An Investigation on The Mechanical Properties of FSP Processed Silicon Nitride Reinforced Aluminum Composite

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https://doi.org/10.5109/7151680

出版情報：Evergreen. 10 (3), pp.1330-1340, 2023-09. 九州大学グリーンテクノロジー研究教育センター
バージョン：

An Investigation on The Mechanical Properties of FSP Processed Silicon Nitride Reinforced Aluminum Composite

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(Received July 30, 2022; Revised April 18, 2023; accepted July 4, 2023).

Abstract: In the present investigation, friction stir solid state process has been used to develop a functionally graded composite material reinforced with Si_3N_4 (Silicon nitride). The main aim of the current investigation is to develop a low density high strength silica reinforced aluminum based composite material develop by friction stir processing (FSP) process for aerospace and defence applications. FGM (Functionally Graded Composite Material) has been prepared by taking two dissimilar aluminium plates with Si_3N_4 as reinforcement content through friction stir processing (FSP) technique. To evaluate the mechanical properties of developed composite material tensile testing, hardness testing, thermal expansion and corrosion test have been performed. The tensile testing revealed the tensile strength, yield strength and percentage elongation of the developed composite material. Further the hardness testing estimated the scratch resistance. Microstructures results of Si_3N_4 reinforced FGM composite showed the proper mixing of the ceramic particle in functionally graded materials (Al6063 and Al8011) at optimum FSP parameter. Effects of FSP parameters like Cutting fluid, Tool shape, Tool speed on tensile strength and hardness of FGM composite reinforced with Si_3N_4 ceramic particles were studied by using Taguchi's technique L9 Orthogonal Array. The experimental results revealed that the highest tensile Strength is observed as 133.05 N/mm^2 at 1460 rpm for square shaped tool with kerosene as lubricant. Further lower values of tensile Strength i.e., 73.65 N/mm^2 at 920 rpm and 78 N/mm^2 at 2260 rpm are observed. Tensile Strength was good for kerosene with square tool shape and least for triangular and circular tool shape. The hardness of fabricated composite was good for circular tool shape and low speed (i.e., 920 rpm) but continuously decreases when tool shape is different. Hardness is good for brine solution at any tool speed but least for kerosene with any tool speed. The hardness of FGM composite was good for brine solution with any tool shape.

Keywords: Functionally graded composite; tool shape; rotational tool speed; cutting fluid; corrosion resistance.

1. Introduction

Friction stir process or FSP is a process in which a rotating tool is fixed in the tool holder which moves into the workpiece in the required direction. FSP is quite a reliable method in order to develop or synthesize the composites by reinforcing the base material with various reinforcing agents[1-2]. In present investigation, Si_3N_4 has been used as reinforcement. The reason that makes FSP unique and novel is its ability to improve the mechanical and surface properties of the base material such as hardness[3], ductility[4], resistance to corrosion[5], toughness[6] etc. in such a manner that the bulk properties of the material does not gets altered. A concise notice on cutting tools and the drawback of their use is also given in order to understand the significance of wear rate in FSP of high melting temperature and hard surface alloys. The core processes are explored with recorded frameworks in the enhancing of friction stir treated composite coating. There are certain discrepancies in surface composite analysis through FSP, including the manufacture of error composites, the customization of microstructures, the production of robust and cost-effective instruments, and perception of enhancing pathways Significant examples are proposed for more investigation on successful surface composite manufacturing by FSP[7-8].

Fabrication of composite material by stir casting method[9-10] is one of the most traditional methods[11-12]. However, various defects such as blow holes[13], accumulation of reinforcement[14] have been observed in the developed composite material developed by stir casting a technique[15-16]. These defects form inside the composite during the solidification process. Friction stir process is the solid-state process. In this process, composite materials developed in the solid-state. Formation of porosity and blow holes in the solid-state technique is negligible[17)].

Nowadays, aluminium is used in various sectors for manufacturing different parts. Aluminium alloys are usually used in the aircraft industries and automobile sectors. Aircraft industries and automobile sectors prefer lightweight material with good strength[18)]. Aluminium alloys are light in weight but have lower mechanical characteristicslike tensile strength and hardness[19)]. There are various attempts performed to improve the mechanical characteristics of aluminium alloys by developing the composite material. In this study, an attempt has been made to prepare composite material by using friction stir processing method.

In the stir casting technique, most of the cases single matrix material is used with one or more than one reinforcement content. In the present study, matrix materials (Al6063 and Al8011) have been used as matrix material.

Manufacturing processes, in particular for structural ceramics, is evolving as an unavoidable necessity for the versatile integration of advanced ceramics. Exceedingly high ceramic toughness, though, renders traditional machining very challenging or even unlikely[20-21)]. The continued need to optimize mechanical properties has contributed to the creation of products. The creation of innovative novel composite materials. Admittedly, a novel term in composite alloys, high temperature alloys, classified as composites consisting of 5 and sometimes more primary components in equimolar proportions, has been introduced during the first twenty years of 21st century[22)]. After its conception, High entropy alloys (HEAs) have influenced the academic society's findings, opinions and ideas, whose assumptions vary from marginally skeptical to aggressively positive. Composite materials are traditionally produced utilizing specific methodologies such as lasers technologies, infrared laser ionization methodologies, spraying coating methodologies, compression molding, cutting, compression moulding, centrifugal casting, etc. by dissipating small or nano-sized reinforcing particles (RPs) m in a matrix material. Huang et al.[23)] used direct friction stirring process to fabricate the composite and investigated its Microstructure and surface mechanical property. Such limitations include a method of composite production and sheet properties conversion that can be done at temperatures underneath the base material melting temperature. The goal can be both enhancement and improvement[24-25)]. The construction and development challenges of composite materials and FGS. FSP is an ideal option for a method that has demonstrated characteristics for composite production and layer alteration[26)].

Through the literature review, it has been found that very few researchers used Si_3N_4 as reinforcement material with Al6063 and Al8011. Barmouz et al. [27)] has employed this method to process or improve the properties of copper-based alloys by reinforcing with some other materials, Shamsipur et al.[28)] used titanium and Ghasemi-Kahrizsangi and Kashani-Bozorg[29)] have further advanced the FSP technique by using steel as a base material. Mishra et al.[30)] had also reinforced the layer of Silicon Carbide (SiC) on Al 5083 to synthesize / fabricate the composite through FSP technique. A large number of Mg, Cu, Ti and steel-based composites have been developed so far but a very limited literature work based on FSP is available so far to develop a composite which uses unconventional reinforcing agents like Si_3N_4. Meng et al.[31)] and Kumar et al.[32)] studied the ongoing development regarding the regulation of intrinsic problems in friction stir welding (FSW). In this study, Al8011 and Al 6063 based composite material has been developed using Si_3N_4 as ceramic particles by friction stir processing that clearly justifies the novelty of the current work.

Now a day the demand of low density high strength material is too high in the field of medical[33)], aerospace[34)], automobile[35)] and defense[36)]. Further the FSP technique has been better than traditional stir casting process[37)]. This technique is free from different types of casting effect generated from stir casting process. The light weighted high strength component can be generated by using the Si_3N_4 reinforced aluminum based (Al 6063 and Al 8011) metal composite.

Aluminum 6063 are alloyed with magnesium and silicon. Further aluminum 6063 are easy to machine and have good welding properties but the strength of 6000 series is less than 2000 and 7000 series. The aluminum 8011 are alloyed with iron, magnesium silicon, copper etc. which are not covered by other series. In the current investigation two dissimilar grades of aluminum were used to fabricate FSP composite material. The objective of the study was to enhance the tensile strength, hardness and thermal properties of the developed composite materials.

2. Materials and Methodology

2.1 Matrix Materials

In this study, two matrix materials Al6063 (Si -0.4%, Mg – 0.7%, rest aluminum) and Al8011 (Si -0.6%, Fe – 0.7%, rest aluminum) have been chosen as matrix material in the development of composite. Al6063 is mainly used in architectural applications such as sign frames, roofs, door frames and window frames. Al8011 aluminium alloy is relatively hard to cast as compared to Al6063 aluminium alloy. Al8011 aluminium alloys are used in aerospace industries. The prepared composite can be used in aerospace and automobile industries.

2.2 Reinforcement Particles

In this investigation, Si_3N_4 was selected as reinforcement material (filler metal between plates) in the fabrication of functionally graded (FGM) composite material. Silicon nitrides (Si_3N_4) attribute an excellent combination of material properties[4]. It is nearly as light (density 3.21 g/cm^3) as silicon carbide (SiC). In the current scenario, Silicon nitrides (Si_3N_4) is a good replacement of silicon carbide (SiC) in the fabrication of composite material due to its lightweight and excellent thermal shock resistance.

2.3 Experimental Procedure

Friction stir process has been used to develop the Si_3N_4 reinforced Al6063 and Al8011composite material. Si_3N_4 ceramic particles were used as interfacial as shown in Figure 1. Three input parameters have been selected to obtain the better mechanical properties of composite material as shown in Table 1. Taguchi L9 has been employed to obtain the optimum FSP parameters. Table 2 shows the design matrix Table for experimenting to achieve better tensile strength and hardness. The tensile test was performed on the prepared specimen. The samples were prepared as per ASTM-E8-04 standard. Hardness test was performed using Rockwell hardness testing machine. Further the protocol used for the hardness test was IS:1757(Pt-1)-2014.Corrosion test was performed by keeping the test specimen in the 3.5% NaCl solution for 5 days.

Table 1. Input Parameters

Parameters	Level 1	Level 2	Level 3
Cutting fluid	Kerosene	Brine solution	Lubricant oil
Tool shape	Square	Triangular	Circular
Tool speed	920	1460	2260

Table 2. Experimental Design

S. No	Tool shape	Tool speed	Cutting fluid
1.	Square	920	Brine solution
2.	Circular	1460	Brine solution
3.	Triangular	2260	Brine solution
4.	Square	1460	Kerosene
5.	Circular	2260	Kerosene
6.	Triangular	1460	CO_2 Coolant
7.	Square	2260	CO_2 Coolant
8.	Circular	920	CO_2 Coolant
9.	Triangular	920	Kerosene

Figure 1 shows the layout diagram of FSP process to develop the composite material. The tool rotates in between the two aluminium plates filled with interfacial reinforcement particles. When the non-consumable tool rotates between the two aluminium plates, heat is generated between the two plates. Resulting material tends to make plastic deformation. Resulting interfacial material mixed with the matrix material in a solid-state.

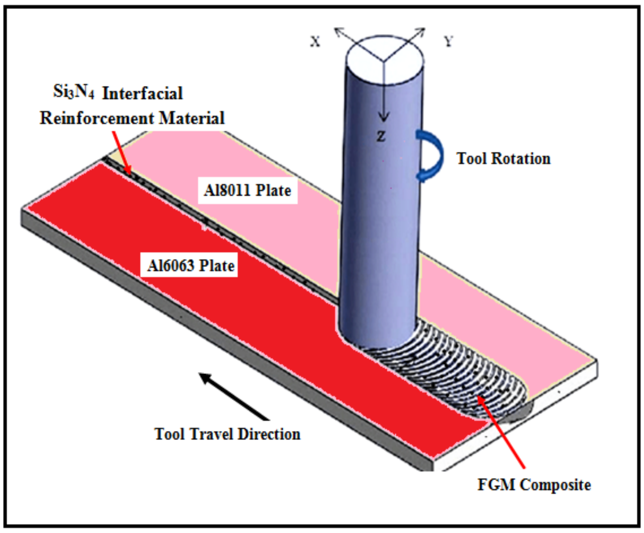

Figure 1. Development of FGM (Al6063 and Al8011) composite using Si_3N_4 reinforcement material [21]

3. Results and Discussion

3.1 Tensile Strength Examination

Table 3 illustrates the tensile test results by experimenting on UTM as shown in figure 2(a). Table 4 shows the ANOVA Table for Si_3N_4 reinforced composite material with two different matrices (Al6063 and Al8011).

Figure 2(b) illustrates the main effect plots for S/N ratio and means. Figure 3(a) shows the influence of tool shape and tool speed on tensile strength in the contour plot. Figure 3(b) and Figure 3(c) illustrates tensile strength analysis for tool speed and cutting fluid and tensile strength study for cutting fluid and tool shape respectively. Figure 3 illustrates the key outcome analysis for the SN ratio. The tensile strength of Si_3N_4 reinforced FGM composite is improved for medium rotational tool speed i.e. 1460 rpm with square tool shape and the slightest tensile strength can be observed for square tool shape with both 920 rpm and 2260 rpm. The transverse speed of the tool in the current investigation was 24 mm/min. The strength of the composite is good for brine at 1460 rpm but continuously decrease when rpm of tool increase or decrease. However, tensile strength is enhanced for kerosene with circular tool shape and least for both triangular and square tool shape. In Square shaped tool the displacement of particles is higher as compared to the circular ones thus the workpiece particles gets displaced and merged to other locations of the workpiece resulting the overall increment in the homogeneity of workpiece. Kerosene is making a layer of fluid between workpiece and the tool, and acts as a coolant by creating a cooling environment at 1460 rpm uniform speed is attained while at lower speeds particles attains a heterogeneity while at higher speeds temperature at the tool workpiece interface becomes higher thus the lubricating agent i.e., kerosene is not sufficient enough to create required cooling temperature. The heat generated overpasses the boiling point of kerosene (170-180°C) therefore midrange values of speed i.e., 1460 rpm is optimum for obtaining themaximum tensile strength.

At the lower speed (920 rpm) the required friction has not been generated for proper FSP. Hence, the required strength has not been achieved. The results reveled that below 1460 rpm tool speed the strength of the developed composite material is less. These results are in line with the findings of Ghandourah et al [6] and Chakravarthi et al. [37].

A substantiation testing is executed by conducting a test using a specific grouping of the factors and levels previously evaluated. Then the estimated mean is

$$\mu_{1,2} = T + (A_2 - T) + (B_2 - T) \quad (1)$$

After analyzing and calculation the parameter which is optimized after the analysis is sample no- 2. The best combination of parameters for Tensile Strength is found in sample no 2 and the values are:

A_1: square
B_2: 1460 rpm
C_3: kerosene
Predicted value = T+ $(A_1$-T) + $(B_2$-T) + $(C_3$-T)
T (average) = 95.36 MPa
A_1= Not significant
B_2= 118.6 MPa

The tensile strength findings of current investigation have been compared with literature and mentioned in table-8

C_3= Not significant

Figure 4 illustrates the SEM (Scanning electron microscope) of the FGM composite for tensile strength. Reinforcement content Si_3N_4 are properly dispersed inside the matrix material. Presence of Si_3N_4 particles inside the matrix material is accountable for enhancing the tensile strength of FGM composite. It has been also found that Si_3N_4 particles can be used as reinforcement. It can be also concluded from the SEM images that Al6063 and Al8081 can be used simultaneously as matrix material with Si_3N_4 ceramic particles in the fabrication of composite material by friction stir method.

Table 3. Tensile test results

Experimental run	Tensile strength (N/mm^2)		
	Sample 1	Sample 2	Average
1.	73.6	73.7	73.65
2.	128.5	137.6	133.05
3.	77.9	78.1	78
4.	94.5	93.9	94.2
5.	102.6	105.1	103.85
6.	88.99	93.2	91.095
7.	78.1	73.2	75.65
8.	118.7	120.4	119.55
9.	88.3	90.06	89.18

Table 4. Analysis of Variance for SN ratio of Tensile Strength

Source	Degree of freedom	Sum of square	Mean Sum of square	F Value	P Value
A	2	0.3083	0.1542	0.24	0.804
B	2	18.9884	9.4942	15.01	0.062
C	2	4.5558	2.2779	3.60	0.217
Residual error	2	1.2651	0.6325		
Total	8	25.1176			

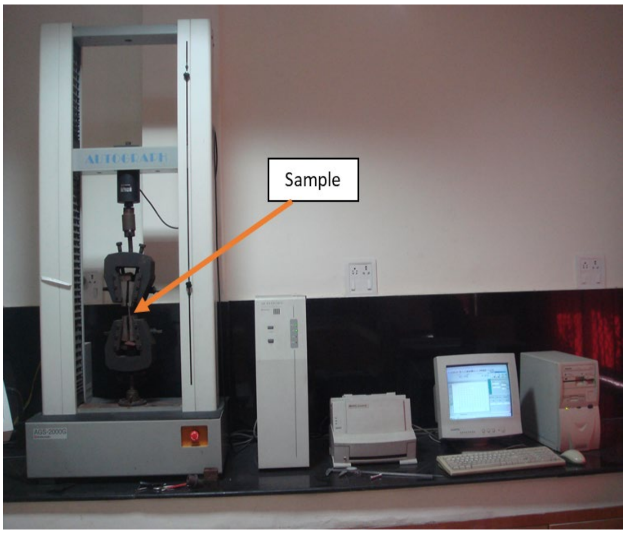

Figure 2(a). Tensile Testing of Developed Composites

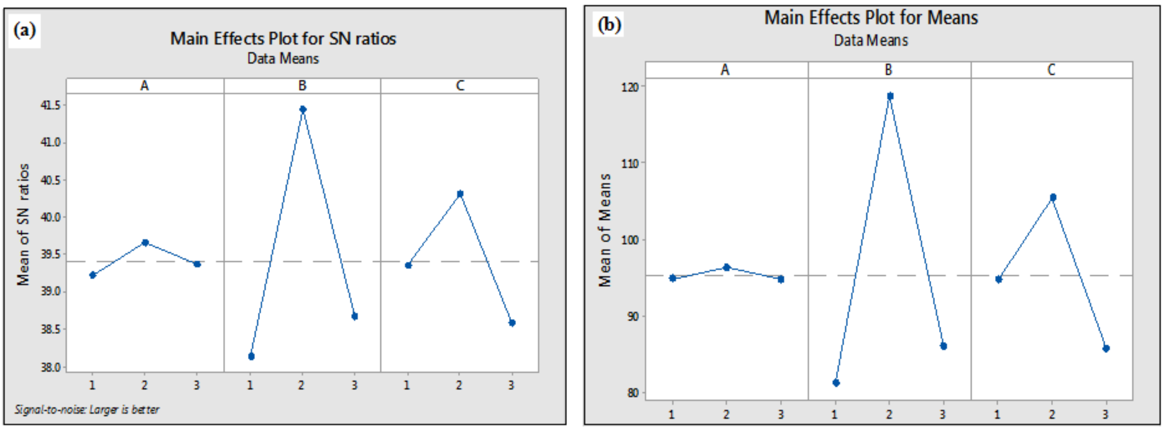

Figure 2(b). Main effect plots; (a) for S/N ratios, (b) for means.

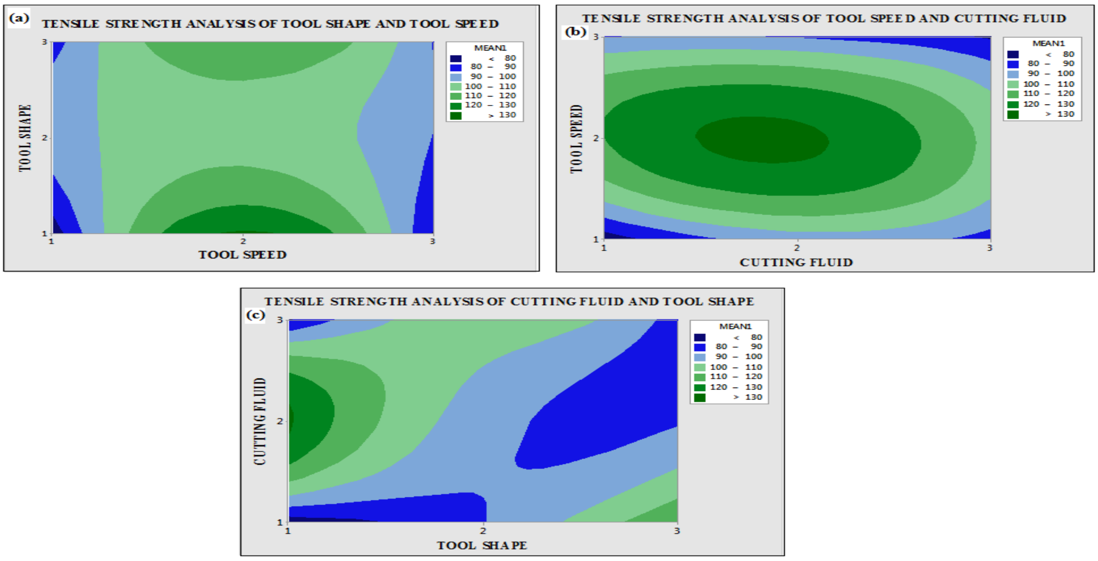

Figure 3. Contour plot; (a) Contour curves of tool speed and tool shape, (b) Contour curves of cutting fluid and tool speed, (c) Contour curves of cutting fluid and tool shape.

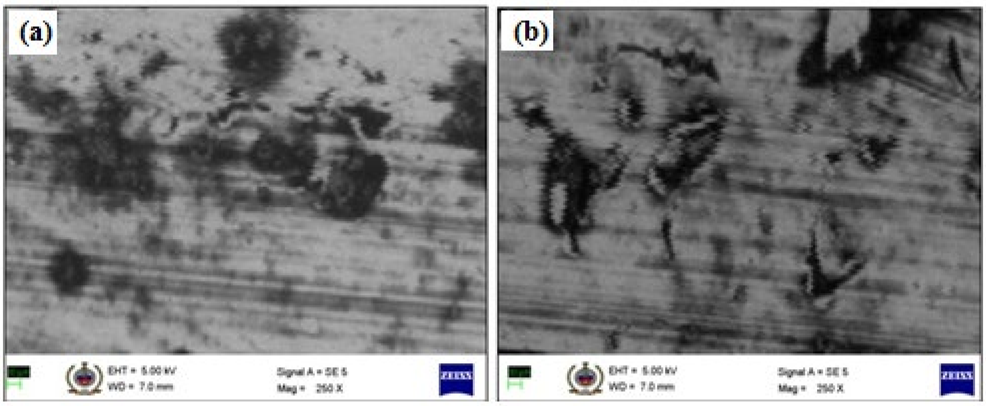

Figure 4: SEM images of tensile strength as response

3.2 Hardness Investigation

Hardness examination of composite reinforced with Si_3N_4 was also evaluated on hardness testing machine as shown in figure 5(i). Table 5 shows the hardness test results of FGM composite. Table 6 explains the ANOVA Table for FGM composite for hardness. Figure 5(ii) illustrates the main effects analysis of FSP parameters on hardness. Figure 6 shows the contour plots for hardness. It can be observed that the hardness is better (59 HV) for circular tool shape with the low speed i.e., 920 rpm but continuously reduces when the tool shape is different i.e. triangular and square with same speed. Least hardness was found to be for square tool shape (52HV) with 1460 rpm and square tool shape with 920 rpm(52HV). The hardness of FGM composite is good for brine solution at any speed of tool but least hardness for kerosene with any tool speed. The possible reason for such kind of observed trend is that circular tool with lower rpm uniformly distributes the workpiece particles thus enhancing the homogeneity of the base metal. Brine solution has comparatively lower boiling point as compared to that of kerosene oil therefore as there in an increment in the shear stress the brine solution viscosity tends to be constant ensuring that there is no change in temperature and pressure. While, the hardness of FGM composite is better for brine solution with any tool shape, and least for kerosene at any tool shape. However, hardness is more for minimum and maximum rotational.

Table 5. Hardness test result

Experimental run	Hardness (HV)				
	Sample 1	Sample 2	Sample 3	Sample 4	Average
1.	52	51	52	53	52
2.	57	59	59	57	58
3.	56	55	56	54	55
4.	52	51	51	52	52
5.	59	57	58	58	58
6.	58	60	59	58	59
7.	59	58	59	60	59
8.	60	59	59	59	59
9.	53	55	54	53	54

Figure 5(i). Hardness Testing of Developed Samples

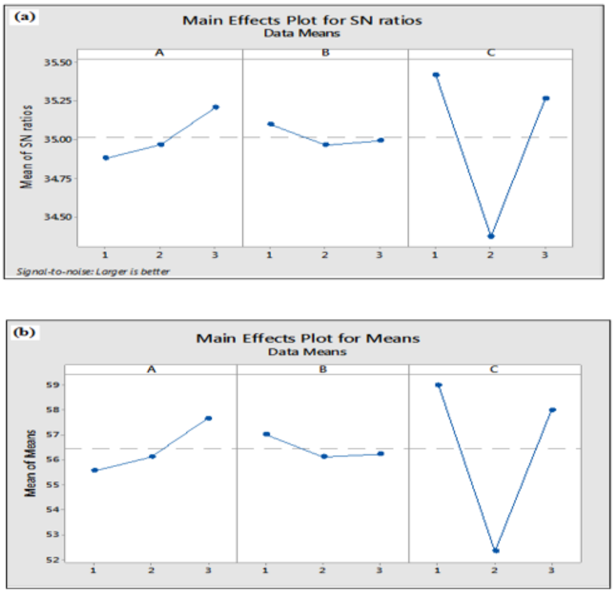

Figure 5 (ii). Main effect analysis of hardness; (a) for S/N ratio, (b) for means

Brine solution has comparatively lower boiling point as compared to that of kerosene oil therefore as there in an increment in the shear stress the brine solution viscosity tends to be constant ensuring that there is no change in tool speed i.e., 920 rpm and 2260 rpm and least for 1460 rpm with any tool shape. Further, the hardness of FGM composite reinforced with Si_3N_4.

At the lower speed (920 rpm) the required friction has not been generated for proper FSP. Hence, the required hardness has not been achieved. The results reveled that below 1460 rpm tool speed the hardness of the developed composite material is less. These results are in line with the findings of Srivastava et al. [8] and Gupta et al. [9].

Figure 7 shows the SEM micro graph of developed composite material. Further micro graph revealed the fair distribution of reinforced particles in the aluminum based matrix. The fair distribution of silica based reinforcement enhanced the hardness of overall developed composite.

The tensile strength findings of current investigation have been compared with literature and mentioned in table-8

FGM composite reinforced with Si_3N_4 is found in sample no. 2 and the values are:

A_1: square
B_2: 1460 rpm
C_3: kerosene
Predicted value = H+ (A_1-H) + (B_2-H) + (C_3-H)
H (average) = 56.30
A_1= Not significant
B_2= 58.9, C3= Not significant

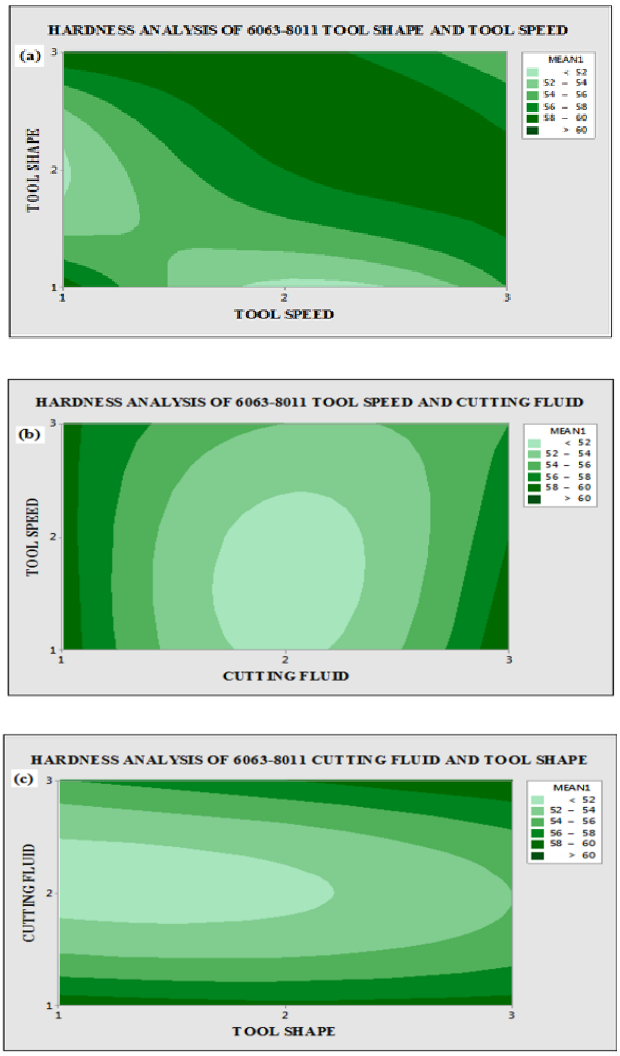

Figure 6. Contour curves of; (a) tool speed and tool shape, (b) tool speed and cutting fluid, (c) cutting fluid and tool shape

Table 6. Analysis of Variance for SN ratio of Hardness

Source	Degree of freedom	Sum of square	Mean Sum of square	F Value	P Value
A	2	0.17417	0.0870	185	0.351
B	2	1.9068	0.9534	20.24	0.047
C	2	0.0298	0.0149	0.32	0.760
Residual error	2	0.0942			
Total	8	2.2050			

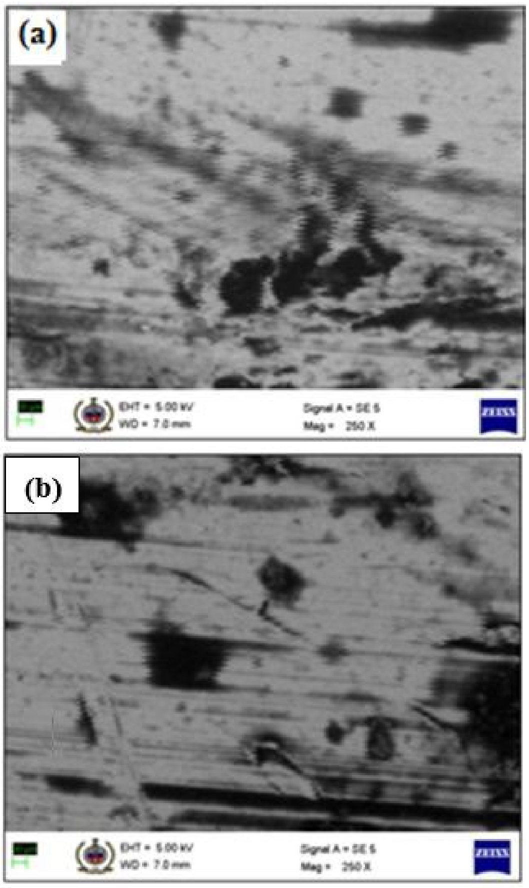

Figure 7. Microstructure Image of composite for hardness.

3.3 Thermal Expansion Analysis

The analysis of thermal expansion was done to know that if this composite is kept at higher temperature then how much difference will be made on its dimension. To perform this test, first the dimensions of the sample were well measured. After this the sample was kept at 450 degree centigrade for 72 hours. Seeing the results, it can be said that this material can be used in high temperature environment.

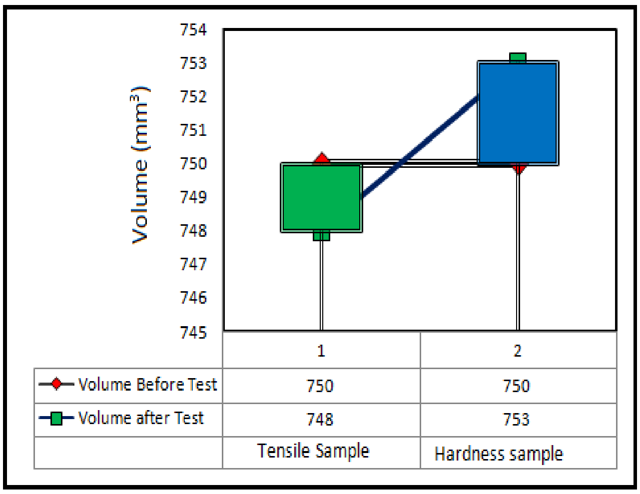

Figure 8. Thermal Expansion of composite reinforced with Si_3N_4

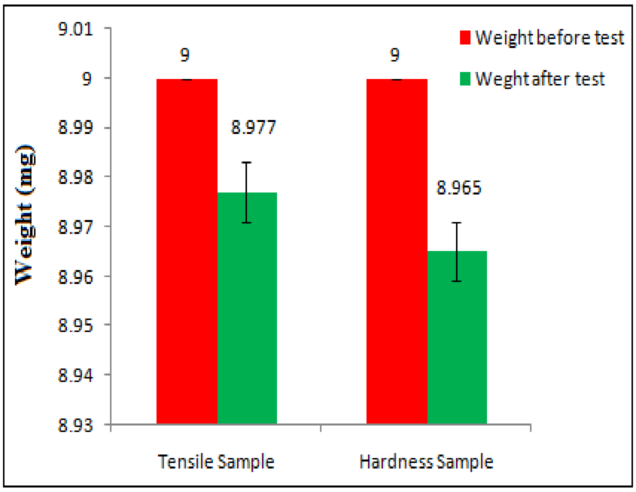

Figure 9. Corrosion behavior of composite reinforced with Si_3N_4

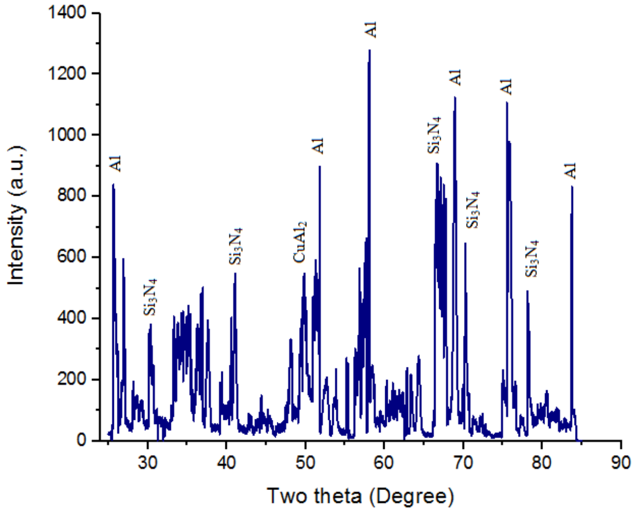

Figure 10. XRD analysis of composite reinforced with Si_3N_4

3.4 Corrosion Testing

Corrosion test has been performed of the prepared samples to identify the ceramic particles Si_3N_4 addition in the aluminium alloys. Corrosion test of FGM composite was performed in 3.5 wt.- % sodium chloride for 5 days. Results revealed that incorporation of Si_3N_4 ceramic particles in the aluminium alloys enhanced the material sustainability in the acetic environment. Figure 9 shows the corrosion loss for the samples.

Corrosion weight loss has been observed 0.023 mg fortensile strength specimen (Sample no. 2) and 0.035 mg for hardness specimen (Sample no. 8). The corrosion rate (CR) was calculated by using the following expression and depicted in table 7:

CR = (Weight loss x K)/ (Density x Exposed area x Exposed time)

Table 7. Corrosion Rate Analysis

Sample No.	Wt. loss (gm)	K	Exp. Density (g/cc)	Exposed Area (cm^2)	Exposed Time(hr)	Corrosion Rate mm/year)
1	0.033	87500	2.71	9	120	0.986572366
2	0.023	87500	2.73	9	120	0.682573599
3	0.021	87500	2.75	9	120	0.618686869
4	0.025	87500	2.77	9	120	0.731214066
5	0.019	87500	2.79	9	120	0.551739015
6	0.025	87500	2.81	9	120	0.720805325
7	0.027	87500	2.83	9	120	0.772968198
8	0.035	87500	2.85	9	120	0.994964263
9	0.032	87500	2.87	9	120	0.903342367

3.5 XRD Analysis of Composite

XRD (X-ray diffraction analysis) of FGM composite reinforced with Si_3N_4(figure 10) developed by FSP technique has been investigated to observe the different produced. Figure 10 shows the XRD of fabricated phases material. XRD of composite material developed at optimum FSP parameters shows the presence of Al, Si_3N_4 and $CuAl_2$ (Copper aluminide) phases.

3.6 Compare with previous published work

Table 8 shows the present study results with previous published work. It

Table 8. Comparison of Present Investigation with Previous Published Work

Ref. No.	Base Mat.	Developed Composition	Tensile strength (N/mm^2)	Hardness (HV)
Present work	Al	Al6063+Al8011, Si_3N_4	133.05	59
2	Al	Al6063+ B_4C	264	92
7	Al	A359+Si_3N_4	468	119
8	Al	A359+Si_3N_4	484	115
12	Al	Al6063+ MgO+SiC	183.6	67
14	Al	Al6061+SiC	298	46
38	Al	Al6351+ Si_3N_4	210	94

4. Conclusion

Conclusive points are given below.

- Aluminium based FGM composite with Si_3N_4 is successfully developed using the friction stir process and can be used in automobile and aerospace industries. Further, the need for a suitable fixture and key holes at ends limit the use of this investigation.

- Tensile Strength (133.05 N/mm^2) is better for 1460 rpm with circular tool shape and least for 920 rpm (73.65 N/mm^2) and 2260 rpm (75.65 N/mm^2) with square tool shape. Tensile strength is better for kerosene at 1460 rpm but continuously reduces when rpm of tool increase and decrease. Tensile Strength is more for kerosene with square tool shape and least for triangular and circular tool shape.
- The hardness of is better (59 HV) for circular tool shape and low speed (i.e. 920 rpm) but continuously decrease when tool shape is different (i.e., triangular & square) and least hardness (51 HV) for square tool shape with 920 rpm. Hardness is good for brine solution at any tool speed but least for kerosene with any tool speed. The hardness of FGM composite is better for brine solution with any tool shape and least for kerosene at any tool shape.
- The Si_3N_4 reinforcement particles improve the thermal corrosion properties of the developed composite material.
- XRD of the fabricated composite illustrated the occurrence of Al, Si_3N_4 and $CuAl_2$ phases. Presence of these hard phases may be responsible for improving the hardness and tensile strength and of the material.
- It has been suggested for the future work that more than one reinforcement material with dissimilar aluminium alloy can be also used to observe the FSW parameters on tensile strength and hardness.

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