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Effect of sintering temperature on the microstructure and performance of a ceramic coating obtained by the slurry method

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ABSTRACT

In this paper, SiO₂, Cr₂O₃, Al₂O₃, and MgO were used as ceramic aggregates, and a small amount of Al powder was added. A ceramic coating was prepared on a Q235 steel substrate. The effect of the sintering temperature on the coating microstructure, phase structure and wear resistance was studied by Scanning Electron Microscope (SEM), X-ray Diffraction (XRD) and friction and wear testing. The results show that the tensile strength of the ceramic coating is increased after sintering, the structure becomes dense, and the size of coated micropores is increased to release the internal tensile stress. With the increase of the sintering temperature and tensile stress, the micropores begin to release the excess tensile stress in the form of crack initiation and expansion. The mineralization of MgO, Cr_2O_3 , nMgO and mSiO₂ phases can be achieved by sintering the coating at 200 °C; the oxygen in the atmosphere migrates along the micropores in the coating to react with Fe in the steel substrate, forming FeO, and the resulting FeO reacts with the SiO₂ in the coating to free SiO₄ phase. The coating has the best wear resistance after being sintered at 400 °C, and the abrasion resistance of the sample is 6.7 times higher than that of the sample dried at room temperature.

1. Introduction

The advantages of ceramic materials include their high temperature stability, high melting point, good wear resistance and excellent corrosion resistance. The preparation of a layer of ceramic coating on a metal surface can be used to obtain both the strength and toughness of the metal and ceramics, high temperature resistance, abrasion resistance, corrosion resistance and other advantages of the composite material and has become a fast developing direction of material development and application [1–3]. Metal-based ceramic coating materials have been successfully applied to the aerospace, aviation, defense, chemical, mechanical, electronics, metallurgy, mining and other industries [4,5] and will be used increasingly deeply and widely in civil and military fields. Ceramic coatings are even the only way to achieve the functionality of certain parts in the fields of aviation, aerospace and other special areas [6].

The preparation of ceramic coatings by the slurry method has the advantages of the use of simple construction technology, low substrate requirements, no harmful effect on the environment and low cost. The coatings are widely used because of their excellent performance [7,8].

The slurry preparation method for ceramic coatings can be roughly divided into three processes: mixing, coating, and sintering. The sintering of the ceramic coating usually refers to the process of removing the volatiles from the ceramic coating under high temperature, with an increase in the porosity, an increase in the particle contact area and an improvement of the mechanical strength and mechanical parameters. Meanwhile, it is also the final process of ceramic coating production. Therefore, it is of great significance to study the sintering phenomenon of ceramic coatings and the relationship between the mechanism of diffusion and the structure of the material during the heating process.

After more than 60 years of development of a solid phase sintering model, many scholars have done much work and put forward various models and assumptions [9]. Frenkel [10] simplified the complex particle system into a two ball model for the first time and studied the viscous flow of crystal particles. The kinetic equation of the growth rate of the sintered neck was derived. Kuczynski [11,12] used the ball-plate model to establish a theoretical model of sintering dynamics. Based on this, the sintering initiation stage of the sintering initial stage was deduced based on volume diffusion, surface diffusion, grain boundary diffusion and the evaporation and agglomeration mechanism, which

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Table 1

Basic characteristic parameters of ceramic particles.

Particle name	Purity	Particle size range	Shape feature
SiO_2	≥ 99%	$70\mu m-150\mu m$	Sharp edges and corners of an irregular body
Cr_2O_3	≥ 99%	0.2 μm – 0.5 μm	Spherical
Al_2O_3	≥ 99%	20 μm – 70 μm	Spherical or nearly spherical
MgO	≥ 99%	10 μm – 50 μm	Spherical or nearly spherical
Al	$\geq 99\%$	25 μm – 80 μm	Spherical or nearly spherical

laid the foundation for sintering diffusion theory. Coble [13,14], through detailed experimental observation and analysis, proposed a three-stage Coble sintering model of early sintering, sintering, and late sintering. Ashby studied the formation of a double-ball sintered neck prior to sintering [15]. Evans theoretically analyzed porosity, grain boundary segregation and coarsening in the middle and late sintering stages [15].

In this paper, SiO_2 , Cr_2O_3 , Al_2O_3 , and MgO were used as ceramic aggregates, and a small amount of Al powder was added to prepare a ceramic coating on a Q235 steel substrate. The effects of the sintering temperature on the phase structure and microstructure of slurry coatings were studied. At the same time, the wear resistance of ceramic coatings sintered at different temperatures was studied.

2. Experimental materials and methods

2.1. Experimental materials

The substrate of this experiment is Q235 carbon steel, and the size is 50 mm * 25 mm * 5 mm. The basic characteristics of the ceramic particles of the ceramic coating paste are shown in Table 1. The SEM morphologies of the SiO₂ and Cr₂O₃ powders are shown in Fig. 1. Fig. 1(a) and (b) show that the SiO₂ particles exhibit an irregular body with sharp edges and corners, while the Cr₂O₃ particles exhibit a spherical body. The coarseness and angular irregularity of the SiO₂ particles lead to the formation of a mosaic structure within the coating, which effectively increases the strength and abrasion resistance of the coating. The cross-sectional morphology of the ceramic coating after sintering at 400 °C was observed. The irregular SiO₂ particles with thick and sharp edges and corners, shown in Fig. 2(a), were coated with fine Cr₂O₃ and MgO particles. After hardening, the coating had a similar concrete structure. Mixed with fine spherical Cr₂O₃ and MgO particles of the sol as the matrix phase, SiO₂ particles are one of the strengthening phases. At the same time, due to the presence of coarse particles of SiO₂, the accumulation of SiO₂ particles easily leads to the formation of holes in the ceramic coating during the spraying process, as shown in Fig. 2(b), resulting in decreased coating density and corrosive media

channels.

2.2. Coating preparation

Q235 carbon steel was cut into a small size of 50 mm * 25 mm * 5 mm by a wire cutter. The surface of the sample was treated with oil removal, rust removal, and surface blasting. The sand blasting grade was Sa2.0. The finished sample was placed in a dryer for standby.

The powder was weighed in the proportions shown in Table 2 and then mixed with a homemade low-sintering point sol as a solvent at a powdery sol ratio of 6:4 in a planetary ball mill for 4 h to form a ceramic coating slurry. Then, an air compressor spray gun was used to spray the slurry evenly onto the Q235 steel substrate pre-treated with rust removal, degreasing, and surface blasting. The thickness of the spray was controlled at 180 \pm 10 μ m. After drying at room temperature for 6 h, the slurry was sprayed under 200 °C, 400 °C, 600 °C and 800 °C box-type resistance furnace sintering for 2 h, forming a test ceramic coating.

2.3. Coating characterization

The preparation and observation process of the ceramic coating SEM morphology sample are as follows: The sample was cut into a 10 mm * 10 mm square sample in central area by a wire cutter, and the other surface of the sample was sealed with a Bakelite powder on a metallographic sealer. The observation surface was ground with a series of SiC paper, polished by using diamond paste to obtain a mirror smooth surface. The surface and cross section morphology of the coating was observed by the FEI Quanta 250 scanning electron microscope (SEM) with an energy dispersive spectrometer (EDS).

The phase composition of the coating was determined by a Nippon Science & Technology's TTR III Multifunctional X-ray Diffractometer. The test wavelength was Cu K_{ct} , measuring the angle range from 10° to 90° at each 0.2° step. The surface morphology of the coating was observed with a Japanese Kehnens VHX-2000 stereomicroscope and taken with the accompanying software. The wear resistance test of the coating was carried out on an HH-600 type friction and wear tester using a ball disc model. A WC-Co cemented carbide ball with a radius of 6 mm was used for the mill. The friction load was 5 N, the friction radius was 2 mm, the rotational speed of the turntable was 1200 r/min, and the wear time was 10 min. The weights of the samples before and after wear were determined using an ML 20-type electronic balance of 0.1 mg manufactured by METTLER TOLEDO. The average value of the specimen from three weighing readings was used as the weight.

The thermal shock test was carried out in a box-type resistance furnace. The coated sample was heated to 700 °C, then incubated for 20 min and placed in cold water at room temperature to observe



Fig. 1. SEM image of ceramic powder: (a) SiO₂ powder; (b) Cr₂O₃ powder.

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