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## A novel approach for fabrication of Cu-Al<sub>2</sub>O<sub>3</sub> surface composites by Friction Stir Processing

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### Abstract

In this study Copper- Al<sub>2</sub>O<sub>3</sub> surface composites are produced with different volume percentages using micron sized particles via friction stir processing in order to enhance surface mechanical properties. Tool rotational speed and traverse speeds were fixed at 900 rpm and 40 mm/min respectively. The fabricated surface composites have been examined by optical microscope for dispersion of reinforcement particles and found that Al<sub>2</sub>O<sub>3</sub> particles are uniformly dispersed in the stir zone. It is also observed that the microhardness at the higher volume percentage increases due to presence of hard Al<sub>2</sub>O<sub>3</sub> particles. The tensile properties of the surface composites increased with the increase in the volume percentage of the Al<sub>2</sub>O<sub>3</sub> particles. This is due to the addition of the reinforcement particles which increases the temperature of recrystallization by pinning of grain boundaries of the copper matrix and blocking the movement of the dislocations. The observed mechanical properties have been correlated with microstructure and fracture features.

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*Keywords:* Friction Stir Processing, Al<sub>2</sub>O<sub>3</sub> reinforcement particles, mechanical properties, microstructure and fracture features.

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**Nomenclature**

|                                  |                                   |
|----------------------------------|-----------------------------------|
| FSW                              | Friction stir welding             |
| FSP                              | Friction stir processing          |
| Al <sub>2</sub> O <sub>3</sub> p | Alumina particles                 |
| SiCp                             | Silicon carbide particles         |
| CASCs                            | Copper-Alumina surface composites |
| BM                               | Base metal                        |
| SEM                              | Scanning Electron Microscopy      |
| SZ                               | Stir zone                         |
| DRX                              | Dynamic recrystallization         |

**1. Introduction**

Friction stir welding (FSW) is a solid state joining technique invented by The Welding Institute (TWI), UK in 1991 and was first applied to aluminum alloys (Thomas W.M et al., 1991 and Mishra R.S & Ma.Z.Y., 2005). In FSW, a non consumable rotating tool with a pin and shoulder is plunged into the material and traversed along the joint line of the plates to be joined (Mishra R.S & .Ma. Z.Y., 2005). FSP based on the principle of friction stir welding, is a solid state technique used for material processing in order to modify the microstructure, mechanical properties and used for fabrication of surface composites (Chang C.I et al., 2007). Friction Stir Processing (FSP) is a new processing technique and it was developed by Mishra et al. (Mishra R.S., et al., 2003) in which, a rotating tool with pin was inserted into a single piece of material for microstructural modification and traversed along the desired line to cover the region underneath the shoulder. Friction between the tool and work piece results in localized heating that softens and plasticizes the work piece. A volume of processed material is produced by the movement of material from the front of the pin to the rear of the pin. During this process, the material undergoes intense plastic deformation and thus results in the grain refinement. FSP has the following advantages: It is a solid state process and prevents problems associated with liquid metallurgy. Good mixing and refining of constituent phases takes place in the material due to severe plastic deformation and the process does not produce any deleterious gas, material distortion. Pure copper is used in many industrial applications due to its high thermal and electrical conductivity, plasticity, softness and formability. Although pure copper is mostly used in electrical applications such as high-performance electric switches and sliding contact materials (Kaczmar J.W., et al., 2000). However, due to its high softness besides low hardness and wear resistance of pure copper limits its structural applications. Ceramics, such as alumina (Al<sub>2</sub>O<sub>3</sub>) are good choice for the improvement of high hardness and wear resistance of pure copper. Since, alumina is not insoluble in the copper matrix, its original particle size nor spacing is neither altered even at high temperature. This characteristic of alumina makes the strength and conductivity of the resulting composite stable even at elevated temperature (Shojiro. O., 1994). Cu-Al<sub>2</sub>O<sub>3</sub> composites (CASCs) are widely used in welding as electrodes, heat exchangers, rotating source neutron targets and rocket nozzles (Groza, J.R., & Gibeling.J.C., 1993). Sun Y.F.,& Fujii (2011) have fabricated Cu-SiC surface composite via FSP in one and two passes, the joints processed after two passes resulted in a particle-rich and free region formed in the stir zone (SZ), the particle rich region consists of refined grain structure due to the SiC particles (SiCp) stimulated nucleation in the dynamic recrystallization (DRX) of copper during the FSP process. Barmouz.M., et al., (2011) fabricated the Cu/SiC surface composites via FSP with 5µm and 30 nm SiCp by varying the volume fraction. From the result, it is observed that the FSP of pure copper with SiCp, the tensile properties of the surface composites were slightly lower than the base metal, where as for FSP of pure copper without powder reinforcement there was an increment in percentage of elongation and decrease in tensile strength. Morisada. Y., et al.,(2006) fabricated AZ31-SiC surface composite via FSP. They found that, the microhardness of the SZ with the SiCp increases. Mahmoud. E.R.I., et al., (2009) produced AA1050 based MMC reinforced with SiCp using FSP. The study was focused on the effect of tool

geometry and processing parameters such as tool rotation and travel speeds. Modified surface composites were obtained, however, there were significant channel like defects that deteriorate the surfaces and preclude its industrial use. From the reported literature, it is observed that the influence of  $\text{Al}_2\text{O}_3$  reinforced particles on mechanical properties of CASCs via FSP was not studied.

Hence the objective of present investigation is to achieve the surface composite via FSP and also to study the influence of  $\text{Al}_2\text{O}_3$  reinforced particles on microstructure and mechanical properties of CASCs fabricated by single pass FSP.

## 2. Experimental procedure

In this study a pure copper plate with dimensions of 200mm x 100 mm x 3 mm was used. In order to produce surface composites  $20\ \mu\text{m}$   $\text{Al}_2\text{O}_3$  particles ( $\text{Al}_2\text{O}_3\text{p}$ ) were contrived in a square groove. The square groove was made in the advancing side which is 1 mm far away from the centre line of the tool rotation on the copper plate (Devaraju.A., et al., 2013). The groove size was varied along with the volume percentage (4, 8 and 12%). The  $\text{Al}_2\text{O}_3\text{p}$  were compressed into the groove and the upper surface of the groove was closed with a FSP tool without pin to prevent  $\text{Al}_2\text{O}_3\text{p}$  to escape from the groove. In the next stage the tool is plunged with the pin into the plate to stir the material along with the reinforcement (SZ) to produce the surface composites. The schematic sketch of FSP to produce surface composite is shown in Fig.1. The optimum rotational and traverse speeds, resulting in an optimum level of mechanical properties were taken as 900 rpm and 40 mm/min respectively (Kumar.A.,& Suvarna Raju.L.,2012). Mechanical properties of the base metal (BM) are presented in Table 1. H-13 tool steel with shoulder diameter, square pin diameter and length of the pin are considered as 24, 8 and 2 mm respectively. The specimens were clamped on to backing plate and fixed by the bolts. Single pass FSP was used to fabricate the CASCs which are shown in Fig. 2.

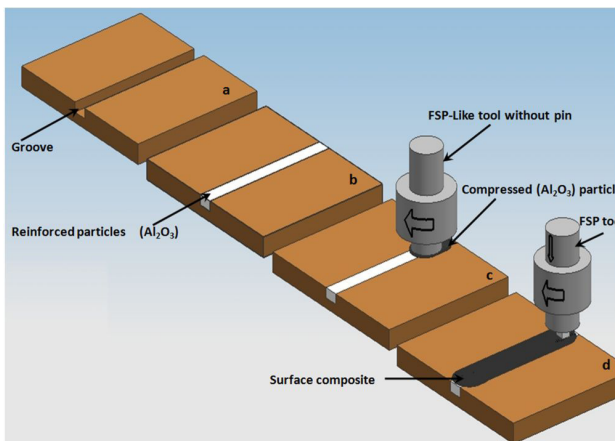


Fig.1. Schematic diagram of FSP

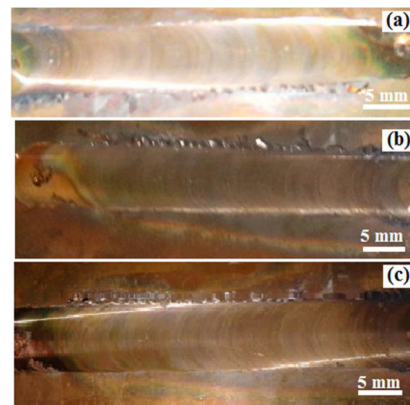


Fig.2. Surface morphologies of the CASCs made with different volume percentages (a) 4% (b) 8% (c) 12%.

After FSP, microstructural observations were carried out at the cross section of SZ of the surface composites normal to the FSP direction, mechanically polished and etched with 100 ml distilled water, 15ml HCl and 2.5g ferric chloride. Microstructure changes were observed by optical microscope in the SZ. The tensile specimens were prepared by Wire cut Electrical Discharge Machine to the required dimensions which is normal to the FSP direction.

The tensile test was conducted with the help of a computer controlled universal testing machine at a cross head speed of 0.5mm/min. Similarly the impact specimens were taken in transverse to the processing direction. The charpy ‘V’ notch impact test was carried out using pendulum type impact testing machine at room temperature. The schematic sketch of both tensile and impact specimens were shown in Fig.3 The fractured surfaces of the tensile and impact tested specimens were analyzed using a scanning electron microscopy (SEM- Hitachi, SU 6600) to study the fracture morphology and establish the nature of the fracture.

Table 1. Mechanical properties of base metal

| Material    | UTS(MPa) | YS (MPa) | % EL | IT (J) | Microhardness (HV) |
|-------------|----------|----------|------|--------|--------------------|
| Pure copper | 260      | 231      | 31   | 18     | 110                |

Microhardness test was carried out by using Vickers digital microhardness tester (Model: Autograph, Make: Shimadzu) with a 15 g load for 15 s duration at the cross section (SZ) of surface composites normal to the FSP direction.

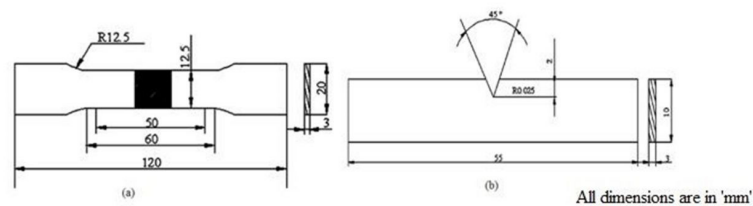


Fig. 3. Schematic diagram of (a)Tensile and (b)Impact specimens

### 3. Results and Discussion

#### 3.1. Microstructure

The specimens for metallographic examination were sectioned to the required size from the SZ which is transverse to the processing zone. The optical micrographs of the defect free FSPed specimens produced by one pass FSP and BM are shown in Fig. 4. It is observed that at higher volume percentage (12%) the reinforced particles are dispersed uniformly in the processed zone which is shown in Fig. 5. This is due to the position of the groove exactly tangential to the tool pin. It is also observed that, severe plastic deformation and frictional heating in the SZ during FSP resulted in generation of a recrystallized equiaxed microstructure which is due to the occurrence of dynamic recrystallization (DRX) (shafiei-Zarghani. A. et al., 2009). It can be seen that in the FSPed specimens with  $Al_2O_3$ p, the pinning effect of  $Al_2O_3$ p reduces the grain size in the processed zone. This may be due to the uniform dispersion of  $Al_2O_3$ p in the processed zone which enhances the pinning effect of  $Al_2O_3$  particles in the SZ. The SEM micrographs of  $Al_2O_3$ p after one pass FSP are shown in Fig.6. It is found that the size of the reinforced particles was reduced in size i.e. approximately 1.48  $\mu m$  than the as received particle. This may be due to the tool which provides

a shear force and breaking of reinforcement particles in the processed zone and causes intense plastic deformation (Mishra R.S. et al., 2003). It is considered that a fine and equiaxed grain structure could be obtained by the FSP with the uniform dispersion of smaller  $\text{Al}_2\text{O}_3$ p.

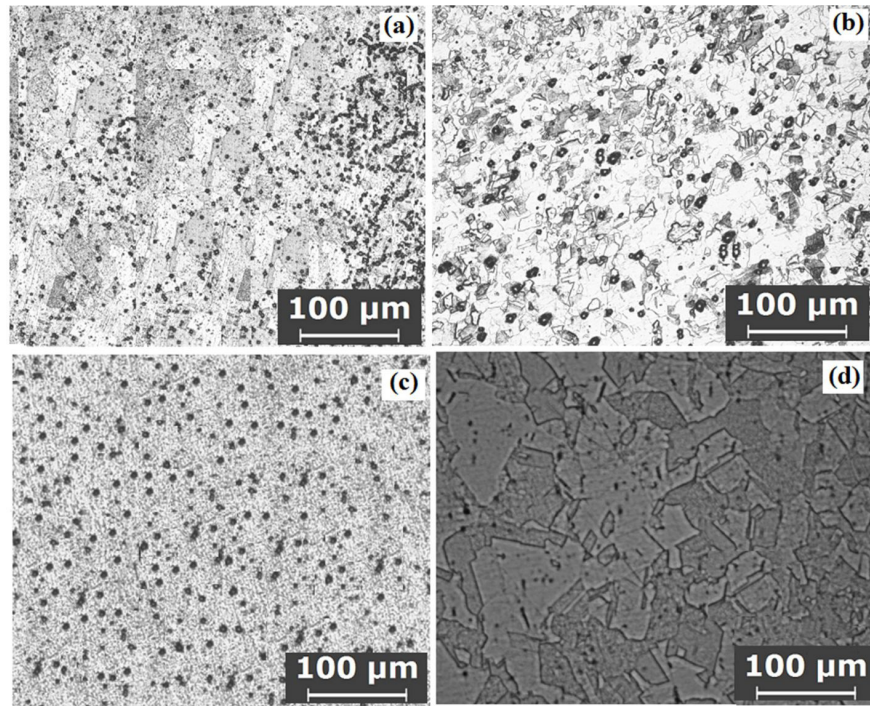


Fig. 4. Optical microstructures of CASCs made with different volume percentages  
(a) 4% (b) 8% (c) 12% (d) base metal

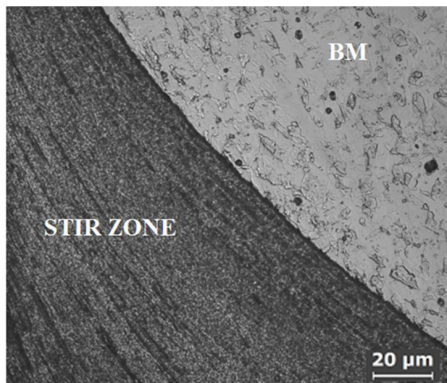


Fig. 5. Micrographs of stir zone (dispersed  $\text{Al}_2\text{O}_3$  particles) of CASCs fabricated by FSP at higher volume percentage (12%).

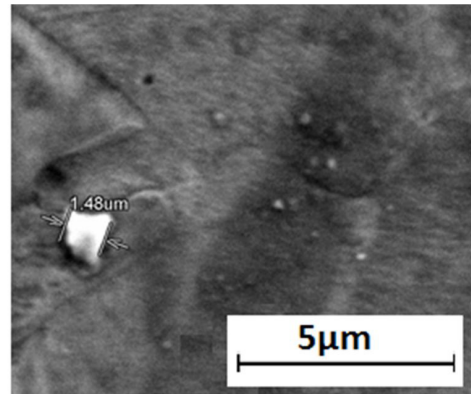


Fig. 6 SEM micrograph of  $\text{Al}_2\text{O}_3$  reinforced particles after one pass FSP

### 3.2. Mechanical properties

Microhardness tests and tensile tests were conducted to evaluate the hardness distributions and tensile strength of the surface composites. At each condition three specimens were tested and average of the results of three specimens is presented. The processing parameters and tool dimensions used to fabricate via FSP are presented in Table 2. Microhardness survey was measured on the FSPed samples across the transverse cross-section of the surface CASCs and are presented in Fig. 7. The average hardness of matrix material is 110 HV. The highest hardness value of 137 HV have been observed in the SZ for the sample processed at the highest volume percentage. It is observed that hardness increases with increased volume of  $Al_2O_3$ p in the SZ, it is due to the presence and pinning effect of hard  $Al_2O_3$ p ( Devaraju. A & Kumar.A. 2011). The presence of  $Al_2O_3$ p is considered for more effective formation of fine grain structure due to the continuous DRX.

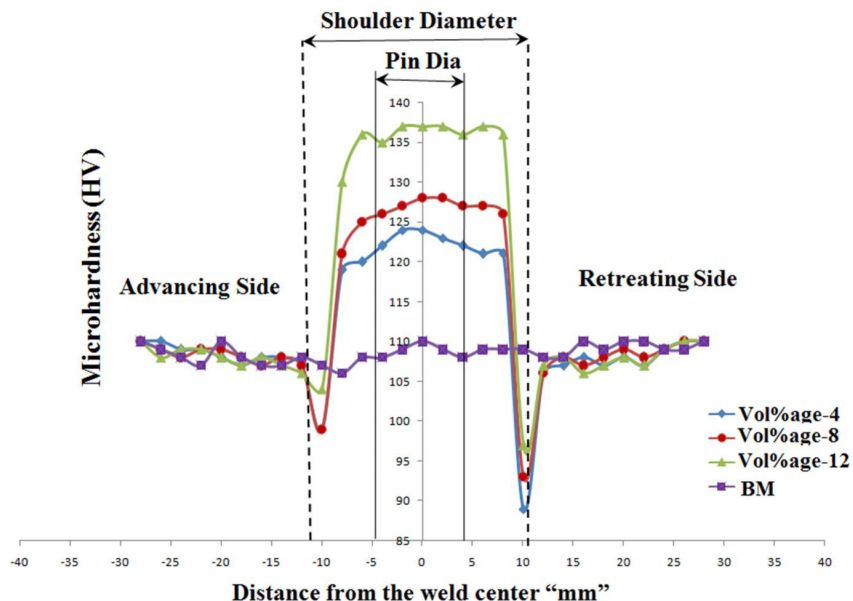


Fig. 7 Microhardness distribution of FSP samples for different volume percentages (4, 8 and 12) and the base metal.

The tensile properties such as UTS, YS and %EL are presented in Table 3. The increase in UTS and YS of the surface composites is accompanied by a decrease in ductility with increase in volume fraction of the  $Al_2O_3$ p (KhederI A.R.I. et al. 2011). The increased UTS of the surface composites over the pure copper matrix is due to the grain refinement of copper in the surface composite which can be related to the interaction between the powder particles and dislocations within the matrix. It is also observed that there is an increase in the joint efficiency ([ultimate tensile strength of the weld / ultimate tensile strength of the base metal] X 100) with increase in the addition of  $Al_2O_3$  reinforcement particles. This is due to increase in the recrystallization temperature by pinning grain boundaries of the copper matrix and blocking the movement of dislocations and thus improving the strength at elevated temperature (Lianga. S. et al., 2004). The elongation of the friction stir processed (FSPed) specimens with the addition  $Al_2O_3$ p were reduced as compared to the pure copper (Barmouz. M. et al., 2011). This may be due to increase in volume percentage of reinforced particles which increases the effective slip distance of dislocations

during the deformation. Impact toughness of the CASCs was evaluated with different volume fraction of the reinforced particles. It is observed that at higher volume fraction of the reinforced particles in the copper matrix exhibited higher impact toughness value. This may be attributed to the addition of reinforced particles which causes softening of the matrix due to frictional heat of the tool shoulder and pin.

Table 2. Constant process parameters and tool dimensions

| Parameters                       | Value                                    |
|----------------------------------|--|
| Tool rotation speed              | 900 rpm                                  |
| Tool travel speed                | 40 mm/min                                |
| Tool pin profile                 | Concave shoulder with square pin profile |
| Tool tilt angle ( $\theta$ )     | 2 (degrees)                              |
| Tool shoulder diameter ( $D_s$ ) | 24 mm                                    |
| Tool pin diameter ( $D_p$ )      | 8 mm                                     |
| $D_s/D_p$ ratio                  | 3 mm                                     |
| Pin Length (L)                   | 2 mm                                     |
| Number of passes                 | 1  |

Table 3. Mechanical properties of copper -  $Al_2O_3$  surface composites

| Reinforced particles (Volume%) | UTS (MPa) | YS (MPa) | % El | Micro hardness (HV) | Impact Toughness (J) | Joint Efficiency (%) |
|--------------------------------|-----------|----------|------|---------------------|----------------------|----------------------|
| 4                              | 180       | 135      | 8.5  | 123                 | 8                    | 70                   |
| 8                              | 211       | 157      | 9.1  | 128                 | 10                   | 81                   |
| 12                             | 270       | 208      | 13.6 | 137                 | 14                   | 104                  |

### 3.3 Fractography

The tensile and impact fracture surface of the BM and FSPed samples made at different volume percentages such as 4%, 8% and 12% respectively were studied using SEM to understand the mode of failure and the results are presented in Fig.8 and Fig.9 respectively. The fractured surface of the BM failed in a ductile manner by the micro void coalescence mechanism which shows fine dimple structure at the fractured surface of the BM. The tensile and impact fracture surfaces of FSPed samples obtained at higher volume percentage (i.e. 12%) shows ductile fracture as compared to 4% and 8%. This may be due to the presence of many fine voids and dimples formed at fractured surfaces in which the grain size is finer for FSPed specimen made at optimum condition. Fracture surface of the FSPed samples made at 4 % and 8% of volume fraction conditions shows both ductile and brittle failure. This is due to dispersion of very large number of reinforcement  $Al_2O_3$ p which severely limits the movement of dislocations and decreases the ductility significantly.

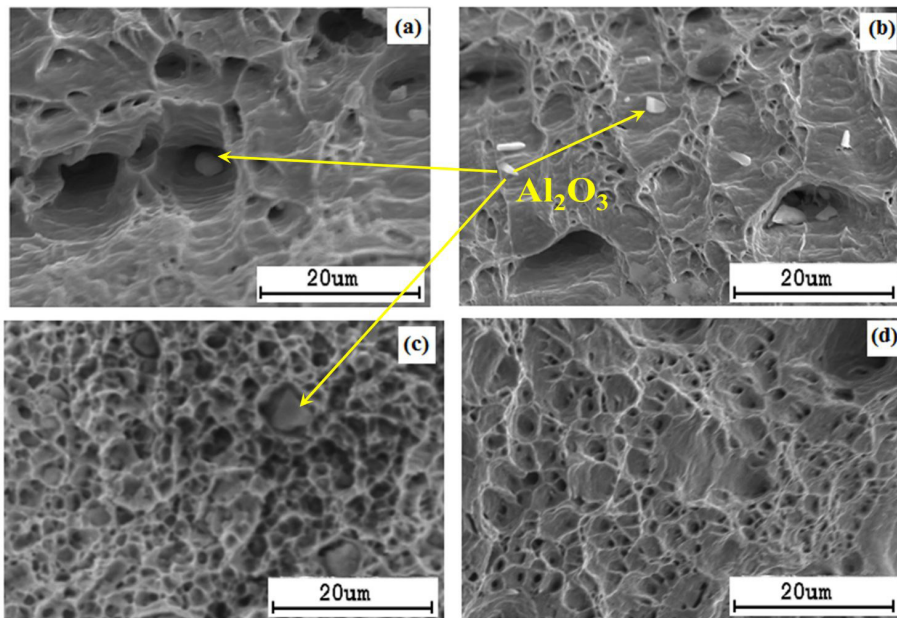


Fig. 8. . Fractographs of tensile (a-d) of FSP samples at different volume percentages (a) 4 % (b) 8 % (c) 12 % and (d) base metal.

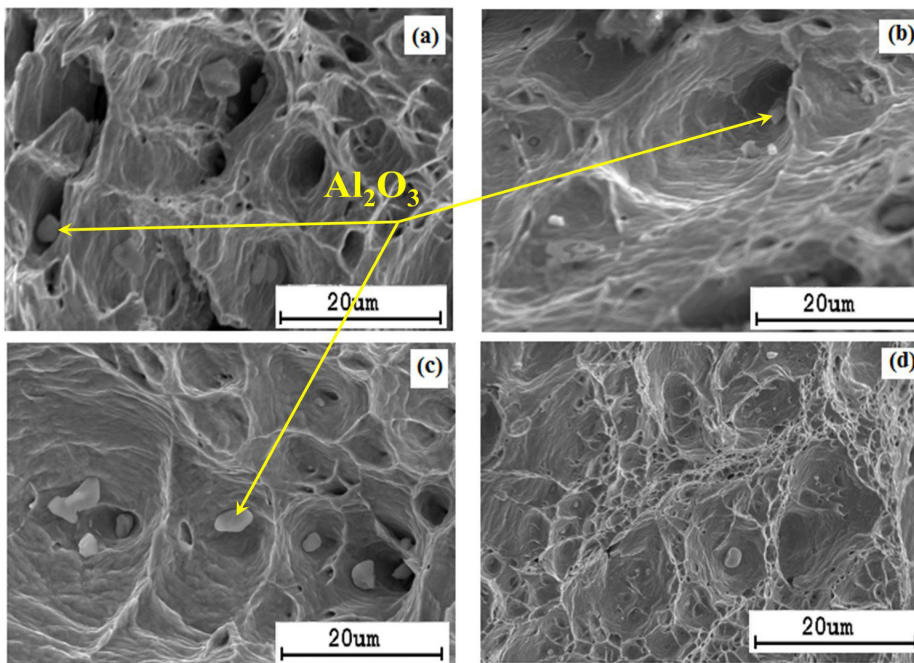


Fig. 9. Fractographs of impact (a-d) of FSP samples at different volume percentages (a) 4 % (b) 8 % (c) 12 % and (d) BM.



#### 4. Conclusions

mechanical The influence of reinforcement particles varying the volume percentage of  $\text{Al}_2\text{O}_3\text{p}$  on microstructure and properties of Cu- $\text{Al}_2\text{O}_3$  surface composites fabricated via FSP were investigated and the following conclusions were drawn.

- (1) Tensile properties of CASCs increased with increase in the volume percentage of reinforcement particles. This is due to the addition of the hard  $\text{Al}_2\text{O}_3$  reinforcement particles which increases the recrystallization temperature by pinning grain boundaries of the copper matrix and blocking the movement of dislocations.
- (2) The microhardness values of all the CASCs increased as compared with the average hardness of the copper matrix due to the presence of hard reinforcement particles.
- (3) The reinforcement particles ( $\text{Al}_2\text{O}_3\text{p}$ ) were distributed uniformly in the processed zone. This may be due to the position of the groove exactly tangential to the tool pin.
- (4) Severe plastic deformation and frictional heating in the SZ during FSP resulted in generation of a recrystallized equiaxed microstructure which is due to the occurrence of DRX.
- (5) The tensile and impact fracture surfaces of FSPed samples made at higher volume percentage (i.e. 12%) shows ductile fracture as compared to other volume percentages such as 4% and 8%. This is due to the presence of fine equiaxed grains which resulted in the formation of fine voids and dimples at fractured surfaces.

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#### References

- [1] Barmouz, M., Asadi, P., Besharati Givi, M. K., & Taherishargh, M., 2011. Investigation of mechanical properties of Cu/SiC composite fabricated by FSP: Effect of SiC particles size and volume fraction, *Mater. Sci. and Eng. A.* 528, pp.1740-1749.
- [2] Barmouz, M., Besharati Givi, M.K., & Seyfi, J., 2011. On the role of processing parameters in producing Cu/SiC metal matrix composite via friction stir processing: Investigating microstructure, microhardness, wear and tensile behaviour, *Materials Characterization.* 62(1), pp108-117.
- [3] Chang, C.I., Du, X.H., & Huang, J.C., 2007. Achieving ultra fine grain size in Mg–Al–Zn alloy by friction stir processing, *Scripta Mater.* 57, pp.209-212.
- [4] Devaraju, A., Kumar, A., & Kotiveerachari, B., 2013. Influence of rotational speed and reinforcement on wear and mechanical properties of aluminum hybrid composites via friction stir processing, *Mater and Des.* 45, pp.576-585.
- [5] Devaraju, A., & Kumar, A.(2011) Dry sliding wear and static immersion corrosion resistance of aluminum alloy 6061-T6/SiCp metal matrix composite prepared via friction stir processing, *Int J Adv Res Mech Eng.* 1(2), pp.62–68.
- [6] Groza, J.R.; & Gibling, J.C. (1993) Principles of Particle Selection for Dispersion Strengthened Copper, *Mater. Sci. and Eng. A.* 171, pp.115-125.
- [7] Kumar A., & Suvarna Raju, L.,2012. Influence of tool pin profile on friction stir welding of copper, *Materials and Manufacturing Process.* 27(12), pp.1414-1418.
- [8] Kheder, A.R.I., Marahleh, G.S., & Al-Jamea, D.M.K., 2011. Strengthening of Aluminum by SiC,  $\text{Al}_2\text{O}_3$  and MgO, *Jordan Journal of Mechanical and Industrial Engineering.* 5, pp.533 – 541.
- [9] Kaczmar, J.W., Pietrzak, K., & Wlosinski, W., 2000. The production and application of metal matrix composite materials, *J. Mater.Pro.Tech.*, 106, pp.58-67.
- [10] Lianga, S., Fana, Z., Xua, L., & Fangb, L., 2004. Kinetic analysis on  $\text{Al}_2\text{O}_3/\text{Cu}$  composite prepared by mechanical activation and internal oxidation. *Composites Part A.* 35, pp.1441-1446.
- [11] Mahmoud, E.R.I., Takahashi, M., Shibayanagi, T., & Ikeuchi, K., 2009. Effect of friction stir processing tool probe on fabrication of SiC particle reinforced composite on aluminum surface, *Sci and Tech of welding and Joining.* 14(5), pp.413-425.
- [12] Morisada, Y., Fujii, H., T. Nagaoka., & M. Fukusumi., 2006. Effect of friction stir processing with SiC particles on microstructure and

- hardness of AZ31, *Mater. Sci. and Eng. A.* 433, pp.50-54.
- [13] Mishra, R.S., & Ma, Z.Y., 2005. Friction stir welding and processing, *Mater. Sci. Eng. R.* 50(1-2), pp.1-78.
- [14] Mishra, R.S., and Ma, Z.Y., & Charit, I.,2003.Friction Stir Processing: A novel technique for fabrication of surface composite, *Mater. Lett.* 341, pp.307-310.
- [15] Sun, Y.F., & Fijji, H.,2011. The effect of SiC particles on the microstructure and mechanical properties of friction stir welded pure copper joints, *Mater. Sci. and Eng. A.* 528, pp.5470-5475.
- [16] shafiei-Zarghani, A., kashani-Bozorg S.F., & zarei-Hanzaki. A.,2009. Microstructures and mechanical properties of Al/ Al<sub>2</sub>O<sub>3</sub> surface nano-composite layer produced by Friction Stir processing, *Mater Sci Eng A.* 500, pp.84-91.
- [17] Shojiro,O.(1994) *Metallic Properties of Metallic Composites*, New York, Marcel Dekker Inc.Thomas,
- [18] W.M., Nicholas, E.D., & Needham, J.C.,1991. International Patent Application No. PCT/GB92/02203; December.