

Effect of Number of Friction Stir Processing Passes on Mechanical Properties of SiO₂/5083Al Metal Matrix Nano-Composite

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Abstract

Nano-composites manufacturing is one of advance manufacturing technique in the history of materials. Friction stir processing (FSP) is an emerging surface engineering technology that can produces nano-composites and homogenous microstructures locally, thereby improving the mechanical properties. This paper presents the effect number of passes on Mechanical Properties of SiO₂/5083 Al Metal matrix nano-composites fabricated by friction stir processing and microstructure. Fabrication of in situ SiO₂/5083 Al Metal matrix nano-composites using multi-pass (One Pass, 2 Passes, 3 Passes, and 4 Passes) at rotation speed 400 rpm, and tool travel speed 100 mm/min. In general it is found that, FSP optimum condition it single pass, at this condition increases the ultimate tensile strength (UTS) up to about 110.6 % elongation percentage up to about 132.1 % micro-hardness number up to about 131.7% and impact energy up to about 125.4 from the base material. Also, coarse second phase particle fragmentation and grain refining are observed. Both macrostructure and microstructure observations indicate homogenous microstructures locally of SiO₂/5083 Al metal matrix nano-composite Fabricated by friction stir processing.

Keywords

Nano-Composite; Metal Matrix Nano-Composite; Friction Stir Processing; 5083Al alloy; Mechanical properties; Microstructure Modification.

Introduction

Friction stir welding "FSW" is a solid state joining process invented at The Welding Institute "TWI", UK in 1991 to join Al alloys [1]. Friction stir processing "FSP" is similar, but it does not join materials, but selectively modifies the microstructure to improve local properties [1-3]. In FSP a specially designed rotating tool is plunged into the selected area. Tool shoulder is of a large diameter, while the concentric tool pin is of a smaller one. As the rotating pin descends into the processed part deformation and friction heat is generated. When the shoulder contacts the work surface, its rotation generates additional frictional heat and plasticizes a larger cylindrical metal column around the pin. The area to be processed and the tool move relative to each other, until the selected area is processed to a fine grain size, and the material is transported from the leading to the trailing side of the pin. On cooling, the processed zone forms defect free and re-crystallized fine grained structure. The process is used to modify microstructure of cast metals to improve their mechanical properties, and sometimes to induce super-plasticity [1-4]. FSP of cast A356 and A319 alloys

refined the microstructure, and increased the yield strength [1, 2, 4]. Moreover, ductility and hardness of A319 alloy were increased [1, 2]. Super-plasticity was also observed on FSP of A356 and Al7075 alloys [1].

Aluminum alloys are important lightweight alloys used in industry. Their properties, as low density, ability to be recycled, high ductility and formability, make it suitable for a wide range of industrial applications. However, the strength of conventional cast aluminum alloys is too low. Cast alloys have also many defects as porosity that makes them difficult to be used in many applications. FSP can be used in such cases for localized change of microstructure, and hence to improve mechanical properties.

The effect of FSP on mechanical and microstructural properties is not fully understood. Also, the effect of various friction stir processing parameters is yet to be clarified. Grain fragmentation in stir zone increases with increasing rotation speed. High tool rotation speed increases the temperature, and porosity is almost removed from FSP zone. Increasing rotation speed in friction stir welding of high strength Al-alloy increases grain size [1, 5]

Integrated thermo-mechanical study [1, 2] of FSP of light weight alloys indicates that, grain size

increases with increasing rotation speed [1, 2, 6]. Similarly, grain size increases with increasing ω/v ratio during FSW of AZ31 alloy [6, 7]. At higher rotation speed, more heat is generated, and a higher temperature is attained, and a larger grain size is formed [1]. Similar observation is made for FSW of copper [1, 8].

The effect of rotation speed on hardness of FSP and FSW zones was studied [1, 7-9]. Hardness decreases with increasing rotation speed, for FSP of AZ31 Mg alloys [1], Al alloys [1, 10], and light weight alloys [1]. Also, the effect of travel speed v on hardness is opposite to that of rotation speed ω [1, 2].

FSP and FSW leads to micro-structural modification and hardness increase [1, 6, 11]. Ultra-fine-grained Al–Al₂Cu composite produced by FSP, revealed a significant increase in hardness of stir zone than that of base metal. This is also true for FSW stainless steel [1].

Surface nano-composites are novel materials, where the second phase is spatially distributed near the surface. The phase composition is linearly graded as a function of distance from surface. Several techniques are used to fabricate surface nano-composites, especially friction stir welding and processing (FSW & FSP) [1, 2, 6, 12, 13], which are solid state techniques to obtain fine-grained microstructures. FSP uses the same approach as FSW, in which a rotating tool is inserted into a substrate to produce a plastically deformed zone. Stir zone consists of fine and equi-axed grains produced by dynamic re-crystallization [1]. FSP is a grain refining technique, and is very attractive to fabricate composites. Nano particles are found well distributed and bonded in Al matrix surface nano-composites [1, 2, 14, 15]. Examples include wrought Al alloy microstructure conditioning using high strain rate super plastic deformation [1, 16], and microstructure refining to improve ductility of high-strength Al nano-composites [1, 13, 17-22]. Other applications of FSP are to improve cold-workability of rough Al plates [1, 23], and the mechanical properties of both Al castings [1], and fusion welds of wrought Al plates [1, 24].

The objective of present work is to study the effect of FSP technological parameters on mechanical properties of SiO₂/5083Al metal matrix nano-composite.

Experimental Work

Materials

Materials used in present study are given in Table 1.

In present work, FSP of 5083 Al alloy is cut from 10 mm thick plates, it's to agree with the standard range of chemical composition according to ASTM B209M and B928M [25, 26], while its chemical analysis is shown in Table 2.

Test samples 10 mm thick, 70 mm wide, and 200 mm long are cut from the alloy plates. Grooves 5.5 mm deep, 2 mm wide, and 200 mm long are prepared on test samples to be filled with nano particles and processed by FSP, see Figure 1.

The effect of tool rotation rate (ω) and tool travel speed (v) on the microstructure and mechanical properties is studied.

The ultimate strength UTS of 5083 Al alloy is 316 MPa, and its elongation e is 22.08%. The FSP tool used is made of K100 tool steel with hardness HRC 62, and its chemical composition is shown in Table 3.

Table 1 Used materials.

No.	Material	Formula	Supplier
1	5083 Al alloy as plates 10 mm thick, 150 mm wide, and 400 mm long	5083 Al-Mg alloy	Egyptalum (EGAL) Company-Egypt
2	SiO ₂ (10-20 nm) nano powder, 99.5%	SiO ₂	Sigma – Aldrich

Table 2 Chemical composition of 5083 Al alloy.

Elements	Wt%	
Si	0.128	
Fe	0.332	
Cu	0.022	
Mn	0.82	
Mg	4.6	
Cr	0.130	
Zn	0.008	
Ti	0.012	
Others	Each	<0.02
	Total	0.015
Al	93.93	

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Table 3 Chemical composition of K100 tool steel.

Element	C	Si	Mn	Cr	Fe
wt. %	2.00	0.25	0.35	11.50	Rest

Experimental Setup

Friction stir processing to fabricate SiO₂/5083Al metal matrix nano-composite is shown in Figure 2 using multi-pass (1 to 4 passes) at 400 rpm rotation speed, and 100 mm/min tool travel speed. This is carried out using friction stir processing machine in the Faculty of Petroleum and Mining Engineering in Suez.

Cylindrical friction stir processing tool with and without pin are shown in Figure 3.

Grooves made on test specimens are filled with nano powder particles, then friction stir processed using a cylindrical tool without pin (Figure 3), to apply surface groove repair. The fixture used to clamp FSP samples is shown in Figure 4.

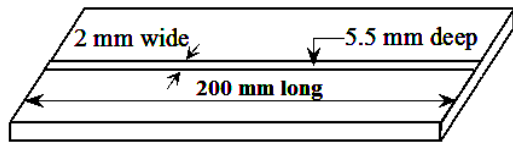


Figure 1 Schematic drawing of groove dimensions.

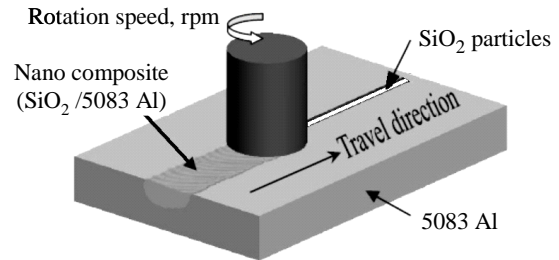


Figure 2 Friction stir processing (schematically).

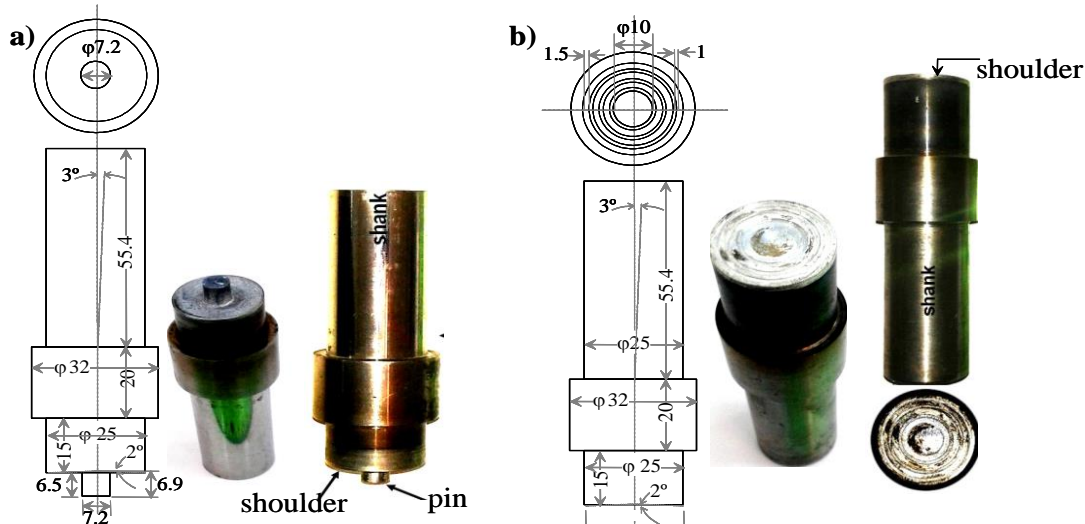


Figure 3 Friction stir processing tool, a) with pin, and b) without pin used in present work.



Figure 4 Used fixture to clamp FSPed samples

Evaluation of Friction Stir Processed Specimens

Evaluation of FSP specimens is carried out using visual inspection, tensile testing, hardness testing, and microscopic examination.

Visual Inspection

Friction stir processed (FSPed) plates are visually inspected. Also, width of nugget and width of discolored (oxidized) area are measured. FSP plates cross-sections are visually examined after sectioning with a hand saw and polishing up to grit 600 SiC emery papers.

Tensile testing

The FSP test specimens are machined by a CNC-milling machine and tested without any top surface modification where a rough surface is induced by the FSP tool, but reduction of thickness done by bottom surface modification.

Tensile test specimen shown in Fig.5 is cut with the nugget in the middle, Figure 5 (a) shown Position selection for the specimens of tensile test of FSP. Tensile testing used 20 tons "Instron" universal testing machine. UTS is determined using the thickness of thinnest area (nugget), Tensile test specimen dimensions shown in Figure 5 (b).

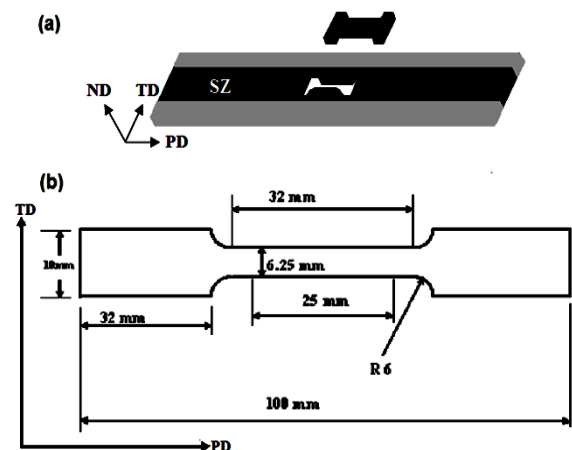


Figure 5 Tensile test specimen (a) Scheme of Position selection for the specimens of tensile test of FSP,(b) Tensile test specimen dimensions in mm (6 mm thick) (ASTEM Standards Part 31).

Micro-hardness Testing

Micro-hardness profile of processed zone is measured on cross-sections perpendicular to FSP direction, and on processed surfaces, using 0.5kg load for 10sec. Hardness measuring is conducted on specimens prepared for metallographic examination.

Microstructure Investigation

Microstructure study is carried out to know the effect of the process on the microstructure, and to show how the process can overcome base metal defects. To reveal Al-Si alloy microstructure, the following regime is employed:

- Planar grinding of the surface with 220, 400, 600, 800, 1000, then 1200 grit SiC emery papers. Water is used for lubrication.
- Initial polishing is done using colloidal silica or alumina suspension (ALU-MIK 0.3 μm) for 20 to 30 seconds. Water is used for cooling to avoid microstructure changes.
- Polishing is finished with 1 μm diamond compounds for 3 or 2 minutes. Water is used for lubrication.

For grinding, a Struers "Knuth-Rotor2" manual grinding machine is used, while polishing is done by Struers "LaboPol", which is a single disc machine with variable rotation speed. Electrolytic polishing and etching are carried out using "Struers Polectrol" machine at 22 volt for 10 seconds at ambient temperature, using a special electrolyte. Also, polished surfaces are examined using "Olympus" optical microscope.

such as frontal advance determination, average water saturation at the breakthrough time determination, and average water saturation and water front.

Results

Macro- and Micro-Structural Observations

Figure 6 shows an overview of FSPed 400 mm long, 100 mm wide, and 10 mm thick plates, indicating the stir zone (SZ) and base metal (BM) regions on both sides. The geometry of the plate and stir zone affects heating and cooling regimes and the microstructure of various zones of specimen.

Typical macrostructure of FSPed specimen using 400 rpm tool rotation speed, 100 mm/min travel speed, and after different number of passes is shown in Figure 7. In addition to the base metal "BM", the FSP test specimen consists mainly of three distinct zones; a fine grain dynamically re-crystallized stir zone (SZ), thermo-mechanically affected zone (TMAZ), and heat affected zone HAZ. During FSP "TMAZ" area experiences both temperature and deformation and is characterized by highly deformed structure. HAZ is a thermally affected zone without any mechanical effects. No porosity can be detected in SZ, TMAZ, and HAZ, while the base metal structure is characterized by porosity. The locations of the advancing side (AS) and the retreating side (RS) are indicated in Figure 7.

The macro-structure on cross-sections of FSP samples processed for one pass or more are shown in Fig.8. All samples processed for more than one pass show defects in the stir zone. The stir zone of the sample processed for one pass is obviously defect-free.

Figure 9 shows an isometric drawing to indicate the location in stir zone center, at which the

microstructure is studied "point c". Figure 10 shows the microstructure of present 5083 Al alloy without FSP processing, and Figure 11 reveals the microstructures of FSP samples after various numbers of processing passes.

For more precise investigation of BM microstructure shown in Figure 10 and that of the central portion of SZ processed under different conditions are shown in Figure 11, that are generated by FSP at 400 rpm rotation speed, 100 mm/min travel speed and after different number of passes. The figure shows various regions obtained by FSP. It also shows that respondents that multi-pass processing leads to the formation of visible defects. However, processing in a single pass leads to no

visible defects. Moreover, the distribution of nano-silicon dioxide powder in stir zone of specimens processed in a single pass is better. Microstructure refining and homogeneity in stir zones of processed specimens are obviously better than in BM, as shown in Figures 10, 11. This agrees well with previous observations[1, 5, 6, 13, 15, 20-22]. Also, porosity is almost removed from FSP stir zone. Severe plastic deformation at relatively high temperature during friction stir processing causes second phase fragmentation and redistribution in the alloy matrix.

Mechanical Properties of FSP Specimens

The Fracture occurs always at the middle of the tensile specimens, as shown in Table 4, which shows atop view and a side view as well as the cross-section of fractured BM as well as FSP tensile specimens. It is observed that, the fracture surface of the stirred specimen is smooth, and that of the non-stirred one is rough. This indicates the grain refining that occurs in the stir zone.

Figure 12 shows the tensile properties of base metal and FSP tensile specimens processed at 400 rpm rotation speed, 100 mm/min travel speed, for different number of passes. It is observed that specimens processed for a single pass show maximum UTS and ductility.

Figure 13 shows the average Microhardness of base metal and FSP tensile specimens. It is obvious that FSP specimens after a single processing pass show the higher Micro-hardness value. In general, tensile properties, elongation and micro-hardness to fracture slightly decrease with increasing number of passes. Figure 14 shows the percentage change of mechanical properties (UTS, ϵ %, VHN) of FSP specimens processed for different number of passes in comparison with corresponding values of the base metal.

For more clear presentation of the effect of friction stir processing on the mechanical properties of friction stir processed SiO₂/5083Al metal matrix nano-composite, measured values of UTS, maximum elongation, and hardness are presented in Table 5. Also, the table includes the ratio between the property value and the corresponding value of base metal. Moreover, it indicates whether the value is a maximum or a minimum among all corresponding results.

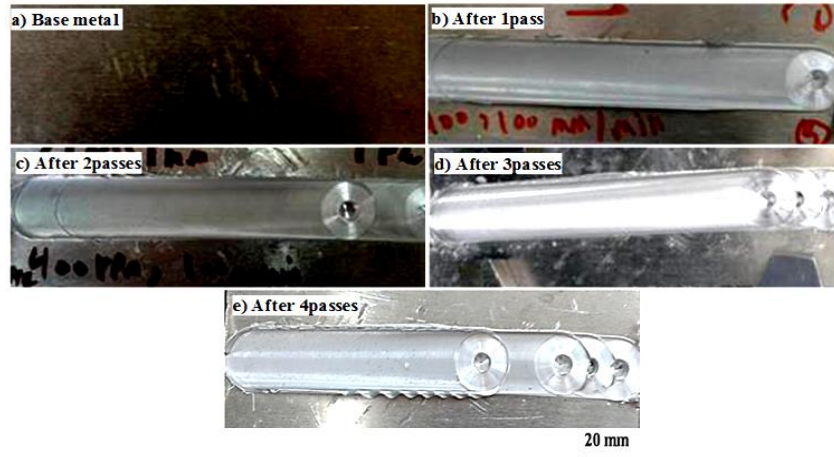


Figure 6 Top views of samples (a) Base metal, and FSP samples after: b) after one pass, c) after double passes, d) after triple passes and e) after quadruple passes, at 400 rpm rotation speed and 100 mm/min travel speed).



Figure 7 Cross section of FSP sample.

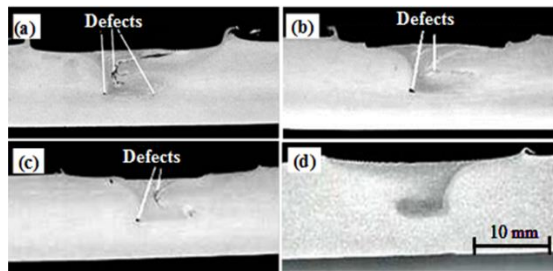


Figure 8 Macrostructure of FSP after: a) quadruple passes, b) triple passes, c) double passes and d) one pass.

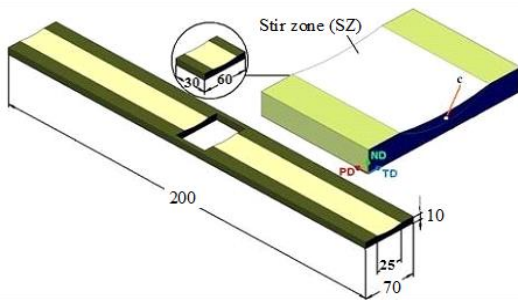


Figure 9 Isometric drawing indicating the location of microstructure study

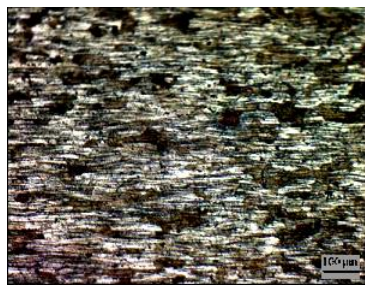


Figure 10 Optical microstructure of present 5083 aluminum alloy without FSP.

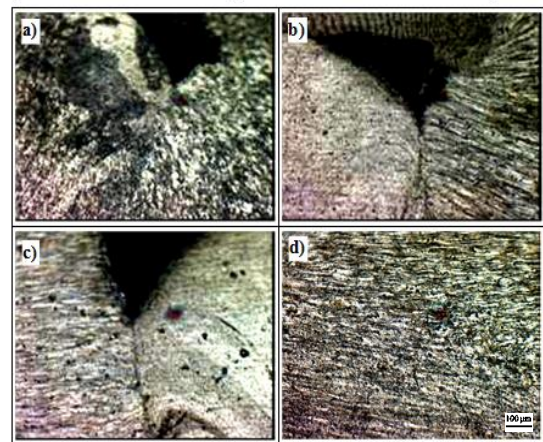


Figure 11 Microstructure of FSP specimens after: a) 4, b) 3, c) 2 passes and d) 1 pass.

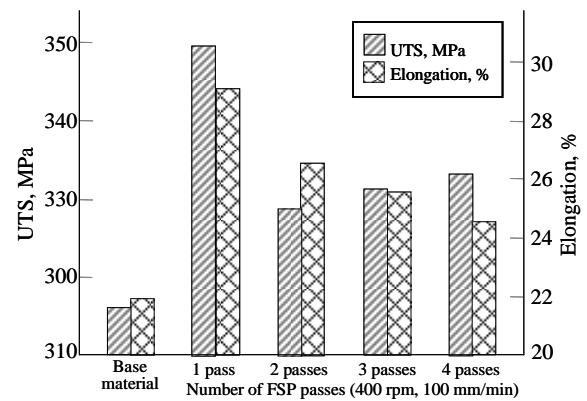


Figure 12 Tensile properties of base metal and friction stir processed specimens.

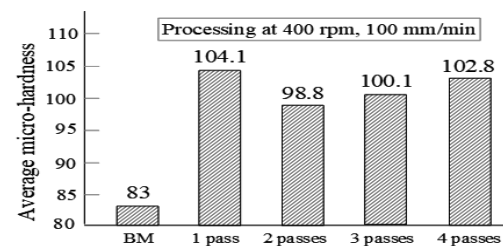
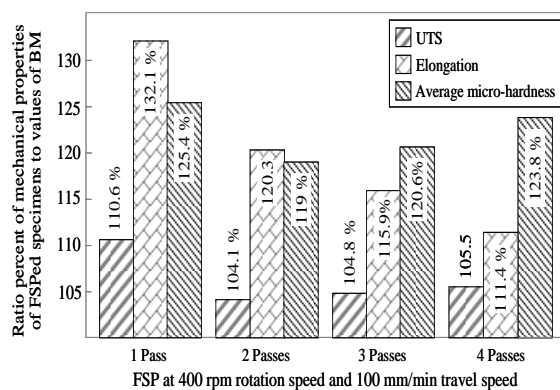
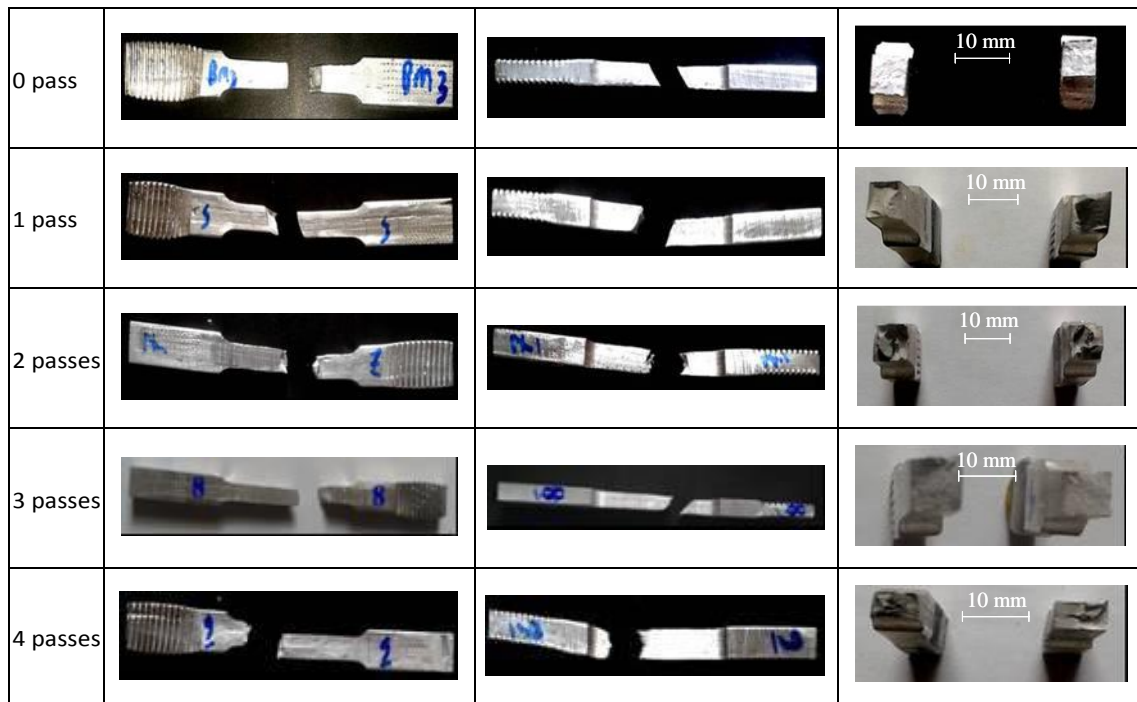


Figure 13 Average micro-hardness of base metal and friction stir processed specimens.

Table 4 Specimens before tension, fracture top view, and fracture side view, for BM and FSP specimens after different number of passes.**Figure 14** Ratio between tensile properties of SZ to those of BM with pass number.

From Table 5 and Figure 14 it is obvious, that using friction stir processing to produce nano-composite material ($\text{SiO}_2/5083\text{Al}$), increases UTS, ductility, and hardness for all processed specimens at 400 rpm rotation speed, and 100 mm/min travel speed for different number of passes (one pass, two passes, three passes and four passes). This agrees well with previous results, [1, 2, 6, 13, 15-18, 20-22].

This may be due to grain fragmentation and porosity removal during FSP. It can also be due to the addition of nano powder particles and its homogeneous distribution in the stirring zone. The increase of the ultimate tensile strength, ductility, and hardness is higher when friction stir processing is carried out in one pass, when 400 rpm tool rotation speed, and 100 mm/min travel speed are applied. Friction stir processing in one pass increases UTS to about 110.6%, the elongation percent to about 132.1%, and the average micro-hardness to about 125.4% of the base material.

Processing in two passes increases UTS to about 104.1%, that of elongation percent is 120.3%, and

that of micro-hardness is 119% to that of the base material, while processing in three passes increases UTS to about 104.8% and the elongation percent to 115.9%, and average micro-hardness to 120.6% of the base material. Finally, processing in four passes increases UTS to about 105.5%, the elongation percent to 111.4%, and micro-hardness to 123.8% of the base material. This could be due to the presence of visible defects in stir zone in the specimens of multi-pass, while such defects are not in specimens made in one pass of friction stir processing.

Conclusions

Based on present observations the following conclusions could be drawn:

- Friction stir processing is an efficient method to fabricate $\text{SiO}_2/5083\text{Al}$ metal matrix nano-composite, characterized by homogeneous micro-structure and high mechanical properties.
- FSP increases the ultimate tensile strength to ~110.6%, the elongation to ~132.1%, and the average micro-hardness to ~125.4% of the base material, at 400 rpm tool rotation speed and 100 mm/min travel speed and in a single pass.
- At 100 mm/min tool travel speed, 400 rpm tool rotation speed, highest mechanical properties are possible by FSP in a single pass.
- Tensile elongation and micro-hardness decrease at higher number of FSP passes.
- Homogenous microstructure is locally formed by a single pass of friction stir processing of $\text{SiO}_2/5083\text{Al}$ Metal matrix nano-composites.

Table 5 Mechanical properties of SZ and BM processed at 400 rpm, and 100 mm/min.

	UTS, MPa	Elongation, %	Micro-Hardness
0 Pass	316 MPa (UTS/UTS ₀)x100=100 %	22.08 % (e%/e ₀)x100=100 %	83 VHN (VHN/VHN ₀)x100=100 %
1 Pass	349.38 MPa (UTS/UTS ₀)x100=110.6 % max	29.16 % (e%/e ₀)x100=132.1 % Max	104.8 VHN (VHN/VHN ₀)x100=125.4% Max
2 Passes	328.84 MPa (UTS/UTS ₀)x100=104.1 % min	26.56 % (e%/e ₀)x100=120.3 % ---	98.77 VHN (VHN/VHN ₀)x100=119% Min
3 Passes	331.09 MPa (UTS/UTS ₀)x100=104.8 % ---	25.58 % (e%/e ₀)x100=115.9 % ---	100.1 VHN (VHN/VHN ₀)x100=120.6% ---
4 Passes	333.33 MPa (UTS/UTS ₀)x100=105.55% ---	24.6% (e%/e ₀)x100=111.4% Min	102.76 (VHN/VHN ₀)x100=123.8% ---

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