Microstructural characterization and sliding wear behavior of Cu/TiC copper matrix composites developed using friction stir processing

I. Dinaharan[a*], E.T. Akinlabi[a], D.G. Hattingh[b]

[a]Department of Mechanical Engineering Science, University of Johannesburg, Auckland Park Kingsway Campus, Johannesburg 2006, South Africa.

[b]Department of Mechanical Engineering, Nelson Mandela University, Port Elizabeth 6001, South Africa.

[*]Corresponding author

E-mail addresses: dinaweld2009@gmail.com (I. Dinaharan), etakinlabi@uj.ac.za (E.T. Akinlabi), danie.hattingh@mandela.ac.za (D.G. Hattingh)

Abstract

The relatively new severe plastic deformation method, friction stir processing (FSP) is a cutting-edge process to synthesize surface and bulk metal matrix composites. The present work is focused to produce Cu/TiC copper matrix composites (CMCs) and investigate the microstructure and sliding wear behavior at room temperature without lubrication. In the beginning of the process, TiC particulates were pressed in a machined groove on the surface of copper plates. The dimensions of the groove were altered to produce four different volume fractions of TiC particulates (0, 6, 12, and 18 vol.%). FSP was accomplished by an optimized set of process parameters. The microstructure was observed using optical microscopy, scanning electron microscopy (SEM) and electron back scattered diffraction (EBSD). The microstructures showed a consistent dispersion of TiC particulates in the copper matrix irrespective of the volume fraction. The dispersion was observed to be uniform across the whole stir zone region. The interfacial bonding with the copper was proper. The reinforcement of TiC particulates enhanced the microhardness and led to a reduction the wear rate of the composite remarkably. TiC particulates changed the wear mechanism and the

geometry of wear debris. Highest hardness and lowest wear rate were observed in Cu/18 vol.% TiC CMC.

Key words: Copper matrix composites; Friction stir processing; Titanium carbide; Microstructure; Wear rate.

1. Introduction

Pure copper and its alloys are comprehensively utilized in industrial applications due to several desirable characteristics which include superior electrical and thermal conductivity, low cost, decent corrosion resistance, excellent formability and simple to manufacture [1–3]. The progress in electronic and electrical industries has created a need to produce connector materials having good strength and wear resistance. The wear resistance of pure copper is poor which restricts its usage in applications where a sliding wear occurs [4,5]. Reinforcing pure copper with hard phase ceramic particulates helps to enhance the wear resistance [6–8]. Such material is universally recognized as copper matrix composites (CMCs). In the midst of a variety of ceramic particulates, titanium carbide (TiC) is a promising ceramic particulate to be used as reinforcement CMCs owing to its high modulus, hardness, melting temperature, low chemical reactivity and density [9–11].

Several researchers made attempts to synthesize and investigate the properties of Cu/TiC CMCs by various casting based and solid-state techniques in the past decades. Chrysanthou and Erbaccio [12] prepared Cu/TiC CMCs by the in-situ reaction between Ti and carbon black with molten copper and reported the microstructure. Lu el al. [13] manufactured Cu/TiC using selective laser melting and found that incorporation of Ni improved the microstructure and surface roughness. Kennedy el al. [14] developed Cu/TiC using self-propagating high-temperature synthesis and estimated the role of C:Ti ratio on the wetting behaviour of TiC. Akhtar et al. [15] obtained Cu/TiC CMCs with high volume fraction of TiC particulates using mechanical alloying of Cu and TiC particulates and analyzed the microstructural evolution and wear behavior. Liang et al. [16] formed Cu/TiC CMCs by applying self-propagating high-temperature synthesis and investigated the reaction mechanism and sequence in the Cu–Ti–C system. Buytoz el al. [17] developed Cu/TiC CMCs using powder metallurgy and elaborated the influence of TiC volume fraction on microstructure and thermal properties. Wang el al. [18] produced Cu/TiC CMCs using sparks plasma sintering and assessed the effect of two step ball milling on electrical and mechanical properties. Li et al. [19] fabricated Cu/TiC CMCs using powder metallurgy method by mixing Cu and Ti2AlC and evaluated the tensile properties. Liang et al. [20] synthesized Cu/TiC CMCs using thermal explosion reaction method and studied the reaction behaviour and microstructure of Cu–Ti–C system under argon and air atmosphere.

It is obvious from the literature survey that it is possible to synthesize Cu/TiC CMCs utilizing a range of techniques including in situ casting, selective laser melting, powder metallurgy, mechanical alloying, sparks plasma sintering, self-propagating high-temperature synthesis, thermal explosion reaction etc. But those synthesizing techniques were constantly producing several defects such as pores [10,13,21], voids [14,18], particle aggregations [12,21] and inconsistent distribution [13,22]. The mechanical and tribological behavior of CMCs are deteriorated due to those defects. Friction stir processing (FSP) has been developed as a capable process for the production of sound metal matrix composites [23], grain refinement [24], inducing super plasticity [25] and repairing defects in metallic materials [26].

FSP is a relatively new severe plastic deformation (SPD) process to develop bulk and surface metal matrix composites [27]. A groove or an array of circular holes of required dimensions are machined on the surface of the metallic plate. The desirable ceramic particulates are afterwards packed in a groove or an array of holes. A non-consumable spinning tool under sufficient axial force is inserted at one end and moved over the groove or holes. The substrate material experiences intense plastic deformation throughout FSP to produce a composite. FSP is a promising method to overcome the common defects encountered in conventional liquid metallurgy and powder metallurgy routes. FSP is a low energy consumption process. The total operation is completed in solid state. FSP method is not sensitive to the kind of ceramic particle and its density gradient with the matrix material [28].

FSP techniques has been effectively applied to synthesize CMCs reinforced with SiC [29], Al_2O_3 [30], TiC [31], B_4C [32], WC [33], Y_2O_3 [34], CNT [35] and graphite [36]. Sufficient literature is not available in the field of CMCs using FSP. Hence, the goal of the current work is to develop Cu/TiC CMCs using FSP and to study the microstructural evolution and sliding wear behavior at room temperature without lubrication.

2. Experimental procedure

Pure copper plates of dimensions 300 mm X 300 mm X 6 mm were used for this investigation. The microstructure of the copper is presented in Fig. 1a. Grooves of 3.5 mm deep were machined into the plate by wire EDM and stuffed with TiC powder. Fig. 1b presents the SEM micrograph of TiC particulates (supplied by M/s Alfa Aeser) used in this work. The average size of TiC particulates was 2 µm. The top opening of the groove was closed using a pinless tool to arrest the escape of particulates while doing FSP. The FSP was performed on an industrial purpose FSW machine (I-STIR) using optimized conditions (Table 1) reported in a literature for CMCs [37]. The same parameters and tool design except pin was employed for capping operation. A schematic of the standard FSP process to synthesize the composite is presented in Fig. 2.

Specimens were prepared across the friction stir processed plates and were polished semi automatically (STRUERS Labopol). The polished specimens were etched with a color etchant containing 20 g chromic acid, 2 g sodium sulfate, 1.7 ml HCl (35%) in 100 ml distilled water. The microstructural features were recorded using an optical microscope (OLYMPUS-BX51M) at various locations within the processed zone and a field emission scanning electron microscope (FESEM, CARL ZEISS-SIGMA HV). Selected specimens were observed using electron back scattered diffraction (EBSD). The microhardness was recorded using a microhardness tester (MITUTOYO HM-220) at 500 g load applied for 15 seconds at various locations within the stir zone.

The sliding wear behavior of Cu/TiC CMCs was measured using a pin-on-disc wear apparatus (DUCOM TR20-LE) at room temperature according to ASTM G99-04a standard. Specimens of dimensions 4 mm x 4 mm x 20 mm were machined within the processed zone using wire EDM. The wear test parameters were sliding velocity–1.5 m/s, normal force – 30 N and sliding distance – 3000 m. The surface of the pin (4 mm x 4 mm) was polished using emery sheets. The counter disc material was a hardened chromium steel disc. The height loss of the wear specimens was registered during wear test and calculated into wear rate. The wear rate was obtained by dividing volumetric loss to sliding distance. Atleast two tests were carried out for each experiment to get a representative data. SEM was applied to observe the worn surfaces and the wear debris.

3. Results and discussion

3.1. Microstructure of Cu/TiC CMCs

Fig. 3 presents the macrographs of the Cu/TiC CMCs at various content of TiC particles. No defects such as tunnels or pin holes observed in the macrographs. The area of the stir reduced with an increase in TiC particulates due to increased flow stress. TiC particulates acts as a hinderance and thermal barrier for the plasticization of copper. Figs. 4a–c reveal the representative SEM micrographs of the synthesized Cu/TiC CMCs with various content of TiC particulates. It is noticed in the micrographs that the TiC particulates are scattered all over the copper matrix. The dispersion of TiC particulates is visualized as reasonably consistent. The frictional heat developed during FSP is sufficient to cause plasticization of pure copper. The plasticized copper is transported from the advancing side (AS) to retreading side (RS) due to the rotary movement of the tool. The movement of plasticized copper mechanically mixes with the TiC particulates initially stuffed in the groove. The groove loses its identity and disappears in the process of developing composite matrix. The plasticized copper mixed with TiC particulates is ultimately consolidated at the heal of the tool with the aid of axial forge force applied along the tool axis. The mixing action of plasticized copper with TiC particulates can be correlated to the material flow due to rotating and mixing action of the FSP tool. The dispersion of reinforcement particulates in the copper matrix is mainly affected by the tool rotational speed [32]. Insufficient tool rotational speed causes poor mixing of particulates and produces particle free regions and particle clusters. Too much rotation of plasticized matrix material induces porosities in the region of particulates in the composite. There are no particle clusters or pores or particle free regions in Fig. 4. It suggests that the tool rotational speed selected in this work is adequate to produce an acceptable dispersion of TiC particulates in the composite. Though grain boundaries are not visible in Fig. 4, it can be thought that majority of TiC particulates are spread within the grains. Few TiC particulates are likely to be on grain boundary in Fig. 4. But there is no continuous arrangement of TiC particulates in the composite to resemble a grain structure. This observation suggests that there are no clusters of TiC particulates beside the grain boundaries. Hence, the dispersion is predominantly intragranular. Clustering reduces the mechanical and tribological performance of the composite. Clustering is likely to occur in

CMCs produced using liquid metallurgy methods. The density gradient between the copper matrix and the ceramic particle cause movement of particulates within the molten copper. Since, FSP is a solid-state method; the unrestricted motion of particulates within the plasticized copper due to density variation is totally nonexistent. As a result, the chances of segregation of particulates are remote. The FSP process has produced the desirable dispersion in the copper matrix.

FSP applies severe plastic strain on the copper matrix which has the tendency to change the geometry of the reinforcement particulates [28,38]. The change in geometry of TiC particulates before and after FSP can be estimated by comparing Fig.1, Fig. 4 and 5. There is no noticeable change in geometry (both size and shape) of TiC particulates after FSP. TiC particulates endured the severe plastic strain and mechanical mixing effort of the spinning tool. The initial size of TiC particle was too small to oppose the flow of plasticized copper and largely flowed along with the plasticized copper during FSP. Further, the initial morphology of the TiC particulates exhibited no major sharp corners around the particulates. Those two factors helped the TiC particulates to avoid breakage and retain its size and shape after FSP.

The SEM micrographs of Cu/TiC CMCs at higher magnification are presented in Fig. 5. Some grain boundaries also revealed in this figure by the etchant used. Fig. 5 also reveals the details of a clear interface region among the TiC particle and the copper matrix. Additionally, it shows that the TiC particulates are not surrounded by any pores or reaction products. This observation suggests that the TiC particulates are fused with the copper matrix. Barmouz and Givi [39] and Sabbaghian et al. [31] noticed pores at the interface of SiC and TiC particulates in respectively Cu/SiC and Cu/TiC CMCs produced using FSP method. A pore in FSP occurs due to insufficient material flow as well as irregular particulates size diverting the smooth flow of plasticized material around it. Thus, the nonappearance of pores can be ascribed to acceptable plasticization of copper and subsequent smooth flow around the TiC particulates. The nature of the interface plays a critical role in structural components and in other applications which includes coating, heterogeneous catalysis, fuel cells and microelectronics. Pores and reaction products around ceramic particulates restrict the effective transfer of load to the particle while tensile load is applied. Therefore, a sound interfacial bonding is an essential requirement to enhance the performance of composites in spite of homogeneous dispersion of reinforcement particulates. Undesirable reaction occurs if the temperature of the processing method is high especially in the case of liquid metallurgy processing. The reaction products generally engulf the interface and deteriorate the interfacial bonding strength. Some investigators provided a detailed insight into the possible interfacial reactions in Cu/SiC and Cu/B4C CMCs [40–42]. The temperature raise and heat generation during FSP is meager to initiate a reaction among TiC particulate and copper. Hence, no reaction products appear around TiC particulates in Fig. 5.

Figs. 6a–f show the optical photomicrographs of Cu/18 vol.% TiC CMC recorded at different places within the stir zone which contains the prepared composite. The evidence suggests that TiC particulates are dispersed evenly within the stir zone. No place within the stir zone which is left without any particulates. The difference in the dispersion of TiC particulates across the stir zone is insignificant. This observation indicates that the dispersion of TiC particulates is independent upon the observed spot in the stir zone. However, some researcher observed significant variation in the dispersion of ceramic particulates within the stir zone [31,33,35]. The insignificant variation in the dispersion in this study can be linked to appropriate plasticization of copper and mixing of material by the spinning tool at the chosen process parameters. A constant dispersion of reinforcement particulates across the bulk composite is required to boost the service life of components. It is complicated to achieve constant dispersion using liquid metallurgy routes [22,43]. The change in the velocity of solidification front within the mold will cause significant variation in dispersion of particulates across the casting. The micrographs (Figs.6e) at the bottom of the stir zone showed no presence of onion rings. This suggests that the temperature gradient from top to bottom of the stir zone is not enough to blend the plasticized material flow to generate onion rings.

Figs. 7a and b shows the EBSD images of Cu and Cu/18 vol.% TiC CMC respectively. Large number of twins is visible (Fig. 7a) in the grain structure of copper. The twins are hardly observed in the composite (Fig. 7b). This can be attributed to the frictional heat which exceeds a certain value for a particular strain present in the stir zone. It is obvious from Fig. 6 that the grains are refined in the composite compared to pure copper. The grain refinement can be ascribed to the subsequent two factors. The generation of fine equiaxed grains in the CMC is the direct effect of dynamic recrystallization because of intense plastic deformation. FSP technique imposes a strain rate which may attain up to 80s–1 at the contact surface of the tool pin and the matrix material. The strain rate is massive considering other conventional SPD techniques [27]. The grains are eventually refined because of the high strain rate. Secondly, the incorporation of TiC particulates contributes to the grain refinement. TiC particulates pin the motion of grain boundaries delaying the rate of grain growth induced by dynamic recrystallization. This is called as pinning effect leading to refinement of the grains [39].

Figs. 8 shows the TEM images of Cu/18 vol.% TiC CMC at different locations in the copper matrix. Several features are observed in the images including fine grains (Fig. 8a), dislocations (Fig. 8b), pinning of dislocations (Fig. 8c) and annealing twins (Fig. 8d). As discussed earlier, the formation of fine grains is related to dynamic recrystallization. The dislocation density is not uniform across the grains. Some grains are saturated with dislocations and some grains do not have dislocation density. This observation hints that the recrystallization was disrupted or discontinuous. The follow factors cause the generation of dislocations in the copper matrix. The plasticized copper is subjected to a cycle of deformation and extrusion until it is consolidated and the tool advances. It is natural for any deformed material to generate dislocations. The dissimilar thermal properties of the copper and TiC particle cause uneven expansion and contraction during heating and cooling of FSP cycle. Dislocations are born to adjust the misfit strain caused by thermal disparity. Pinning of grains and entanglement of dislocations are due to Zener-pinning effect induced by fine TiC particles. Annealing process occurs once the copper grains are recrystallized. The initiation of growth fault transforms into a twin [44].

3.2. Microhardness of Cu/TiC CMCs

Fig. 9a depicts the microhardness of Cu/TiC CMCs showcasing the effect of volume fraction of TiC particulates. The incorporation of TiC particulates into the copper matrix raised the microhardness of the composite. The microhardness rises as the content of TiC particulates in the composite is increased. The microhardness was measured to be 80 Hv at 0 vol.% and 190 Hv at 18 vol.%. The strengthening mechanisms are explained as following. The immediate presence of hard ceramic particulates in a soft metallic matrix leads to an improvement in the hardness. Reinforcement of TiC particulates improves the dislocation density of the copper. The interaction between TiC particulates and dislocation density during indentation enhances the microhardness. More the volume fraction of TiC particulates more will be the dislocation density. Hence, the interaction will be more with higher volume fraction of TiC particulates which improves hardness further. The grain refinement in the composite is a strengthening factor to improve the hardness consistent with Hall-Petch relationship. The consistent dispersion of TiC particulates in the copper matrix provides Orowan strengthening. The above discussed factors lead to an improvement in hardness of the Cu/TiC CMC. The microhardness distribution across friction stir processed copper with 18 vol.% TiC particulates taken 2 mm from the shoulder region is depicted in Fig. 9b. The variation in hardness across the stir zone is minimum due to the consistent dispersion of TiC particulates.

3.3. Sliding wear behavior of Cu/TiC CMCs

Fig. 9a shows the wear rate of Cu/TiC CMCs showcasing the effect of volume fraction of TiC particulates. The raise in the content of TiC particulates lead to reduced wear of the composite. In other words, the wear resistance of the composite constantly improved with increment in TiC particulate content. The wear rate was estimated to be 248 x 10-5 mm3/m at 0 vol.% and 175 x 10-5 mm^3/m at 18 vol.%. The hardness and wear rate relationship are well documented and are related to each other as per Archard's laws of wear. The improvement in hardness improves the resistance to material removal during sliding wear. The copper matrix surrounding the TiC particulates is effortlessly removed during sliding. Subsequently, the net contact surface between the Cu/TiC CMC specimen and the counter disc is lessened compared to unreinforced copper. TiC particulates endure the applied normal load along the specimen axis. The proper interfacial bond between the TiC particle and the copper matrix delays the detachment of particulates. The consistent dispersion of TiC particulates causes a decrease in frictional coefficient. The above discussed factors lead to an increased wear resistance of the composite. The decrease in wear rate is not linear with an increase in TiC particle content (Fig. 9a). The intricate wear mechanisms occurring during sliding of the composite are accountable for the nonlinear behavior of the composite.

Figs. 10a–d show the SEM micrographs of the worn surfaces of Cu/TiC CMCs as a function of volume fraction of TiC particulates. The effect of TiC particle content is well pronounced on the appearance of the worn surfaces. The extent of plastic flow, craters and plowing marks gradually disappear with the rise in the volume fraction of TiC particulates. The worn surface of pure copper displays severe destruction in the form of deep craters and plastic flow. The frictional heat during sliding plasticizes the surface of the copper. The sharp asperities of the counterface effortlessly scratch and penetrate into the subsurface of the unreinforced copper. As a result, material is removed in large amounts at a faster rate. The wear mode is noticed to be adhesion. The reinforcement of TiC particulates strengthens the subsurface and resists the removal of material due to attack of the hard asperities of the counterface and plastic flow of copper. Therefore, the material removal takes place at slower pace and the damage to the worn surface is deferred. The worn surface of Cu/18 vol.% TiC CMC appears to be more uniform due to higher volume fraction of TiC particulates. Numerous debris are observed on the worn surface. No crates or cracks or plastic flow is found. The wear debris are non-sticky on the worn surface due the hard nature of TiC particulates. The wear mode changes to abrasive (Fig. 10c and d) from adhesion (Fig. 10a and b) with the increase in TiC particulates.

Figs. 11a–d show the SEM micrographs of the wear debris of Cu/TiC CMCs displaying the effect of volume fraction of TiC particulates. A considerable variation in the geometry of the wear debris is evident with an increase in TiC particulates. The size of wear debris decreases with increased TiC particulates in the composite. The wear debris of pure copper are so large and flaky in shape. The large size is due to cutting of subsurface of copper by counterface asperities. The size of wear debris reduces above 6 vol.% of TiC particulates in the composite. The size of wear debris is so fine for Cu/18 vol.% TiC CMC. The generation of fine wear debris can be related to the subsequent two causes; (a) the improvement in hardness of the composite due to reinforcement of TiC particulates and (b) the decrease in the actual contact between the wear specimen and the counterface due to TiC particulates. The debonded TiC particulates during sliding are scattered along the wear track. They change from two body abrasion wear into three body abrasion wear which forms fine debris. The mechanism of fine debris generation is equivalent to the principle of operation of high energy ball milling. The milling effect increases with increased volume fraction of TiC particulates leading to the generation of finer debris.

4. Conclusions

- TiC particulates were dispersed consistently in the copper matrix regardless of volume fraction. The microstructure was independent on the location within the stir zone.
- The grains of Cu/18 vol.% TiC CMC exhibited a finer structure compared to coarser grain structure of pure copper. The grain refinement was ascribed to dynamic recrystallization and pinning effect of TiC particulates.
- The initial shape and morphology of the TiC particulates were preserved after FSP due to smaller size and absence of sharp corners.
- The interfacial bonding between the TiC particle and the copper matrix was good. The interface was clear without any pores or reaction products.
- The microhardness of Cu/TiC CMCs increased with raise in content of TiC particulates. The microhardness was measured to be 80 Hv at 0 vol.% and 190 Hv at 18 vol.%.

- The wear rate of Cu/TiC CMCs decreased with increased volume fraction of TiC particulates. The wear rate was estimated to be 248 x 10-5 mm3/m at 0 vol.% and 175 x 10-5 mm3/m at 18 vol.%.

- The increase in volume fraction of TiC particulates transferred the wear mechanism from adhesion to abrasion and caused a reduction in the size and morphology of the wear debris.

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Table 1

FSP conditions.

Parameter	Values
Rotational speed (rpm)	1000
Traverse speed (mm/min)	40
Axial force (kN)	10
Shoulder diameter (mm)	24
Pin diameter (mm)	6
Pin length (mm)	5

Pin shape	Straight cylindrical
Tool material	Hot working steel
Passes (no.)	1
TiC content (Vol.%)	0,6,12 and 18