# **Effect of Particle State on the Adhesive Strength of HVOF Sprayed Metallic Coating**

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*(Submitted 3 January 2001; in revised form 20 April 2001)*

**NiCrBSi and Ni-50Cr coatings were deposited using the high velocity oxygen fuel (HVOF) spray process under different spray parameters with two powders of different sizes to clarify the influence of the melting state of spray particles on the adhesive strength of the coating. The adhesive strength of the coating was estimated according to the American Society for Testing and Materials (ASTM) C633-79. The melting state of the spray droplet was examined from the coating microstructure. It was found that the melting state of spray particles had a significant effect on the adhesive strength of HVOF sprayed Ni-based coatings. The significant melting of the spray particle did not contribute to the increase in the adhesion of HVOF metallic coatings. On the other hand, the deposition of a partially melted large particle contributed to the substantial improvement of adhesive strength of the HVOF coating. The subsequent coating presented a dense microstructure and yielded an adhesive strength of more than 76 MPa, which was double that of the coating deposited with completely molten particles. It can be suggested that the good melting of the spray particle is mainly related to the mechanical interlocking effect, which reaches the limited and approximately defined adhesive strength up to 40-50 MPa.**



# **1. Introduction**

High velocity oxygen fuel (HVOF) is characterized by a high flame velocity up to 2000 m/s. Such high flame velocity consequently results in the formation of spray particle streams with high velocity. It is generally believed that the high velocity of spray particles improves the adhesion of the coating to the substrate. Until now, many investigations of the adhesion of HVOF coatings mainly have been concerned with the cermet coatings. The adhesive strength of HVOF cermet coatings, such as WC- $Co^{[1]}$  and  $Cr_3C_2-NiCr,^{[2]}$  to mild steel substrate can generally exceed 70 MPa, which corresponds to the strength of the adhesives used in a pull-out test. According to the pin test result, a high adhesive strength up to 150 MPa may be achieved,  $[3,4]$  although the test results will be greatly influenced by coating thickness and the diameter of the pin used in the test.[5] Such results evidently confirm the excellent adhesion of a coating obtained by the HVOF process.

Therefore, the HVOF process has become a very popular process to deposit a dense coating with excellent adhesion. In addition to cermet coatings, the HVOF process is also used to deposit various metallic coatings such as NiCrBSi self-fluxing alloy and nickel- and cobalt-based alloys. It has been reported that the performance of HVOF deposited NiCrBSi coatings is comparable to that produced by the conventional spray-fusing process.[6,7] A recent study into the recrystallization behavior of an HVOF deposited amorphous NiCrBSi coating clearly revealed that the control of the microstructure through annealing treatment could further enhance the performance of HVOF NiCrBSi coatings.[8] Therefore, HVOF deposited metallic coatings are also of great industrial application potential.

However, there are few papers that deal systematically with the adhesion of HVOF deposited metallic coatings. A systematic investigation into adhesion of HVOF Tribaloy 800 (T-800) material using the experimental design method yielded the adhesive strength ranging from 42-52 MPa.<sup>[1]</sup> HVOF titanium coating yielded the average adhesive strength of 35.6 MPa with the range from 27.7-40.3 MPa, which was still larger than that of HVOF copper coating.<sup>[9]</sup> Previous systematic study confirmed that the adhesion of HVOF NiCrBSi coating deposited with well melted droplets is limited to about 40 MPa.<sup>[10]</sup> The result is consistent with that for a similar coating deposited by Jet-Kote, although the coating deposited with the JP-5000 HVOF system yielded some high adhesion of 60 MPa, $^{[11]}$  which is still much lower than the results for WC-Co cermet coating. The results imply that the HVOF process does not always deposit metallic coatings with adhesive strength comparable to cermet coatings.

An investigation into the deposition behavior of WC-Co experimentally revealed that the deposition of HVOF sprayed WC-Co coating takes place through solid-liquid two-phase droplets in which WC carbide particles are in a solid state, whereas the cobalt-based binder phase is in liquid state.<sup>[12]</sup> Therefore, it can be believed that the high adhesive strength of HVOF WC-Co coating is primarily associated with the characteristics of the solid-liquid two-phase droplet deposition process. This implies that the melting condition of a metallic particle before deposition will influence the adhesion of an HVOF metallic coating.

The objective of the present article is to clarify the correlation of the adhesive strength of HVOF metallic coatings with the melting state of the spray particles through an investigation into the influence of the melting state of the spray droplet on the

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adhesive strength. In this study, the melting state of the spray droplet was modified from completely melting to partially melting states using HVOF spray parameters and spray powders of two different particle sizes.

# **2. Materials and Experimental Procedures**

NiCrBSi and Ni-Cr powders were used as spray materials. Each powder was screened into two size fractions: 45-74 and 75-104 µm, respectively. Two NiCrBSi powders of different sizes were assigned as type A and type B, whereas two Ni-Cr alloy powders were assigned as type C and type D. Table 1 shows the classification of the powders and their grain sizes.

The NiCrBSi powder has a nominal composition of Ni, balance; Cr, 20-22; Fe, 5; B, 4.0; Si, 4.0; C, 0.6, whereas Ni-Cr powder has a nominal composition of Ni-50Cr. All powders were manufactured by the gas atomization method and exhibited a near-spherical morphology.

Mild steel was used as a substrate. Prior to the spraying, the substrate was sandblasted using 20 mesh  $Al_2O_3$  grit. Consequently, the substrate surface had a roughness of  $R_a$  5.9  $\mu$ m.

The spraying was carried out with the CH-2000 HVOF system developed in Xi'an Jiaotong University. Propane was used as a fuel gas. During spraying, the pressures of the propane and oxygen were fixed at 0.35 and 0.55 MPa, respectively. The flow of the oxygen was set to 360 L/min, whereas the flow of the propane was changed from 28 to 45 L/min. Nitrogen gas was used as a powder feed gas, which was operated at a pressure of 0.35 MPa. The spray distance was kept at 210 mm throughout spraying. The average thickness of the coatings was about 250 µm.

The tensile adhesive strength of the coating was estimated according to American Society for Testing and Materials (ATSM) C633-79. For each experiment, five specimens were used. A commercially available adhesive (E-7 adhesives, Shanghai Research Institute of Synthetic Resins, China) was used during the test. The microstructures of coatings were examined using an optical microscope to reveal the melting state of the spray droplets on impact with the substrate. The morphologies of the as-sprayed coating surface and the fractured surface after tensile test were examined with scanning electron microscopy (SEM).

To investigate the effect of particle velocity on the adhesive strength, the velocity of the particles was estimated using a particle velocity measurement system developed on the basis of thermal emission of the spray particles. The detailed measurement setup was given elsewhere.<sup>[13]</sup>

**Table 1 Classification of Powders Used in the Experiment**

<b>Type</b>	Alloy	Particle Size, um
Type A	NiCrBSi	$+45$ to $-74$
Type B	<b>NiCrBSi</b>	$+75$ to $-104$
Type C	$Ni-50Cr$	$+45$ to $-74$
Type D	$Ni-50Cr$	$+75$ to $-104$







**Fig. 1** Typical microstructure of HVOF sprayed NiCrBSi coatings under different propane gas flows: **(a)** 28 L/min; **(b)** 36.5 L/min; **(c)** 45 L/min



**Fig. 2** Effect of propane flows on the adhesive strength of HVOF NiCrBSi coatings

## **3. Results**

### *3.1 The Effect of Propane Flow on the Adhesive Strength of NiCrBSi Coatings*

Figure 1 shows typical microstructures of HVOF NiCrBSi coatings sprayed at different propane gas flows with type A powder. There exist many particles in near-sphere shape in the coating deposited under the low propane flow of 28 L/min, which indicates that many spray particles were in a partially melted state during deposition. Nevertheless, the coating is evidently dense owing to the high particle velocity. With the increase in propane flow, the coating tends to be composed of well-flattened splats. When the propane flow was increased up to 45 L/min, the coating presented the classic lamellar structure, which implies that the particles were sufficiently melted before deposition.

Figure 2 shows the influence of propane flow on the adhesive strength of NiCrBSi coatings deposited by type A powder. The examination of the fractured pattern showed that all specimens fractured at the interface between the coating and the substrate. All NiCrBSi alloy coatings yielded an average adhesive strength of about 40 MPa. Evidently, the fuel gas flow has no significant influence on the adhesive strength, despite the much improved particle melting state with an increase in the fuel gas flow, as shown in Fig. 1. Generally, it is believed that the complete melting of spray particles during thermal spraying contributes to good adhesion between coating and substrate, especially under the traditional low velocity thermal spraying processes such as the conventional plasma spraying process. However, it is clear from the present results that the higher adhesive strength cannot



**Fig. 3** Typical microstructure of HVOF NiCrBSi coating deposited with type B powder

be achieved, although spray particles are sufficiently melted in the HVOF process. With the HVOF NiCrBSi coatings deposited with the powder of particle size from 45-74  $\mu$ m, the previous systematic investigation yielded the maximum average tensile adhesive strength of about 40 MPa,<sup>[10]</sup> which is low compared with the adhesive strength of HVOF WC-Co coatings.<sup>[1]</sup>

#### *3.2 The Effect of Powder Size on the Adhesive Strength of NiCrBSi Coatings*

When the type B NiCrBSi powder was used to deposit the coatings with the same spray parameters as those used for type A powder, it was found that the coating thickness could not be increased to more than several tens of microns when the propane gas flow rate was lower than 36.5 L/min. This fact implies that the melting of spray particles is too poor to be deposited in the coating under those conditions. On the contrary, the erosion of the previously formed coating by unmelted solid particles may occur. However, the coating was successfully deposited under a propane gas flow of 45 L/min. Figure 3 shows a typical microstructure of the coating deposited by type B powder. The coating was clearly built up of partially melted particles and has a dense microstructure that is the result of the high velocity of the spray droplets.

Figure 4 illustrates the influence of powder size on the adhesive strength of HVOF NiCrBSi coatings sprayed under propane flow of 45 L/min. The adhesive strength of the coating deposited with type B NiCrBSi powder exceeded 67 MPa, which was nearly doubled compared with the coating deposited with type A powder. Furthermore, the examination of the fractured surface showed that three among five specimens with type B powder fractured from failure in the adhesives, whereas the other two specimens presented a complicated mixed failure: of adhesives and the inside of the coating, and from a small amount of the interface of the coating with the substrate. The previous study showed that with the increase in the adhesion of the HVOF



**Fig. 4** Effect of powder grain size on the adhesive strength of HVOF NiCrBSi coatings



**Fig. 5** Effect of propane flow on the adhesive strength of HVOF Ni-50Cr coatings deposited with type C powder

NiCrBSi coating, the apparent adhesive strength is dominated by the cohesion of deposited particles in the coating.[10] Therefore, the present result also implies that with the improvement of

the adhesion of HVOF metallic coating, the apparent tensile adhesive strength of the coating tends to be dominated by the cohesion between deposited particles.

## *3.3 The Adhesive Strength of HVOF Ni-50Cr Coatings*

Figure 5 shows the effect of propane gas flow on the adhesive strength of HVOF Ni-50Cr coatings deposited with type C powder. The apparent average adhesive strength was increased from 31-42 MPa when the fuel gas flow was increased from 28-37 L/min. With the further increase in fuel gas flow to 45 L/min, the apparent adhesive strength tended to be decreased, although a high propane flow led to good melting of the spray droplets. All specimens were fractured at the interface between the coating and the substrate during the tensile test. The examination of microstructures of the deposited coating confirmed that the coating deposited under low propane flow was composed substantially of partially melted particles, as shown in Fig. 6. Therefore, it is consistently evident that the improvement of melting state of the spray particles is not associated with the improvement of the adhesion of the metallic coating during the HVOF process.

Figure 7 shows the effect of Ni-50Cr powder size on the adhesive strength of the HVOF coating. The adhesive strength of the coating deposited with large powder (type D) exceeded 57 MPa because all samples were fractured in the adhesive during the tensile test. The result is consistent with results obtained using NiCrBSi powder. It was confirmed that the coating was also deposited with partially melted particles when using type D powder, as shown by the microstructure in Fig. 8.

## **4. Discussion**

The present results reveal that the melting state of spray particles in the HVOF process has a significant influence on the tensile adhesive strength of the NiCrBSi and Ni-50Cr coatings. With the improvement of the particle melting state, the adhesive strength of the coating is not increased when the powders of small particle size are used, such as type A and type C powders in the current study. On the other hand, when the powders of large particle size are used, the coating deposited with partially melted particles consistently yielded a much higher adhesive strength. Therefore, the adhesive strength of the HVOF metallic coating can be significantly improved by the deposition of the spray particles in the partially melted state rather than in the completely molten state.

It has been suggested that the impact of partially melted particles at high velocity on a substrate surface leads to the plastic deformation of the surface, which introduces the peening effect to the substrate surface. As a result, a high level compressive residual stress may be produced in the coating.[14] Such an effect will contribute to the increase of the apparent adhesion of the coating to the substrate. Moreover, a partially melted particle cannot be fully deformed and spread during splatting when it impacts on the surface; the contact area between the droplet and substrate is effectively limited, which results in a very high impinging pressure concentrating on the smaller area. Such high contact pressure contributes to the good contact between the droplet and substrate, and produces good adhesion.







**Fig. 6** Effect of propane flow on the microstructure of HVOF Ni-50Cr coatings: **(a)** 28 L/min; **(b)** 35.5 L/min; **(c)** 45 L/min



**Fig. 7** Effect of powder grain size on the adhesive strength of HVOF Ni-50Cr coatings



**Fig. 8** Typical microstructure of HVOF Ni-50Cr coating deposited with type D powder

On the other hand, the present results revealed that the improvement of the adhesion between substrate and coating through the deposition of partially melted particles also is dependent on the droplet size. The large droplet in the partially melted state considerably improves the adhesion of the HVOF metallic coating to a substrate compared with the small droplet in a partially melted state. The measurement of particle velocity showed that both type C and type D powders yielded approximately similar mean velocities of 366-398 m/s, respectively, under the same propane flow of 45 L/min and present spray distance of 210 mm. Therefore, it can be suggested that the total kinetic energy of a partially melted liquid-solid two-phase droplet also plays an important part in the improvement of coating adhesion. The heavy droplets, such as large droplets resulting from type B and type D powders used in the current study, and WC-Co (with large density), lead easily to the generation of highly localized contact pressure between droplet and substrate, and therefore the formation of an HVOF coating with high adhesive strength.

However, when a well-melted droplet impacts on a substrate, the droplet fluid flattens laterally. As a result, the effective contact pressure is limited to a much-localized area only at the initial impact stage.<sup>[15]</sup> As the flattening progresses, substantial area between splat and substrate develops and there is lack of sufficient contact pressure to ensure the good contact of splat with substrate, which consequently leads to ineffective physical adhesion. On the other hand, the good flattening of droplet fluid may lead to an increase in the substrate surface area covered by subsequent splat. This will contribute to the enhancement of mechanical interlocking, that is, mechanical bonding. Because the mechanical interlocking does not depend on coating materials and mainly depends on the surface roughness and flattening degree of the droplet, the mechanical interlocking effect of splats formed by a completely molten droplet under certain surface roughness conditions would not be influenced largely by droplet state, such as velocity when the splashing behavior during droplet flattening is taken into consideration.<sup>[16]</sup> Therefore, with certain surface roughness, the adhesive strength of a sprayed coating contributed by the mechanical interlocking effect would have a certain value.

The present results revealed that the HVOF coating deposited with well-melted droplets reached an adhesive strength of about 40 MPa. This result was also confirmed by using HVOF MrCrAlY coatings.<sup>[16]</sup> Although the surface conditions in the literature may be different, the adhesive strength of the most HVOF metallic coatings reportedly yielded values from 30 to approximately 50 MPa,  $^{[1, 9-11]}$  which is consistent with the results in the current study. Therefore, it may be suggested that the adhesion resulting from mechanical interlocking is limited to a typical value from 40-50 MPa. This value may be increased slightly with the increase in the surface roughness. The adhesive strength values of different metallic coatings deposited with various spray methods (such as flame spraying, arc spraying, air plasma spraying, and low pressure plasma spraying) at low substrate preheating temperatures fell into the range from 30 to approximately 50 MPa, as shown by the data collected by Pawlowski<sup>[18]</sup> from substantial literature. Those results provide further evidence for the suggestion made above that the adhesion generated by mechanical interlocking is limited to 40-50 MPa. Therefore, the high adhesive strength cannot be expected for the metallic coating even with the HVOF process when the spray particle is subject to substantial melting before impact on the substrate.

It is believed, however, that the effective approach to enhance the adhesion of HVOF metallic coating is to produce spray particles in the solid-liquid two-phase state of partially melting, similar to those produced with WC-Co cermet particles during the HVOF process.

# **5. Conclusions**

The melting state of metallic droplets during the HVOF process was modified using different spray parameters and powder sizes. The influence of the melting state of the droplets on the adhesive strength of the HVOF metallic coating was investigated. The melting state of the spray particle and droplet size significantly influenced the adhesive strength of HVOF metallic coating. The complete melting of the spray particles in the HVOF process did not contribute to the increase in the adhesive strength of the coating. The adhesive strength of the HVOF sprayed NiCrBSi and Ni-Cr coatings deposited with the powder of particle size 45-74 µm reached about 40 MPa and was not significantly influenced by the fuel gas flow rate; that is, the melting state of the droplet, despite the partially melted state of droplets, resulted from the HVOF flame of low fuel gas flow condition. The HVOF metallic coating deposited with large nickel-based alloy powders from 75-104 µm particle size was composed persistently of partially melted particles and presented a dense microstructure, and yielded a much higher adhesive strength of more than 76 MPa, which is about twice that of the coating deposited with the powder of 45-74 µm particle size. The large droplet in the partially melted state is more effective to the improvement of the adhesion of HVOF metallic coating compared with the small droplet, which is also in the partially melted state.

It can be suggested that the complete melting of spray particles is mainly related to the mechanical bonding effect, which reaches the limited adhesive strength up to 40-50 MPa.

### **Acknowledgment**

The present project was financially supported by the National Natural Science Foundation of China (Contract No. 59671061).

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