

# Microstructure Analysis of Cold Sprayed Nickel-based Silicon Carbide Composite Coatings

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## Abstract

In this paper, a systematic study of cold sprayed nickel-based silicon carbide coatings was carried out in terms of microstructure, deposition mechanism, microstructure and strengthening mechanism. The results show that the area share of SiC particles on the three coating cross sections was about 1.8%, 4%, and 6.5%. The maximum average hardness of the coating reaches 339.31 HV1 and the maximum average bond strength reaches 23.665 MPa. The cold spraying technology significantly reduces the problems of poor bonding and high porosity caused by the significant difference in the coefficient of expansion of ceramics and metal, and improves the performance parameters of the coating to a certain extent. Through the detailed analysis of the powder deposition mechanism and in-depth discussion of the principle of ceramic particle refinement, the role of ceramic particles in the coating strengthening process was analyzed.

## Keywords

Metal-ceramic Composite Coatings; Sic; Microstructure Analysis; Mechanical Properties; Wear Resistance; Friction Mechanism; Ceramics.

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## 1. Preface

Ceramics have become one of the hotspots of current research by virtue of its superior performance. However, due to the characteristics of the material itself, the application of ceramics in the field of coating has been greatly restricted. In order to change this status quo, metal-based ceramic composite coatings came into being, and has become one of the hot spots in metal composite coatings[1]. However, due to the large difference between the expansion coefficient of ceramics and metal, the coating performance of the traditional thermal spray technology is not ideal. Especially with plasma arc, fuel and electron beam as the heat source of plasma spraying, supersonic flame spraying and laser cladding technology, the metal-ceramic composite coatings prepared by the residual stress is large, the coating of the internal defects are more, and the coating is easy to fall off[2]. Cold spraying technology is a relatively low temperature in the use of high-speed airflow powder material sprayed to the surface of the substrate, the formation of the coating surface treatment method. Since there is no significant thermal effect on the material during the spraying process, it has an important advantage in spraying heat-sensitive or easily oxidized materials[3].

In the use of cold spraying technology to prepare metal-ceramic composite coating process, the impact of the particles can be induced metallurgical bonding and mechanical interlocking, this is because the powder impact particles have a huge kinetic energy, with the substrate or has been deposited material will be strongly deformed when the impact will occur to produce a particle jet[4], and can be to a certain extent to remove the oxidized layer[5], resulting in a fresh metal surface to promote the occurrence of the materials of the metallurgical bonding[6-8]; when the ceramic particles with the When the ceramic particles and the deposited material impact “embedding mechanism” will

make the SiC particles anchored or embedded in the substrate to produce mechanical interlocking effect[9], thus promoting the material bonding and to a certain extent to enhance the strength of the material, but these ceramic particles can only be retained in the boundary of the metal particles[10], which will to a certain extent impede the two sides of the metal surface direct contact and the formation of metal bonds. However, these ceramic particles can only be retained at the boundary of the metal particles, which will somewhat hinder the direct contact between the two metal surfaces and the formation of metal bonds. This paper discusses the deposition mechanism of nickel-based silicon carbide coatings from the aspect of microstructure, the strengthening mechanism of ceramic additive phase; from the aspect of microstructure, mechanical properties and friction and wear data on the coating friction mechanism has been studied and analyzed.

## 2. Test Preparation

### 2.1 Sample Preparation

The matrix material used for the test is China GB standard steel 45 steel, composition see Table 1, size 200mm \* 120mm \* 10mm. 99.9% purity of dark green silicon carbide and 99.9% nickel powder produced by Kezhou Metal Materials Co. The powder was dried for 12 hours to prepare the spare powder required for the test.

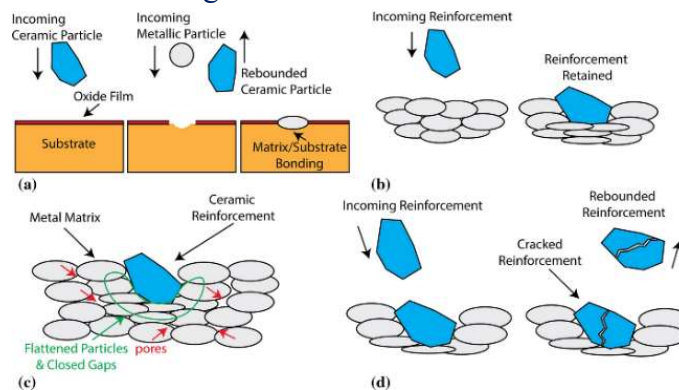
**Table 1.** 45 steel composition

Element	Content	Element	Content
C	0.42~0.50%	Cr	≤0.25%
Mn	0.50~0.80%	Ni	≤0.25%
Si	0.17~0.37%	Cu	≤0.25%
P	≤0.035%	Fe	other
S	≤0.035%		

**Table 2.** Initial powder composition

No.	SiC vol	SiC size(μm)	Ni vol	Ni size(μm)
1#	10%	10	90%	30
2#	30%	10	70%	30
3#	50%	10	50%	30

#### 1.1.1 Spraying Parameters and Bonding Mechanism



**Fig. 1** Schematic representation of the ceramic particle deposition process (a) surface activation, (b) buffering effect of the matrix, (c) ceramic-enhanced tamping effect and (d) erosion[11]

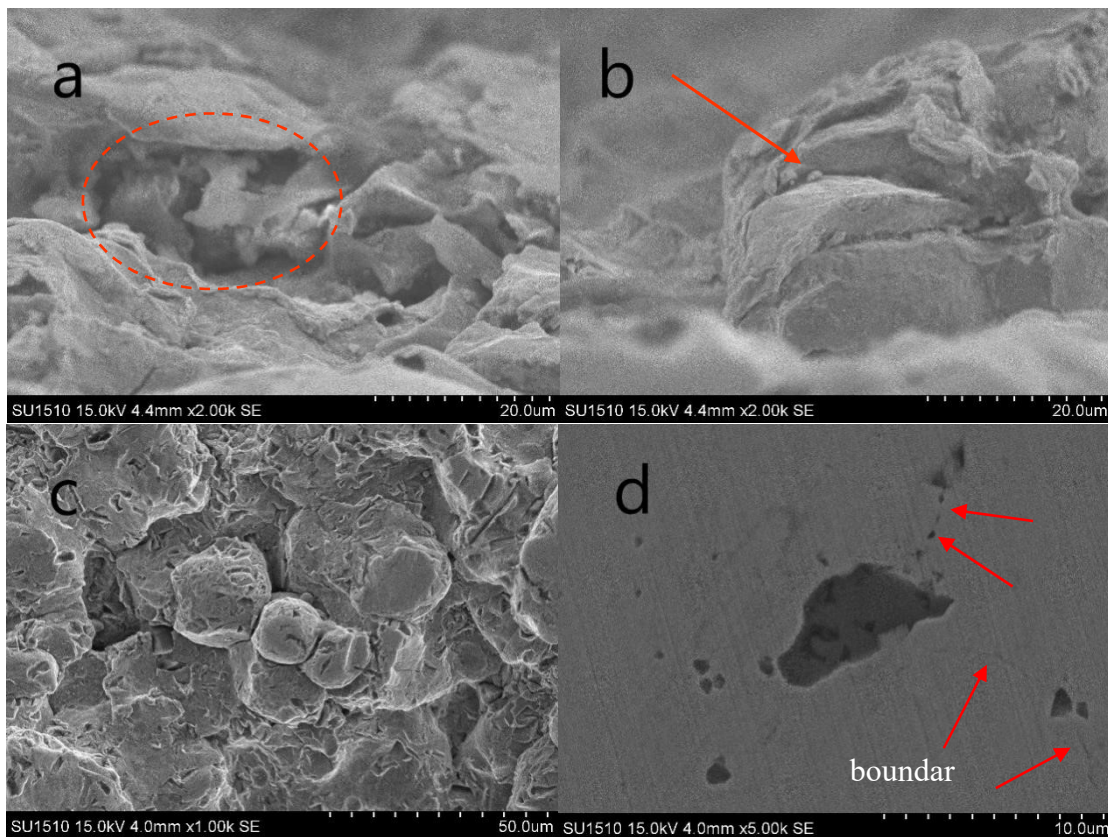
Nitrogen was used as the carrier gas during the spraying process in this test, carrier gas pressure: 3.0 MP, spraying distance: 30 mm, gun head moving speed: 300 mm/s, powder feeding rate: 2 r/min, preheating temperature: 600 °C, number of spraying passes: 6 passes, size of the spraying area: 120 mm × 30 mm. the deposition of the SiC particles in the coating was as shown in Fig. 1.

In the deposition process, SiC particles and the substrate and the deposited material impact can be removed to a certain extent of the oxide layer, part of the SiC particles will be crushed due to impact, in the form of finer particles remain in the coating, “embedding mechanism” so that the ceramic particles are mainly in the form of mechanical embedding in the coating. The subsequent material will cover the deposited material, the rapid deformation of the adiabatic nature of the powder leads to a local temperature increase[12,13], which in turn causes dynamic recrystallization[14,15], and the internal grains of Ni powder will also be different degrees of refinement due to the impact, and generate a large density of dislocations in the interfacial region to adapt to the plasticity[16]. The interfaces between the different particles will undergo some fusion in the adiabatic state, resulting in metallurgical bonding[17].

### 2.2 Structural Characterization and Mechanical Property Testing

HITACHI (Hitachi) field emission scanning electron microscope was used to observe the powder microscopic morphology and composition analysis; bonding strength test was carried out by CTM8050 microcomputer-controlled electronic universal testing machine at a speed of 50mm/min for the tensile test; MHV-1000Z automatic turret digital microhardness tester produced by Beijing Times Peak Science and Technology Co. 1kg load holding time 15s.

### 3. Microstructure Analysis

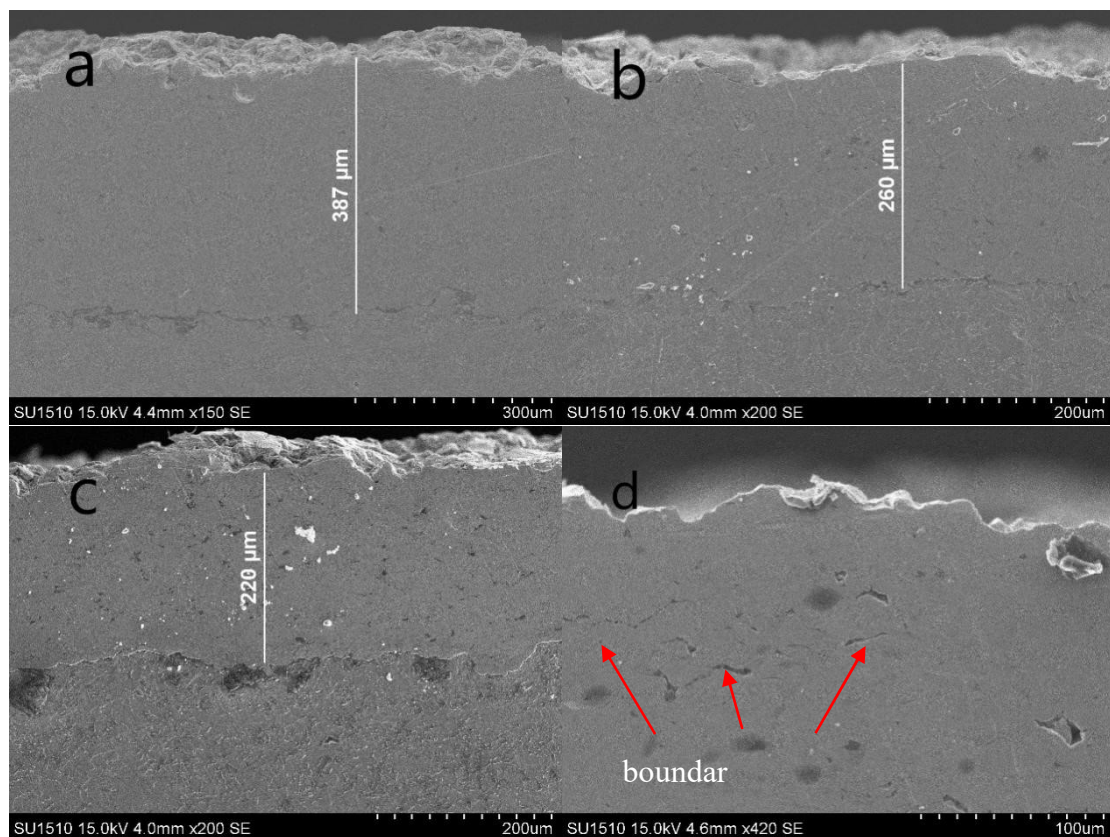


**Fig. 2** SEM images of the coated surface, (Figs. a, b, and c show the original surface after spraying, and Fig. d the surface after sanding to remove SiC particles)

Fig. 2 shows the SEM image of the coating surface formed after powder deposition, which visualizes the changes of the powder during the deposition process. The slit produced by the fracture of the part

due to excessive deformation in Fig. 2a proves that the metal particles produced obvious plastic deformation during deposition, and the traces after the collision-produced particle jets can also be seen at the broken position indicated by the arrow in Fig. 2b. The gaps generated during deposition also provide deposition locations for the tiny particles generated in the collision; this also explains the diffuse distribution of SiC particles in the coating and the grain refinement of the coating.

Figs 2c, 2d show the SEM images after removing the surface SiC particles using ultrasound. Analysis of the SEM images in conjunction with Fig. 2d shows that the SiC particles underwent significant fragmentation during deposition and were deposited inside the coating, while the deposition location was the boundary of Ni. At a magnification of 15,000 $\times$ , it can be observed that smaller SiC particles were detached around and inside the larger SiC particle pits. The inner wall of the pits after SiC particle detachment was smooth, and no obvious bonding was seen. This further indicates that the SiC particles underwent crushing and refinement during the deposition process, existed inside the coating in a mechanically embedded manner, and did not form metallurgical bonds with the nickel powder. After being processed and analyzed by Image J software, it was found that the area share of SiC particles on the three coating cross sections was about 1.8%, 4%, and 6.5%.



**Fig. 3** SEM image of coating cross section

Fig. 3 shows the SEM image of the coating cross-section, in which it can be seen that the average thickness of the coating gradually becomes thinner with the increase of SiC content, which is about 387 μm, 260 μm, 220 μm, respectively, and the porosity also increases slightly with the increase of SiC content, but the average porosity of the three kinds of coatings are not more than 3%. Figure 3d is a local cross-section of the No. 1 coating, from which it can be observed that a more obvious gap in the particle boundary, the study found that the reason for its generation is that the compaction of the material is not tight enough during deposition, and the second is the powder deposition, the rebound of the particles caused by the “Antarctic” problem.

#### 4. Mechanical Properties

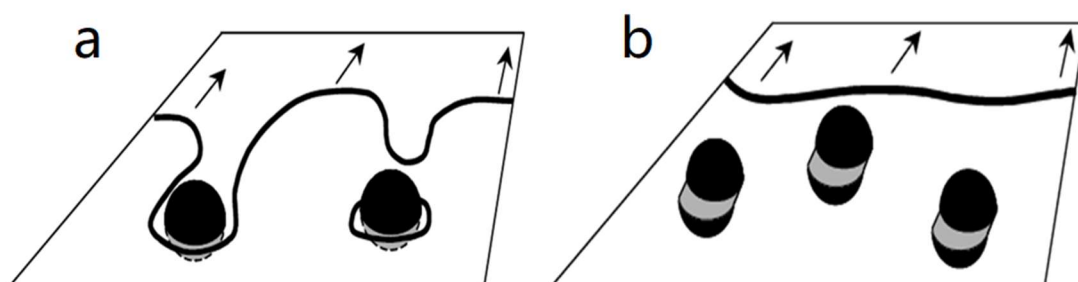
Hardness test on the sample continuously take 15 points to remove the deviation of the larger value, take its average value. For bond strength test, three groups of parallel tests are conducted, three times for each group. Before the tensile test, the samples were bonded and fixed with E7 adhesive, left at room temperature for 24 hours, cured at 130°C for 150 minutes and cooled with the furnace, and then carried out the room temperature tensile test.

**Table 3.** Coating surface hardness and coating bond strength

No.	1#	2#	3#
Average hardness (HV1)	309.48	315.26	339.31
Bonding strength (MPa)	14.034	21.841	23.665

The test results in Table 3 show that the coating enhances the hardness of the substrate by up to about 70%, further illustrating the enhancement of the mechanical properties of the coating by SiC particles. Combined with the deposition mode and refinement of SiC particles in the coating in Fig. 2, it can be seen that the enhancement of the coating is due to the combined effect of tamping and diffusion strengthening.

Dispersive strengthening is that the tiny SiC particles inside the coating enhance the mechanical properties of the substrate by restricting the dislocation movement as well as limiting the slip of grain boundaries[18], and the principle of enhancement is as follows: (1) under the action of external force, the dislocations inside the substrate will move freely to the grain boundaries, but due to the presence of oxide particles in the substrate, these dislocations will be hindered and pinned inside the grains, and only when the external force is further increased, the dislocations will be able to move by bypassing the grain boundaries and the dislocation will not be able to move by the way of the grain boundary. Only when the external force is further increased, the dislocations can move to the grain boundaries again by bypassing (Fig. 4a) or shearing (Fig. 4b), when a large number of oxide particles and uniformly dispersed, this pinning effect will be greatly enhanced, thus improving the internal deformation resistance of the substrate; (2) At the same time, under the action of the external force, the grain boundaries will be slipped, and the material will be fractured when the slipping distance is more than a certain range, while the oxide particles at the grain boundaries can effectively impede the slipping motion, which can effectively impede the slipping motion, the grain boundaries at the grain boundaries of the oxide particles. The oxide particles at the grain boundaries can effectively hinder the slip motion, thus improving the deformation resistance at the grain boundaries. This way of enhancing the mechanical strength of the material is still effective even at high temperatures, and the mechanical properties can be continuously enhanced by choosing different oxides or preparation methods<sup>[6]</sup>.



**Fig. 4** Schematic representation of the pinning action of oxide particles (a) Orowan mechanism (b) Shear mechanism[19]

## 5. Results and Conclusion

Cold spraying technology shows unique advantages in the preparation of ceramic - metal composites, and its application prospect is very broad. In this paper, the relationship between microstructure, mechanical properties and silicon carbide content of nickel-based silicon carbide coatings is investigated. In order to meet the industry needs in the field of modern mechanical wear resistance, the structure and properties of cold sprayed nickel-based silicon carbide composite coatings need to be further optimized. Based on the research results of this paper, future research directions are proposed:

- (1) Improve the spraying process to increase the deposition rate of the powder; explore new powder preparation methods to enhance the bonding strength between the metal and ceramic powder in the coating and reduce the shedding of ceramic particles.
- (2) Optimize the powder preparation process, and prepare a composite coating with a high content of nano-ceramic particles and metal powder through a suitable process to improve the diffusion strengthening of the ceramic phase in the coating.
- (3) Through the optimization and control of the cold spraying process, obtain large-size bulk diffusion-reinforced metal-ceramic composites and study their low-temperature and high-temperature mechanical properties and corrosion resistance. To study the effects of material anisotropy and dispersion strengthened anisotropy on the service performance of the materials brought by the cold spraying process.

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