The effect of SiC content on the tribocorrosion performance of spark plasma sintered Al–SiC nanocomposites

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Abstract

As a follow-up study, the present work evaluates the tribocorrosion behavior of Al–SiC nanocomposites produced using spark plasma sintering. The tribocorrosion tests were carried out using a ball-on-disk tribometer with the sliding contact fully immersed in 3.5 wt% NaCl solution. Post-mortem characterization studies of the worn surfaces included scanning electron microscopy (SEM) coupled with an energy dispersive spectroscopy. From the results, oscillations in chronopotentiometry values characterize the in situ tribocorrosion behaviors of the composites. Shifts to the cathodic region at the beginning of sliding and immediate shift to the anodic region of the curve as sliding continues accounts for the oscillations in chronopotentiometry values. The SEM analyses infer the formation of adherent and modified tribolayers in the worn surfaces of the composites during sliding. The inert character and increasing resistance of the tribolayers enhance the tribocorrosion performance of the composites compared to the unreinforced Al alloy.

Keywords Tribocorrosion · Spark plasma sintering (SPS) · Coefficient of friction (COF) · Chronopotentiometry · Nanocomposites · Energy dispersive spectroscopy (EDS)

1 Introduction

The automotive industry uses Al–SiC composites for components such as pistons, bearings, cylinder liners, driveshafts and prop shafts and most especially for brake systems owing to properties such as low density, good electrical and thermal conductivities, high damping capacity and good corrosion resistance [1–3].

Tribological properties of aluminum matrix composites (AMCs) depend on intrinsic and extrinsic factors such as reinforcement volume fraction, particle size, applied load, temperature, speed etc. Several studies on the influence of reinforcement volume fraction on wear resistance of AMCs agree that wear resistance improved as volume fraction increased [4–6]. Some studies suggested that wear rate tend to increase beyond an optimum volume fraction [7, 8]. Investigations on the effect of reinforcement particle

size also indicated that wear resistance increased with increased particle sizes [9, 10]. Conversely, other reports [11–13] suggested that adding nano- or submicron-sized reinforcement particles improved wear resistance of AMCs as well. Although individually, the reinforcement volume fraction and particle size intrinsic properties improve wear resistance, interactions between these two properties at high volume fraction and coarse reinforcement particle size negatively affect wear resistance of Al–SiC composites [14]. Diler and Ipek [14] highlighted that Al–SiC composites exhibit the best performance against wear if the reinforcement particle size is smaller than that of the matrix particles.

Generally, the wear resistance of materials closely relates to its hardness property. In metal matrix composites (MMCs), the addition of hard ceramic reinforcing phases (such as SiC) improves the hardness of the

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composites by acting as load-carrying constituents in the composite. Nonetheless, the enhancement of MMCs hardness property has been reported to hinge on the interfacial bonding and characteristics between the metal matrix and reinforcing ceramic phase [15]. In the event of inadequate interfacial bonding resulting from fracture or fragmentation at the interface, some reinforcing particles are susceptible to be catastrophically pulled out from the matrix during sliding contact and may function as third body abrasives leading to decreased wear resistance [16–18]. With regards to corrosion, flaws or formation of anodic compounds at the metal matrix and ceramic reinforcement interface sets the interface as corrosion initiation sites or can lead to preferential corrosion attacks [19]. According to Silva et al. [20], the electrochemical behavior of composites with hard ceramic reinforcements under sliding condition can exhibit pronounced deviations from the traditional behavior of the passive metals. For instance, Toptan et al. [21] studied the tribocorrosion behavior of Ti reinforced with B_4C in 9 g L^{-1} NaCl solution. The authors inferred that under static conditions, the composites showed localized corrosion on pore sites at areas where the reinforcements agglomerated. However, under sliding, the composites exhibited lower susceptibility to corrosion and lower corrosion kinetics, owing to the load-supporting responsibilities of the B_4C reinforcements and from the decreased localized corrosion after wear debris compacted on the pores. Fang et al. [22] also reported that the increasing amount of Al_2O_3 reinforcements in 6061 Al alloy matrix was detrimental to the corrosion resistance of the composites but had positive effects on wear corrosion. The authors asserted that with increasing Al_2O_3 contents, the less the exposed fresh surfaces of the metallic matrix to the astringent 3.5% NaCl environment during sliding, hence resulting in more noble corrosion potentials.

Al–SiC is a reactive system; hence the fabrication route influences the tribological and mechanical properties through its effects on the matrix grain size, porosity, distribution of the reinforcing phase and the reinforcement–matrix interfacial properties. Additionally, there is limited research on the tribocorrosion of AMCs compared to studies on solely wear and corrosion. Toptan et al. [17] studied the tribocorrosion behavior of Al–Si–Cu–Mg alloy reinforced with B_4C (15 and 19 vol%) produced by squeeze casting in 0.05 M NaCl solution using the reciprocating ball-on-plate tribometer. Jamaati et al. [23] also investigated the tribocorrosion behavior of composites of Al–(0.48–3.55 vol%) Al_2O_3 fabricated by anodizing and accumulative roll bonding (ARB) processes by using a ball-on-plate tribometer in 1 wt% NaCl solution. Furthermore, the tribocorrosion behavior of functionally graded aluminum matrix composites (FGAMCs) with SiC_p reinforcements has also been studied [16, 18, 24].

Therefore, the present study aims to add knowledge to the tribocorrosion behavior of AMCs. The study evaluates the impact of large amounts SiC nano-sized additions on the tribocorrosion behavior of Al–SiC nanocomposites produced using the solid-state process of high energy mechanical milling and spark plasma sintering (SPS) in 3.5 wt% NaCl solution. From our previous study [25], the hardness and corrosion properties of the composites improved with increasing SiC additions. In the present study, the tribocorrosion performances of the composites also improve with increasing SiC contents. The composites form adherent tribolayers, inert and hard enough to resist the synergistic effects of wear and corrosion. Thus, Al–SiC nanocomposites in the present study offer beneficial marine application properties when used for components subject to tribocorrosion such as engine components of high-speed boats and marine diesel engines [26].

2 Materials and methods

Details on the synthesizing of the Al–SiC nanocomposites, their characterization and corrosion behavior are shown in our previous study [25].

For the tribocorrosion tests in the present study, the sintered Al–SiC nanocomposites were mounted, ground and polished to $1\text{ }\mu\text{m}$ mirror-like surface finish on one face. Although in real applications a mirror-like surface finish is not present, a mirror-polished finish before tribocorrosion testing in the laboratory is important because the samples' surfaces need to be flat, smooth and scratch-free. A smooth, clean and scratch-free surface significantly reduce the number of corrosion initiation sites on the composites' surface, which help to understand better and characterize the behaviors of the composites. With the face being the working surface and facing upward, samples were mounted on a ball-on-disc tribometer (Anton Paar tribometer with software Instrum X 7.3.13).

A continuous unidirectional sliding test was used which included a stainless steel 316L counter body of 6 mm diameter mounted vertically on a rotating head, above the sample. A mechanical load of 5 N was applied with a rotating speed of 92 rpm (sliding speed of 2.1 cm s^{-1}). The applied load on the ball drew a circular wear track of radius 2.25 mm on the working surface of each sample. The test was carried out at room temperature (about $25 \pm 1\text{ }^\circ\text{C}$) and humidity of 50%. 3.5 wt% NaCl solution was used as the corrosive environment per ASTM G44 to show better performance of the composites. The 3.5 wt% NaCl solution is the typical salt concentration in seawater and is the most common and abundant corrosive agent on earth. For each run, the composites were immersed in a volume of 5 ml of the NaCl solution. Tribo-electrochemical measurements

were acquired using the three-electrode set-up, where the reference electrode was Ag/AgCl, the counter electrode was graphite, and the various sintered composites were the working electrode. The electrodes were connected to a VersaSTAT 4 potentiostat and linked to a PC with Versastudio software. To study the electrochemical (active or passive) state of the composites, open circuit potential (OCP) measurements were carried out.

The duration of the OCP or chronopotentiometry measurement was 3600 s for each run, which included 1800 s of rubbing. Figure 1 shows the tribocorrosion tests setup and scheme showing the position of the sample, counter body and electrodes.

Characterization of samples after tribocorrosion testing was done using the Zeiss Axio observer optical microscope and the Jeol JSM-7200F scanning electron microscope.

3 Results and discussions

Tribocorrosion, as defined by Landolt et al. [27], is when a material deteriorates permanently as a result of combined effects of mechanical, electrochemical and chemical processes taking place on a material surface in the presence of a corrosive environment. A general overview of the results suggests a stable passivation period, depassivation and repassivation processes and a return to the initial steady

passivation potentials sum up the surface states of the composites, as shown in Fig. 2.

3.1 Passivation period

The formation of stable passive films characterizes the first 900 s of the chronopotentiometry measurements, as shown in Fig. 2. The passive films are products of the chemical interactions occurring between the Al alloy matrix and the corrosive NaCl environment. Innately, Al alloys are known to form protective films during corrosion [28] with respect to the reactions [29]:

where M denotes the matrix Al alloy. SiC as a hard ceramic reinforcement is recognized to be chemically inert [30]. Depending on the fabrication route and SiC quantities, the addition of SiC in some studies has been suggested to be detrimental to corrosion resistance [19, 31]. However, from our previous study [25], the corrosion resistance of the present composites improved with increasing SiC additions. This was attributed to the solid-state processing route, SPS, in which no deleterious anodic phases such as Al_4SiC_4 or Al_4C_3 formed. The formation of such anodic phases at the interface of the Al alloy matrix and SiC reinforcements favor preferentially localized corrosion attacks [32].

3.2 Depassivation and repassivation period

At the onset of sliding, the unreinforced alloy and composites exhibit an instantaneous shift to the cathodic region (lower potentials) of the curve followed by a shift to the anodic region (noble potentials) as seen in Fig. 2. From Fig. 2a the chronopotentiometry value of the unreinforced alloy fluctuates between -0.685 and -0.69 V during sliding for 1800 s. During unloading, the potential drops to -0.70 V before “recovering” to its initial steady potential of about -0.695 V. Figure 2b–f show clearly the immediate anodic shift in chronopotentiometry value for the composites compared to that seen in the unreinforced Al alloy as sliding begins. The height of the shift correlates to the quick recovery or restoration of the formed films on the composites.

The potential shift to the cathodic region indicates an altering in the electrochemical surface states of the composites and is evidence of the removal of protective films on passive metals and alloys [33–35]. During sliding, the exposed new surfaces in contact with the NaCl solution become active and are subject to corrosion. This is because a galvanic cell develops between

Fig. 1 **a** The tribocorrosion setup, **b** scheme showing the position of the sample, counter body and electrodes

Fig. 2 Chronopotentiometry behavior of **a** Al alloy, **b** 15 wt%, **c** 20 wt%, **d** 25 wt%, **e** 30 wt% and **f** 40 wt% SiC reinforced Al composites respectively

the worn (active) and unworn (passive) surfaces. The galvanic coupling promotes anodic dissolution in the worn regions due to the large area ratio of the passive surface to the active surface. Thus, as illustrated in Fig. 3, the worn or exposed new area shows a dark contrast compared to the unworn surface. Conversely, the shift of

chronopotentiometry values to more positive potentials indicate the occurrence of repassivation or re-adsorption in the worn areas by cathodic reactions involving the reduction of dissolved oxygen as summarized by the reaction [29]:

Fig. 3 Optical image of the worn surface of Al-40 wt% SiC in 3.5 wt% NaCl solution

As sliding continues, competing activities due to the repassivation rate and the mechanical depassivation at the contact area of the composites manifest as potential fluctuations, as noticed in Fig. 2. The potentials measured under sliding is a mixed potential resulting from the galvanic coupling of materials found in the worn and unworn areas [27, 36, 37]. Similar chronopotentiometry trends have been reported in many studies in which the studied materials are well known to form passive protective films during corrosion [38, 39].

Furthermore, chronopotentiometry values recorded during sliding for the composites are nobler compared to their initial stable potentials. This is because the increasing SiC contents in the composites tend to reduce the newly exposed surfaces of the Al matrix to corrosion by the 3.5 wt% NaCl solution [22].

3.3 Recovery and restoration period

The potential of the unreinforced Al alloy and the nanocomposites decrease sharply to their initial potential after sliding and removal of the applied load (Fig. 2). Within the shortest time, the stable potential behavior similar to that before sliding manifests, signifying repassivation occurring in the active area and restoration of the wear scars [27].

3.4 Coefficient of friction (COF)

Figure 4 shows the variation in COF during sliding under 5 N load for the unreinforced Al alloy and the Al-SiC

Fig. 4 Coefficient of friction of all composites under open circuit potential during the sliding period in 3.5 wt% NaCl solution

nanocomposites. The average COF of the unreinforced Al alloy is 0.053, whereas those for Al-SiC nanocomposites range from 0.291 to 0.505. The COF for the Al-SiC composites increases from 0 to as high as 0.633 for the composite with 40 wt% SiC. The increase in COF of the Al-SiC nanocomposites compared to the Al alloy may stem from the interaction between the counter body and the hard SiC ceramic reinforcement protruding during sliding. Furthermore, the presence of hard and oxidized wear debris freely moving at the sliding region culminating in third-body wear [40], can also account for the increase in COF.

The lack of smooth sliding due to the presence of reinforcing particles cause staggered motion with particle pull out being the main determinant of the actual wear rate. The direct contact of the stainless steel counter body minimizes with increased amounts of SiC nano-particles in the softer matrix phase, which causes particle pull out to decrease in the composites. The hard SiC particles counteract with the stainless steel surface, and an increase in COF is observed because of these hard particles. It has been reported that AMCs exhibit higher wear resistance than the unreinforced alloy if fracture, fragmentation, and pull out of particles are minimized or avoided [41].

Additionally, the formation of mechanically mixed layers (MML) or tribolayers on the worn surfaces from the transfer of materials from the stainless steel counter body and composites influence variations in COF values as observed in Fig. 4.

In previous studies on Al-SiC_p functionally graded Al matrix composites (FGAMCs) [16, 24], the authors concluded that the role of the SiC reinforcements served not only as load-bearing constituents but also promoted the formation and stability of adherent tribolayers leading to

the improvement of wear resistance of the composites. Also, it is well known in Al–SiC composites, that the wear rate of the Al alloy matrix depends on the formation rate of the adherent tribolayer. Hence, with the increasing rate of formation of the tribolayer, the Al matrix is protected and its degradation by wear decreases [42]. Furthermore, wear-accelerated effects of the NaCl solution influence the tribochemistry on the wear processes. The NaCl solution influences the development and removal of chemical reaction products in the form of debris in the worn areas, which could affect COF.

3.5 Worn surface morphology and composition analysis

Figure 5 shows the SEM micrographs of the worn areas of the unreinforced Al alloy and composites after the tribo-corrosion tests. From Fig. 5a the integrity of the formed passive layers in the worn areas of the unreinforced Al alloy is observed to be severely fractured and less resistant to the mechanical contact from the stainless steel counter

body during sliding. This results in material removal or delamination in certain areas, as shown in Fig. 5b and accounts for the fluctuations observed in both chronopotentiometry values and COF. Additionally, the direction of fracturing is mostly parallel to the direction of sliding. For the Al–SiC nanocomposites, exemplified by Fig. 5c, d, the worn surfaces are smooth. SiC is well known to show a smooth abraded surface [18]; thus the surface smoothness character of the nanocomposites indicate the load-bearing protective role of the nano SiC reinforcements which tend to resist the applied mechanical action and reduce the amount of plastic deformation.

This feature is consistent with some previous studies [31, 43]. Feng et al. [44] reported that the presence of ceramic reinforcing phases in MMCs enhance their corrosion resistance compared to the matrix metal by functioning as an inert physical barrier on the formed passive films. Although the formed film exhibit cracks, which also are responsible for chronopotentiometry and COF fluctuations, the film shows no delamination. This demonstrates that the formed tribolayers in the nanocomposite are

Fig. 5 SEM SE and BSE micrographs showing the wear track of **a**, **b** the unreinforced Al alloy, **c** Al-25 wt% SiC and **d** Al-30 wt% SiC

Fig. 6 SEM-EDX spot analysis of wear track of Al-25 wt% SiC

adherent, hard enough and resistant to the mechanical contact. The resistance of the tribolayers to mechanical contact can be correlated to the composites' respective hardness properties. Consistent with our previous study, the hardness of the composites improved with increasing SiC amounts compared to the unreinforced Al alloy [25].

SEM-EDX spot compositional analyses (Fig. 6) shed further insight into the formed, modified tribolayers in the worn areas of the nanocomposites. The light grey contrast areas (spot 3) comprising the tribolayer are mainly of Al, Si, C and O. The rough dark grey regions (spot 2) are rich in Al, O and C. On the other hand, the white products seen in the composites and unreinforced Al alloy are predominant of Fe. These products are material transfer from the stainless steel counter body resulting from wear and corrosion, which are mechanically alloyed onto the tribolayer during sliding [8]. However, even though the stainless steel 316L counter body slightly corrodes in the NaCl solution, this does not compromise the forces it exerts on the samples. This exerted force is a critical component of the wear tests.

Table 1 shows the elemental quantification from the SEM-EDX spot analysis.

In summary, the present work shows the incorporation of high amounts of SiC in AMCs produced using the solid-state processing route such as SPS has beneficial effects. The study indicates the increase in load-bearing capacity of the Al alloy as a result of the addition of the hard ceramic SiC reinforcing phase. Consequently,

the Al-SiC nanocomposites exhibit an increase in wear resistance. Additionally, the SiC reinforcing phase favors the formation of tribolayers from the transfer of materials from the counter body and protruding SiC phases at the worn areas of the composites. The tribolayers are inert, adherent and hard enough to resist the synergistic effects of wear and corrosion. All of the above observations in this study are consistent with previously published studies involving FGAMCs or cast AMCs with SiC [16, 22, 24, 42].

For future studies, an inert counter body will be considered to avoid any contaminations or deterioration of the counter body during testing.

4 Conclusions

The present study is a follow-up study [25] evaluating the tribocorrosion behavior of Al-SiC nanocomposites with high amounts of SiC reinforcements synthesized using high energy mechanical ball milling and spark plasma sintering (SPS).

The fluctuations in chronopotentiometry values characterize the in situ tribocorrosion behaviors of the Al-SiC nanocomposites in 3.5 wt% NaCl aqueous solution. A shift of chronopotentiometry values to the cathodic region of the curve indicates the removal of formed passive layers and exposure of the new underlying surfaces to the aggressive external environment. The presence of a high-volume fraction of SiC in the composite and wear debris from the counter body form modified tribolayers in the worn surface. The modified tribolayers are adherent and hard enough to resist degradation by the synergistic effects of wear and corrosion. In all, the collective action of the SiC in increasing the load-bearing capacity of the composites and the formation of an inert physical barrier on the passive film in the worn areas of the composite, translate to the increase in wear resistance and improved tribocorrosion performance of the composites.

Table 1 Elemental quantification of SEM-EDX spot analysis (in at%)

Spot	C	O	Na	Al	Si	Cl	Fe
1	17.52	16.21	0.56	10.68	1.17	0.15	53.7
2	11.65	49.61	0.3	29.02	6.28	0.82	2.32
3	31.8	37.83	0.05	15.11	11.83	0.83	2.55

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Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interest to disclose.

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