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MICROSTRUCTURE DEPENDENCE OF AL6061 SURFACE COMPOSITE ON TOOL ROTATION SPEED DURING FRICTION STIR PROCESSING

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Abstract. Aluminium (Al) based alloys are used in aerospace and automotive industries due to their high specific stiffness and high specific strength. To enhance their performance, often their mechanical properties are improved by making composites via incorporation of ceramic particles as reinforcement. For requirements, such as high wear resistance, high surface hardness is essential, and therefore making of their 'surface composites' suffices. Friction stir process (FSP) is an effective technique to produce surface composites. By varying tool rotation speed, microstructure can be controlled to achieve high hardness. In this work, aluminium alloy Al6061 based surface composites containing silicon carbide and alumina microparticles were made by FSP method. Surface composites were produced at three tool rotation speeds (rpm: 600, 800, 1000). Composites were characterized for their microstructure, i.e. grain size, at four distinct zones, namely, nugget zone (stir zone), heat affected zone, thermo-mechanically affected zone and base metal. Microhardness was measured for the composites at their nugget zone (stir zone) and for the base metal. Hardness of the composites was higher than the base metal, due to recrystallized microstructure i.e. reduction in grain size, and uniform distribution of ceramic particles and their strengthening mechanisms. With increase in tool rotation speed, the grain size in the composites decreased and consequently their hardness increased, such that, at the highest speed (1000 rpm), the grain size at the stir zone was smaller by an order of magnitude and the hardness was three times higher, compared to those of the base metal. Dependence of grain size (and concomitant increase in hardness) on tool rotation speed provides an effective route for microstructure control and hardness enhancement during processing of surface composites, without resorting to post-fabrication secondary processes.

Keywords: friction stir process (FSP), Al6061 alloy, ceramic reinforcements, microstructure, microhardness

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Оригинальная статья

ЗАВИСИМОСТЬ МИКРОСТРУКТУРЫ ПОВЕРХНОСТНОГО КОМПОЗИТА AL6061 ОТ СКОРОСТИ ВРАЩЕНИЯ ИНСТРУМЕНТА ПРИ ОБРАБОТКЕ ТРЕНИЕМ С ПЕРЕМЕШИВАНИЕМ

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Аннотация. Сплавы на основе алюминия (Al) используются в аэрокосмической и автомобильной промышленности из-за их высокой удельной жесткости и удельной прочности. С целью улучшения их механических свойств изготавливают композиты, включающие керамические частицы в качестве армирующих элементов. Высокая твердость поверхности, необходимая для получения высокой износостойкости, обеспечивается изготовлением «поверхностных композитов». Обработка трением с перемешиванием (ОТП) является эффективным методом производства поверхностных композитов, поскольку, дает возможность контролировать микроструктуру, изменяя скорость вращения инструмента. В настоящей работе поверхностные композиты на основе алюминиевого сплава Al6061, содержащие микрочастицы карбида кремния и оксида алюминия, были получены методом ОТП. Поверхностные композиты изготавливались на трех скоростях вращения инструмента (600, 800, 1000 об./мин). Приводится характеристика микроструктуры композитов, а именно размера зерна в четырех различных зонах – в зоне точечной сварки (зона перемешивания), в зоне термического влияния, в зоне термомеханического воздействия и в основном металле. Микротвердость композитов измеряли в зоне точечной сварки (зоне перемешивания) и в основном металле. Твердость композитов была выше, чем у основного металла из-за рекристаллизованной микроструктуры, то есть уменьшения размера зерна и равномерного распределения керамических частиц и механизмов их упрочнения. С увеличением скорости вращения инструмента размер зерен в композитах уменьшался и, следовательно, увеличивалась их твердость, при этом на максимальной скорости вращения (1000 об./мин) размер зерен в зоне перемешивания был на порядок меньше, а твердость в три раза выше, чем у основного металла. Зависимость размера зерна (и сопутствующего увеличения твердости) от скорости вращения инструмента обеспечивает эффективный способ контроля микроструктуры и повышения твердости во время обработки поверхностных композитов, не прибегая к их дополнительной обработке после изготовления.

Ключевые слова: обработка трением с перемешиванием, сплав Al6061, керамическое армирование, микроструктура, микротвердость

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Introduction

Friction stir processing (FSP) was developed based on the basic principles of friction stir welding (FSW, a solid-state joining process). Using FSP, control of microstructure can be achieved at surface/near-surface regions of processed metallic components [1, 2]. FSP involves movement of a rapidly rotating, non-consumable tool over the surface of a work piece. As the surface of a metallic material is friction stir processed, it undergoes intense plastic deformation, material mixing and experiences thermal heating owing to the friction caused by the mechanical motion of tool. These occurances cause significant microstructural changes, such as, grain refinement, densification and homogenization of processed zone. FSP is used to produce: (*i*) fine-grained structure, (*ii*) surface alloying, (*iii*) surface composites (*ex situ*), and (*iv*) intermetallic compounds, and thereby *in situ* composites [1, 2].

FSP is a local thermo-mechanical metal-working process, which changes local properties of metallic materials, without affecting their bulk material properties. A schematic illustration of FSP is shown in Fig. 1 [3]. During FSP, a cylindrical non-consumable tool is made to rotate, and the rotating tool is plunged into a selected area on a work piece whose surface is to be modified. Typically, a FSP tool is a pin having small diameter in dimension, and has a shoulder whose diameter is larger than that of the pin (Fig. 1).

When a tool is plunged into a metallic sheet, the rotating pin contacts the surface, and friction generated thereof between sheet material and shoulder rapidly heats a small region of metallic sheet. This also enables transverse movement of the tool over the metallic sheet. Depth of penetration of tool into metallic plate is determined by the height of pin, as penertation is restricted by its shoulder [1 – 3]. During FSP, the area of a metallic sheet to be processed and the tool are moved relative to each other, such that the rotating tool traverses with overlapping passes, until the entire selected area is processed. The processed zone cools as the tool passes, giving rise to defect free, dynamically recrystallized and equiaxed fine-grained microstructure [1 – 3]. Such beneficial changes in microstructure enhance surface mechanical properties of the processed materials. Some of the process variables of FSP include: tool plunge depth, tool rotation speed, tool transverse speed, tool tilt angle, pin geometry (i.e. length, diameter, profile of pin and shoulder), num-

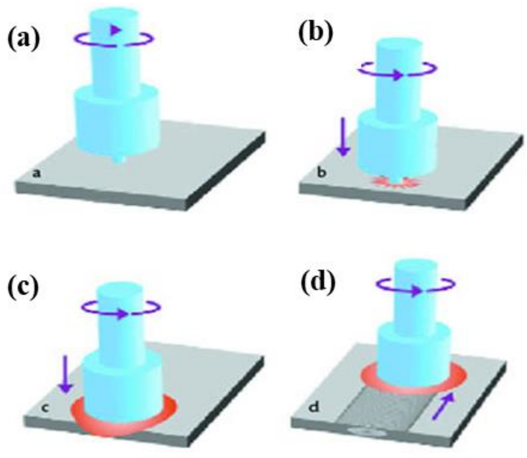

Fig. 1. Schematic illustration of friction stir processing:
a – rotating tool prior to contact with a metallic plate; *b* – rotating tool pin makes contact with the metallic plate, creating heat; *c* – shoulder makes contact, restricting further penetration into the metallic plate; *d* – metallic plate moves relative to rotating tool [3]
Рис. 1. Схематическое изображение обработки трением с перемешиванием:
a – вращающийся инструмент до контакта с металлической пластиной; *b* – вращающийся штифт инструмента контактирует с металлической пластиной, выделяя тепло; *c* – бурт соприкасается, ограничивая дальнейшее проникновение в металлическую пластину; *d* – металлическая пластина движется относительно вращающегося инструмента [3]

ber of passes of tool over surface, thermal and mechanical properties of alloy/matrix, reinforcement type, particle size, reinforcement content etc [1, 2, 4]. FSP is used to modify microstructure and mechanical properties of materials such as aluminium, magnesium, nickel, copper etc.

Aluminium, magnesium and titanium are lightweight materials, i.e. materials with lower densities. These materials have good strength-to-weight ratio. Due in part to this, they are suitable for weight-saving applications [5]. Light-weighting of structures is a major requirement in aerospace, automotive, consumer electronics and sports sectors [6, 7]. However, aluminium and its alloys are relatively soft, mainly due to their face centered cubic (f.c.c) structure, and thus have low hardness, low mechanical strength and low wear resistance [8]. Surface and bulk mechanical properties of Al materials can be improved by modification of their microstructure right during their processing stage [9]. FSP is a suitable method for surface modification of Al materials. Few examples are highlighted here. Castings of A356 Al-alloy usually contain porosity [10], which is undesirable as it reduces strength and ductility, causes lowering of corrosion resistance, and severely limits fatigue life. By friction stir processing cast A356 Al-alloy, its: (*i*) casting porosity can be eliminated, (*ii*) microstructure can be homogenized, and can be recrystallized, and made fine grained, (*iii*) tensile strength can be increased by 18% and (*iv*) ductility can be increased by 6 times [10]. FSP can also improve formability of Al alloys [10]. As an example, when 2519 aluminium plate was subjected to FSP, its formability (in terms of capability to bend without fracture) altered significantly. While the unmodified plate failed upon its bending by 30^o, the friction stir processed plate could be bent even up to 80^o, without cracking.

Using FSP method, surface composites of Al materials can be produced [4, 11]. Dispersion of secondary phase particles over Al metal/alloys is achieved by the stirring action of a tool. Different approaches for incorporating reinforcement particles into Al matrices during their friction stir processing include [12]: *a* – hole drilling approach: particles are filled into holes that are drilled in straight/zig-zag pattern on top of a work piece, *b* – groove filling approach: a groove is created on a work piece and filled with reinforcement particles; *c* – sandwich approach: a layer of reinforced particles is sandwiched between matrix material plates. Mechanical work by tool during preparation of surface composites breaks reinforcement particles, and the increase in number of passes provides uniform distribution of particles in matrix material [4, 10 – 12].

2. Experimental Procedure

2.1 Materials and Processing

Aluminium Al6061 plate was used as the test material. Al6061surface composites were prepared by FSP via groove filling approach. A groove of about 2 mm width was cut on the Al6061 plate and was filled with silicon carbide (SiC, 10 μm) and alumina (Al_2O_3, 4 μm) particles taken in equal weight fraction (3 %). FSP tool was rotated continuously and was allowed to contact the metal surface over the groove filled with ceramic particles. The tool tip (i.e. plunger) was plunged into the metal for a duration of about 8 seconds. FSP was conducted at tool rotation speed (rpm) of 600 (Composite 1), 800 (Composite 2) and 1000

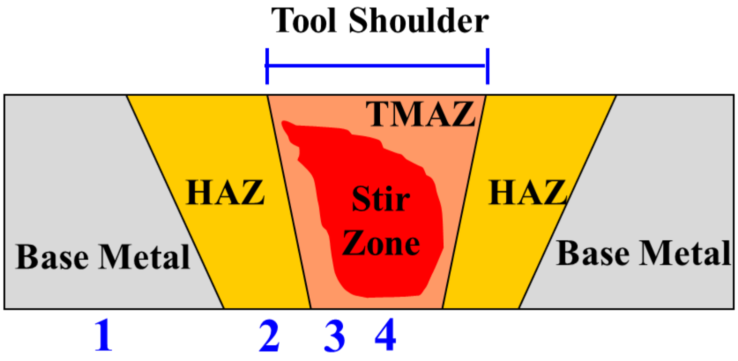

Fig. 2. Four zones observed on a metal surface after friction stir process. Left to right: (1) base metal, (2) heat affected zone (HAZ), (3) thermo-mechanically affected zone (TMAZ) and (4) nugget zone (stir zone, FSP zone)

Рис. 2. Четыре зоны, наблюдаемые на поверхности металла после сварки трением с перемешиванием. Слева направо: (1) основной металл, (2) зона термического влияния, (3) зона термомеханического воздействия и (4) зону точечной сварки (зона перемешивания, зона ОТП)

(Composite 3). Transverse speed of the tool was kept constant at 60 mm/min. Tilt angle of the tool was 2 degrees.

2.2 Microstructure and Microhardness

Surfaces of composites were polished using SiC abrasive sheets (until 3000 grit size), followed by polishing using SiC suspension, and subsequently using diamond suspension. Polished specimens were ultrasonically cleaned in an ethanol bath. Keller's reagent was used to etch the cleaned surfaces. Grain size of the etched specimens was measured using optical microscopy. Vickers microhardness test was performed on the cross-section of the specimens (stir zone and base metal) using 100 g load, for 20 s.

3. Results and Discussion

3.1 Microstructure

FSP composites have four distinct zones (see schematic, Fig. 2), namely: *i* – base metal, *ii* – heat affected zone (HAZ); *iii* – thermo-mechanically affected zone (TMAZ); *iv* – nugget zone (stir zone, FSP zone) [12 – 14]. Microstructural features observed in these four zones of the Al6061 surface composites are discussed in this section.

TMAZ is the region that is in close proximity to the stir zone (Fig. 2). In this zone, the material experiences (*a*) friction generated due to the mechanical motion of the tool and (*b*) heat arising due to the generated friction. So the name 'TMAZ'. Grain size in the TMAZ is in the range 50 μm to 70 μm (Fig. 3, *d*). Grain size is larger in TMAZ than that in the base metal (40 μm) which is due to the higher temperature in the region that causes grain coarsening. However, due to the mechanical work by the tool, grains undergo mechanical deformation and hence, size of some grains is found to be lower (< 70 μm, but ≥ 50 μm) than that in the HAZ (70 μm, Fig. 3, *c*). (d) Nugget Zone (Friction Stir Processed Zone)

Nugget zone is the stir zone. Also known as FSP zone, as this is the region intended to be made into as a surface composite (Fig. 2). When compared to the other regions, stir zone experiences high plastic deformation, mechanical mixing of reinforcement particles with matrix material and generation of high temperature, followed by fast cooling [1, 2, 4, 10, 15]. Area in the zone is highly strain hardened and grains get considerably reduced in their size. In the present case, the grain size is < 25 μm (Fig. 3, *e*), which is 60 % smaller than that of the grains in the base metal (40 μm, Fig. 3, b).

3.1.2 Composite 2: Tool rotation speed 800 rpm

Optical images of Composite 2 are shown in Fig. 4, *a*. Base metal contains agglomerated and interconnected Mg_2Si secondary phase along the grain boundaries. The phase gets broken and becomes finer in size increasingly in the regions that are affected by the mechanical/shearing action of tool, as can be seen in the images of HAZ, TMAZ and nugget zone. Stir zone shows uniformly distributed secondary and reinforcement phases.

(*a*) Base Metal. Average grain size in bulk metal is 70 μm (Fig. 4, *b*), which is larger than that of the grain size in base metal of Composite 1 (600 rpm).

(*b*) Heat Affected Zone (HAZ)

Microstructure of the heat affected zone of Composite 2 is shown in Fig. 4, *c*. Grains have higher aspect ratio (i.e. length/diameter) when compared to those in the HAZ of Composite 1. Grains are found to be elongated along the transverse direction of the tool movement. Higher mechanical work and increased frictional heat generated due to the increase in rotational speed of the tool (compared to that during the formation of Composite 1, 600 rpm) causes the directionality of the grains. Grain size in the zone is in the range 50 μm to 90 μm.

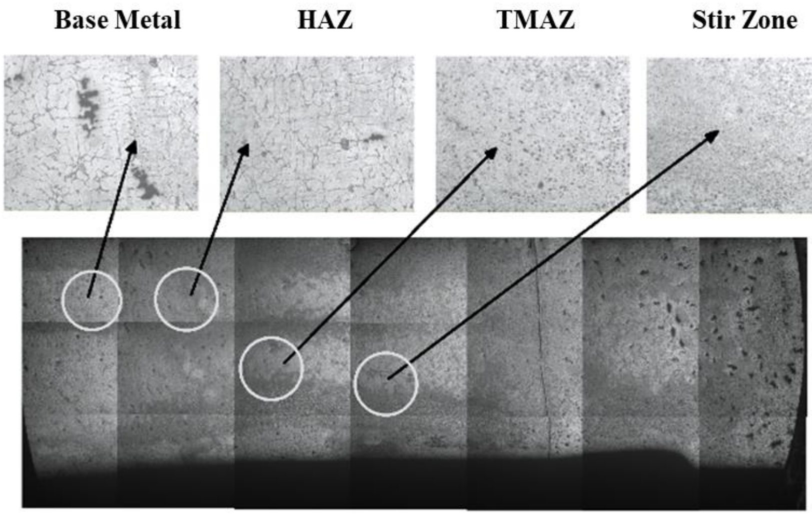

Fig. 3, *a*. Optical images of Composite 1 (processed at 600 rpm) showing various zones, left to right: base metal, HAZ, TMAZ and nugget zone (stir zone, FSP zone); (*a*) Base Metal; Average grain size in bulk metal is 40 µm (Fig. 3, *b*). (*b*) Heat Affected Zone (HAZ)

Рис. 3, *a*. Оптические изображения композита 1 (обработанные при 600 об./мин), показывающие различные зоны слева направо: основной металл, зона термического влияния, зона термомеханического воздействия и зону точечной сварки (зона перемешивания, зона ОТП); *a*) основной металл; средний размер зерна в массивном металле составляет 40 мкм (рис. 3, *b*). (*b*) Зона термического влияния

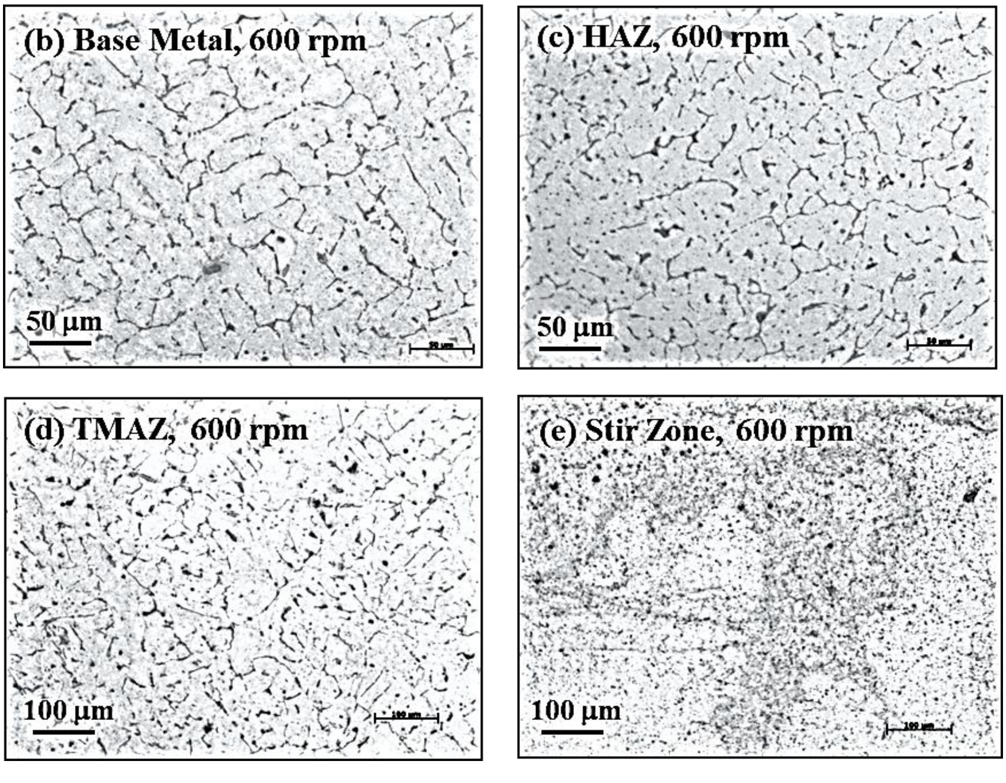

Fig. 3, *b* – *e*. Optical images taken at higher magnification of Composite 1 (processed at 600 rpm) showing various zones: (*b*) base metal, (*c*) HAZ, (*d*) TMAZ and (*e*) nugget zone (stir zone, FSP zone). (*c*) Thermo-Mechanically Affected Zone (TMAZ)

Рис. 3, *b* – *e*. Оптические изображения, полученные при большем увеличении композита 1 (обработанного при 600 об./мин), показывающие различные зоны:
(*b*) основной металл, (*c*) зону термического влияния, (*d*) зону термомеханического воздействия и (*e*) зону точечной сварки (зона перемешивания, зона ОТП). (*c*) Зона термомеханического воздействия

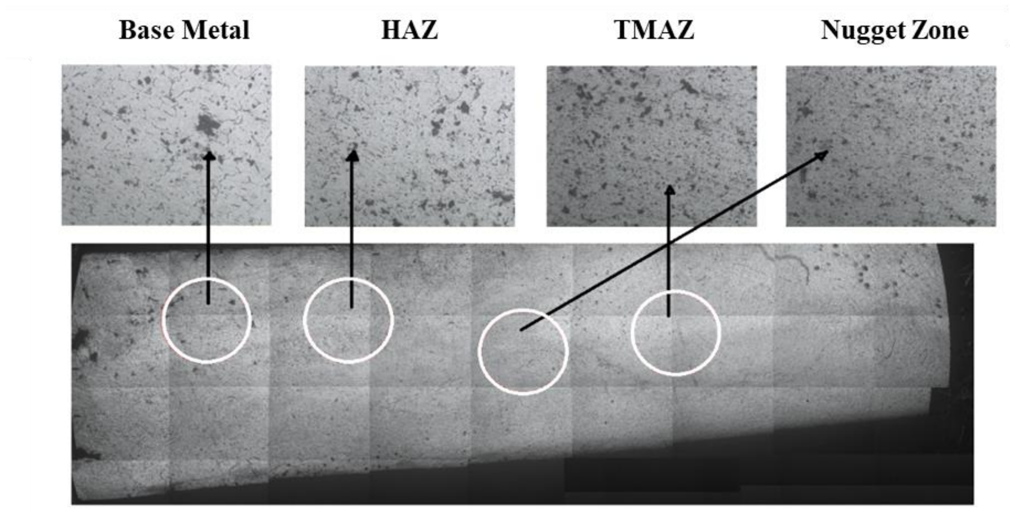

Fig. 4, *a*. Optical images of Composite 2 (processed at 800 rpm) showing various zones:
b – base metal; *c* – HAZ; *d* – TMAZ; *e* – nugget zone (stir zone, FSP zone)
Рис. 4, *а*. Оптические изображения композита 2 (обработанные при 800 об./мин) с различными зонами: *b* – основной металл; *c* – зона термического влияния; *d* – зона термомеханического воздействия; *e* – зона точечной сварки (зона перемешивания, зона ОТП)

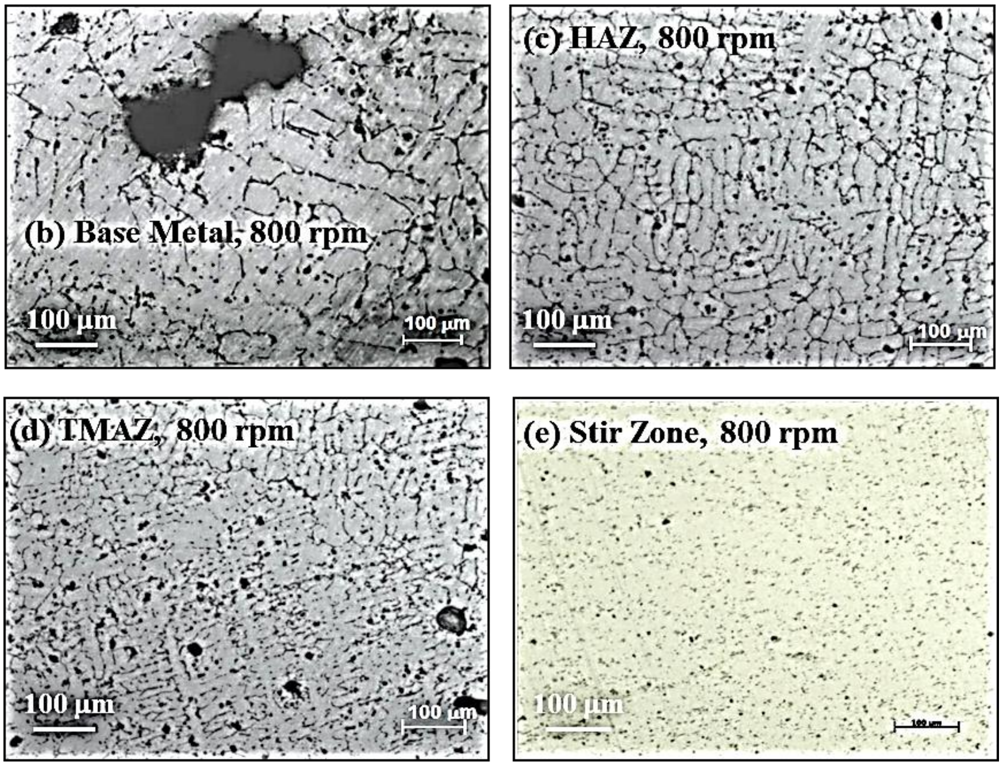

Fig. 4, *b*–*e*. Optical images taken at higher magnification of Composite 2 (processed at 800 rpm) showing various zones:
b – base metal; *c* – HAZ; *d* – TMAZ; *e* – nugget zone (stir zone, FSP zone)
Рис. 4, *b*–*e* Оптические изображения, полученные при большем увеличении композита 2 (обработанные при 800 об./мин), показывающие различные зоны: *b* – основной металл; *c* – зона термического влияния; *d* – зона термомеханического воздействия; *e* – зона точечной сварки (зона перемешивания, зона ОТП)

(*c*) Thermo-Mechanically Affected Zone (TMAZ)

Grain size in the TMAZ is in the range 40 μm to 70 μm (Fig. 4, *d*). Higher thermal input and severe plastic deformation broughtforth by the increase in tool speed (compared to that during the formation of Composite 1, 600 rpm), followed by faster cooling, produces relatively narrower grains.

(*d*) Nugget Zone (Friction Stir Processed Zone)

Microstructure of the nugget zone of Composite 2 is shown in Fig. 4, *e*. Grains have undergone refinement and their size is < 20 µm, which is slightly smaller than that of the grains in the nugget zone of Composite 1 (< 25 µm, Fig. 3, *e*).

3.1.3 Composite 3: Tool rotation speed 1000 rpm

Optical images of Composite 3 are shown in Fig. 5, *a*.

(*a*) Base Metal

Average grain size in bulk metal is 100 µm (Fig. 5, *b*), which is larger than that of the grain size in base metal of Composite 2 (600 rpm). Increase in size of grains in base metal with increase in tool speed has been reported earlier [16, 17], which is attributed to high temperature that causes grain coarsening. Second phase particles (Mg_2Si) are seen distributed along the grain boundaries.

(*b*) Heat Affected Zone (HAZ)

Microstructure of the heat affected zone of Composite 3 is shown in Fig. 5, *c*. Grain size in the zone is in the range 60 to 80 µm, which is a narrower range when compared to that of the range of the grain size in the HAZ of Composite 2 (50 µm to 90 µm, Fig. 4, *c*).

(*c*) Thermo-Mechanically Affected Zone (TMAZ)

Average grain size in the TMAZ is about 60 µm. Grains are found to be elongated along the transverse direction of the tool motion.

(*d*) Nugget Zone (Friction Stir Processed Zone)

Microstructure of the nugget zone of Composite 3 is shown in Fig. 5, *e*. Grains have undergone significant grain refinement (grain size < 10 µm). Grain size is lower by one order of magnitude compared to the size of grains in the base metal (100 µm, Fig. 5, *b*). Second phase and reinforcement particles have become finer in size due to their shearing caused by mechanical work of tool [11, 18], and they are uniformly distributed due to the stirring motion of tool [4, 14, 18].

Measured grain size at the four distinct zones of Al6061 surface composites are given in Table 1.

3.2 Microhardness

Microhardness values of base metal and stir zone of the surface composites as a function of tool rotation speed (rpm) is shown in Fig. 6. Trends show that with the increase in tool rotation speed (*a*) hardness of base metal decreases and (*b*) hardness in stir zone increases. In the range of the tool speed considered in the present work, at the highest speed (1000 rpm), the hardness in stir zone is (*a*) 3 times higher than that of base metal and (b) 1.8 times higher than that in stir zone of the composite processed at the lowest speed (600 rpm).

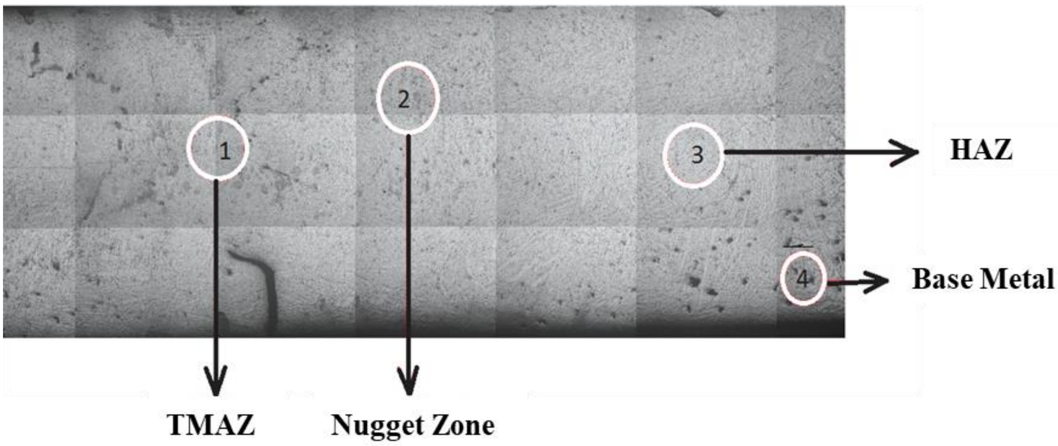

Fig. 5, *a*. Optical images of Composite 3 (processed at 1000 rpm) showing various zones:
b – base metal; *c* – HAZ; *d* – TMAZ; *e* – nugget zone (stir zone, FSP zone)

Рис. 5, *a*. Оптические изображения композита 3 (обработанные при 1000 об./мин), показывающие различные зоны:
b – основной металл; *b* – зона термического влияния; *d* – зона термомеханического воздействия; *e* – зона точечной сварки (зона перемешивания, зона ОТП)

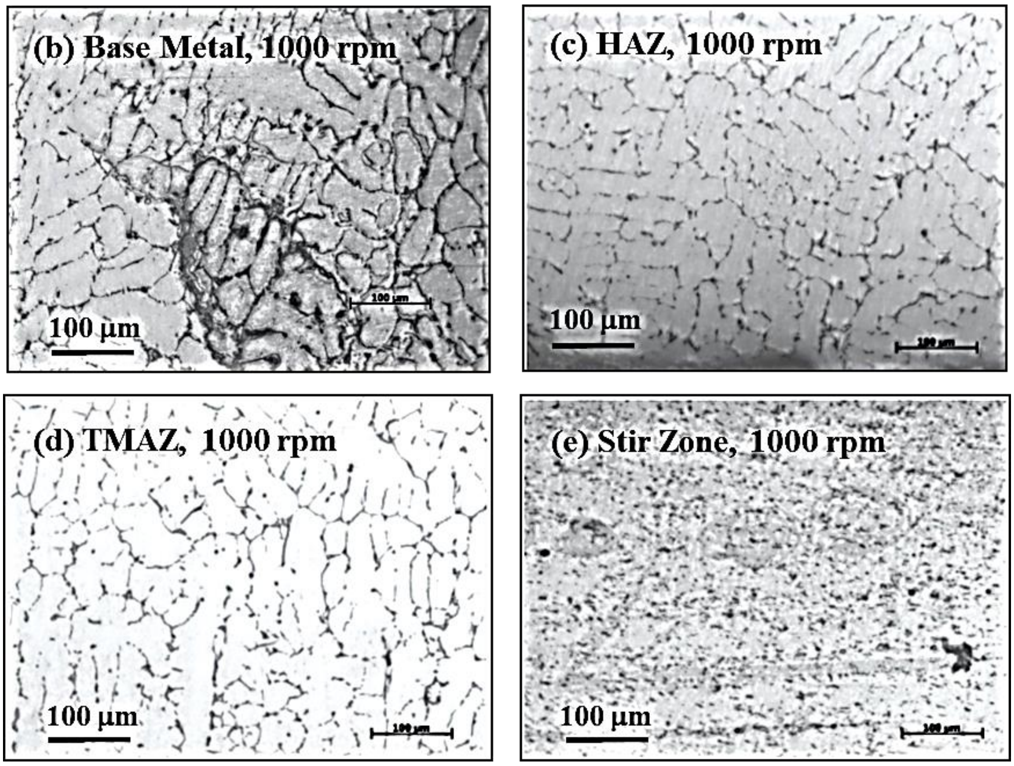

Fig. 5, *b*–*e*. Optical images taken at higher magnification of Composite 3 (processed at 1000 rpm) showing various zones: *b* – base metal; *c* – HAZ; *d* – TMAZ; *e* – nugget zone (stir zone, FSP zone)
Рис. 5, *b*–*e*. Оптические изображения, полученные при большем увеличении композита 3 (обработка при 1000 об./мин), на которых видны различные зоны: *b* – основной металл; *c* – зона термического влияния; *d* – зона термомеханического воздействия; *e* – зона точечной сварки (зона перемешивания, зона ОТП)

In Al6061 alloy, Mg_2Si is the secondary phase that increases hardness and strength of the alloy, when compared to pure Al. As the alloy is friction stir processed, high heat is generated at stir zone due to frictional heating and plastic deformation. During the processing of Al6061 surface composites, extraction of heat by the bulk of the alloy at the vicinity of tool contact causes a rise in temperature in the base metal. Consequently, (*a*) grain size in the base metal increases and (*b*) partial dissolution of the secondary phase occurs [11]. Together, these two occurances result in the decrease in hardness value of the base metal. With increase in tool rotation speed, grain size increases further, causing further decrease in hardness values of the base metal (Fig. 6).

Considering grain size (Table) and hardness values at stir zone (Fig. 6), it is seen that (*a*) hardness is inversely dependent on grain size and (*b*) increase in tool rotation speed decreases grain size that eventually increases hardness.

Surface composites show high hardness in stir zone due to (*a*) presence of inherently hard SiC and Al_2O_3 ceramic particles and (*b*) grain refinement. Friction stir processing of Al6061 alloy to form surface composites gives rise to:

i – uniform distribution of ceramic particles in stir zone, which act as heterogenous sites for grain nucleation;

Measured grain size in Al6061 surface composites (Composite 1 – 600 rpm, Composite 2 – 800 rpm and Composite 3 – 1000 rpm)
Измеренный размер зерна в поверхностных композитах Al6061 (композит 1 – 600 об./мин, композит 2 – 800 об./мин и композит 3 – 1000 об./мин)

Tool Speed, rpm	Grain Size, µm			
	Base Metal	Heat Affected Zone (HAZ)	Thermo-Mechanically Affected Zone (TMAZ)	Stir Zone (SZ) or Nugget Zone
600	40	70	50 to 70	< 25
800	70	50–70	50–90	< 20
1000	100	60–80	60	< 10 (Very Fine)

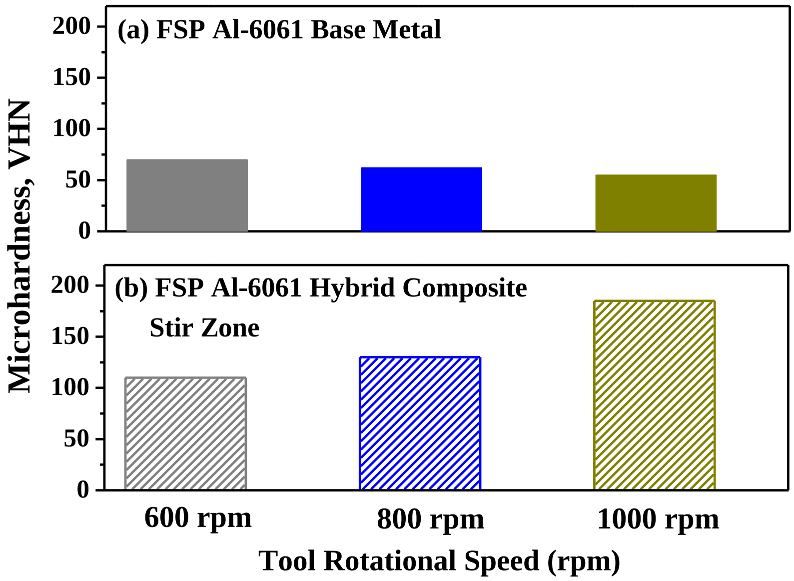

Fig. 6. Microhardness values of (*a*) base metal and (*b*) stir zone of the surface composites as a function of tool rotation speed (rpm)

Рис. 6. Значения микротвердости основного металла (*а*) и зоны перемешивания (*b*) поверхностных композитов в зависимости от скорости вращения инструмента (об./мин)

ii – higher heat extraction and severe plastic deformation at stir zone. This aids in nuleation of fine grains at the stir zone, resulting in dynamically recrystallized microstructure;

iii – breakdown in size of Mg_2Si secondary phase network;

iv – high heat generation at stir zone that enhances binding of reinforcement with matrix;

v – Orowan strengthening: hard ceramic particles obstruct dislocation motion during deformation (i.e. strain hardening) [19].

By increasing hardness of composites, their wear resistance can be increased, as wear of a material is inversely related to its hardness, as according to Archard's law [20].

Processing of Al–Mg alloys/composites by conventional liquid-state methods (e.g. stir casting), fusion techniques (e.g. thermal spraying), laser surface modification has certain disadvantages, namely (*a*) porosity and (*b*) formation of brittle Al_4C_3 phase due to interaction of SiC with molten Al. Both these occurances are undesirable, as they adversely affect mechanical properties of materials. In FSP, (*a*) porosity is absent/eliminated and (*b*) extraction of heat from processing zone by the surrounding region is fast, and thereby the interaction time is less, and as a result undesirable phases (e.g. Al_4C_3) do not form [21].

Conclusions

Al6061 surface composites containing SiC and Al_2O_3 microparticles were fabricated by friction stir processing method, at three tool rotation speeds (rpm: 600, 800, 1000). Composites were characterized for their grain size and hardness. Conclusions that could be drawn from the work are:

FSP is an effective method to produce surface composites;

rotational speed of tool influences microstructure that consequently alters hardness;

increase in tool rotation speed reduces grain size at stir zone, attributed to (*a*) dynamically recrystallized microstructure and (*b*) reinforcement particles acting as grain nucleation sites;

microhardness at stir zone increases with increase in tool speed, due to: (*a*) grain refinement and (*ii*) Orowan strengthening.

It is expected that reduced grain size and enhanced surface hardness would provide better wear resistance to the processed materials.

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