

Article

Microstructural Characterization and Mechanical Properties of AA5083/Coal Composites Fabricated by Friction Stir Processing

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Abstract: This study evaluates the development and characterization of AA5083/Coal composite joints using Friction Stir Processing (FSP) technology. The primary findings reveal significant improvements in the grain structure, with the utilization of FSP leading to an average mean grain size of 31.173 μm , representing a reduction of 50.8598% compared to the AA5083-H111 base material. This grain refinement contributed to a notable increase in hardness, achieving an average of 91.42 HV for the AA5083/Coal composite. The highest tensile strength recorded was 280 MPa, with a yield strength of 225.6 MPa. Additionally, flexural strength analysis indicated a significant difference between face and root specimens, with face specimens demonstrating a maximum ultimate flexural strength of 747.53 MPa. However, the agglomeration of coal particles and non-uniform particle distribution negatively impacted the mechanical properties, resulting in a slight reduction in the ultimate tensile strength compared to the AA5083-H111 base material. This work offers valuable insights into the fabrication and characterization of AA5083/Coal composite joints, contributing to the development of lightweight and cost-effective materials. The study underscores the importance of optimizing process parameters to minimize defects and enhance mechanical performance.

Keywords: AA5083; coal; friction stir processing; composite; microstructure; mechanical properties; tensile strength; flexural strength; hardness



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1. Introduction

Composite materials have become increasingly important across various industries due to their ability to combine the desirable properties of different constituent materials. Aluminum metal matrix composites (AMMCs), in particular, offer a compelling combination of high strength-to-weight ratio, high thermal expansion, stiffness, and excellent corrosion resistance, making them attractive for applications ranging from aerospace, automotive, and marine [1]. Among the numerous aluminum alloys, AA5083 stands out due to its favorable combination of weldability, formability, and high strength, making it a suitable choice for lightweight and high-performance applications [2,3]. However, like other aluminum alloys, AA5083 has limitations that affect its efficiency in high-performance applications. One major drawback is its relatively low hardness and wear resistance, which makes it prone to material loss under abrasive and high-stress conditions. This is particularly critical in applications where continuous friction, impact, or heavy loads are present.

Additionally, while AA5083 offers good strength compared to other non-heat-treatable aluminum alloys, it is still inferior to stronger structural materials, like steel and titanium alloys. Another challenge is the alloy's thermal stability, as its mechanical properties degrade at elevated temperatures, limiting its use in high-heat environments, such as engine components and aerospace structures.

To address these inefficiencies, a second-phase reinforcement is introduced into the AA5083 matrix to enhance its mechanical performance. By incorporating hard ceramic particles (e.g., SiC, Al₂O₃) [4], intermetallic compounds, or carbon-based reinforcements, such as coal-derived particles, significant improvements can be achieved. These reinforcements enhance strength and hardness through mechanisms like grain refinement [5–8], the Hall–Petch relationship [9–11], Orowan strengthening [12], and precipitation hardening, where dispersed particles hinder dislocation movement and refine the microstructure. Additionally, the addition of a second phase improves wear resistance, reducing material degradation in abrasive environments. For applications that require high-temperature performance, second-phase materials help maintain the structural integrity of AA5083, preventing premature softening and ensuring long-term reliability.

Friction Stir Processing (FSP) is a solid-state joining technique that has emerged as a promising method for fabricating AMMCs, derived from Friction Stir Welding (FSW) [13–15]. FSP offers several advantages over traditional fusion-based techniques, including the ability to refine grain structure and improve material homogeneity and mechanical properties [16,17]. However, processing aluminum alloys using FSP can present challenges, such as grain growth and softening in the heat-affected zone. The addition of reinforcement particles during FSP has the potential to mitigate these challenges. The reinforcement particles can act as grain refiners, promoting the formation of a finer grain structure, and as reinforcing agents, contributing to increased strength and stiffness. Furthermore, the presence of coal may enhance the wear resistance of the composite.

Numerous studies on the fabrication of AMMCs utilizing FSP with various reinforcements have been published [6–8,12,18–21]. The literature shows that the incorporation of secondary phase reinforcements via friction stir processing (FSP) in AA5083 alloys is expected to optimize mechanical properties, enhancing strength, hardness, wear resistance, and thermal stability. By refining the microstructure and acting as barriers to dislocation movement, these reinforcements improve the alloy's performance under high-stress and high-temperature conditions. This makes AA5083-based composites more durable and suitable for high-performance applications in industries such as aerospace, marine, and automotive engineering, where superior mechanical properties are essential.

Shahraki et al. [6] looked into employing FSP to create AA5083 composites enhanced with ZrO₂ nanoparticles. Utilizing optical microscopy and scanning electron microscopy (SEM), microstructural characterization was utilized to investigate the shape and distribution of ZrO₂ nanoparticles in the AA5083 matrix. Tensile and hardness tests were also used in the study to thoroughly assess the composites' mechanical properties. ZrO₂ nanoparticles were successfully integrated into the AA5083 matrix, according to the results. The addition of ZrO₂ nanoparticles significantly increased the composite's hardness and wear resistance compared to unreinforced AA5083, demonstrating the positive impact of ZrO₂ nanoparticles on the hardness and wear resistance of AA5083 composites.

Jain et al. [7] investigated the effects of incorporating various ceramic reinforcement particles—specifically, TiC, B₄C, and SiC—on the microstructure, mechanical properties, and wear resistance of AA5083 surface composites fabricated through friction stir processing. This research involved the fabrication of three unique surface composites reinforced with TiC, B₄C, and SiC particles using the FSP technique. Significant grain refinement was found in the matrix by microstructural examination, with a dense particle distribution seen

close to the stir zone's advancing and retreating sides. Remarkably, there were areas in the stir zone with bands free of particles, yet there was also good particle–matrix bonding. The microstructure observed is attributed to the severe plastic deformation induced by friction stir processing, which facilitates the mixing and refinement of the material's constituent phases. Mechanical property evaluations, including microhardness and tensile tests, revealed that the incorporation of B_4C , SiC, and TiC particles significantly enhanced the hardness and tensile strength of the AA5083 matrix compared to the unreinforced base metal. While the B_4C -reinforced composite showed bimodal fracture behavior, the SiC- and TiC-reinforced composites showed a ductile mode of fracture, according to fracture analysis. These results demonstrate how important ceramic reinforcements are to the microstructural development, wear resistance, and mechanical properties of AA5083 surface composites made using friction stir processing.

Karmiris-Obratański et al. [8] explored the effects of multi-pass friction stir processing on the mechanical, tribological, and microstructural properties of AA5083 composites reinforced with TiO_2 nanoparticles. Analysis using optical and scanning electron microscopy was conducted; the results revealed considerable grain refinement, with grain sizes reducing from 20 μm to 3 μm after the first pass and stabilizing around 7 μm after four passes. Optimal rotational speeds balanced enhanced mechanical properties and defect prevention, with mechanical tests showing significant increases in yield strength (up to 192 MPa), ultimate tensile strength (up to 359 MPa), and consistent microhardness (103 HV0.1). Energy absorption improved slightly, although it remained slightly lower than the base material's. Wear resistance was markedly enhanced with increasing FSP passes, and tribological studies indicated a reduction in the friction coefficient by up to 22.95%. Fracture mechanisms transitioned to a mixed ductile-brittle behavior from the predominantly ductile behavior of the base material.

Jain et al. [12] looked at how the speed at which the tool rotated affected the microstructure and mechanical properties of AA5083 reinforced with Fe-Al intermetallics that were produced in situ. This study adopted a different technique by using a slot in the base metal that was filled with powdered Fe-Al reinforcement. This single-pass FSP was performed at tool rotational speeds of 710 r/min, 900 r/min, and 1120 r/min, utilizing a cylindrical pin tool with a scrolled shoulder. Grain sizes inside the AA5083 matrix were finer as the rotational speed increased, according to microstructural studies. Grain refining and the in situ production of Fe-Al intermetallics greatly improved the composite's mechanical properties. The study observed that the UTS increased to 225.8 MPa after the first pass, with significant improvement in microhardness, attaining 123.3 HV as the highest value. These findings indicate that the tool rotational speed notably influences the mechanical properties and microstructure of AA5083 composites reinforced with in situ-generated Fe-Al intermetallics.

Yuvaraj & Aravindan [18] used FSP to study the tribological characterization and production of Al5083/ B_4C surface composites. This research incorporated B_4C particles, ranging from micro- to nanoscale dimensions, as reinforcing elements. Optical microscopy and scanning electron microscopy were used to analyze the microstructure of the produced composites. The research examined the influence of B_4C reinforcement particle size and the number of friction stir processing passes on the resulting microstructure and material mechanical properties. The mechanical properties were evaluated via tensile and microhardness tests, with comparisons made to the base metal. Additionally, pin-on-disk wear tests assessed the tribological performance of the surface composites. The findings indicated that a surface composite layer produced with three FSP passes and nano-sized B_4C particles exhibited superior hardness, tensile strength, and wear resistance compared to the base metal, which overall shows improvement.

Khan et al. [19] investigated the impact of inter-cavity spacing in friction-stir-processed (FSP) Al 5083 composites reinforced with carbon nanotubes (CNTs) and boron carbide (B_4C) particles. This work used a single-pass method to create hybrid surface composites by adding boron carbide particles and carbon nanotubes to the AA5083 matrix at 10 mm and 8 mm inter-cavity spacing. To determine the distribution of reinforcements and spot any flaws, microstructural characterization using optical and SEM was conducted. According to the study, inter-cavity spacing had a major impact on the distribution of reinforcement and the final microstructure. The inter-cavity spacing had a major impact on the distribution of reinforcement and the final microstructure, according to the investigation. The composite with a 10 mm inter-cavity spacing exhibited up to 38% improvement in tensile strength and 18% enhancement in performance when reinforced with B_4C particles, compared to other configurations. Additionally, U-bend ductility tests were used in the study to assess the composite's cold formability. Because of reinforcement clustering and insufficient material compensation for the cavities, composites with carbon nanotubes and an 8 mm inter-cavity spacing showed significant cracking. This highlights the vital function of material compensation in maintaining the integrity of the composite. The study concluded that a 10 mm inter-cavity spacing facilitated optimal reinforcement sinking, minimizing clustering and maximizing material compensation, resulting in better mechanical properties and increased formability in the AA5083/carbon nanotube/boron carbide hybrid composites.

Amra et al. [20] investigated the microstructural characteristics and wear performance of Al5083 surface composites reinforced with nano-sized CeO_2 , SiC, and a hybrid combination of both, fabricated via friction stir processing (FSP). Microstructural analysis was conducted in this study and microstructural analysis results revealed significant grain refinement and uniform distribution of reinforcement particles within the nugget zone. Wear resistance was assessed using a pin-on-disk wear tester, demonstrating that all composite samples exhibited improved wear resistance and hardness compared to the unreinforced base metal. Notably, the hybrid composite (Al5083/ CeO_2 /SiC) exhibited the lowest friction coefficient and the highest wear resistance, while the Al5083/SiC composite demonstrated the highest hardness, achieving a value 1.5 times that of the base metal.

Kaya et al. [21] examined how key FSP process parameters, such as axial load (6 kN, 8 kN, and 10 kN) and tool rotational speed (560 r/min, 710 r/min, and 900 r/min), affected the microstructural, mechanical properties, and wear characteristics of AA5083-H111/SiC surface composites. The investigation found that process parameters had a significant impact on the composite's properties. An axial load of 0.8 kN and a rotational speed of 900 rpm were identified as optimal parameters (Sample N8), resulting in a 38% increase in hardness and a 42% improvement in wear resistance compared to the base metal under a 15 N load. Although a slight decrease in tensile strength was observed relative to the base metal, the sample exhibited an acceptable performance level at 97%.

While these studies highlight the potential of various reinforcements, the use of coal as a secondary-phase reinforcement in AA5083, particularly processed by FSP, remains relatively unexplored. Coal, being an abundant and cost-effective resource, presents an attractive alternative to more expensive reinforcements. However, the use of coal in AMCs also poses challenges, primarily related to achieving uniform dispersion within the aluminum matrix and ensuring adequate interfacial bonding between the coal particles and the matrix. Achieving a homogeneous distribution of the reinforcement is essential for realizing the full potential of the composite material. This study investigates the effect of coal addition on the microstructure, and specifically, the hardness, tensile strength, and flexural strength of AA5083/Coal composites fabricated by FSP. This focus allows us to directly assess the influence of coal on the properties most relevant to structural applications involving bending and tensile loads. Additionally, the study assesses the potential

challenges associated with using coal as reinforcement, such as particle agglomeration and interfacial reactions.

This study's findings will help to advance the understanding of coal's potential as a feasible and cost-effective reinforcing material for AA5083 composites. This study will pave the road for the possible use of these innovative composites in a variety of industries, including marine, aerospace, and automotive, where high-performance and lightweight materials are in great demand.

2. Experimental Procedure

Plates made of aluminum alloy AA5083-H111 with a thickness of 6 mm were used in this investigation. For the purpose of ensuring that these plates are compatible with the friction stir welding (FSW) fixture, they were precisely cut to measurements of 530 mm by 70 mm. Table 1 summarizes the base material's chemical composition, which was determined using an HLC Belec Compact Spectrometer manufactured by the Belec Spectrometry Opto-Electronics GmbH, Georgsmarienhütte, Germany. Table 2 displays the AA5083-H111 alloy's mechanical properties. For this study, powdered coal particles were used as the reinforcing material. Figure 1 depicts an SEM image of the powdered coal particles, while Table 3 details their chemical makeup.

FSW was utilized to produce the weld by joining the aluminum alloy plates. A modified Lagun FU.1-LA universal milling machine, produced by Lagun Machine Tools S.L.U. in Gipuzkoa, Spain, was utilized to perform Friction Stir Welding (FSW), creating AA5083/AA5083 FSWed joints. Coal particles were added to the FSW process using a four-step procedure. The FSWed joint was first drilled using blind holes that were 4.5 mm deep and 2.5 mm in diameter. These holes extended from the joint's initiation point to its endpoint. There was a constant 15 mm gap between these holes.

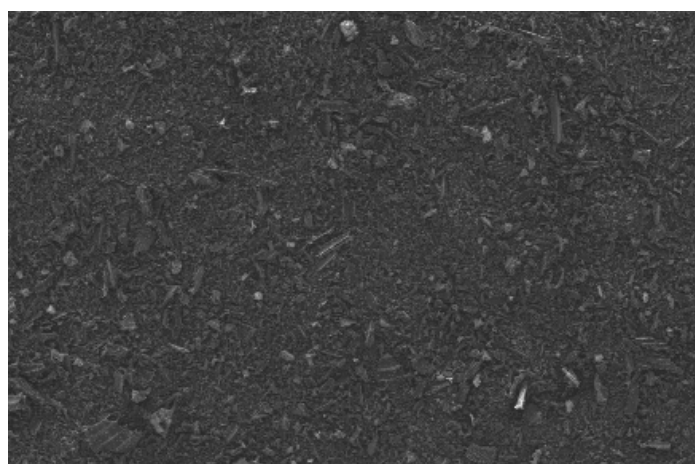


Figure 1. SEM picture of the utilized coal powder particles @ 200 μm .

Table 1. AA5083-H111's chemical composition with respect to weight.

BM	Cu	Cr	Fe	Mg	Mn	Si	Ti	Zn	Al
AA5083-H111	0.010	0.040	0.153	4.339	0.649	0.139	0.011	0.013	Bal

Table 2. AA5083-H111's mechanical properties.

Type	Tensile Strength (MPa)	Yield Strength (MPa)	PE (%)	Bending Test (MPa)	Hardness (HV)	Mean Grain Size (μm)
BM	311	248.8	58.65	415.2	90.57	61.29

Table 3. The powdered coal particles' chemical composition.

Reinforcement	O	Br	C	Si
Coal	5.4	1.3	91.7	1.6

Coal particles (5% volume) filled these blind holes entirely in the second stage. The drilled holes were then sealed in the third stage using a pin-less tool configuration. The FSP procedure was executed in the last step, employing the same tool used for the FSW operation. The process parameters and tool parameters utilized for FSW/FSP are outlined in Tables 4 and 5. Figure 2 depicts the pin-equipped and pin-less tools used in the composite fabrication process, while Figure 3 illustrates the 2D Solidworks diagram of the tools drawn using Solidworks 2024 Version.

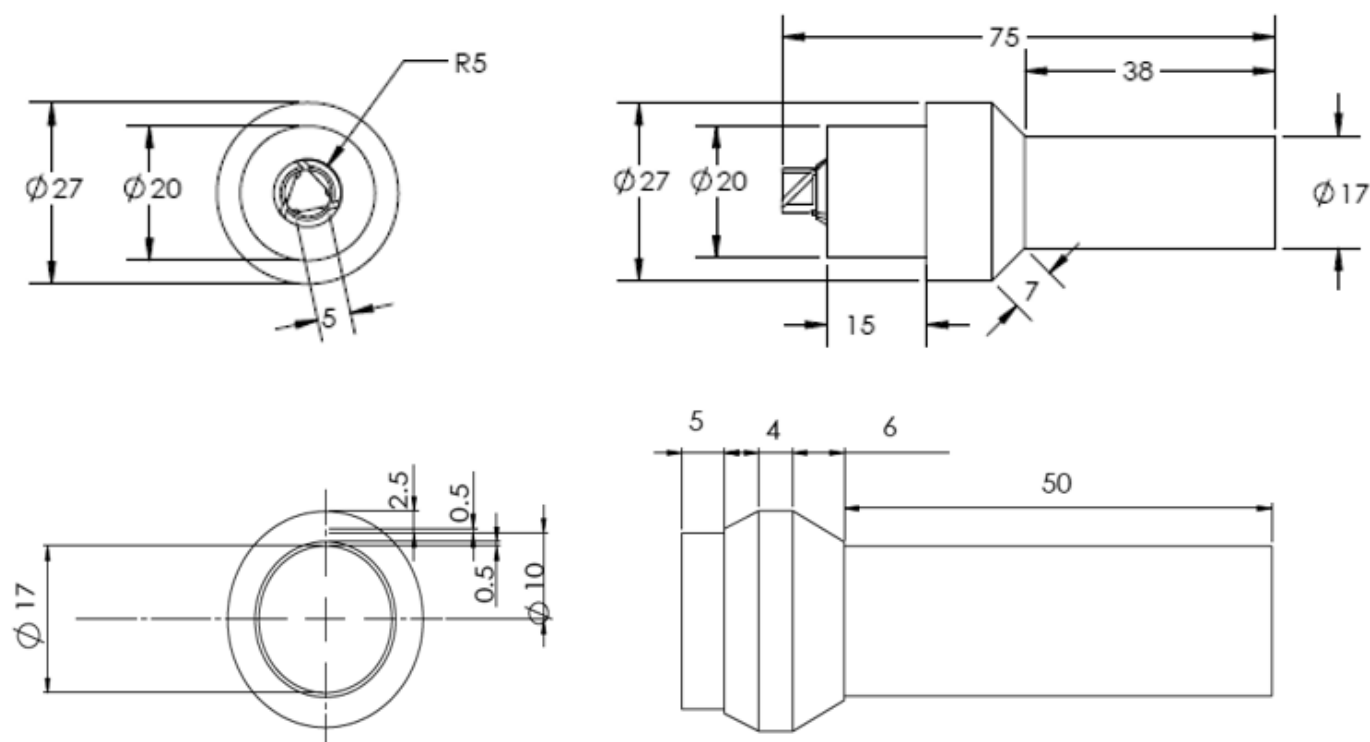
**Figure 2.** The tools with and without pins utilized in the composite production process.**Figure 3.** The tool's 2D SolidWorks diagram with dimensions in mm.

Table 4. Process parameters used for FSW/FSP procedures.

Rotational Speed (r/min)	Traverse Speed (mm per min)	Tilt Angle (°)	Axial Force (kN)	Vertical Force (kN)	Dwell Time (s)
900	60	2	4	20 tan (2) = 0.698	20

Table 5. Tool parameters.

Shoulder Diameter	Probe Diameter	Pin Length	Plunge Depth
20 mm	7 mm	5.8	5.8

To conduct microstructural analysis, bending tests, tensile tests, and hardness measurements, specimens were precisely cut from the produced plates in compliance with the designated ASTM standards. In preparation for microstructure analysis, the specimens were prepared by mounting them using a multi-fast Aka-Resin phenolic, followed by grinding, polishing and etching with a 2% aqueous NaOH solution (100 g distilled water and 2 g sodium hydroxide). The microstructure specimens' measurements are shown in Figure 4a. After etching, the microstructure specimens were examined with a Motic AE2000MET microscope manufactured by The Motic Europe S.L.U. based in Barcelona, Spain. The line intercept approach was used to measure the grain size. in accordance with ASTM E112-12 [22], and the data were analyzed using ImageJ software.

Hardness testing was carried out using an InnovaTest Falcon 500 (manufactured by the INNOVATEST Europe BV Manufacturing Maastricht in the Maastricht, The Netherlands) in line with ASTM E384-11 [23], the traditional approach for testing materials' microindentation hardness. A 0.3 kg load was applied, and indentations were made at 1 mm intervals along three parallel lines spaced 2 mm apart, resulting in 25 indent measurements per joint. A 10× objective lens was employed to focus the specimen. The experimental configuration used for the hardness indentation test is displayed in Figure 4b. The dimensions and shape of the specimens used for hardness testing were identical to those prepared for microstructural analysis. This ensured consistency in specimen preparation and allowed for a streamlined testing process, as depicted in Figure 4a.

Bend tests were conducted on face and root joint specimens to assess material ductility, adhering to the ASTM E290-14 standard [24]. When welding, the “face” is the surface that comes into contact with the tool, and the “root” is the region that comes into contact with the welding machine bed. Finally, ASTM E8M-04 was followed while conducting the tensile tests [25]. Figure 4c illustrates the dimensions of the flexural specimens, while Figure 4d depicts the tensile test specimens. All mechanical tests were conducted using a Hounsfield 25 K machine.

To explore potential differences in the material properties along the weld as seen in Figure 4e, specimens were systematically cut from the AA5083/Coal composite plate at three unique locations: the weld's start (S), middle (M), and finish (E). This method enabled a thorough study of the material characteristics and behavior by constantly analyzing the material properties at these exact places across all testing. It is important to note that only one specimen was tested for each condition (base material, start, middle, and end of the FSP joint). While this does not allow for statistical analysis, the data provide valuable preliminary insights into the relative mechanical behavior across the different regions.

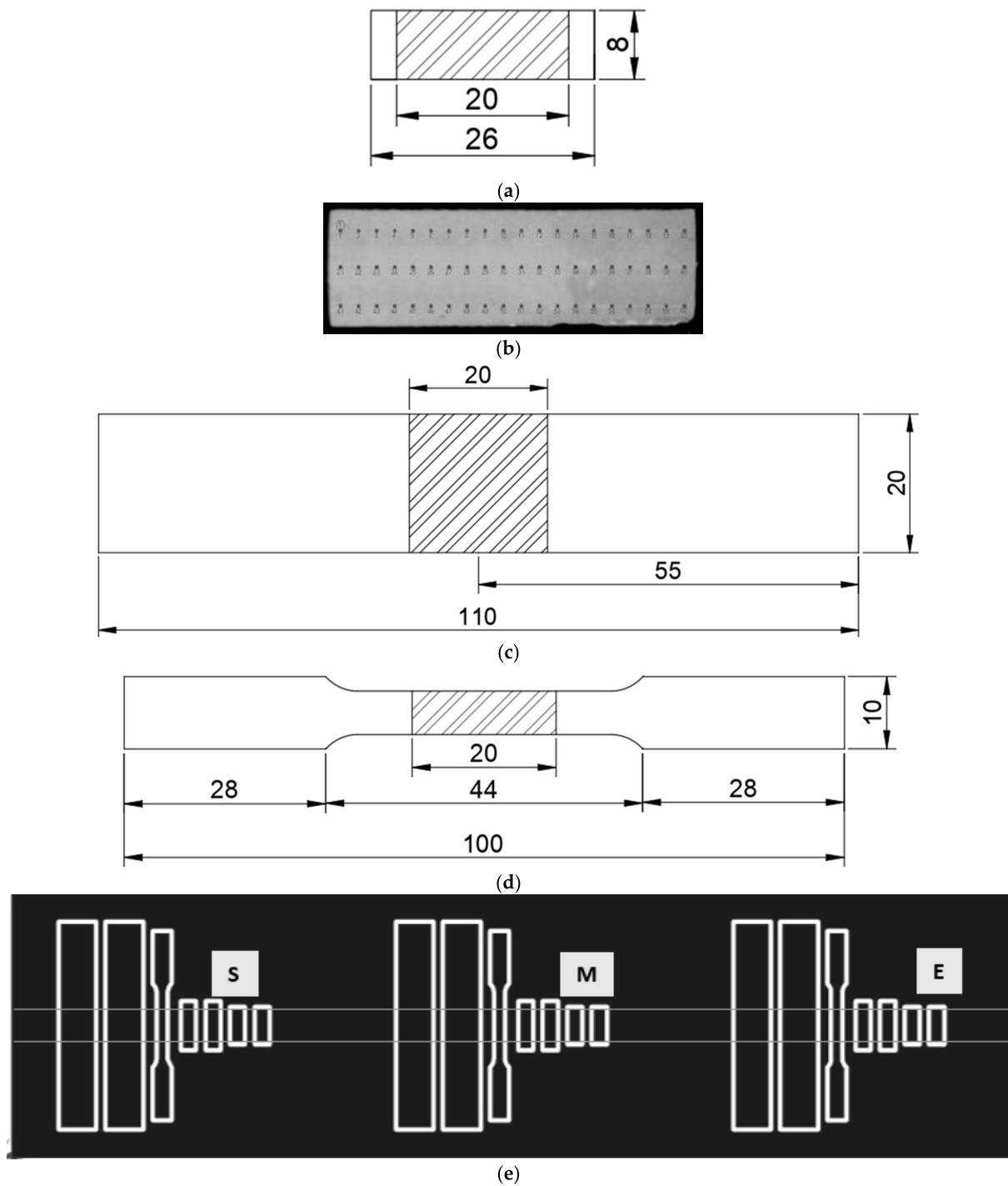


Figure 4. (a) Specimen of microstructure; (b) Setup for the hardness test; (c) Specimen of flexural test; (d) Specimen of tensile test; (e) Specimen positioning. [All dimensions are in mm].

3. Results and Discussion

3.1. Macrostructural Analysis

The nugget zone (NZ), the thermomechanically affected zone (TMAZ), and the heat-affected zone (HAZ) are all visible in the macrographs of the AA5083/Coal composite joints that are depicted in Figure 5. The microstructural behaviors and property changes going on inside the joints may be better understood with the help of these macrographs.



Figure 5. Macrographs: AA5083/Coal composite joints: (a) start, (b) middle, (c) end (scale bar is 2 mm).

Macrographic analysis revealed defects in specimens a, b, and c, including cracks, pinholes, and tunnel defects, as highlighted by red circles in Figure 5. The pinhole defects observed in specimens a, b, and c are likely attributed to inadequate heat input and incomplete material flow during the FSP process [26]. Specimen a (Figure 5) also exhibited tunnel defects, which are similarly attributed to inadequate heat input and incomplete material flow [27,28]. Furthermore, tool traverse speeds that are too fast before sufficient material deposition can contribute to void formation, leading to the development of tunnel defects. These defects can significantly compromise the composite mechanical properties [29]. Specimens b and c exhibited minor cracks, which might be related to insufficient heat generation, thermal stresses, improper cooling rates, or material brittleness [26]. Overall, processing defects, such as pinholes, cracks, and tunnel defects, are highly dependent on the FSP parameters, including the tool rotational speed, traverse speed, tilt angle, and axial force.

3.2. Microstructural Analysis

Figures 6 and 7 present optical micrographs of the AA5083-H111 base material and micrographs of the nugget zone in AA5083/Coal composite joints at a 20× magnification, which reveal the distribution of coal particles within the AA5083 matrix. As seen from the results, the AA5083 base material had an average particle grain size of 61.292 μm and a standard deviation of 10.8674 μm . The AA5083/Coal composite joints had a standard variation in grain size ranging from 4.8828 μm to 5.9413 μm and a mean grain size of 27.515 μm to 33.802 μm . The average particle grain size measured 31.173 μm . A 50% reduction in grain size observed in the AA5083/Coal composites processed by FSP can be explained by several interacting mechanisms.

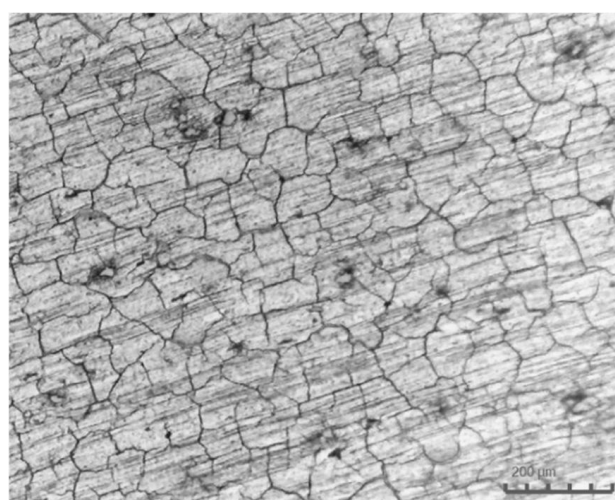


Figure 6. Parent material micrographs: AA5083-H111 @ 20× magnification and 200 μm .

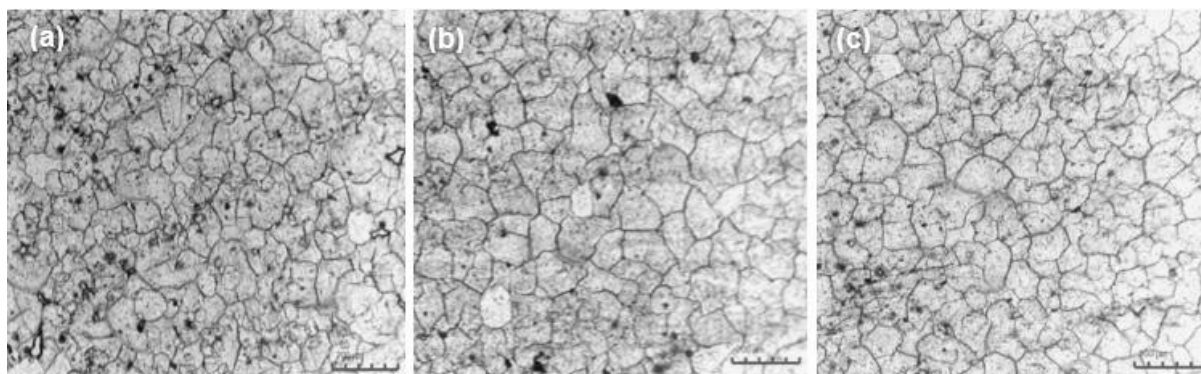


Figure 7. NZ optical micrographs @ 20× magnification and 200 μm: AA5083/Coal composite: (a) start, (b) middle, (c) end.

Primarily, the coal particles act as heterogeneous nucleation sites for dynamic recrystallization during FSP. These particles provide numerous locations for new grains to form, resulting in a finer grain structure compared to the base material. Additionally, the coal particles can pin grain boundaries, restricting their movement and inhibiting grain growth, further contributing to grain refinement. The presence of the coal particles also influences material flow during FSP, potentially promoting more intense plastic deformation and thus a finer grain structure. Finally, localized strain and temperature gradients created by the coal particles can affect the recrystallization process and contribute to the observed grain refinement [5–8]. It is important to acknowledge that the specific contributions of these mechanisms can vary depending on factors such as the size, distribution, and volume fraction of the coal particles, as well as the FSP parameters employed.

Figure 7 reveals variations in the coal particle distribution within the AA5083 matrix. While some areas exhibit relatively uniform dispersion, others show agglomeration. Despite these variations, the addition of coal significantly alters the AA5083 matrix microstructure. However, the resulting grain size of 31.173 μm, larger than conventional values, likely indicates the need for additional thermal-mechanical treatments, such as secondary severe deformation or thermal cycling, to promote finer recrystallization. Future research must explore such treatments to achieve ultrafine grain sizes. Figure 8 shows the average grain size, while Table 6 summarizes the grain sizes and standard deviations.

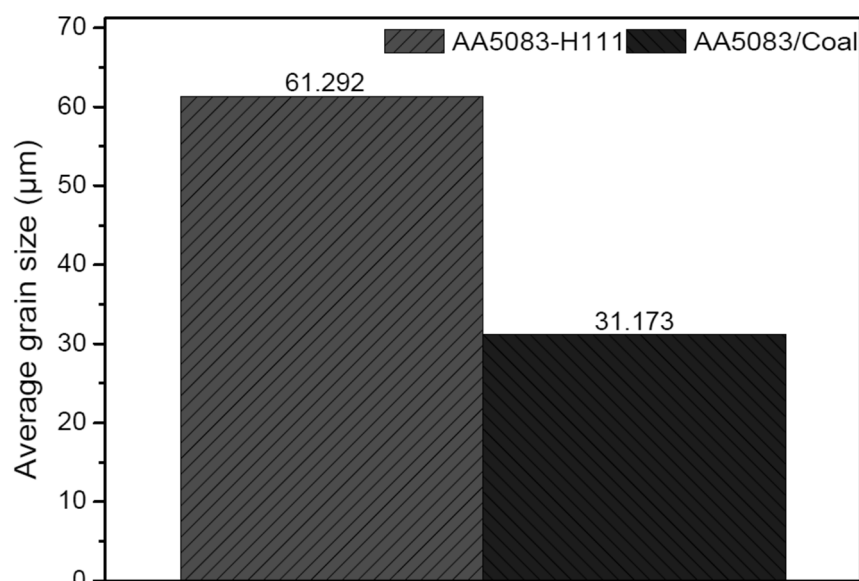


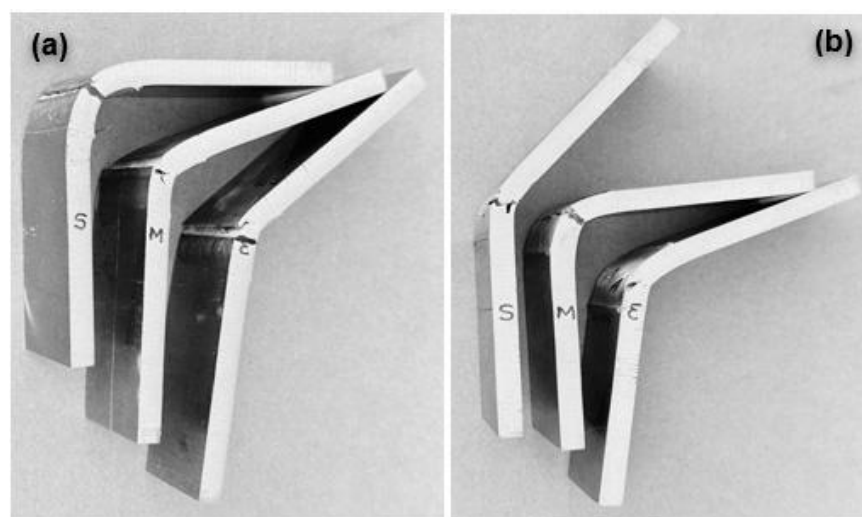
Figure 8. Average mean grain size.

Table 6. Standard deviations and grain sizes.

Type	Mean Grain Size (μm)	Standard Deviation (μm)
Base material		
AA5083-H111	61.292	10.868
AA5083/Coal composites joints		
Start	27.515	5.0464
Middle	32.203	4.8828
End	33.802	5.9413

3.3. Flexural Properties

Flexural testing of AA5083/Coal composite joints, conducted on both face and root specimens, resulted in failure within the middle of the nugget zone for all specimens. As previously discussed, examination of the post-flexure test specimens (Figure 9) revealed potential failure mechanisms, including pre-existing defects, agglomeration of coal particles, and poor interfacial bonding between coal particles and the base material. The low bending angle observed in the AA5083/Coal composite joints, along with the presence of central zone processing defects, likely stems from a combination of material properties, processing parameters, and inherent defect formation. The material's inherent properties, specifically, the tendency for coal particles to agglomerate and distribute non-uniformly, can negatively impact ductility and lead to premature failure during bending [21,30–34].

**Figure 9.** Post-bending specimens: (a) AA5083/Coal composite root; (b) AA5083/Coal composite face.

Furthermore, the friction stir processing (FSP) parameters, including the tool rotation speed, traverse speed, and axial force, play a crucial role in determining the material's microstructure and mechanical properties. Suboptimal parameter selection can result in defects and consequently reduce the achievable bending angle. These processing defects, such as cracks, pinholes, or tunnel defects, act as stress concentrators, initiating failure during bending [12,21,30–34]. To address these issues and increase the bending angle, several strategies can be employed. Material optimization focuses on improving the particle distribution within the AA5083 matrix to reduce agglomeration and enhance interfacial bonding between the coal particles and the matrix to improve load transfer.

Optimizing the FSP parameters, including the tool rotation speed, traverse speed, and axial force, is crucial for achieving a balance between heat generation, material flow, and proper consolidation, thereby minimizing defect formation. Finally, defect mitigation strategies, such as preheating the base material, optimizing tool design (pin profile and

shoulder geometry), and implementing controlled cooling strategies, can further improve material flow, reduce stress concentrations, and minimize thermal stresses, ultimately preventing cracking during the FSP process.

Figure 10a,b demonstrates the ultimate flexural strength (UFS) and strain curves for the AA5083/Coal composite joints. Flexural testing was conducted on both face and root specimens. For the root specimens, the ultimate flexural strength ranged from 410.5972 MPa to 631.9514 MPa at strain rates of 8.70588% and 22.2353%, respectively (Figure 10a). For the face specimens, the UFS range was 404.6944 MPa to 729.85 MPa at strain rates of 5.4471% and 22.9412%, respectively (Figure 10b). At the fracture point, the minimum and largest flexural strain rates for root specimens were 16.23529% and 29.4118%, respectively, whereas the range for face specimens was 14.9412% to 28%.

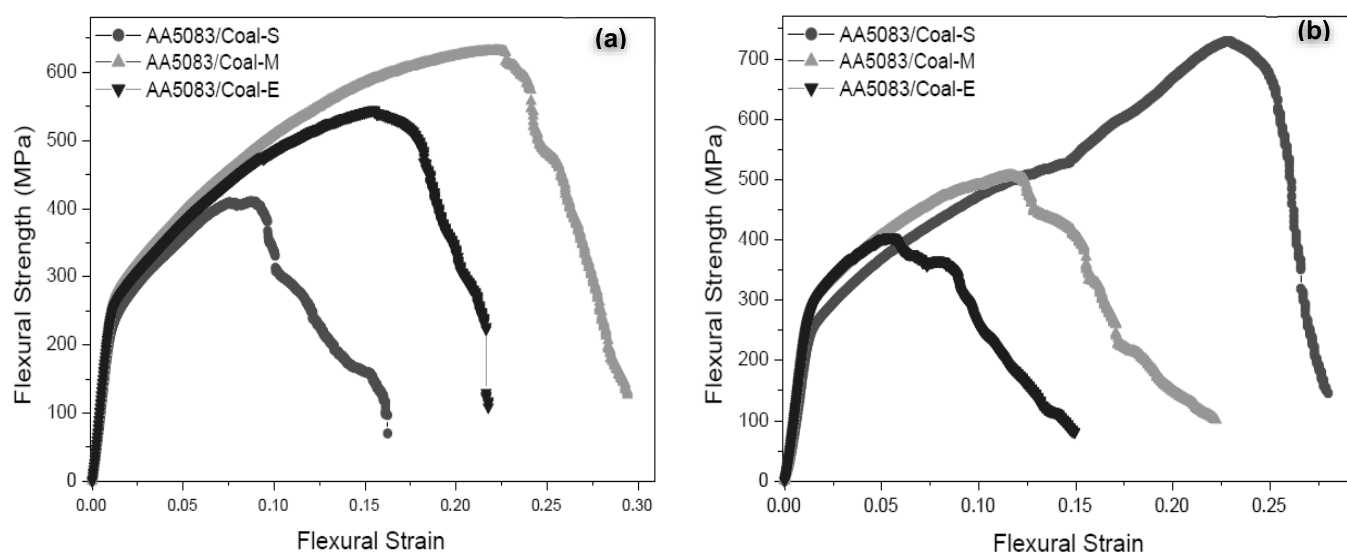


Figure 10. Flexural strength–strain curves: AA5083/Coal composite joints: (a) Root; (b) Face.

The face specimens of the AA5083/Coal composite joints exhibited higher UFS values compared to the root specimens. This observation aligns with previous research findings by Takhakh [35] and Sorger et al. [36], which demonstrated superior performance of face specimens compared to root specimens in FSP joints. While the face specimens exhibited a clear trend in flexural strength and strain, no distinct trend was observed in the root specimen results. The complete bending properties for Figure 10 are summarized in Table 7.

Table 7. Bending properties of the joints.

Type	Ultimate Flexural Strength (MPa)	Flexural Strain (%)
Base material		
AA5083	415.2014	0.268235
AA5083/Coal composite joints		
Root specimens		
S	410.5972	0.087058
M	631.9514	0.222353
E	544.8264	0.155294
Face specimens		
S	729.5833	0.229412
M	747.5278	0.228235
E	404.6944	0.054471

Overall flexural strength is a critical property for structural components in marine environments where materials experience bending loads. In this study, the AA5083/Coal

composite exhibited a maximum flexural strength of 747.5833 MPa for face specimens and 631.9514 MPa for root specimens, compared to 415.2014 MPa for the AA5083-H111 base material. This represents a 1.8005 improvement in flexural strength for face specimens and a 1.5220 improvement for root specimens.

3.4. Tensile Properties

Figure 11 illustrates the post-tensile specimens for the AA5083/Coal composite joints. Fracture analysis revealed that all specimens failed within the nugget zone, which is near the TMAZ, where the hardness is less. This suggests that the failure was initiated in the weaker regions of the composite, such as the TMAZ. The observed reduction in tensile strength was mostly due to the agglomeration of reinforcing particles of coal powder in the nugget zone. The lack of inter-particle spacing within these agglomerates [16,37,38] creates localized stress concentrations, acting as nucleation sites for crack initiation and ultimately leading to premature failure [39,40]. Furthermore, the non-uniform distribution of reinforcement particles further weakened the composite by creating stress concentrations and hindering effective load transfer [41]. Within the context of FSP, agglomeration can have a particularly detrimental effect on the nugget zone, where the extreme plastic deformation and localized heating can exacerbate particle clustering. Additionally, inadequate tool penetration during the FSP process can result in incomplete mixing and further contribute to non-uniform particle distribution and potential weaknesses within the nugget zone [12,31,32]. To mitigate this, strategies such as multi-pass FSP, vibration-assisted FSP, or ultrasonic-assisted FSP could be employed. These methods have been shown to enhance particle homogeneity, reduce agglomeration, and improve load transfer efficiency within the composite matrix.

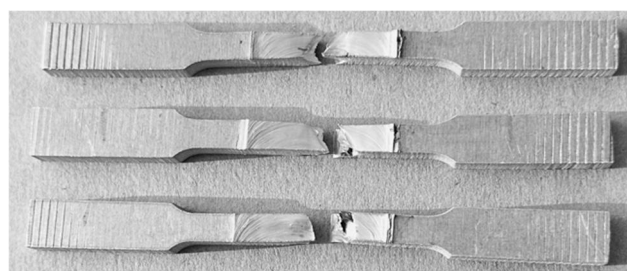


Figure 11. Post-tensile specimens: AA5083/Coal composite joints.

The tensile stress–strain curves for AA5083/coal composite joints are depicted in Figure 12. The results demonstrate that the composite achieved a maximum ultimate tensile strength (UTS) of 280 MPa, which is comparable to the 311 MPa UTS of the base AA5083-H11 material. The corresponding tensile strain rate was 33.48%, and the maximum yield strength at the start of the joint was 225.6 MPa. In contrast, the minimum UTS measured was 180 MPa, which occurred near the joint's end. The equivalent tensile strain rate was 15.17%, with a yield strength of 144.0 MPa. At the fracture point of the AA5083/coal composite joints, the highest tensile strain rate was 34.11%, and the lowest was 15.82%. There was no consistent trend observed in the tensile strength and strain rate for the AA5083/Coal composite joints. Overall, the ultimate tensile strength observed was lower by 9.9678% compared to the base material (AA5083-H111), which might be explained by the presence of these agglomerates which initiate crack formation during tensile loading, which can negatively impact the tensile strength or ductility.

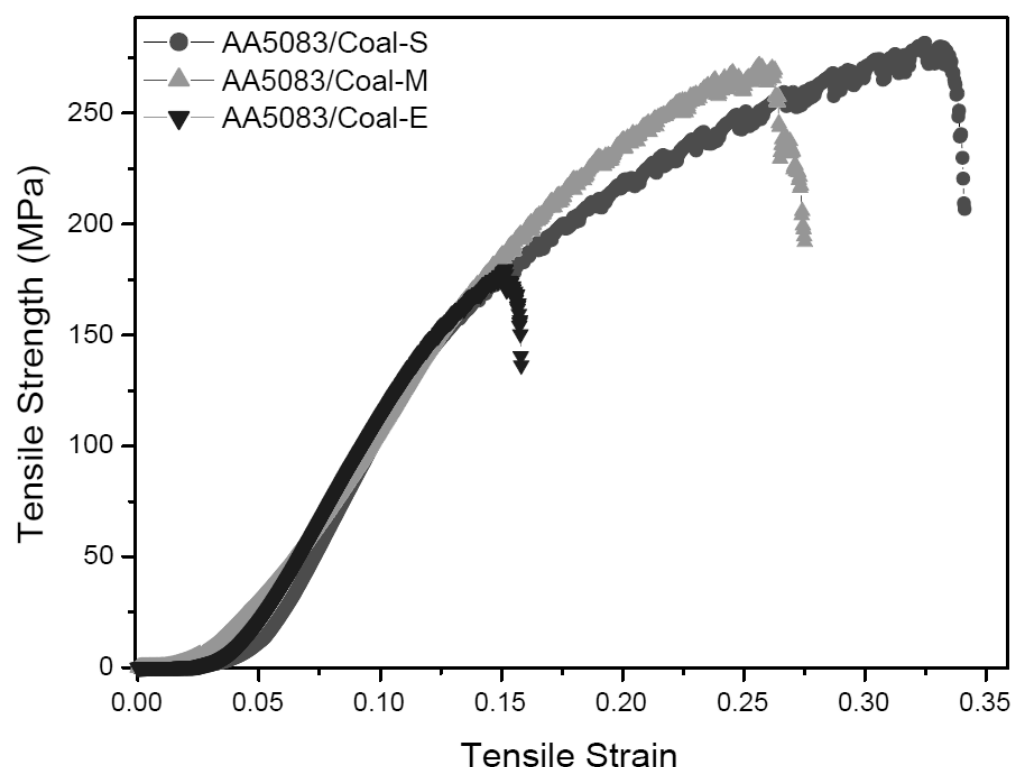


Figure 12. Tensile stress–strain curve: AA5083/Coal composite joints.

It is important to recognize that the AA5083/Coal composite is a complex system, and its tensile properties are influenced by a combination of factors, including the grain size, particle distribution, porosity, residual stresses, and potentially other localized microstructural variations. While grain size plays a significant role, the other factors mentioned above can also significantly influence the tensile properties, leading to the observed variations as seen in Table 8. Variations in the coal particle distribution, ranging from agglomerated regions (see Figure 11) to areas of more uniform dispersion (see Figure 7), create localized differences in strength due to stress concentrations [39,40]. Similarly, variations in porosity and other defects, such as microcracks, can act as stress concentrators and contribute to premature failure [39,40]. Residual stresses induced by the FSP process, although not measured here, also likely play a role. Finally, localized microstructural variations, such as differences in intermetallic particle size/distribution or texture, may exist despite a similar overall grain size and can further influence tensile properties.

Table 8. Tensile strength properties of the joints.

Type	Ultimate Tensile Strength (MPa)	Yield Strength (MPa) @ 0.2% Offset	Strain Rate (%)	Fracture Location
Base material				
AA5083	311	248.8	58.65	N/A
AA5083/Coal composite joints				
S	280	225.6	33.48	NZ
M	272	217.6	25.64	NZ
E	180	144.0	15.17	NZ

3.5. Fractography

Figure 13 illustrates the post-tensile fracture morphology of the AA5083/Coal composite joints, revealing a combination of ductile and brittle failure mechanisms. The fractured

surfaces displayed features such as microvoids, rough surfaces, dimples, and particle clusters. The balance between ductile and brittle failure was influenced by factors including the microstructural arrangement, and the extent of reinforcement particle agglomeration [42]. Ductile fracture was evidenced by the existence of dimples of different sizes, highlighted with a sample red arrow, and microvoids, indicated by a sample yellow arrow. Conversely, brittle fracture was characterized by small planes and rough patches, as marked by a sample yellow circle. These observations are consistent with prior research, which reported similar ductile-brittle fracture behavior in FSP-processed composites [8,12,21,40–45].

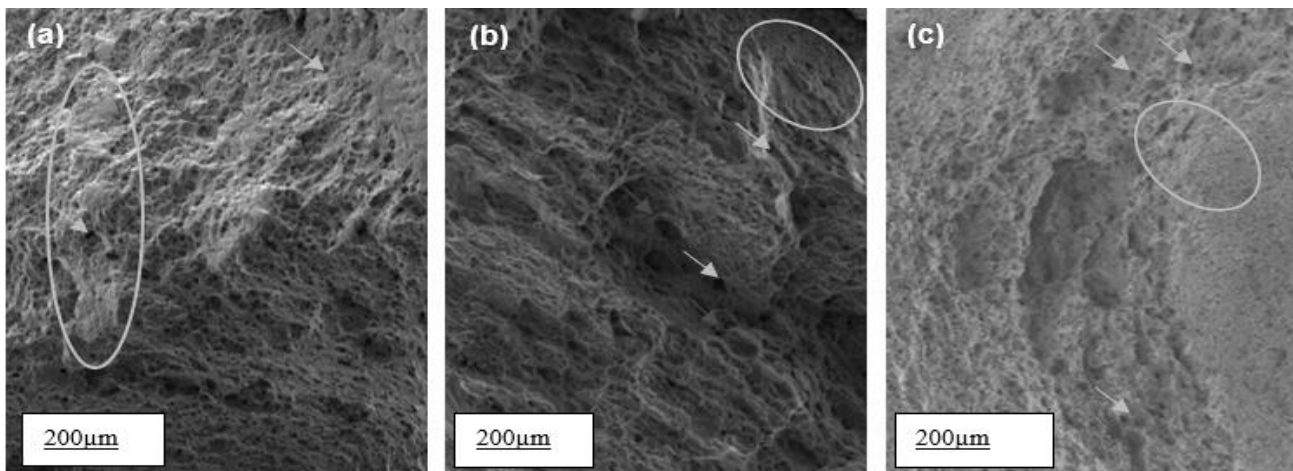


Figure 13. Post-tensile fracture morphology: AA5083/Coal composite joints: (a) Start, (b) Middle, (c) End.

3.6. Microhardness

Figure 14 presents the Vickers microhardness (HV0.3) profile of the AA5083/Coal composite joints. The hardness of the composite ranged from 89.24 HV0.3 to 91.6 HV0.3, with an average value of 91.42 HV0.3. This represents a 1% increase in hardness compared to the base material, which exhibited a hardness of 90.57 HV0.3. This improvement can be attributed to the grain refinement achieved through FSP, as the grain size was reduced from 61.292 μm in the base material to 31.173 μm in the composite [6,21,46]. This reduction in grain size leads to an increase in the grain boundary area, which provides more obstacles to dislocation motion, resulting in enhanced strengthening, as described by the Hall–Petch relationship [9–11]. While the Hall–Petch relationship suggests that this reduction in grain size should lead to significant strengthening, its effect might be less pronounced at these larger grain size values.

Furthermore, the presence of dispersed coal particles contributes to dispersion strengthening, which further increases the hardness. Micrographs (Figure 7a–c) indicate relatively good interfacial bonding between coal particles and the AA5083 matrix, which is crucial for effective load transfer and contributes to the observed increase in hardness [9,12]. However, the non-uniform distribution of coal particles and their tendency to agglomerate likely limits the overall strengthening effect. The results indicate that the nugget zone (NZ) exhibited the highest hardness values, followed by the thermomechanically affected zone (TMAZ) and the heat-affected zone (HAZ). The superior hardness of the NZ can be attributed to its refined microstructure, which is a result of dynamic recrystallization [6,21,46] and intermetallic particle fragmentation/redistribution [9,20,47]. This refined microstructure contributes to the high hardness of the NZ, as explained by the Hall–Petch relationship [9–11].

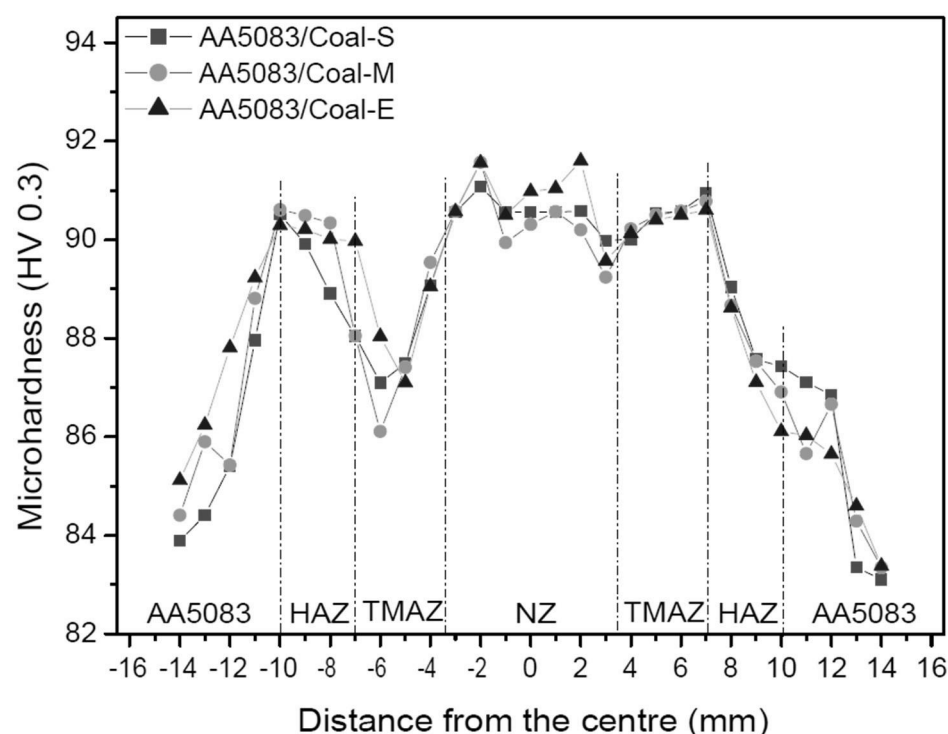


Figure 14. Hardness profiles AA5083/Coal composite joints.

While the AA5083/Coal composite joints had greater hardness values at the NZ, the hardness decreased towards the lower areas, most likely because of the decreasing intensity of the FSP process and the less homogenous dispersion of the reinforcing particles. Previous investigations have revealed similar patterns in microhardness [5,21,48]. The microstructural investigation verified the dense dispersion of coal reinforcement powder particles in the NZ, which matched the reported greater hardness values [5,7,12,31,45–47]. Figure 15 shows the related hardness summary. As demonstrated, the addition of coal reinforcement powder particles greatly improved the hardness of the AA5083/Coal composite over the AA5083-H111 base material. The Hall–Petch connection, which links hardness with grain size refinement, is the cause of this hardness increase [9–12], and the Orowan mechanism [12]. It is important to note that while Orowan strengthening might play a minor role, it is not the dominant factor.

While FSP resulted in a significant reduction in grain size (from 61.292 μm to 31.173 μm), the corresponding increase in hardness was relatively modest (from 90.57 HV to 91.42 HV). This suggests that grain refinement, while contributing to strengthening, is not the sole factor influencing the hardness of the AA5083/Coal composite. Several factors likely contribute to the limited hardness increase despite significant grain refinement. The non-uniform distribution and agglomeration of coal particles create stress concentration sites, weakening the material and hindering effective load transfer. Additionally, the FSP process may introduce porosity or microcracks, further reducing hardness. While the micrographs suggest some degree of interfacial bonding, potentially weak bonding could limit load transfer and reinforcement strengthening. Finally, the presence of residual stresses, although not directly measured in this study, could also influence hardness. These factors collectively counteract the strengthening effect of grain refinement.

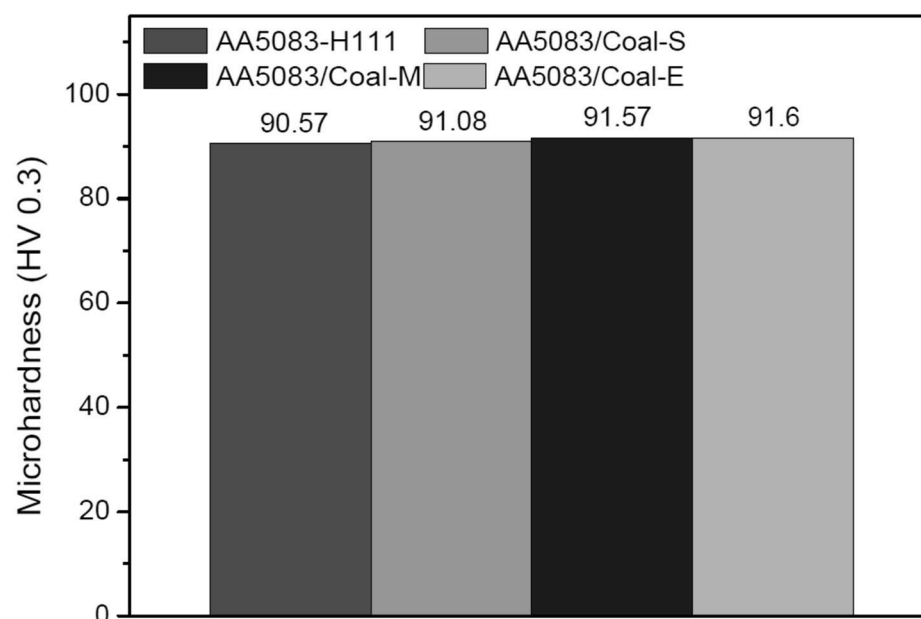


Figure 15. Microhardness average summary.

4. Conclusions

This study looked at the manufacturing and characterization of AA5083/Coal composite joints utilizing the friction stir processing (FSP) process. Key findings are as follows:

- **Microstructure:** FSP effectively induced dynamic recrystallization in the AA5083/coal composite joints, leading to significant grain refinement in the nugget zone due to dynamic recrystallization and coal particle presence. However, challenges remain in achieving uniform coal distribution, with agglomeration observed in some areas. The average grain size of the composite joints was 31.173 μm , representing a 50.86% reduction compared to the base material (61.292 μm).
- **Tensile Properties:** The AA5083/Coal composite achieved a maximum ultimate tensile strength (UTS) of 280 MPa and a corresponding tensile strain rate of 33.48%. This represents a 21.3% decrease compared to the base material's UTS of 311 MPa. Despite this reduction, the composite's tensile properties remain comparable to the base material. Overall, the tensile tests showed variations in strength across the joint, possibly due to inconsistent coal distribution and porosity.
- **Hardness:** The inclusion of coal particles increased the hardness of the composite substantially when compared to the base material. The average hardness of the AA5083/Coal composite joints was 91.42 HV, indicating a significant increase in hardness. A 50% reduction in grain size should, according to the Hall–Petch relationship, lead to a noticeable increase in hardness. The fact that the hardness only increased by roughly 1% (91.42 HV vs. 90.57 HV) suggests that other factors are counteracting the strengthening effect of grain refinement.
- **Flexural Properties:** Flexural strength was significantly higher in the composite, particularly in face specimens, indicating enhanced load-bearing capacity. The greatest UFS for face specimens was 747.53 MPa, whereas root specimens reached 631.95 MPa.
- **Failure Mechanisms:** Agglomeration of coal particles within the nugget zone was identified as a major contributor to strength reduction. Agglomerates acted as stress concentrators, initiating cracks and leading to premature failure. The composite's mechanical properties were significantly reduced due to the non-uniform particle dispersion.

These findings suggest that FSP can effectively produce AA5083/Coal composites with improved mechanical properties, but further research is needed to optimize coal

distribution and explore advanced FSP techniques for enhanced performance. Additionally, wear resistance and fatigue properties should be investigated to broaden understanding of the composites' capabilities. Overall, this study contributes to the development of high-performance AA5083/Coal composites for various engineering applications.

5. Limitations and Future Work

This study, while providing valuable preliminary insights into the microstructure and mechanical properties of FSPed AA5083/Coal composites, has certain limitations. Most notably, tensile testing was conducted with only one specimen per condition, limiting the ability to draw statistically significant conclusions. While the data suggest a possible trend of decreasing UTS from the 'Start' to the 'Middle' of the FSP joint, future work with a statistically significant number of replicates is crucial to validate these observations and establish a more robust understanding of the tensile behavior. Furthermore, this study primarily investigated a limited set of FSP parameters. A more comprehensive exploration of the parameter space, including the tool rotation speed, traverse speed, and axial force, coupled with a systematic analysis of the resulting microstructures and mechanical properties, is needed to gain deeper insights into the defect formation mechanisms and their mitigation.

Future research will explore advanced FSP methods like Laser-assisted FSP (LA-FSP), Ultrasonic-assisted FSP (UA-FSP), and Friction Stir Extrusion (FSE), alongside new assisted processes, such as Multi-pass FSP and Vibration-assisted FSP. These techniques aim to improve particle dispersion, reduce grain size, and ultimately enhance the composite's mechanical properties. The research objectives should include investigating the effects of these advanced methods on microstructure, mechanical properties, and wear behavior, optimizing process parameters for each method, comparing performance against traditional FSP, and developing a comprehensive understanding of the relationships between process parameters, microstructure, and properties. Expected outcomes after addressing these limitations include the development of high-performance AA5083/Coal composites with superior properties, optimized fabrication processes, a fundamental understanding of structure–property relationships, and the potential for wider industrial application.

Additionally, future work will address the challenges related to particle agglomeration and non-uniform distribution observed in the current study, which negatively impact the mechanical properties.

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