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## Tribology characteristics of ex-situ and in-situ tungsten carbide particles reinforced iron matrix composites produced by spark plasma sintering

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### ABSTRACT

In this paper, ex-situ (adding the particles reinforcement phase into the matrix materials directly) and insitu (the particles were synthesized directly from elemental powders of W and C during the fabrication) tungsten carbide particle reinforced iron matrix (WC/Fe) composites were well fabricated by spark plasma sintering (SPS) with the particle volume fraction of approximately 30%. The main phases were ferrite, WC, W<sub>2</sub>C, Fe<sub>3</sub>W<sub>3</sub>C and pearlite. The content of Fe<sub>3</sub>W<sub>3</sub>C in ex-situ WC/Fe composites was much higher than that in in-situ WC/Fe composites, and some of which spread throughout particles in ex-situ WC/Fe composites. The homogenous distribution of WC particles within the iron matrix was obtained with strong bonding to the matrix. The mean WC grain size was about 24 µm and 13 µm for ex-situ and in-situ WC/Fe composites, respectively. Compared with the traditional martensitic wear-resistant steels, these two type composites presented the more excellent wear resistance which was enhanced at least six times. Moreover, due to the better particles size and interfacial microstructure, the in-situ composite had the lower specific wear rate ( $2.5 \times 10^{-5}$  mm<sup>3</sup>/Nm) which was about 65% to that of the ex-situ composite  $(3.8 \times 10^{-5} \text{ mm}^3/\text{Nm})$ . The dominant wear mechanism for the in-situ and ex-situ WC/Fe composites was a combination of abrasive wear and oxidation wear, which was different from the micro-ploughing mechanism of the martensitic wear-resistant steel. For the ex-situ composites, coarse-grained WC and higher content of brittle phase Fe<sub>3</sub>W<sub>3</sub>C increased the wear rate and reduced the wear-resistance.

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#### 1. Introduction

Ceramic particles reinforced metal matrix composites (MMCs) not only possess high strength, good plasticity and impact toughness of metal material, but also possess high hardness, high wear resistance of ceramic materials, overcoming the problem of the contradiction of traditional wear-resisting materials between resistance and toughness [1–3]. MMCs have been used in applications requiring high wear-resistance and good toughness, such as tool steels, machine elements and rock drilling, dies as well as high temperature oxidation resistance components.

Owing to extremely high hardness, wear resistance and good wettability between WC and molten metal [4], WC particles find applications in manufacturing cemented components employed in various wear applications, such as WC-Co [5], WC-Fe/steel [6], WC-Ni [7]. Compared with other WC particle reinforced MMCs, iron or steel matrix composites are desirable materials due to low cost and good mechanical properties [8]. Several techniques have been used to prepare WC-reinforced Fe-based composites [9,10]. For instance, Li et al. [9] prepared WC particle reinforced Fe-based surface composites on gray cast iron substrates using a vacuum evaporative pattern casting infiltration process, which gave different volume fractions of WC particles. The results of abrasive wear tests showed that the wear resistance of the composites reached the maximum when the volume fraction of WC was 27%. There are also many other techniques, such as multi-track overlapping laser induction





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hybrid rapid cladding [11,12], vacuum infiltration casting technique [13], supersonic laser deposition [14], vacuum evaporative pattern casting [15], hot isostatic pressing [16], casting method [17], plasma melt injection [18], laser melting deposition [19,20], the gas tungsten arc welding process [21]. These techniques are based on adding the WC particle reinforcement phase into the matrix materials directly. The in-situ processing technique also shows many advantages for producing particulate composites, such as fine grain size and uniform distribution of reinforcements, free of contamination and strong interface between reinforcements and matrix. Zhong et al. [22] designed iron matrix reinforced with a columnshaped WC-Fe composite which was fabricated by a novel in situ method. Experimental results showed that WC particulates improved the wear resistance of the composite, and the highest wear resistance was ~96 times higher than that of gray cast iron under a load of 20 N with SiC abrasive particles. Additionally, several other techniques such as in-situ formed WC particles on gray cast iron substrate by centrifugal casting [23], WC reinforced iron-based surface composites by solid-phase diffusion [24], in-situ synthesized WC particles through the reaction between tungsten wires and molten gray cast iron [25], have been used to fabricate WC-reinforced Fe-based composites.

Ex-situ and in-situ composites have their own advantages. Few people have compared microstructure and properties between these two kinds of composites. In this paper, samples with iron matrix reinforced by WC were designed and fabricated by SPS.

#### 2. Experimental procedure

The particulate composites were prepared by SPS from a mixture of commercially pure WC, Fe, C, W and Cu (Cu can reduce sintering temperature and promote sintering densification [26]) powders with particle size of 25 µm. Table 1 shows the compositions of different powder mixture for ex-situ and in-situ WC/Fe composites. The powders of in-situ WC/Fe particulate composites containing 25 vol% WC were mixed. The two kinds of powders were blended homogeneously using ball milling (QM-3SP04 planetary ball mill from Nanjing University instrument plant, China). After ball milling, a spark plasma sintering furnace (SPS-1050, Japan) was used to produce the composites. The sintering samples with dimensions of 30 mm in diameter and approximately 10 mm in height were sintered under a pressure of 50 MPa stayed warm for 5 min. Sintering temperature were shown in Table 1.

The Archimedes method was used to determine the density of the sintered materials. The microstructures, elemental analysis and phase compositions were investigated by a scanning electron microscope-secondary electrons (SEM-SE) equipped with an energy dispersive X-ray spectrometer (EDS) and X-ray diffractometer (XRD). The Brinell hardness was measured by a hardness tester at a load of 1470 N. The samples for compressive strength measurement were cut from the sintered material with dimension of  $\Phi$  5 × 10 mm. The fracture toughness for the composites was measured using the three point bending test (3 PB). The dimensions of samples which contained pre-existing cracks (cut the transverse of samples into a certain depth of incision by molybdenum wire) are shown in Fig. 1.



Fig. 1. The three point bending specimen used for fracture toughness measurement.

The abrasive wear tests were carried out on an MMU-10G pinon-disc wear tester (from Jinan Tribology Testing Technology Co., Ltd, China). Two kinds of particulate composites, cut into cylinder specimens with the size of  $\Phi 4 \times 15$  mm (pin), were applied a load of 80 N and a grade 240 mesh alumina abrasive paper used as counterface at room temperature, relative sliding speed of 120 rpm/ min and a sliding distance of ~4752 m. The worn alumina abrasive paper was replaced for every sliding time of 2 min to ensure fresh supply of abrasive particles to the pin specimens. The weight loss of the pin specimens were measured for every sliding time of 10min by an electronic balance with an accuracy of 0.0001 g.

Specific wear rate,  $\beta$ , was used to evaluate the abrasive-wear properties of the WC/Fe composites. This value can be obtained by first computing the abrasive volume, which is defined as [27]:

$$\Delta V = \frac{\Delta M}{\rho} \tag{1}$$

where  $\Delta V$  is the abrasive volume (mm<sup>3</sup>),  $\Delta M$  is the weight loss of composites (g), and  $\rho$  is the density of the test material (g/cm<sup>3</sup>). The specific wear rate can be expressed as:

$$\beta = \frac{\Delta V}{F_{n} \cdot d} \tag{2}$$

where  $F_n$  is the normal load applied to the disc (N), d is the sliding distance (mm).

#### 3. Results and discussion

#### 3.1. Densification and microstructure of the composites

The two kinds of composites sintered by SPS method have been fabricated. The densities of the composites in Table 1 were apparently greater than theoretical values. This was likely due to some potential volatilization of the matrix during sintering, which would slightly alter the theoretical density, and was in agreement with previous studies on TiC-316L stainless steel cermets [28,29].

The XRD pattern of the composites is shown in Fig. 2. It can be

Table 1

 $Compositions \ of \ different \ powder \ mixtures \ (wt\%) \ and \ sintering \ temperature \ used \ in \ SPS \ experiments.$ 

Material	Compositions					Sintering temperature
	W	С	WC	Cu	Fe	
Ex-situ WC/Fe composite In-situ WC/Fe composite	_ 37.6 ± 0.01	$1 \pm 0.01$ $3.4 \pm 0.01$	40 ± 0.01 -	$1.5 \pm 0.01$ $1.5 \pm 0.01$	Bal. Bal.	1080 ± 5 980 ± 5

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