Review



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Journal of Materials Science & Surface Engineering



# Role of Friction Stir Processing on Copper and Copper based Particle Reinforced Composites – A Review

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Article history

### Abstract

Received: 24th-April-2015 Revised: 28th-May-2015 Available online: 29<sup>th</sup> May, 2015

Keywords: FSP, Copper, Surface composites, Microstructure, Mechanical properties Friction stir processing (FSP) is a green, energy efficient, one- step, solid-state surface modification technique. FSP is an emerging metal working technique and an effective method for producing fine-grained structure and surface composite, modifying the microstructure of materials, and synthesizing the composite. The objective of this review article is to provide the current state of understanding and development of friction stir processing technology on copper. This paper also presents the critical reviews on copper based particulates reinforced surface composites using friction stir processing route by various researchers in recent years. The researcher's findings with influencing parameters for obtaining successful copper based surface composites are given and discussed.

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# Introduction

Friction stir processing (FSP) is developed based on friction stir welding (FSW) technology [1]. Friction stir welding, a solid state joining technique invented in 1991 by The Welding Institute (TWI), is extensively used in joining of Al, Mg, Cu, Ti and their alloys [2]. In FSW, a cylindrical-shouldered tool, with a profiled probe or pin is rotated at a constant speed and fed at a constant traverse rate into the joint line between two pieces of sheet or plate material, which are butted together as shown in Fig. 1(a).



Figure 1: Schematic representation of (a) FSW and (b) FSP [3]

FSP is a green, energy efficient, one-step processing route and an emerging surface-engineering technology. It has been successfully applied to alter the grain structures of various metals and alloys to change the surface properties without influencing properties of the bulk material and also producing surface level composites [1-2]. It can also be used as a repair tool for sensitive parts. Actually, FSP is a novel grain refinement method based on strong couplings of thermo-mechanical phenomena, applied to light metal alloys for various end applications [4]. Fig.1 (b) depicts FSP and how it differs from FSW as in Fig.1 (a). FSP uses a specially designed, nonconsumable tool with shoulder and pin. The tool rotates at a constant rotational speed against the work surface moving at a fixed traverse (processing) speed to develop a friction stir processed zone. When the pin descends to the work piece and the shoulder contacts the work surface, heat is generated by friction between the tool shoulder and the top of the work piece causing plastic flow by the rotation of the tool pin. As the processed zone cools at ambient condition, it forms a defect free recrystalized fine grain microstructure. Fig.2 illustrates the schematic representations of step by step working principle of FSP.



**Figure 2**: Steps of FSP (a) rotating tool prior to contact with the plate (b) tool pin makes contact with the plate, creating heat (c) shoulder makes contact, restricting further penetration while expanding the hot zone and (d) plate moves relative to the rotating tool, creating FSP zone [5]

This thermo-mechanical conditions introduce three distinct regions namely stir zone (SZ), thermo-mechanically affected zone (TMAZ) and heat affected zone (HAZ) during FSP. The processed zone with various regions is illustrated in Fig.3.The formations of

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these regions are affected by the material flow behavior under the action of non-consumable tool. The degree of plastic deformation and the heat generation during FSP are the dominant factors in determining grain refinement at processed zone [2]. In FSP, heat is generated by a combination of friction and plastic dissipation during deformation of the metal. The dominating heat generation mechanism is influenced by the process parameters, thermal conductivities of the workpiece, pin profiles, tool geometry and the backing plate. Nevertheless, the amount of heat generation during FSP is a decisive issue to produce a defect-free FSPed zone. FSP has been applied to Al, Mg, Cu, Fe, and alloys with resulting property improvements. Fig. 4 shows a list of attributes and links to the FSP processes with potential applications. The benefits and limitations of FSP in different aspects are presented in Table 1.



Figure 3: Different regions in FSP (a) unaffected base metal (BM) (b) heat affected zone (HAZ) (c) thermo-mechanically affected zone (TMAZ) and (d) stir zone (SZ) [6]



Figure 4: Schematic view of list of attributes and links to the FSP processes [1]

Table 1: Benefits and limitations of FSP

Benefits

v	
Technical	One-step processing technique
	Good dimensional stability and repeatability
	Depth of processed zone can be controlled by the pin
	length
	Suitable for automation
	No surface cleaning required
Metallurgical	
Wietanurgiear	Solid state process
	Grain refining and homogenization
	Minimal distortion of parts
	No chemical effects and no cracking
	Excellent metallurgical properties
	Possibility to treat thermally sensitive materials
Energy	
	Energy efficient technique
	Low energy consumption route for surface composite
	fabrication
	Energy efficiency competing with fusion based processes
<b>F</b> 1	as laser
Environmental	Green technique
	No fumes produced
	Reduced noise
	No solvents required for surface degreasing and cleaning
Limitations	
Linnanons	New technique and availability of data
	Lack of predictive models in FSP
	Keyhole at the end of each pass
	Need of a backing plate and suitable fixture

#### Friction stir processing of copper

Today, copper and copper alloys remain one of the major groups of commercial metals, ranking third behind only iron/steel and aluminum in production and consumption. Copper and its alloys have found extensive applications because of its high thermal and electrical conductivity, plasticity, softness and formability. However, copper in pure form has poor strength, wear and fatigue resistance and hence is unsuitable for high end applications like contact terminals of electrical switches and sliding surfaces. Friction stir processing (FSP) overwhelmed the above limitations of the pure copper. Mishra et al [7] have developed this innovative solid-state processing technology, which is being used to enhance locally the mechanical properties of conventional materials by producing ultrafine grained structures, which is rather attractive in situations where strength and/or fatigue crack initiation are serious concerns. FSP is a unique process to modify the microstructure and other mechanical properties at selective locations [1-2]. The current understanding of FSP of copper and its alloys, with particular concern for the effects of process parameters, microstructure evolution, microstructure-property relationships and modeling are summarized in this section.

Surekha and Els-Botes, (2011) [8] have developed a high strength and high conductivity copper by FSP at low –heat input conditions by varying the traverse speed (50-250 mm/min) at constant rotation speed (300 rpm) . Grain size of the nugget decreased from 9 to 3  $\mu$ m and the hardness increased from 102 to 114 HV by increasing the traverse speed from 50 to 250 mm/min. Fig.5 implies that the yield strength (YS), ductility (% Elongation) and ultimate tensile strength (UTS) of the processed zone is higher compared to the base metal and the YS and UTS increased with the increase in traverse speed, the heat input and the grain size decreased and hence the mechanical properties improved and the yield strength obeyed Hall–Petch relationship.





Figure 6: Grain structures in the FSPed copper [9]

Jian-Qing Sua et al (2011) [9] have conducted FSP on commercial OFHC (C10100) copper to study the characteristics of microstructures at various locations behind the pin tool extraction site in copper with continuous quenching to enhance cooling rates. The substructures initially formed around the pin tool consist of very small crystallites having sizes of a few tens of nanometers (refer Fig.6). The formation of microband structures around pin tool appear as nanoscale elongated crystallites surrounded by highangle boundaries. The resulting microstructures consist of equiaxed, nearly random oriented grains surrounded by high-angle boundaries. The grain size ranges mainly from 50 to 300 nm with an average size of about 109 nm (number-weighted) and 174 nm (area-weighed), respectively.

Salar Salahi and Vahid Rezazadeh (2012) [10] have investigated the fracture mechanism in friction stir processed annealed pure copper samples along with microstructure and improvement in ductility. By varying the traverse speed from 40 to 100 mm/min at rotation speeds of 300 and 600 rpm, the ultrafine grain microstructures were achieved as depicted in Fig.7. Defects were observed in rotational speed of 300 rpm. By increasing traverse speed at constant rotational speed of 600 rpm, grain size of the stir zone decreased and ductility increased. Fracture mechanism of processed pure copper significantly depends on grain size and cavity formation during process. Grain size is controlled by heat input and cavity formation decreases with higher plastic deformation, produced by tool.



Figure 7: (a) Boundary between SZ and HAZ in sample processed in adequate heat input; (b) cavities and cracks in samples processed at tool speed of 300 rpm and (c-d) microstructure of SZ in sample processed at tool speed of 600 rpm and traverse speed of (c) 40 mm/min (d) 100 mm/min [10]

Galvao et al (2012) [11] have used friction stir processing technology to enhance locally the mechanical properties of phosphorus deoxidized copper (Cu-DHP) sheets. In their work, 1 and 3 mm-thick copper-DHP plates were processed with the aim of simulating surface (SFSP) and bulk/in-volume (VFSP) processing. It was found that the tool geometry, which has a close relation with the plastic deformation and dynamic recrystallization kinetics inside the stirred volume, the processing parameters and the heat exchange conditions, which determine the extent of dynamic recrystallization and annealing phenomenon, are determinant in FSP. In fact, for the range of processing parameters tested in this work, from Fig.8, it was found that grain size increases with decreasing traverse speed, in bulk processing, and with increasing rotational speed, in surface processing.

Xue et al (2012) [12] have prepared ultrafine-grained (UFG) copper with high strength and tensile ductility using a simple friction stir processing technique with additional cooling. Low tensile ductility owing to the insufficient strain hardening is the main drawback for UFG materials, which restricts their practical applications. Enhanced strain hardening capacity, which is effective in blocking and accumulating dislocations, was achieved in the present recrystallized UFG microstructure. UFG pure copper with high strength/ductility was successfully prepared by FSP. The engineering tensile stress-strain curves of the FSP Cu samples (FSP-1 and FSP-2), as well as the coarse grained (CG) Cu reference material, are compared in Fig. 9(a). The CG Cu exhibited low yield strength of ~60 MPa and an elongation of ~40% than other two samples. The sound tensile properties of the processed copper samples were attributed to the enhanced strain hardening capacity in the FSP Cu samples. which can be observed from the true stress-strain curve (Fig. 9b).It is obvious that the stress of the 16-pass equal channel angular processed (ECAP) Cu sample decreased quickly after a small plastic strain ( $\sim 2\%$ ). This work provides a strategy for designing UFG materials with good mechanical properties.



Figure 8: Evolution of the average grain size (GS) of the processed structures with the processing parameters [11]



Figure 9: (a) Tensile engineering stress-strain curves of FSP Cu and coarse grained Cu samples and (b) true stress-strain curves of FSP Cu and 16-passes ECAP Cu [12]

Xue et al (2013) [13] have successfully prepared a large-area bulk ultrafine grained (UFG) pure Cu by multiple-pass (5 pass) overlapping friction stir processing as shown in Fig.10(a) at a tool rotation rate of 400 rpm and a traverse speed of 50 mm/min under additional water cooling. Overlapping FSP did not exert a significant effect on the microstructure and mechanical properties of the FSP UFG Cu. Similar average grain size was achieved in the transitional zone (TZ) of the multiple-pass FSP sample compared to that in the stir zone of the single-pass FSP sample. Very weak softening occurred in the TZ of the multiple-pass FSP UFG Cu, resulting in a relatively uniform hardness distribution throughout the whole processed zone (Fig.10b).



**Figure 10**: (a) 5-pass overlapping FSP with cross-sectional macrostructure and (b) hardness distribution [13]

Salar Salahi et al (2013) [14] have investigated the microstructural evolution characteristics of friction stir processed pure copper. Copper plates were processed to the depth of 3.4 mm at different process conditions by varying the traverse speed from 30 to 120 mm/min at rotation speeds of 400 and 600 rpm. Defects were observed in rotational speed of 400 rpm. Table 2 shows the mechanical test results of various samples. Grain size of processed zone depended significantly on plastic deformation and heat input value. By increasing traverse speed at constant rotational speed of 600 rpm, grain size of the stir zone decreased and the hardness increased. Ultimate tensile strength increased with decrease in grain size. FSP was found as an effective method to develop fine-grained microstructure in copper plates.

 
 Table 2: Different experimental process conditions and mechanical test results [14]

Specimen number	Tool rotation speed (Rpm)	Traverse speed (mm/min)	Ultimate tensile strength (MPa)	Hardness on nugget zone (HV)	Grain size of NZ (μm)
1	400	30	210	78	32
2	400	120	219	84	23
3	600	30	245	81	49
4		50	255	87	41
5		70	259	90	35
6		90	263	91	30
7		120	269	99	24

Barmouz et al (2013) [15] have investigated the tensile behavior of friction stir processed pure copper including ultimate tensile stress, yield stress, and elongation under different processing parameters and FSP pass. It was observed that the processing parameters and pass number could have considerable effects on the tensile deformation properties of the pure copper such as 300% and 47% enhancements in elongation and ultimate strength, respectively. It was demonstrated that higher passes resulted in the development of ultrafine grains (up to 700-800 nm) in the specimens. Fig.11 (a)-(d) show the OM and SEM images of FSPed samples at different process parameters with 4 passes. The fracture surface morphology was also used to further support the elongation results.

Barmouz et al (2013) [16] have successfully fabricated ultrafined grain (UFG) structure in pure copper plate by FSP, implemented in single and two passes. The dislocation densities of both specimens are estimated by hardness indentation size effect method and it was used for clarification of the tensile deformation results. Multi-pass friction stir processed Cu had lower dislocation densities compared to single-pass one. Tensile properties of both specimens were examined in different strain rates and it was indicated that (as in Fig.12), the lower dislocation densities in multi-pass FSPed metal resulted in more significant alteration in its tensile behavior compared to single-pass FSPed specimen. Yield drop phenomenon also only occurred in highest strain rate of twopass FSPed metal.



Figure 11: OM images of the SZ of samples with 4 passes at (a) 630 rpm + 40 mm/min (b) 630 rpm + 315 mm/min, and (c) 1600 rpm + 40 mm/min (d) FESEM image of (a) [15]



Figure 12: True stress-strain curves of FSPed samples in different strain rate of (a) single pass (b) double pass[16]



**Figure 13**: (a) Average grain size vs. annealing duration (b) hardness vs. annealing duration at annealing temperatures of 600, 800 and 1000 °C [17]

Amir Mostafapour et al (2014) [17] have investigated the effects of annealing heat treatment on the microstructure characteristics of pure copper during friction stir processing. Samples processed in annealing temperature of 600°C had smaller grains relative to those processed at 800°C and 1000°C. The grain size of samples decreased with the increase in the duration of annealing treatment at the temperatures of 600 and 800°C (refer Fig.13a). Grain coarsening was observed in all samples annealed at 1000°C. The grain size of samples increased with the increase in the duration of annealing treatment at the temperature of 1000 °C. Grain size and dislocation density were major factors controlling hardness variations. Generally, grain size was dominant factor and samples processed at annealing temperature of 600 °C had higher hardness due to finer microstructure as shown in Fig.13 (a) and (b).

Cartigueyen and Mahadevan (2014) [18] have studied the effect of friction stir processing technique on pure copper with six different tool pin profiles (plain cylindrical - PC, threaded cylindrical -TC, triflute -TF, triangle -TR, square - SQ and hexagonal - HE) at 350 rpm as tool rotation speed and 50 mm/min as traverse speed to achieve the low heat input condition during the FSP. K-type thermocouples were embedded in the Cu plate to monitor the heat generation during FSP.Of the six tool pin profiles, the four pin profiles (threaded cylindrical, triflute, square and hexagonal) show successful formation of FSP surface on copper. From the investigation, it was observed that pin profiles are also responsible for heat generation during processing at stir zone. Threaded cylindrical (TC) pin profile tool is more effective in bringing about a favourable mechanical modification as revealed in Fig.14 in pure copper than other pin profiles under low-heat input condition.



Figure 14: Tensile properties of FSPed samples [18]



Figure 15: Grain refinement - function of heat input [19]

Cartigueyen et al (2015) [19] have investigated friction stir processing of pure copper experimentally to explore the effect of process parameters in two different combinations on thermal histories, resulting microstructure and mechanical properties. Ktype thermocouples were used to measure the temperature history at two different locations on workpiece. The results showed that the heat generation during FSP strongly depends on process parameters. The peak temperature obtained was 65% of the melting point of parent metal. Optical micrographs in Fig.15 revealed grain refinement depends strongly on the heat input during FSP. The microhardness of the processed copper plates was influenced by their grain sizes. The grain sizes were monitored by heat input during processing. High heat generation leads to grain growth in the stir zone which lowers the microhardness values. Fig.16 (a) and (b) show the effect of rotational and transverse speed on the resulting hardness of processed zone and compared with base metal value.

Cartigueyen and Mahadevan (2015) [20] have studied the influence of rotational speed on the formation of friction stir processing zone in commercial pure copper at low heat -input conditions. The experiments were conducted using K-type thermocouples to record the temperature history at different locations on the work piece. The results suggest that the temperature achieved during processing plays an important role in determining the microstructure and properties of the processed metal. FSP produced very fine and homogenous grain structure and it is observed that smaller grain size structure is obtained at lower rotational speed whereas a tunnel defect was formed at lower speed of 250 rpm. It is also observed that the hardness of the processed copper depends strongly on the rotational speed (heat input) during FSP as revealed in Fig.17. Tensile tests were carried out, and the tensile strength of the FSPed samples was compared to that of the base metal. For a successful FSP at low-heat input condition, the minimum rotational speed was found to be 350 rpm.



Figure 16: Variation of the surface hardness of FSPed sample with (a) rotational speed and (b) transverse speed [19]



Figure 17: (a) Hardness of BM and FSPed samples and (b) hardness as a function of tool rotational speed [20]

# Modeling of friction stir processing of copper

Despite the large number of studies, most of the work that has been done in the FSP field focuses primarily on experimental work. Only limited modeling attempts on temperature distribution, mechanical properties and strain rate analysis of FSP on copper and its alloys have been conducted. Modeling offers great potential for reducing experimental effort in the development of welding/processing parameters, tool design and many other areas and at the same time reduces cost and time. Several works on numerical modeling of heat transfer during FSW/P with moving heat source technique have been performed [21-26] and observed that the use of a moving heat source technique is proved to be a reliable method to simulate FSP [25]. One of the most important issues that hold back the wide spread use of FSP is the lack of predictive models than FSW. As compared to Al and Mg alloys, FSP on copper based materials have received very less attention in the numerical modeling domain so far. Only limited work has been done in the field of FSP modeling on copper.

Cartigueyen et al (2014) [27] have investigated the heat generation during friction stir processing of pure copper by numerical modeling and experimentation in two different combinations of processing parameters. They observed that the grain refinement depends strongly on the heat input during FSP. A three dimensional (3D), transient, non-linear thermal model was developed using ANSYS 11.0 software to simulate the thermal history during FSP of copper. The simulated temperature distributions (profile and peak temperature) are in good concurrence with that of experimental results. The results as shown in Fig. 18 showed that the heat generation during FSP strongly depends on both rotational and transverse speed where the peak temperature was observed to be strong function of the rotational speed while the rate of heating was a strong function of the transverse speed.



Figure 18: Simulation results of temperature profiles for pure copper at (a) different rotational speeds (constant transverse speed of 30mm/min);(b) different transverse speeds (constant rotational speed of 700rpm) [27]

# Copper based surface composites by friction stir processing route

For many engineering applications, the useful life of components often depends on their surface properties than bulk properties [28-29]. In these situations, it is desirable that only the surface layer of components is reinforced by ceramic phases while the bulk of components retain the original composition and structure with higher toughness. Friction stir processing (FSP) has evolved as a novel solid state, low-energy consumption route to prepare surface composites [30-31]. FSP has been used in various types of particle (SiC,  $B_4C$ , TiC, WC and  $Al_2O_3$ ) reinforced copper composite materials. This technique has been effectively explored by several investigators to fabricate surface composite on aluminum [32–35], magnesium [36-38], steel [39] and titanium alloys [40].

Most of the published work is focused on the effect of processing parameters on surface characteristics and techniques to evaluate the performance of modified surfaces. Nevertheless, the reinforcing particles deposition method is relevant in terms of structural and chemical homogeneity, agglomeration free and depth of the modified layer which influence the final surface performance [41]. Different methods for depositing reinforced particles have been reported such as (i) mixing reinforcing particles or powders with a volatile solvent such as methanol or a lacquer, in order to form a thin reinforcement layer, preventing reinforcing powders to escape [30,42] (ii) machining grooves of required depth and width in the substrate, compact with reinforcing particles and process the zone with a non consumable FSP tool in a single pass or in multiple passes along the groove [32-40],(iii) mixing reinforcing particles with substrate powders and coated on the substrate surface using thermal spray system and process the coated zone [43,44] and (iv) designing a net of blind holes of

required diameter and depth in the substrate, compact with reinforcement particles, plunge the tool and traverse along the FSP direction [57,58].

Barmouz et al (2011) [45-49] have successfully applied the FSP technique to fabricate copper based surface metal matrix composites (SMMC) in recent times. They developed copper based surface composites by FSP route with different FSP tool pin profiles and observed that the tool pin profile significantly influenced the formation of Cu/SiC surface composite. The straight cylindrical (SC) pin profile produces lower heat input rather than the square pin (SQ) profile because of its smooth geometry which results inhibition of grains growth. A straight cylindrical pin profile produced a uniform distribution of SiC particles (as depicted in Fig.19a-b) and finer grain size in the stir zone, increased hardness (Fig.20a) and wear resistance compared to the square pin profile [45].



Figure 19: Distribution of SiC particles with (a) square and (b) straight cylindrical pin profile [45]

Barmouz et al [46] have produced Cu/SiC surface composites using micron sized SiC particles via friction stir processing at different process parameters to enhance the surface properties of composites. The effect of traverse speed as the main processing variable on microstructure and microhardness of MMC layers was investigated. Microstructural evaluation indicated that an increase in traverse speed and a decrease in rotational speed cause a reduction in the grain size of stir zone (SZ) for the processed specimens without SiC particles. Higher traverse speeds resulted in poor dispersion of SiC particles and consequently reduced the microhardness values of MMC layers (as shown in Fig.20b-20c). It was found that upon addition of SiC particles, wear properties and friction coefficients were improved. It was also observed that, the MMC layer produced by FSP showed lower strength and elongation than pure copper while a remarkable elongation was observed for FSPed specimen without SiC particles.

Barmouz et al [47] have developed copper based surface composites by FSP route with micro and nano size of SiC particles. The size of the SiC particles considerably influenced the grain size and wear rate of Cu/SiC surface composite. Nano size SiC particles yielded finer grains and lower wear rate compared to micro size SiC particles. Increased volume fraction of both micro and nanosized SiC particles enhanced the wear resistance of the surface composites. Increasing the volume fraction or decreasing the reinforcing particle size enhances the tensile strength and wear resistance and lowers the percent elongation.

Barmouz et al [48] have used multi-pass friction stir processing (MFSP) for improvement of microstructural and mechanical properties of in situ Cu/SiC composites. Multi-pass FSP notably enhances the separation and dispersion of SiC particles and also reduces the grain size in the composite matrix, SiC particles size and porosity contents. Fig.21(a) and 21(b) show the increase in microhardness and remarkable enhancement of tensile properties caused by multi-pass FSP respectively. It was also found that the average friction coefficients of composites fabricated by multi-pass FSP were noticeably reduced compared to the pure copper.

Addition of SiC particles led to enhancement of electrical resistivity of pure copper. A negligible difference between the electrical resistivity of composites fabricated by 1, 4 and 8-pass FSP was also detected.



**Figure 20**: Microhardness profile (a) by different pin profiles [45] (b) of FSPed samples without SiC and (c) samples with SiCp in different traverse speed [46]



Figure 21: (a) Microhardness and (b) stress–elongation curves for pure Cu and multi pass FSPed Cu [48]

Barmouz et al [49] have produced successfully Cu/SiC nanocomposite layer using friction stir processing technique. FSP homogenizes and refines the copper structure and creates a

JMSSE Vol. 2 (2), 2015, pp 133-145

microstructure with nano-sized SiC particles (30 nm) distributed in the pure copper matrix. Results show that increase in traverse speed leads to a decrease in grain size and an increase in hardness value of FSPed specimens without SiC particles (Fig.22a). However, it has an opposite effect on the grain size and hardness of specimens with SiC particles as shown in Fig.22(b). In order to obtain a microstructure with fine grains and uniform distribution of reinforcing SiC particles, lower traverse speeds should be used. Cu/SiC nanocomposite shows a higher wear resistance  $(3.931 \times 10^{-5} \text{ mm}^3/\text{Nm})$ . However, SiC particles leads to a slight increase in friction coefficient because of obstacle action of SiC particles as shown in Fig.23 (a-b).



Figure 22: Microhardness profile of samples (a) without SiCp and (b) with nano SiCp [49]



Figure 23: Variation of friction coefficient for (a) pure Cu and (b) nanocomposite [49]

Zhang et al (2012) [50] have investigated situ Al<sub>3</sub>Ti/Al-5.5Cu composites fabricated by powder metallurgy and subsequent forging which were subjected to multiple pass friction stir processing with and without active cooling. The forged sample exhibited lower strength and ductility due to the presence of coarse Al<sub>3</sub>Ti clusters with a size range of 50-100 µm and coarse matrix grains. Four-pass FSP in air results increase in strength and ductility of the composites due to grain refinement and redistribution of the Al<sub>3</sub>Ti clusters. Further it was observed that the coarse Al<sub>2</sub>Cu particles dissolved and re-precipitated due to a relatively long duration of thermal exposure. Additional two pass FSP with rapid water cooling (FSP water) dissolved most of the Al<sub>2</sub>Cu into the matrix and retained the solutes in solution due to the short duration of thermal exposure. Ultrafine matrix grains with a high density of dislocations were also obtained. These microstructural changes led to significant increase in strength and a decrease in ductility in the FSP-water sample. After aging, the FSP-water sample exhibited further increased yield strength and ultimate tensile strength due to the precipitation of metastable Al<sub>2</sub>Cu phases. The ductility of the aged FSP-water sample did not decrease due to the reduction in dislocation density after aging compared with that of the FSP-water sample. Fig.24 (a) shows the differential scanning calorimetric (DSC) curves of FSP-air and FSP-water samples and Fig. 24(b) shows thermal profiles of FSP with 25 mm/ min in air and 200 mm/ min in water.



**Figure 24**: (a) DSC curves of FSP-air and FSP-water samples and (b) thermal profiles of FSP with 25 mm/ min in air and 200 mm/ min in water [50]

Sarmadi et al (2013) [51] have used friction stir processing technique to produce copper-graphite (Gr) surface composites. Five FSP tools with different pin profile were employed in order to achieve comprehensive dispersion of Gr particles. Results show that the tool with triangular pin gives a better dispersion of Gr particles. Furthermore, four copper-graphite composites containing different graphite content (1G to 4G) were prepared using triangular tool through repeating the process passes. Fig.25 (a) represents the microhardness behavior of Cu/Gr surface composites. The hardness is reduced when graphite content increases which is due to presence of softer graphite particle in comparison with the copper matrix. Wear loss of the composites was also decreased with increase in graphite content (Fig.25b). This is related to change in wear mechanism from adhesive to delamination wear and reduction of friction coefficient. Wear test indicates that the friction coefficients of composites were lower than pure annealed copper and decreased with increase in graphite content (Fig.25c). The reduction in friction coefficient is due to decrease in metal-metal contact points, originated from the presence of graphite particles as a solid lubricant.



Figure 25: (a) microhardness behavior (b) wear rate against % graphite and (c) friction coefficient behavior [51]

Sathiskumar et al (2013)[52-55] have recently fabricated boron carbide (B<sub>4</sub>C) particulate reinforced copper surface composites by friction stir processing route successfully. The effect of FSP parameters such as tool rotational speed, processing speed and groove width on microstructure and microhardness of Cu/B<sub>4</sub>C surface composites were investigated [52]. A groove was contrived on the 6mm thick copper plates and packed with B<sub>4</sub>C particles. FSP was carried out using five various tool rotational speeds, processing speeds and groove widths. Fig.26 shows that higher tool rotational speed and lower processing speed produced an excellent distribution of B<sub>4</sub>C particles and higher area of surface composite due to higher frictional heat, increased stirring and material transportation. Table 3 shows that the selected FSP parameters significantly influenced the area of surface composite and grain size of the surface composites. The B<sub>4</sub>C particles were bonded well to the copper matrix and refined the grains of copper due to the pinning effect of B4C particles.



Figure 26: (a) macrostructure of FS zone and (b) uniform distribution of B<sub>4</sub>C at 1200 rpm+40mm/min+0.7mm [52]

Table 3: Effect of process parameters on the properties of copper surface	ace
composites [52]	

FSP parameter	Value	Area of the surface composite (mm <sup>2</sup> )	Average grain size (µm)	Theoretical volume fraction (Vol. %)	Actual volume fraction (Vol. %)
Tool rotational	800	27	5	11.7	6.7
speed (rpm)*	900	30	6	11.7	5.8
	1000	33	8	11.7	5.3
	1100	37	10	11.7	4.7
	1200	43	13	11.7	4.1
Traverse speed	20	47	14.5	11.7	3.7
(mm/min)**	30	41	11	11.7	4.3
	40	33	8	11.7	5.3
	50	29	6.5	11.7	6.0
	60	24	4	11.7	7.3
Groove width	0	44	13	0.0	0.0
(mm)***	0.35	37	11.5	5.8	2.4
	0.7	33	8	11.7	5.8
	1.05	27	5.5	17.5	9.7
	1.4	24	2	23.3	14.6

Sathiskumar et al [53] have also investigated the effect of  $B_4C$  particles and its volume fraction on microstructure and sliding wear behavior of Cu/B<sub>4</sub>C surface composites via FSP route. A groove was prepared on 6 mm thick copper plates and packed with B<sub>4</sub>C particles. The dimensions of the groove was varied to result in five different volume fractions of B<sub>4</sub>C particles (0, 6, 12, 18 and 24 vol.%). A single pass FSP was done using a tool rotational speed of 1000 rpm, travel speed of 40 mm/min and an axial force of 10 KN. The volume fraction of B<sub>4</sub>C particles influenced the area of the surface composite. The macrostructure of friction stir zone containing B<sub>4</sub>C particles at different volume fractions are shown in Fig. 27. The area of the surface composite was found to be 44 mm<sup>2</sup> at 0 vol.% and 24 mm<sup>2</sup> at 24 vol.%. Particles were distributed

uniformly irrespective of the volume fraction used. The number of particles increased as well as the spacing between particles reduced when the volume fraction was increased. Fig.28 (a) shows the increase in microhardness when the volume fraction of  $B_4C$  particles is increased. The wear rate decreased (as shown in Fig.28b) when the volume fraction of  $B_4C$  particles is increased. Results indicated that the  $B_4C$  particles significantly influenced the area, dispersion, grain size, microhardness and sliding wear behavior of the Cu/B<sub>4</sub>C surface composites. When the volume fraction of  $B_4C$  was increased, the wear mode changed from microcutting to abrasive wear and wear debris was found to be finer.



Figure 27: Macrostructure of stir zone containing  $B_4C$  in vol.%: (a) 0 (b) 6 (c) 12 (d) 18 and (e) 24 [53]



Cu/B<sub>4</sub>C surface composites [53]

Sathis kumar et al (2014) [54] have evolved a novel method to fabricate copper based surface metal matrix composites using  $B_4C$ ceramic reinforcements. The  $B_4C$  powders were compacted into a groove of width 0.5 mm and depth 5 mm on a 9.5 mm thick copper plate. A single pass friction stir processing was carried out using a tool rotational speed of 1500 rpm, processing speed of 40 mm/min and axial force of 10 KN. A defect free interface between the matrix and the composite layer was achieved due to higher tool rotational speed and lower processing speed and produced an excellent distribution of  $B_4C$  particles. Fig.29 shows the optical and scanning electron micrographs revealed a homogeneous distribution of  $B_4C$  particles which were well bonded with the matrix. The hardness of the friction stir processed zone increased by 26% higher to that of the matrix material.

Sathiskumar et al (2014) [55] successfully applied FSP to prepare copper surface composites reinforced with variety of ceramic particles such as SiC, TiC,  $B_4C$ , WC and  $Al_2O_3$ . Empirical relationships are developed to predict the effect of FSP parameters on the properties of copper surface composites such as the area of the surface composite, microhardness and wear rate. The FSP parameters which influence the properties of surface composite are shown in Fig.30.



Figure 29: (a) OM image of stir zone and (b) SEM image of stir zone [54]

A central composite rotatable design consisting of four factors and five levels is used to minimize the number of experiments. The factors considered are tool rotational speed, traverse speed, groove width and type of ceramic particle. The type of ceramic particle did not make a significant variation in the area of the surface composite. Higher tool rotational speed and lower traverse speed produced a fine distribution of ceramic particles in the surface composite. The groove width and the type of ceramic particle did not influence the distribution of ceramic particles significantly. Lower tool rotational speed, higher traverse speed, maximum groove width and  $B_4C$  ceramic particle resulted in higher microhardness and lower wear rate of the surface composite. Fig.31 (a) and (b) show the effect of ceramic particles on microhardness and wear rate of fabricated surface composites respectively.



Figure 30: FSP parameters influencing the properties of surface composite [55]



Figure 31: Effect of ceramic powders on (a) microhardness and (b) wear rate of copper surface composite (rotational speed = 1000 rpm, traverse speed = 40 mm/min, groove width = 0.7 mm) [55]

Jeganathan Arulmoni and Mishra (2014) [56] have investigated the process parameters affecting the friction stir processed copper and enhancement of the mechanical properties of the Cu/Gr composite material. The results showed that the grain size of fabricated composite is reduced and also the microhardness of friction stir processed composites in stir zone (SZ) increased significantly. The results obtained also indicated that the selected FSP parameters significantly influence the area of surface composite by the distribution of material particles. Higher tool rotational speed and lower processing speed produce an excellent distribution of graphite particles and higher area of surface composite due to higher frictional heat, increased stirring and material transportation which is similar to the observations made by Sathiskumar et al [52].

Akramifard et al (2014) [57] have successfully fabricated Cu/SiC surface composites by friction stir processing route. In order to improve distribution of reinforcing SiC particles, a net of holes( diameter = depth = 2mm) were designed by drill on the surface of pure Cu sheet as shown in Fig.32 (a). It was observed that designing a net hole was very effective to achieve uniform distribution of SiC particles and prevent agglomeration of SiC particles. Microstructural observation confirmed fine and equiaxed grains in the stir zone (SZ) and fine particles had a good distribution in SZ as depicted in Fig.32 (b). In the SEM micrographs, porosities in some SiC particles were detected as microstructure defects. Fig.33(a) shows that no intermetallic compound was found in Cu/SiC composite. Microhardness measurements showed that surface hardness was two times as high as that of substrate (Fig.33b). The use of SiC particles enhanced wear resistance and increased average friction coefficient of pure Cu.



Figure 32: (a) FSP with net of holes on Cu sheet (b) SEM image of SiC particles in stir zone [57]

Sabbaghian et al (2014) [58] have fabricated a copper based surface composite using TiC reinforcement particles via friction stir processing route. Holes with the diameter and depth of 2mm were made on the surface of a pure copper specimen and filled by TiC powders. Fig.34 (a) revealed that FSP produced a fine grain microstructure with a homogeneous distribution of particles on the surface with some porosity. Reinforcing particles showed a good bonding with the base metal which increased the microhardness of composite. Maximum hardness in the stir zone was 117 Vickers, while the hardness of pure copper was 65Vickers. Fig.34 (b) shows that wear resistance of fabricated composite was increased than pure copper as a result of grain refinement and the existence of TiC particles in the FSP zone. No intermetallic compounds found in the stir zone.



Figure 33: (a) XRD analysis of Cu/SiCp composite (b) microhardness profile across the section [57]



Figure 34: (a) TiC distribution in stir zone (b) wear test results of Cu and Cu/TiC composit [58]

Bahram et al (2014) [59] have produced copper reinforced metal matrix composite using micron sized chromium (Cr) particles via friction stir processing in order to study effects of adding Cr particles to copper based matrix by FSP. Microstructures, microhardness and wear properties were studied in order to evaluate the microstructures and mechanical properties of fabricated composites. The results showed that the grain size of fabricated composite reduced at different process parameters. Good bonding between the copper matrix and Cr particles was shown in Fig.35. Lower traverse speeds lead to more uniform dispersion and smaller grain size. It is shown in Fig.36 that higher traverse speed of 80 mm/min lead to relatively higher values of microhardness in comparison with traverse speed of 40 mm/min and also wear resistance of specimen with traverse speed of 80 mm/min is higher than (10.6 x  $10^{-5}$  mm<sup>3</sup>/Nm) specimen with traverse speed of 160 mm/min (8.9 x  $10^{-5}$  mm<sup>3</sup>/Nm).



Figure 35: OM images of Cu/Cr composites at different process parameters [59]



Figure 36: Microhardness profile measured on cross-sectional planes of specimens with rotational speed of 1600 rpm and traverse speeds of 40, 80 mm/min respectively [59]

Avettand-Fenoel et al (2014) [60] have conducted a study aims to show for the first time the ability of friction stir processing in incorporating yttria ( $Y^2O^3$ ) particles into copper to produce an oxide dispersion strengthened (ODS) material. The powder was found to be distributed in the Cu matrix as confirmed at various length scales from the micrometric to the nanometric level as shown in Fig.37. The increase of the number of FSP passes leads to a more homogeneous and finer distribution of the particles as it favored the dissociation of the clusters of initial powder particles and the intergranular fracture of individual elemental particles. In spite of their very low volume fraction, these 10 nm sized fragments which present the highest density among the various size classes of particles, exert a strengthening and work hardening effect.



Figure 37: Distribution of Y<sup>2</sup>O<sup>3</sup> particles in the nugget of (a) Cu-ODS-3pass-0.2mm thick and (b) Cu-ODS-9 pass-0.2 mm thick samples (TEM) plus (c) EDX analysis of particles Y<sup>2</sup>O<sup>3</sup> [60]

Suvarnaraju and Kumar (2014) [61] have fabricated Cu/Al<sup>2</sup>O<sup>3</sup> surface composites via friction stir processing route and studied the influence of three factors, such as volume percentage of reinforcement particles (Al<sup>2</sup>O<sup>3</sup>), tool tilt angle and concave angle of shoulder. Taguchi method was used to optimize these factors for maximizing the mechanical properties of surface composites. The fabricated surface composites were examined by optical microscope and it was found that Al<sup>2</sup>O<sup>3</sup> particles are uniformly dispersed in the stir zone ( as shown in Fig.38). The tensile properties of the surface composites increased with the increase in the volume percentage of the Al<sup>2</sup>O<sup>3</sup> reinforcement particles. This is due to the addition of the reinforcement particles which increases the temperature of recrystallization by pinning the grain boundaries of the copper matrix and blocking the movement of the dislocations. The observed mechanical properties are correlated with microstructure and fracture features.



Figure 38: Uniform distribution of Al<sup>2</sup>O<sup>3</sup> particles in stir zone at different process parameters [61]

Cu/WC composites by applying the 1, 2, and 4-pass friction stir processing technique. The results indicated that increasing the pass numbers was also used to improve the dispersion of WC particles and consequently to intensify the mechanical properties of composite layers. The grain size in these composites shows the promising reduction to the 1.2  $\mu$ m in 4-pass friction stir processed one, and microhardness values reach to a considerable amount up to two times more than the pure copper. Wear rate and friction coefficient evaluation of the composites in different sliding rates demonstrated the composites resistance against weight loss and high reliability of the 4-pass friction stir processed composites in higher wear distances compared to that of fabricated by 1-pass. Moreover, the thermal expansions of the composites were examined up to 500 °C which is indicative of the composites stability in higher temperatures.

Jahangir Khosravi et al (2014) [62] have fabricated good quality

Rajashekhara Shabadi et al (2015) [63] have reported the effect of microstructural change brought by the FSP and presence of yttria particles on the thermal conductivity (TC) and coefficient of thermal expansion (CTE) of copper material. Thermal properties of samples with or without powder were investigated for the temperature ranges between 40° and 240°C as a function of the number of FSP passes as depicted in Fig.39. The reduction of CTE in the samples without powder is attributed to the decrease of the grain size during FSP (Fig.39b). CTE values are rather close at the lower temperatures i.e., 40°-80°C, but, as the temperature rises to higher values there is a clear distinction between pure copper and samples with a modified microstructure. An impressive 27% reduction in the CTE was observed for a sample with uniformly distributed yttria particles in copper (i.e.; 9 passes with yttria) in comparison to pure copper. Thermal conductivity at 240°C are slightly higher for the copper modified 3 passes and 9 passes with yttria powder when compared to pure copper (Fig.39a). It was found that distribution of yttria particles in the copper matrix play a significant role in stabilizing the microstructure and hence reduce the CTE with no loss in thermal conducting ability of copper. However, conductivity values were still on the higher side for the samples with the yttria and the major influence being the uniform distribution of the particles in the copper matrix thereby providing a stable microstructure. Fig. 40(a) presents yttria particles pinning down the grain boundary in a 9 pass+yttria sample after heat treatment. Yttria particles have been incorporated without much defects in the copper matrix as can be observed in Fig. 40(b).







**Figure 40**: Role of yttria particles in Cu+ yttria, 9 pass FSPed sample (a) grain boundary pinning, and (b) well integrated particle in the matrix [63]

Jalal Jafari et al (2015) [64] have investigated the effect of incorporation of carbon nanotubes(CNT) into pure copper through friction stir processing. FSP has been performed using one and three passes. The changes in peak temperature of one-pass friction stir processing performed by tools with different shoulder diameters have been recorded. It was found that for the tool with smaller shoulder, the process temperature peak has been significantly reduced. Remarkable reduction in grain size of Cu/carbon-nanotube nanocomposites was observed as compared with the pure copper. It was observed that higher extent of degradation of carbon nanotubes upon increasing the friction stir processing passes. On the other hand, microhardness and wear results showed that as the carbon nanotubes are introduced to the pure copper, hardness of composites processed via one- and threefriction stir processing passes showed enhancements of 65 and 105 %, respectively, and the weight losses were also decreased 31 and 68%, respectively. It was also observed that friction coefficient of one-pass processed composite is lower than that of the pure copper due to the presence of carbon nanotube clusters. Whereas, the friction coefficient of three-pass processed composite was increased regarding to the pure copper.

#### Conclusions

Friction stir processing (FSP) is a solid state surface modification technique since the working temperature is lower than the melting temperature of metals and alloys. The primary research on friction stir processing of copper and its alloys are focused and studied. This technology has found applications in modifying the microstructure of particles reinforced metal matrix composite materials also. The recent state of understanding and developments in particulates reinforced copper based metal matrix surface composite has been addressed. After detailed study of various works, the following conclusions have been drawn:

- FSP is an efficient technique for grain refinement in metals and alloys. It is a successful microstructural modification technique in copper to alter the surface properties of copper in a localized area.
- FSP is a low energy consumption route to produce surface level copper based metal matrix composites.
- Fracture mechanism of FSPed pure copper significantly depends on grain size and cavity formation during process. Grain size is controlled by heat input and cavity formation decreases with higher plastic deformation, produced by tool.
- The selected FSP parameters and particulates deposition technique significantly influence the area of surface composite, distribution of material particles and micro hardness of the surface composites.
- Higher tool rotational speed and lower processing speed produce an excellent distribution of particles and higher area of surface composite due to higher frictional heat, increased stirring and material transportation.
- The microhardness values of the surface composites increased with increasing reinforcement particles content.
- Addition of SiC particles led to enhancement of electrical resistivity of pure copper.
- Reinforcement particles increased the resistance of composites against wear.
- The average friction coefficients of composites fabricated by multi-pass FSP were noticeably reduced compared to the pure copper.
- The hardness values and the wear behaviour of the surface composites strongly depend on reinforcement particles, process parameters, heat generation, and particles deposition technique and tool geometry.

There are some major challenges for improving the FSP technology.

- Lack of predictive models in FSP than FSW.
- Lack of development in modeling and simulation of FSP on copper.
- Tool wear in processing reinforced composite materials.
- How to improve the fatigue property of FSP composite materials.

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