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Mechanical and microstructural characterization of Ti-SiC reinforced AA5083 surface composites fabricated via friction stir process

Md Ziyaur Rahman1, Zahid A Khan1, Arshad Noor Siddiquee3, Mustufa Haider Abidi1, Mohamed K Aboudai1 and Abdulrahman Al-Ahmari2

1 Department of Mechanical Engineering, Faculty of Engineering and Technology, Jamia Millia Islamia, New Delhi-110025, India
2 Raytheon Chair for Systems Engineering, Advanced Manufacturing Institute, King Saud University, Riyadh-11421, Saudi Arabia
3 Industrial Engineering Department, College of Engineering, King Saud University, Riyadh-11421, Saudi Arabia

E-mail: mabidi@ksu.edu.sa

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Abstract

A mixture of Titanium and silicon-carbide powders was embedded in the AA5083 matrix by Friction Stir Processing (FSP). Experiments were performed as per Taguchi L8 orthogonal array, and the effect of reinforcement on hot strength (at 100 °C), processed zone (PZ) geometry, and microstructure were investigated. The effect of PZ geometry on the surface properties was also analyzed. The effect of heating the tensile test specimens to 540 °C on the strength at 100 °C was also separately investigated. It was observed that surface hardness was significantly enhanced by FSP, and the highest mean hardness of 90.4 HV was observed. Furthermore, it was observed that the surface properties also significantly depend on PZ geometry. From experimental results, it was found that the specimens with the lowest width to depth ratio bears the highest hardness and vice versa. A clear effect of parameters was evident on the geometry of processed zones with a deep bowl, and shallow cup-shaped zones were formed with smaller and larger shoulder diameters, respectively. The samples were processed at 355 rpm, 63 mm min⁻¹, 17 mm shoulder, and 355 rpm, 80 mm min⁻¹. The 20 mm shoulder showed high tensile strength 292 MPa and 294 Mpa, respectively. The strength of these samples did not reduce much even after heating to 540 °C.

1. Introduction

The increasing demand for lightweight and cost-effective design in aeronautical, space, and shipbuilding applications has led to the development of materials having high strength to weight ratio and high reliability [1–3]. However, Al alloys have a limitation of lowering of strength with the rise in temperature [4]. To encounter such challenges, one of the several alternatives to enhance Al alloys’ surface and bulk properties is to incorporate hard components/particles into the base alloy via metal matrix composites (MMC). The carbides, oxides, and nitrides of elements such as tungsten, silicon, titanium and boron, etc., hard particles are common reinforcements. A number of techniques, including casting are used to incorporate hard components into the base to enhance surface/bulk properties. However, the fusion routes have various limitations, such as segregation of micro phases and creation of micro phases during processing, and a number of solidification defects [5]. The techniques such as powder metallurgy proved to be helpful to some extent, but the non-uniform distribution of reinforcement during processing limits its applicability [6]. To address these challenges, a recent Friction Stir Processing (FSP) technique which is based on the principle of Friction Stir Welding (FSW), has proved to be very effective. FSP is also considered as an advanced technique to tailor the surface properties of base materials [7–10]. The schematic of FSP is shown in figure 1(a). The reinforcement in powder form is generally introduced by filling it in the groove and subsequently packing it with the aid of pinless tool to confine the reinforcement within the processed region during processing. With the optimized process parameters, the final FSP is performed with the aid of a FSP tool. The stirring action of the tool mixes the materials and also
subjects them to severe plastic deformation (SPD). The stirring action distributes the reinforcement uniformly and embeds it into the matrix within the processed zone.

FSP as a thermomechanical process involving the SPD and very high strain rates (HSR) at elevated temperatures giving rise to distinct zones. FSPed specimens are characterized by four distinct zones: stir zone (SZ), thermos-mechanically affected zone (TMAZ), heat affected zone (HAZ), and base material (BM) as shown in figure 1(b). The SZ undergoes SPD and dynamic recrystallization (DRX) and is characterized by fine equiaxed grains. On the other hand, in TMAZ, the grains are relatively large oriented in the stirring direction owing to partial plastic deformation of the material in this region. The HAZ surrounding the TMAZ is characterized by coarsened grains more or less similar to BM due to high thermal cycles encountered during FSP with no plastic deformation.

Shaik et al\(^\text{[11]}\) investigated the effect of reinforcement (carbides of boron, tungsten, and silicon) on wear resistance of AA6061 using FSP, and it was observed that boron carbide (B\(_4\)C) reinforced specimen exhibited the better wear resistance than tungsten carbide and silicon carbide reinforced specimens. Srivastava et al\(^\text{[12]}\) studied the effect of silicon carbide reinforcement on surface properties of AA5059 using FSP, and it was claimed that by incorporating the silicon carbide powder, the surface hardness (HV) was enhanced by 55% as compared to base alloy. Vignesh et al\(^\text{[13]}\) studied the effect of FSP parameters on the wear behavior of AA5083 and reported that friction stir processed (FSPed) specimens possessed higher wear resistance than the base alloy. Shahraki et al\(^\text{[14]}\) investigated the effect of ZrO\(_2\) reinforcement on mechanical properties of AA5083 and it was claimed that FSPed specimens possessed improved hardness and tensile strength.

A large number of studies have been performed on surface property improvement in a number of heat-treatable and non-heat-treatable aluminum alloys (although mostly non-heat-treatable) by incorporating different reinforcements using FSP. Most investigations utilize ceramics and hybrid (mixture of ceramics) as reinforcement in powder form. However, very few studies also reported that the use of metal powders mixed with ceramics particles might provide added benefits due to mechanical alloying and \textit{in situ} formation of intermetallic compound (IMC) hard-phase \cite{15, 16}. Keeping in view a stronger among the non-heat-treatable AA5083, which finds emerging applications in the aerospace application (Boeing commercial airplanes) \cite{17},

\begin{figure}[h]
\centering
\includegraphics[width=0.8\textwidth]{figure1.png}
\caption{(a) The principle of FSP; (b) The macrostructure of transverse cross section of FSPed specimen.}
\end{figure}
was reinforced with Ti and SiC by FSP as a maiden investigation on post FSP properties AA5083 reinforced surface composite.

In this study, L8 Taguchi orthogonal array (L8 OA) was used to perform experiments and to study the effect of FSP parameters on microstructure and bead geometry of Ti + SiC reinforced surface properties produced by FSP. Along with tensile strength, the fractographic examination of the hot tensile strength (at 100 °C) of the FSPed specimens was also investigated. The present work was performed by selecting two levels each of tool rotational speed, traverse speed, and shoulder diameter (refer to table 1) in order to fully explore the effect of these process parameters on the surface properties of AA5083.

### 2. Materials and methods

The AA5083 plates (nominal composition as 4.37Mg-0.5Mn-0.16Fe-0.08Si-0.07Cr-0.031Cu-0.013Ti-0.002Zn-Rest Aluminum.) of dimensions 200 mm × 70 mm × 7 mm were used for surface composite fabrication. The ultimate tensile strength (UTS), percentage elongation, and hardness of the AA5083 alloys are 317 MPa, 16%, and 80 HV, respectively. Rectangular grooves of cross-section 2.5 mm × 2 mm were machined at the middle of the plate. The grooves were packed with the reinforcement comprising of mixture of SiC (particle size of 400 μm) and Ti (particle size of 70 nm) in the ratio of 3:10 by weight. The ratio was established through pilot experiment and the ratio of 3:10 gave distribution without agglomeration. Before packing, the reinforcement mix was blended at 50 rpm for 4 h. While developing Ni particles reinforced 5083 Al composite using FSP, it was reported that tools with distinctive threads provide better distribution of particles than the plain tool [18]. While reinforcing B4C particles in AA7075 using FSP, it was reported that in comparison to samples processed with the cone pin, those treated with the pinless tool showed a more homogenous distribution of reinforcement in the outer layer of the material [19]. The slots were packed with the reinforcement comprising of a mixture of SiC (particle size of 400 μm) and Ti (particle size of 70 nm) in the ratio of 3:10. The slots packed with powder were covered by using pinless FSP tool (refer to figure 2(a)). After packing, the FSP runs were performed on a robust vertical milling machine adapted to perform FSP, by using H-13 steel tool having cylindrical pin with right-hand threads having a Pitch of 0.8 mm (refer figure 2(b)–(c), by using L8 OA. (refer to table 2).

The various test coupons were machined by using wire electric discharging machine (WEDM) across transverse section from the FSPed samples. The specimen for microstructural investigation were prepared by cold mounting procedure and subsequently progressively ground on water resistant abrasive paper of 100, 200, 400, 600, 800, 1000, 1200 and 1500 grit. The finical polishing was done by diamond polishing. The polished samples were finally chemically etched using 12 ml HCl, 16 ml HNO3p, 1 ml HF, 34 ml distilled water and 4.8 grams of CrO3. The microstructure of specimens was revealed using optical microscopy (OM) and scanning electron microscopy (SEM). The micro-hardness of specimens was measured with the aid of Vickers micro-hardness tester (Micro WhizHard, Mitutoyo, Japan) at a regular spacing of 0.5 mm under a load of 0.5 kgf for 15 s dwell time. The bead geometry expressed as width and depth of processed zones were measured using ImageJ software. The ultimate tensile strength (UTS) of FSPed specimens (refer to figure 3) was evaluated with the aid

![Figure 2.](image_url)
computer interfaced tensometer. To investigate the effect of heat treatment on UTS of BM and FSPed specimens, the tensile specimens were heat treatment to 540 °C.

3. Results and discussion

3.1. Effect of Ti-SiC on micro-hardness

To explore the effect of Ti-SiC reinforcement on hardness behavior of AA5083, the hardness values measured across the processed zone at a distance of 1 mm below the top surface were plotted against the distance from the weld center. Figures 4(a) and (b) show the variation of micro-hardness within the processed region measured across the weld cross-section. The hardness of the base alloy (AA5083) was obtained as 80 HV using Vickers micro-hardness tester. From figure 4, it can be observed that the FSPed zones have higher hardness than the base alloy. This hardness behavior is due to the combined effect of the thermomechanical effect of FSP (grain refinement) and Ti-SiC reinforcement leading to the positive effect on hardness [20]. The variation in the values
of micro-hardness on both AS and RS as shown in figure 4, is attributed to the nature of particle distribution in the processed zone due to FSP [21, 22]. However, the sharp peaks in hardness values in SZ of all the specimens may be attributed to the combined effect of hardness of reinforcement particles, new phase formed, SPD and DRX. The mean micro-hardness values were also calculated for each FSPed specimen. The maximum and minimum average hardness values were obtained in S5 (90.4 HV) and S8 (82.4 HV), respectively. These observations infer that micro-hardness at tool rotation speed of 355 rpm and tool traverse speed 63 mm min\(^{-1}\) are better than those observed at tool rotational speed of 450 rpm and tool traverse speed of 80 mm min\(^{-1}\). This may be due to the fact that optimal combination of tool rotational speed and welding speed leads to more uniform distribution of particles during FSP [23]. In S5, the peak value of hardness (115.2 HV) was obtained in SZ on RS whereas the lowest value of hardness (82.6 HV) was observed in SZ on AS. In the case of S8, the peak value of hardness (87.7 HV) was observed on AS whereas the lowest hardness value (64.4 HV) in SZ on RS.

### 3.2. Macro and micro-structural characterization

Figure 5 shows the macrostructure of the transverse cross-section of S5 and S8 FSPed specimens bearing the maximum and minimum micro-hardness obtained at different combinations of process parameters (refer to table 2) of the variation in the properties of the material in various zones bears the characteristic effect of tool geometry and optimal combination of process parameters such as tool rotational speed, tool traverse speed, tool tilt angle etc. [24]. During FSP, as the tool advances, the rotational action of the tool moves the plasticized material along with reinforcement particles from AS to RS of the processed region (refer to figure 5). As can be observed from figure 5 that there is a more or less uniform distribution of reinforcement particles on AS whereas a significant agglomeration of these particles can be noticed on RS. The SZ is wider near the shoulder and undergoes a higher degree of SPD, complex material mixing, higher strain rates and extreme frictional heat due to its direct interface with the shoulder. This is evident from figure 5 that reinforcement particles within the SZ occur near to top processed surface. The SPD, higher strains, and complex material flow cause effective embedment of Ti-SiC particles within the metal matrix resulting in hybrid MMCs. The hybrid MMCs bear the properties of both Ti and SiC particles leading to the overall improvement in surface properties.

The optical microscopic (OM) images of the S5 and S8 FSPed specimens are illustrated in figure 6 and figure 7, respectively. It can be observed that SZ consists of fine and equiaxed grains (refer figure 6(b) and figure 7(b)) is attributed to DRX whereas the grains in TMAZ are deformed and elongated oriented in the direction of material flow (refer figures 6(a), (c) and figures 7(a), (c)). The interface between the SZ and TMAZ on AS is more distinguishable than RS due to more heat generation and higher strain rates on AS. The degree of homogenous/heterogeneous distribution and severity of agglomeration of reinforcement particles within the processed zone during FSP is dependent on tool geometry, nature of particles, matrix chemistry and process parameters [25, 26]. Defect free cross-section of FSPed specimens were obtained in both S5 and S8 specimens. In S5, more or less uniform distribution of particles in SZ can be observed leading to better hardness distribution (refer figure 6(b)). In case of S8, defect free processed region is obtained, however a significant agglomeration of
the Ti-SiC powder occurs (refer figure 7(b)). In addition, it can be observed from figure 7(b), that the particles are heterogeneously distributed in SZ. The occurrence of agglomeration and heterogeneous distribution of Ti-SiC particles leads to overall reduction in hardness in the processed region [27].

3.3. Scanning electron microscope (SEM) characterization
To analyze the behavior of microhardness evolution across the transverse cross-section of processed zones, scanning electron microscopic (SEM) characterization of S5 and S8 FSPed specimens were performed. In figure 8 and figure 9, the locations ‘a’, ‘b’ and ‘c’ corresponds to the regions in the processed zone where maximum, peak, and minimum hardness values where obtained, respectively. Figure 8(a) shows the
A homogenous distribution of Ti-SiC particles corresponds to high hardness values, whereas figure 8(c) shows the agglomeration with poor bonding of reinforcement with the matrix, which corresponds to lower hardness values. The SPD coupled with DRX and homogenous distribution of reinforcement particles in SZ (refer figure 7(b)) peaks of hardness [28]. Therefore, it can be observed that hardness depends on the distribution of the reinforcement particles in the processed region. It can be observed from figure 9(a) that Ti-SiC particles are uniformly distributed (but somewhat to a lesser extent than that observed in S5), leading to high hardness. On the other hand, the agglomeration of reinforcement particles, as shown in figure 9(c), corresponds to the lowest hardness values. In contrast (refer figure 9(b)) the uniform distribution of Ti-SiC particles in SZ of the processed region results in sharp peaks of hardness. It can be observed from figure 8 and figure 9 that more uniform distribution of Ti-SiC particles in S5 than S8 has led to overall higher surface hardness in S5.
3.4. Effect of bead geometry on surface properties

Figure 10 shows the bead geometry of eight FSPed specimens obtained at different combinations of process parameters. The bead geometry is expressed in terms of the depth and width of the processed zone. The bead geometry depends on the tool geometry and process parameters such as tool rotation, tool traverse speed, tool tilt, etc. [29]. It can be observed from figure 10 that two different bead shapes (bowl which is deeper and cup...
which is shallower) are formed. Table 3 illustrates the bead shape parameters, width, and depth of the SZ. Figure 11 shows the relation between the average hardness and width/depth ratio obtained for eight FSPed specimens. FSPed specimens S1, S2, S3, and S4 fabricated by shoulder size of 17 mm whereas specimens S5, S6, S7, and S8 were fabricated by shoulder size of 20 mm. From figure 11, it can be observed that specimens having the lowest weight to depth ratio bear the highest average hardness, whereas the specimens bearing the highest hardness values.

Figure 9. The SEM images of S8 specimen corresponding to the locations of (a) high hardness, (b) peak hardness, and (c) lowest hardness values.
width to depth ratio have the lowest hardness. However, it can be illustrated from figure 11 that the width to depth ratio also depends on the shoulder diameter. For instance, it can be observed that S3 bears the lowest width to depth ratio has high hardness, whereas specimen S1 having the highest width to depth ratio has the lowest hardness. Both S1 and S3 were fabricated by shoulder size of 17 mm. On the other hand, specimens S5 and S8 fabricated under shoulder size of 20 mm having the lowest and highest width to depth ratio bears the highest and lowest hardness values, respectively. So, it can be inferred from results that the specimens bearing the lowest width to depth ratio have the highest hardness and vice versa.

![Figure 10. The bead geometry of eight FSPed specimens fabricated at different combinations of process parameters.](image)

![Figure 11. The variation of mean hardness with width to depth ratio obtained for eight FSPed specimens.](image)

| Sample No. | Depth (mm) | Width (mm) | Area (mm²) | Width/depth | Hardness (HV) |
|------------|------------|------------|------------|-------------|---------------|
| S1         | 5.26       | 16.45      | 47.32      | 3.126188    | 84.7          |
| S2         | 5.62       | 16.69      | 46.3       | 2.971305    | 87.3          |
| S3         | 5.52       | 16.71      | 44.93      | 2.876543    | 89            |
| S4         | 5.58       | 16.72      | 44.87      | 2.995520    | 85.8          |
| S5         | 5.9        | 19.4       | 57.30      | 3.288136    | 90.4          |
| S6         | 5.69       | 19.25      | 50.47      | 3.382425    | 88.6          |
| S7         | 5.65       | 19.69      | 52.62      | 3.484425    | 83.6          |
| S8         | 5.25       | 19.87      | 47.66      | 3.782600    | 82.4          |
3.5. X-Ray diffraction (XRD) results

As shown in figure 12, the XRD pattern for samples with highest strength (experiment 6) shows that the peaks corresponding to aluminum plans (111), (200), (220), (311), and (222). The FCC phase continues due to the higher concentration of Al in SZ which suggests that poor mixing of reinforcement could not take effect. The lattice parameter and lattice strain were calculated as 4.05 Å and 8.77 × 10⁻⁵. Furthermore, the evolution of new phases and the intermetallic compounds was not observed.

3.6. Effect of heat treatment on tensile strength

To study the effect of reinforcement and heat treatment on mechanical properties, the tensile testing of BM and FSPed specimens was performed. The tensile testing of specimens was performed in two different conditions; in the first case, the tensile test was performed on BM and FSPed specimens at 100 °C (the strength so obtained is specified as UTS100), whereas in the second case, the specimens were heated to 540 °C (the strength so obtained is specified as UTS540) and were held at that temperature for 20 min, subsequently cooled in the air to 100 °C and the tensile tests were performed at this temperature (100 °C). Figure 13 shows the tensile test results. It can be observed that there is an appreciable decrease in the UTS of BM and FSPed specimens, which were heated to 540 °C. This behavior may be attributed to the coarsening of grains and of β-phase (Al3Mg2) due to high temperature to which the tensile samples were heated [30]. In addition, a complete dissolution of β-phase at high temperatures and subsequent nucleation of sub-micron β-phases during cooling in air results causes lower tensile strength [31]. The higher values of UTS for samples that were not heated prior to the test at 100 °C are due to smaller grain size [32].

Figure 14(a) and figure 14(b) show the fractured tensile specimens heated to 100 °C and 540 °C, respectively. As shown in figure 14, all the specimens fractured within the central processed zone, which may be attributed to the agglomeration or high density with lack to interfacial bonding of hard reinforcement particles with matrix within this zone. In both heating conditions, the highest UTS values were observed in S6 and the lowest UTS values were obtained in S4 (refer figure 13). The fractographic analysis of specimens bearing the lowest and
The highest tensile strength can be helpful in analyzing the effect of reinforcement and heat treatment on FSPed specimens (refer to figure 15). The fractured surface of S4 (refer to figure 15) shows the small and large ridges indicating the brittle mode fracture. The formation of large deformation zones corresponds to the lowest strength [33, 34]. On the other hand, the fractured surface of S6 (figure 15(b)) shows the cleavage fracture and quasi cleavage dimples indicating both brittle and ductile modes failure and hence the highest tensile strength. Figures 15(c)–(d) illustrates the fractography of specimens that were heated to 540 °C. Figure 15(c) shows the large ridges’ brittle mode of fracture bearing the lower UTS, whereas in the case of S6 (figure 14(d)), both brittle and ductile modes of failure indicate higher UTS.

3.7. Bending test results
In this experimental study, the three point bending test was carried out as per the ASTM standard (E290-97a). The bending test of all the eight specimens was performed on the face side of the joint. The bending test results indicated that only specimen S1 and specimen S6 has passed the bending test while the other specimens has failed in the stir zone which is in line with the behaviour observed in tensile analysis. The crack were initiated at the center of the processed zone and extended to the bottom of the joint. However, it was observed that the failure of specimens was obtained at different bend angles, which may be correlated with the distribution or agglomeration of hard reinforced particles within the processed region.
4. Conclusions

To analyze the effect of Ti SiC reinforcement on metallographic and mechanical properties of base alloy (AA5083), FSP was performed at several combinations of process parameters as per Taguchi L8 orthogonal design. From the critical examination of results of microhardness, microstructure, SEM, and bead geometry, the following important conclusions are drawn:

- Microhardness examination of FSPed specimens shows a significant improvement in hardness by reinforcement of Ti-SiC powder. The maximum and minimum average hardness was observed in S5 and S8 specimens, respectively.
- The macrostructural investigation of FSPed specimens reveals the defect-free processed region.
- The microstructural examination demonstrates the significant grain refinement in SZ and; diffused and elongated grains in TMAZ. In addition, the microstructural results indicate the uniform distribution of Ti-SiC particles in the S5 specimen, whereas agglomerations of these particles were observed in the S8 FSPed specimen.
- XRD results indicated that no new phases were evolved.
- The SEM images of processed zones demonstrate denser packing of Ti-SiC precipitates in S5 specimen and relatively less dense packing of these particles in S8 specimen.
- The bead geometry of processed specimens indicates the lower values of width to depth ratio in S5 having a high average hardness, whereas the higher value of width to depth ratio was obtained in S8 having the lowest average hardness.
- The lowest and highest tensile strength of FSPed specimens was obtained in S4 and S6 specimens, respectively. The UTS was found to decrease with heat treatment.
- The bending test results show that all the specimens fractured in the SZ of the processed region during bending except the specimens S1 and S6.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

ORCID iDs

Arshad Noor Siddiquee @ https://orcid.org/0000-0002-3573-8385
Mustafa Haider Abidi @ https://orcid.org/0000-0001-5609-5705

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