBed material just outside the stir zone. FSW was reported to suppress large reductions of hardness in the ARBed material, although the stir zone and the TMAZ experienced small reductions of hardness due to dynamic recrystallization and recovery. Consequently, FSW effectively prevented the softening in the ARBed alloy which had an equivalent strain of 4.8.

Sansui et.al (2006)studied friction-stir processing of a composite aluminum alloy (AA1050) reinforced with titanium carbide powder. In this study, the friction-stir processing (FSP) technique was applied for the development of surface composites of aluminum alloy (AA 1050) reinforced with titanium carbide (TiC) powder of particle size range below 60 μm compressed into the groove. Rotational speeds of 1200 min–1 and 1600 min–1 and the travel rates of (100, 200 and 300) mm/min were used for the process. This study investigates the effect of processing parameters on the wear-resistance behavior of friction-stir processed Al-TiC composites. This was achieved through microstructural characterization using optical and scanning electron (SEM) microscopes equipped with Oxford energy-dispersion spectrometry (EDS) (Tuscan), micro hardness profiling and wear resistance tests. From the results, it was found that the processing parameters influenced the distribution of the TiC particles. The micro hardness profiling of the processed samples revealed an increase in the hardness value compared to the parent material. The wear-resistance test results confirmed the FSP technique enhanced properties in surface engineering. The hardness tests showed an improvement and an enhancement on the integrity of the processed materials due to the improvement in the evolving microstructure. The wear-resistance test results confirm the feasibility of using FSP for the optimization of surface properties of the composites produced.

COPPER

Sua et.al(2011) studied the developments of nanocrystalline structure in Cu during friction stir processing (FSP). It was performed in Copper plate thickness of 6mm. H13 steel and hardened to HRC 52 which was fabricated by the tool with shoulder diameter7.5mm, pin diameter2.5mm and pin length of 2.0mm. Copper plate(6mm) was reduced to 2mm in three rolling passes with 30 min anneals at 500 ◦C. after each rolling pass, providing a final annealed microstructure with a grain size of 70 nm. The tool was tilted to 2.5◦ whose direction is opposite to the traveling direction during a single processing pass on the Cu sheet at 800rpm and a travel speed of 120mm min^-Using small tools and imposing rapid cooling, nanocrystalline structures were successfully produced in pure copper in a single. step. The resulting microstructures consist of equiaxed, nearly random oriented grains surrounded by high-angle boundaries. The grain size ranges mainly from 50 to 300nm with an average size of about 109nm and 174nm respectively.

With aims to predict the effect of tool rotation rate and traverse speed on strain hardening behavior of friction stir welded copper joints were investigated using hardening capacity and strain hardening exponent concept by Khodaverdizad eh et.al(2012) Kocks–Mecking type plots were used to show different stages of strain hardening. FSW samples reveal higher hardening capacity and lower strain hardening exponent relative to the base metal. With increasing rotation rate and/or decreasing traverse speed, FSW samples show higher hardening capacity and lower strain hardening exponent. The strain hardening behavior was discussed by dislocation density and grain size variation during FSW.

Prediction of mechanical and wear properties of copper surface composites fabricated using friction stir processing was presented by Satishkumar et.al(2014) Pure copper plates of length100mm, width-50mm and thickness6mm & five different ceramic particles such as SiC, TiC, WC, and Al2O3 were used. A tool made of double tempered H13 hot working steel with a cylindrical pin profile. They aimed towards successfully applying FSPed to prepare copper surface composites reinforced with a variety of ceramic particles such as SiC, TiC, B_4, C, WC, and Al_2O_3. Empirical relationships were developed to predict the effect of FSP parameters on the properties of copper surface composites such as the area of the surface composite, micro hardness and wear rate. B4C reinforced composites have higher micro hardness and lower wear rate. Higher tool rotational speed, lower traverse speed, and minimum groove width yielded higher area of the surface composite.

Ahuja et.al (2015) studied Friction stir forming to fabricate copper tungsten composite. Oxygen free copper (C1100) blocks with a dimension of 80 mm×50 mm×12 mm was used for conducting the FSP. 100 mm long problems tool with an 8◦ concave shoulder was manufactured from H13 tool steel. A mid-range value of 0.05 mm plunge depth, 3◦ tilt angle, 970 rpm tool rotation speed and 100 mm min−1 tool traverse speed, was set as the reference parameter combination. Tungsten Copper composite of copper was fabricated through probe less tool aided friction stir forming (FSF). Preliminary FSF of copper was performed by varying the tool plunge depth and tilt angle. Tool rotation and traverse speed were kept constant at 970 rpm and 100 mm min−1 respectively. No plastic deformation occurred at the low plunge depths of 0.025 mm and 0.05 mm with 1◦ tool tilt angle. Substantial encapsulation of the cavity can be observed at the higher tool rotation speed. Grain refinement induced work hardening was observed in the copper close to the interface. The bond strength of the Cu–W mechanical interlock fabricated by FSF was determined to be 130 MPa.

Shen et.al (2010) studied the effect of welding speed on microstructure and mechanical properties of friction stir welded copper, Materials, and Design. The base metal (BM) used in the experiment was a pure copper plate (under the 1/2H condition) of 3mm thickness. The rotation tool was made of high-speed tool steel, with a pin (φ/3 x 2.85 mm) having standard right hand threads and a shoulder (/12 mm) having a concave profile. FSW was conducted at a constant rotation rate of 600 rpm together with different welding speeds of 25, 50, 100, 150 and 200 mm/min. Influence of welding speed on microstructure and mechanical properties of the joints was investigated. As the welding speed increased, the grain size of nugget zone first increased and then decreased, the thermomechanically affected zone became narrow and the boundary between these two zones got distinct, but the heat affected zone was almost not changed. The ultimate tensile strength and elongation of the joints increased first and decreased finally with increasing welding speed, but the effect was little when the welding speed is in the range of 25–150 mm/min.

Effect of Single and Multiple Pass Friction Stir Processing on Microstructure, Hardness and Tensile Properties of a 99.99% Cu with Carbon Nano Tubes was studied by Arulmoni et.al(2015). Dimensions of the copper plate that was used for processing were 200 mm x 74 mm x 5 mm. The tool Material used was H13 steel with shoulder diameter 15mm, threaded pin diameter 8 mm, pin length 2.5 mm. Tool rotational speed 960 rpm, tool angle 20 and table traverse speed 25 m / min. The states of development of FSP for processing of Copper with carbon nanotubes are addressed. This paper investigates the parameters affecting the friction stir processed copper with carbon nano tubes and enhancement of the microstructure, hardness and tensile properties of the composite material. The behavior of Copper with carbon nano tubes has been studied with a single pass, double passes, and triple passes.

CARBON STEEL

Microstructure and mechanical properties of steel/TiC composite surface layer produced by friction stir processing by Ghasemi-Kahrizsangi et.al (2012). A steel/TiC composite surface layer with ultra fine grains of less than 600 nm was fabricated on a mild steel substrate by introduction of TiC powder into the stir zone employing four passes of friction stir processing. TiC clusters were formed after the first pass. The sequential break-up of clusters and refinement of matrix grains were caused by subsequent FSP passes. A near uniform dispersion of nano-sized TiC particles was achieved after the fourth pass. The fabricated nano-composite layer exhibited a maximum micro hard- ness value of ~ 450HV; this is much greater than 185 and 130HV of the friction stir processed layer without the introduction of TiC powder and as-received substrates, respectively. Moreover, a significant improvement in wear resistance of the composite layer was observed as compared with that of the as-received sub- state. The enhanced properties are attributed to the uniform dispersion of hard nano-sized TiC reinforcements in a matrix of Ultra fine dynamically recrystallized grain.

Aldajah et.al(2009) studied the effect of Friction Stir Processing on tribological performance of High carbon steel. Friction stir processing (FSP) was applied to 1080 carbon steel as a means to enhance the near-surface material properties. The process transformed the original pearlite microstructure to martensite, resulting in significant increase in surface hardness. This surface hardening produced a significant benefit for friction and wear behavior of the steel as measured by unidirectional sliding ball-on-flat testing. Under dry sliding, FSP reduced friction coefficient by approximately 25% and wear rate by an order of magnitude. Under oil lubrication, FSP had only a marginal effect on friction, but it reduced wear rates by a factor of4.The improvement in tribological performance of 1080 steel by FSP technique is attributed to the reduced plasticity of the near-surface material during sliding contact. Friction stir processing when applied to 1080 carbon steel resulted in significant changes in the near surface microstructure from pearlite to martensite up to a depth of 5 mm. This microstructural change resulted in increased hardness of 1080 steel from 3 GPa to about 7 GPa as expected since martensite is much harder than pearlite. The improvement in surface mechanical properties was observed to translate to improvement in friction and wear behavior as measured by a unidirectional sliding ball-on-flat contact configuration. Under dry contact, FSP reduced friction coefficient by about 25% and wear rate by an order of magnitude. Under oil lubricated conditions, FSP had only a marginal impact on friction since it depends largely on the lubricant. Significant reduction in wear rate was observed in FSP surfaces even under oil lubrication.

Effect of titanium additions to a low carbon, low manganese steels on sulphide precipitation was studied by Aminorroaya-Yamini (2008) . If sufficient amounts of titanium are used, titanium carbide can provide more precipitation strengthening than either Nb or V carbides. However, because higher levels of precipitation strengthening are generally associated with reduced toughness, grain refinement would be necessary to improve toughness. Titanium is a moderate grain refiner (compared to Nb and V in hot rolled steels), and the high levels of precipitation strengthening of titanium micro alloyed steels result in a severe penalty in toughness. The use of only titanium as a strengthener in high strength hot rolled strip has resulted in unacceptable variability in mechanical properties.

Enhanced mechanical properties of medium carbon steel casting via Friction Stir Processing and subsequent annealing studied by Xue et.al(2016). This study provides an effective surface processing technology to increase the local mechanical properties of medium carbon steel castings. The ultrafine dual-phase structure of the ferrite and the martensite was obtained in the processed zone with a depth of 1 mm via submerged friction stir processing (FSP). Significantly enhanced yield strength (YS) of 2070 MPa was achieved in the FSP steel compared to that of the base material (BM) with a relatively low YS of 590 MPa, but a low uniform elongation (UE) of 3.0% was achieved compared to that of the BM (9.4%). After annealing, obvious carbide precipitation was observed in the original quenched martensite phases. Therefore, good strength-ductility synergies with high YS of 1020 MPa and 925 MPa, and acceptable UE of 5.9% and 9.3% were achieved in the FSP steel after annealed for 2 h at 500 °C and 600 °C, respectively. Medium carbon steel was successfully friction stir processed at a tool rotation rate of 400 rpm and a traverse speed of 50 mm min 1 with additional rapid water cooling, and an obvious PZ with a depth of about 1.5 mm was obtained. The FSP steel was characterized by the ultrafine dual-phase structure of the ferrite and the martensite, which exhibited quenched state.

3. Background and Motivation

3.1 Motivation

Many research has been done in studying the microstructures produced by severe plastic deformation in metals including those produced by Solid State technique generally aluminum and magnesium as discussed by Tarrassov et.al(1999) . More research is required in the field of Friction Stir Processing in different materials especially the high melting point ones. Most of the literature available is focusing on the effect of mechanical properties of the material being modified by FSP, very few literatures are available on the effect of usage of different tool probes on the mechanical and tribological properties of the material being modified by FSP. This work aims at studying the fabrication of surface composite of medium carbon steel (C45) via Friction Stir Processing and determining its mechanical and tribological characterization (Wear, Micro hardness, etc) and also examine its microstructure. The fabrication has been performed by using different tool probes (Square, Circular, and Triangular) and also the effects have been compared.

3.2 Material Specifications:

<u>International grades of the steel used:</u>

BS: C45, 080M46, 50CS

AFNOR: C45, 1C45, AF65C45

SAE: 1045

Application:

1045 is a medium carbon steel used when greater strength and hardness is desired than in the rolled condition. It is used for mechanical engineering and automotive components. It is Used in gears, shafts, axles, bolts, studs, and machine parts.

Chemical Composition (Typical analysis in %):

C	0.42-0.5
Si	<0.40
Mn	0.50-0.80
P	<0.045
S	<0.045
Cr	<0.40
Mo	<0.10
Ni	<0.40

Table 0.1 Chemical Composition of AISI 1045 Steel

4. Experimental Procedure

4.1 Process Parameters

Based on literature review and trial works the following process parameters were fixed for my experimental work :

Table 5.1: Process Parameters used in Friction Stir Processing

Process Parameters	Value
Transverse Speed of the Tool	48 mm/min
No of Passes of Tool	1
Rotational Speed of the Tool	800

Table 4.1 Process Parameters used in Friction Stir Processing

4.2 Machine Setup:

An indigenously developed friction stir welding machine (R V machine tools, FSW-4T-HYD, shown in Fig. 4.1) is used for Friction Stir Processing. Specifications of the machine are as follows:

- Power – 15 HP
- Load capacity – 40 KN
- Rpm – 3000
- Clamps – hydraulic actuated. The clamps are used to hold the job firmly

- Colet used for holding the tool could hold a cylindrical (tapered/un-tapered) tool of diameter 20mm.

Figure 4.1 **Friction Stir Welding Machine**

4.3 FSP Tool Specifications

The tool used during Friction Stir Processing has following specifications:

- Tool Material: Tungsten Carbide
- Shoulder diameter: 20 mm
- Pin size: 6 mm (diagonally)
- Pin profile: Cylindrical, Square, and Triangular
- Pin length: 2 mm

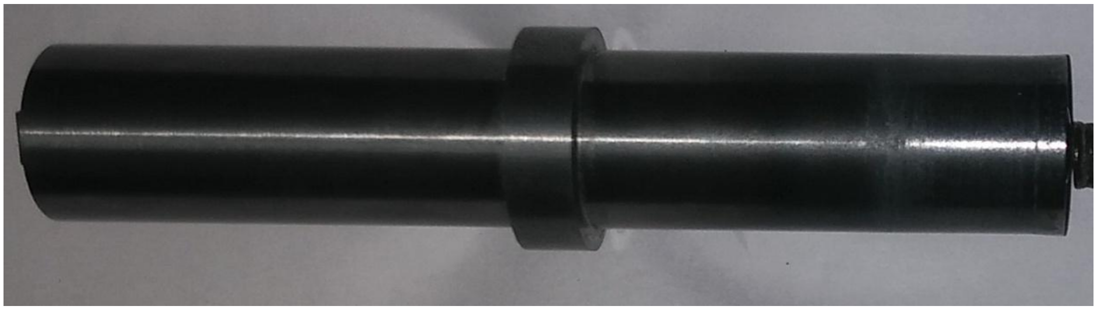

Figure 4.2 A Manufactured FSP tool

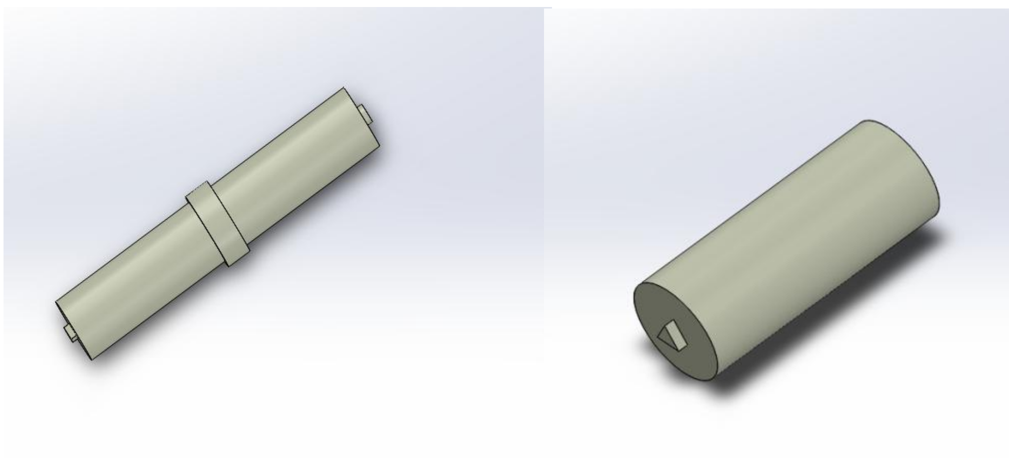

Figure 4.3 Solid Works Model of FSP Tool

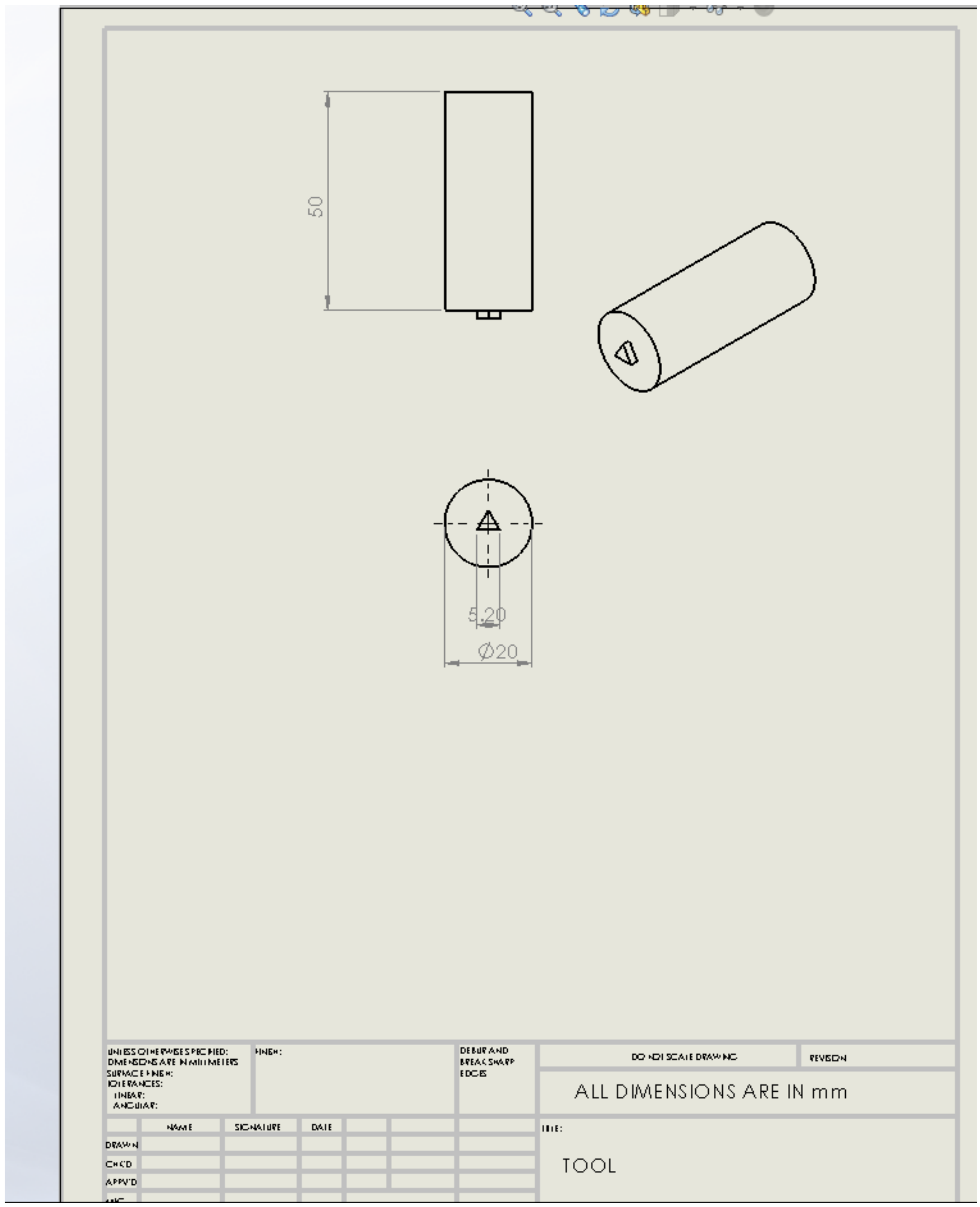

Figure 4.4 Draft of Design tool (Triangular Probe) using Solid Works

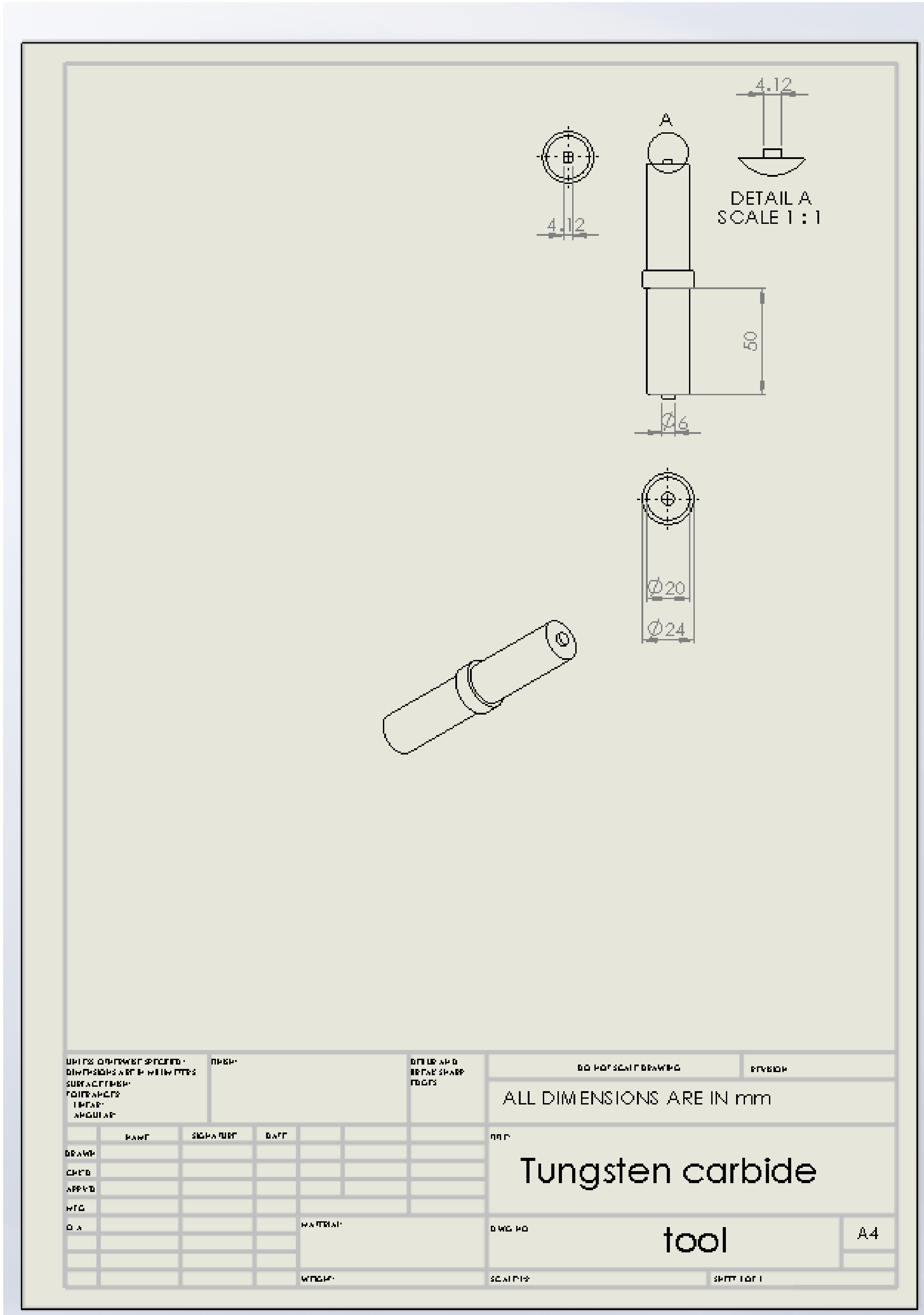

Figure 4.5 Draft of designed tools (Square and Circular)

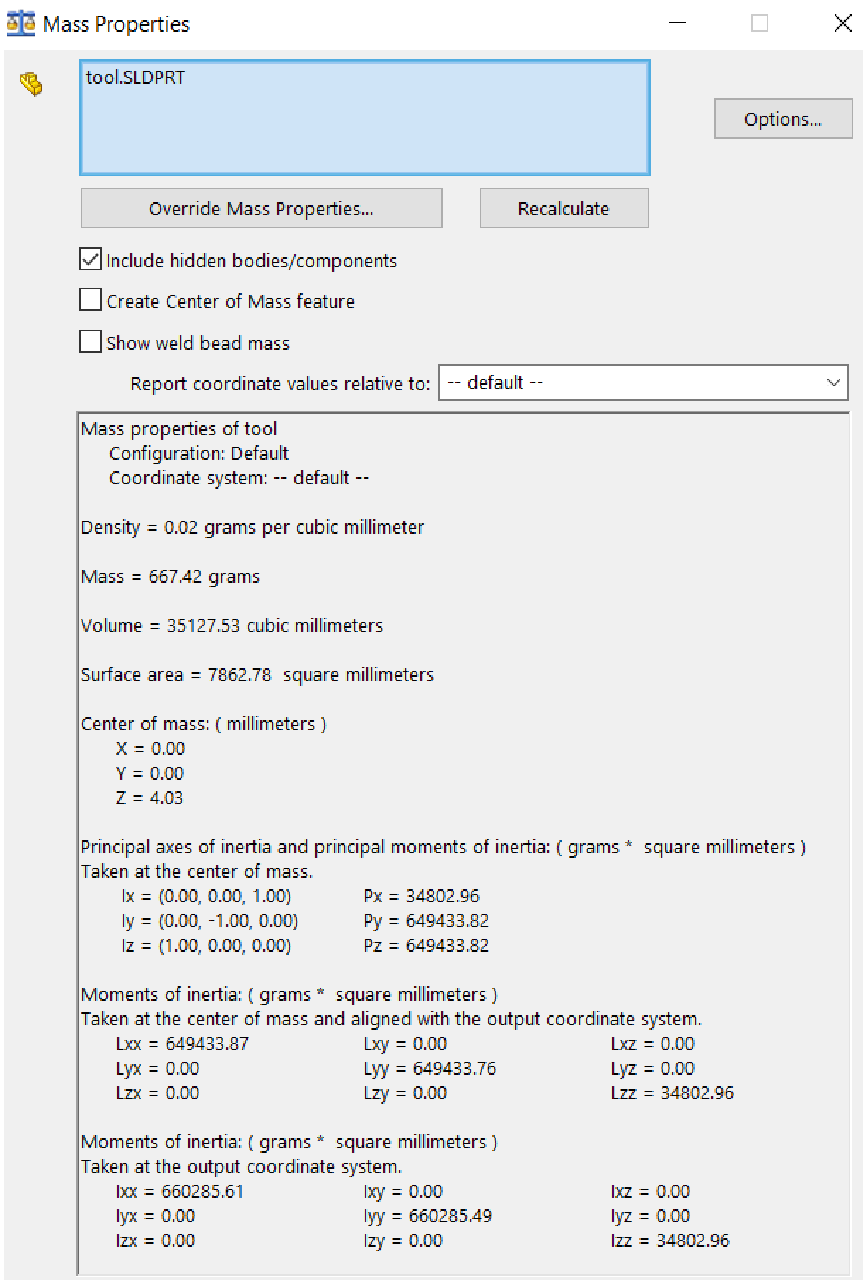

Figure 4.6 Mass properties of tool using Solid Work

4.4 Volume fraction calculation

The volume fraction of Titanium carbide (TiC) particles used during FSP was calculated from the following expressions given below:

$$Volume\ Fraction = \frac{Area\ of Groove}{Projected\ Area\ of\ Tool\ Pin} \times 100$$

Where, $Area\ of\ the\ Groove = Width\ of\ Groove\ \times\ Depth\ of\ the\ Groove$

$$Project\ area\ of the\ \ Tool\ Pin = \ Pin\ size\ \times\ Pin\ length$$

From the above expressions, volume-fraction of TiC particles comes out to be nearly 13.33%.

4.5 Sample Preparation

To study microstructure, micro hardness and wear; samples were cut in transverse direction from the mid portion of the stirred zone of the processed surface with the help of wire EDM as shown in figure 4.7 Two on circular samples each from the three processed surface having size 8 mm and 10×10 mm were cut for microstructure observation and micro hardness study. Moreover, two circular samples, two square samples, and two triangular samples were taken. Also, processed surface having 10 mm diameter were taken for the wear test.

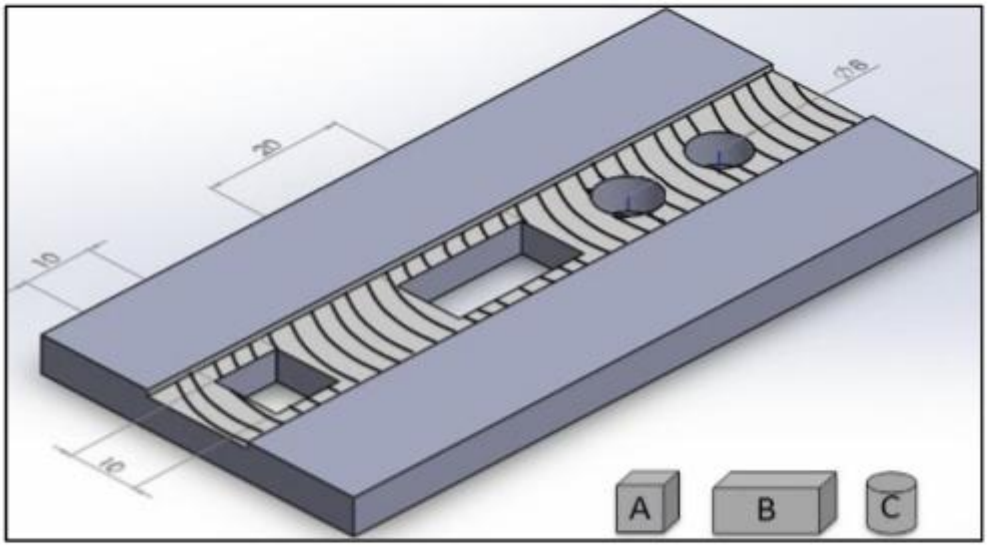

Figure 4.7 FSP specimens for : (A) Microstructure (B) Microhardness and (C) Wear studies

4.6 Microstructural Studies

Figure 5.4 shows the samples cut from the stirred zone for microstructure study. This section consists of Polishing, Etching, Optical Microscopy (OM) investigation, Scanning Electron Microscopy (SEM) study, etc.

Before doing the polishing operation, samples were mounted with the help of resin and hardener. Mounting was done for easy holding of the samples, as the samples were small and cannot be held during polishing resulting in uneven and improper polishing. Now, the mounted samples were dry polished using different grades of emery paper, progressively coarser to finer one (100, 120, 220, 320, 400, 600, 1200, 2000 grits) in the metallurgical laboratory.

After performing dry polishing, initially wet polishing was done by making the samples contact against the rotating disc having fine grit emery paper. Finally, wet polishing was done in a rotating disc of proprietary cloth (velvet type) with the application of alumina powder having grade I, II and III. Intermittent drying of samples was also done by the hand dryer provided in the machine. Finally, Ethanol was used to clean the surface to remove the contaminants.

To reveal the grain structure, the samples were finally etched using nital solution. After chemical etching, the left over layer remained on the metal surface was removed by immersing it in ethanol.

The microstructural evolution during the FSP was characterized using an Olympus Optical Microscope (OM), model GX 41 equipped with image analysis software, a camera having 10x, 20x, 50x and 100x lens and a computer. Indentations in the specimen which was developed during micro hardness test were also visualized by OM. OM images were taken for all three H1 alloy types through the 10x objective lens.

Scanning electron microscope (SEM) was used to observe the dispersion of the reinforced particles (TiC) in the AISI1045 medium carbon steel. Worn out surfaces during wear testing of all three specimens were also analyzed using SEM to understand the wear mechanism.

4.7 Micro Hardness Test

The Vicker's micro hardness testing machine (Model- MVK-H1, Mitutoyo) having specification as hardness ranging from 20 Hv to 1500 Hv and load ranging from 10gm to 1000 gm was used for micro hardness test. Before the hardness measurement, samples were polished up to 1200 grit fine emery paper to remove the oxide and other scales for clear observation of indentation mark. The Vickers micro hardness values of the processed regions were calculated along the processing direction (along with the depth of specimen) using the load of 50g with a dwell time of 10 s.

4.8 Wear Test

4.8.1 Wear Sample Preparation

Wear samples of 10mm diameter, were engraved from the middle of stirred zone of the processed region.

The sliding wear test was carried out using pin-on-disc tribometer (model TR 20, manufactured by Ducom, Bangalore, India) in the room temperature condition. Pins of 8 mm diameter were made to slide against the rotating counter discs of diameter 160 mm as shown in figure 4.8. The counterpart disks were made of EN-31(100Cr6 , AISI 52100) steel with a hardness value of about 60 HRC (746 HV). Sliding track diameter on the disk surface was 100 mm. Before wear test, each pin specimen was ground down to 1000 grit abrasive paper A constant track diameter of 100 mm and a constant sliding velocity of 2 m /s, were used in all wear sample testing. Moreover, sliding distance of 2500 m was fixed in all tests which give the 1000 seconds duration of each wear test. For every wear test, the sample was cleaned with acetone and weighed to an accuracy of 0.001 mg by electronic weighing balance before and after wear test. The load was fixed for 40 N and 10N for all the samples. Sensor output in the machine measured the frictional force in N and cumulative wear loss in mm as a function of time. Wear rate in mg/m was calculated from weight loss during wear. The friction coefficient between the pin-specimen and the disc was calculated by determining the ratio of frictional force (obtained from sensor output) and normal load (40 N or 10 N). All the worn sample surfaces were cleaned by acetone and examined under SEM.

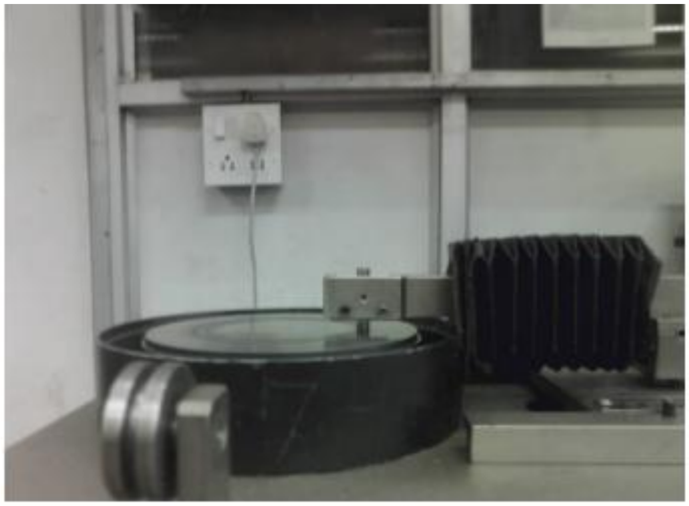

Figure 4.8 Pin-on-disc Tribometer

4.9 Experimental Procedure

4.9.1 AISI 1045 Medium Carbon Steel Plate

ISI 1045 steel is characterised by good machinability, good weldability and high impact and strength properties in either the normalized or hot rolled condition. The size of plates is (200 X 80 X 1) mm as shown in fig. 4.9.

Figure 4.9 Slotted Plate

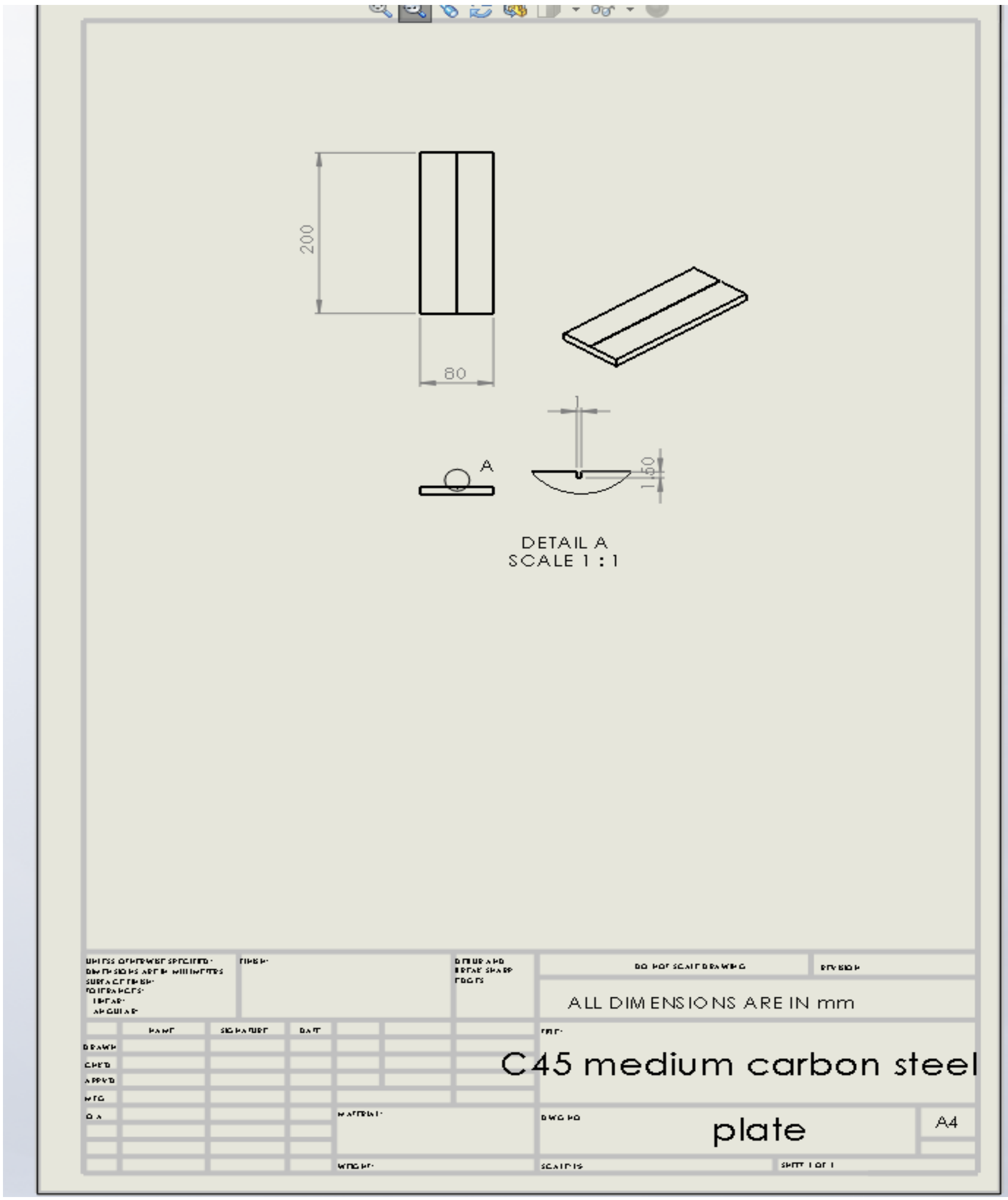

Figure 4.10 Draft of designed plates

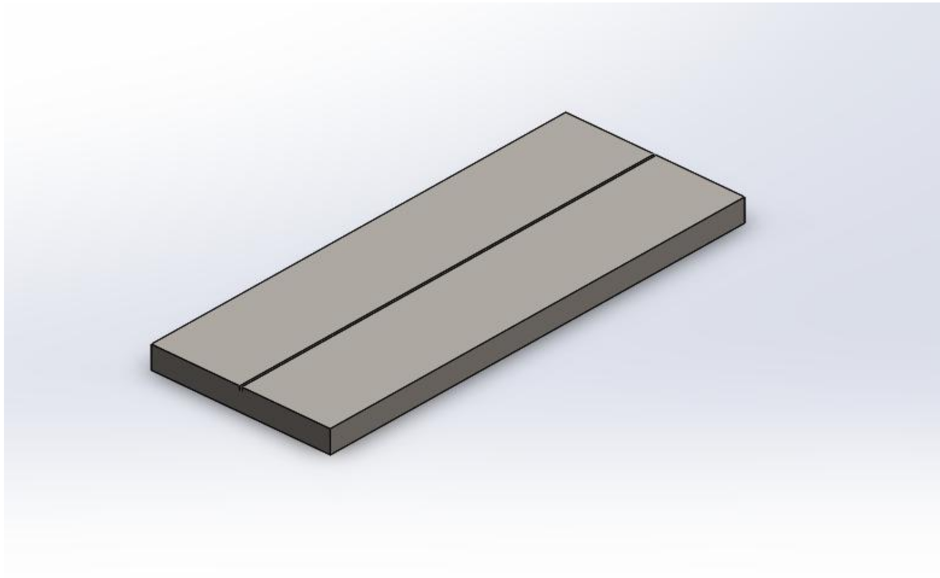

Figure 4.11 Solid Works Design of Plate

The slot of 1mm deep is made in the plate. Then the plate is being leveled from the sides by using vertical milling machine. We processed it with different tool probes (Square, Circular, and Triangular) with mixture of Titanium Carbide particles

Physical Properties:

Physical Properties of work-piece (plate) is shown in table below:

Density 10^3 Kgm^{-3}	**7.85**
Thermal conductivity($Jm^{-1}K^{-1}s^{-1}$)	48
Thermal expansion ($10^{-6}K^{-1}$)	11.3

Young's modulus (GNm^{-2})	210
Tensile strength (MNm^{-2}	600
% elongation	20

Table 4.2 **Physical Properties of AISI 1045**

4.9.2 Composite Fabrication

For fabrication of surface composites, a groove of 1mm width and 1.5mm depth was made on a C45 medium carbon steel plate with the help of milling machine (Fig 4.12.(a)). Then the plate was mounted on the bed of FSP machine rigidly with the help of clamps.After this, the groove was properly cleaned with acetone and Titanium Carbide (TiC) particles were introduced into the slot by groove filling and closing method (Fig 4.12.(b)). In this method, first of all, groove was filled with TiC particles and then groove was closed with the help of a probeless tool to prevent escaping of micro particles (Fig 4.12.(c)). Then the tool pin was made to get inside the slot in the plate and axial load is given to plate via tool shoulder. At last, the plate was subjected with an FSP tool as shown in fig. 4.12 to produce a surface composite. Tool moved forward doing extrusion and forging together. The specimen was then allowed to be cooled to the room temperature and all the experiments were carried out at room temperature. One portion of the plate was also subjected to simple FSP without the addition of TiC particle. The FSP procedure to produce the surface composite is schematically shown in figure 4.12

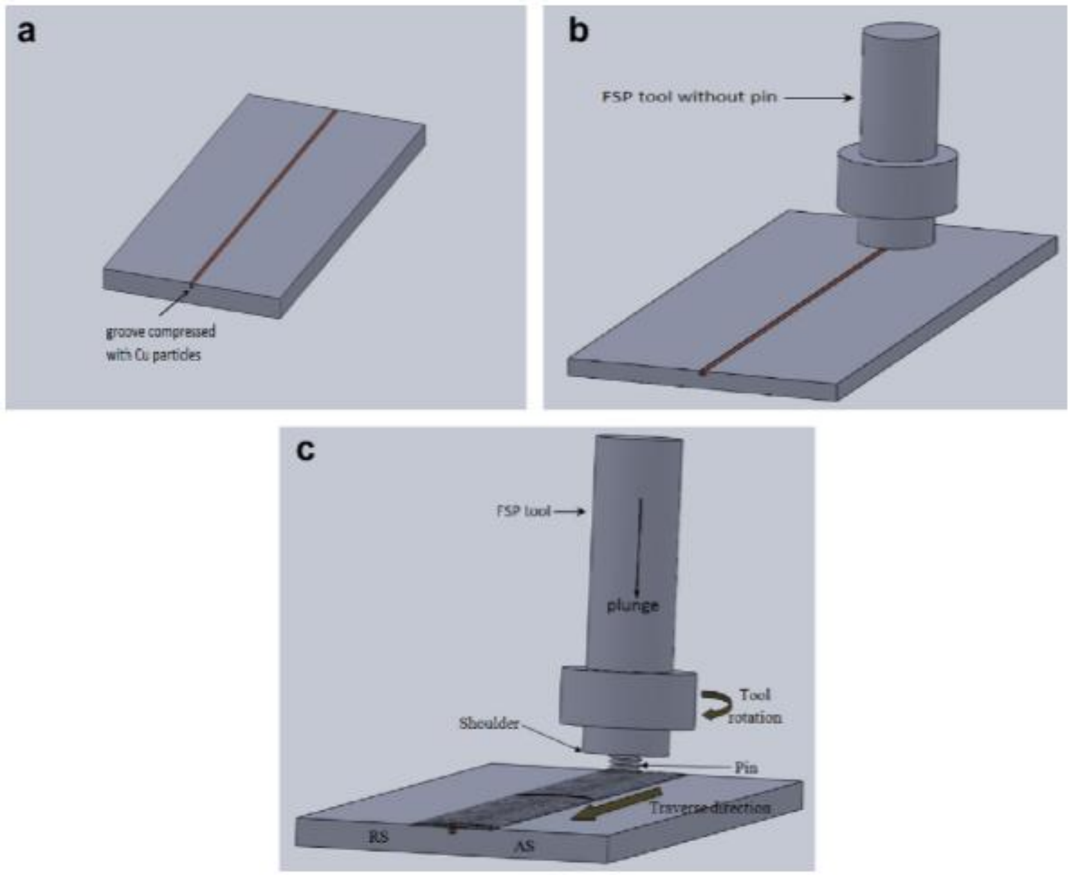

Figure 4.12 **(a) Plate with slot (b) Plate with the tool (c) Process schematic view**

The processing was repeated using different tool profiles – Circular, Square and Triangular keeping others parameters same. The pin length was 2mm and the pin cross-section was kept 6mm diagonal.

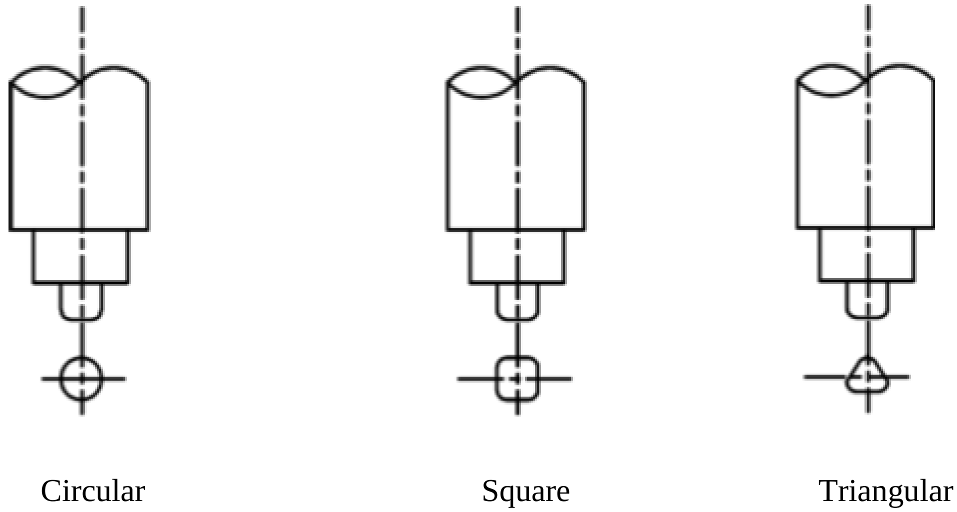

Figure 4.13 Different types of Tool Profile

5. Result and Discussion

This chapter deals with the effect of FSP on the microstructure, microhardness and wear properties of medium carbon steel and Titanium Carbide (TiC)surface composite.

5.1 Micro Hardness

The average hardness of the base material and FSPed surface using different tool probes is given below:

PIN PROFILE	DESIGNATION	HARDNESS
Base Material	BM	233
FSPed Without TiC	FSP	260
FSPed (Square)	Sq	316
FSPed (Triangular)	Tr	302
FSPed (Circular)	Cr	298

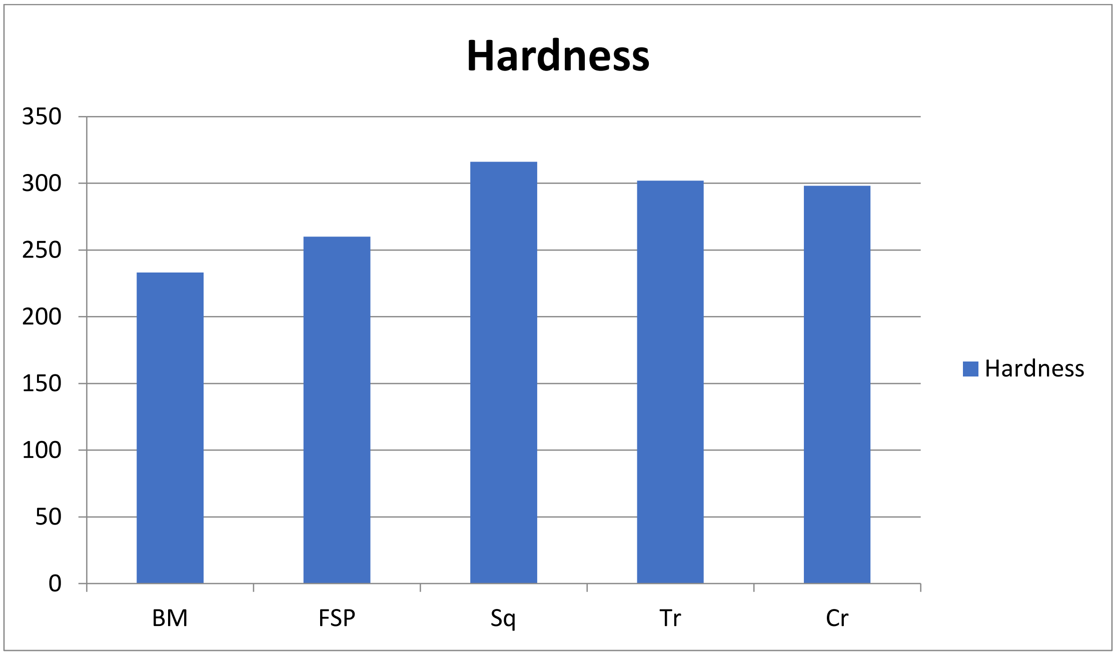

Figure 5.1 Hardness

As we can see from the above results, FSP resulted in an increase of hardness from 233Hv to 316HV by the incorporation of reinforcement particles. The increase of hardness due to FSP can be explained by Hall-Petch relationship. According to Hall-Petch relation:

$$\sigma y = \sigma i + kd0.5$$

Here, ‘σy’ represents the yield strength of the polycrystalline material, ‘σi’ represents the yield strength of the same material at infinite grain size, ‘k’ represents hall-pitch constant and’. Tic reinforcements have double effects on hardness value. The artifact is because of its hard nature. The second arises from the role of reinforcements on grain boundary pinning.

The Hardness achieved using squared tool probe was found to be maximum. The specimen engraved out of the middle-stirred zone of the processed zone (using square tool probe) will be performed for microstructural and wear studies.

5.2 Microstructure Characterization

FSP resulted in successful fabrication of carbon-steel/Tic surface composite. Figure below represents the microstructural images of the stirred zone (SZ) of the medium carbon steel which was examined by optical microscope. The samples were across-sectioned perpendicular to the processing direction, polished and then etched in 5% natal solution for 5 s the track exhibited sound appearance and smooth quality. Almost no prominences, void, cracks or depression were found

Cross section of the fabricated layer after the FSPaexhibitsamaterialaflow pattern. This assists mixing of TiCaparticlesawith the plasticised material. Employing higher magnification on unetched cross section, small regions with bright contrast were revealed. These areas are titanium carbide. However,the particles made clusters of various sizes within theastir zone. The agglomerative nature of fine particles due to their high cohesive energy leads to an increase in theatotal surface area and increases their tendency to clump together forming agglomerates and clusters. Etched cross section of the fabricated surface layer using one pass FSP with the introduction of TiC showed fine grain microstructure. Thus, a FSP refined theamicrostructure of the surfacealayer. FSP and FSW wereainitially applied to lightaalloys (mainly, Al andaMg alloys). For heavierametallic materials, tougheratools are required toaproduce relatively greateraheat inputs and to withstandawear. However, tool wearadebris is inevitable andareported in the FSPed layers in a scattered fashioned.

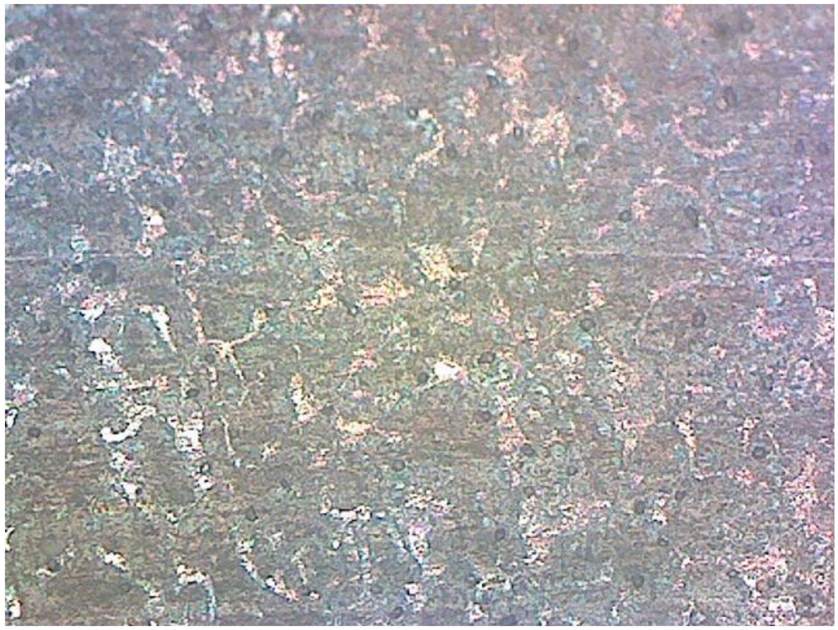

Figure 5.2: **OM images of transverse cross-sectional of C45 steel- 10X**

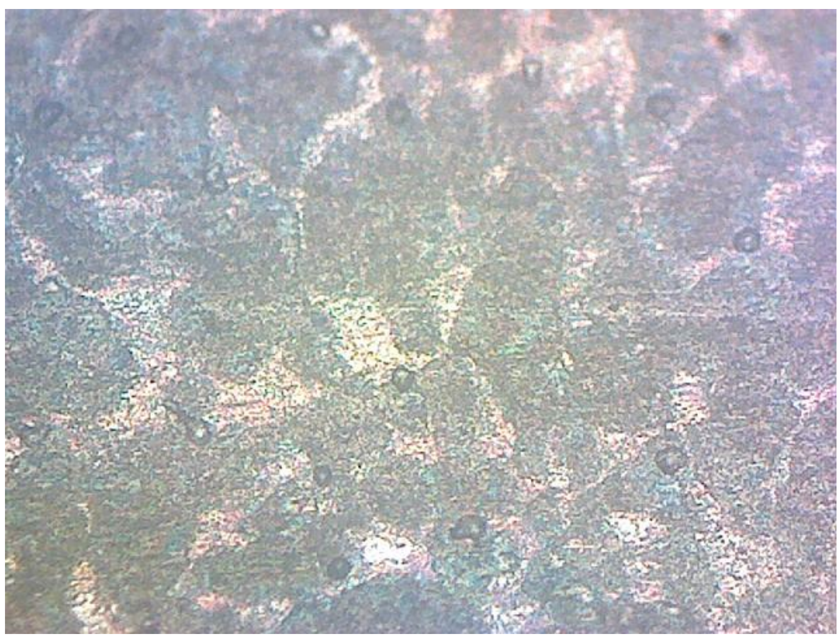

Figure 5.3**:OM images of transverse cross-sectional of C45 steel - 20X**

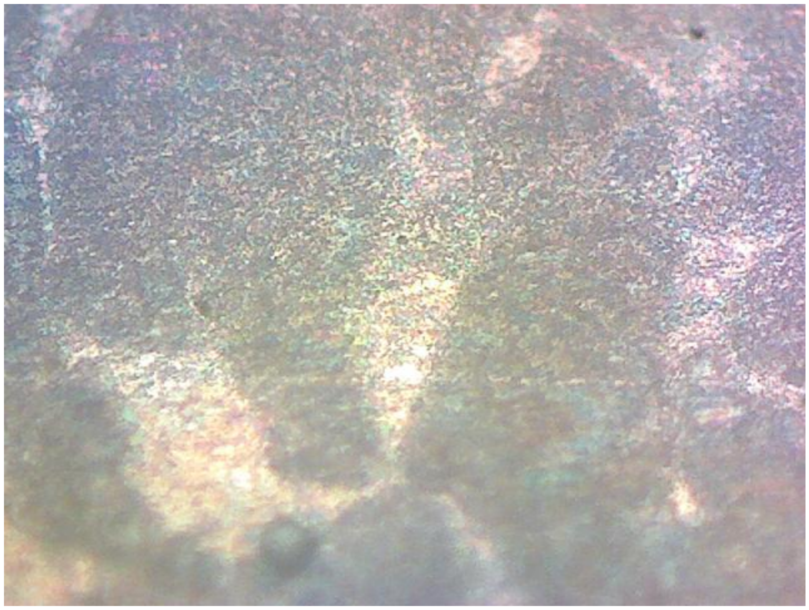

Figure 5.4 **:OM images of transverse cross-sectional of C45 steel - 50X**

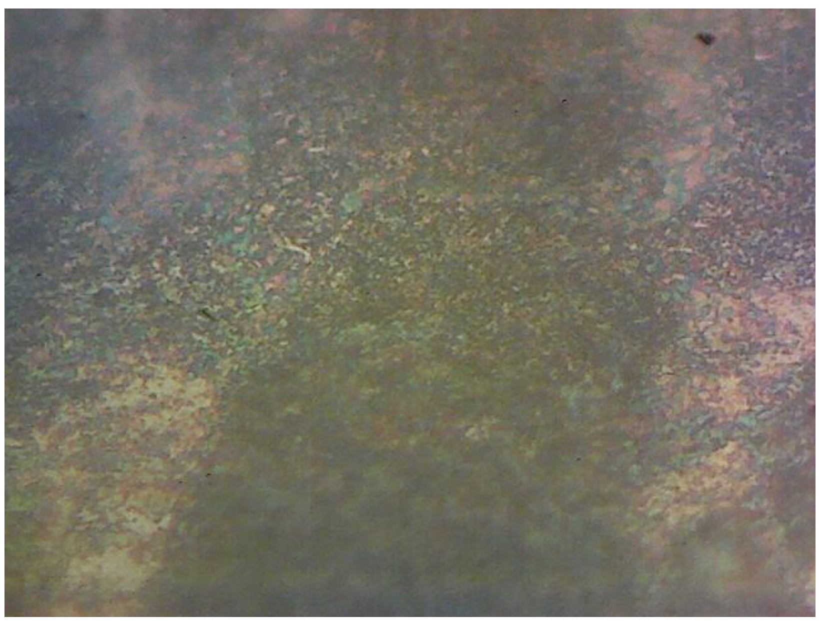

Figure 5.5 :**OM images of transverse cross-sectional of C45 steel- 100X**

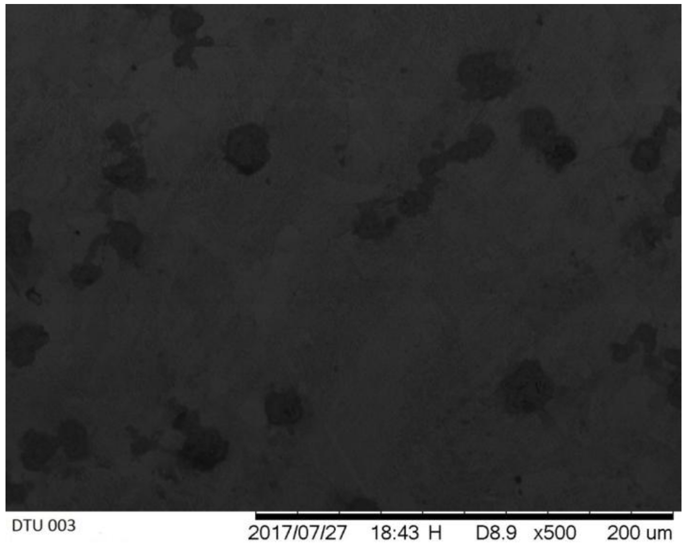

Figure 5.6 SEM micrographs of C45 Steel FSPed with TiC micro sized particle-500X

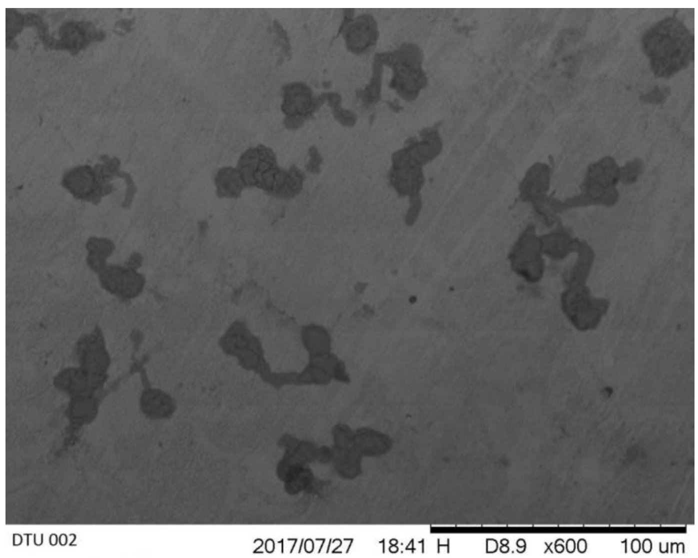

Figure 5.7 :SEM micrographs of C45 Steel FSPed with TiC micro sized particle-600X

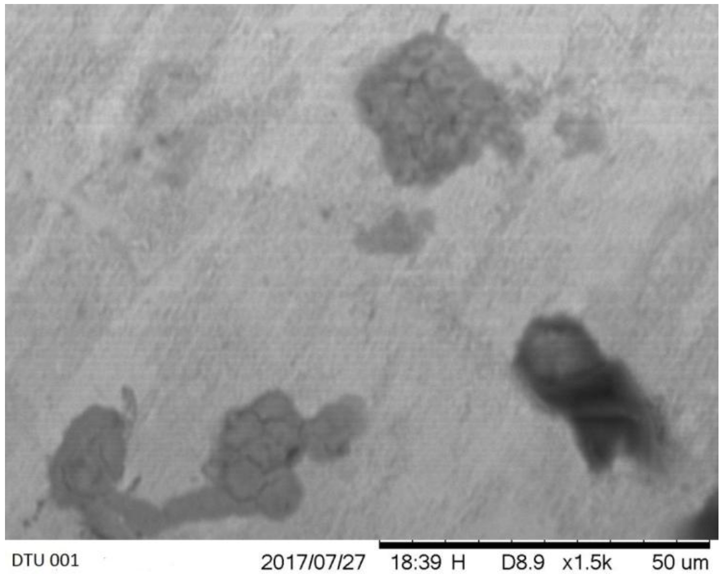

Figure 5.8 SEM micrographs of C45 Steel FSPed with TiC micro sized particle-1500X

5.3 Wear Properties

The wear behavior of the as received for C45 Steel, FSPed with TiC particle was evaluated by a pin on disc tribometer. Wear test was conducted at 40N. Table presents the complete wear test results, primarily mentioning wear rate (mg/m) and friction coefficient. Here, friction coefficient is calculated as the ratio of average friction force and load. Wear analysis was performed on two sample and the average of both has been mentioned in the table

Steel Condition	Load	Weight Loss (g)	Wear Rate (mg/m)	Friction Coefficient
Reinforced with TiC	40N	.032	0.0128	0.33514

Fig 5.9 represents the variation of weight loss (mg) with the sliding distance (m) for the tested wear samples. The decreasing trend of wear rate from base metal to FSPed specimen with particle at 40 N. It can be inferred from the results, that samples containing hard TiC particles in the steel exhibit less weight loss

Fig 5.9 shows the comparison of wear rate and microhardness of the corresponding samples. This variation clearly supports the Archard Equation [26] which is

given by:

$$W = kLP/3H$$

where W is volume of materials worn out, k is the wear coefficient, L is the sliding distance, P the applied load and H is the bulk hardness of the materials. It can be inferred from above equation that wear rate is inversely proportional to hardness. This indicates that sample having less hardness will have more wear rate and vice-versa. Therefore, base metal was found to be having more wear rate as compared to the FSPed samples with particle. Now, it is evident from above discussion that FSPed alloy with reinforcement particle has shown better wear resistance, at load 40 N, compared to Base Metal. The variation of friction coefficient with sliding distance at an applied load of 40 N is shown in fig 5.9. The base metal alloy has shown higher value of friction coefficient compared to other condition of alloy. This is due to the presence of hard reinforcenemnt particles TiC present in the FSPed composite which decreases the direct load, thus reducing friction coefficient. Moreover, these TiC particles act as lubricant, thus reducing friction coefficient.

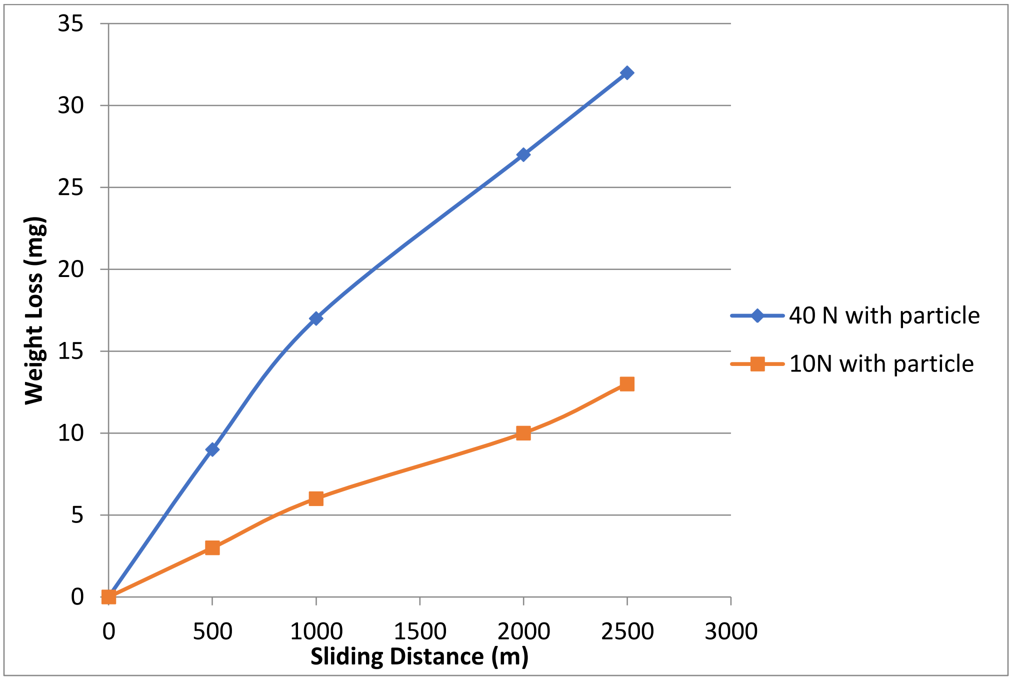

Figure 5.9 Variation of weight loss (mg) with the sliding distance (m) for fsp ith the square shaped tool probe at 40N, 10N

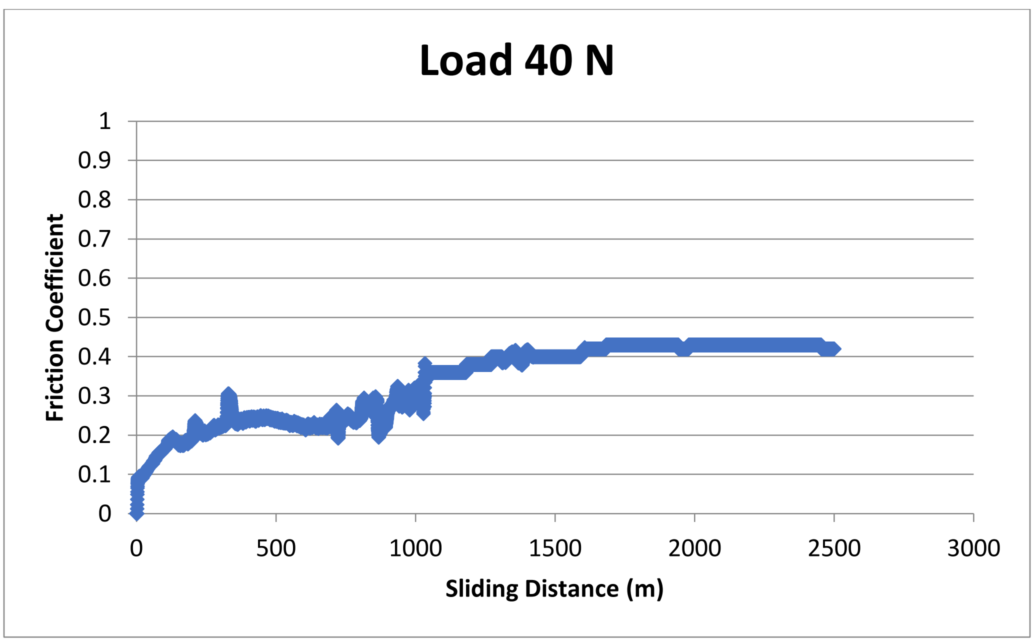

Figure 5.10 Variation of Friction Coefficient with Sliding Distance at 40 N load

6. Conclusion

Medium carbon steel was successfully synthesized using the novel method of Friction Stir Processing without any formation of defects. The Fabrication of surface composite was performed using different tool probes- square, circular and triangular.

The hardness of FSP zone fabricated using square profiled tool was found to be higher than the FSP zone fabricated using other tool probe .FSPed (square pin) steel with TiC particle exhibited the hardness of 316 Hv as compared to the 233 of Base material due to reduction in grain size as explained by Hall-Petch relationship. Hence, the specimen of stirred zone of the surface composite fabricated using square profile tool was selected and engraved out for further studies of microstructure and wear . The FSP resuled in significant grain refinement of particles and uniform distribution of TiC particles in the composite. The wear rate at 40N load, 2m/s and sliding distance of 2500m was found to be 0.0128 mg/m and the friction coefficient was 0.33514.

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