

FRICION-STIR PROCESSING OF A COMPOSITE ALUMINIUM ALLOY (AA 1050) REINFORCED WITH TITANIUM CARBIDE POWDER

UPORABA MEŠANJA S TRENJEM ZA IZDELAVO KOMPOZITA ALUMINIJEVE ZLITINE (AA1050), OJAČANE S TITANOVIM KARBIDOM V PRAHU

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In this study the friction-stir processing (FSP) technique was applied for the development of surface composites of aluminium alloy (AA 1050) reinforced with titanium carbide (TiC) powder of particle size range below 60 μm compressed into the groove. Rotational speeds of 1200 min^{-1} and 1600 min^{-1} and the travel rates of (100, 200 and 300) mm/min were used for the process. This study investigates the effect of processing parameters on the wear-resistance behavior of friction-stir processed Al-TiC composites. This was achieved through microstructural characterization using optical and scanning electron (SEM) microscopes equipped with Oxford energy-dispersion spectrometry (EDS) (Tescan), microhardness profiling and wear resistance tests. From the results, it was found that the processing parameters influenced the distribution of the TiC particles. The microhardness profiling of the processed samples revealed an increase in the hardness value compared to the parent material. The wear-resistance test results confirmed the FSP technique enhanced properties in surface engineering.

Keywords: aluminium alloy, friction-stir processing, microhardness, microstructure, surface composite, TiC

V študiji je bila uporabljena tehnika mešanja s trenjem (angl. FSP) za razvoj površinskih kompozitov iz aluminijeve zlitine (AA 1050), ojačanih s titanovim karbidom (TiC) v prahu, z velikostjo delcev v območju pod 60 μm , stisnjenih v utor. Pri tem postopku so bile uporabljene hitrosti vrtenja 1200 min^{-1} in 1600 min^{-1} in hitrosti pomikanja (100, 200 in 300) mm/min. Študija raziskuje vpliv procesnih parametrov na odpornost kompozitov Al-TiC proti obrabi s trenjem. Izvedena je bila mikrostrukturalna karakterizacija z uporabo svetlobnega in vrstičnega elektronskega mikroskopa (SEM), opremljenega z Oxford energijsko disperzijsko spektrometrijo (EDS) (Tescan), profiliranja mikrotrdote in preizkusa obrabe. Iz rezultatov je bilo ugotovljeno, da so parametri obdelave vplivali na distribucijo TiC delcev. Profiliranje mikrotrdote na vzorcih je pokazalo, da je vrednost trdote povečana v primerjavi z osnovnim materialom. Rezultati testa odpornosti obrabe so potrdili, da FSP-tehnika izboljša lastnosti površine.

Ključne besede: aluminijeve zlitine, tehnika mešanja s trenjem, mikrotrdota, mikrostruktura, površinski kompoziti, TiC

1 INTRODUCTION

Friction-stir processing (FSP) is a newly emerging technology based on the friction-stir welding (FSW) method that allows the near-surface regions of a material to be selectively processed and currently used to enhance the mechanical properties of conventional materials.¹ This process has been applied to microstructural modifications for enhanced mechanical properties through intense plastic deformation using a non-consumable rotating tool that causes material mixing and thermal exposure and results in grain refinement of the material being processed.^{2,3} The benefits of the FSP process include significant microstructural refinement, densification, and homogeneity of the processed zone, homogenization of precipitates in various aluminium alloys and composite materials.⁴⁻⁶ Moreover, the severe plastic deformation (SPD) and material flow in the stirred zone of the FSP can be utilized to achieve surface or bulk alloy modification by the mixing of other elements into

the stirred alloys where the stirred material can become a metal matrix composite or an intermetallic alloy with much higher hardness and good wear-resistance properties.⁷ Applications of the FSP/FSW are widespread for the aerospace, automotive and the transportation industries. Research studies have been reported on the friction-stir process that grain refinement can be achieved in this way. The microstructure and mechanical behaviour of lightweight materials subjected to the FSW/FSP are being studied extensively, which include the processing of the microstructure amenable to high-strain-rate superplasticity.⁸ Y. Kwon et al. produced 1050 aluminium alloy with an ultra-fine grain size through the friction-stir process (FSP). They studied the influence of tool rotation speed on the temperature profile, microstructure and mechanical properties of the friction-stir processed zone (FZ) and they concluded that FSP is very effective in producing an ultra-fine-grained material with excellent mechanical properties.

Aluminium matrix composites (AMCs) reinforced with ceramic particles exhibit high strength, high elastic modulus, and improved resistance to wear, creep and fatigue, which make them promising structural materials for aerospace and automobile industries.⁹ These composites can suffer a great loss in ductility and toughness due to the incorporation of non-deformable ceramic reinforcements, which limits their wide applications to a certain extent.^{10,11} However, the nature of the technique used to fabricate particle-reinforced metal-matrix composites (MMCs) has been shown to have a significant effect on the product's mechanical properties and hence the applications for which it is suitable.^{12–15} Surface-modification techniques such as high-energy laser beam, plasma spraying, cast sinter and electron beam irradiation have been developed over the past two decades to fabricate Surface Metal Matrix Composites (SMMCs).^{16–19} It has been shown that friction-stir processing (FSP) can be effectively used to homogenise the particle distribution in Al-based in-situ composites. R. Mishra et al.¹² confirmed the application of FSP technology to fabricate AA5083/SiC SMMC. The incorporation of SiC particles on the surface was successful and bonded well with the matrix. Subsequently, they confirmed FSP as a promising method to fabricate SMMCs.² Mahmoud et al. fabricated AA1050/SiC SMMC, their results showed that the distribution of the SiC particles became more homogeneous with an increasing number of passes and with the combination of tool rotational speed and the processing speed resulted in the formation of defects, such as voids and tunnels in the FSP zone.²⁰ E. R. I. Mahmoud et al.²⁰ also fabricated AA1050/SiC SMMC and assessed the effect of the tool pin size and profile. From their results, the wear rates of the samples produced with the flat surface tools were higher than that of the samples produced with the round-shaped tools and the samples produced with the square profile pin yielded a finer distribution of the SiC particles.²¹ R. Bauri et al.²² used FSP effectively to homogenise the particle distribution in Al-based in-situ composites. The process was employed on the as-cast composite to uniformly distribute the TiC particles in the Al matrix. The composite was subjected to single- and double-pass FSP and its effect on the microstructure and properties was evaluated. From their results, they confirmed that a single pass of FSP was enough to break the particle segregation from the grain boundaries and improve the distribution and two passes of FSP resulted in complete homogenization and elimination of casting defects. The grain size was also refined after each FSP pass. This led to a significant improvement in the mechanical properties after FSP. A. Kahrizangi and S. Kashani-Bozorg²³ works on a mild-steel substrate by the introduction of nano-sized TiC powder into the stir zone using four passes of friction stir processing and the TiC clusters were formed after the first pass. Sequential break-up of the clusters and refinement of the matrix

grains were caused by subsequent FSP passes. A near-uniform dispersion of nano-sized TiC particles was achieved after the fourth pass. A significant improvement in the wear resistance of the nano-composite layer was observed as compared to that of the as-received substrate and the enhanced properties are attributed to the uniform dispersion of hard nano-sized TiC reinforcements in a matrix of ultra-fine dynamically recrystallized grains. A. Thangarasu et al.¹⁰ fabricated TiC particulates ($\approx 2 \mu\text{m}$) and reinforced them with an aluminium matrix composite (AMC) using FSP. The powders were compacted into a groove and a single pass FSP was carried out using a tool rotational speed of 1600 min^{-1} and a processing speed of 60 mm/min . They analysed the microstructure and microhardness of the fabricated AMC and from their results, revealed a uniform distribution of TiC particles which were well bonded to the matrix alloy with an increase in the hardness value of the AMC compared to that of the matrix alloy. E. Akinlabi et al.³ investigated the effect of the processing parameters on the wear-resistance behaviour of friction-stir processed Al-TiC composites. From their findings it was revealed that an increase in the hardness value, which was a function of the TiC particles incorporated when compared to the parent material and the wear resistance property, was also found to increase as a result of the TiC powder addition. The right combination of the processing parameters was found to improve the wear resistance property of the composites produced. Kurt et al. incorporated SiC particles into the commercially pure aluminium to form particulate surface layers by using friction-stir processing (FSP). The samples were subjected to the various tool rotating and traverse rates with and without SiC powders. Microstructural observations were carried out by employing optical microscopy of the modified surfaces. Mechanical properties like hardness and plate bending were investigated. The results showed that increasing rotating and traverse rate caused a more uniform distribution of the SiC particles. Uygur studied commercially pure (CP) aluminium using the friction stir welding technique (FSW) to weld the specimens. The welding process was carried out by rotating 1500 min^{-1} and by moving 200 mm/min . Under a constant friction force this involved four different shoulder diameters of (20, 25, 30 and 40) mm. During welding, temperature measurements were performed using a non-contact laser thermometer at various parts of the plates from the welding centre outwards. Microscopic and mechanical tests were used to characterize the sample.

In this paper, a further research study was conducted on AA1050 to uniformly distribute the TiC particles into the Al matrix using friction stir processing. The produced composites were investigated through the microstructural evolution, wear resistance behaviour and the hardness distribution. The research study is aimed at providing a better understanding of the materials behaviour

after FSP using different combinations of rotational speeds and travel rates.

2 MATERIALS AND METHODS

In this study, aluminium alloys (AA1050) with rectangular elements with dimensions of (200 × 160 × 3) mm were used for this experiment. The experimental setup showing the clamping fixture and the backing plate system is presented in **Figure 1**. The chemical composition of the aluminium alloy used is presented in **Table 1**. The V-groove of about 5 mm was machined and filled with TiC powder with the aim of strengthening the material. Some 99.5 % TiC with a particle size range below 60 µm ball milled powder with irregular shape was used. The powder was compressed into the groove using a pinless tool across the surface of the material and

a second tool, comprising a pin and shoulder, was plunged into the workpiece after compressing the TiC powder. The micrograph of the TiC powder used is shown in **Figure 2**. The tools were machined from H13 tool steel and hardened to 52 HRC. The workpieces were securely clamped on a rigid, smooth, mild-steel backing plate with dimensions of (650 × 265 × 25) mm, to make the material withstand the significant perpendicular and lateral forces developed during the friction-stir process. The weld matrix employed with the processing parameters is presented in **Table 2**. The rotational speeds employed were 1200 min⁻¹ and 1600 min⁻¹ and the travel rates were (100, 200 and 300) mm/min, both representing the low, medium, and high settings, respectively. **Figure 3** shows a schematic drawing of the V-groove machined into the base metal.

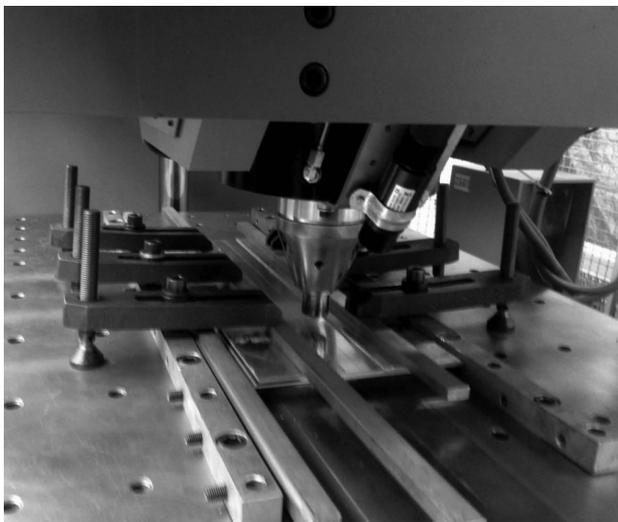


Figure 1: Experimental set up
Slika 1: Eksperimentalni sestav

Table 1: Chemical composition of AA1050

Tabela 1: Kemijska sestava zlitine AA1050

	Al	Si	Fe	Cu	Mn	Mg	Zn	V
AA1050	99.5	0.25	0.4	0.05	0.05	0.05	0.05	0.05

Table 2: Processing-parameters matrix of friction-stir processing

Tabela 2: Matrika procesnih parametrov pri obdelavi mešanja s trenjem

Designation	Spindle speed (min ⁻¹)	Travel rate (mm/min)
FsP 1200_100	1200	100
FsP 1200_200	1200	200
FsP 1200_300	1200	300
FsP 1600_100	1600	100
FsP 1600_200	1600	200
FsP 1600_300	1600	300

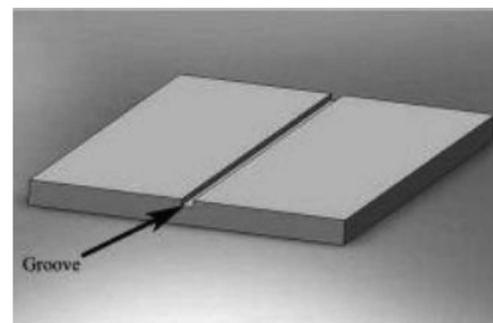
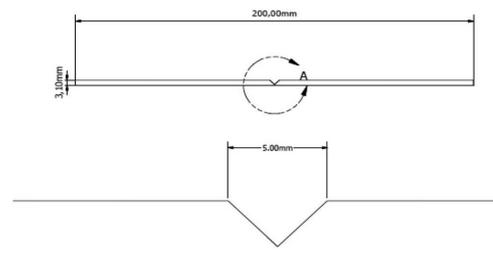


Figure 3: A schematic drawing showing the V-groove machined into the base metal

Slika 3: Shematski prikaz in posnetek klinastega utora v osnovni kovini

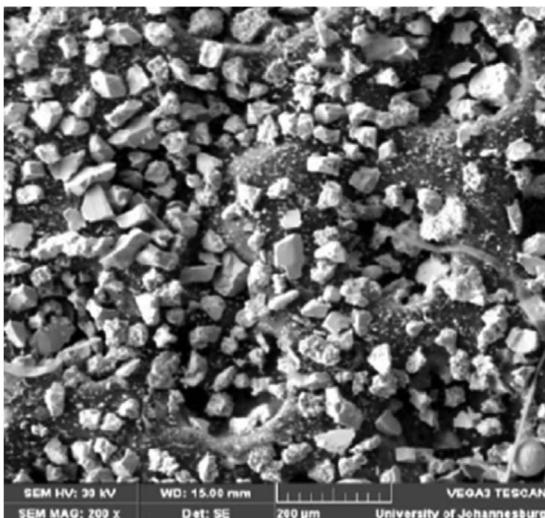


Figure 2: SEM micrograph of the titanium carbide powder

Slika 2: SEM-posnetek prahu titanovega karbida

All the processed samples and the as-received materials were cut into the required sample sizes with the aid of the water-jet cutting technique for microstructural characterization and microhardness measurements. The processed specimens were examined with a scanning electron microscope (SEM) equipped with Oxford energy-dispersion spectrometry (EDS) (Tescan) were used to characterize the samples. The microhardness measurements were conducted using a Vickers microhardness tester with a diamond indenter using 300 g load and a dwell time of 15 s at the surface of the processed samples and the parent material. The wear tests were evaluated on both the as-received and processed samples using a tribometer (CETRUMT200). The wear resistance test was performed under dry condition using a ball-on-disk arrangement on both the parent material and the materials processed by FSP. The material of the ball is a tungsten carbide of 10 mm diameter and at a load of 25 N with a reciprocating frequency of 20 Hz and for a 2000 m sliding distance.

3 RESULTS AND DISCUSSIONS

3.1 Surface appearance characterisation

Figure 4 shows the upper surface appearances of the samples processed at a rotational speed of 1200 min^{-1} and travel rates of 100 mm/min and 300 mm/min and **Figure 5** shows the appearance of samples processed at a rotational speed of 1600 min^{-1} and travel rates of 100 mm/min and 300 mm/min. From the figures, good smooth and quality surfaces were observed after the FSP, the groove is effectively bonded and no defects such as voids and cracks were observed on the surface. **Figure 6** shows the root of sample C at a rotational speed of 1200 min^{-1} and a travel rate of 300 mm/min. The root indicates that a thorough stirring was achieved during the FSP.

3.2 Microstructural evolution

Figures 7 and **8** show the SEM micrographs of the distribution of the TiC particles in the AA 1050 aluminium produced at rotational speeds of 1200 min^{-1} and 1600 min^{-1} , respectively. **Figure 7** shows that the grains in the FSP zone of the specimen were refined; this caused a high plastic strain in the stirred zone in which

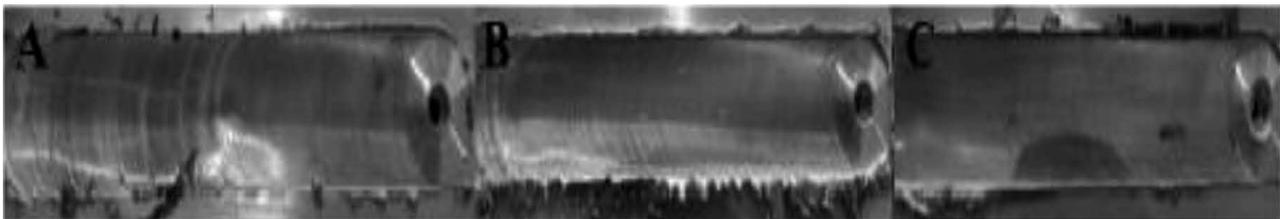


Figure 4: Photograph of samples processed at a rotational speed of 1200 min^{-1} and travel rates of: a) 100 mm/min, b) 200 mm/min and c) 300 mm/min

Slika 4: Fotografija vzorcev, obdelanih pri vrtilni hitrosti 1200 min^{-1} in hitrosti pomikanja: a) 100 mm/min, b) 200 mm/min in c) 300 mm/min



Figure 5: Photograph of samples processed at a rotational speed of 1600 min^{-1} and travel rates of: d) 100 mm/min, e) 200 mm/min and f) 300 mm/min

Slika 5: Fotografija vzorcev obdelanih pri hitrosti vrtenja 1600 min^{-1} pri hitrosti pomikanja: d) 100 mm/min, e) 200 mm/min in f) 300 mm/min



Figure 6: The root of sample C produced at a rotational speed of 1200 min^{-1} and travel rate of 300 mm/min

Slika 6: Koren vzorca C, obdelanega pri hitrosti vrtenja 1200 min^{-1} pri hitrosti pomikanja 300 mm/min

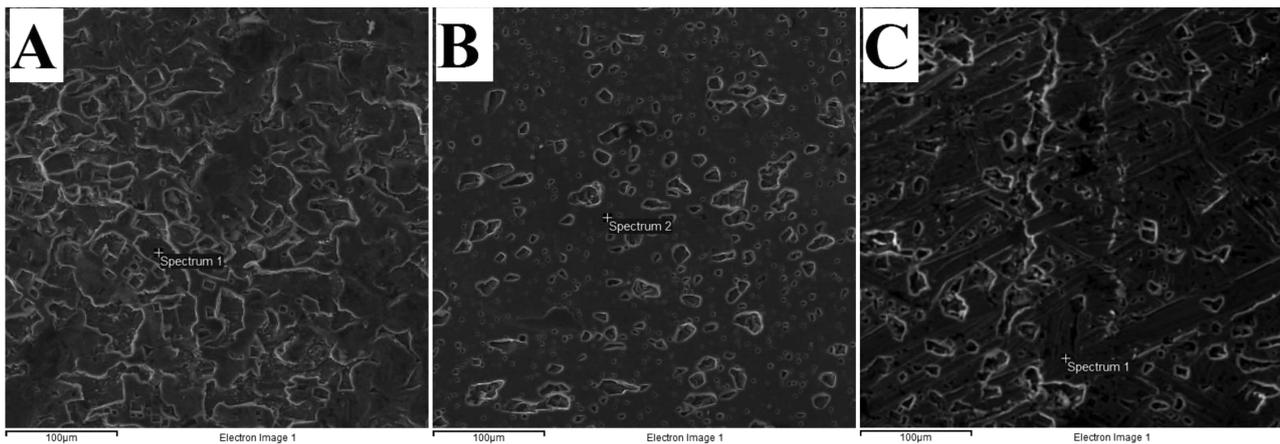


Figure 7: SEM photograph of grains after processing at a rotational speed of 1200 min^{-1} and travel rates of: a) 100 mm/min, b) 200 mm/min, c) 300 mm/min

Slika 7: SEM-posnetek zrn po obdelavi s hitrostjo vrtenja 1200 min^{-1} in hitrostjo pomikanja: a) 100 mm/min, b) 200 mm/min, c) 300 mm/min

severe plastic deformation was introduced into the workpiece through the rotating pin and shoulder, which results in a redistribution of the TiC particles. This can be attributed to the heat generated by the tool rotation speed and the traverse speed that resulted in lots of dilution of the TiC and the Al. A homogenous distribution of the TiC particles is essential to attain higher mechanical properties in the composite of aluminium alloy (AA 1050) and the TiC. The mechanism of grain refinement in AA1050 is that of dynamic recrystallization.^{22,24} It was observed in **Figure 7** that the TiC particles were clustered together at a rotational speed of 1200 min^{-1} and a travel rate of 100 mm/min compared to the samples produced at travel rates of 200 mm/min and 300 mm/min. This indicates that when the travel is at a slower rate, the TiC particles are not well distributed. It was observed in **Figure 8** that the size of the TiC particles is not uniform throughout the FSP zone. It was observed that some of the TiC particles were clustered in the samples produced at a high rotational speed of 1600 rpm. This can be attributed to the elevated temperature

generated by the tool rotation speed and the traverse speed employed, which is in agreement with the published literature, as reported in some work by researchers in aluminium matrix composites.^{25–27} Particles other than the TiC and aluminium were also observed in the processed specimens, using energy-dispersive X-ray spectroscopy (EDS).

3.3 Energy-dispersive X-ray spectroscopy (EDS)

Energy-dispersive X-ray spectroscopy (EDS) analysis was conducted to evaluate the morphological features of the debris found in the processed specimens. **Figure 9** shows the EDS of the debris of an FSP specimen processed at rotational speeds of 1200 min^{-1} and 1600 min^{-1} . The EDS pattern, shown in the figures, reveals that some particles are comprised of elements like Si, C, Fe and Ag. The presence of these elements indicates that the debris consist of elements present in the tool as well as in the backing plate (e.g., Fe, C). The figures also reveals that the extent of elements like Si, C, Fe and Ag are

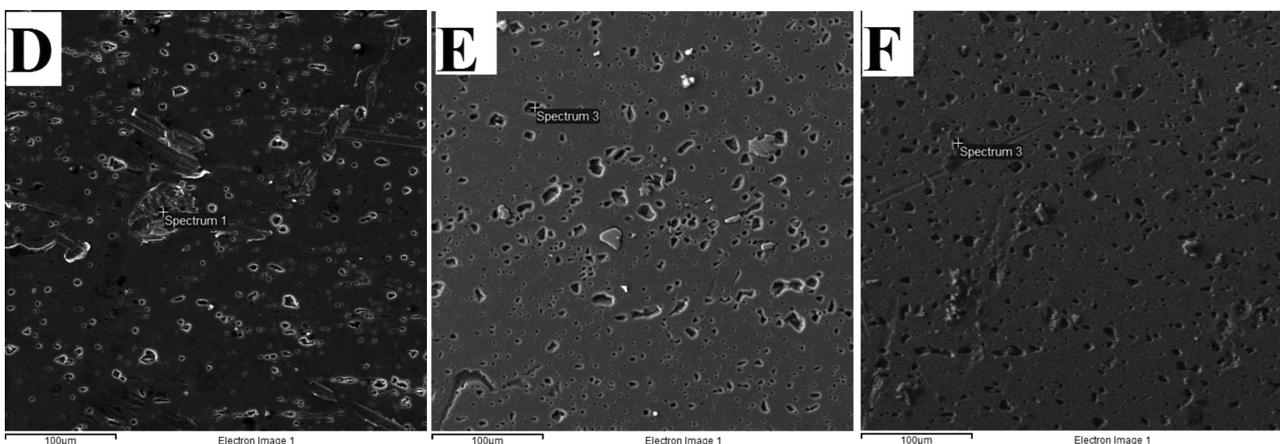


Figure 8: SEM photograph of grains after processing at a rotational speed of 1600 min^{-1} and travel rates of: d) 100 mm/min, e) 200 mm/min, f) 300 mm/min

Slika 8: SEM-posnetek zrn po obdelavi s hitrostjo vrtenja 1600 min^{-1} in hitrostjo pomikanja: d) 100 mm/min, e) 200 mm/min, f) 300 mm/min

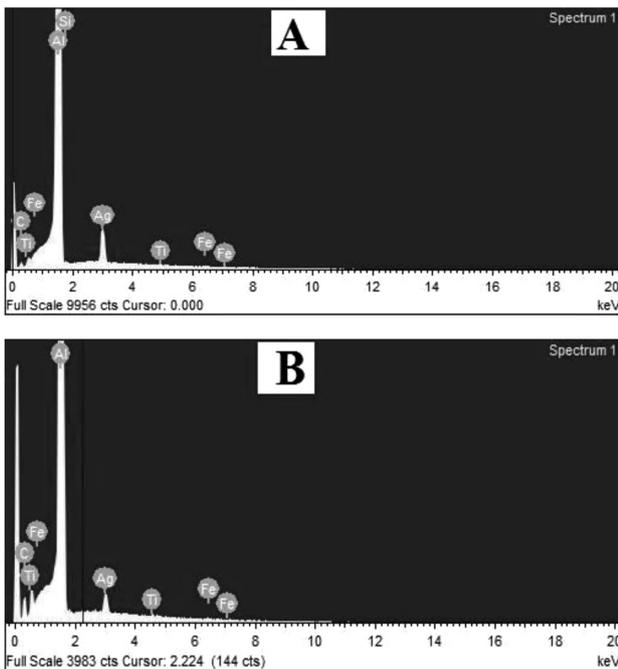


Figure 9: EDS of debris of FSP specimen processed at rotational speed of: a) 1200 min⁻¹ and b) 1600 min⁻¹

Slika 9: EDS-zlomnine iz FSP vzorca, obdelanega s hitrostjo vrtenja: a) 1200 min⁻¹ in b) 1600 min⁻¹

present in the specimen, which implies that the worn out particles/debris mainly result from the tools/backing plate and/or dissociation of the TiC. However, tool-wear debris is inevitable during FSW/FSP and reported in the FSP specimen layers in a scattered fashion by A. Kahri-zsangi and S.vKashani-Bozorg and Y. Wang et. al.^{23,28}

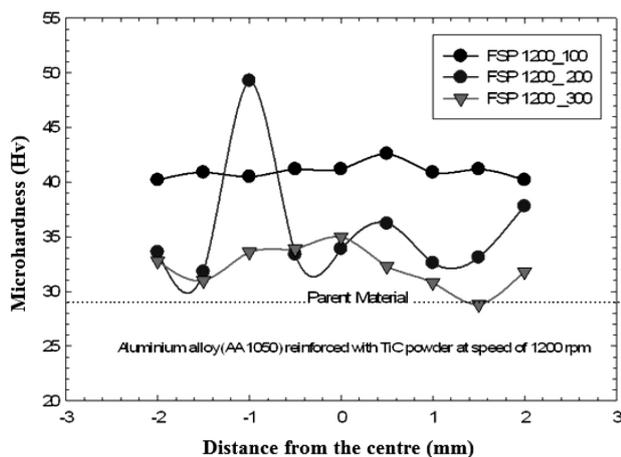


Figure 10: Vickers microhardness of friction-stir processed AA1050 reinforced with TiC powder processed at the rotational speed of 1200 min⁻¹ and different travel rates: the lower dashed line shows the parent material's condition

Slika 10: Vickers mikrotrdota predelanega AA1050 z mešanjem, s trenjem in ojačanega s TiC v prahu, obdelanega pri hitrosti vrtenja 1200 min⁻¹ in različnih hitrostih premika: spodnja črtkana črta kaže pogoje pri izvorni zlitini

3.4 Hardness profiling

Figures 10 and 11 shows the Vickers microhardness of friction-stir processed AA1050 reinforced with TiC powder processed at the rotational speeds of 1200 min⁻¹ and 1600 min⁻¹, respectively, at different travel rates of (100, 200 and 300) min⁻¹. The microhardness was measured on the matrices of the parent material and friction-stir processed materials with the lower dashed straight horizontal line indicating the homogeneity of the parent material. The hardness shows a significant improvement of the friction-stir processed material compared to the parent metal. The increase in the hardness can be attributed to the presence of the TiC particles in the matrix of the composite and the grain refinement of the matrix. The hardness across the friction-stir processed zone is not constant and the peak hardness value was observed at the point where the TiC was spotted. The average hardness values of the processed materials and the parent material are shown in **Figure 11**. The average hardness value of the parent materials is 28.9 HV and the average hardness of the processed materials after using 1200) min⁻¹ and at (100, 200 and 300) mm/min are (41, 36 and 32) HV, respectively. The hardness results of the processed materials after using 1600 min⁻¹ and at (100, 200 and 300) mm/min for processing were (44, 34 and 32) HV, respectively.

3.5 Ultimate tensile strength

D. Tabor²⁹ has shown that the ratio of ultimate tensile strength (UTS) to the Vickers hardness is related to the strain hardening coefficient. The relation between these parameters was expressed using Equation (1):

$$UTS = \left(\frac{H}{C}\right)^{1-n} \left(\frac{12.5n}{1-n}\right)^n \quad (1)$$

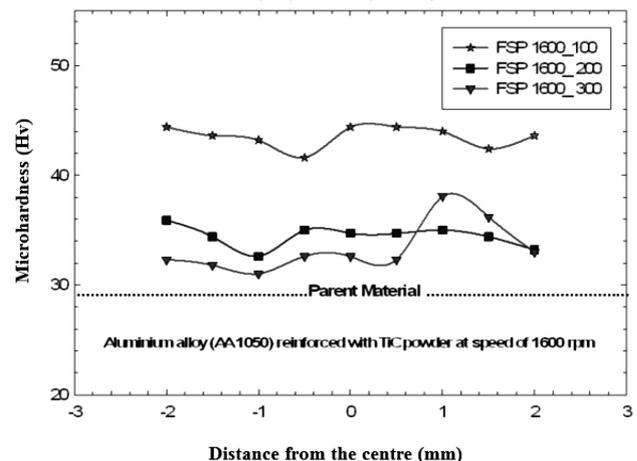


Figure 11: Vickers microhardness of friction-stir processed AA1050 reinforced with TiC powder processed at the rotational speeds of 1600 min⁻¹ and different travel rates: the lower dashed line shows the parent material's condition

Slika 11: Vickers mikrotrdota obdelanega AA1050 z mešanjem s trenjem, ojačanega s TiC v prahu, obdelanega pri 1600 min⁻¹ in pri različnih hitrostih pomikanja: nižja črtkana črta prikazuje osnovni material

Where UTS is the ultimate tensile strength, H is the Vickers hardness, n is the strain-hardening coefficient, and C is a constant that has a value of 2.9 for mild steels and ~ 3.0 for copper alloys and aluminium. This relation was further improved by J. R. Cahoon³⁰ in the form of Equation (2):

$$UTS = \left(\frac{H}{2.9} \right) \left(\frac{n}{0.217} \right)^n \quad (2)$$

where H is the Vickers hardness and n is the strain-hardening coefficient.

Using Equation (2), the UTS were calculated and plotted in **Figure 13**. From **Figure 13** it was observed that the parent material has a lower ultimate tensile strength compared to the materials processed by friction-stir processing reinforced with the TiC powder. It was also observed that the materials processed using rotational speeds of 1200 min⁻¹ and 1600 min⁻¹ and a travel rate of 100 have higher ultimate tensile strengths compared to the others. The trends in **Figure 13** also correlate with the hardness results in **Figure 12**.

3.6 Wear-volume losses

The wear-volume loss of the samples after the wear test is shown in **Figure 14** as a function of the process parameters for loads of 25 N with a sliding distance of 2000 m. It has been shown theoretically that the mass loss can be estimated using the Archard Equation (3):

$$V = K \frac{SL}{3H} \quad (3)$$

where V is the volume worn away during testing, S is the total sliding distance, L is the normal load, H is the hardness of the softer surface, and K is a dimensionless wear coefficient specific to the sample under test. It is reasonable to expect from Equation (1) that the materials after FSP will experience a smaller mass loss

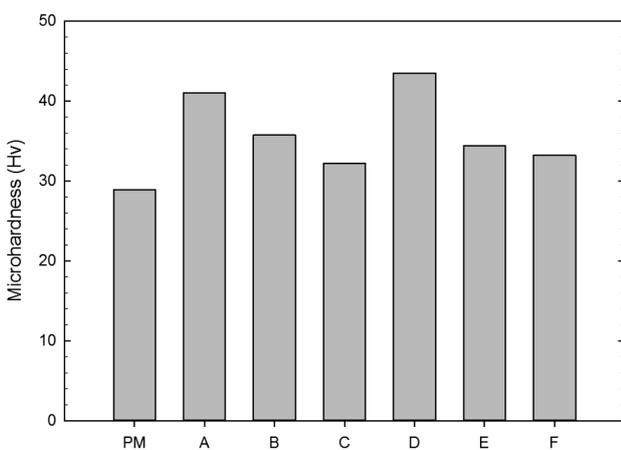


Figure 12: Average microhardness of parent material and friction-stir processed AA1050 reinforced with TiC powder

Slika 12: Povprečna mikrotrdota osnovnega materiala in AA1050, ojačanega s TiC v prahu po obdelavi z mešanjem s trenjem

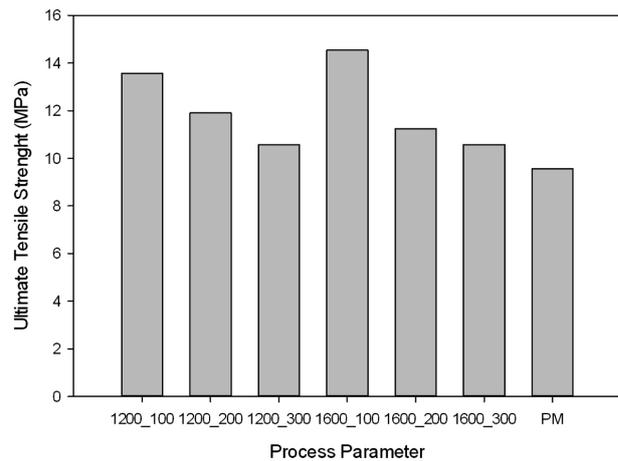


Figure 13: The ultimate tensile strength of parent material and friction-stir processed AA1050 reinforced with TiC powder

Slika 13: Natezna trdnost osnovnega materiala in AA1050 ojačanega s TiC, obdelanega z mešanjem s trenjem

and it shows in **Figures 9 to 11** that the samples processed by FSP have a higher hardness than the parent material sample, which also shows in **Figure 14** that the samples processed by FSP exhibit a lower mass loss compared to the parent material. Wear resistance is usually better for the MMC than the base metal for all the process parameters. This is due to the fact that the composite surface layer provides better wear resistance and it is harder than the base material.

The dimensional wear coefficient, k , is widely used to compare the wear rates in different classes of materials and is defined by Equation (4):

$$K = \frac{V}{SL} \quad (4)$$

The calculation suggests that under a load of 25 N and at a 2000 m sliding distance, the dimensional wear coefficient for the samples processed and the parent

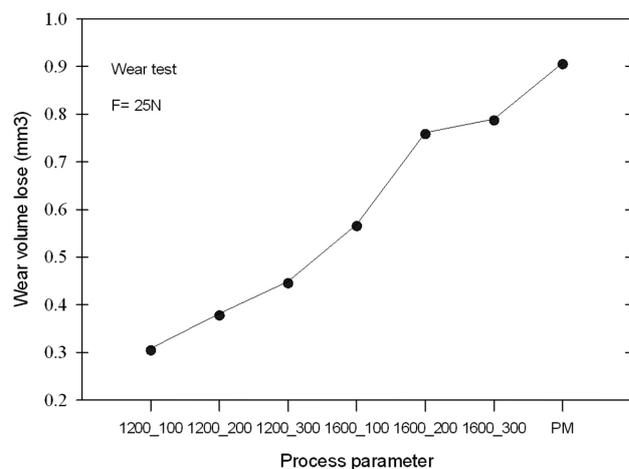


Figure 14: Wear-volume loss of processed samples for different parameters

Slika 14: Volumske izgube pri obrabi vzorcev, obdelanih z različnimi parametri

material are shown in **Table 3**. These results confirm the feasibility of using FSP with larger bulk samples, especially for the optimization of surface properties such as the wear-resistance behaviour.

Table 3: Dimensional wear coefficient

Tabela 3: Koeficient dimenzijske obrabe

Designation	Dimensional wear coefficient (mm ² N ⁻¹)
FsP 1200_100	6.1712×10^{-6}
FsP 1200_200	7.6321×10^{-6}
FsP 1200_300	8.977×10^{-6}
FsP 1600_100	1.378×10^{-5}
FsP 1600_200	1.5227×10^{-5}
FsP 1600_300	1.580×10^{-5}
Parent Material	1.815×10^{-5}

4 CONCLUSION

In the present study, FSP was performed on an aluminium alloy (AA 1050) with TiC powder in order to investigate the evolving microstructural and mechanical properties. A surface composite layer was successfully fabricated on the aluminium sheets and the following conclusions were drawn from the test outcomes:

- The microstructural evolution of the AA1050 during FSP showed that the TiC particles were more uniformly distributed in the AA1050 after FSP using 1200 min⁻¹ and at 100 mm/min, while at a high rotational speed of 1600 min⁻¹, it was observed that the TiC particles were clustered in some parts of the stirred zone after the FSP.
- The hardness tests showed an improvement and an enhancement on the integrity of the processed materials due to the improvement in the evolving microstructure.
- The wear-resistance test results confirm the feasibility of using FSP for the optimization of surface properties of the composites produced.

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