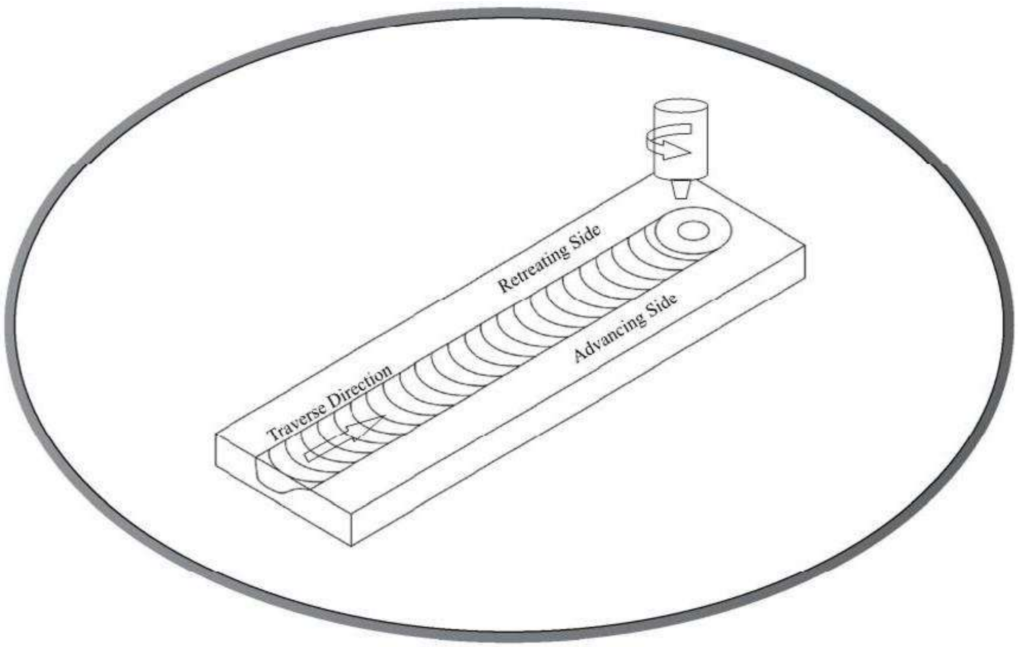
Chapter 2

Literature Review



In this chapter, current trends, various issues and strategies followed for surface composites fabrication via FSP have been reviewed. Various factors involved in the process of SCs fabrication are discussed and classified. Also, variation of microstructural, mechanical, wear and electrical characteristics with these factors is reviewed.

Chapter concludes with the discussion on the scope and objective of the present investigation.

2.1 Introduction

Surface composites (SCs) are most appropriate materials for engineering applications where only surface interactions are involved and the rest material doesn't require any property change. Friction stir processing (FSP) is a novel technique capable of surface modification of materials along with surface and bulk composite fabrication. SCs display improved surface properties such as hardness, wear, corrosion resistance, and resistance to fatigue crack initiation without affecting the bulk properties of materials. FSP can be used for production of surface composites of various ferrous and non-ferrous alloys like aluminum, magnesium, copper, titanium and steel. In this chapter, current trends, various issues and strategies followed for the fabrication of surface composites via FSP have been reviewed. Various factors involved in the process of SCs fabrication are discussed and classified. Also, variation of microstructural, mechanical, wear and electrical characteristics with these factors is reviewed.

2.2 FSP for surface composites fabrication

Due to relative motion between tool shoulder and workpiece, frictional heating occurs which leads to plasticization and softening of the materials. The material flows from the advancing side to retreating side of the tool. The combined effect of material flow and stirring action of the tool at elevated temperatures mixes the reinforcing particles (RPs) with plasticized base metal (BM). As the rotating tool traverses in the desired direction, the material is forged beneath the tool shoulder and results in processed zone. In this way, the process of composite fabrication completes. Metal matrix composites can be fabricated by two methods via FSP namely ex situ and in situ. In ex situ composites, RPs are preplaced in/over the plate before FSP whereas, in the case of in situ composites, the as prepared particulate reinforced composites are processed by selecting the parameters in such a way that it lead to reaction of matrix and particulates. The mechanism of ex situ composites fabrication is relatively simpler and less time consuming in comparison to in situ composites. In ex situ technique of composite fabrication by FSP, particulates are preplaced on the plate and then FSP is performed. Various methods of pre-placement of reinforcements are in practice which will be discussed in detail in the section reinforcement strategy. In this chapter, ex situ composites fabrication are discussed along with factors involved and properties enhancement mechanisms.

Mishra et al. (Mishra et al., 2003) used FSP to fabricate ex situ SC of aluminum alloys for the very first time in 2003. They mixed silicon carbide (SiC) with methanol and then coated over AA 5083 plate. The SiC coated plates were then friction stir processed. The microstructural features of the processed plate in the stir zone (SZ) revealed uniform dispersion of SiC particles in aluminum matrix. The hardness in the SZ of the composite improved significantly as compared to base aluminum and was found to be approximately 1.8 times higher than the unprocessed aluminum. This work proved to be landmark in the sense that it made way for various possibilities of particles reinforcement in the metal matrix for manufacturing of SCs of different alloys by FSP. Since then, it has come along a long distance and a lot of studies have been reported and are currently in progress for fabrication of SCs. Earlier, it was basically used for aluminum alloys. But, now it is not limited to aluminum alloys only. It has been successfully extended to manufacturing of SCs of other alloys such as copper (Barmouz et al., 2011b, Dehghani and Mazinani, 2011), magnesium (Dadashpour et al., 2016), titanium

(Shamsipur et al., 2011), and even steel (Ghasemi-Kahrizsangi and Kashani-Bozorg, 2012, Ghasemi-Kahrizsangi et al., 2015).

2.3 Factors involved in composite fabrication via FSP

The properties of the materials to be processed play important role in selection of processing parameters. For example, high melting point materials such as steel, titanium and copper alloys require high heat input for their processing (Heidarzadeh et al., 2015). The mechanical properties such as yield strength (YS), ductility and hardness of base metal are important in regards to plastic deformation during friction stir welding (Balasubramanian, 2008). It was found that aluminum alloys having lower YS and hardness, and higher ductility was easy to process as compared to one which has higher YS and hardness, and lower ductility. Thermal aspects of materials govern the peak temperature reached during processing of a material. Materials having high thermal conductivities require more heat input for defect free processing (Xu et al., 2014). High thermal conductivities materials dissipate heat more rapidly by conduction (Khandkar et al., 2003). To manipulate thermal properties, a backing plate beneath the specimens is generally used.

Various factors involved in composite fabrication via FSP is schematically shown in Fig. 2. 1.

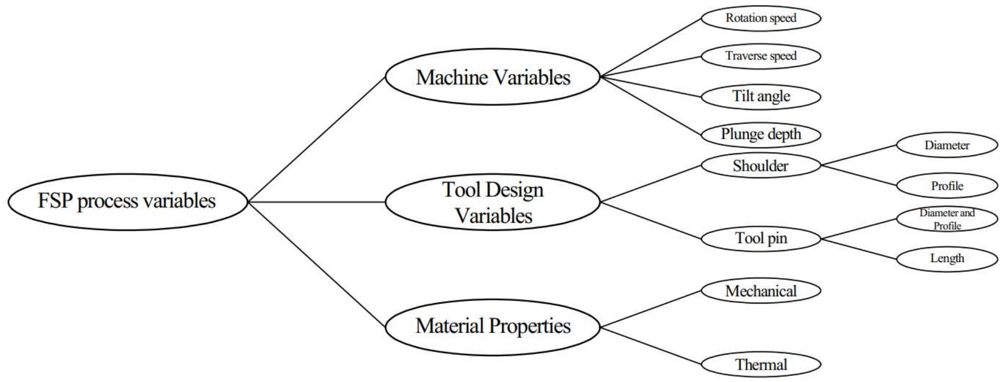


Figure 2. 1 Classification of FSP variables involved in SC fabrication

Variables involved in SCs fabrication by FSP can be broadly classified into three different categories namely: machine variables, tool variables and reinforcement variables. Contribution of each variables is different as some have larger influence on properties enhancement than the others. These variables will be discussed in detail in the following sections.

2.4 Machine variables

The variables which are controlled by machine settings are termed as machine variables. The rotational speed of the spindle and traverse speed are considered to be main machine variables which affect the amount of heat production and material flow during the process. Numerous results on SCs fabrication have shown that combination of higher rotational speeds and lower traverse speed leads to higher heat input, better material flow and consequently improvement in particle dispersion (Azizieh et al., 2011, Morisada et al., 2006a, Rathee et al., 2017a). Also, torque reduction with higher temperatures and vice-versa are observed with higher tool rotation speed (Węglowski and Dymek, 2013).

In fabrication of SiC/AZ91 composite, it was reported that an increase in rotational speed leads to an increase in grain size while increase in traverse speeds leads to decrease in grain size. This was explained by noting that increasing traverse speed leads to a decrease in the time of exposure to the process heat (Asadi et al., 2010). Material flow in SZ enhances with increase in rotational speed. FSP of Al_2O_3/AZ31 surface composite revealed that the cluster size of alumina particle clusters decrease as the rotational speed increases (Asadi et al., 2011). Increased heat input due to high rotational speed increased the grain size but nano-particles of alumina were homogeneously distributed due to shattering effect of rotation. The successful combinations of rotational and traverse speeds reported for surface composites are listed in Table 2.1.

Table 2. 1 Successful combination of rotational and traverse speed for surface composites fabrication via FSP

Surface composite	**Rotational speed (rpm)**	**Traverse speed (mm/min)**	**References**
($SiC+MoS_2$)/A356	1600	50	(Alidokht et al., 2011)
SiC/AZ91	900	63	(Asadi et al., 2010)
Al_2O_3/AZ31	800	45	(Azizieh et al., 2011)
SiC/Cu	900	40	(Barmouz et al., 2011b)
Nano-clay/Polymer	900	160	(Barmouz et al., 2011c)
SiC/AA5052	1120	80	(Dolatkhah et al., 2012)
TiC/Al	1000	60	(Bauri et al., 2011)
($SiC+Al_2O_3$)/AA1050	1500	100	(Mahmoud et al., 2009b)
Al_2O_3/AZ91	1600	31.5	(Faraji et al., 2011)
HA/Ti-6Al-4V	250	16	(Farnoush et al., 2013)

TiC/Steel	1120	31.5	(Ghasemi-Kahrizsangi and Kashani-Bozorg, 2012)
SiO_2/AZ91	1250	63	(Khayyamin et al., 2013)
SiC/AA1050	1000	15	(Kurt et al., 2011)
MWCNT/AA1016	950	30	(Liu et al., 2013)
Ni/AA1100	1180	60	(Qian et al., 2012)
(TiC+B4C)/AA6360	1600	60	(Rejil et al., 2012)
SiC/AA6061	1600	40	(Salehi et al., 2012)
B_4C/Cu	1000	40	(Sathiskumar et al., 2013)
SiC/Ti	800	45	(Shamsipur et al., 2011)
(SiC+MoS_2)/AA5083	1250	50	(Soleymani et al., 2012)
Al_2O_3/AA2024	800	25	(Zahmatkesh and Enayati, 2010)
Al_2O_3/AA6082	1000	135	(Shafiei-Zarghani et al., 2009)
Cu/AA5083	750	25	(Zohoor et al., 2012)
Cu/AlN	1000	40	(Dinaharan et al., 2017c)
Cu/AlN	1000	30	(Thankachan and Prakash, 2017)
Cu/B_4C	700	60	(Ahn et al., 2017)
Cu/Al_2O_3	900	40	(Raju and Kumar, 2014)

Shahraki et al. (Shahraki et al., 2013) observed that the distribution of ZrO_2 particles in the SZ of AA5083 alloy was not uniform at low rotational speeds or high traverse speeds. A combination of low rotational speed and high traverse speed results in poor plastic flow of the material, poor distribution of particles, and formation of porosities. Further, at low rotational speeds, the amount of heat produced is not sufficient for the material to become soft enough to go for high traverse speeds.

Barmouz et al. (Barmouz et al., 2011b) showed that effect of traverse speed on grain size was reversed in the fabrication of Cu/SiC surface composite as compared to FSP of the material. The SiC particles tend to agglomerate in the SZ on increasing traverse speed. As traverse speed decreased, the grain size decreased in composite, whereas, in FSP of Cu without SiC particles, grain size increased with the decrease in traverse speed because of increase in heat input. This may be attributed to the uniform dispersion of SiC particles in lower traverse speeds which enhance the pinning effect of SiC particles in the SZ.

Although, it has been reported that increasing speed of rotation leads to improved particle dispersion in metal matrix, a few studies have also reported that a defect free stir zone and better particle dispersion was achieved at relatively lower speeds of rotation (Mahmoud et al., 2009a). However, no proper explanation for the same was provided. Other machine related parameters are axial force (Langlade et al., 2016), tool tilt angle and plunge depth (Rathee et al., 2016, Rathee et al., 2017b) which have relatively less influence on particle distribution. An axial force is necessary element for holding the tool and stirred material (during material flow) against the workpiece. Also it influences particle distribution in metal matrix. Low axial force results in inhomogeneous particle distribution whereas, high axial force leads to particulates ejection on the surface of the plate. Therefore, for better result an optimum axial force is required (Wang et al., 2009b, Yang et al., 2010).

The inclination of tool from the vertical axis (tool tilt angle) and plunge depth are interrelated machine parameters (Mehta and Badheka, 2016). On one hand, higher tool tilt angle implies lesser contact area between tool shoulder and the workpiece whereas, higher plunge depth means higher contact area and vice-versa. So to have desired contact area between tool shoulder and workpiece, tool tilt angle and plunge depth is needed to be optimized simultaneously. Higher tool tilt angle with constant plunge depth, lesser surface of the tool shoulder will be in contact with the plate leading to reduced heat generation. On the other hand, increased plunge depth will counterbalance the reduced contact area and heat generation. Asadi et al. (Asadi et al., 2010) reported in their study that the optimum values of plunge depth required for tool tilt angles of 2.5°, 3°, and 3.5° should be 0.22, 0.30, and 0.40 mm, respectively. At low tool tilt angle defect like voids even at higher tool rotational to traverse speed ratios were observed. Tool tilt angle generally varies from 0° to 3° for all the materials in SCs fabrication.

2.5 Tool variables

Tool geometry mainly includes shoulder diameter, shoulder profile, probe shape, probe size and probe profile. The tool geometry plays a vital role in the prediction of heat generation, material drift, particulate dispersion and properties of the fabricated SCs in addition to rotational and traverse tool motion (Chen et al., 2009a). General tool geometry of FSP tool is shown in Fig. 2.2.

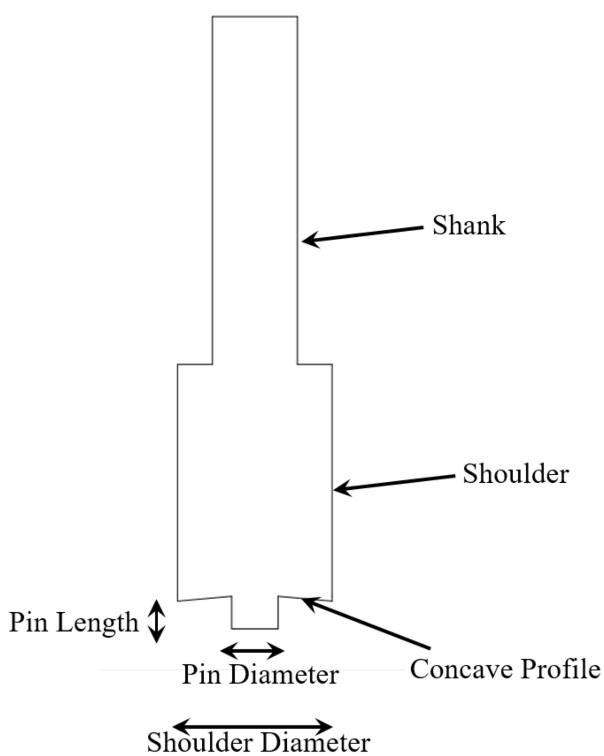


Figure 2. 2 Schematic of tool geometry of FSP tool

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The heat generation by the rotating tool is because of friction between the workpiece and rotating tool, and due to plastic deformation which occurs when the workpiece material stirs, mechanically mix and forge due to the stirring action of the tool (Mishra and Ma, 2005). Besides, aforementioned roles, second phase particles can also be reinforced into the metal matrix by the tool (Rathee et al., 2017a). Tool design variables include mainly two aspects, i.e. shoulder and pin design. Shoulder design consists of its diameter, and end surface angle whereas, tool pin design consists of tool pin diameter, tool pin length, and shape of the pin. The characteristic attributes of these parameters related to tool design are discussed in this section.

Shoulder diameter is considered to be an important process parameter because it generates most of the heat and is also responsible for forging of materials under it. Increasing the shoulder diameter while maintaining remaining processing parameters constant leads to higher heat generation. This can be attributed to higher contact area of the tool shoulder with the workpiece. But, for better results, there should be some optimum value of shoulder diameter. To optimize the shoulder diameter, a lot of work has been done on different types of materials. A common conclusion drawn from experimental results is that tool shoulder diameter should follows the straight line equation with workpiece thickness having a slope of 2.2 and intercept value of 7.3 mm (Reynolds and Tang, 2001). In addition to shoulder diameter optimization, shoulder to pin diameter (D/d) ratio should also be needed to optimize for better properties. In friction stir welding (FSW), most commonly utilized shoulder to pin diameter ratio is 3 for desirable weld properties (Chen et al., 2009a, Prado et al., 2001). Vijayavel et al. (Vijayavel et al., 2014) proposed same results during FSP also. In their study, they used FSP on LM25AA–5% SiC composites and varied D/d ratio from 2 to 4 mm in steps of 0.5 mm. Out of the five ratios (D/d), one having value of 3 exhibited better microstructural and mechanical properties. Shoulder surface can be flat, concave and convex. This affects the material flow during FSP and hence the properties of the processed materials. Flat surface design is easy as compared to concave and convex but in some cases it may cause excessive flash which is not the case with concave shoulder. In addition to end surface angle, the end surface may be having different profiles such as scroll, spiral etc. to enhance the performance of the tools (Zhang et al., 2012).

Another aspect of the tool is its pin. In tool pin, the shape of the pin plays important role as compared to other aspects. Numerous types of pin profiles have been used in which characteristics of different pin profiles have been discussed extensively. Till date mainly cylindrical, circular, conical, triangular and square tool pin profiles have been used for FSW/FSP. Also, the tool pin profiles can be threaded and having flutes on the surface. The tool pin profile plays important role in material flow and particulate distribution in metal matrix during surface composite fabrication via FSP which in turn affects the properties of the fabricated SCs. In general, it is believed that larger pin surface area results in more favorable material flow as it leads to increased frictional heating, higher temperatures and reduced flow stress in stir zone (Nandan et al., 2008). However, the value of the optimum D/d ratio limits the size of the pin diameter. Different shapes of pins are shown in Fig. 2.3.

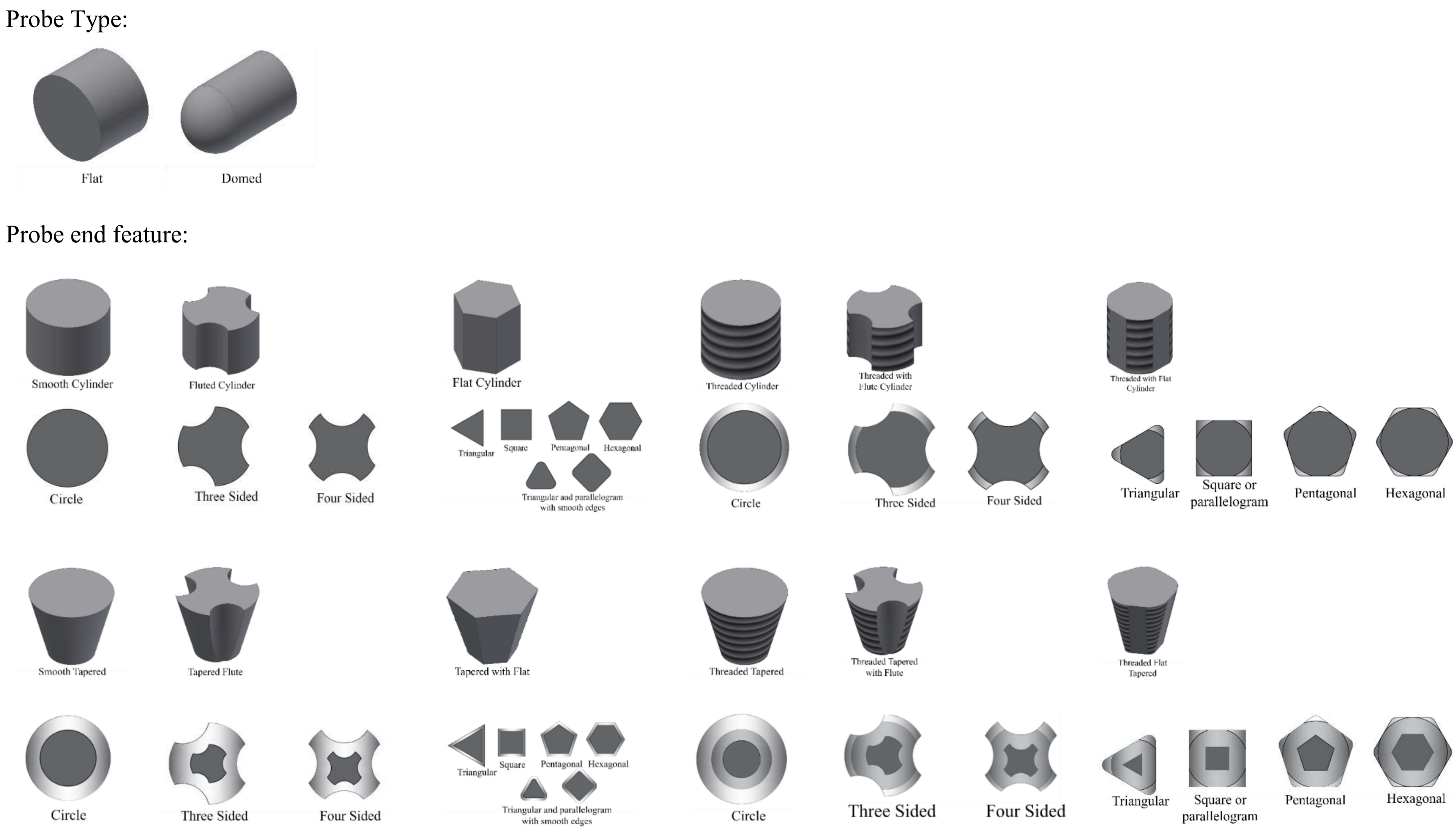


Figure 2. 3 Schematic of different pin profiles used in FSW/P

Numerous results have been reported to explain the effect of tool pin profiles on material flow, particulates dispersion in the matrix and the properties of the fabricated SCs. The results of these studies can be summarized as: The particle distribution in the matrix was found to be more homogeneous with a square tool pin profile in comparison to circular and triangular pins in case of Al/SiC SCs (Mahmoud et al., 2010). Elangovan and Balasubramanian (Elangovan and Balasubramanian, 2008a, Elangovan and Balasubramanian, 2008b) reported similar results in their studies in which they found that square tool pin profile resulted in defect free welds along with superior mechanical properties as compared to other tool pin profiles. Also it has been reported that square tool pin profile results in more grain refinement as compared to circular pins which may be attributed to pulsating action of flat faces of a square pin (Eftekharinia et al., 2016).

It was observed by Azizieh et al. that a uniform particle distribution can be obtained with pin having threads on the surface as compared to plain shape or with flutes (Azizieh et al., 2011). Due to the larger contact area of the threads with the surrounding deformation zone, higher amount of heat is produced as compared to smoother surface tool pin profile which leads to proper material flow and results in better particle distribution in the matrix.

Moreover, threaded tool pin profiles are inherited with vertical material movement characteristics. In case of threaded tool pin profile, material tends to flow in upward direction in the deformation zone whereas, tool shoulder forces the material to flow in the downward direction close to pin. Therefore, vertical vertex flow occurs which leads to better particulate distribution in the matrix (Guerra et al., 2002, Shojaeefard et al., 2018, Avettand-Fènoël and Simar, 2016).

In their investigation, Shojaeefard et al. (Shojaeefard et al., 2018) observed that particulate dispersion in the A356 alloy using threaded tool pin profile is more homogeneous as compared to cylindrical and square tool pin profile. The maximal velocity with which material flows might be higher in case of threaded tool pin profile. Yu et al. (Yu et al., 2012) proposed that the maximum flow velocity of material increased up to 30% in case of threaded tool pin profile as compared to plain tool pin profile using computational fluid dynamics (CFD) modelling during FSP of magnesium alloys. So a properly designed tool pin profile results in sound SCs.

Hot worked tool steel (H-13) is generally used for FSW/P of non-ferrous metal alloys whereas, tungsten carbide is in common practice for ferrous metals. Moreover, it can be observed that tool wear is not a matter of concern in case of non-ferrous metal alloys. However, in case of FSP/W of ferrous metals (like steels), severe tool wear takes place which needs special consideration (Ghasemi-Kahrizsangi and Kashani-Bozorg, 2012, Siddiquee and Pandey, 2014, Rai et al., 2011, Çam, 2011).

2.6 Reinforcements variables

The following reinforcement variables play pivotal role in the properties of the fabricated SCs:

- Reinforcement materials
- Size and volume fraction of the reinforcements and
- Reinforcement strategy

2.6.1 Reinforcement Materials

Varieties of materials are used as reinforcements, the majority being hard ceramic particles such as silicon carbide (SiC) (Najafi et al., 2008), titanium carbide (TiC) (Yuvaraj and Aravindan, 2017), boron carbide (B_4C) (Rejil et al., 2012, Sathiskumar et al., 2013), aluminum oxide (Al_2O_3) (Du et al., 2016), silicon oxide (SiO_2) (Dadashpour et al., 2016), titanium oxide (TiO_2), AIN (Kashani-Bozorg et al., 2015), Si_3N_4 (Kashani-Bozorg et al., 2015), titanium diboride (TiB_2) (Eskandari et al., 2016, Narimani et al., 2016), zirconium diboride (ZrB_2) (Zhao et al., 2016), carbon particles like graphite (Thapliyal and Dwivedi, 2016), carbon nano-tubes (Johannes et al., 2006), nano-hydroxyapatite (Farnoush et al., 2013), industrial and agricultural wastes like Fly ash (Dinaharan et al., 2016a, Juang and Xue, 2015), rice husk ash (Dinaharan et al., 2017a), zircon sand (Rahsepar and Jarahimoghadam, 2016, Sharma et al., 2001) etc., for improvement of surface properties. Selection of reinforcing materials for composites fabrication is based on different factors like processing route, end used, economy, environmental threats etc. for example industrial and agriculture wastes used as reinforcement led to reduction in processing cost along with environmental threats. Although, use of ceramic particle as reinforcement led to higher processing cost but in application such as heat sink selection of waste as reinforcement will not be fruitful due to low thermal and electrical conductivity.

Mishra et al. (Mishra et al., 2003) reported the first successful results on production of MMCs via friction stir processing. The ceramic particle SiC was mixed with volatile solvent such as methanol and applied on the surface of aluminum plate. They stated that the SiC particles were dispersed uniformly in aluminum matrix along with clean interface. The bonding between SiC particles and aluminum matrix was excellent.

Barmouz et al. (Barmouz et al., 2011b) conducted studies on Cu/SiC surface composite fabricated by FSP for thermal and electronic applications. The authors reported that the hardness of pure copper increased from 60 HV to 90 HV in SZ of the fabricated surface composite. They also reported that the increase in traverse speed resulted in decrement in hardness of the composite along with asymmetric material flow.

2.6.2 Size and volume fraction of RPs

Surface composites are generally fabricated by utilization of reinforcements of size in the range of micrometer to nanometer. The shape and size of reinforcements play important role on the properties of surface composites fabricated by FSP. (Akramifard et al., 2014) and (Sabbaghian et al., 2014) observed interruption as pores around SiC and TiC particles respectively in copper matrix surface composite fabricated via FSP. The interruption at the interface is observed due to uneven shape of the reinforced particles which could not allow proper flow of plasticized matrix all over the surface of particles. The properties of the composites such as microstructural features, hardness, and mechanical strength are greatly influenced by the size and volume fraction of the reinforced particles (El-Kady and Fathy, 2014, Thangarasu et al., 2015, Sahraeinejad et al., 2015). Experimentally it has been established that smaller the size of reinforcements better will the properties of the composites which is in agreement with the relation shown in Eq. 1 (Kouzeli and Mortensen, 2002).

$$\lambda = \frac{1 - V_p}{N_i} \qquad (1)$$

λ is inter-particle distance, V_p is volume fraction of reinforced materials and N_i is intercepts number of particles per unit length. Inter-particle distance is defined as distance between adjacent particles and is directly proportional to particle size and inversely proportional to volume fraction of the particles reinforced.

The reinforced volume fraction influence grain size. According to Zener–Holoman parameter, higher volume fraction of particles led to decreased grain size due to pinning of grain growth (Asadi et al., 2011, Shafiei-Zarghani et al., 2015). Grain growth is restricted by reinforced particles after recrystallization which can be observed by Fig. 2.4 shown below.

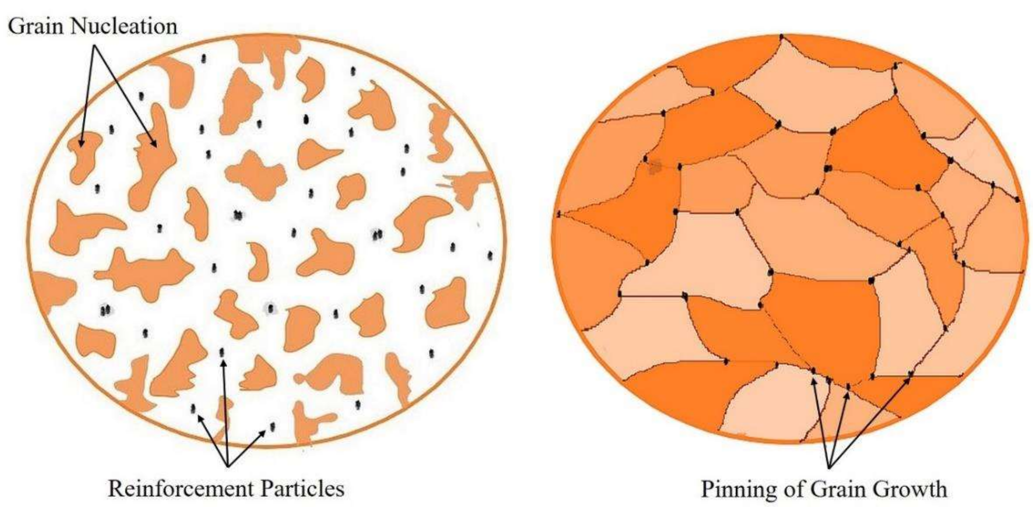


Figure 2. 4 Pinning effect: (a) nucleation of new grains at grain boundaries; (b) Blocking of the grain growth by reinforcement particles in MMCs after recrystallization

However, higher volume fraction of particles may led to agglomeration of particles (Rathee et al., 2017c). Hence, a proper volume fraction of particles is needed to choose for optimum results. Mathematically, grain size (Zener's) is estimated by the Eq. 2 (Shafiei-Zarghani et al., 2009).

$$D_z = \frac{4r}{3V_f} \qquad (2)$$

Where, D_z is critical radius, r is radius and V_f is volume fraction of the reinforced particles.

From this equation, it can be infer that increase in volume fraction of the particles will result in reduction in grain size of the composite.

Another model for calculation of grain radius was predicted by Rios et al. (Rios, 1987) in which grain radius was proposed to vary with radius and volume fraction of particles to be reinforced. Mathematically, it is given by

$$D_z = \frac{r}{6V_f} \tag{3}$$

Where D_z is the critical grain radius.

Some results have been reported in which Rios model was found to be more closely to the experimental data as compared to Zener's model. For example, in the work reported by Shamsipur et al. (Shamsipur et al., 2011), it was found that resulting mean granular size agree closely to Rios model rather than Zener's model.

2.6.3 Reinforcement strategies

Methods followed for pre-placing of reinforcement particles on the matrix is termed as reinforcement strategies. In addition to above mentioned processing parameters, suitable strategy chosen is compulsory for fabrication of defect free surface composites by FSP. After the establishment of FSP as a potential surface composite fabrication technique, numerous studies were conducted by various researchers to investigate the suitable reinforcement strategy. It started with the work of Mishra et al. (Mishra et al., 2003) in which they successfully fabricated Al/SiC surface composite via FSP by direct pasting of reinforced particles. Later on, it was discarded due to shortcomings encountered in the form of limitations of amount to be reinforced and thickness of composite layer fabricated as mentioned earlier. To overcome the limitations faced by direct pasting methods followed by

Mishra et al. (Mishra et al., 2003), different reinforcement strategies were proposed. During the investigation of other reinforcement strategies focus was also on prevention of wastage of reinforcements due to sputtering of particles during processing. (Miranda et al. (Miranda et al., 2013) reinforced SiC and Al_2O_3 in AA5083-H111 alloy matrix by FSP in order to fabricate functionally graded materials. The authors proposed three different reinforcement strategies for preplacing of particulates and reported that direct placing of particulates on the matrix is a quick process in which additional work like tool preparation and hole preparation is not required. However, tool preparation is required for hollow tool technique and produced thicker but less homogeneous surface layers as compared to other techniques.

The strategy followed for pre-placing of particulates on metal surface for fabrication of surface composites via FSP may be broadly divided in to three heads as shown in Fig. 2.5.

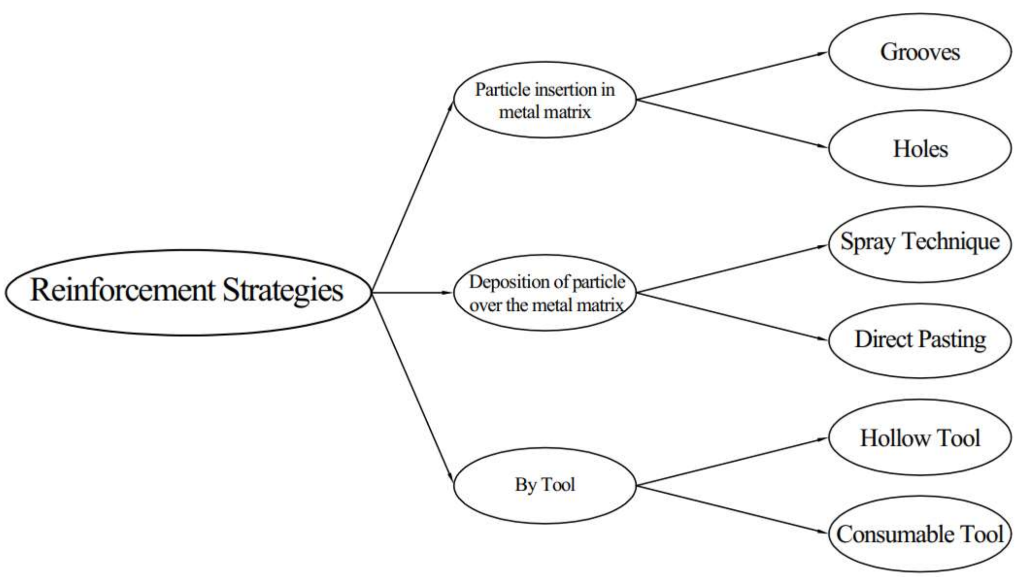


Figure 2. 5 Classification of reinforcement strategies

- Direct placement of particles on the substrate after mixing with some volatile solvent like methanol (Miranda et al., 2013, Kurt et al., 2011) or by spraying of particulates on metal surface by an appropriate technique like plasma spray (Li et al., 2016), high velocity oxy fuel spray, etc. (Zahmatkesh and Enayati, 2010, Anvari et al., 2013, Mazaheri et al., 2011).
- Placement of particulates in substrate through grooves (Dolatkhah et al., 2012, Sharifitabar et al., 2011, Salehi et al., 2012) / profiles (Bahrami et al., 2014b, Bahrami et al., 2014a) /holes of different geometries on metal surface (Yang et al., 2010, Arora et al., 2012, Akramifard et al., 2014)
- By utilising hollow and consumable tools with drilled holes (Huang et al., 2014)

It is well known that the uniform dispersion of particulates in the matrix is essential for better performance of the composites. The effect of reinforcement methods followed during surface composite fabrication via FSP with their characteristics are discussed here.

In the direct pasting of particles on the surface of substrate after mixing with some volatile solvent such as methanol, limitations over amount of reinforcement and thickness of processed layer were faced as mentioned earlier. In continuation of this method, another method was proposed in which through spray technique reinforcements were placed on the surface of substrate and then FSP was performed. In this method, particles dispersion was in wider region as dispersion of particles were governed by tool shoulder in place of tool pin. Zahmatkesh and Enayati (Zahmatkesh and Enayati, 2010) fabricated AA2024/Al2O3 surface composites via FSP and found to have uniform dispersion of alumina particles in aluminum matrix. The authors sprayed Al_2O_3 particles on the surface of aluminum matrix through plasma spray and deposited a layer of thickness 200 µm. Then FSP was performed on the coated plate. The hardness of the processed plate was improved approximately 2.5 times of the matrix. In another study, Mazaheri et al. (Mazaheri et al., 2011) used high velocity oxy-fuel spraying method to deposit 5 vol% of Al_2O_3 on A356 substrate in order to fabricate A356/Al_2O_3 surface composite. After that FSP was performed on the coated plates which led to consolidation of composite layer without any defects. Hodder et al. (Hodder et al., 2012) used cold spray technique along with FSP to deposit Al_2O_3 on aluminum matrix and subsequently it was processed. The maximum volume fraction of particulate deposited was 48%. The hardness of the composite improved significantly (1.5 times).

The most frequently used reinforcement technique for surface composite fabrication is groove technique (Devaraju et al., 2013, Asadi et al., 2011, Shafiei-Zarghani et al., 2011). The schematic illustration of the groove technique has been depicted in Fig. 2.6.

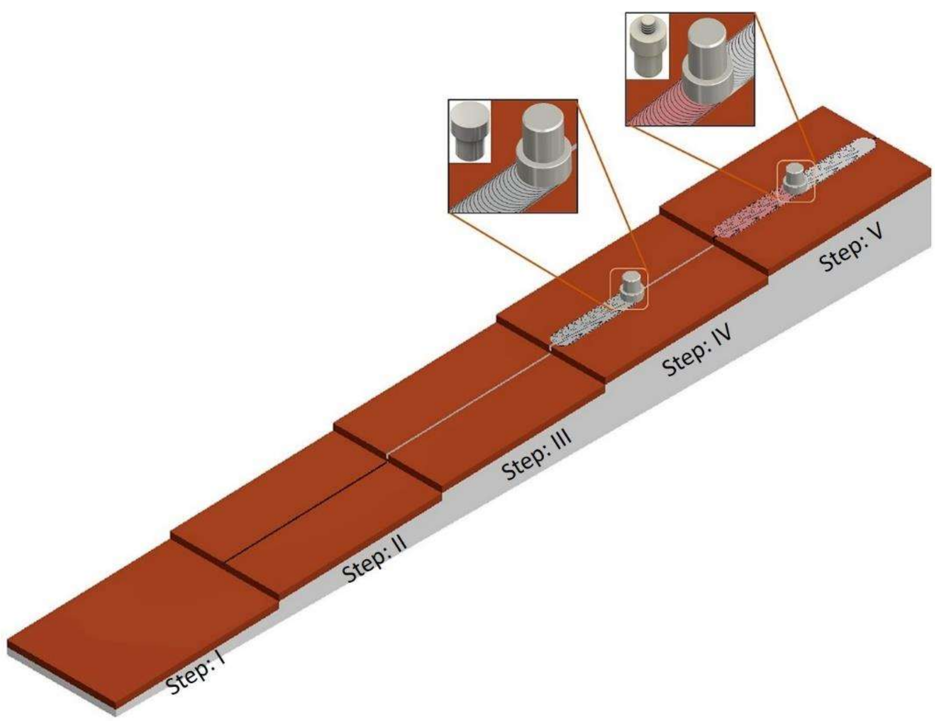


Figure 2. 6 Steps in SC fabrication using the groove technique

In this technique, a groove of known dimension (according to volume fraction of the particulate to be reinforced) is machined in the middle of the plate to be processed. Then, particulates are compacted carefully in the grooves. A pin less tool is used for closing the grooves generally known as capping for prevention of wastage of particulate by sputtering during processing stage. Finally, in the last stage, FSP is performed with a tool which has pin. Some studies have been reported in which, instead of using capping stage, groove or drilled holes were closed by using a thin sheet of same material as substrate to avoid the wastage of particulates during processing (Lim et al., 2009, Avettand-Fènoël et al., 2014, Don-Hyun et al., 2013, Mahmoud et al., 2008, Sharma et al., 2016) as shown in Fig 2.7 (a) and (b).

Sharma et al. (Sharma et al., 2016) fabricated AA7075/MWCNT surface composite by FSP in which they used AA6111 plate of 1.1 mm thickness as cover plate to prevent sputtering of particles from the surface of plate during processing. In another study, Avettand-Fènoël et al. (Avettand-Fènoël et al., 2014) used 0.2 mm thick cover plate to minimize wastage of Y_2O_3 particles on the surface of copper plate surface during processing of Cu/Y_2O_3 surface composite. In place of thin sheet, utilization of thick cover plate has also been reported in fabrication of surface composite by FSP. For example, Mahmoud et al. (Mahmoud et al., 2008) used a 2 mm thick aluminum plate as a cover plate to prevent the escapement of particles on the surface during processing. Utilization of an aluminum tape instead of thin/thick cover plate for prevention of particles sputtering on surface of plate have also been reported (Don-Hyun et al., 2013). However, this may create problem in mixing and bonding of cover plate with the matrix which need to be observed carefully.

Apart from cover plate, for elimination of capping stage during processing of surface composite by FSP, some results have been reported where blind drilled holes techniques were used (Akramifard et al., 2014, Li et al., 2013). Fig. 2.7 (b) shows the schematic illustration of blind drilled holes techniques used for fabrication of surface composites by FSP. Li et al. (Li et al., 2013) used blind drilled holes techniques for fabrication of surface composite. Blind holes of 1 mm diameter were machined at a distance of 0.5-2 mm from each other. Reinforcements were compactly filled in the holes and then FSP was performed. Capping of drilled blind holes were automatically occurred by the half part of shoulder ahead of traversing pin. Akramifard et al. (Akramifard et al., 2014) used twin drilled holes arrays on copper plate for the fabrication of Cu/SiC composite.

Although, sputtering of particulates on the surface of plate was eliminated by the groove method or hole method but, it takes a lot of time and effort in machining the groove or holes on the surface of plates. Huang et al. (Huang et al., 2014) proposed an alternate method for fabrication of surface composite via FSP in which there is no need to machine groove or hole on the surface of plate and hence reducing effort and time. In this technique, direct FSP can be performed as shown in Fig. 2.7 (c). In this technique, they used a hollow tool without pin having 8 mm diameter through hole at the center for fabrication of AZ31/SiC composite. SiC particles were filled in the tool initially which slowly moved to fill the space between shoulder and deformed zone. The SiC particles are then inserted in the specimen as tool traverse longitudinally. However, this method has limitations over depth of fabricated surface composite layer.

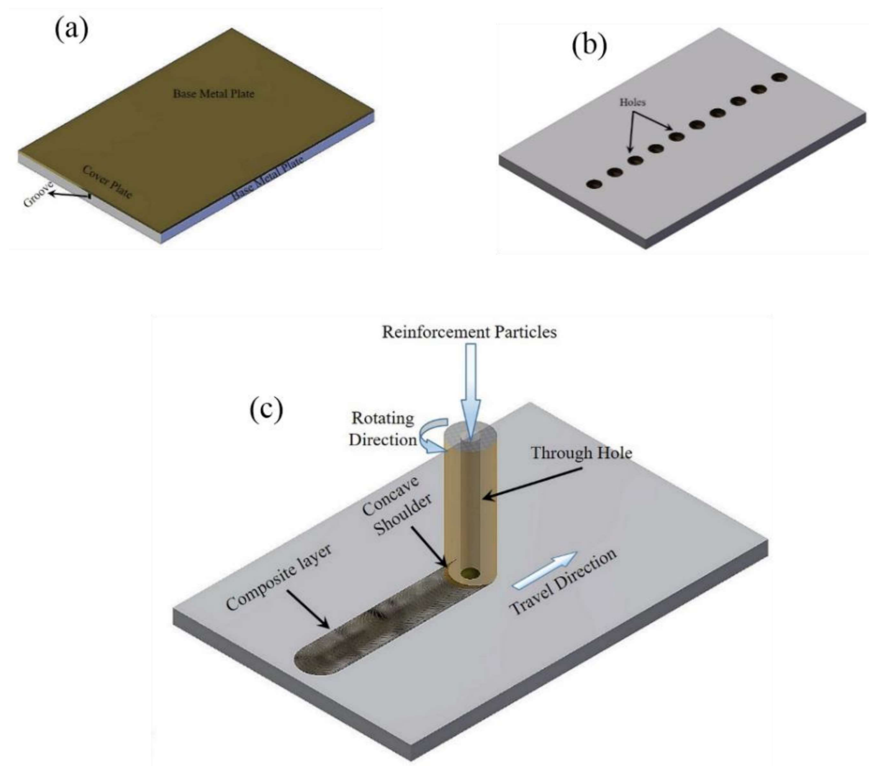


Figure 2. 7 Schematic view of: (a) groove covered with thin cover plate (b) drilled hole pattern and (c) introduction of reinforcements via a hollow tool

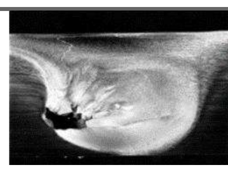
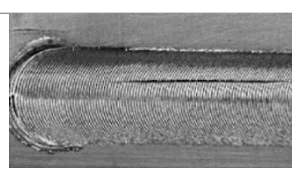
2.7 Defects in FSPed composite surfaces

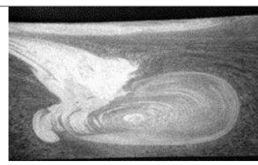
Table 2.2 depicts the common defects encountered during FSW/P of materials. The processing parameters are the main cause behind the appearance of defects in FSPed zone during FSP/W. Improper selection of processing parameters lead to formation of defects in SZ of FSPed materials. The reinforced particles may influence the material flow during FSP of surface composites which may increase the chances of formation of defects. The defects generally encountered during FSW/P are worm hole, scalloping, ribbon flash, cavity, surface lack of fill, nugget collapse and surface galling ((Arbegast, 2008).

Due to insufficient flow of material during FSP/W, defects such as cavity and tunnels are formed. As mentioned earlier, rotational speed, traverse speed and plunge depth are the vital parameters for defect free processed zone during FSW/P

(Elangovan et al., 2008, Padmanaban and Balasubramanian, 2009). Defects are prone to occur on the advancing side as there occurs an abrupt microstructural transition from SZ to TMAZ. However, this transition is gradual on retreating side (Nandan et al., 2008). Kim et al. (Kim et al., 2006) indicated defects like flash and cavity during their study on FSW of cast aluminum ADC 12 alloy. They pointed the reason for flash formation as excess heat input whereas, cavity formed due to insufficient stirring of the material. Elangovan et al. (Elangovan et al., 2008) indicated that tool probe can also be responsible for defect formation in FSP/W which they observed during their study on AA2219 alloy. Triangular cavities near the SZ in SiC/AA5083 surface composites were observed by (Gandra et al., 2011). They also observed clustering of particles in SZ of the fabricated SiC/AA5083 surface composite along with cavity formation on the advancing side. The cavity formation on the advancing side was due to insufficient material flow.

Table 2. 2 Common defects encountered in FSW/P (Arbegast, 2008)

Defect		Explanation	Reasons
Worm Hole		An advancing side tunnel of inadequately consolidated and forged material running in the longitudinal direction	Excessive travel speed for given rotational Speed Cold weld Too low weld pitch
Surface lack of fill		A continuous or intermittent top surface void on the advancing side	Insufficient flow arm formation across top surface Insufficient forge pressure Improper backside support Insufficient plunge depth

Ribbon flash		Excessive expulsion of material on the top surface leaving a corrugated or ribbon like effect along the retreating side	Excessive forge load or plunge depth Excessively hot weld Too high weld pitch
Surface galling		Galling and tearing of the metal on the top surface of the weld beneath the pin tool	Sticking of metal to pin tool Excessively hot weld Too high weld pitch
Nugget collapse		Improper formation of dynamically recrystallized nugget shape	Excessive Flow Arm Formation and injection of material into advancing Side Excessively hot weld Too high weld pitch

2.8 Microstructural characterization

Materials experience extensive stirring action of the tool, frictional heating due to relative motion between tool and work piece, and plastic deformation during FSP which leads to dynamic recrystallization (DRX) (Morisada et al., 2006b, El-Rayes and El-Danaf, 2012, Karthikeyan et al., 2010) . Due to aforementioned action experienced by materials i. e. stirring action of the tool, frictional heating and plastic deformation with DRX, new grains at favorable sites are generated and hence material results into fine and equiaxed grain structure due to which grain boundaries transform from lower to higher angle. Furthermore, due to the presence of reinforcement, recrystallization enhances through multiple heterogeneous nucleation sites (Nia and Nourbakhsh, 2016).

During FSP, grain refinement or grain size enhancement can occur simultaneously due to annealing effect. There are two dominant factors which affects the grain size structure during FSP which are, 1) annealing effect due to frictional heating, and 2) pinning effect due to reinforcements. Both these phenomena has contradictory effect on grain structure. On one hand, frictional heat may facilitate grain growth during FSP (Faraji and Asadi, 2011), and on another hand, the reinforced particulates may act like hurdle against grain boundaries, thereby restricting granular growth (El-Rayes and El-Danaf, 2012). Also reinforcements act as nucleating sites for new grains to generate which further hinder the grain growth and results into fine grain structure.

Kumar et al. (Kumar et al., 2016) fabricated 5083 Al–W surface composite by FSP. The authors stated that the initial grain size of the matrix was 25 µm which reduced to 3 µm after FSP of 5083 Al–W surface composite. They argue that the grain size refinement was due to dynamic recrystallization.

Saravanakumar et al. (Saravanakumar et al., 2017) synthesize Cu/AlN (0, 6, 12, 18 Vol%) copper matrix composite on pure copper substrate through FSP. Initially grain structure of pure copper was coarse and elongated with average grain size of 28 µm. The grain structure of the fabricated surface composite was found to be fine and equiaxed and the average grain size was estimated to be 2.8 µm. They stated that three factors affected the grain structure of the composite, i.e. frictional heat, plastic deformation and incorporation of AlN particles in the matrix. The frictional heat and plastic deformation led to dynamic recrystallization which reduce the grain size. The reinforced AlN particles act as nucleating sites for new grains to form which hindered the grain growth during recrystallization and led to grain refinement.

Sabbaghian et al. (Sabbaghian et al., 2014) fabricated Cu-TiC surface composite by FSP. The grain size of the pure copper was reduced drastically during FSP of Cu-TiC surface composite (150 μm to 5 μm). The reduction in grain size of the composite was due to dynamic recrystallization. The reinforced particles act as nucleating sites for new grains to generate which further hindered the grain growth and resulted into fine grain structure. Table 2.3 shows the grain size reduction during FSP of various surface composites fabricated.

Table 2. 3 Grain size reduction in stir zone of fabricated surface composites

Matrix used	Reinforcement	Grain size (μm) Base Material	Grain size of (μm) Matrix in composite	References
Al5052	SiC	243	0.9	(Dolatkhah et al., 2012)
Al7075	TiN	78	1.4	(Hussain et al., 2016)
AZ31	SiC	16.57	1.24	(Huang et al., 2014)
AZ31	Al_2O_3	70	2.2	(Azizieh et al., 2011)
Al 5052	Al_2O_3	25	0.94	(Sharifitabar et al., 2011)
Cu	SiC	50	5.3	(Barmouz et al., 2011a)
Cu	SiC	120	6	(Akramifard et al., 2014)
Cu	B_4C	35	2	(Sathiskumar et al., 2013)
Cu	TiC	150	3	(Sabbaghian et al., 2014)
Cu	AlN	28.8	2.8	(Saravanakumar et al., 2017)
Cu	RHA	30	1.8	(Dinaharan et al., 2017a)

2.9 Mechanical characterization

Enhancement of mechanical properties of the SCs fabricated via FSP can be attributed to different strengthening mechanisms which individually or collectively come in to play during FSP. If a material offers higher resistance to dislocations movement, it is considered to be of higher strength. From literature it can be seen that various researchers have reported strengthening mechanism in their own way (Lloyd, 1994, Kim et al., 2013, Zhang and Chen, 2008). Four different strengthening mechanisms reported by Lloyd (Lloyd, 1994) in their study are as follows: Orowan,strengthening, grain refinement strengthening mechanism (Hall–Petch relationship), work hardening and variation in thermal expansion coefficients (CTE) of metal matrix and reinforcement. Zhang and Chen (Zhang and Chen, 2008) evaluated strengthening mechanisms effect for metal matrix nanocomposites (MMNCs) and reported that Orowan strengthening plays a major role in MMNCs as the size of nanoparticles decreases. It increases to its maximum level for critical particle size. Any further decrease in size of particle amounts to a decreased Orowan strengthening effect. Critical nanoparticle size in the case of Mg/Al_2O_3 and Ti/Y_2O_3 nanocomposites was found to be 5.44 times the Burger vector or atomic diameter of the matrix. Also, it was reported that critical particles size is independent of volume fraction of RPs. However, impact of Hall–Petch strengthening has not been taken into account in this approach.

Out of several available modeling methods for predicting the strength of MMCs, Zhang and Chen approach are more reliable for MMNCs. The Clyne approach can be trusted for prediction of results closer to experimental ones for micro-composites. But, the Hall–Petch effect has not been considered by both Zhang and Chen method which may amount to deviation in predicted results from actual ones (Sanaty-Zadeh, 2012). In another study, Sanaty-Zadeh (Sanaty-Zadeh, 2012) considered four mechanisms: Orowan strengthening, Hall–Petch, load bearing and CTE difference by following the Clyne method. Proposed model findings were validated with experimental results on Mg nanocomposites using Al_2O_3 and Y_2O_3 reinforcement. They reported the Hall–Petch relationship to be major strengthening mechanism for micro composites also. The major mechanical properties are microhardness and ultimate tensile strength. Similarly, (Shafiei-Zarghani et al. (Shafiei-Zarghani et al., 2015) considered four strengthening mechanisms: Orowan strengthening, Hall–Petch, load bearing and CTE difference in Ti/Al_2O_3 nanocomposites fabricated using FSP. They fabricated SCs at three different groove widths of 0.8 mm, 1.2 mm and 1.6 mm. Two sizes of RPs viz. 80 nm and 20 nm were used.

2.9.1 Microhardness

As it is well established fact that during FSP of materials, two contradictory parameters are involved which need to be considered before estimation of microhardness of the fabricated surface composites. Friction stir processing of materials lead to drastic change in grain size of the material due to dynamic recrystallization which results in enhanced hardness of the material according to well-known Hall-petch equation (Ma, 2008). Contrary to this, as FSP involves relative motion between tool shoulder and the work piece which increase the temperature of the work piece due to frictional heating along with plastic deformation amounting to annealing of material (Mishra and Ma, 2005). Because of this annealing effect during FSP, dislocation density and compressive residual stress of the material decreases which reduces the hardness of the materials. So if the hardness of the processed material is higher than the unprocessed materials then the effect of annealing is dominated by the grain refinement effect and vice-versa (Dolatkhah et al., 2012). Besides this, improvement in hardness value due to secondary particle strengthening is also important in case of surface composites fabricated by FSP. Thus, the strengthening is the combined effect of grain refinement and uniform dispersion of reinforcements imposed by FSP (Izadi et al., 2014). Dolatkhah et al. (Dolatkhah et al., 2012) reported that the change in microhardness is a combined effect of reduction of grain size, the presence of hard phase RPs and quench hardening effect which is because of different thermal contraction coefficients of matrix and RPs.

Dinaharan et al. (Dinaharan et al., 2017b) fabricated AA6061/FA surface composite via FSP by using different volume fractions (0-18 Vol%) of FA particles in aluminium matrix. They stated that particle distribution was uniform throughout the SZ of the fabricated surface composite along with clean interface between FA particles and aluminium matrix. The hardness of the composite was found to be increasing with increasing volume fraction of FA and it was maximum at 18 Vol% (127 HV). The improved hardness of the composite was due to incorporation of hard phase FA particles in aluminium matrix, drastic grain refinement due to dynamic recrystallization and difference in coefficient of thermal expansion between matrix and particles which generates additional dislocations.

Saravanakumar et al. (Saravanakumar et al., 2017) manufactured Cu/AlN surface composite via FSP. They used different volume (0-18%) fractions of AlN particles in copper matrix. The results showed increasing trend of hardness value with increasing volume fraction of particles and the maximum value was at 18 Vol%. The improvement in the hardness of the fabricated surface composite is ascribed to grain refinement and uniform dispersion of AlN particles in copper matrix.

H. Sarmadi et al. (Sarmadi et al., 2013) used FSP to fabricate copper-graphite surface composite for its application as bearing materials. They used different pin profile to see the effect of pin profile on particle dispersion in copper matrix. The results showed that triangular pin profile produce more homogeneous particle dispersion. The hardness of the surface composite decreased with increasing volume fraction of the graphite. The decrement in hardness with increasing volume fraction was due to the presence of softer graphite particles in comparison with copper matrix.

Sabbaghian et al. (Sabbaghian et al., 2014) used FSP to reinforce TiC particles in copper matrix in order to fabricate copper based surface composite. The processing parameters traverse speed and rotational speed were 50 mm/min and 1000 RPM. The results showed that TiC particles were dispersed uniformly in copper matrix along with excellent bonding. The hardness of the composite improved significantly (1.7 times).

Dinaharan et al. (Dinaharan et al., 2017a) used waste (Rice husk) as reinforcement in copper matrix for fabrication of copper based surface composite. The authors used different volume fraction (0-18 Vol %) of rice husk particles. The results showed that hardness of composite improved with increasing volume fraction of the particles and maximum hardness was recorded at 18 Vol%.

2.9.2 Tensile strength

Tensile properties of any composite material is usually governed by many metallurgical parameters including grain size, bonding between matrix and the reinforcements and also the dislocation density that exists between matrix and reinforcements (Sun and Apelian, 2011).

If the tensile strength of SCs is low then this can be accounted to any of the aforementioned reasons but the most influencing of them is agglomeration of hard particulates, causing the reduction in elongation and existence of dimples within the RPs. However, if the strength of the fabricated composite is improved which is generally the case, it may be attributed to the presence of uniformly distributed particulates in metal matrix. It is a well-established fact that grain size of the matrix undergoes remarkable refinement during FSP mainly because of recrystallization and the pinning effect. Smaller sized reinforced particles have a tendency to stop sliding of grain boundaries (GBs), if the particles are resting on the GBs (B.T. Balakrishna, 1980). Additionally, they restrict the dislocation movement resulting in improved strength, if the particles are inside the grains (Ibrahim et al., 1991).

During tensile testing, dislocation develops a tendency to move inside grains owing to increasing tensile stress. This slipping is restricted by RPs. Dislocation line forms a loop around RPs resulting in increased dislocation density thereby necessitating requirement of higher stresses to cause material deformation. Thus, uniform dispersion of smaller RPs results in enhancement of tensile properties of SCs.

Barmouz and Givi (Barmouz and Givi, 2011) fabricated Cu/SiC surface composite by utilizing different size and volume fraction of SiC particle in copper matrix via FSP. Results showed that FSP without reinforcement increases percentage elongation significantly but decreases tensile strength of copper. Incorporation of reinforcement showed decreased tensile strength but increasing volume fraction and decreased particle size yielded improved tensile strength and decreased percentage elongation.

Dinaharan et al. (Dinaharan et al., 2017b) fabricated and characterized AA6061/18 Vol% RHA AMC by FSP. The authors reported that RHA particles dispersion in aluminum matrix was uniform throughout the SZ along with excellent interfacial bonding. Fragmentation of RHA particles happened during FSP. The tensile strength of the fabricated composite improved as compared to base aluminum.

Raju and Kumar (Raju and Kumar, 2014) fabricated Cu-Al_2O_3 surface composite by FSP. The authors reported that volume fraction of particles, tool tilt angle and concave angle of shoulder influence the properties of the fabricated composite. The mechanical properties of the composite such as hardness, ultimate and yield strength of the composite improved significantly. The improvement in mechanical properties of the composite was believed to be due to reinforcement of Al_2O_3 particles which increases the temperature of recrystallization by pinning the grain boundaries of copper matrix and blocking the movement of dislocations.

2.10 Tribological behaviour

Various engineering components involves surface interactions due to their running environment, and friction and wear have become an important issue in such circumstances. Although, wear of materials is considered to be affected by various factors such as materials properties, running conditions, geometry of contacting surfaces and environment but hardness of the material is considered as most influencing factors. So, improvement in hardness is considered as most promising method in protection against friction and wear. In this regard, MMCs where soft metal matrix is reinforced with hard and stronger particulates has got popularity across the globe.

In dry sliding wear of MMCs against a harder face (hardened steel), four types of wear are mainly observed: (i) abrasive wear (ii) adhesive wear (iii) oxidative wear and (iv) delamination (Sannino and Rack, 1995, Zhang et al., 1995, Singh and Chauhan, 2016). The occurrence and prevalence of a particular type of wear is governed by two main factors namely applied load and sliding distance (Zhang et al., 1995). At low loads, the counter-face is quickly abraded by hard asperities until a steady state is reached. This is called running-in.

The friction coefficient increases to a maximum and then drops down to a steady state. Consequently wear loss increases rapidly initially and then drops off to a steady state. Initial increase in wear loss is due to the fact that the hard reinforcements protrude out and abrade the counter-face like a tool. At the same time, the asperities from the steel surface abrade the sample surface and form grooves. This abrasion last for very short period of time and wear loss reaches to a steady state. The examination of the worn surfaces and wear debris produced in this regime revealed formation of oxides. The temperature rises due to the frictional heat and oxidation of the steel surface readily occurs. The oxide particles or an oxide film lubricate the surface and bring down the friction coefficient and the wear loss (Zhang et al., 1995). With increasing sliding distance and/or load, the asperities deform and the mating surfaces make adhesive contact. The deformed asperities generate wear particles which are typically flaky type (Zhang et al., 1995, Singh and Chauhan, 2016). Cross-transfer of material from the sample to the steel surface and vice versa also takes place due to the adhesion. Therefore adhesive wear can be characterized by flaky type wear debris and presence of the counter-face material on the worn surface of the sample. The friction coefficient and volumetric wear increases due to material removal in this regime. The generation of wear particles transforms the wear from two-body to a three-body abrasion wear. It may be also noted here that a tribo-layer forms just beneath the worn surface due to severe deformation and material flow and mixing. This layer is also known as mechanically mixed layer (MML) and consists of materials from both the mating surfaces (Venkataraman and Sundararajan, 1996). The MML layer also contains finer fragmented reinforcement particles in case of MMCs. At high loads subsurface cracks grow due to large strain. In MMCs, the crack can initiate at the particle matrix interfaces. As the worn material is removed from the surface layer, the crack approaches the surface and causes material removal by delamination. Delamination wear causes excessive material removal in chunks leading to entry in the severe wear regime.

Mild wear is observed in MMCs and unreinforced matrix at low loads and their wear resistance does not show much difference. However, at higher loads, wear mechanism change from two body abrasion to severe delamination wear. The transition of wear mechanism from mild to severe depends on load. In severe wear zone MMCs show better wear resistance as compared to unreinforced metals and its alloys. The transition from mild wear to severe wear is not prohibited by the high strength reinforcements rather it is delayed at higher loads and the transition load is found to increase with increasing volume percentage of reinforcements (Sannino and Rack, 1995, Singh and Chauhan, 2016).

Wear behavior of various surface composites fabricated by FSP has been investigated (Raaft et al., 2011, Eskandari et al., 2016, Devaraju et al., 2013, Soleymani et al., 2012, Ghasemi-Kahrizsangi and Kashani-Bozorg, 2012, Rejil et al., 2012, Qu et al., 2011, Shafiei-Zarghani et al., 2011, Mahmoud et al., 2010, Hashemi and Hussain, 2015, Golmohammadi et al., 2015, Lu et al., 2013). In most of the cases wear characterization was performed under dry sliding condition using a pin on plate unidirectional apparatus wherein the specimens were used as the pin and a hardened steel disc as the counter-face and it was found that the wear rate of

SCs were lower than the unreinforced substrate fulfilling the requirement for which they are manufactured. The improved wear behavior of the SCs is generally ascribed to the scattering of hard ceramic particles and associated increment in the hardness. Mathematically, the relationship between hardness and wear rate is expressed by Archard's equation (Archard, 1980).

$$V = Ʀ \frac{NS}{H} \tag{4}$$

Where V is the volumetric loss because of wear, N is the applied normal load, S is the sliding distance, H is the hardness, and Ʀ is a dimensionless constant known as wear coefficient. It can be seen that wear loss is inversely proportional to the hardness. So as hardness of the composite increases due to reinforcement of hard ceramic particles, wear rate decreases. As grain refinement leads to improvement in hardness according to Hall-Petch relation, it also contributes to decrement in wear loss. Due to load-bearing capability of the reinforced stronger ceramic particles, wear resistance of the surface composite also improves (Bauri and Surappa, 2008). The hard and strong reinforcements bear the load during the wear process and reduce the direct contact between the specimen and the disc (counter-face).

Saravanakumar et al. (Saravanakumar et al., 2017) reinforced aluminum nitrate (0, 6, 12, 18 Vol%) in copper matrix via FSP and assessed the wear properties of the surface composite. In their study, they reported that wear resistance of the composite improved significantly at 18Vol% of the reinforcement. They infer that improved wear resistance of the surface composite is because of the improved hardness of the composite. AlN particles protrude out of the surface of the sliding specimen after a brief initial period of sliding and bear the normal load. The net contact area on the counter disc is reduced. The load bearing action of the

AlN particles further decreases the coefficient of friction. The separation of AlN particles from the surface is delayed due to intimate interfacial bonding with the copper matrix.

Thankachan and Prakash (Thankachan and Prakash, 2017), in another study, used AlN as reinforcement in copper matrix by FSP and adjudged mechanical and tribological behavior of the composite. They also reported improvement in the wear resistance of the fabricated composite. The reason for this drastic improvement was uniform distribution of AlN particles into the copper matrix, enhancement in hardness value of the developed surface composites and reduction in copper surface contact area with rotating disc as these AlN particles carry away the load henceforth reducing the matrix material's wear rate.

Dinaharan et al. (Dinaharan et al., 2017a) manufactured Cu/RHA (0, 6, 12, 18 Vol%) copper based surface composite by FSP. They assessed performance of fabricated composite in terms of microstructural features, hardness and wear. The results showed that grain structure of the composite in SZ was fine and equiaxed. The hardness of the composite improved significantly along with wear resistance and maximum value of hardness and wear resistance was found at 18 volume percent reinforcement of RHA particles.

Dinaharan et al. (Dinaharan et al., 2016b) produced copper matrix surface composite by FSP. The authors used four different particulate in copper matrix namely: SiC, Al_2O_3, B_4C and TiC. Constant volume fraction and processing parameters were used for fabrication of composites. The results indicated improvement in hardness and wear resistance of the composites.

2.11 Electrical conductivity

The electrical conductivity of a material is measured as materials ability to conduct electrons through it. At microscopic level, electric current is related to the movement of electrons through it. It is commonly expressed as %IACS, which is the acronym for International Annealed Copper Standard corresponding to the electrical conductivity measurement as a percentage of a standard conductivity of pure copper at 25^0C. The electrical conductivity of materials depend on electron movement. In other word, we can say that it is the function of crystalline structure of exiting phases as well as on the crystal defects such as voids and interstitials, dislocations and twins and grain boundaries. Grain size also affect electrical conductivity due to total length of grain boundaries per unit area. (Bautista et al., 2010) argued that electrical conductivity depend on mechanical strain. However, experimentally it's been established that electrical conductivity does not change significantly until a strain of 1.8 (Faria, 2010).

Zou et al. (Zou et al., 2017) stated in their study on TiB_2 particulate reinforced copper matrix composites fabricated by casting method, based on in situ precipitation reaction between B and Ti elements to form TiB_2 particles in molten copper that the electrical conductivity decreased from 99.92% IACS for pure copper to 73.43% IACS for the Cu-1.5 wt%TiB_2 composite. The reason for the decrement in electrical conductivity of the composite was cited to be more scattering of electrons due to impurities, defects, dislocations and grain boundaries.

Lu et al. (Lu et al., 2015) fabricated W–Cu/CeO_2 composites from Cu-coated W and CeO_2 composite powders. When the addition of CeO_2 reaches 0.25 wt%, the composites perform the best electric conductivity of 69.41% IACS, which is higher than the national standard (W–Cu) of 65.26%. However, with further addition of CeO_2 to 1 wt% and 2 wt%, the composites show numerous pores and united particles and exhibit a lower density, which is prone to damage the properties of the alloys.

2.12 Scope of the present work

Surface engineering is getting popularity across the globe for engineering applications where surface interactions are involved. The surface of such components is either protected or modified in such a manner that suits the interaction at the surfaces. In this regard, several technologies have been developed. The research fraternity has participated in a revolution as far as the shaping of surface properties is concerned. The shaping of surface properties largely involves physical, chemical and solid-state mechanical treatments. However, challenges are there related to economic issues, environmental and energy aspects and material performance. Constraints are there to minimize production costs, to have low impacts on environmental emissions and minimize solid and liquid disposals along with low energy consumptions. Therefore, green processes, efficient from an energy point of view are required.

Pure copper is inherited with superior electrical and thermal conductivity, high formability, good ductility, and excellent oxidation and corrosion resistance. Due to these characteristic attributes, copper drew a lot of attention from several industries which were not limited to optical, thermal and electrical industries. However, copper is known to have low strength and hardness, poor wear resistance and inferior arcing resistance. Therefore, for the applications such as bearing bushes, nozzles, electrical connectors, railway overhead current collector etc. those properties require considerable improvement.

Conventionally, the surface of light metals such as aluminum, magnesium, copper etc. can be significantly improved by putting hard coating layer on the surface through techniques like physical vapor deposition, hard anodizing and ion beam enhanced deposition. However, the coating layer produced by these processes are too thin to sustain high load as it breaks easily with deformation of the matrix. Moreover, these processes are costly, time-consuming and have a harsh effect on the environment due to expensive consumables, long processing time and toxic emissions. Surface properties of light metals can also be improved by reinforcing hard ceramic and intermetallic particles in the surface of the materials. So far, different methods such as laser cladding, plasma spraying and micro-arc oxidation have been used to produce surface composites. As laser cladding is a melt based process where melting of metals occur which leads to several defects such as porosity, cracking, anisotropic and dendritic grain coarsening and formation of some detrimental phases due to the interfacial reaction between particulate and matrix. Whereas, in the case of plasma spraying and micro-arc oxidation techniques, there is an obvious stratification between composite layer and substrate and interface strength is limited. In order to overcome the problems associated with these aforementioned techniques, more advanced technologies are still desirable.

Solid-state technologies, known from the middle of the twentieth century, were revisited to meet these new requirements. The development of the friction stir welding concept and its applications to surface modification opened up new possibilities to improve the surface properties of components produced by conventional technologies. Friction stir processing (FSP) has been intensively investigated in recent years as a solid-state process with an enormous potential to modify material surfaces.

So in lieu of above, present work aim to fabricate copper based surface composites through friction stir processing route. As the cost of MMCs are major issue in their applications so an attempt is made to use wastes (FA and Zircon sand) as reinforcements in copper matrix. A known ceramic zirconia has also been used as reinforcement to compare the performance of Cu/FA and Cu/Zircon surface composite with Cu/Zirconia surface composite. The performance of the fabricated surface composites have been adjudged through microstructural features, mechanical, tribological and electrical characterizations. The leach test of the fabricated composite (Cu/FA) was also conducted to assess its environmental impact.

2.13 Objective of the present investigation

- To utilise wastes as reinforcement in copper based surface composites
- To assess the performance of the composites
- To compare the performance of composites having wastes as reinforcement with that having known ceramic as reinforcement.