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Effects of heat input and particulate deposition on Cu/SiCp surface composite processed by friction stir processing

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Friction stir processing is a novel, green and low energy consumption route used to prepare surface level composites. In this study, a single pass friction stir processing was utilized to prepare copper based surface composite using silicon carbide particles at a constant rotational speed of 1000 rpm and processing speed of 50 mm/min. A cluster of blind holes 2 mm in diameter and 4 mm depth is used for particulate deposition during composite fabrication. The effects of heat input and particulate deposition technique on microstructure and mechanical properties of fabricated surface composite are studied. Temperature distributions are measured using K-type thermocouples. Optical and scanning electron micrographs reveal a uniform dispersion of SiC particles without any agglomeration problem. X-ray diffraction study shows that no intermetallic compound is formed after processing. The microhardness of the surface composite is remarkably enhanced than that of base metal.

Keywords–Surface composite, Temperature, Particulate deposition, Microstructure, Microhardness

Copper (Cu) and copper alloys are the widely used industrial and functional metal for various high end applications. This owes to its high thermal and electrical conductivity, plasticity, softness, formability and outstanding corrosion and oxidation resistance. But the major limitations of copper and its alloys are of low strength and poor wear resistance[1]. In many engineering applications, the surface properties decide the life of the components than its bulk properties. Hence, it is of interest to alter the surface of the component by reinforcing with ceramic particles while the inner matrix retains its bulk properties. The modified surface layer reinforced with ceramic particles is normally called as surface composite[2]. Silicon carbide (SiC) particles are of great technological importance because of their application as reinforcement for metal matrix composites and structural ceramics with exceptional thermal shock resistance qualities[3]. In recent years, copper based surface composites are gaining wide spread importance in several applications due to their good mechanical, thermal and tribological properties. An extensive study on copper based surface composites is therefore needed without much loss in bulk properties of the matrix material[4].

Though several techniques are available to prepare surface composites, friction stir processing (FSP) is a simple, green and low energy consumption route based on the principles of friction stir welding (FSW) to prepare surface composites. Recently, FSP has been successfully attempted to prepare surface composites with good results[5-9]. In FSP, the heat generated due to friction between tool and work-piece, softens the metal matrix and the intense stirring action of the tool, aids in distribution of the reinforcement particles within the plasticized metal matrix zone. Mishra *et al*.[10] and Adem Kurt *et al*.[11] developed aluminium based surface composite successfully via FSP by applying SiC powder mixed with small amount of methanol to the surface of the parent metal. The most common method of particulate deposition technique followed to produce surface composite using FSP is to make a groove of required depth, pack it with ceramic particles and plunge the tool and traverse it along the groove. FSP route has been explored effectively by a number of investigators to produce surface metal matrix composite on aluminium, magnesium, copper and steel.

Barmouz and Givi[12] investigated that electrical resistivity of Cu/SiCp composite was increased due to the presence of hard SiC particles produced by FSP than copper. Barmouz *et al.*[13,14] successfully used the FSP route to produce Cu/SiCp surface composite and observed that the distribution of SiC particles were significantly influenced by the processing speed and

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tool pin profile. Higher processing speed led to poor distribution of SiC particles and vice versa[13]. A straight cylindrical tool pin profile produced a uniform distribution of SiC particles than other pin profiles[14]. Sathishkumar *et al*.[15,16] developed Cu/B_4Cp surface composites using FSP and found that the distribution of B_4C particles and the area of the surface composite were strongly influenced by process parameters[15]. They observed that higher rotational speed at lower traverse speed produced fine distribution of ceramic particles in the surface composites[16]. Akramifard *et al*.[17] investigated the effectiveness of designing net holes instead of conventional groove method in successful fabrication of Cu/SiCp surface composite with enhanced mechanical properties. Sabbaghian *et al*.[18] developed a Cu/TiCp surface composite using a set of fine holes for TiCp on the Cu sheet and investigated the effect of TiC particles on the mechanical properties and the microstructure.

However, still there is lack of information regarding particles dispersion mechanism, agglomeration and deeper/wider region of reinforcement particles in surface composites by FSP[17,19]. The agglomeration of reinforcement particles during FSP deteriorates microstructure and mechanical properties of surface composites[12,20]. The successful research on the effect of heat input and design of particulate deposition on the microstructure and mechanical properties of copper based surface composites through FSP are rarely reported till now. In this work, FSP was employed to prepare a copper based surface composite by means of micro sized SiC particles using a cluster of blind holes as particulate deposition technique on the surface of Cu plates. The effects of heat input and particulate deposition on the microstructure, hardness, temperature and the particles distribution characteristics of the developed surface composite were investigated.

Materials and Methods

In this study, commercial pure copper plates of 120 mm × 50 mm × 6 mm size were used as work-piece. The chemical compositions of copper were 0.005Mg-0.005S-0.001Ti – rest Cu (in wt%). Commercially available SiC powders with an average particle size of 12 µm were used as reinforcement particles. The OM of commercial copper plate and the SEM image of the anisotropic shape of SiC particles used are shown in Fig. 1(a) and 1(b), respectively.

A single pass FSP experiments were carried out in a conventional vertical milling machine (3 HP and 2000 rpm ratings). A high carbon high chromium (HCHCr) tool with a flat shoulder of 18 mm diameter (D) and a straight threaded cylindrical pin of 6 mm diameter (d), 5.6 mm long (L) was made followed by hardening and tempering process to increase the hardness to 55-58 HRC. Figure 2(a) shows the FSP tool with its geometry.

For producing wider surface level composite layer, a net of blind holes of diameter 2 mm and depth 4 mm was designed on the surface of copper plate as shown in Fig. 2(b) and the SiC particles were compacted into them before processing. The FSP steps to produce surface level composite were schematically depicted in Fig. 3(a)-(c). FSP was performed using a constant rotational speed of 1000 rpm, processing speed of

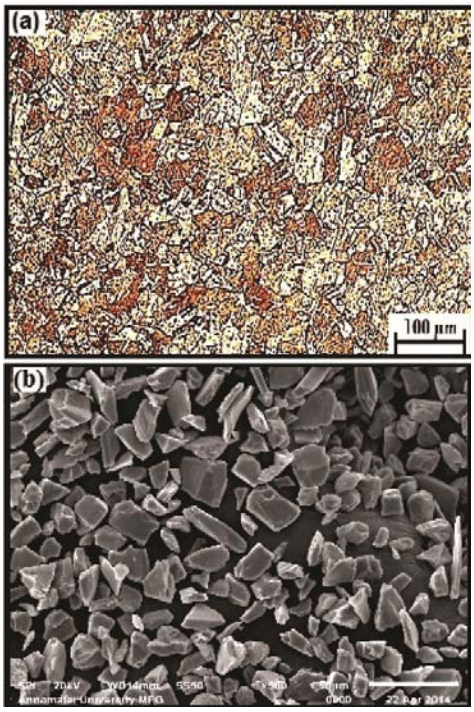

Fig. 1—Micrograph of as-received (a) pure copper and (b) silicon carbide particles

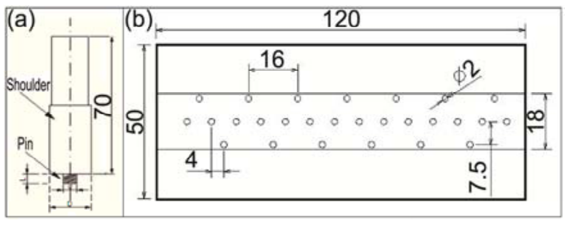

Fig. 2—(a) Tool geometry and (b) schematic representation of design of blind holes on Cu sheet (depth 4 mm)

50 mm/min and tool tilt angle of 2.5°. Two K-type thermocouples (1.6 mm diameter) were used to measure the temperature distribution below the FSP tool. Thermocouples were inserted in blind holes drilled from the bottom of the copper sheet near the perimeter of FSP tool shoulder and orthogonal to the path of the tool. Figure 3(d) schematically shows the actual locations of thermocouples (TC1 and TC2) inserted at the advancing side (AS) and the retreating side (RS) prior to FSP runs. A four channel data logger was used to record the temperature readings. Microstructural observations were carried out at the processed samples by optical microscopy (OM) and scanning electron microscopy (SEM) as per ASTM standards. X-ray diffractometer was used to examine the phase composition for both SiC particles and the base metal. Microhardness profile of the stir zone was measured across the cross-section of the sample with a constant load of 0.25 kg_f with a dwelling period of 15 s.

Results and Discussion

Temperature distribution

Normally higher rotational speed and lower transverse speed produce an excellent distribution of ceramics particles in metal matrix composites[16]. The selected process parameters (1000 rpm, 50 mm/min) are sufficient to produce an adequate amount of heat generation to get defect free FSP region. Figure 4 shows the plot of temperature versus processing time in both advancing and retreating side of FSP. The heat generation is due to friction between tool and the work-piece. The following important information were obtained from these curves: (i) The temperatures on the advancing side were slightly higher than those on the retreating side of the processed zone; and (ii) the peak temperature at the advancing side reached during FSP was 830°C which is 0.77 times the melting point of pure copper (~1080°C). It indicates that no melting of work-piece has taken place during the process and that the FSP is a solid state process for surface composite fabrication. The peak temperature, the heating and cooling rates are very important because it affects the grain growth during the process. These results are used to interpret the microstructure characterization of the process along with the refined SiC particles.

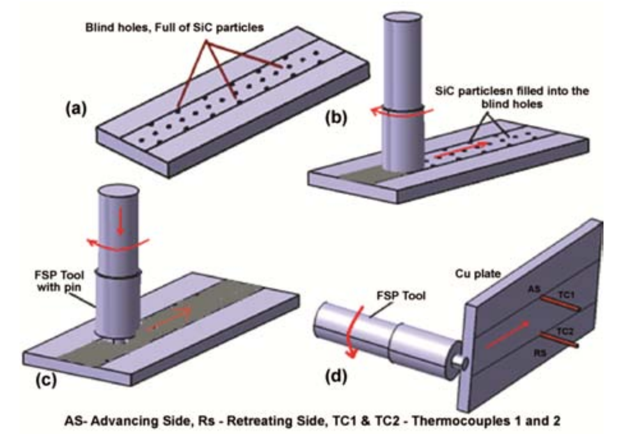

Fig. 3—(a)-(c) Schematic representations of FSP steps and (d) thermocouple locations

Microstructural analysis

The microstructural observations of Cu/SiCp composite are shown in Fig. 5(a-d). The different FSP regions like stir zone (SZ), thermo-mechanically affected zone (TMAZ) and the unprocessed base metal (BM) were observed but due to high thermal conductivity of copper, the heat affected zone (HAZ) was not observed. The microstructure of base metal in Fig. 1(a) has both equilateral and twinned grains of beta phase. The presence of twinning is due to cold working/drawing of base metal which has nearly equiaxed grains with a size of approximately 40 μm.

Figure 5(a) shows the interface between base metal and TMAZ zone. The image shows the plastic flow of base metal due to thermal effect. Owing to rapid movement of the tool, dynamic recrystallization has not taken place due to insufficient deformation strain but it was seen that the grains have fragmented. Mishra and Ma[5] reported the similar trend that recrystallization did not occur due to insufficient deformation strain. TMAZ is the region containing elongated grains in the direction of material flow next to the stir zone on both the sides of a processed area due to plastic deformation and limited to a high density of sub-boundaries. It was found in the shoulder zone and bottom of stir zone, which bcannot

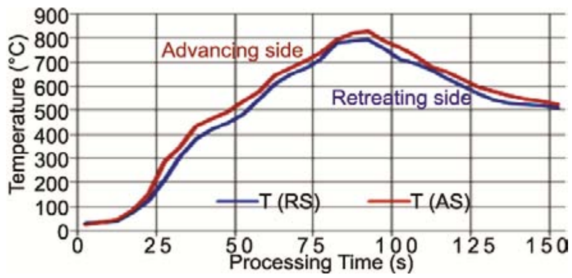

Fig. 4—Temperature profile in AS and RS during Cu/SiCp surface composite fabrication

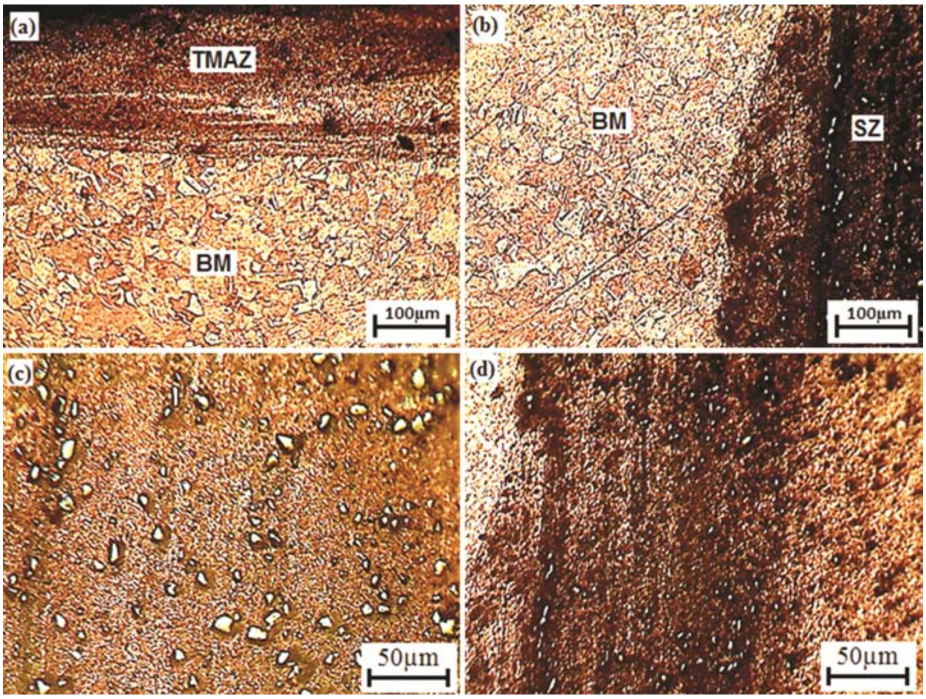

Fig. 5—Microstructure of interface between (a) BM and TMAZ, (b) BM and SZ, (c) uniform distribution of SiCp in stir zone and (d) reduction in size of SiCp after FSP

be observed in stir zone side regions[18]. The same was observed by comparing Fig. 5(a) with 5(b). This trend could be related to heat generation and its transfer during friction stir processing as follows:

(i) The bottom area of stir zone has more heat transfer than side areas of stir zone. This is due to the physical contact of bottom region of stir zone with backing steel plate, consequently, the side regions of stir zone has a good possibility to be coarse-grained[21].

(ii) The heat generation and plastic deformation in the bottom area of stir zone are much higher than side region of stir zone. This is because of (a) the tool pin in the side region of stir zone has a line contact with the work-piece and (b) the tool pin in bottom has a planer contact with the work-piece[22].

In general, the plastic deformation does not occur in heat affected zone (HAZ). As cited before, the HAZ was not observed in this study. Hence, the distribution of SiC particles in this region was deficient[23]. However, the occurrence of HAZ may be expected based on process parameters, type of reinforcement particles and its size[18,24]. Figure 5(b) distinguishes the interface between base metal and stir zone where the stir zone region reveals the alignment of SiC particles along the boundary. A parallel band-like distribution of SiC particles is observed. The Cu/SiCp surface composite was well bonded to the copper substrate[24]. It is more obvious from Fig. 5(b), that there were no discontinuities or flaws along the boundary.

Characterization of SiCp distribution in stir zone of Cu/SiCp surface composite

Apart from frictional heat generation, when the tool rotational speed is high (1000 rpm), the amount of stirring and material flow are increased and the corresponding FSP zone is subjected to high plastic strain. These high plastic strains enhanced the stirring action and particles deposition technique (set of blind holes) has helped to avoid the agglomerations of SiC particles in the copper matrix. The processing speed (50 mm/min) determines the stirring of the FSP tool and affects the transportation of materials from advancing side to retreating side. The severe plastic deformation and increase in heat generation during FSP produces fine grain structure[24]. The stir zone could be characterized by its microstructure with fine grains and dispersed SiC particles. The uniform distribution of SiC particles in stir zone of copper matrix as seen in Fig. 5(c) was attributed to intense stirring (1000 rpm) and sufficient material flow (50 mm/min) which has reduced the possibility of formation of agglomeration[16,20]. Normally, the agglomeration of particles in surface composite by FSP is reduced when the rotational speed increases or the processing speed decreases[25]. From Fig. 5(d), it was observed that the size of SiC particles was reduced to approximately 5 µm to 7 µm which was much less than as-received SiCp size (~12 µm). It is evident that a single pass FSP with a cluster of blind holes as particulate deposition technique was adequate to break the SiC particles and enhance the distribution[6].

The SEM micrographs as presented in Fig. 6 shows the agglomeration-free fine distribution of SiC particles in Cu/SiCp composite. Figure 6(a) shows the region of uniform distribution of SiC particles in stir zone as the rotating FSP tool gives adequate heat generation and necessary circumferential force to distribute SiC particles to flow in a wider region[26]. Figure 6(b) shows higher magnification of Fig. 6(a). However, the existence of porosities in some SiC particles as in Fig. 6(a) and 6(b) in stir zone was observed. This is due to the variation of physical properties between copper and SiC particles. But in Fig. 6(c), it was observed that a higher magnification of a SiC particle which had good interfacial bonding with copper matrix at the same process parameter conditions. The presence of pores, limited the recrystallization process by pinning the dislocations and boundaries[17,18]. Many researchers observed similar defects[17,23] and reported that by increasing the number of FSP passes and changing the direction of tool rotation in between the passes, the occurrence of porosities can be reduced[13,24].

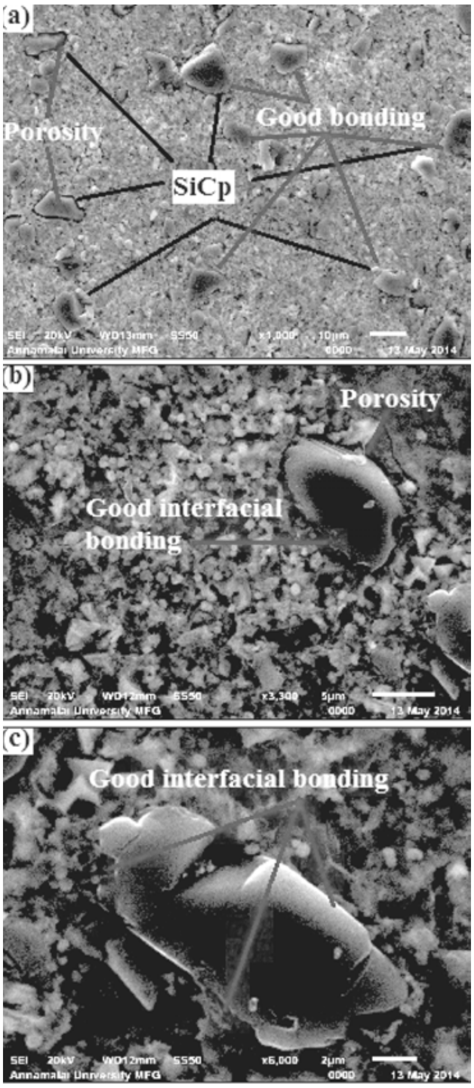

Fig. 6—SEM images of Cu/SiCp composite (a) agglomeration-free composite, (b) higher magnification of (a) with porosity and (c) good interfacial bonding of SiCp in Cu substrate

XRD analysis

The XRD patterns of the selected SiC powder and the prepared Cu/SiCp surface composite are presented in Fig. 7. The diffraction peaks of SiC particles and its

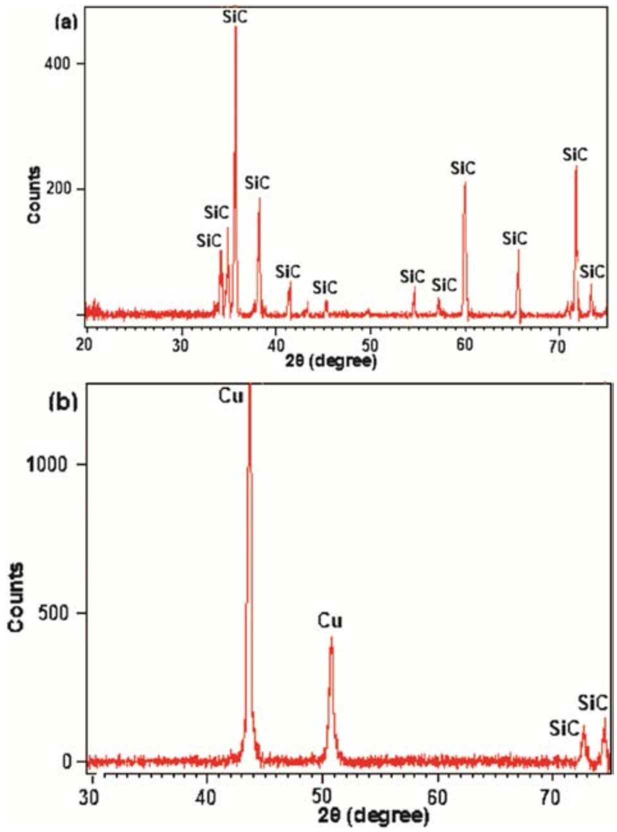

Fig. 7—XRD analysis of (a) SiC micro powder (b) Cu/SiCp surface composite

presence in the composite were seen in Fig. 7(a) and 7(b), respectively. The peaks of SiC particles in Cu/SiCp composite seem weak because the volume fraction of SiC particles is smaller than that of copper[27]. It is also observed from Fig. 7(b) that no evidence of new phases (intermetallic compounds) which were attributed to an adequate heat generation during the FSP and also some SiC particles reflections were disappeared, due to the good dispersion and particle size reduction of SiC. Akramifard *et a*l.[17] and Sabbaghian *et al.*[18] have not reported any intermetallic compound in their Cu/SiCp and Cu/TiCp composite respectively, produced via FSP similarly.

Microhardness

Figure 8 reveals the microhardness of the base metal and the distribution of microhardness in the cross-section of Cu/SiCp surface composite. The average microhardness of commercial pure copper was 82 Hv. The microhardness profile shows increase in hardness at the stir zone of composite than that of copper matrix. In general, during FSP, the hardness in the side regions of stir zone has been reduced owing to annealing-induced grain growth. While, for the samples prepared at lower processing speeds (50 mm/min), SiC particles were split well and as a result, an intense pinning effect of hard SiC particles occurs in stir zone, leading to remarkable enhancement of microhardness values. Increasing hardness in stir zone region during FSP with the addition of SiC particles was due to: (i) Hard phase dispersion of SiC particles (Orowan strengthening mechanism); (ii) Grain refinement in stir zone (Hall-Petch relationship); (iii) Quench hardening (due to the variation in thermal reduction between SiC particles and pure copper matrix); and (iv) Work hardening (caused by the strain misfit between the elastic SiC particles and the plastic pure copper).

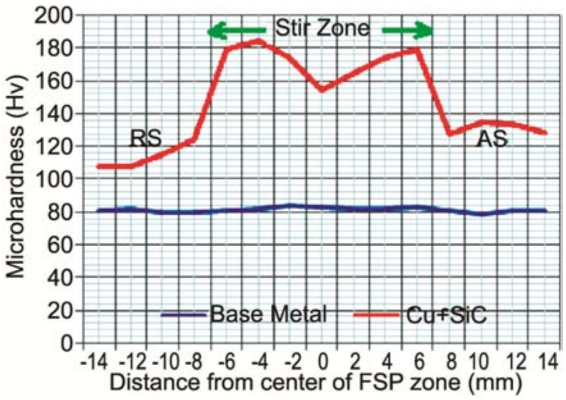

Fig. 8—Microhardness distribution in the base metal and Cu/SiCp composite

Figure 8 shows that in composite specimen, hardness is not equal in both sides of process line. The material flows in a complex fashion and the material flow pattern varies in different sections of stirred zone. This will cause variations of strain rate, strain and temperature in stirred zone and this phenomenon will intensify microstructural features in different locations of stirred zone[28,29]. Since, the stirring action during FSP takes place away from the center[28,29], the center may experience less deformation compared to advancing and retreating sides. This may be responsible for the hardness drop at the center. The peak hardness is observed away from the center at the advancing and retreating sides. A similar kind of hardness profile showing peak hardness away from the center of the stir zone was reported earlier by Yadav and Bauri[28] and Thangarasu *et al.*[29]. The microhardness value of advancing side is slightly higher than retreating side. This is due to a better distribution of SiC particles in advancing side than retreating side[21]. The enhancement of microhardness in surface composite by FSP was also reported in similar earlier works[14-21,24]. The average hardness of FSP zone of Cu/SiCp was about 165 Hv which is 100% higher than that of the pure copper.

Conclusions

In the present work, the effect of heat input and a cluster of blind holes as particulate deposition technique on microstructure and hardness were investigated in Cu/SiCp surface composite produced by FSP. The following conclusions can be drawn from the results obtained:

(i) Cu/SiCp surface composite with widely dispersed SiC particles can be fabricated using energy efficient FSP technique.
(ii) Designing a cluster of blind holes was very effective to achieve agglomeration- free surface composite where the SiC particles were well-bonded with copper matrix.
(iii) The existence of porosities in some SiC particles in stir zone was observed in microstructure and it can be reduced by increasing FSP pass and changing the tool rotational direction between FSP pass.
(iv) The hardness of the FSP zone increased by 100% higher than that of the pure copper was due to the introduction of SiC particles reinforcement.

(v) No intermetallic compounds were observed in surface composite because of the adequate heat input condition during FSP.

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