MANUFACTURING OF NANO/MICRO COMPOSITES USING FRICTION STIR PROCESSING

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INTRODUCTION

Aluminum and its alloys are used extensively in aerospace and automotive industries because of its low density and high strength to weight ratio. However, a poor resistance to wear and erosion is of serious concern for prolonged use. Metal matrix composites are most promising new class of materials that exhibit good wear and erosion resistance properties, higher stiffness and hardness at a lower density as compared to the matrix. This is due to the presence of nano and micro-sized reinforcement particles into the matrix. Aluminum matrix composites (AMCs) reinforced with particles and whiskers are widely used for high performance applications such as in automotive, military, aerospace and electricity industries because of their improved physical and mechanical properties In the composites relatively soft alloy like aluminum can be made highly resistant by introducing predominantly hard but brittle particles such as Al_2O_3. Hard particles such as Al_2O_3, SiC], TiC, TiO_2, Cr_2O_3, or mixture of them, and others are commonly used as reinforcement in the composites. The application of Al_2O_3 particle reinforced aluminum alloy matrix composites in the automotive and aircraft industries is gradually increasing for pistons, cylinder heads, connecting rods etc. where the tribological properties of the materials are very important. However, the presence of the ceramic particles in the metallic matrix result in higher strength and hardness, often at the expense of some ductility which makes the matrix brittle.

In this regard, it may however be noted that wear is a surface dependent degradation mode, which may be improved by a suitable modification of surface microstructure and/or composition.

Hence, instead of bulk reinforcement, if the ceramic particles would be added to the surface, it could improve the wear and erosion resistance without sacrificing the bulk properties. The enhancement of mechanical properties in the novel nano-particle reinforced MMCs has been reviewed recently. Dispersion of the nano-reinforcements particles on metallic substrate surface and the control of its distribution in a uniform manner is a critical and difficult to achieve by conventional surface treatments

Fabrication Of Metal Matrix Composites

Conventional Methods

Fabrication of MMCs had been carried out using various methods. Those methods are based on surface modification techniques which include casting, cast sinter, high-energy electron beam irradiation, high-energy laser melt treatment. plasma spraying. In Laser technique, metal-matrix composites using either carbide powder (SiC, TiC, or WC), or combination of carbide powders and a binding material (Co, Al, or Ni) could be obtained.

In the above-mentioned techniques, it is hard to avoid the interfacial reaction between reinforcement and metal- matrix and the formation of some detrimental phases because these processing techniques are based on liquid phase processing at high temperatures. Furthermore, critical control of processing parameters is necessary to obtain ideal solidified microstructure in surface layer. Moreover,

using conventional surface modification techniques makes it difficult to achieve successful dispersion of fine ceramic particles in a surface layer. Obviously, if processing of

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surface composite is carried out at temperatures below melting point of substrate, the problems mentioned above can be avoided.

Friction Stir Processing Method

Recently, much attention has been paid to a new surface modification technique named friction stir processing (FSP). FSP is a solid state processing technique to obtain a fine-grained microstructure. It has been developed for microstructural modification by Mishra et al. based on the basic principles of friction stir welding (FSW).

FSW is a relatively new solid state joining process developed initially for aluminum alloys by The Welding Institute (TWI) of UK. FSW uses a non-consumable rotating tool with a specially designed

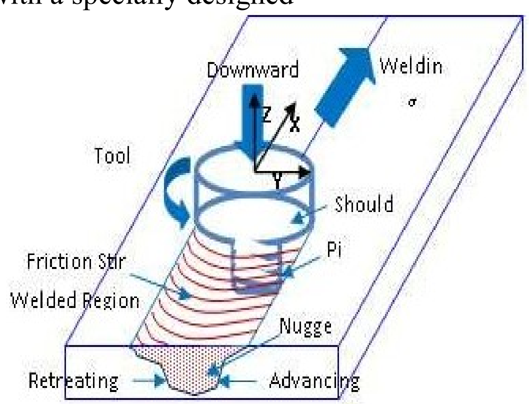


Figure 1: Principals of FSW.

pin and shoulder is plunged into the interface between two plates to be joined and traversed along the line of the joint as shown in **Figure 1**.

Localized heating is produced by the friction between the rotating tool and the workpiece to raise the local temperature of the material to the range where it can be plastically deformed easily. As the rotating tool traverses along the joint line, metal is essentially extruded around the tool before being forged by the large down pressure. It is well known that the stirred zone consists of fine and equiaxed grains produced due to dynamic recrystallization. Though FSP has been basically advanced as a grain refinement technique, it is a very attractive process for also fabricating composites. Mishra et al fabricated the Al/Si C surface composites by FSP, and indicated that Si C particles were well distributed in the Al matrix, and good bonding with the Al matrix was generated.

Manufacturing of Nano/Micro Composites using Friction Stir Processing

Nano/Micro Metal Matrix Composites

Metal matrix composites containing nano-sized reinforcement particles inserted into the matrix are promising materials due to the enhancement in mechanical properties. Friction stir processing technique is used in manufacturing nano composites. Various reinforcement particles materials were applied in the metals matrices (metallic substrates) which were also varied from aluminum alloys to copper alloys and other easy friction stir processed metal alloy.

Metallic substrate Aluminum alloys

Aluminum alloys are the most widely applied metallic substrate for producing nano composites. There also applied in case of micro composites. Aluminum alloy AA5083 and other aluminum alloys such as AA2618, AA5052 and aluminum magnesium alloy were the most aluminum alloys received attention from researchers in the recent years concerning metal matrix composites.

Other Metals and Alloys

Copper was used as a metal substrate in manufacturing metal matrix composites. Magnesium, Titanium and other alloys were also used as a metal substrate in producing metal matrix composites.

Reinforcement Particle

Alumina (Al_2O_3) was the main reinforcement particle used with metal substrate regardless its type. Silicon carbide (SiC) was used extensively also as reinforcement particles with different metal matrices Mixture of both Al_2O_3 and SiC was also used. Other carbides such as T_iC ,or oxides such as TiO or Cr_2O_3,or compound such as Al-Cr-O were applied as a reinforcement particles.

Process and Joint Design

There are various designs for the process followed for producing matrix metal composites MMCs using friction stir process, FSP and different designs for the joint or specimen used in this process. There are mainly two types of joint design followed in producing MMCs, first using flat plate, second using two plates to form a joint to be welded. In case of flat plate there were two main methods for inserting the reinforcement; first making groove(s) all through the plate length, second making holes in the substrate in two parallel line having specific distance apart between each line or each hole wall. In case of the joint with two separate plates, groove was made at one edge of one plate and then been joined to the other plate.

Tool used for FSP was mainly from hard steel alloy or tool steel such as H-13, or WC-Co alloy, etc. The tool design including shoulder diameter, pin shape and diameter(s) and length were varied to have columnar or conical shape which either threaded or un-threaded pin/probe.

Friction stir process parameters were chosen according to the plate thickness, substrate material and tool used. Values varied in the range from 600 up to 1600 rpm for aluminum alloys, while traversing speed were in the range of 30 upto 180 mm/min.

Special case was recorded using 3000 rpm and 348 mm/min for cylindrical specimen with holes. For cupper, it was 900-1000 rpm and 40-50 mm/min. In case of Mg and Mg alloys, it was 800-1500 rpm and 20-45 mm/min.

Material of substrate was mainly from aluminum and its alloys, copper, magnesium and its alloys. Some cases dealt with mild and stainless steel, and titanium alloy.

Table 1 summarizes some of the above-mentioned details concerning joint and process design extracted from numerous researches. It includes also material of substrate and reinforcement type used in those researches.

Examinations

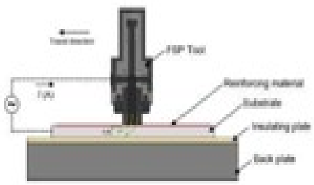
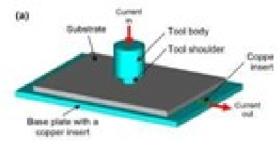
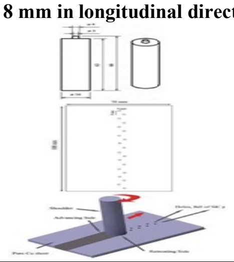
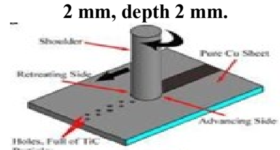
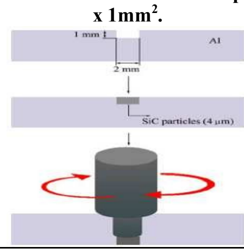
Metallurgical and mechanical characteristics of metal matrix composites were studied extensively by researchers to evaluate the effect of adding and inserting the reinforcement particles to the matrix. Examinations were carried out to study the effects of many factors on mechanical and metallurgical characteristics of the composite.

Those factors include volume percent of the reinforcement, number of passes, tool design, process parameters, such as rotational and traverse speeds. The effect of using mixture of reinforcements had been also investigated. There were some researches which dealt with the friction stir process as a cure for the previously produced composites by other methods such as powder metallurgy, laser cladding, and stir casting. The effect of reinforcement in general was the dominating factor studied. The main reinforcements used for producing the composites were Al_2O_3 and SiC or mixture of them. Other oxides or carbides of titanium have been also used as reinforcements.

Metallurgical

Optical and scanning examinations and x-ray diffraction in some cases were the main analysis techniques followed to determine the metallurgical properties. Main results of using FSP and nano/micro reinforcements showed grain refinement and even distribution of reinforcements. Grain refinement and better distribution of the reinforcement played the main role for enhancement of mechanical properties in general including wear rate decrease and hardness increase as explained below. The grain refinement was also enhanced with increasing both number of passes and tool rotational speed. The FSP cured the problem of grain growth associated with stir casting or laser cladding by breaking down the carbides resulted after those processes. In the same time eliminated the porosities existed after the process of stir casting. It was also noticed that intermetallic compounds were not existed neither in the SZ nor in the TMAZ which helped in strengthening the composite and homogenizing its properties all through.

Table 1: Various Process and Joint Designs According to Numerous Researches.

Substrate	Thickness, mm	Reinforcement	Grain Size, μm	Method	FSP Condition: Joint Design	FSP Condition: TravelSpeed, mm/min	FSP Condition: RotationalSpeed, rpm
AA5083-H111	8	Al_2O_3	45	FSP Using electric current circuit 12V with 720A  Alumina: fixed with glue in spray Layers: several upto 200 μm thick.		180	1120
		SiC & Al_2O_3	35 & 45	FSP Tool: H-13 hardened tool steel with threaded concave pin, Shoulder: dia. 19 mm,tilt: 0 & 2° Alumina: fixed with glue in spray. Layers: several upto 200 μm thick.	---	180, 224, 355	1120, 710, 355, 1800
	5	SiC	25	FSP Tool: shoulder dia. 16 mmwith conical pin, Pin: dia. 3 & 4 mm, length3 mm, Tilt: 3°, Net of holes was used forfilling the reinforcement.	Groove dimensions: No groove. Net of holes wasused with zigzag shape having 4 mm distance between holes'center in transverse direction and 8 mm in longitudinal direction. 	50	1000
	6	TiC		FSP Tool with shoulder dia. 16mm with conical pin, Pin: dia. 4 & 3 mm, length3 mm, To prevent reinforcementfrom scattering: Tool withpinless shoulder was used.	Groove dimensions: no groove. Holes were drilled along the surfaceof the sustrate. Hole dia. 2 mm, depth 2 mm.  		
A356	4	SiC	4	FSP Tool: H-13, Shoulder: columnar withthreaded pin, To prevent the sputtering of powder: aluminum tapeis used to close the gap, Passes: double pass (back side and front side).	Groove dimensions:width x depth: 2 x 1mm^2. 	127	180 0 20

SS 304	---	SiC (reactive) & Al_2O_3 (non-reactive)	2-3 & 0.5	FSP Tool: WC-Co alloy, Shoulder: columnar dia. 12mm, Pin: dia. 7 mm, length 2.4mm, Fill the groove: Plane toolwith 12 mm dia. Under lower load was used.	Groove dimensions: width x depth: 1 1 mm^2.	1000 (1000 kgf constant load)	100	40
Ti-6Al-4V	3	Hydroxyapatite, HA	nano	FSP Tool: Tungsten carbide,dia. 16 mm, Tilt: 3°, To prevent the stir zone and tool from oxidizing:argon gas shrouding system was used, Max temp: 850-900 °C.	Groove dimensions: width x depth: 2 x 1 mm^2(3 grooves separated from each other by a 2 mm distance).	16	250	43
		TiC	5.5			---	---	30

Mechanical

Mechanical properties were measured through tensile test, hardness test, wear and in limited cases fatigue and impact tests. The main results indicated increases in hardness/microhardness and consequently wear resistance, mechanical properties including yield strength, ultimate tensile strength, compression strength, bending strength, elongation, ductility and stiffness, fatigue life, toughness and impact strength. Mechanical properties were affected directly by the metallurgical characteristics as explained above. Grain refinement and uniform distribution of the reinforcement were the reasons behind the enhancement of the mechanical properties including hardness and wear resistance. Tensile properties were also improved including yield and tensile strength, in the same time elongation and ductility. In specific cases compression and bending strength were measured and showed better records after FSP. It is worth noting that all wear tests of material composites showed abrasivewear mainly.

CONCLUSIONS: Friction stir process is used recently as a surface modification method. It has the advantage of being solid-state process where the melting point of the material is not reached. The superplasticity condition of the stirred zone encouraged many researchers use it in manufacturing the metal composites where substrate is being grooved and reinforcement is put inside it, then the FSP is applied. This reinforcement is either in micro or nano size. The resulted microstructure has specific characteristics metallurgical and mechanical. The researchers studied the effect of various factors on such characteristics. Those factors were mainly concentrated on the reinforcement volume percentage, the reinforcement and substrate material, the process conditions such as number of passes, tool design and rotational and traverse speeds. The results can be summarized in the following points:

* Friction stir process showed grain refinement and improved mechanical properties such as yield and ultimate tensile strength, compressive and bending strength, toughness and fatigue life, and hardness, wear and corrosion resistance.
* Main reinforcements applied for composite manufacturing were SiC and Al_2O_3, whether the substrate materialwas aluminum or its alloys or other material such as cupper or steel.
* Hybrid reinforcement where two types were used had shown good results concerning microstructure and mechanical properties.
* Increasing number of passes or tool rotational speed showed more microstructure refinement and better mechanical properties for the composite.
* Threaded tool was the best among other tool designs like three-flute or non-threaded design where fine microstructure and higher mechanical properties were recorded.
* Friction stir process could improve the resulted microstructure of metal composites manufactured by other methods like stir casting or laser cladding where large grains have been broken into fine grains and porosities were limited after FSP application.