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Characterization of AA6061-ZrO_2-C friction stir welded composite joints

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In present work, investigation of metallurgical and mechanical characterization of Friction Stir Welded MMC material, the influence of micro structural properties on mechanical properties using different heat inputs were conducted. These heat input generation depend on the FSW process parameters such as Tool Rotational Speed, Welding Speed and Axial Load. The FSW process parameters were set such as the tool rotational speed in the range of 800 to 1000 rpm, axial load in the range of 4 to 6 kN and a constant welding speed 50 mm/min to fabricate the FS welded specimen of MMC materials. These process parameters play a major role in determining grain growth, phase transformation and change in microstructure of different zones of weldment like welded nugget zone (WNZ), thermo- mechanically affected zone (TMAZ), heat affected zone (HAZ) and base metal (BM). Moreover, the effects of changes in the microstructure help in improvements of mechanical properties like tensile strength and hardness. The weld joints obtained with medium heat input produced the fine grains and maximum tensile strength of 198 MPa and hardness of 56 HRB at the tool rotational speed of 800 rpm, welding speed of 50 mm/min and axial load of 5 kN. The optical microscope (OM) and EDX analysis are employed to identify the presence of the matrix, reinforcement particulates in HMMCs and to confirm defect free welds attained at the WZ, TMAZ and HAZ during the medium heat input condition.

Keywords: MMC, FSW, Metallurgical Characterization, Mechanical Characterization

Introduction

Advanced lightweight engineering applications like Aerospace, Automotive, Energy, Military, Electronics and Packaging industries mostly make use of Friction Stir Welding process (FSW). The FSW is a low heat input solid-state welding process invented by The Welding Institute, the UK in 1991 [1]. The FSW process produces plastic deformation in the material by generating an immense amount of heat. The welding process of aluminium based MMC is very delicate due to aluminium being difficult to weld than other metals. However, in the case of aluminium based MMC, joints made by Fusion Welding processes possess many defects and are not suitable to serve its purposes. Based on the previous research work carried out on fusion welding on Aluminium Matrix Composites (AMCs), both aluminium alloy matrix and hard ceramic reinforcing phase create some problems during fusion welding like high thermal expansion, high thermal conductivity, solidification shrinkage, incomplete mixing of parent and filler materials, coarse grain structure at weld zone due to high heat input, high solubility of gases in the molten state, presence of oxide inclusions [2-4]. To this end, several investigations were carried on the FSW of Aluminum Metal Matrix Composites (AMMCs) and its welding process parameters, studying their microstructures and mechanical properties of these joints.

Li et al. [5] investigated the microstructure and mechanical properties of friction stir welded B_4C / Al6061 joints, and found the uniform distribution of B_4C reinforcement in the excited zone. Compared with base metal (BM) grain size, the weld nugget zone (WNZ)'s grain size was smaller due to this nature and better mechanical properties were observed in the WNZ. Shojaei et al. [6] studied the microstructure and mechanical properties of FSW'ed aluminum based MMC with bronze material joints, the improvement of mechanical properties from 10 to 15% in FS welded joints. Also found the WNZ's microstructure was completely differed from BM's microstructure. Guo et al. [7] investigated the microstructure and mechanical properties of dissimilar FSW'ed AA1100 / B_4C MMC and AA6063 alloy joints, found better improvements in mechanical properties such as tensile strength and hardness. The maximum hardness and different grain sizes were observed in the WNZ, also studied single – sided FSW of thin or dense AMMCs plates. Satish et al. [8] studied and reported the effects of FSW process parameters of 5083 aluminum alloy joints, also known WNZ had achieved better mechanical properties with fine grain size. Vijayavel and Balasubramanian [9] investigated the effects on mechanical properties for different tool pin profile designs and justified the

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square pin profile which produced superior tensile strength compared with other design of profile pins. Seyed et al. [12] studied the microstructure (OM, SEM and EDS) and mechanical (microhardness and tensile strength) properties of friction stir welded Mg-$_{12}$Li-$_{1}$Al Alloy. The simultaneous improvement in grain structures (19 μm to 73 μm) was to improve the hardness and tensile strength properties up to 13%.

The survey revealed the most common types of reinforcement materials such as TiC, Tib_2, B_4C, Al_2O_3, SiC, Si_3N_4 etc and it also found that the including usage of ZrO_2 based reinforcement materials in Aluminium MMC were limited. Based on the technical gaps noted from the literature survey, the objectives have been framed for the present investigation on enhancement of mechanical and metallurgical characterization of Friction Stir Welded AA6061- ZrO_2-C AMMC. The present work consists, the investigation of metallurgical and mechanical characterization of friction stir welded MMC material, study of the influence of micro structural properties on mechanical properties in the effect of three different heat inputs generation such as low, medium and high of FS welded MMC materials. The range of FSW process parameters were set as the follows: Tool Rotational Speed of 600 rpm, 800 rpm and 1,000 rpm, a constant Welding Travel Speed of 50 mm/min and Axial Load levels of 4 kN, 5 kN and 6 kN to evaluate the optimum heat input conditions and to obtain superior mechanical properties (tensile strength and hardness) to produce a defect free joint. Stir Casting method was used to fabricate the 92 wt% of AA6061, 6 wt% of ZrO_2 and 2 wt% C of MMC materials.

Materials and Methods

MMC Materials preparation

A 92 wt% of AA6061 aluminum alloy was used in this investigation along with 6 wt% of Zirconium Di-oxide (ZrO_2) as the primary reinforcement particulate and 2 wt% of Graphite (C) as the secondary reinforcement particulate, the primary reinforcement particulate of ZrO_2 is more sought after in the multi-industrial and technological applications due to its low co-efficient thermal expansion, low cost and high thermal conductivity [10], also it possess hard refractory ceramic quality and better compatibility with aluminum alloys. For example, ZrO_2 has a thermal conductivity of 175 W/mK which is less than the 250 W/mK for SiC. In this regard, the materialistic stability of ZrO_2 is better because it is more heat resistant than SiC. In addition, ZrO_2 possesses hard refractory ceramic compatibility with Al alloy. The secondary reinforcement particulate, Graphite (C) has much flexibility (though not elastic), valuable electric as well as thermal conductivity and high wear resistance. The specification of reinforcement particulates is of 99.5% purity, 11-39 μm particulate size of ZrO_2 and 98% purity, a 325 mesh size for C to fabricated the MMC materials.

To fabricate the aluminum metal matrix composite (AMMC) by stir casting method, AA6061 was chopped into smaller pieces and placed inside the induction furnace crucible. It was melted at a furnace temperature of 850 °C. In this molten stage the primary and secondary reinforcement particulates were gradually mixed with AA6061 matrix before mixing the particulates it were preheated to 350 °C for removed moisture contents. The semi-liquid composition was stirred well for the time duration of 10 minutes at constant speed of 450 rpm to obtain a homogeneous distribution of particulates in the mixture [11]. Then the stirred semi-liquid composition material was poured into a rectangular mold to cast the required number of work pieces in the size of 100 × 50 × 6 mm.

Friction stir welding of MMCs

The stir cast MMC plates were successfully welded by friction stir welding (FSW) process by using square profile tool as shown in Fig. 1. There are three main process parameters that highly influence the quality of FS welded joints in MMC such as tool rotational speed, welding speed and axial load [12-14]. The process parameters and their respective levels are presented in Table 1.

Heat input calculation

The square pin profile design tool was used to fabricate the FS welded MMC material to cause the plastic deformation in the stirred zone by producing an immense amount of heat during at FSW process. As a result, the micro structures' changes were observed in the weldments. Eqs. (1) and (2) were used to calculate the amount of total torque and heat input values, and Eq. (3) were employed for finding the average heat input under the tool shoulder pin [15]. The total frictional torque and heat average input were developed by the tool rotational speed (rpm) are given by

$$T_{Total} = T_S + T_P \quad (1)$$
$$T_{Total} = 2\mu F\,(r_o/3 + r_i 2/r_o 2 * h) \quad (2)$$
$$Q_{in} = \eta\,(2\pi NT/f) \quad (3)$$

where, T_S = Torque generated by the tool shoulder (in N-m), T_P = Torque generated by the tool pin (in N-m), μ = Co-efficient of Friction, F = Applied Axial Force (in N), r_i = Radius of the tool shoulder (in m), r_o = Radius of the tool pin (in m), h = height of the tool pin (in m), T_{Total}= Total torque (in N-m), η = Efficiency, N = Tool rotational speed (in rpm), f = welding speed (in mm/min) and Q_{in}= Average heat input (in kJ/mm). Table 1 presents the amount of heat generated by the FSW tool for different welding conditions, also each welding conditions and corresponding amount of heat generations were influenced in grain growth, phase transformation in the microstructure and hardness of

Table 1. FSW process parameters and their responses.

Sample (Heat I/P)	Tool Rotational Speed (rpm)	Welding Speed (mm/min)	Axial Force (kN)	Heat Input (kJ/mm)	Tensile Strength (MPa)	Weld Nugget Hardness (HRB)
1 (low)	600	50	4	24.922	73	46
2 (Medium)	800	50	5	41.537	198	56
3 (High)	1000	50	6	62.306	124	49.5

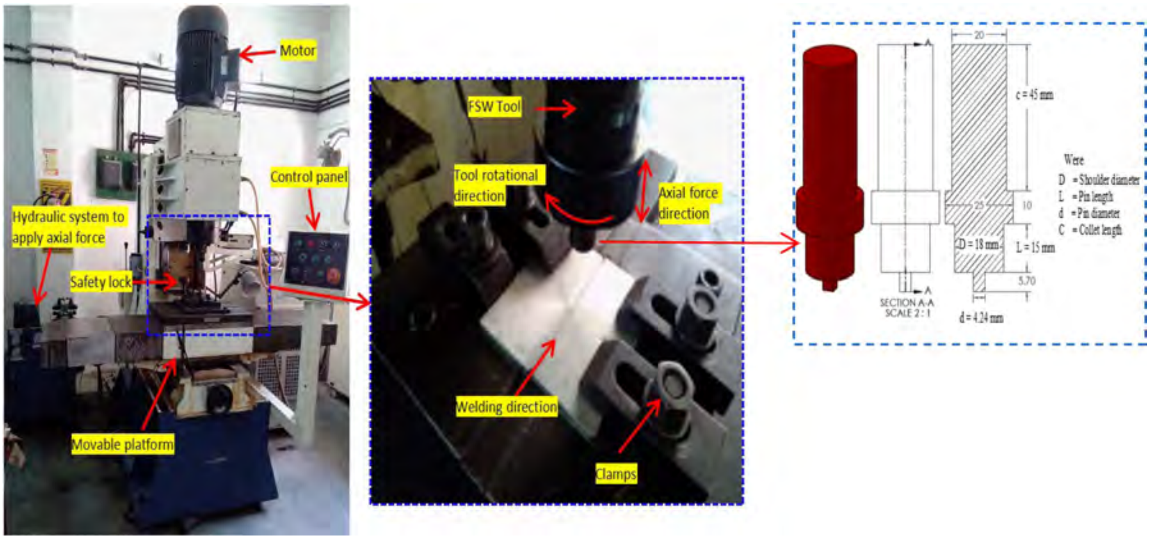

Fig. 1. Experimental setup of FSW.

welded and heat-affected zones. In this study, three levels of heat inputs were considered to better understand the effects of heat input in metallurgical characterization. From Table 1, they can be seen as, 24.922 kJ/mm as low, 41.537 kJ/mm as medium and 63.306 kJ/mm as high level of heat inputs generated by FSW tool.

Metallurgical and mechanical properties

The metallurgical study was carried out along various zones across the cross sections of FS welded specimens by using of a metallurgical microscope in different magnification ranges. The chemical composition of the AMMC's samples was detected by using of SEM with EDX spectrum analysis. The FS welded MMC specimen in the size of 15 mm × 15 mm × 10 mm was cut and polished with emery sheets, having the grits in the range of 220 to 1,200. The specimen surface was further polished using a diamond polishing disc of 6 μm, 3 μm, and 0.5 μm. Further polishing was made again by applying the alumina solution to give a glossy finish to the specimen. Finally, the specimen was etched with the help of Keller's agent. The specimen was well prepared to observe the Base Composite (BC), Heat Affected Zone (HAZ), Thermo Mechanically Affected Zone (TMAZ) and Welded Nugget Zone (WAZ) under different magnification levels (100X / 100 μm, 100X / 50 μm, 200X / 50 μm) on an optical microscope and EDX analysis (Zhang et al., 2015; Vijay et al., 2010).

The FSW joints are tested for their mechanical properties as per ASTM standards E08 M-04 for the tensile test and E10 for the hardness test. The tensile test characterizes the elastic-plastic behaviour of a material by applying a uniaxial tensile load. All the tensile tests are conducted in a displacement control mode at a rate of 0.1 mm/min. The localized strain variations in the tensile samples were measured using an extensometer, it is used to measure the change in length of the FS welded MMC. Engineering stress-strain relation is calculated for each specimen.

The hardness test specimen samples were made in rectangular shape using the wire-cut EDM process, as per the specification mentioned earlier (15 mm × 15 mm × 10 mm). These samples were placed on the hardness testing machine and subjected to an axial load of 500 g using a ball indenter for a dwelling period of 10 s. The spherical indentation induced by the 1/16' diameter indenter was measured on various locations in the sample surface. The micro hardness survey is made at different positions in various zones of welded joints.

Results and Discussions

Microstructure friction stir welded MMCs

The Scanning Electron Micrograph (SEM) image of 92% Wt% of AA6061, 6 Wt% of ZrO_2 and 2 Wt% of C particulate stir cast aluminum matrix composite materials is shown in Fig. 2. It clearly shows the uniform dispersion of reinforcement particulate in stir cast

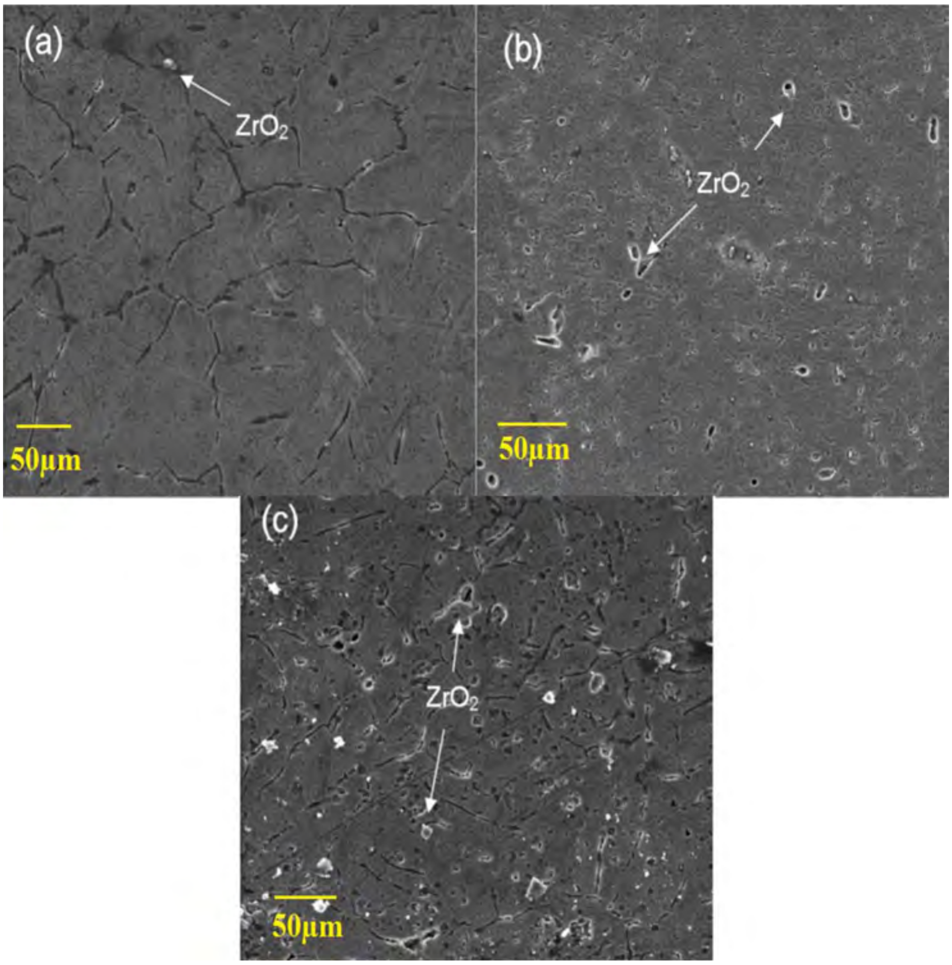

Fig. 2. SEM micrograph of stir cast MMC material (a) sample 1, (b) sample 2, (c) sample 3.

aluminum AA6061, ZrO_2 and C reinforced composite materials. It is noticed that the particulate agglomeration was reduced considerably. It makes sure that proper stirring was employed during fabrication of MMC. The random orientation of particulates provides uniform strength to cast material in all directions.

Fig. 3 shows that the optical microscopy images of friction stir welded joints with different zones like base metal (BM), thermos-mechanically affected zone (TMAZ), weld nugget zone (WNZ) and heat affected zone (HAZ), also represents the size, shape and distribution of the grain structure of reinforced particulates. Compared all samples, the sample 1 weld zone had poor penetration and columnar dendrites along with pores and it causing lower tensile strength. Also it has more recrystallized structure in the TMAZ surface. Sample 2 & 3 obviously shows that the cluster and even distribution of C particulates in the FSW'ed WNZ surface that occurred throughout the matrix, similarly ZrO_2 particulates were also evenly distributed. The FSW'ed MMCs of WNZs were presenting columnar dendrites towards the welding direction, and the TMAZs were found that the distribution of particles across the matrix was very fine and uniform. But some amount of coarsening grains was observed in the friction stir welded TMAZ surfaces of samples 1 & 2. Compared with sample 1, samples 2 & 3 ensuing improved mechanical and metallurgical properties of the welded joints. The SEM with EDX spectrum analysis clearly shows the presents of other elements like Al, Zr, SiC, Mg, C, etc. Fig. 4(a-c) shows the SEM image of the weld nugget interface zone and also shows the matrix and reinforcement particulates in friction stir welded MMC and highlights the defect-free joints of the cross weld. The SEM analysis of FS welded MMC illustrates the other weld zone like WNZ, TMAZ, HAZ and BM. As observed, in comparison to BM, the stir zone produces finer grain structure.

The matrix of this zone exhibits a fine grain size and has low segregation. The grain structure is evenly dispersed in this case when compared to Low heat input and High heat input conditions. Furthermore, as observed in section (ii) of Fig. 3, the thermo mechanically affected zones of the matrix under Low heat input and High heat input conditions are very much affected by the process parameters, thereby resulting in uneven particulate dispersion. The composite matrix under the Medium Heat input condition has better reinforcement

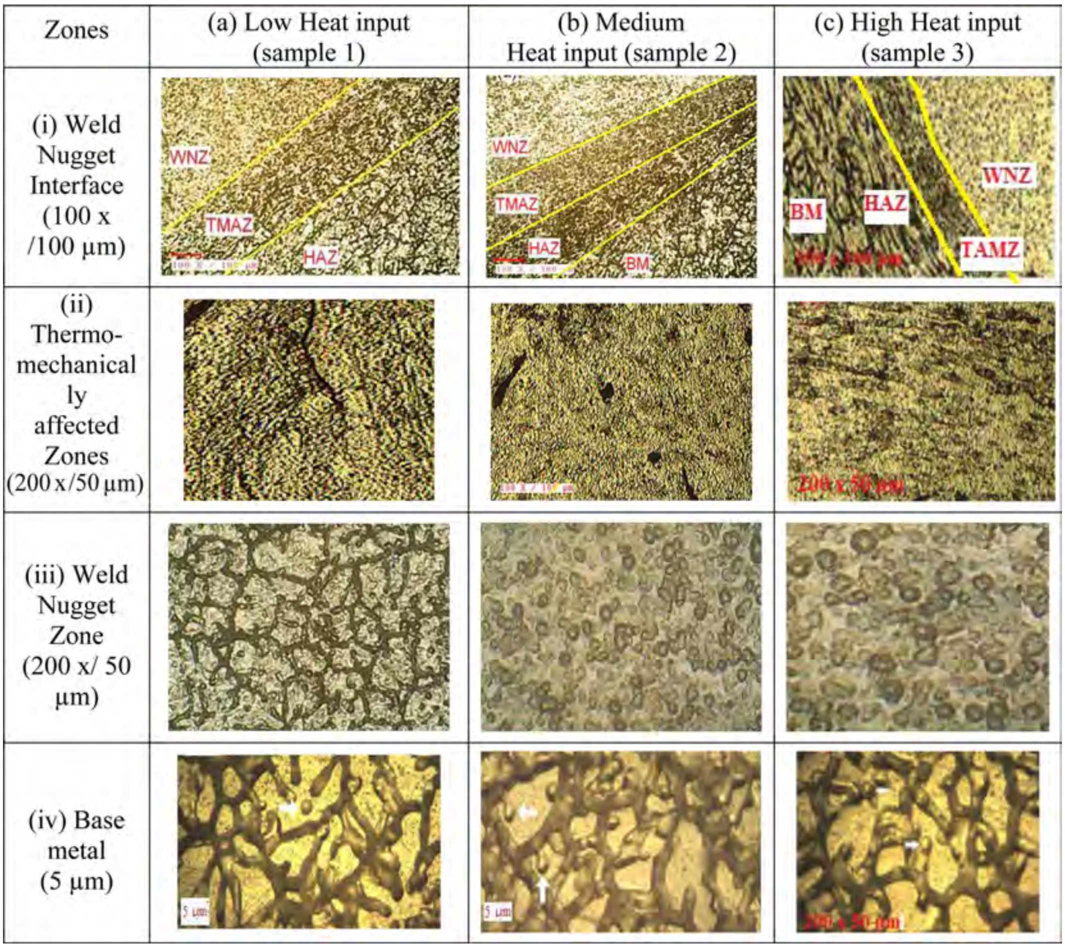

Fig. 3. Micro structural analysis FS welded MMC (a) sample 1, (b) sample 2, (c) sample 3.

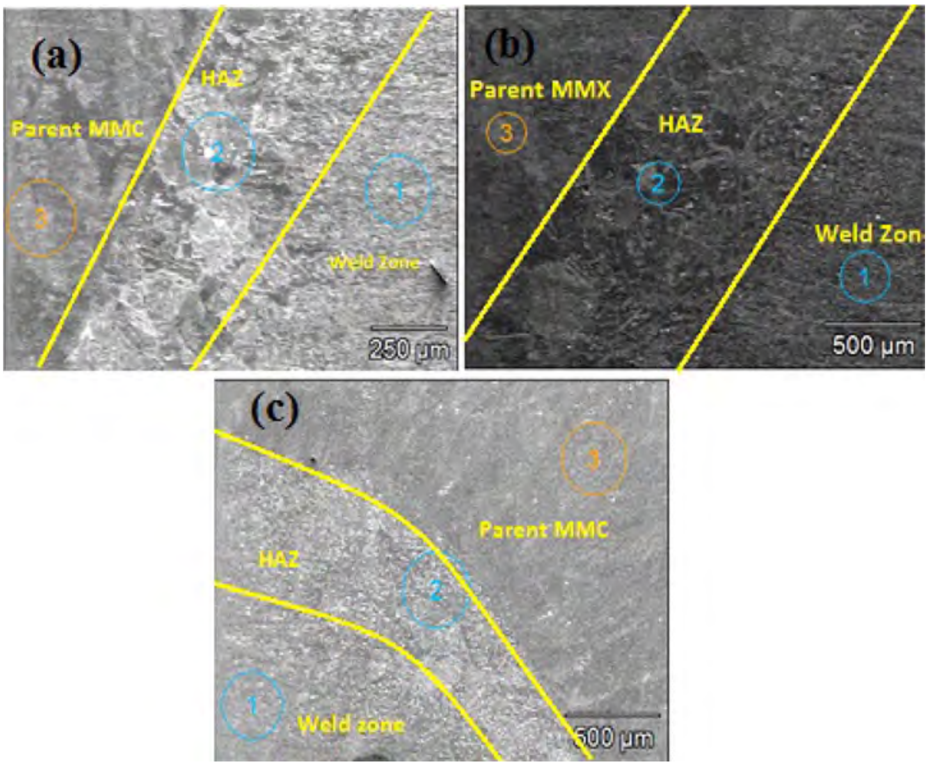

Fig. 4. SEM micrographs of FSW’ed MMCs of (a) Sample 1, (b) Sample 2, (c) Sample 3.

grain structure dispersion and fewer particulate dislocations. Section (iii) of Fig. 3 compares the Weld nugget zone of the composite matrix under Low, Medium and High heat inputs. Here the Medium heat input zone is observed to have better phase dispersion between the base metal matrix and particulate reinforcements. Section (iv) compares the base metal under similar conditions as above, therefore there are no significant differences observed in cases of heat input.

Chemical composition

The chemical composition of the FSW'ed MMCs was measured in the WNZs, and it presented the amount of alloying elements. Fig. 5 shows the XRD analysis of FS welded AMMC with different heat input conditions like low, medium and high. It also shows the presence of reinforcement particulates in FS welded AMMCs materials. Fig. 6(a-c) shows the spectrum analysis of three samples, and the presence of matrix and oxides can also be noticed. The zones are marked in SEM image Fig. 6(a-c) at lactation 1, 2 and 3. Table 2(a-c) shows the presence of elements in FS welded MMC at three different weld zones. The EDS analysis clearly shows the present alloying elements such as Al,

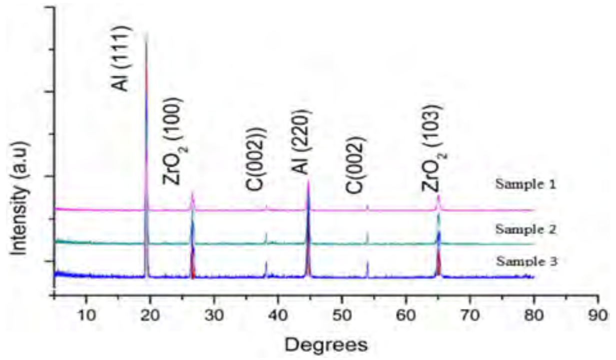

Fig. 5. XRD analysis of FSW'ed MMCs of (a) sample 1, (b) sample 2, (c) sample 3.

Table 2(a). Identified elements in low heat input of FS welded MMC.

	C-K	*Si-K*	*Al-K*	*Mg-K*	*Zr-L*
Location1	----	0.8	92.33	3.83	3.84
Location2	0.62	0.2	92.08	4.19	3.12
Location3	0.81	0.78	88.84	7.22	3.14

Table 2(b). Identified elements in medium heat input of FS welded MMC.

	C-K	*Si-K*	*Al-K*	*Mg-K*	*Zr-L*
Location1	0.57	0.28	92	4.50	2.93
Location2	0.70	-	91	4.84	3.46
Location3	1.02	-	87.97	7.18	3.83

Table 2(c). Identified elements in high heat input of FS welded MMC.

	C-K	*Si-K*	*Al-K*	*Mg-K*	*Zr-L*
Location1	0.99	0.62	91.05	4.19	3.15
Location2	0.86	0.77	90.66	4.41	3.3
Location3	--	---	88.26	8.03	3.72

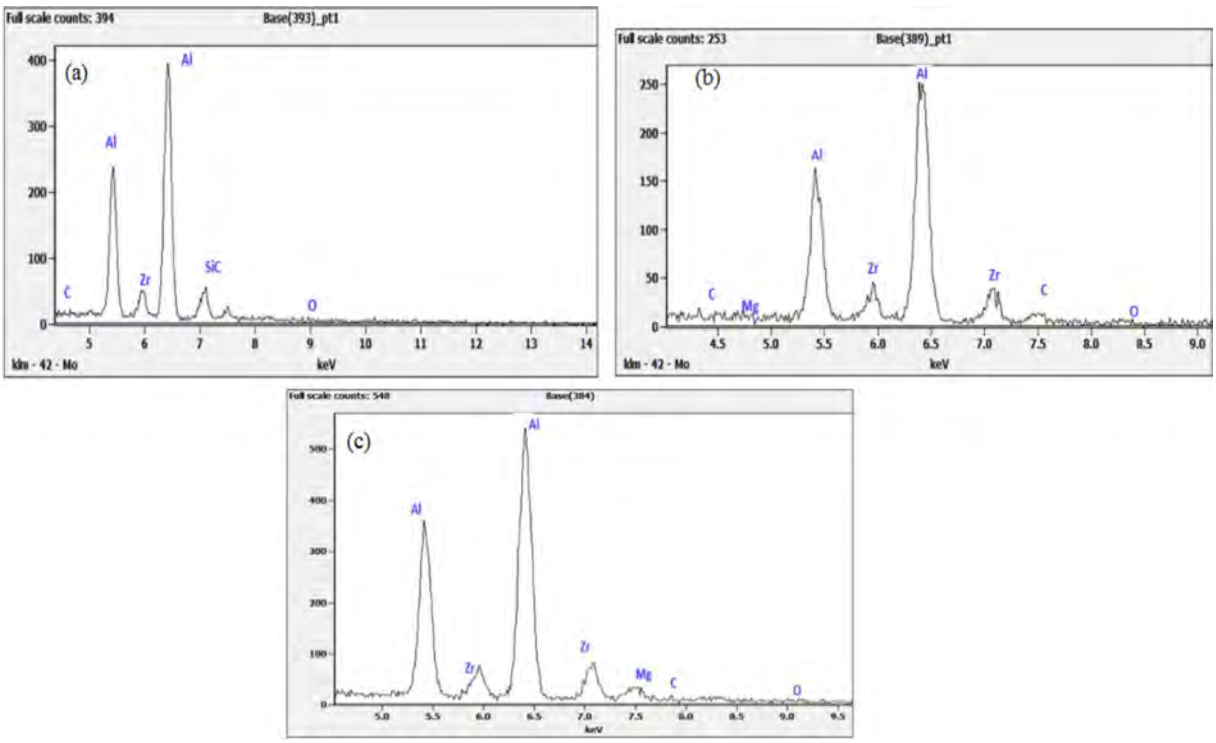

Fig. 6. Spectrum analysis of FSW'ed MMCs of (a) sample 1, (b) sample 2, (c) sample 3.

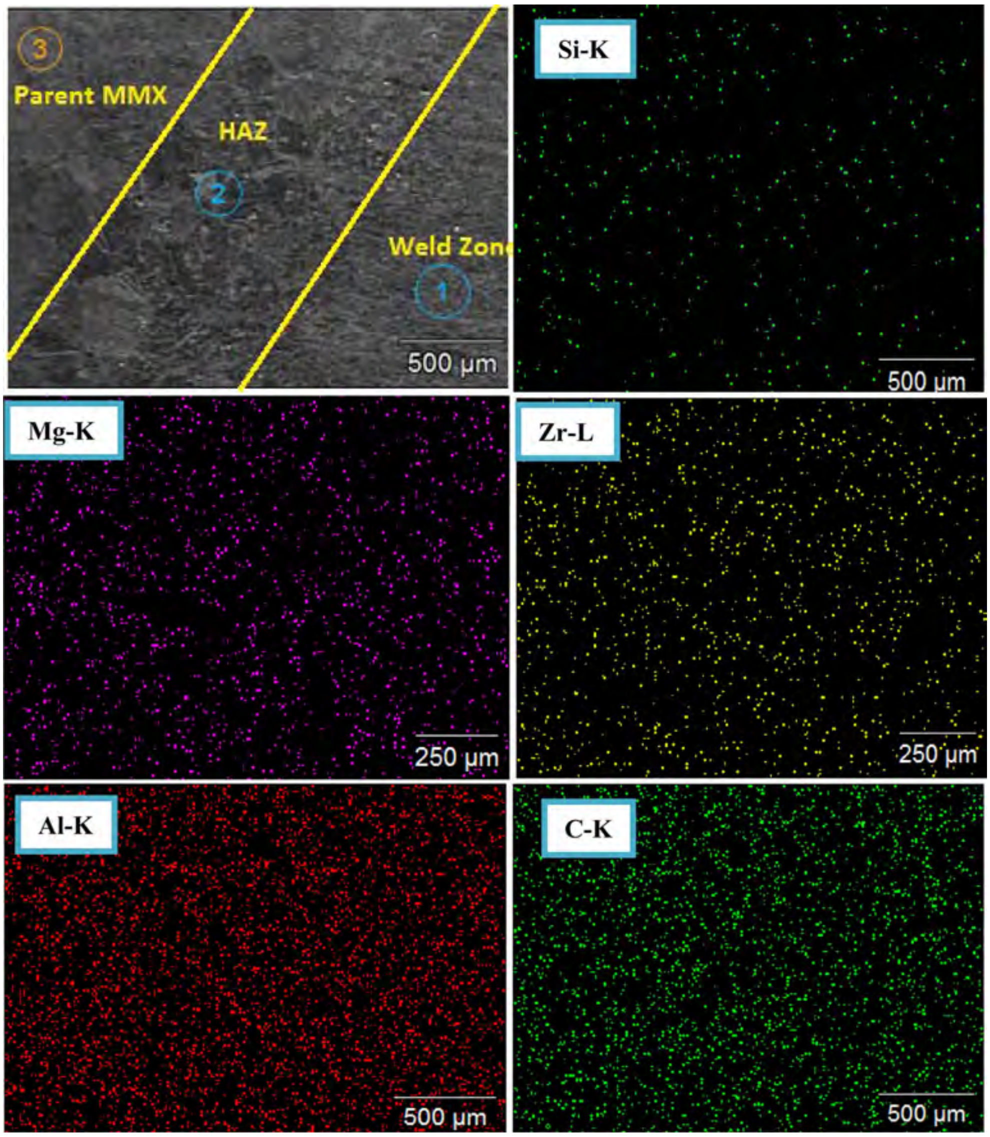

Fig. 7. (a) SEM images of FS welded MMC, (b)–(f) EDAX results from designated area for element mapping.

Zr, Si, C, Mg, etc. in the MMCs welded zones. It has proven the uniform distribution of matrix and uniform micro structure of FSW'ed zones. Fig. 7(a) shows that the matrix elements of sample 1, and few amount of matrix were present in uneven distribution (uneven mixing) due to insufficient heat input, having poorer mechanical properties. But sample 2 & 3 shows that the uniform distribution of matrix (even mixing) because of sufficient heat input, and it has improved mechanical and metallurgical properties. The identified alloying elements of FS weld MMCs were recorded in Table 3, Fig. 7(b-f) were showed friction stir welded MMCs EDAX element mapping.

Mechanical properties

The mechanical properties such as Tensile Strength and Hardness were measured after FSW'ed MMCs samples. By comparing three samples, the sample 1 resulting lower tensile strength comparing with other two samples (sample 2 & 3) due to insufficient heat input and uneven distribution of matrix. Sample 2 & 3

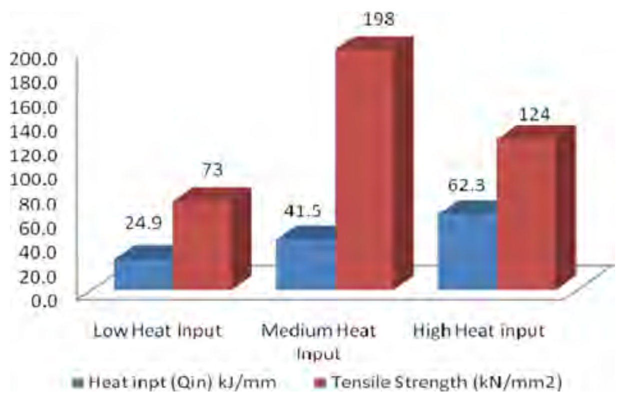

Fig. 8. Tensile strength Analysis of FS welded MMC.

ensuing higher tensile strength because of fine micro structure and even distribution of matrix. These are all shown in Fig. 8.

The significant contribution of reinforcement in the MMC can be analyzed from Fig. 9. It is noticed that the variation in load-bearing capacity of FS welded MMC is decreased by increasing the heat input generation during FSW process. The maximum breaking load of FS welded MMC is improved by lowering of heat generation to the intermediate (Medium) conditions. Similarly the maximum displacement and displacement at the breaking load of FS welded MMC is reduced by increasing of heat generation and dispersion of reinforcement due to stirring action of the FSW process. Due to ceramic particulate reinforcement, it loses its strength rapidly during plastic deformation.

The hardness levels of the FSW'ed MMCs joints are shown in Fig. 10. The higher hardness values were observed in the sample 2 & 3 due to columnar dendrites with fine and uniform mixing of matrix. Sample 1 has the resulting lower hardness value compared to other samples because of its uneven mixing of matrix due to the insufficient heat input.

The process parameters for these conditions were as follow: For Low Heat Input condition, the parameters were: Tool Rotational Speed -600 rpm, Welding Travel Speed- 50 mm/min, Axial Force- 4 kN and the obtained Tensile strength and Hardness values were 73 kN/mm2

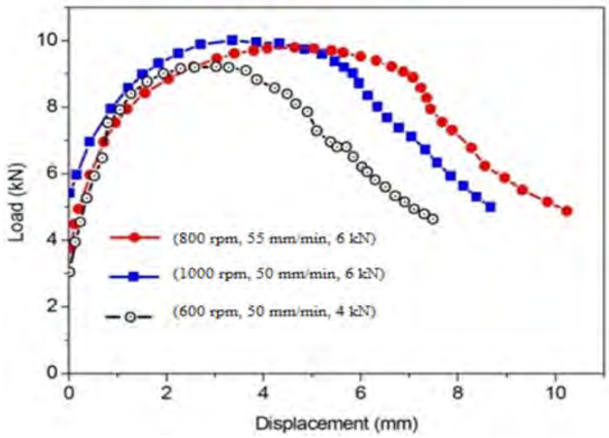

Fig. 9. Load-displacement curve of FS welded joint in MMC.

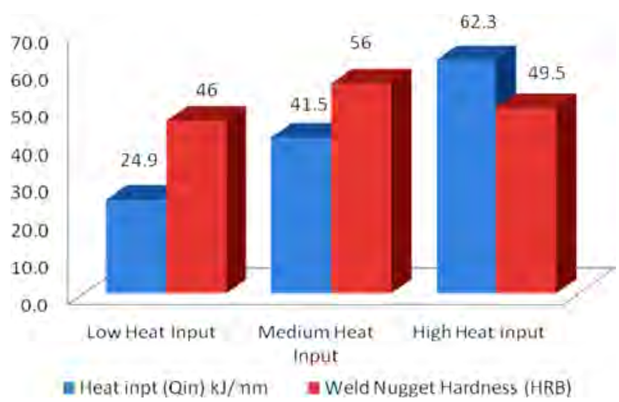

Fig. 10. Hardness analysis.

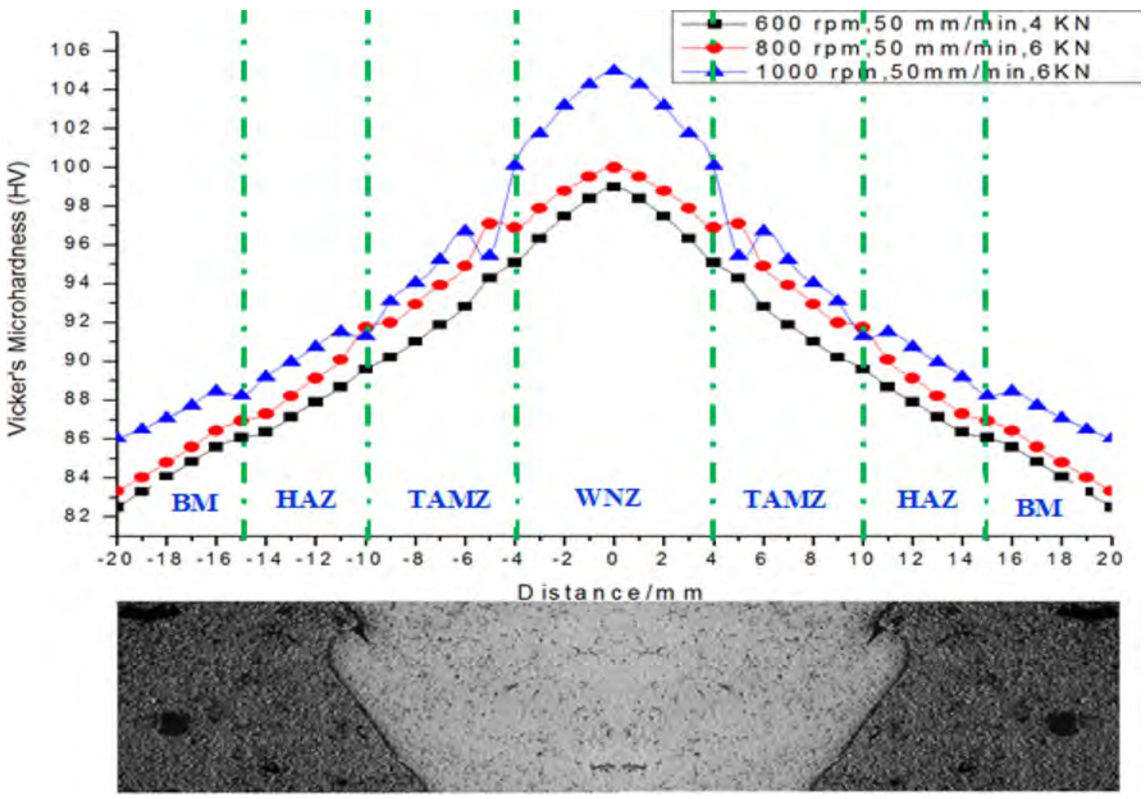

Fig. 11. Micro-hardness survey Analysis of FS welded MMC.

and 56 HRB respectively. Correspondingly, the process parameters for the Medium Heat Input condition were: Tool Rotational Speed - 800 rpm, Welding Travel Speed- 50 mm/min, Axial Force- 5 kN, observed Tensile strength -198 kN/mm2 and Hardness of 46 HRB. In the case of High Heat Input condition, the parameters were, Tool Rotational Speed -1000 rpm, Welding Travel Speed- 50 mm/min, Axial Force- 6 kN which resulted in the observed Tensile strength of 124 kN/mm2 and Hardness of 49.5 HRB.

While Fig. 11 shows that the micro-hardness survey of friction stir welded MMCs, it also proves the uniform mixing of matrix and uniform heat inputs during entire friction stir welding process. In micro-hardness survey analysis, the whole surface of FSW'ed joints was measured against the affected zones. The result of the survey provides accurate and detailed information about the surface features of FSW'ed AMMCs that have fine microstructure and multi-phases of homogeneous matrix. The weld nugget zone (WNZ) was considered as the centre-line for the hardness measurement of test sample's surface, based on the centre-line the hardness values that were measured on both sides (negative and positive) of the surface against the zones. From the survey result, found the WNZs had highest hardness value compared with other zones, also found the hardness values gradually reduced from centre-line to tail ends on both sides and zones had approximately equal value range on both sides. Based on the hardness values, the hardness range of the zones are in the manner of BM < HAZ < TMAZ < WNZ > TMAZ > HAZ > BM. Furthermore, it was found that Sample 2 & 3 ensuring higher hardness value at all zones in comparison with sample 1 because of the uniform mixing of matrix due to the sufficient heat inputs. The hardness is measured in Vickers Hardness scale (HV).

Conclusion

This investigation has dealt with the metallurgical characterization of Friction Stir Welded MMC and has observed that, the homogeneously distributed reinforcements and recrystallized grain structures were the predominant characters of the weld zone. In the FSW'ed joints, the weld nugget zones resulted in columnar dendrites along with even mixing of matrix and fine microstructure, also observed the grains coarsening in the TMAZ of the FSW'ed joints of AMMCs. During the FSW process, an optimum (Intermediate) heat input resulted in even mixing of matrix, obtained higher mechanical and metallurgical properties of the joints whereas, insufficient heat input lead to uneven mixing of matrix, obtained lower mechanical and metallurgical properties of the joint.

Metallurgical characterizations of the welded MMC were done by SEM, EDX and EDX analyses. Micro-structure comparison was done between the WNZ, TMAZ, HAZ and BM under three heat input levels: Low heat input, Medium heat input, and High heat input conditions. The Tensile strength and Hardness are higher in the case of the Medium Heat Input condition when compared to low and high heat input conditions. The process parameters for the Medium Heat Input condition were: Tool Rotational Speed - 800 rpm, Welding Travel Speed- 50 mm/min, Axial Force- 5 kN, observed propertied were: Tensile Strength -198 kN/mm^2 and Hardness of 46 HRB. The weld nugget hardness was found on the WNZ, TMAZ, HAZ, and BM. The maximum micro hardness of 62.3HRB was observed under 800 rpm Tool Rotational Speed, 4 mm/min Welding Travel Speed, 6 kN of Axial Force.

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