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SURFACE NANOCOMPOSITE FABRICATION ON AA6063 ALUMINIUM ALLOY USING FRICTION STIR PROCESSING: AN INVESTIGATION INTO THE EFFECT OF THE TOOL-SHOULDER DIAMETER ON THE COMPOSITE MICROSTRUCTURE

IZDELAVA NANOKOMPOZITA NA POVRŠINI ALUMINIJEVE ZLITINE AA6063 Z UPORABO VRTILNO-TRENJSKEGA PROCESA: RAZISKAVE VPLIVA PREMERA DRŽALA ORODJA NA MIKROSTRUKTURO KOMPOZITA

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In this work, surface metal matrix composites (SMMCs) were fabricated on AA6063 base metal through friction stir processing (FSP). In all the samples for surface composites, grooves of 2 mm × 2 mm were made along the centreline of plates and TiB_2 powder (~80 nm) was filled and compacted in these grooves. A pinless tool was employed to initially cover and compact the grooves filled with TiB_2 particles to prevent it from sputtering during FSP. Tools of different shoulder diameters (16, 18, and 20) mm with anti-clockwise scrolls on the shoulder surface were used for the FSP with constant pin diameter and pin length. The tool rotational speed of 900 min^{-1}, traversing speed of 40 mm/min and tilt 2°, respectively, kept constant for all the experiments. Macro, optical micro images and micro hardness tests were used to evaluate the particle distribution. Powder agglomeration was observed in the retreating side of samples processed with 16 mm and 18 mm shoulder diameter tools. On the other hand, significant improvement in particle distribution and excellent bonding with the substrate were observed for the sample processed with 20 mm shoulder diameter tool. The findings of this investigation are important and provide knowledge for better tool design and effective tool selection to bring out better distribution in a single pass.

Keywords: friction stir processing, aluminium alloy, shoulder diameter, microstructure

V tem prispevku avtorji opisujejo izdelavo in raziskavo izdelanega nanokompozita s kovinsko osnovo (angl.: SMMCs; Surface Metal Matrics Composites) na površini aluminijeve zlitine vrste AA6063, izdelanega s pomočjo vrtilno (rotacijsko) trenjskega procesa (angl.: FSP; Friction Stir Processing). Vsi vzorci površinskega kompozita širine 2 mm in globine 2 mm so bili izdelani vzdolž središčne linije kovinske plošče. Pri tem so bili med FSP dodajani približno 80 nm delci TiB_2 prahu v nastajajoče brazde. Za začetno prekrivanje in kompaktiranje brazd napolnjenih s TiB_2 so uporabili orodje brez trna in s tem preprečili razprševanje delcev med FSP. Za izdelavo SMMCs so uporabili držala različnih premerov (16, 18, in 20) mm z valjčki, nameščenimi na površini držal, ki so se vrteli v nasprotni smeri urnega kazalca. Pri tem so za FSP uporabili trn s konstantnim premerom in dolžino. Pri vseh preizkusih so za izbrani FSP uporabili hitrost vrtenja 900 min^{-1}, vzdolžno hitrost potovanja orodja 40 mm/min, in nagib 2°. Da bi ugotovili porazdelitev delcev TiB_2 v izdelanih SMMCs so izvedli metalografske preiskave in določili mikrotrdoto kompozitov. Opazili so aglomeracijo prašnih delcev na vzorcih izdelanih s 16 mm in 18 mm premerom držala orodja. Po drugi strani pa so dosegli pomembno izboljšanje porazdelitve prašnih delcev in njihovo odlično vezavo s kovinsko osnovo pri vzorcih, ki so bili izdelani z 20 mm premerom držala orodja. Ugotovitve te raziskave omogočajo boljše oblikovanje in učinkovito izbiro orodja za doseganje optimalne porazdelitve nanodelcev v kovinski osnovi pri FSP z enim samim prehodom F.

Ključne besede: proces rotacijskega trenja, zlitina na osnovi aluminija, premer držala, mikrostruktura

1 INTRODUCTION

FSP is based on the principles of Friction Stir Welding (FSW) developed at "The Welding Institute (TWI), UK" in 1991.[1] In FSP a cylindrical shouldered tool with a profiled probe or pin is rotated and plunged into base metal (BM) and traversed on the workpiece surface in the processing direction (**Figure 1**). The rubbing action of the tool shoulder generates frictional heat and softens the material under the shoulder, which also undergoes severe plastic deformation at high strain rate by the rotating pin (called stirring).[2,3] During FSW/FSP, material is subjected to a combination of metal working processes, e.g., friction, extrusion and forging.[3–5] FSP is evolving as a promising surface modification technology for surface composite fabrication mainly because it is a solid-state process and a green process by virtue of being free from use of consumables and evolution of effluent. One of the major challenges of the process, however, is the inhomogeneous distribution of reinforcement particles. A large number of research works have been focused on achieving a homogeneous distribution of particles, elimination of agglomeration of particles, overcoming of tunnel-like defects and achieving a wide

Table 1: Chemical composition of AA6063-T6, in mass fractions (*w*/%)

Element	Al	Mg	Si	Cu	Mn	Fe	Ti	Cr	Zn	Ni
AA6063	98.71	0.499	0.424	0.022	0.034	0.25	0.015	0.005	0.028	0.003

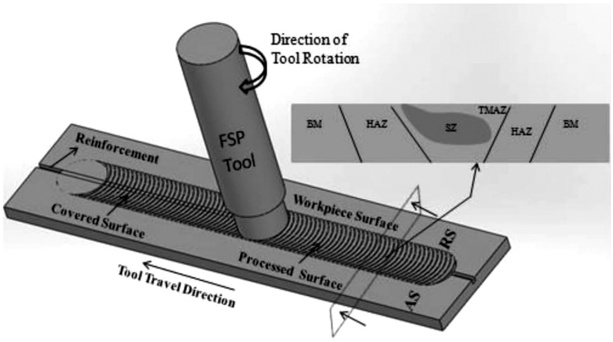

Figure 1: Schematic diagram of FSP

composite zone by utilizing various strategies such as applying multiple number of FSP passes, change of tool rotation between passes, process hybridization such as electric current assisted FSP, etc.[6–10] All these, result in loss of time and energy and often loss of substrate properties as well. Multiple passes not only increase the energy input, production time but every pass also lowers the material properties, especially in case of heat-treated materials.

Studies with a specific focus on material flow and reinforcement particle distribution have been reported in which the effects of pin profile have been considered.[8,11–12] Few researches have used concave and scrolled shoulder for surface composite fabrication without investigating specifically the effect of the shoulder diameter on the response.[9–10,13] Tool shoulder and pin control the heat generation caused by friction during FSP. Of the two, the shoulder is the main heat generation source since the contact area between shoulder and BM is higher than the pin contact area.[14] It is pertinent that the tool shoulder diameter is important to materials movement as well and hence an important factor in obtaining adequate particle distribution. AA6063 is a material of choice in naval and automobile applications mainly due to it good formability, specific strength and resistance to corrosion.[15–18] Some researchers have fabricated AA6063/TiB_2 composites by using liquid metallurgy and FSP process.[19–20] However, this study is performed with a specific objective to understand the distribution of TiB_2 particles in the AA6063 alloy for which the effect of tool shoulder diameter has been investigated. In this paper tool shoulder diameter was varied and their effect on particle distribution and hardness were analysed.

2 MATERIALS AND METHODS

The chemical composition of the 6063-T6 aluminium alloy used in the investigation is presented in **Table 1** and its mechanical properties are presented in **Table 2**. The FSP experiments were performed on samples of 170 mm × 50 mm × 4.75 mm dimensions. In all samples for surface composites, grooves of 2 mm × 2 mm were made along the centreline of plates and TiB_2 powder (~80 nm) was filled and compacted in these grooves. The scanning electron micrograph (SEM) and X-ray diffraction (XRD) pattern of TiB_2 reinforcement powder is shown in **Figure 2**. A pinless tool was employed to initially cover and compact the grooves filled with TiB_2 particles to prevent it from sputtering during FSP.

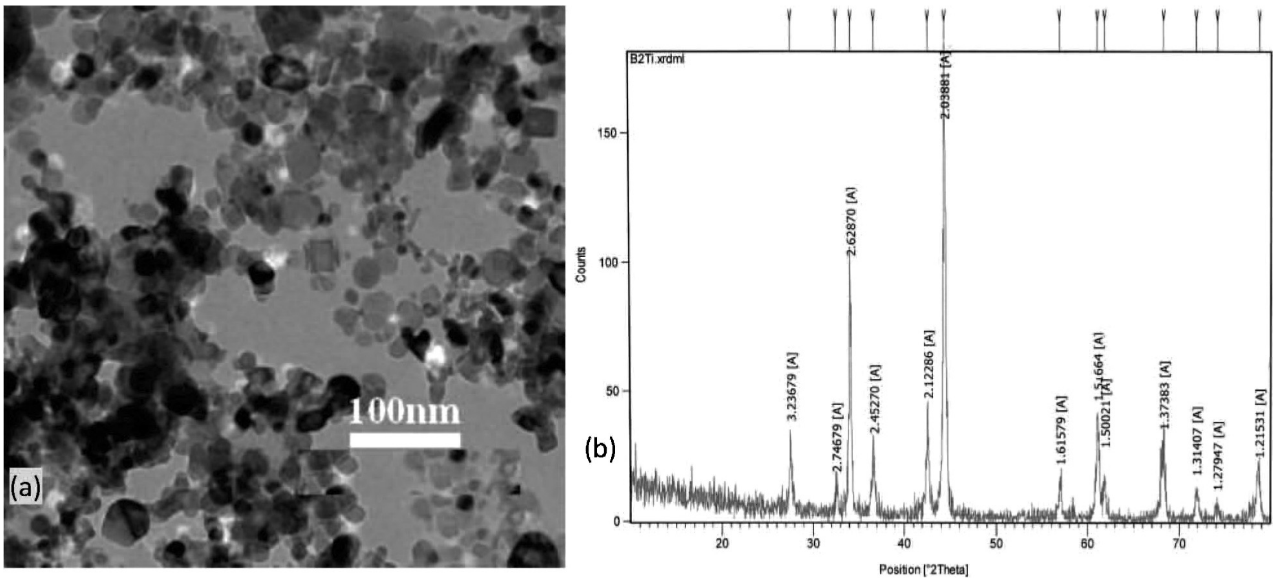

Figure 2: a) SEM micrograph, and b) XRD pattern of titanium diboride (TiB_2) nano powder

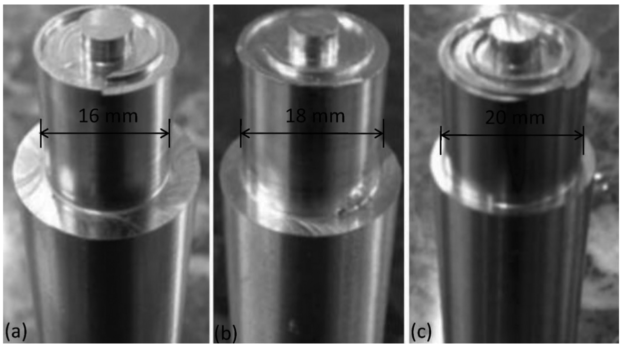

Figure 3: FSP tools having shoulder diameter: a) 16 mm, b) 18 mm, and c) 20 mm

Table 2: Mechanical properties of as-received AA6063-T6

UTS (MPa)	Yield strength (MPa)	Elongation (%)	Microhardness (Hv)
220	110	14	72.6

The FSP was carried out on an indigenously retrofitted FSW machine. FSP tools made of high-carbon high-chromium (HCHCr) steel as shown in **Figure 3** were used in this study. (16, 18 and 20) diameter tools with anti-clockwise (ACW) scrolled shoulder surface having cylindrical pin with 6 mm diameter and 2.5 mm in length were used.

For all the experiments the rotational speed, traverse rate, tool tilt and tool plunge were fixed at 900 min^{-1}, 40 mm/min, 2° and 2.42 mm respectively. The friction stir processed plates are shown in **Figure 4**.

After FSP, microstructural analyses of processed zone (PZ) were carried out for which specimens were prepared using a standard metallographic procedure. The metallographic samples were subsequently etched with extended flick reagent (15 ml hydrochloric acid, 10 ml hydrofluoric acid and 90 ml distilled water) for 3 min. Macroscopic images were taken using a Stereozoom microscope (Focus, Japan). Microstructural observations were carried out by employing OM (QS Metrology, India). The micro-hardness of the samples was measured under a load of 0.1 N and for a dwell time of 15 seconds using micro-hardness tester (Mitutoyo, Japan).

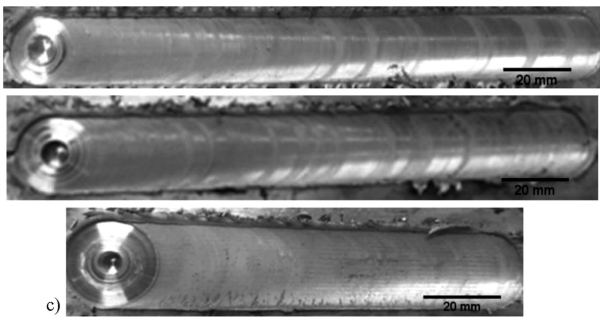

Figure 4: Friction stir processed plates with tool shoulder diameter of: a) 16 mm, b) 18 mm, and c) 20 mm

3 RESULTS AND DISCUSSION

Macro and micro-structural, bead geometry, analysis along with indentation test were carried out to investigate the influence of the tool shoulder diameter on the fabricated surface composites.

3.1 Macro and micro-structure

Macrograph of cross-section showing reinforced zone (RZ) of all the samples is shown in **Figure 5** and the microstructure of its various regions is shown in **Figures 6** to **8**. It is also evident from the micrographs that the advancing side (AS) interface in all samples generally possesses better distribution and good bonding of the reinforcement with the substrate material. However, the accumulation of TiB_2 particles was witnessed on the retreating side (RS) interface of the samples (especially in those which were processed with 16 mm and 18 mm diameter shoulder tool). Whereas no accumulation was found in the sample that was processed

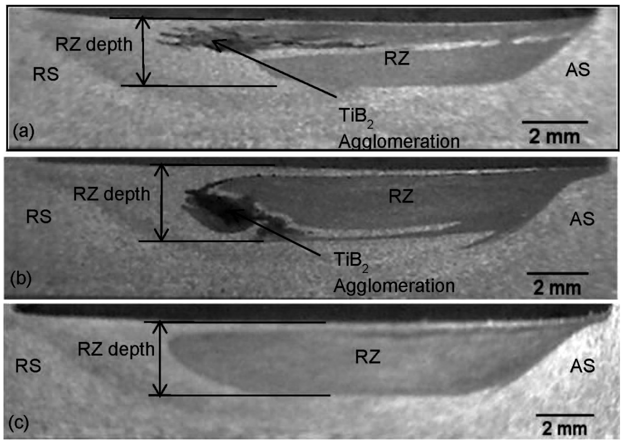

Figure 5: Macrograph of cross-section of PZ processed with tool shoulder diameter of: 16 mm, b) 18 mm and c) 20 mm

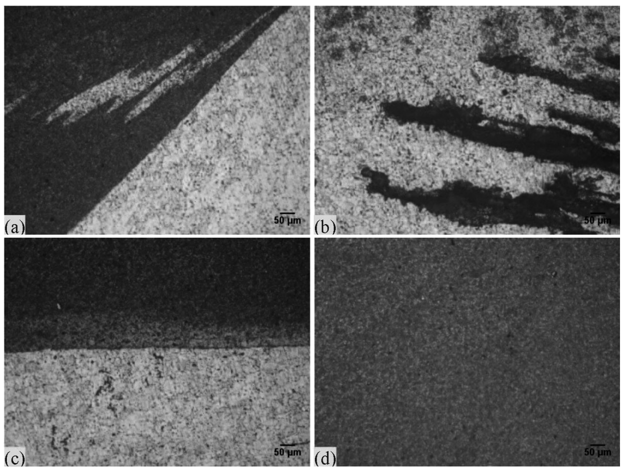

Figure 6: Microstructure of various regions of sample processed with 16 mm tool shoulder diameter showing: a) AS interface, b) agglomerated region in RS side, c) bottom interface and d) SZ

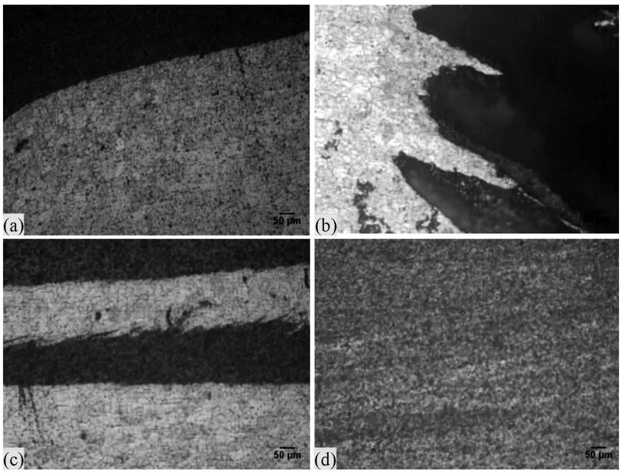

Figure 7: Microstructure of various regions of sample processed with 18 mm tool shoulder diameter showing: a) AS interface, b) agglomerated region in RS side, c) bottom interface and d) SZ

with a 20 mm diameter shoulder tool. Significant improvements in terms of particle distribution were achieved in samples processed with a 20 mm diameter shoulder. Bands of reinforced and unreinforced regions were found in samples processed with 16 mm and 18 mm tools, as shown in **Figures 6a** and **7c**. No such bands were observed in samples processed with 20 mm tool diameter and the reinforced zone in these samples exhibited homogeneous particle distribution as well as good bonding with the substrate material (**Figure 8a** to **8d**). The PZ dimensions are given in **Table 3**. The results indicated that the depth and area of RZ increase with the increase in the shoulder diameter in all the samples.

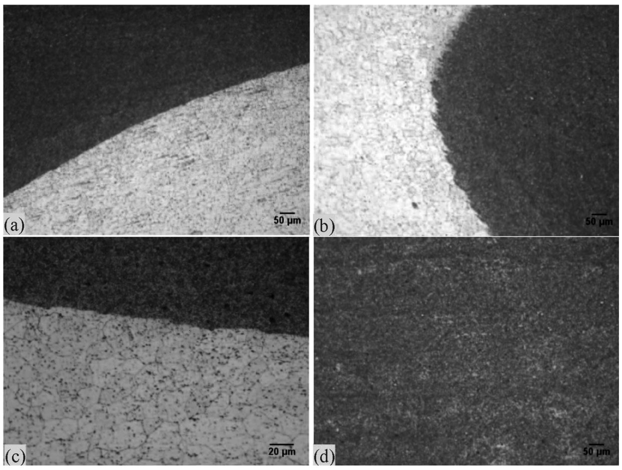

Figure 8: Microstructure of various regions of sample processed with 20 mm tool shoulder diameter showing: a) AS interface, b) RS interface, c) bottom interface and d) SZ

Table 3: Processed zone dimensions

Tool shoulder diameter (mm)	Reinforced zone depth (mm)	Particle agglomerated area (mm^2)	Reinforced zone area (mm^2)
16	2.43	1.03	19.37
18	2.67	1.35	21.74
20	2.71	–	26.62

The tool shoulder has a direct relation with the heat generation during processing due to the higher frictional contact area. For a larger shoulder diameter the heat generation is higher because of the larger contact area and vice versa.[21] Small shoulder diameter produces inadequate frictional heat and consequently provides insufficient plasticized base material flow under the shoulder. Also, the shoulder is responsible for nearly one-third of the material transport in the upper portion of processing zone.[22] Kumar and Kailas demonstrated that the combined effect produced by shoulder and pin driven material flow is responsible for the resultant particle distribution and the microstructure of SZ. The shoulder facilitates bulk material transfer, while the tool pin is responsible for layer-by-layer material flow.[23] The additional features on the shoulder surface such as scrolls also play an exceptionally important role by providing additional frictional treatment and better material flow.[24–25] In the present investigation, the combined effect of shoulder-driven and pin-driven flow has been observed in all samples. It can be inferred that the tool shoulder diameter of 16 and 18 mm could not generate sufficient frictional heat and material flow, which results in the agglomeration of reinforcement particles in RS of SZ due to higher flow stresses and poor material flow. However, 20 mm shoulder diameter tool was able to generate sufficient frictional heat, lower flow stresses, proper consolidation of material behind the tool and better material flow, therefore, it results in a homogeneous particle distribution in the SZ, larger reinforced zone depth and area without any microscopic defect.

3.2 Microhardness

The microhardness profile was generated along the width of PZ at 1 mm equidistant point and the same is shown in **Figure 9**. The microhardness profile was traced 1 mm below the top surface.

The average micro-hardness of PZ of samples processed with 16, 18, and 20 mm diameter shoulder along the horizontal direction of the cross-section were found to be 107.65, 111.33, and 113.27 HV, respectively.

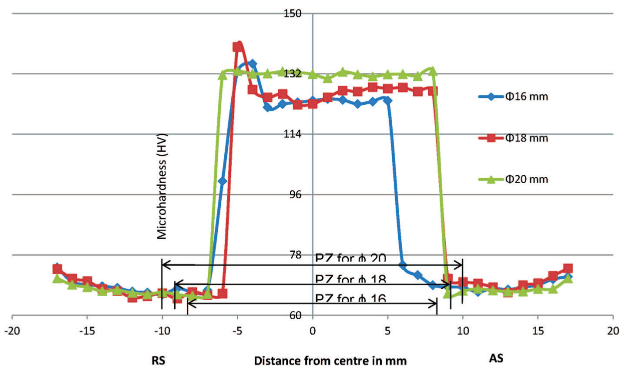

Figure 9: Hardness profiles

Narimani et al.[20] also fabricated AA6063/TiB_2 composite and reported an increase in hardness to a significant value due to higher inherent hardness (2500 kg/mm^2) of the reinforcement particles. Fine-grained matrix and the Orowan strengthening are the two major contributors to the improvement of the micro-hardness of fabricated composites.[19,20] Surface composites (SCs) fabricated in this study exhibited significantly higher hardness than the base metal due to the presence of hard TiB_2 particles. The average micro-hardness of the sample processed with 20 mm diameter tool is highest among all the samples. The highest and more uniform distribution of hardness profile of this sample was attributed to homogeneous distribution of TiB_2 particles in processed zone.

4 CONCLUSION

The AA6063/TiB_2 composites were fabricated successfully using FSP. The effect of tool shoulder diameter on the microstructure and micro-hardness of the metal matrix composite was studied. The obtained results can be summarized as follows:

- The hardness of processed zone is increased in all the samples due to inherent higher hardness of the reinforcement.
- The reinforced region depth and area are increased with an increase in the diameter of tool shoulder.
- No agglomeration of particles was observed in the sample processed with a 20 mm diameter shoulder tool.
- The composite processed with 20 mm diameter tool shoulder exhibited excellent particle distribution, superior micro-hardness and good bonding with the substrate material.

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