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Effect of reinforcement content on the density, mechanical and tribological properties of $\text{Ti}_3\text{SiC}_2/\text{Al}_2\text{O}_3$ hybrid reinforced copper-matrix pantograph slide

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Abstract: As an important part of the pantograph and catenary system, the pantograph slide receives increasing attention from investigators, mainly focused on the interfacial bonding and characteristics, electrical conductivity, and friction and wear characteristics under current-carrying circumstances. In the article, hybrid reinforcements of titanium silicon carbide (Ti_3SiC_2) and nano-alumina (nano- Al_2O_3) particles pretreated by electroless plating process were firstly blended with copper powders and then prepressed and vacuum hot pressed to fabricate the hybrid reinforced copper-matrix pantographs (HRCs). The relative density, mechanical and electrical properties of $\text{Ti}_3\text{SiC}_2/\text{Al}_2\text{O}_3$ HRCs were calculated and measured. The tribological performance of the HRCs reinforced with 1 wt%–5 wt% of alumina particles and 20 wt%–40 wt% of Ti_3SiC_2 reinforcements was also analyzed.

Keywords: hybrid reinforced copper-matrix pantograph (HRC); pantograph and catenary system (PCS); pantograph slide; titanium silicon carbide (Ti_3SiC_2); tribological performance.

1 Introduction

The high-speed rail industry has become an eye-catching subject in China's manufacturing industry, a part and parcel of its modern railway undertaking and a first choice in public transport. As an important component of high-speed rail, the pantograph and catenary system (PCS) is not only the most common running modality in today's high-speed railway but also the most effective online running system [1–3]. Usually, the PCS is composed of a pantograph and catenary operated in the energized state, and their stability and reliability are very crucial for the PCS. In order to prolong the service life of the catenary which has a high replacement cost, the pantographs under electrical wear condition should possess a combination of good mechanical strength, electrical and thermal conductivity, self-lubricating characteristics, etc. The copper-matrix pantograph is the most widely used material in operation, but the friction between the pantograph and catenary is very serious, while the addition of reinforcements into the pantograph is one of the most common measures undertaken. Titanium silicon carbide (Ti_3SiC_2), as one of the newly emerging MAX ternary compounds ($\text{M}_{n+1}\text{AX}_n$, where M=early transition metal, A=group IIIA or IVA element, and X=C and/or N, $n=1-3$), possesses the excellent performance of both metallic and ceramic characteristics, which has easy machining properties and good thermoconductivity, excellent oxidation resistance and thermal stability at high temperatures [4, 5], and its electrical conductivity is roughly two orders of magnitude higher than that of graphite [6–8]. The Ti_3SiC_2 nano-laminates can easily shear over other materials and form a film on the Ti_3SiC_2 tribo-surface owing to the hexagonal close packing structure of the MAX phases [9, 10]. The wear and friction property of dense Ti_3SiC_2 ceramics have been well explored, and it has been reported for Ti_3SiC_2 compound wearing against steel and Al_2O_3 with a steady state friction coefficient value of 0.4–0.5 [11], against Ti_3SiC_2 and diamond with the friction coefficients of 1.16–1.43 and below 0.1, respectively [12],

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and against Si_3N_4 with the friction coefficient of 0.31–0.48 and wear rates ($0.34\text{--}2.1 \times 10^{-5} \text{ mm}^3 \text{ N}^{-1} \text{ m}^{-1}$) from 25 to 800°C [13], etc. The tribological behavior of Ti_3SiC_2 -based material was also studied, and the wear mechanism was concluded as the fracture and delamination of Ti_3SiC_2 -based materials followed by adhesive wear of steel sliders [14]. All studies mentioned above illustrate the good self-lubricating performance of the Ti_3SiC_2 compound.

On the other hand, Ti_3SiC_2 does not have good wear resistance under higher applied loads which can generate delamination at the basal plane, owing to the weak Si layer in its ternary layered structure [15]. Thus, researchers attempt to improve its poor wear resistance by adding some enhanced phases (e.g. TiC , SiC , Al_2O_3 and TiB_2) to the Ti_3SiC_2 matrix. Among these reinforcements, nano-sized alumina particles (nano- Al_2O_3) are potential materials for application in the pantograph as a function of its prominent performance because it has excellent wear resistance and antioxidant properties, which can provide adequate support for the pantographs and lower the wear loss of the catenary to a minimum. What is more, incorporating nano-materials into the matrix can usually improve the mechanical properties, emerging as a promising research field of nano-composite. The effect of plastic deformation on the Ti_3SiC_2 -toughened Al_2O_3 was analyzed, and the interactions between crack deflection and plastic deformation have been discussed in the literature [16].

Moreover, the wettability between metals and ceramics plays an important role in the fabrication of metal-ceramic composites and joints. The wetting behavior of the $\text{Cu}/\text{Ti}_3\text{SiC}_2$ system by the sessile drop technique under a vacuum atmosphere was investigated, and the results showed that two distinct reaction layers consisting of different contents of Cu , TiC_x , Ti_3SiC_2 and Cu_xSi_y intermetallic compounds were formed at the interface of Cu and Ti_3SiC_2 [17]. To prevent the formation of unfavorable intermetallic compounds along the interface and improve the contact behavior between copper matrix and Ti_3SiC_2 particles, an electroless plating process is usually introduced for preparing the composites, and the formation of a new compound layer at the interface is a more efficient way to improve the wetting [18, 19].

In this work, the hybrid pantographs reinforced with 20%–40% of Ti_3SiC_2 particulates and 1%–5% of nano- Al_2O_3 particles were fabricated through a vacuum hot pressing procedure, and the effect of the reinforcements pretreated by electroless plating process on the density, mechanical, and tribological properties of hybrid pantographs was also investigated. To increase the wettability between the copper matrix and the reinforcements, the surface of the reinforcements were coated with copper atoms through an

electroless plating process in order to strengthen the interfacial structure and organization of the HRCs, which will improve their overall performances.

2 Materials and methods

2.1 Experimental procedure

In the experiment, as-received copper particles (average particle size $20 \mu\text{m}$, 99.7% in purity, Changzhou Yalang Chemical Co., Ltd., China, as shown in Figure 1), as-received Ti_3SiC_2 particles (average particle size $30 \mu\text{m}$, 99.2% in purity, Hebei Jinghe Titanium Silicon Carbon Materials Co., Ltd., China, exhibited in Figure 2) and nano-alumina (nano- Al_2O_3) particles (average particle size 20 nm , 99.99% in purity, Guangzhou GBS High-Tech and Industry Co., Ltd., China) after copper plating process (presented in Figure 3) were used as starting materials. The Ti_3SiC_2 particulates were treated with an electroless copper plating process, which was conducted at 70°C in a bath with the following composition: 10 g/l of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ (98%, AR, Wuxi Yasheng Chemical Co., Ltd, China), 31 g/l of ethylenediaminetetraacetic acid (EDTA)-2Na (99%, AR, Zhejiang Brandt Chemical Co., Ltd, China), 10 g/l of NaOH (99.9%, AR, Kunshan Southeast Chemical Co., Ltd, China), 1 ppm of 2,2'-dipyridyl (99%, AR, Shanghai J&L Biological Co., Ltd, China) and 15 ml/l of HCHO (99%, AR, Dongguan Qiaoke Chemical Co., Ltd, China). The pH of the bath was adjusted to 13 by the addition of NaOH. The detailed composition of the pantograph slide with a different fraction of Ti_3SiC_2 , copper and nano- Al_2O_3 is shown in Table 1, and

Figure 1: Scanning electron microscopy micrograph of as-received copper powders.

Figure 2: SEM presentation of as-received Ti_3SiC_2 particles under (A) low and (B) high magnification.

Table 1: Composition of pantograph slide with different fraction of Ti_3SiC_2 , copper and nano- Al_2O_3 .

Al_2O_3 wt%	Ti_3SiC_2 wt%	Cu	Experiment code
1%	20%	bal.	$\text{A}_1\text{B}_2\text{C}$
1%	30%	bal.	$\text{A}_1\text{B}_3\text{C}$
1%	40%	bal.	$\text{A}_1\text{B}_4\text{C}$
3%	20%	bal.	$\text{A}_3\text{B}_2\text{C}$
3%	30%	bal.	$\text{A}_3\text{B}_3\text{C}$
3%	40%	bal.	$\text{A}_3\text{B}_4\text{C}$
5%	20%	bal.	$\text{A}_5\text{B}_2\text{C}$
5%	30%	bal.	$\text{A}_5\text{B}_3\text{C}$
5%	40%	bal.	$\text{A}_5\text{B}_4\text{C}$

all specimens were blended for 10 h. For the purpose of protecting the coating of plating particles in the process of mixing, the rotation speed should be lower than 200 rpm, and make sure the mixing procedure is sufficient. After the blending procedure, the particles mixed were compressed in the steel mold under the pressure of 300 MPa to prepare the preformed compact and then hot pressed (sintering apparatus: RRY vacuum hot pressed sintering furnace, Shenzhen Shidimu Technology Co., Ltd., China) with the following parameters: $15^\circ\text{C}/\text{min}$ of heat rate, 850°C of hot pressing temperature, 30 MPa of hot pressure and 2 h of packing time.

2.2 Characterization

After the hot pressing procedure, the mechanical and electrical properties of hybrid reinforced copper-matrix pantographs (HRCs) were analyzed. The relative densities of the HRCs were measured in accordance with Archimedes'

Figure 3: Transmission electron microscopy micrograph of nano-alumina particles after electroless copper plating.

principle. Hardness measurements were carried out on a Brinell hardness testing machine (THB-3000MD-F), using an indenter ball with 2.5 mm diameter at a load of 25 kg, and the mean values of at least five measurements conducted on the different areas of each sample were obtained. Electrical conductivity of all specimens was tested by a four-point contact method.

Testing of friction and wear behavior of $\text{Ti}_3\text{SiC}_2/\text{Al}_2\text{O}_3$ HRCs was carried out on the MM-200 high-speed current-carrying wear machine at room temperature with the humidity of 25%, which was controlled with a stepless current regulator ranging from 1 A to 400 A. The investigation of friction and wear behavior was carried out under the loading of 8 N at the speed of 200 rpm, with the duration of 40 min. The coefficient of friction was

automatically collected by a wear tester, while wear loss and the characteristics of the pantographs were detected by a Micro-XAMTM non-contact 3D surface topography instrument, and surface morphology of the pantographs were performed with an S-3500N scanning electron microscope (SEM) equipped with an energy dispersive spectrometer (EDS).

3 Results and discussion

3.1 Relative density and microstructure

Based on the relationship between theoretical and measured density, relative density of the HRCs was calculated on the basis of Archimedes' principle, and the testing results are presented in Figure 4. As shown in the figure, the relative density of hybrid reinforced pantographs decrease with the increase of the fraction of titanium silicon carbide particulates, and the relative density of the HRCs as a whole exhibits a declining trend. The relative density of pantograph composites reinforced with 1 wt% of alumina particles is much higher than that reinforced with 5 wt% of alumina particles. When the fraction of titanium silicon carbide particles within the HRCs increases from 20 wt% to 40 wt%, the densities of pantograph composites almost decrease significantly.

Generally speaking, relative density is an important criterion for powder metallurgy products, which not only reflects the quality of hot pressing parts but also reveals the correlations of different components indirectly. From the results mentioned above, it can be concluded that the hybrid reinforcements in the composites are inclined to

Figure 4: Relative density of $\text{Ti}_3\text{SiC}_2/\text{Al}_2\text{O}_3$ HRCs.

Figure 5: Schematic diagram of the formation of pore in the pantograph.

aggregate with the increase of titanium silicon carbon particles and form internal voids, and the gas trapped inside is very hard to discharge from the material, as a result affecting the density of the pantograph slide. The schematic diagram of pore formation in the pantograph is shown in Figure 5. On the other hand, aggregation of Ti_3SiC_2 particles hinder the thermo-forming fusion and hot pressing process, as a result increasing the porosity of the pantograph slide and decreasing the density of the composites.

Figure 6 exhibits the interfacial status of a hybrid reinforced pantograph slide. As can be seen, the ternary Ti_3SiC_2 particles pretreated by the electroless copper plating process are well meshed with the copper matrix, and the coating layer plays the transition role in the composites, which is vital for the properties of pantograph slides, because the good interfacial adhesion can effectively improve the mechanical and electrical performance of the materials and lower the effect of electron scattering [20].

3.2 Microhardness test

The testing results of microhardness of the HRCs reinforced with different fractions of nano-alumina and titanium silicon carbide particles are exhibited in Figure 7. As

Figure 6: Interfacial bonding of HRCP slide.

Figure 7: Microhardness of $\text{Ti}_3\text{SiC}_2/\text{Al}_2\text{O}_3$ HRCs.

illustrated in the figure, the microhardness of the pantograph composite is much higher after the addition of nano- Al_2O_3 and Ti_3SiC_2 reinforcements when compared with that of the pure carbon pantograph slide (72HB). Owing to the augment of the fraction of Ti_3SiC_2 particles ranging from 20% to 30%, the microhardness of the composites on average is effectively strengthened. According to the basis of the Orowan strengthening theory [21], a large number of nano- Al_2O_3 and Ti_3SiC_2 particles are dispersed in the copper matrix with the increase of the fraction of reinforcements, and the reinforcements acting as load bearing points are in contact with the friction pair. The intensive resistance of the HRCP slide decreases the tendency of plastic deformation, which will increase the microhardness of the pantograph composites. If the fraction of the Ti_3SiC_2 particles is increased continually, microhardness of the pantographs declines slightly; that is to say that the reinforcements at high levels reduce the hardness of the composites. The reason lies in that the increase of the reinforcements content could result in the agglomeration of reinforcing particles, and the voids inside the pantograph composite have an obvious tendency to increase with the Ti_3SiC_2 content ranging from 30 wt% to 40 wt%, which leads to the decreasing density of the composite material, as a result reducing the capability of resisting external stress and lowering the microhardness of the pantographs.

On the other hand, the hardness of pantograph slide shows an upward trend with the increase of the nano-alumina fraction. In the light of the testing results, strengthening mechanisms for the pantographs can be explained as that the applied load transforms from the matrix to reinforcements, while the reinforcements acting as load bearing play an important role in the composite; as a result, the load transfer mechanism is very important for the

hybrid reinforced composites. According to the Hall-Petch equation (1), when the size of grain (d) in the composites is lessened, the yield strength (σ_y) is always much higher. The pantograph slide exhibits the greater occurrence of the pinning effect with higher nano-alumina fraction, so the hardness of the composites is inclined to increase.

$$\sigma_y = \sigma_0 + k_y / \sqrt{d} \quad (1)$$

3.3 Electrical resistance measurement

The electrical conductivity of the pantographs with different fraction of nano- Al_2O_3 and Ti_3SiC_2 particles is exhibited in Figure 8, and the electrical conductivity of a commercial pure carbon pantograph slide was also analyzed and taken for comparison. In the experiment, when the fraction of nano-alumina particles increased from 1 wt% to 3 wt% and 5 wt%, the electrical resistivities of the pantograph slide were $0.12 \mu\Omega\cdot\text{m}$, $0.17 \mu\Omega\cdot\text{m}$ and $0.22 \mu\Omega\cdot\text{m}$, respectively. It can be analyzed that, because hybrid reinforcements of nano- Al_2O_3 and Ti_3SiC_2 particles are surface-coated by electroless copper plating process, a better connection between the reinforcements and copper matrix is created, which weakens the function of electron scattering, so the discrepancy of electrical resistivity is not obvious. Besides, with the increase of the fraction of reinforcements, the internal interfaces in the pantograph composites grow exponentially, which abates the effect of electron scattering, increases the electrical conductivity of the pantograph slide and reduces the electrical resistivity.

Usually, electrical resistance arises essentially from static lattice distortion and thermal vibration caused by defects or impurities generated by dynamic distortion,

Figure 8: Electrical resistivity of $\text{Ti}_3\text{SiC}_2/\text{Al}_2\text{O}_3$ HRC.

while electrical scattering is the reason why the materials have electrical resistance. Different phases in the composite have a scattering function for conductive electrons, which results in an increase in electrical resistivity of the composites and decrease in their conductivity. In addition, solid solution strengthening phases in the matrix can also cause distortion of lattice, so that the scattering effect is enhanced. In order to ensure that the pantographs have good conductivity, hybrid reinforcements should play an important role in the pantograph slide and enhance the synergistic effect between the nano- Al_2O_3 and Ti_3SiC_2 particles. On the one hand, the quantity of free electrons in the pantograph slide is reduced so that electrical resistivity is enhanced with the increase of the fraction of nano-alumina and titanium silicon carbide particles; on the other hand, while increasing the fraction of hybrid reinforcements, the augmentation of interface geometry and enhanced ability of electron scattering also results in the decreased conductivity of the HRCs.

3.4 Friction and wear performance

Generally speaking, wear can be divided into three stages [22]: running-in stage, normal wear stage and rapid wear stage. The best running stage for the specimen is under the normal wear stage, because the surface of the sample is integrated, resulting in slow wear and tear at this stage. The primary goal is to take measures to prolong the lifetime of the normal wear and tear stage, which will ensure the stable operation of an electric locomotive.

Table 2 exhibits the friction coefficient and wear loss of pantographs with different fractions of reinforcements. For comparison, a pure carbon pantograph was introduced here in order to investigate the discrepancy between a pure carbon pantograph and a copper-matrix pantograph, and their friction coefficient and wear rate

Figure 9: The relationship between friction coefficient of pantograph and wear time.

were also tested. As can be seen in Figure 9, friction coefficient of the pantographs gradually reduce with the duration of time, and the friction coefficient of pure carbon is lower than that of copper-matrix pantograph slide. Because the bonding strength of the C-C bond is very low, it makes the pantographs wear out easily, resulting in the attachment to the contact surface of a friction pair in the course of friction. As a result, double-sided lubrication comes into being, which reduces the friction coefficient of the pure carbon pantograph slide; that is the reason that the friction coefficient of pure carbon slide is so low. But for the HRCs, the nano- Al_2O_3 and Ti_3SiC_2 reinforcements play an important role in the process of friction and wear, and the frictions coefficient of the HRCs is much higher than that of the pure carbon pantograph slide.

Figure 10: Wear loss of hybrid reinforced copper-matrix composite pantograph under carrier wear.

Table 2: Friction coefficient and wear loss of the pantographs.

Experiment code	Friction coefficient	Wear loss (μm^3)
A_1B_2C	0.415	4.23×10^6
A_1B_3C	0.401	3.57×10^6
A_1B_4C	0.387	3.14×10^6
A_3B_2C	0.424	4.06×10^6
A_3B_3C	0.408	3.31×10^6
A_3B_4C	0.392	3.01×10^6
A_5B_2C	0.452	3.92×10^6
A_5B_3C	0.434	3.24×10^6
A_5B_4C	0.423	2.89×10^6
Pure carbon	0.317	8.48×10^6

Figure 10 exhibits the wear volume loss of the pantographs reinforced with nano-alumina particles, ranging from 1 wt% to 3 wt% and to 5 wt%, respectively. It can be concluded that wear loss of the pantograph reinforced with 1 wt% of nano-alumina particles is much higher than that reinforced with 3 wt% or 5 wt% of reinforcements, and the wear loss of all tested pantograph slides shows a downward tendency with the increase of titanium silicon carbon particles. Titanium silicon carbide particles in the composite have special ternary structure, which plays a very important role in the lubrication; while nano-alumina particles primarily assume the role of load bearing, alumina particles can reduce the wear of the pantographs and make the wearing surface of the pantograph more flattened [23]. As a result, the higher the nano-alumina content, the lower the wear rate of the pantographs.

Complicated working circumstances for the electric locomotive determine the complexity of wear mode; as a result, pantograph slides are inclined to take the function of mechanical, adhesive and electrical wear under normal

circumstances. Figure 11 exhibits the wear surface morphology of the pantograph slides under current-carrying conditions. Because the hardness of the copper-matrix pantograph is much higher than the pure carbon pantograph slide, the reinforcements falling off from the pantographs will remain on the surface of the pantograph slide and the friction pair in the process of friction and wear, which will form three-body friction and cause abrasive wear and a certain depth of furrow. On the other hand, ablation products are commonly presented on the surface of the composites, because the arc erosion phenomenon is so obvious for the pantograph which generates high temperature, melts the materials with low melting point in the pantograph, and separates the reinforcements from the pantograph slide in the process of friction and wear. If hybrid reinforcements peel off from the composites, they will lose their supposed to function, while the pure carbon pantograph slide does not have this characteristic because of its monophase carbon.

It is believed that the ablation products were formed on the surface of the specimens in the process of friction

Figure 11: SEM micrograph of HRCP slide under current-carrying wear. (A) Pure carbon; (B) A_1B_2C ; (C) A_3B_3C ; (D) A_5B_4C .

Figure 12: EDS mappings of the pantograph slide after wear test.

and wear, because the copper matrix around the reinforcements has been dissolved, which results in the dispersion of hybrid reinforcements on the surface of the pantographs. Therefore, the reinforcements rub against the copper-matrix pantograph slide and friction pair, and accumulate on the surface of the pantograph. The maximum amount of wear loss usually happens at the position such as offline, contact surface and defects, etc., and the defects in the pantograph slide are the most seriously worn position. It can be easily seen from Figure 11C that the traces of ablation products are presented on the slide surface, accompanied by the presence of adhesive wear. Elemental analysis of ablation products marked A in Figure 11C is shown in Figure 12, and the testing results have showed that it contains Cu, C, O, Ti, Si, Al and other elements.

4 Conclusions

The following are the conclusions of this study:

1. Hybrid reinforced copper-matrix pantographs, composed of 1 wt%–5 wt% of the nano- Al_2O_3 particles and 20 wt%–40 wt% of Ti_3SiC_2 ternary compound, were successfully fabricated with the vacuum hot pressing procedure.
2. The microhardness of the pantograph composites were firstly strengthened, then decreased as a function of the content of Ti_3SiC_2 particles ranging from 20% to 40%, and increased with the nano-alumina increasing from 1% to 5%.
3. The electrical resistivity of HRCs was obviously lower than that of pure carbon. For the reinforcements proceeded with electroless copper plating process, the effect of electron scattering was alleviated

when compared with the pantographs with a higher fraction of reinforcements.

4. The tribological performance of HRCs was presented to be considerably better than that of a pure carbon plate, which was enhanced with the increase of the reinforcements content. The reinforcements acting as load bearing elements can absorb the applied energy from the friction pair and prevent the materials from peeling off the surface of the pantograph slide.
5. For all the pantographs prepared, the specimen consisting of 30 wt% of Ti_3SiC_2 and 3 wt% of nano- Al_2O_3 particles is the most prominent material for its overall properties.

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