Research Article

Friction and Wear Response of Friction Stir Processed Cu/ZrO₂ Surface Nano-Composite

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The present work aims to develop Cu/ZrO₂ surface composite by friction stir processing and analyse the effect of zirconia incorporation on microstructure, mechanical, and tribological behaviour of developed copper matrix composite. Microstructural observations indicated that grains were equiaxed and fine in the stir zone of the composite and zirconia particles were uniformly dispersed in the copper matrix with excellent bonding. The test results for mechanical and wear behaviour showed increment in hardness and wear resistance as compared to copper which may be because of the effect of zirconia presence and grain refinement. The fabricated composite displayed higher value of average friction coefficient in comparison to as received copper. The worn surface observed by SEM revealed the predominance of adhesion and delamination wear mechanism in base copper.

1. Introduction

Copper has been prime material for applications in industries where electrical and thermal conductivity is a major concern [1]. This is due to satisfactory electrical and thermal conductivity, workability [2], and high oxidation and corrosion resistance [3] of copper. However, poor mechanical and wear properties limit its applications [3]. Therefore, ceramic particles are usually introduced in copper and their alloys to improve their strength and wear resistance [4–6]. Although ceramic particle reinforcement causes enhancement in mechanical and wear properties but by virtue of the occupation of hard (non-deformable) ceramic particles, metal matrix composites (MMCs) face problems in terms of loss in ductility and toughness [7]. It is observed that in various applications, only surface needs to make harden and wear resistant without disturbing the bulk configuration and structure and hence retaining the ductility and toughness [8, 9].

At present, liquid phase methods like stir casting, squeeze casting, and infiltration are being used for MMCs fabrication [9, 10]. As these are melt-based processes, temperature during processing is very high which leads to initiation of interfacial interaction between the reinforcements and the matrix and hence generation of some disturbing phases that finally hamper the performance of the composites [11, 12]. The ill effects encountered during melt-based processes may be eradicated by using solid-based processing techniques where temperature rise is not fair to be considered a threat for initiation of interfacial reactions. Recently, FSP, a variant of FSW, is in trend for manufacturing of MMCs due to its solid-state nature [13]. For example, S. Sarvankumar et al. [5] used FSP for the synthesis of Cu/AlN surface composite and investigated the effect of volume fraction on microhardness and wear behaviour. The results showed that increased volume fraction resulted in improved hardness and wear properties of the fabricated composite.
Friction stir processing is considered solid-state process as temperature rise does not melt the materials during processing and work on the same principle as friction stir welding which was first introduced by The Welding Institute (TWI) of UK in 1991 for microstructural modification [7, 14]. As mentioned earlier, its capability is not only limited to grain refinement but it has also gathered attention globally for surface/bulk composite fabrication. In the case, a given dimension plate with machined grooves/holes on the surface generally in the middle along the length with carefully packed ceramic particles is used. Then, a hard tool, generally non-consumable with distinctively developed shoulder and pin, is inserted in the plate. The material underneath the shoulder get plasticised due to heat generated due to plastic deformation and friction at the contacting point. After sufficient plasticization, it starts moving from advancing side to retreating side because of the rotational motion of the tool. Because of the synergetic effect of material flow and its mixing during the process, it becomes possible to incorporate reinforcements and hence fabricate composites [7, 15].

Particulates like AlN, SiC, TiC, and TiB₂ have been used as particulates for the manufacturing of copper matrix composites. ZrO₂ is also a ceramic material and has been used as filler in the copper matrix through various processing techniques [16–19]. It possesses high hardness, melting temperature, elastic modulus, and low adhesion friction along with very low reactivity with metals [20, 21]. As far as the available literature is concerned, there is nothing related to processing of Cu/ZrO₂ by FSP. Therefore, the present work is focused to fabricate Cu/ZrO₂ surface composite via FSP and to investigate the associated microstructure, hardness, and dry sliding wear behaviour.

2. Materials and Method

2.1. Manufacturing of the Composite. A plate of dimension 150 x100 x 6 mm³ was considered for the present investigation. The chemical composition of the copper was as follows: elements (Wt.%) P: 0.0003, As: 0.0005, Sb: 0.0004, Te: 0.0002, and remaining copper. To fill the powder of the reinforcement, a groove of dimension 1.5 mm wide and 3.5 mm deep with rectangular cross-section was machined on the surface of the plate. To maintain the accuracy, CNC end mill cutter was utilised for machining of grooves. Grooves were in the centre of the plate along the length.

The grooves were fully packed with zirconia powder. To ensure it was fully packed, multiple times, plates were lightly hit in the side region of the plate. Before processing, groove opening was closed by a tool having no pin so that during processing, loss of powder can be minimised.

Then, it was processed for the composite fabrication with the equivalent tool which had pin.

The tool material was super alloy IN718 in peak aged form [22]. The tool shoulder was 18 mm in diameter. For smooth flow of material during processing, cylindrical pin contour with thread was used. Keeping plate and groove dimensions in mind, the length and diameter of pin were kept 6 mm and 4 mm, respectively. The entire fabrication was carried out on a modified vertical milling machine. After rigorous trials and exhaustive literature survey, the processing parameters were decided which were kept constant for the entire experimentations. The processing parameters were as follows: 920 RPM, 40 mm/min, and 2.5°, rotational speed, bed movement, and tilt angle, respectively.

A schematic of the experimental steps is presented in Figure 1.

2.2. Sample Extraction and Characterisation. Figure 2 depicts schematically how specimens were extracted for characterisation.

For microstructure characterisation, specimens were machined perpendicular to the length of the processed plate. The machined specimens were ground and polished carefully as per standard metallographic practice. An etchant with composition 10g chronic acid, 1 g sodium sulphate, 0.85 ml hydrochloric acid, and 50 ml distilled water was prepared. The polished specimens were etched and detected beneath optical microscopy for microstructural features. The microstructural features were also observed under scanning electron microscope (SEM) (ZEISS EVO 18 RESEARCH, 20KV) but with un-etched samples. The polished samples were also analysed by electron back scattered diffraction (EBSD) analysis. The samples were electro-polished in a solution prepared by phosphoric acid and methanol at -12°C and 11 V. To observe the hardness at the various sections of the processed plate, Vickers microhardness tester was used. The processed plates were machined by 1 mm from the top for hardness indentation and indented cross-sectionally. Between every indentation, a gap of 0.5 mm was maintained. 300 gram load was used for 10 seconds during indentation for all the specimens. The chemical analysis of the plate before experimentation and after investigation was done by X-ray diffraction technique.

Friction coefficients and wear properties of plates before investigation and after experimentation were evaluated by a pin on disk tribometer at room temperature. Cylindrical specimens of size 4 mm in diameter and 30 mm in length were machined from the centre of SZ of the fabricated composite. A hardened steel which was in disc form, used as counter face. The EN-32 steel used had hardness of 65 HRC. Before experimentation, specimens were polished with various grades of emery papers. To ensure the maximum conformity, disc was also polished with surface grinder. The test parameters were kept constant during the experimentations. A normal load of 20 N and velocity of 1 m/s were maintained for all the specimens. The sliding distance (1000 m) was also kept constant for all the specimens. The friction coefficients of the specimens were determined by the machine automatically. Wear rate of the specimens was calculated by cumulative weight loss method. Before the commencement of test, samples were weighted. After the test for 100 m of sliding distance, again it was weighted. This was repeated ten times so that 1000 m of sliding distance could be completed. Then, the cumulative weight loss was plotted on the vertical axis and distance on horizontal axis to calculate wear rate. The wear out surfaces post-test was
3. Results and Discussion

3.1. Surface Appearance. Figure 3 displays the macroscopic top view of the fabricated Cu/ZrO₂ surface composite. The macrograph resembles the typical ring-like patterns which is the characteristic of FSP. There are no defects like depressions or discontinuities visible on the surface of processed plate. Ring-like patterns without defects obviously justify the effectiveness of the chosen process parameters for the experimentation. Such type of surface appearance in the case of FSPed plate is required for defect-free SZ. Defects on the surface of processed plates accompany corresponding defects in the SZ of the composite. Frictional heat generated due to relative motion between tool shoulder and workpiece soften copper and at elevated temperature and copper suffered severe plastic deformation. The severely plastically deformed copper moves from one side of the tool pin to other side of the tool pin which is also known as advancing and retreating side, respectively. The simultaneous movement of materials in translation and rotation led to formation of ring-type pattern on the processed plate.

3.2. Macrostructure. The cross-sectional macrostructure of fabricated Cu/ZrO₂ surface composite is depicted in Figure 4. The SZ region in which particles have been incorporated is quite clearly visible from the macrograph. It can be observed that the groove machined for particle compaction has completely disappeared from the copper plate which indicates that the composite formation process has been completed and material flow during composite fabrication was continuous. The figure also indicates that defects like tunnels and worm holes are not there. The macroscopic image shows that SZ is free from the irregularities.

The absence of defects in macrostructure may be because of ample flow of plastisised copper from one side of the pin to other, leads grooves to crash and mixed the filled zirconia with plastisised copper and eventually resulted into defect free SZ of FSPed Cu/ZrO₂ surface composite. The macrostructure of SZ of the composite is alike basin. That means the shape is not uniform. Its width is larger at the top and lesser at the bottom. This is believed to be varied nature of material flow during FSP. The tool being varied shape and size at different positions affects materials differently during

Figure 1: Illustrative view of the process (a) plate without groove. (b) Plate with groove. (c) Plate filled with powder. (d) Groove closing. (e) Final processed plate.

Figure 2: Diagrammatic view for specimens extraction from the plate.
processing. The top material is affected by shoulder having larger diameter and the bottom by pin with lesser diameter and hence width changes from top to bottom.

3.3. Microstructure. Figure 5 presents SEM image of zirconia particles and corresponding size distribution.

The average particle size of zirconia was estimated to be 1.21 \( \mu \text{m} \). The particles are small in size having smooth surfaces with no sharp corners.

Figure 6 depicts optical image of SZ of the composite at various area.

The microstructural features of the fabricated composite can be separated into two distinct zones (Figure 6(f)) comprising SZ and base metal (BM).

SZ is the region characterised by an exceedingly deformed structure with fine grains and evenly sputtered particles (Figures 6(b)–6(e)). Figure 6(f) shows the interface between SZ and BM. Thermally mechanically affected zone (TMAZ) and heat affected zone (HAZ) are not visible in the micrograph (Figure 6(f)). TMAZ is the region developed adjacent to SZ and is characterised by a highly deformed structure. TMAZ also gone through heat and deformation during FSP but owing to inadequate strain rate, recrystallization could not complete in this region. However, in the case of heat-affected zone (HAZ), plastic deformation did not occur during FSP, but due to annealing, grain growth is favoured. The obscene of TMAZ and HAZ in the present investigation may be thought of due to more heat flow along the depth of the plate in comparison to side of the plate. The heat flow/dissipation is favoured with high thermal conductivity of the materials. As in the present case, the backing plate used was copper and hence it might have promoted more heat dissipation along the depth of the plate in comparison to transverse section of the plate. Onion rings were not visible in the composite (Figures 6(b)–6(e)). M. Sabbagian et al. [23] observed onion rings in the fabrication of Cu/TiC composite through FSP. The reason for the same they have given was different temperature and flow nature of material moving and coming together from the shoulder to pin zone at the bottom.

Electron back scattered diffraction technique was utilised for grain structure analysis. The outcome of the analysis, i.e., IPF and grain boundaries map, is represented in Figure 7.

The effect of FSP and zirconia incorporation on grain structure of the composite is visible from the micrograph. The structural feature of grain for base copper was coarse and elongated (Figure 7(a)). Before processing, the size of grain was estimated to be 107.4 \( \mu \text{m} \) for base copper. The size of grain for the composite changed drastically and reduced up to nano-scale. The grain size reduction in the SZ of the fabricated composite may be because of pinning effect of reinforced zirconia particles which curb the grain growth by covering grain boundaries sliding. Moreover, heat and extremely high deformation due to tool movement promoted dynamic recrystallization which creates new nucleating sites and resulted in grain reduction.

Figures 8(a)–8(c) represent the SEM image for the composite captured at varied magnifications.

The whole SZ is comprised of zirconia particles. The zirconia particles are reasonably apart from each other.
Particle-free zones are not visible in the micrographs. Hence, safely it may be assumed that sputtering of reinforcements was fairly even in SZ. This was due to the fact that high stirring rate of tool mixed the compacted zirconia and heavily deformed copper together and led to uniform dispersion of ZrO₂. Contrary to this, some results have been reported in which un-even scattering of reinforcements in the matrix was reported due to no-suitable variables chosen during processing [13, 24]. The variation in zirconia particle dispersion in SZ was not observed (Figures 8(a)–8(c)). It might have been because of non-melting feature of the process.

The reinforced zirconia particles could not move freely due to high viscosity of plasticised copper matrix. Figure 8(c) depicts SEM image of the composite captured at high magnification. The interface of zirconia and copper is continuous without any interruption. Tiny size and continuous surface of reinforcement (Figure 5(a)) allowed smooth flow of plasticised copper all over the zirconia particles. There was no appreciable change in morphology of the reinforcement during processing (Figures 5(a) and 8(c)).

Literatures are available which shows appreciable changes in the morphology of the reinforcement before and after processing. [24–26]. During FSP, materials experience high strain rates owing to frictional heating and high rotational speed of the tool. Ceramic reinforcements are having brittle nature so they cannot store potential energy like metals and hence whenever experience force gets fractured directly.

Zirconia did not break in the present investigation which may be due to the following reasons. Firstly, due to tiny size, they could not provide much resistance to the flowing copper matrix.

Secondly, due to the absence of sharp edges in the reinforced zirconia, stress concentration did not happen. Lastly,
the surface of the reinforced zirconia particulates was smooth, so plasticised copper matrix flowed smoothly without any hindrance.

3.4. Chemical Composition. The X-ray diffraction peaks (Figure 9) of the fabricated Cu/ZrO₂ surface composite displayed only copper and zirconia peaks.

No other peaks related to discernible reactional products were detected in the pattern. It clearly shows that no zirconia particle/copper matrix confluence reactions occurred during processing. Temperature rise during FSW is affected by process parameters especially the ratio of tool RPM to bed movement. In the present case, the selected parameters were sufficient for fairly even scattering of particulates.

The highest temperature reported during FSP of copper that could reach with such parameters has been reported to be 0.8 Tₘ of copper [27] which was not adequate for initiation of reaction between copper and zirconia. Further, low reactivity of zirconia with metals and short exposure duration during FSP may be another reason behind the denial of in-situ products in the composite.

3.5. Microhardness. The hardness distribution of the fabricated Cu/ZrO₂ composite is illustrated in Figure 10.

The average hardness of base copper was estimated to be 64 ± 2.7 HV whereas it was 116 ± 4.6 HV for the composite. The side region of the plate has lower hardness than the processed one. This may be because of the higher heat input.
in the region which reduces dislocation density and compressive residual stress. The improved hardness of the composite may be thought of because of the presence of hard zirconia particles. Being a hard phase, it not only increased the hardness but also impede the grain growth and hence reduction in grain size which further contributed in increased hardness.

As per the Hall-Petch relation, grain size is inversely proportional to hardness. Additionally, generated dislocations due to thermal mismatch between copper and zirconia also contributed to enhancement in hardness by hindering the motion of dislocations [28]. Moreover, the Orowan mechanism may also be responsible for higher hardness [29]. As reported by this mechanism, zirconia particles acted as a barrier to dislocation movement and hence causing improvement in hardness.

3.6. Tribological Behaviour. Figure 11 depicts the plot for friction coefficient V/S time graph. From the figure, it is obvious that the average value of friction coefficient in the case composite is slightly more in comparison to un-processed copper. Also, the fluctuation is more for the composite compared to base copper.

As mentioned earlier, incorporation of zirconia in copper matrix and grain refinement due to FSP caused enhancement in hardness of the composites significantly. The increased hardness improved the abrasion resistance of the composite. Hence, we can say that the presence of zirconia led to gain in sliding resistance; therefore, the average value of friction coefficient elevated for the composite. The thin tribolo-layer pounded on the surface of specimens and counter disc because of depreciation of wear debris reduced the fluctuation in friction coefficient for base copper. However, this development of tribolo-layer was not favoured in the case of the composites because of the presence of hard zirconia particles which abraded the layer.

Figure 12 shows the results of wear test.
Figure 8: SEM image for composite at varied magnifications within SZ.

Figure 9: XRD pattern of the fabricated composite.
It can be observed that the weight loss in the case of the fabricated composite (Cu/ZrO$_2$) is less in comparison of as received copper. It is also worthy to note that as the distance of sliding is increasing, the weight loss for the composite is increasing almost linearly. However, this is not the case with matrix. Figures 13(a) and 13(b) present SEM image of wear out surfaces for base copper and composite, respectively.

It can be seen from the micrographs that adhesion is dominant in the case of base copper in comparison to the fabricated composite. Delamination and grooves due to ploughing effect are quite clearly visible in the micrograph (Figure 13(a)). Due to the dominance of adhesion, delamination pits formed in as received copper which caused relentless material eviction from copper surface. However, the presence of hard zirconia particles in fabricated composite enervated the outcome of adhesion. That’s why the loss due to adhesion was less in the case of composite. Further, due to higher hardness of the composite as compared to as
received composite, resistance towards abrasive wear was also higher. The elevated value of abrasion resistance farther reduces the wear loss of the composite.

4. Conclusions

In this work, copper-ZrO2 composite was manufactured via FSP. Also, the effect of ZrO2 reinforcement on microstructure, mechanical, and wear behaviour was adjudged. Outcomes of the investigation are summarised below:

(i) The dispersion of zirconia particles was fairly even with no obvious defects. The grain structure in the case of composite was fine comparatively

(ii) The size and shape of the particulate have not changed during processing. Also, there is no interruption at the interface of the composite which shows excellent bonding between copper and zirconia

(iii) The hardness for the composite was observed more in comparison to base copper. Also, the hardness value in vicinity of processed section was found less than that of base copper

(iv) The value of friction coefficient was more for the composite with respect to base copper

(v) Wear loss for the composite was less in comparison to copper
**Data Availability**

The datasets generated during and/or analysed during the current study are available from the corresponding author on reasonable request.

**Conflicts of Interest**

The authors declare that they have no conflicts of interest.

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**References**


