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**ORIGINAL ARTICLE**

# Effect of multipass friction stir processing on mechanical properties of A356/SiC metal matrix composite

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## Abstract

This study investigates the effects of multiple-pass friction stir processing (MPFSP) with SiC reinforcement particles on the microstructure and mechanical properties of A356 aluminum alloy. The MPFSP process induces severe plastic deformation (SPD) and dynamic recrystallization (DRX), resulting in refined recrystallized grains in the stir zone (SZ). Homogeneous dispersion of SiC particles enhances mechanical properties, including hardness and ultimate tensile strength (UTS), through grain refinement and increased resistance to dislocation movement. MPFSP/SiC demonstrates a significant increase in Vickers hardness, reaching a maximum of 126 HV after 5 FSP passes at 500 rev/min. The UTS improves with increasing FSP passes, with values ranging from 260 MPa to 325 MPa, attributed to grain refinement during DRX. The highest UTS is achieved at a rotational speed of 1500 rev/min with 5 FSP passes. Moreover, maximum hardness values are observed in the 5th pass FSP at 500 rev/min with SiC reinforcement particles. The mean indentation hardness increases from 868 HV in the base metal to 126 HV after 5 FSP passes. These findings demonstrate the effectiveness of MPFSP with SiC reinforcement particles in enhancing the mechanical properties of A356 aluminum alloy, offering insights for the development of advanced aluminum matrix composites.

## 1. Introduction

In many applications, the surface properties of components play a crucial role in determining their useful life, especially when it comes to factors like tensile strength, hardness, and wear resistance. In this context, friction stir processing (FSP) has gained significant attention as a surface modification technique [1, 2]. FSP is a solid-state joining and surface modification technique that involves the frictional heating and plastic deformation of materials. It is commonly used to improve the surface properties of aluminum and its alloys, including AA5083, in various industries such as marine, armor plate, and transportation [3]. FSP can enhance the tensile properties and wear resistance of aluminum components by refining the microstructure and introducing beneficial changes to the surface layer. During FSP, a specially designed tool is rotated and traversed along the material surface, generating heat and causing severe plastic deformation. This process leads to the breakdown of coarse microstructural features, the formation of fine-grained structures, and the redistribution of alloying elements. The resulting refined microstructure and improved alloy distribution contribute to enhanced wear resistance and other desirable surface properties [4, 5]. By utilizing FSP, manufacturers can extend the useful life of components subjected to wear and reduce the need for frequent replacements or repairs. FSP offers the advantage of operating

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at lower temperatures compared to some composite manufacturing methods, ensuring that the material remains in a solid state. This eliminates the problems associated with high process temperatures while also promoting grain refinement and enhancing the material's properties [6]. To produce Metal Matrix Composite (MMCs) using FSP, the reinforcement powder is introduced into the process. Mishra et al. [7] applied the powder directly to the surface of the plate before initiating the FSP. This allows the powder to be mixed and distributed within the material during the FSP process, resulting in the formation of a metal matrix composite with enhanced properties. Mostafapour et al. [8] involve creating a groove in the material where the reinforcement powder is filled. This ensures that the powder is effectively incorporated into the material during FSP. A pin-less tool is used, and a surface repair process can be performed to cap the groove to prevent the powder from escaping, effectively containing the reinforcement powder within the material. By incorporating reinforcement powder into the FSP process, MMCs can be produced with tailored properties, such as improved strength, stiffness, and wear resistance, by taking advantage of the synergistic effects of the reinforcement and the refined microstructure achieved through FSP. Yuvaraj et al. [9] investigated the wear characteristics of Al5083 surface hybrid nanocomposites produced using FSP. The hybrid nanocomposites were created by incorporating nanoparticles into the aluminum matrix during the FSP process. They observed that the resulting composite exhibited superior mechanical properties and wear resistance compared to the base alloy. This indicates that the incorporation of nanoparticles through FSP enhanced the overall performance of the material. Adem Kurt et al. [10] focused on incorporating SiC particles into commercially pure aluminum using FSP to form particulate surface layers. They examined the effects of rotational and traverse speeds on the distribution of SiC particles in the composite. Their findings revealed that increasing the rotational and traverse speeds led to a more uniform distribution of SiC particles within the aluminum matrix. Conventional and powder metallurgy methods for fabricating bulk metal matrix composites have drawbacks: uneven distribution of reinforced particles (agglomeration), interfacial reactions between particles and matrix, and particle segregation along grain boundaries. These drawbacks negatively impact the performance of the composites, leading to poor ductility and toughness [11-13]. Mirjavadi et al. [14] utilized the FSP technique to fabricate AA5083/ZrO2 nanocomposites. Through an increase in the number of FSP passes, the hardness values of the composites gradually improved due to microstructure refinement. For eight FSP passes, a hardness of 130 HV was achieved. Another approach in surface reinforcing using FSP is the in-situ reinforcing method, which involves synthesizing desired reinforcements during the processing itself. In-situ composites offer several advantages, including defect-free reinforcement-matrix interfaces, thermodynamically stable reinforcements, improved compatibility, and higher bonding strength between the reinforcements and the matrix [15,16].

In this work, A356 was used as the base material in the form of plates. SiC nanoparticles were employed as the reinforcing material, which was incorporated into the aluminum matrix through the multipass FSP process. The addition of SiC nanoparticles to an aluminum matrix offers improved mechanical properties, enhanced thermal conductivity, and increased wear resistance, making it a popular choice for various applications, including aerospace, automotive, and electronic industries. Specimens with varying numbers of FSP passes were successfully produced and subjected to analysis using optical microscopy (OM), scanning electron microscopy (SEM), and X-ray diffraction (XRD) techniques. These analyses allowed for a comprehensive examination of the microstructural characteristics. The microhardness distribution was calculated, evaluated, and correlated with the observed microstructural features. Experimental trials were conducted to determine the optimal parameters, including a rotating speed of 1600 rpm, transverse speed of 25 mm/min, tool tilt angle of 2, and multiple passes. These parameters were selected to ensure a sound FSP region and achieve a homogeneous dispersion of the particulates in the stirred zone.

## 2. Materials and Methods

The base material in this study is A356 Al alloy, which consists of the following elements in weight percent: 6.5% Si, 0.28% Mg, 0.18% Fe, 0.21% Cu, 0.09% Mn, 0.08% Zn and the remaining portion is aluminum. The base plate was then cut into smaller plates measuring 180 mm in length and 50 mm in width. SiC nanoparticles were employed as the reinforcement particles. This SiC nanoparticle was commercially available and had an average particle size of 80 nm and a purity of 99%. Friction Stir Processing (FSP) was performed using a conventional milling machine with a tilt angle of 3° on the machine's head. The plates were securely fixed during FSP with a robust fixture.

In order to create a composite, a 2 mm deep groove with a width of 0.9 mm was made on the plates. The groove was then filled with the SiC nanoparticles. Based on a previous study [6], the following parameters were chosen for the FSP: a traveling speed of 25 mm/min and a rotational speed of 1250 rpm. The rotating tip surface of the pin was kept in contact with the surface of the plate for 30 seconds before plunging to ensure that the temperature reached a sufficient level for completing the process without any defects. After the plunge of the pin into the plate with a traveling speed of 25 mm/min was completed, in this study, an H13 steel FSP tool with a hardness of 51 Rc, an 18 mm shoulder diameter, and a 6 mm threaded pin was used. After filling the groove with powder, a pinless tool was used to close the groove.

Samples were taken from the stir zone of the FSPed to evaluate the microstructure. These samples were then machined using an end mill tool. The surface roughness left by the machining process was removed by using SiC abrasive papers of 400 to 3000 grit size. Afterward, the samples were mechanically polished and etched using a modified Poulton's reagent consisting of 2.5 ml HF, 40 ml HNO3, 30 ml HCl, 12 gr

$H_2CrO_4$, and 42.5 ml $H_2O$. Optical microscopy (OM) and scanning electron microscopy (SEM) were used to capture macro and micrographs of the cross-sections of the samples. Grain size analysis of both the samples and the base metal was performed using Clemex image analysis software. To assess the effect of the process on the tensile strength of the samples, a tensile test was conducted. Tensile samples were cut from the stir zone of the processed parts for this purpose. The dimensions and shape of the tensile test specimens were determined according to ASTM-E8 standards. After removing the excess areas from the top and bottom of the processed region, the samples were cut out of the processed path using an electrical discharge machining wire cut. The tensile test was conducted at a crosshead speed of 1 mm/min at an ambient temperature of 24 °C. Micro-hardness measurements were also performed on the samples. A load of 100 grams was applied to the nugget zone for 15 seconds. On each sample, the micro-hardness test was conducted at various distinct points. The average hardness value was calculated and will be reported in the results.

## 3. Results and Discussion

### *3.1 Tensile Strength*

The consequence of FSW parameters on metallurgical characterization of MPFSP/SiC samples has been investigated. The tensile test specimens were tested using a UTM according to ASTM E8 standard, and the stress-strain value of MPFSP/SiC of A356 at TRS of 500 to 1500 rpm and constant TS of 65 mm/min was shown in Table 1. The mechanical properties included UTS, % strain, and microhardness. The absence of reinforcement particles, the grain size mainly controls the mechanical properties of the FSP samples. When reinforcement particles such as SiC are added. Various factors also play an significant role in determining the mechanical properties of the material. These factors affect the load transfer mechanism and the stress distribution in the composite, which in turn affects the overall mechanical behavior of the material. Therefore, it is important to carefully consider these factors when designing and fabricating reinforced composites. These factors imply that the addition of reinforcement particles can significantly affect the mechanical properties of the FSP samples beyond just the grain size [8]. Table 1 shows the stress-strain value of an AMC subjected to MPFSP with the addition of SiC nanoparticles. The tensile stress of the MPFSP/SiC increased from 410 MPa to 525 MPa as the FSP increased from 1 to 5, indicating an improvement in the material's strength. Previous research had reported that the coarse grain structure and minimum hardness of the TMAZ and HAZ contributed to the fracture location of the material. However, the addition of SiC nanoparticles and the resulting grain refinement and equiaxed grain structure of the MPFSP/SiC improved its UTS beyond that of the parent material.

*Table 1: Mechanical Properties of FSPed joint A356*

| Run | TRS (rev/min) | FSP pass | Traverse speed (mm/min) | UTS (MPa) | % Strain | Joint efficiency (%) | Hardness (HV) |
|---|---|---|---|---|---|---|---|
| 1 | 500 | 1 | 75 | 264.803 | 12.61 | 120.61 | 119.25 |
| 2 | 500 | 3 | | 271.465 | 14.75 | 123.64 | 108.75 |
| 3 | 500 | 5 | | 273.917 | 14.12 | 124.75 | 126 |
| 4 | 1000 | 1 | | 268.742 | 15.87 | 122.41 | 123 |
| 5 | 1000 | 3 | | 283.661 | 14.74 | 129.19 | 117.75 |
| 6 | 1000 | 5 | | 295.745 | 16.17 | 134.69 | 103.5 |
| 7 | 1500 | 1 | | 286.872 | 15.57 | 130.65 | 93.75 |
| 8 | 1500 | 3 | | 299.031 | 16.74 | 136.19 | 89.25 |
| 9 | 1500 | 5 | | 321.989 | 19.55 | 146.65 | 100.5 |

It also suggests that the ductility and strength of defect-free MPFSP/SiC depend on the thermal properties of the base metal. As the number of FSP passes increased, the proof resilience of the MPFSP/SiC also improved, indicating a potential improvement in the material's ability to withstand plastic deformation without permanent damage. Overall, the results suggest that MPFSP with the addition of SiC nanoparticles can improve the mechanical properties of AMC, particularly its strength and hardness. Overall, the results suggest that MPFSP/SiC can be an effective technique for improving the mechanical properties of A356. MPFSP can lead to increased dispersion and fragmentation of SiC reinforcement particles, as well as improved dislocation movement resistance.

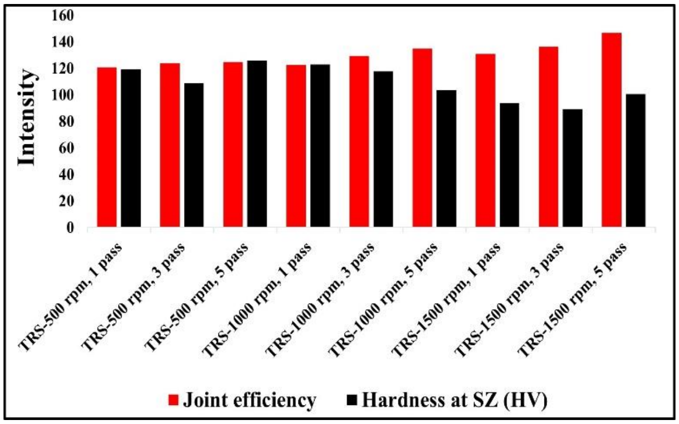
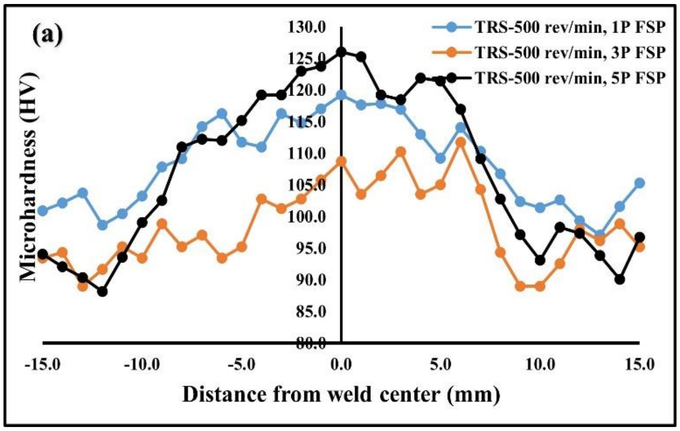


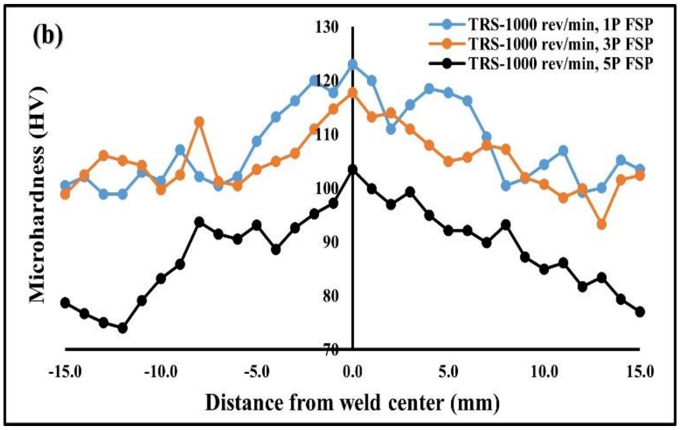
*Figure 1: Comparison of joint efficiency and hardness at SZ of MPFSP/SiC of A356*

The MPFSP process induces intense DRX, material plasticization, fragmentation, and dispersion of SiC with A356 composite, which can contribute to improved strength and ductility. However, it is important to note that the tendency for fragmentation of SiC reinforcement particles may also have some negative effects on the overall properties of the material, and additional studies may be needed to fully understand the trade-offs between these different factors. The use of MPFSP with SiC reinforcement particles on A356 resulted in an increase in tensile properties as the FSP passes increased. The minimum UTS (428.83 MPa) was perceived at TRS of 500 rev/min with 1P FSP, while the maximum UTS maximum UTS (521.44 MPa) were observed at a TRS of 1500 rev/min, with 5P FSP. This improvement was attributed to the materialization of refine grains during DRX reveals in table 1.

### *3.2 Micro-hardness*

Hardness values rise with an increasing number of FSP passes due to various factors, including grain fragmentation and refinement of SiC reinforcement particles in the aluminum matrix composite (AMC) shown in Fig. 2. The Hall-Petch relationship, which links smaller grain size to increased strength, is applicable. The thermal input from MPFSP incorporates hard fragmented and reinforcing SiC particles, refining the grain size in the AMC and boosting the hardness of base metal A356.





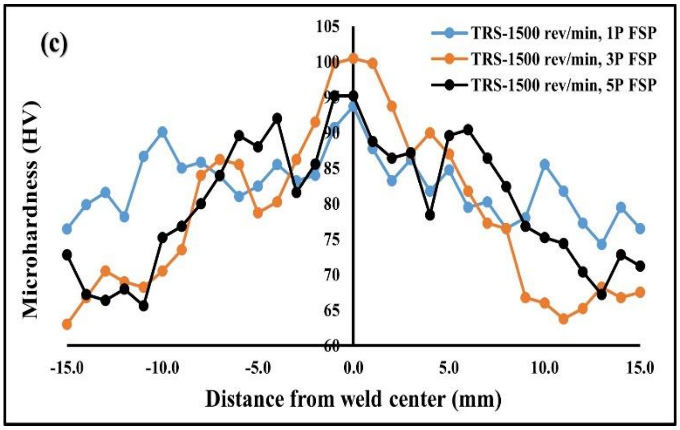


*Figure 2: Variation of microhardness of multipass FSP/SiC of A356, (a) TRS-500 rev/min, (b) TRS-1000 rev/min (c) TRS-1500 rev/min*

Additionally, the inclusion of SiC reinforcement particles in FSP further enhances A356 hardness. Maximum hardness along the centerline (SZ) was observed in the 5th pass FSP at 500 rev/min with SiC reinforcement particles. The base metal A356 exhibited a mean indentation hardness of 128 HV, which increased to 168 HV in the 5th pass FSP with SiC reinforcement particles. Dislocation density and grain refinement in MPFSP/SiC increased obstacles to dislocation movement, elevating hardness. Grain size refinement reduced grain boundary distance, enhancing material strength. Base metal and reinforcement particle dispersion in all MPFSP SZ contributed to hardness consistency. Overall, the combination of grain refinement, dislocation density enhancement, and reinforcement particle dispersion and intermixing leads to an improvement in hardness during the MPFSP/SiC process. MPFSP generates intense plastic strain and heat, which leads to DRX, resulting in very refine grains and mechanical rupture of inherent grain boundaries. The dispersion of SiC reinforcement particles in the AMC leads to an increase in hardness through the mechanism of dispersion strengthening. The SiC particles serve as obstacles to the movement of dislocations, leading to an increase in strength and hardness. Additionally, the grain refinement in the AMC due to the incorporation of fragmented and refined SiC particles also contributes to the improvement in hardness. The accumulation of SiC particles during MPFSP and their role as preferential sites for new grain formation after DRX also lead to a very refined grain structure in the 5th pass FSP/SiC. Overall, the combination of grain refinement, dispersion strengthening, and the hard nature of SiC particles leads to an improvement in micro-hardness in the MPFSP/SiC samples. The SiC particles increase the hardness of the composite by resisting plastic deformation, and dispersion strengthening occurs when dislocations interact with the reinforcement particles, causing them to resist movement and strengthen the composite. Overall, the combination of these factors leads to an enhancement in hardness during MPFSP/SiC. The fragmentation of SiC reinforcement particles during MPFSP allows for grain boundaries to migrate, leading to the materialization of a very refine grain structure. The fragmentation of SiC reinforcement particles during MPFSP allows for the grain's boundary migration and the materialization of a very refine grain structure, which further contributes to the enhancement of micro-hardness.

### *3.3 Microstructure analysis*

The microstructure analysis of A356/SiC composites was fabricated using MPFSP. Fig. 3 shows the optical microstructure of the base material A356 with coarse grains of an average size of 74±15 µm. This refinement is due to severe plastic deformation (SPD) and DRX induced by the stirring action of the FSP tool. The average grain size of the base metal and MPFSP samples was analyzed using image J software. The results show that as the FSP passes increases, the grain size decreases. Specifically, the grain size of the one-pass FSP sample was found to be 19.8 µm, whereas the grain size of the three-pass and five-pass FSP samples was found to be 11.85 µm and 2.8 µm with TRS of 1500 rpm. This suggests that multiple passes of FSP with SiC can lead to further refinement of the microstructure of A356. Overall, FSP with SiC is a promising method for refining the microstructure of A356, and further studies could be conducted to explore the potential applications of this technique in the manufacturing of high-performance components. The microstructure analysis revealed that the uniform dissemination of refine grains in the SZ led to the maximum hardness and UTS of the MPFSP/SiC samples. The SPD and DRX induced by the tool stirring action led to significant refinement of the microstructure in the SZ, TMAZ, and HAZ regions. The uniform dissemination of refine grains in the SZ led to the maximum hardness and UTS of the MPFSP/SiC samples.

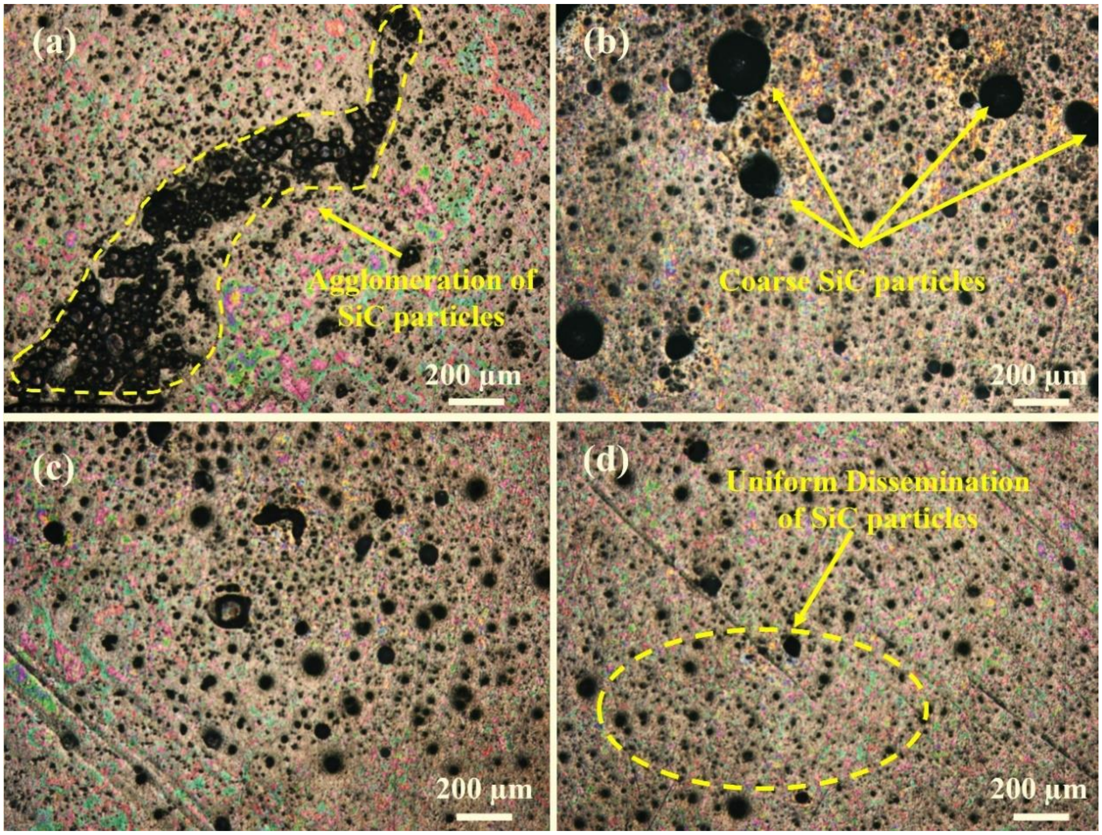


*Figure 3: Optical microstructure of multipass FSP/SiC, (a) 1Pass FSP, (c) 2Pass FSP, (d) 3 Pass FSP, (d) 5 pass FSP*

The refinement of the grain structure in the SZ during MPFSP/SiC Nanocomposite is mainly attributed to the DRX process. DRX is induced due to the intense plastic deformation and heat generated by the rotating tool during FSP. The SiC reinforcement particles dispersed in the base metal A356 play a crucial role in the refinement of grain structure. During MPFSP/SiC, the SiC reinforcement particles are fragmented, which allows grain boundaries to migrate, resulting in the materialization of refine and equiaxed grains. The accumulation of SiC particles acts as the preferred location for the formation of new refine and equiaxed grains after DRX. The uniform dissemination of refine grains in the SZ is observed due to adequate softening of the material. The MPFSP/SiC Nanocomposite shows a sharp grain structure with refine and equiaxed reinforcement particles, which are observed in the TMAZ and SZ as compared to the base metal. The refinement of the grain structure in the SZ leads to an enhancement of micro-hardness and UTS in the MPFSP/SiC Nanocomposite.

### *3.4 Fracture Analysis*

The observation of ductile failure with refine and equiaxed dimples in MPFSP/SiC samples indicates that the addition of SiC reinforcement particles enhances the ductility and toughness of the composite revealed in Fig. 4. The uniform dispersion of reinforcement particles during MPFSP/SiC results in effective material mixing and refinement of grain structure. The refine grain structure leads to an increase in the strength and hardness of the material, while the ductile failure with refine dimples suggests an improvement in the toughness and ability of the material to absorb energy before failure. The honeycomb dimples observed in one pass FSP/SiC samples indicate that the ductile fracture occurred with the materialization of micropores, which may be attributed to the inadequate dispersion of SiC reinforcement particles and incomplete refinement of grain structure. The uniform and finer microstructure of the composite with five passes FSP/SiC shows that the MPFSP process with multiple passes is effective in achieving a higher level of refinement and uniform dispersion of SiC reinforcement particles, resulting in improved mechanical properties of the composite. However, the fracture analysis also reveals that the MPFSP/SiC samples still exhibit a tendency to fail in the HAZ or TMAZ region due to the presence of coarse grains and material softening in these regions.

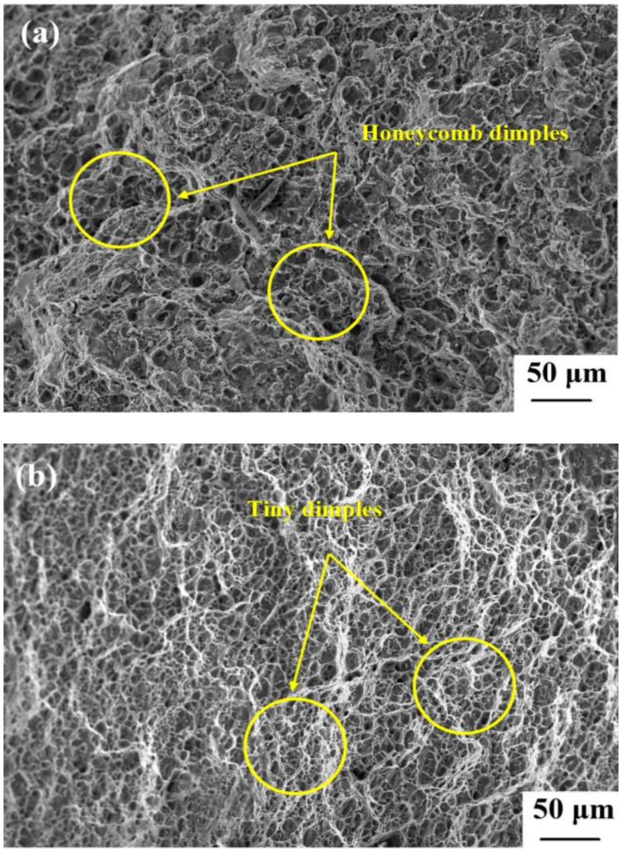


*Figure 4: SEM images of fractured specimens of FSP/A356/SiC, (a) single pass, (b) fifth pass*

## 4. Conclusions

The fabrication of MPFSP was successfully done with reinforcement particles SiC and found the following the conclusions.

- The change in microstructure of A356 to a refine recrystallized grains structure in the SZ after implementing MPFSP with SiC reinforcement particles is a result of the SPD that occurs during the FSP process. This deformation leads to DRX, which is a process of grain refinement and restructuring due to the generation of high strain and temperature.
- The homogeneous dissemination of SiC reinforcement particles is one of the key factors that contribute to the improvement of mechanical properties in the composite material. The SPD and mechanical stirring during the FSP process help to achieve this homogenous dispersion of reinforcement particles, which results in a strengthening effect and an improvement in mechanical properties such as hardness. Additionally, the reduction in grain size due to SPD also contributes to the improved mechanical properties.
- MPFSP with reinforcement particles of SiC on A356 has a positive effect on the UTS of the material. The improvement in UTS is due to the combined effect of several factors. The uniform dissemination of SiC reinforcement particles in the AMC and the reduction of grain size contribute to the strengthening effect. Additionally, the dispersion of SiC particles in the matrix increases the resistance to dislocation movement, leading to an increase in UTS. The enhancement of UTS during MPFSP/SiC is a result of the combination of these strengthening mechanisms.
- The Vickers hardness values increased as the number of FSP passes increased, with a maximum value of 126 HV observed after 5 FSP passes at 500 rev/min. This increase in hardness can be attributed to the refinement of the microstructure and the homogeneous dissemination of SiC reinforcement particles, which results in the hindrance of dislocation movement and grain boundary strengthening.
- The use of MPFSP with SiC reinforcement particles on A356 resulted in an increase in tensile properties as the FSP passes increased. The minimum UTS (264.80 MPa) was perceived at TRS of 500 rev/min with 1P FSP, while the maximum UTS maximum UTS (321.98 MPa) were observed at a TRS of 1500 rev/min, with 5P FSP. This improvement was attributed to the materialization of fine grains during DRX.
- The maximum hardness values along the centerline (SZ) were observed in 5th pass FSP with TRS of 500 rev/min with SiC reinforcement particles. The mean indentation hardness of the base metal A356 was 86 HV, while this value-enhanced up to 126 HV in 5th pass FSP with the corporation of a reinforcing agent of SiC reinforcement particles.

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