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Microstructure and mechanical properties of steel/TiC nano-composite surface layer produced by friction stir processing

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ABSTRACT

A steel/TiC nano-composite surface layer with ultra fine grains of less than 600 nm was fabricated on mild steel substrate by introduction of nano-sized TiC powder into the stir zone employing four passes of friction stir processing. TiC clusters were formed after the first pass. Sequential break-up of clusters and refinement of matrix grains were caused by subsequent FSP passes. A near uniform dispersion of nano-sized TiC particles was achieved after the fourth pass. The fabricated nano-composite layer exhibited a maximum micro hardness value of ~450HV; this is much greater than 185 and 130HV of the friction stir processed layer without introduction of TiC powder and as-received substrates, respectively. Moreover, a significant improvement in wear resistance of the nano-composite layer was observed as compared with that of the as-received substrate. The enhanced properties are attributed to the uniform dispersion of hard nano-sized TiC reinforcements in a matrix of ultra fine dynamically recrystallized grains.

1. Introduction

Friction stir processing (FSP) was developed for microstructural modification of metallic materials [1]. Surface layers can be achieved with refined grains [2,3]. In addition, by introduction of hard ceramic powder into the stir zone, composite surface layers were produced on Al [4,5], Mg [6–10], Cu [11] and Ti [12] alloys. Moreover, it was shown that nano-composite surface layer can be achieved on an Al alloy substrate by introduction of nano-sized Al_2O_3 powder using multiple FSP passes [13]; enhanced hardness and wear properties were reported [14]. Initially, FSP was restricted to lower melting temperature materials due to severe conditions encountered by the tool [15,16]. Later on, hard heat resistance tools were used for friction stir welding/processing of substrates with relatively higher melting points such as iron [17], mild steel [18], high carbon tool steel [19–21], stainless steel [22,23] and titanium [24,25]. During FSP, work piece temperature is locally raised up to ~0.6–0.7Tm (melting point temperature of the work piece) because of significant friction between the rotating tool and work piece [2]. At such high temperatures, the material is easily plasticized resulting in the formation of fine grains via dynamic recrystallization or dynamic recovery phenomena [26].

Mild steel is used as a structural material in industry and construction. However, the wear resistance of mild steel is considered to be poor in certain applications. Dispersion of hard ceramic particles in the mild steel matrix can improve strength and wear resistance compared to those of the monolithic counterparts [27].

Titanium carbide (TiC) is widely used in coatings due to its high hardness value [28,29], high strength, high rigidity [28,29], good wear resistance, low friction coefficient [28,30], high melting point and high chemical stability [30]. Due to the mentioned features, TiC is extensively used as a reinforcing phase in metal matrix composite coatings, such as steel based composites [31–33]. Iron and steels are relatively low cost material and considered as a coating matrix with potential application prospect. Ariely et al. [32] produced TiC reinforced steel coatings on the surfaces of Armco iron, AISI 1045 and 1095 steels using laser surface alloying that exhibiting relatively great hardness values. Tassin et al. [33] introduced TiC into a surface laser melt zone of AISI 316 L stainless steel, which substantially improved sliding wear resistance. Jiang and Molian [31] increased life of die-casting dies by laser surface processing with micrometer- and nanometer-sized TiC powder.

In this study, nano-sized TiC powder was used as a reinforcement material. The technique of FSP was employed for the production of steel/TiC nano-composite surface layer on a mild steel work piece. Microstructure of the fabricated composite layers was characterized. In addition, mechanical assessment of the layers was carried out using hardness and pin-on-disk wear testing.

2. Material and methods

A mild steel plate with the nominal chemical composition presented in Table 1 was used as the substrate material. Eight work pieces were prepared with a thickness, width, and length of 10, 100, and 150 mm, respectively. A groove was made in a straight line along the middle length of each work piece; its width and depth were 1 and 2 mm, respectively. In addition, a "technological hole" was drilled on

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Table 1
Nominal chemical composition of the mild steel substrate (wt.%).

Fe	C	Si	Mn	P	S	Cr	Mo	Ni	Al	Co	Cu	Nb
99.2	0.131	0.0244	0.452	0.0097	0.0078	0.0043	0.0069	0.0332	0.0446	0.0083	0.0322	0.0070

the mild steel plate in the beginning of the groove. This hole can ease the process and decrease tool wear. The groove of the four work pieces (first set) was filled with nano-sized TiC powder with an average particle size of ~70 nm before FSP experiment. The remaining work pieces (second set) were friction stir processed without introduction of TiC powder. Two tungsten carbide tools were fabricated with a shoulder and pin diameters of 16 and 5 mm for two sets of experiments (with and without TiC powder), respectively. Their pin length was 3 mm. The tool was tilted 3° from the substrate normal direction. Friction stir processing of both sets of work pieces (with and without TiC powder) was carried out using a tool rotation and substrate advancing speeds of 1120 rpm and 31.5 mm/min, respectively. Process parameters such as tool rotation and substrate advancing speeds, tool material and its dimensions, and groove dimensions were selected according to a detailed experimental work which is beyond the scope of the present paper [34]. Each set of work pieces was subjected to numbers of FSP passes from one to four. Fig. 1 shows the schematic of FSP experiment.

Microstructure of the fabricated surface layers was investigated using a scanning electron microscope linked with an energy dispersive spectrometer. Mean matrix grain size was measured by ASTM linear intercept method [35]. Volume fraction of TiC particulates was obtained using Clemex image processing software [36]. Micro hardness values of the fabricated surface layers were measured by a Vickers indenter under a load of 200 g for 15 s.

Wear test was conducted employing a pin-on-disk tribometer. Most requirements of ASTM standard G99-04 were followed. Nevertheless, several modifications were introduced, mainly regarding the pin shape (a flat shape disk was used instead of spherical one, i.e. pin and disk were in contact on a plane). Cylindrical pin specimens with a diameter of 5 mm were made from the as-received mild steel and fabricated surface layers. The counterpart disks were made of 100Cr6 (AISI 52100) steel with a hardness value of about 60 HRC. Sliding track dimension on the disk surface was 40 mm. Before wear test, each pin specimen was ground down to 1000 grit abrasive paper. Wear tests were performed under dry sliding condition using a sliding velocity of 0.5 m/s for 2000 s. A constant load of 15.5 N was applied. Both pin specimens were cleaned in acetone and weighed to an accuracy of ± 0.1 mg prior to testing and at different sliding distances during the test. Friction coefficient between the pin specimen and disk

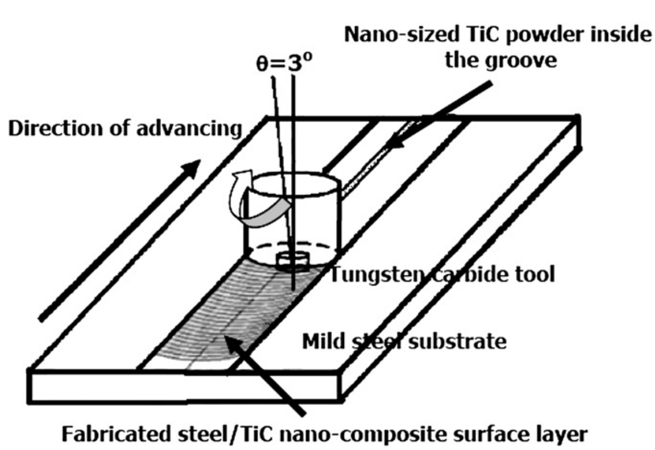

Fig. 1. Schematic of friction stir processing for the fabrication of steel/TiC nano-composite surface layer.

was determined by measuring frictional force using a stress sensor. Worn-out surfaces were studied by scanning electron microscopy and X-ray diffractometry.

3. Results and discussion

3.1. Friction stir processing

Top view of a typical fabricated surface layer was shown in Fig. 2. Both tracks exhibited sound appearance, uniform chevron marks and smooth quality. Almost no prominences, void, cracks or depression were found. Further macroscopic and microscopic studies of track cross sections at three places confirmed their defect-free status.

Tool wear was found to be a serious problem; after four number of FSP passes (i.e., a total processing length of 400 mm), pin length and diameter of the tool used in the first set of experiment (without introduction of TiC powder) were reduced about 20% of its initial dimensions. However, this was found to be slightly greater in the case of the second pin tool (used with introduction of TiC powder). It can be deduced that TiC-reinforced layer provided harder material.

3.2. Dispersion of TiC particles

Cross section of the fabricated layer after the first FSP pass exhibits material flow pattern (Fig. 3a); this assists mixing of TiC particles with the plasticized material. Employing higher magnification on un-etched cross section, small regions with bright contrast were revealed; energy dispersive spectroscopy analyses on these regions showed that they are rich in titanium (e.g., Fig. 3b). Thus, these areas are titanium carbide. However, the initial nano-sized particles made clusters of various sizes within the stir zone (Fig. 4a). The agglomerative nature of fine particles due to their high cohesive energy leads to an increase in the total surface area and increases their tendency to clump together forming agglomerates and clusters [37,38]. Break-up of these clusters to smaller ones occurred after the second FSP pass (Fig. 4b). Material flow was proposed to be complex in FSP [39,40]. The complexity arises from material motion at various directions; these are circumventing motion of surface material around the tool shoulder, torsional motion due to rotational motion of surface material within the interaction layer under the tool shoulder, and vortex motion associated with the flow of thickness material due to the action of the tool pin [40]. Such material motions are responsible for the break-up of TiC clusters and their dispersion in the mild steel matrix (Fig. 4b). A nearly uniform dispersion of nano-sized TiC reinforcements was achieved after the fourth FSP pass (Fig. 4c). Thus, dispersion and size of TiC reinforcements were found to be a function of number of FSP passes.

3.3. The matrix microstructure of the fabricated layers

Optical metallography of the as-received mild steel substrate exhibited dominant grainy microstructure with a mean grain size of ~15 μm (Fig. 5a). In addition, minor areas with a dark contrast were observed. Scanning electron microscopy, using secondary electron imaging revealed the existence of minor lamellar features in a dominant grainy structure (Fig. 5b); these are consistent with a mild steel alloy [41]; the grainy and lamellar structures are ferrite and pearlite, respectively.