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# Fabrication of $TiC_p/Ti-6Al-4V$ surface composite via friction stir processing (FSP): Process optimization, particle dispersion-refinement behavior and hardening mechanism

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#### ABSTRACT

After the process optimization experiments according to a series of principles stated in the paper, the TiC particle-reinforced surface composites were successfully fabricated via friction stir processing (FSP) method on the Ti–6Al–4V alloy substrates, using a surface-reservoir reinforcement placement method for embedding TiC powder into the substrate. A relatively uniformed dispersion and an ultra-refined average size of the introduced TiC reinforcements were obtained. The reinforcement behaviors of dispersion and refinement were investigated in consideration of the plasticized Ti-matrix material-flow characteristics during FSP. Nano-sized martensite- $\alpha'$  phase and ultra-refined TiC particles were found in the TiC<sub>p</sub>/Ti–6Al–4V surface composites. The average micro-hardness of the produced surface composites was  $680Hv_{0.2}$ . The thicknesses of hardening surface layer with the hardness upon  $650Hv_{0.2}$  reached  $900 \,\mu$ m. It was further discussed on the hardening mechanisms including ceramic particle strengthening, nano-sized refinement pinning effect and matrix martensite- $\alpha'$  influence.

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#### 1. Introduction

Titanium and its alloys, owing to their attractive properties of high specific strength, outstanding corrosion resistance, good biocompatibility and sufficient stiffness, have been extensively utilized in aerospace, automobile, chemical, medical and defense industries [1]. Titanium alloys, however, suffer from limitations such as relatively low hardness and poor wear resistance that have limited their application in a number of industrial products. In this respect, titanium matrix composite (TMC) coatings reinforced with hard and stiffer ceramics for strengthening the surface properties of titanium and its alloys are accordingly fabricated to solve the problem.

Particularly, the discontinuously reinforced titanium matrix composites (DRTMCs), e.g. ceramic particle-reinforced titanium matrix composites (CPRTMCs), have generated increasing research interests in recent years, due to their excellent and isotropic mechanical properties, easy fabrication and low cost [2–5]. Several ceramic particles, such as SiC, TiN, TiC and TiB, were frequently used as reinforcements in TMCs [1–3]. For preparing TMC coatings on titanium and its alloys, various techniques are developed as the surface modification methods. The laser, electron beam, tungsten

inert gas, and plasma radiation processing methods have been demonstrated as a high capability in producing thick TMC coatings with high performance for some wear applications [6–9]. Nevertheless, these techniques are expensive and require specific conditions, e.g. high vacuum [7]. Additionally, during the metallic melting procedures, several process defects, such as cracking, porosity, anisotropic and dendritic grain coarsening, reinforcement dissolution and harmful interfacial reaction, were easy to produce. It was believed that the solid-state fabrication or processing method for producing surface composites could solve the problems during the metallic melting procedures, due to the relatively lower processing temperature. However, Very few solid-state surface modifications were reported introducing ceramic reinforced-particles into the titanium alloys or fabricating the bulky CPRTMCs in the absence of melting.

Recently, a novel solid-state surface modification technique, friction stir processing (FSP), is emerging as a very attractive method used to provide localized microstructure modifications in the surface layer of metal components or to produce a new composite surface. FSP technique was firstly proposed by Mishra et al. [10] and initially developed for Al-alloys based on the basic principles of friction stir welding (FSW), which is a relatively new solid state joining invented by The Welding Institute (TWI) of UK [11]. The FSW is considered as the most significant development in metal joining in a decade, and has been successfully used to produce joints in aluminum, magnesium, titanium, steel and other alloys [11,12]. It has been demonstrated that

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the titanium alloys can be soundly jointed via FSW without melting but with the processing temperature upper the  $\beta$ -transus of  $\alpha$ -Ti [13]. And it has also been proved that the surface engineering process of FSP can successfully form aluminum matrix composites by friction stirring ceramic particles into the surface to obtain a relatively uniform particle distribution, resulting into a developed surface performance of hardness and wear-resistance. In addition, this solid-state surface modifications technique has also been attracted in designing high power SLED [14–16]. In this respect, the FSP method, as a solid-state surface modification technique to produce surface composites, could offer a solution to the problem of that titanium and its alloys have low hardness and poor wear resistance, resulting in the limitation to application in the related industries.

From the view of FSP technical advantages for producing surface composites on titanium and its alloys, our interests in the research aiming to prepare sound surface CPRTMCs on titanium alloys are strongly motivated by multiple points of the FSP method. They can be summarized as follows:

- The prepared surface CPRTMCs with a relatively uniform particle distribution can improve the surface hardness and wear-resistance without sacrificing the bulky ductility of substrate.
- The ceramic reinforced-particles can be severely fractured and decreased in size to obtained fine reinforcements mixed and embedded in the matrix.
- The reinforcement content of surface CPRTMCs can be tailored via the control of powder placement methods and FSP process parameters.
- Unlike most other surface engineering techniques, this process can form very thick surface layers on substrate, up to centimeters in thickness, according to the designed length of FSP tool-pin.
- Due to the inherent material continuity during FSP, the novel structure of the surface composite can avoid delamination, debonding and interlayer reaction between the upper coating and beneath substrate.
- Dendrite growth, cracking and dissolution of ceramic reinforced particles can be significantly limited in the produced CPRTMCs due to relatively low process temperature and short heating period via the solid-state FSP method in the absence of titanium melting.
- Significant environmental, energy and cost benefits contributed FSP technique to be more attractive than most other surface engineering techniques.

In the present investigation, the FSP method was successfully employed to the fabrication of TiC particle (TiC<sub>p</sub>) reinforced composite on the surface of a general- purpose titanium alloy, Ti–6Al– 4V. The blind-hole form of surface reservoirs for the introduced reinforcements was utilized before FSP procedure. The FSP process optimization, TiC particle dispersion-refinement behavior, microhardness, tensile mechanical properties and hardening mechanisms of the produced TiC<sub>p</sub>/Ti–6Al–4V zone were focused.

#### 2. Experimental details

#### 2.1. Materials and processing setup

3 mm thick rolled plates of Ti–6Al–4V alloy (6.01 wt% Al, 3.84 wt% V, 0.3 wt% Fe, 0.1 wt% C and bal. Ti), with  $\alpha$ + $\beta$  duplex phases after annealing treatment, were used as substrate material. The average size of reinforcement TiC particles introduced in to substrate was ~5.5 µm.

FSP experiments were performed using a professional FSW machine and a stir-tool of WC-13 wt% Co matrix material. The

tool-shoulder diameter was 15 mm. The tool-pin was tapered from the root diameter of 6 mm to the top diameter of 4 mm. The pin length was 2.2 mm to produce a surface FSP-zone with a thickness of slightly more than 2.2 mm. Numerous blind-holes as the reservoirs for holding the TiC particles were prepared on the substrate plate surface of Ti–6Al–4V via automatically mechanical drilling methods. The holes could be arranged according to certain geometry rules as shown in Fig. 1a. The hole depth was set as 0.5~2 mm. The diameter of the holes was unified as 1 mm. Multipass FSP method using the consistent process parameters could be employed to obtain a large scaled processed zone [11,14].

In the present research, only the single-pass FSP procedure for surface composite fabrication of TiC<sub>p</sub>/Ti-6Al-4V zone was investigated. The mean process parameters during FSP included tool rotation speed (n, r/min), tool traveling speed (v, mm/min) and the tool-shoulder plunge depth (d, mm). Differently from the FSP for the Al-alloys, the d value of FSP for titanium alloy should be lower as no more than 0.05 mm to reduce the tool-shoulder wear and ensure the required friction heat-input. The tool rotating direction (RD) was set as clockwise. During the FSP procedure, a specialized Shielding Gas-circulating System was utilized to continuously introduce a shielding atmosphere of Ar<sub>2</sub> gas (99.9% purity) surrounding the rotating tool and upper the processed zone aiming to prevent the high-temperature oxidation of titanium alloy. It has been found that the Ti-6Al-4V alloy can be soundly jointed via FSW without melting but with the processing temperature, ~1100 °C, upper the  $\beta$ -transus [13]. The processing temperature would accelerate the oxidation of substrate without shielding atmosphere. Therefore, anti-oxidation prevention device was necessary.

#### 2.2. Temperature monitoring

Furthermore, an Infrared Temperature Measurement System was used to monitor the temperature changes of the substrate surface during FSP on Ti–6Al–4V alloy plate without the introduction of TiC<sub>p.</sub> contributing to investigate the relationship between the heating generation and the microstructure revolution of Ti-matrix. The Infrared Temperature Probe (IRTP) was statically



Fig. 1. Schematic diagrams of surface-reservoir reinforcement placement method (a) and FSP procedure for fabrication of TiC<sub>p</sub>/Ti-6Al-4V surface composites (b).

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