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Effect of Ceramic Particle Velocity on Cold Spray Deposition of Metal-Ceramic Coatings

A. Sova, V.F. Kosarev, A. Papyrin, and I. Smurov

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In this paper, metal-ceramic coatings are cold sprayed taking into account the spray parameters of both metal and ceramic particles. The effect of the ceramic particle velocity on the process of metal-ceramic coating formation and the coating properties is analyzed. Copper and aluminum powders are used as metal components. Two fractions of aluminum oxide and silicon carbide are sprayed in the tests. The ceramic particle velocity is varied by the particle injection into different zones of the gas flow: the subsonic and supersonic parts of the nozzle and the free jet after the nozzle exit. The experiments demonstrated the importance of the ceramic particle velocity for the stability of the process: Ceramic particles accelerated to a high enough velocity penetrate into the coating, while low-velocity ceramic particles rebound from its surface.

Keywords cermets, cold spray, composite coating, particle velocity

1. Introduction

Cold spray coatings are deposited by plastic deformation of the sprayed material. Therefore, only plastic materials such as metals can form coatings. Besides, certain critical relationships between the particle parameters such as size, velocity, and temperature must be achieved. Otherwise, particles do not coat the surface but provoke its erosion as a result of a high-speed impact (Ref 1-3). Pure nonplastic materials (ceramics, oxides, etc.) produce no coating, but erode the surface. Metal coatings with ceramic inclusions were formed from previously prepared cermet mixtures in Ref 4-13. Some essential differences between spraying pure metal and cermet mixtures must be noted. For example, it is known that the deposition efficiency of a cermet mixture depends strongly on the mass ratio between components and, under certain conditions, can considerably exceed that of pure metals (Ref 5, 7). Hard ceramic particles create microasperities that favor the bonding of the incoming Al particles and increase the contact area between the coating and the substrate (Ref 7). It was also shown that the deposition efficiency of a metal-ceramic mixture strongly depends on the ceramic particle granulometry (Ref 8).

Different properties of the cold spray cermet coatings such as adhesion, wear resistance, corrosion resistance, and so forth were studied (Ref 4, 7, 9-13). For example, the addition of Al_2O_3 to Al powder is reported to enhance the coating-to-substrate adhesion (Ref 7). Lee et al. (Ref 9) demonstrated that the adhesion and density of a coating depends on the quantity and size of the ceramic material. The ceramic particle shape could have an influence on the coating properties (Ref 11). References 10 and 12 report that the porosity of copper-alumina and Ni-20Cr coatings could be significantly diminished by increasing the amount of Al_2O_3 particles in the initial powder mixture. Also, the Al-Al_2O_3 coatings proved to be as efficient as pure Al coatings in providing corrosion protection under exposure to alternate immersion in saltwater and against salt spray environment (Ref 7).

Al_2O_3 particles alone cannot form a coating under cold spray conditions. It is suggested that they peen and roughen the layers by a high-velocity impact during mixture deposition. However, their inclusion in the coatings is observed and is limited to about 25 wt.% (Ref 7, 8).

Actually, the presence of the ceramic particles in the coating is explained by mechanical ‘‘wedging’’ and following ‘‘riveting’’ by the metal particles of the sprayed mixture. Consequently, the ceramic particles should have rather a high velocity to penetrate into the metal matrix formed by the metal particles. Thus, the ceramic particle velocity influences the process of the cermet coating formation, and the coating properties as well. Also, it is clear that the ‘‘penetration velocity’’ should have some

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A. Sova and **I. Smurov**, Ecole Nationale d'Ingénieurs de Saint-Etienne (ENISE), DIPI Laboratory, 58 rue Jean Parot, 420023 Saint-Etienne, France; **A. Sova** and **V. F. Kosarev**, Khristianovich Institute of Theoretical and Applied Mechanics SB RAS (ITAM SB RAS), 630090 Novosibirsk, Russia; and **A. Papyrin**, Cold Spray Technology, LLC, Albuquerque, NM. Contact e-mail: smurov@enise.fr.

correlation with the ceramic particle size. The objective of this study is to assess how the velocity of different-size ceramic particles influences the process of the cermet coating formation and some coating properties (ceramic content and microhardness). The optimal way to detect the influence of the ceramic particle velocity on the process of cermet coating formation by cold spray is to spray metal-ceramic mixtures at fixed impact parameters of the metal component and different velocities of the ceramic particles. In this study, the variation of the ceramic particle impact velocity was realized by the displacement of the ceramic powder injection zone in the direction of the nozzle exit. It is known that the particle impact velocity in cold spray depends on the length of the supersonic part of the nozzle (Ref 3). At practically the same gas flow parameters, the ceramic particle impact velocity is smaller for a shorter acceleration path. At the same time, the metal powder should be always injected into the subsonic part of the nozzle to ensure stable impact parameters of the metal particles.

2. Experimental Procedure

In the present study, three configurations of the ceramic powder injection were tested (Fig. 1).

In configuration A, a MOC nozzle developed by Cold Gas Technology was used. MOC nozzle has critical section diameter 2.7 mm and exit diameter 6.5 mm. Mechanical mixtures of ceramic and metal powders were injected in the prechamber of the MOC nozzle.

In configuration B, an ejector nozzle developed in DIPI and ITAM described in Ref 8, 14, and 15 was used. ITAM nozzle has critical section diameter 3 mm and exit diameter 6.5 mm. This nozzle makes it possible to feed powders in the subsonic part as well as in the supersonic part. In the present study, metal powder was injected into the subsonic part, and ceramic particles were injected in the supersonic part. In this case, erosion of the nozzle walls in the critical section is avoided (Ref 8). The ceramic powder was injected at different zones all around the diameter of the nozzle, the injection angle was 90°. The ceramic powder carrier gas flow rate was 0.1 m^3/min. The ceramic and metal powders were injected by means of two independent feeders (feeder CGT-PF4000 and domestic feeder manufactured at ITAM).

In configuration C, the ceramic powder was injected in the free jet after the exit of the MOC nozzle, whereas the metal powder was injected in the prechamber of the nozzle. Two separate powder feeders were also used in this configuration. The ceramic powder carrier gas flow rate was 0.1 m^3/min. Ceramic powder was injected into the supersonic jet through the 3 mm tube. Therefore, the ceramic powder injection jet had smaller dimensions than the supersonic jet of the MOC nozzle (exit diameter of MOC nozzle is 6.5 mm).

It should be noted that the use of two different types of nozzle (ejector nozzle and MOC nozzle) does not ensure stable impact parameters of the metallic particles since geometry of the ejector nozzle differs slightly from geometry of the MOC nozzle. In consequence, the current experiments permitted only evaluation of the influence of the ceramic particle velocity and definition of its basic trends. Unfortunately, it was impossible to use the ITAM nozzle in configuration A. The walls of the ITAM nozzle made from steel are strongly eroded by the ceramic particles in the critical section. As for configuration C, both types of nozzles could be used. It was expected that the difference of the ceramic particle velocity between configurations A and C would be more significant than the difference between configurations A and B and B and C. Therefore, the comparison of configurations A and C could demonstrate the influence of ceramic particles velocity on formation of metal-ceramic coatings in the clear way. Thus, it was particularly important to keep constant the impact parameters of the metal powder in experiments with configurations A and C. Hence, a MOC nozzle was chosen for configuration C because this nozzle was already used for configuration A.

In all spray configurations, the carrier gas flows from the powder feeders was set at room temperature.

Experiments were performed in DIPI laboratory (ENISE, St-Etienne, France) on cold spray equipment

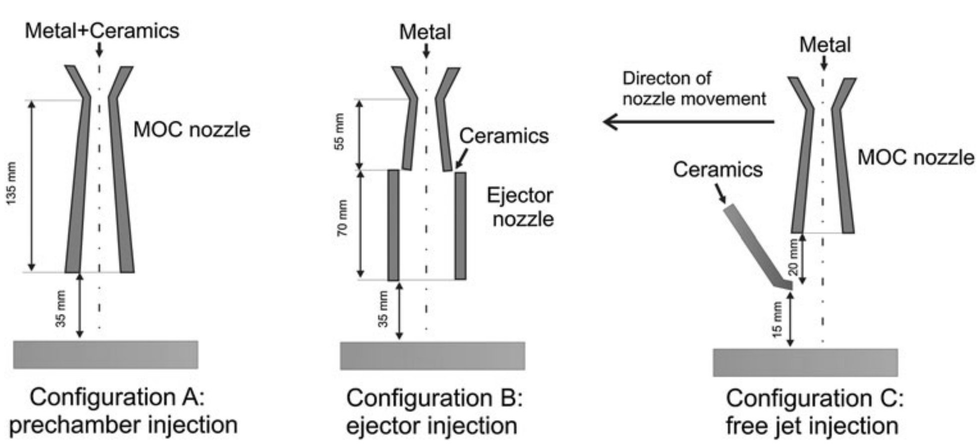

Fig. 1 Powder injection method

Table 1 Powder characterization

Powder	Mean size, μm (volumetric statistics)	Standard deviation, μm	Shape
Al-25	25	12	Spherical
Cu-39	39	13	Irregular
Al_2O-19	19	8	Irregular
Al_2O_3-127	127	14	Irregular
SiC-25	25	11	Irregular
SiC-135	135	18	Irregular

Kinetiks 4000 (CGT GmbH, Amfing, Germany) mounted on a robot IRB 4400 (ABB, Saint Ouen L'Aumône, France).

Experiments were conducted with two metal powders (aluminum and copper) and four different ceramic powders (two types of aluminum oxide and silicon carbide). Particle size was analyzed in DIPI laboratory (ENISE, St-Etienne, France) by an optical granulomorphometer Alpaga 500 Nano (OCCHIO SA, Angleur, Belgium), which is an optical sieving system, and Callisto image analysis software (OCCHIO, Angleur, Belgium). Parameters of the tested powders are presented in Table 1. To distinguish the ceramic powders between them, their names will be followed by a hyphen with the average value of the particle size, for example, Al_2O_3-19, SiC-135, and so forth.

The substrate material was aluminum, sandblasted before spraying. Four passes with 90° spraying angle and 20 mm/s nozzle speed were applied for all the types of mixtures. Powder feeding rate in configuration A was 1.2 and 0.9 g/s for copper-ceramic and aluminum-ceramic mixtures, respectively. In configurations "B" and "C," copper and aluminum feeding rates were 0.9 and 0.4 g/s, respectively. The feeding rates of ceramic powders in configurations B and C were set aiming to obtain the 50 vol.% metal + 50 vol.% composition of the mixtures (0.45 and 0.5 g/s for fine and coarse SiC, respectively; 0.5 and 0.7 g/s for fine and coarse alumina, respectively).

Nitrogen was used as working gas with 3 MPa stagnation pressure in all the tests. Gas stagnation temperature was 500 K for the aluminum-ceramic mixtures and 700 K for the copper-ceramic mixtures.

The powder mass flow rate was measured by weighing the powder injected into a closed volume after 10, 30, and 60 s time intervals. Nitrogen was used as a carrier gas with 5 m^3/h flow rate.

The particle velocity before impact was measured experimentally by a domestic CCD-camera-based diagnostic system of DIPI exploiting the well-known PTV method (Ref 16, 17). The optical device consists of a nonintensified image sensor Exview HAD CCD by Sony Inc. and achromatic telescopic lens providing a real-time monitoring area of 23 × 30 mm of the powder jet. The capturing of gray scale images was performed with resolution 1038 × 1366 points (Ref 16, 17). Particle velocity measurements (10 μs exposure time) were conducted in the free jet zone situated 5 mm from the nozzle exit. All measurements were performed without a substrate in a free jet in the zone situated 2 mm from the assumed position of the substrate. Particle luminosity was insufficient to apply optical diagnostics because of the low temperature. Consequently, an external illumination source was used.

Table 2 Ceramic content in coatings sprayed at different configurations

	Ceramic content, vol.%		
Spraying mixture	Config. A	Config. B	Config. C
Al + SiC-25	~14	~13	~4
Al + SiC-135	~11	~12	<1
Al + Al_2O_3-19	~13	~13	~5
Al + Al_2O_3-127	~10	~9	<1
Cu + SiC-28	~16	~15	~5
Cu + SiC-135	~10	~10	<1
Cu + Al_2O_3-19	~14	~13	~5
Cu + Al_2O_3-127	~9	~9	<1

The ceramic content in the coatings was evaluated by optical microscope image analysis based on the percentage of the metal and ceramic particles area in the coating cross sections (Carl Zeiss AxioScope microscope with Axiovision 4.7 image analysis software). Polished nonetched cross sections of coatings were used in these measurements. Ten images of each cross section with magnification 10×, 20×, and 50× were chosen for evaluating ceramic content. Image analysis software permits one to distinguish ceramic particles and coating porosity; therefore it is supposed that the porosity influence on accuracy of measurements does not exceed a few percent.

Microhardness measurements were carried out as an average of 10 measurements with a Buehler Micromet 5104.

3. Results

3.1 Ceramics Content

Volumetric content of ceramics in the deposited coatings is given in Table 2. It is obvious that the ceramic content in the coatings depends on the configuration of the ceramic powder injection in the gas flow. Injection of the ceramic particles in the prechamber of the MOC nozzle or in the supersonic region of the ejector nozzle allows deposition of cermet coatings aluminum + ceramics and copper + ceramics with more than 10% volumetric content of ceramics.

Typical microstructures of the coatings deposited using configurations A and B are shown in Fig. 2(a) and (b). It is clear that only a small portion of ceramic particles embed the coating. Metal particles form a metal matrix with uniformly distributed separate ceramics inclusions. Ceramic particles cannot be plastically deformed; they just interlock with the metallic matrix. This structure of the metal-ceramic coatings is typical for the cold sprayed metal-ceramic mixtures (Ref 5-13). Coating porosity was evaluated about 1-1.5%. It also should be noted that large ceramic particles >100 μm were not found in the coating when spraying coarse powders containing particles up to 150 μm and

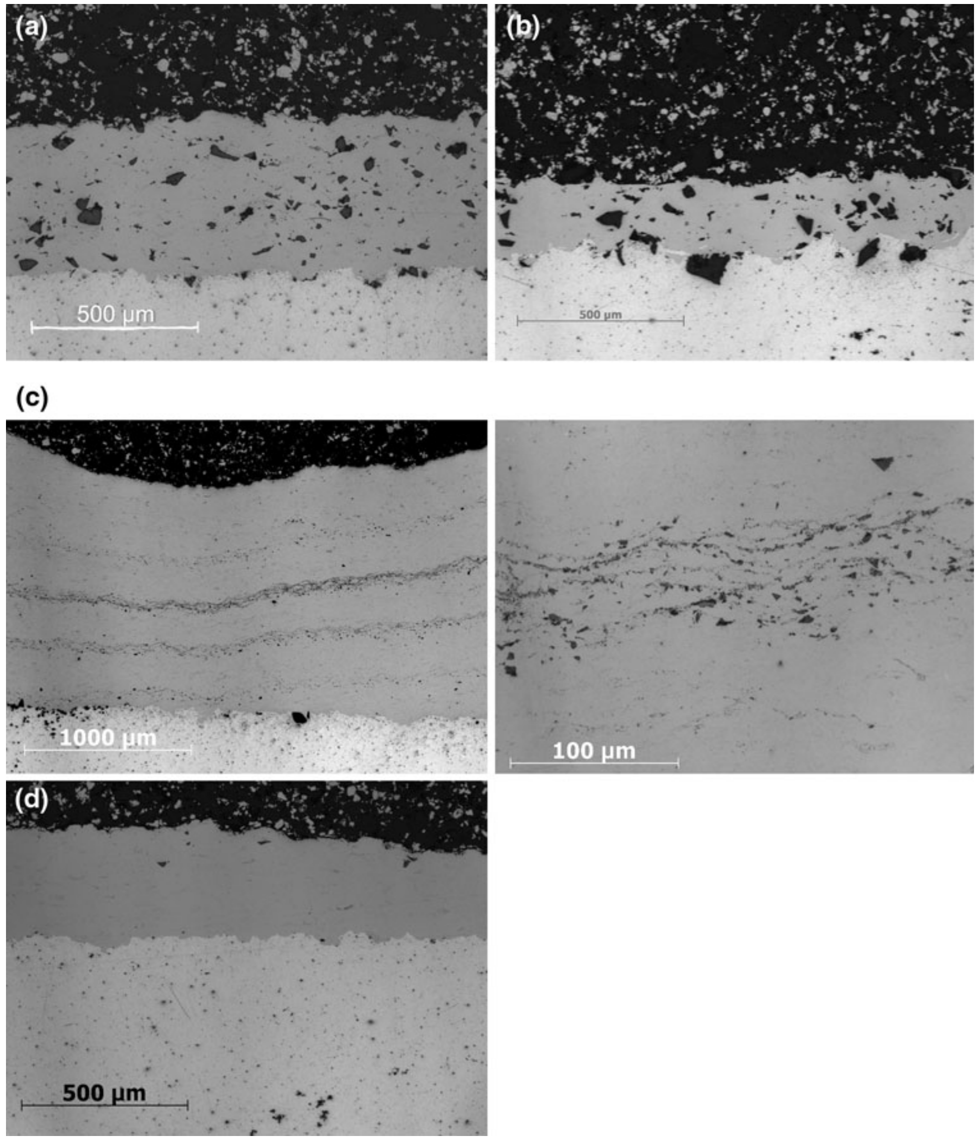

Fig. 2 Optical micrographs of the deposited cermet coatings. (a) Cu + SiC-135, configuration A. (b) Cu + Al_2O_3-127, configuration B. (c) Cu + SiC-25, configuration C, two different magnifications. (d) Cu + SiC-135, configuration C

larger. Some authors suggest that this phenomenon is explained by defragmentation of the large ceramic particles as the result of a high-velocity impact (Ref 13). One can suggest that the velocity of large (more than 100 µm) ceramic particles is not enough for stable penetration into the metal matrix, small (25-35 µm) metal particles cannot ''rivet'' them into the coating, and large ceramic particles just rebound after impact with the obstacle surface.

Injection of ceramics into the free jet of the nozzle significantly decreases the content of ceramic particles in the coating. Microstructures of Cu + SiC-25 and Cu + SiC-135 coatings sprayed at configuration C are presented in Fig. 2(c) and (d) as examples. It is possible to conclude that, in the case of mixtures with fine ceramic particles, the ceramic content does not exceed 5%. Coating porosity was evaluated to be about 1.5-2%. In the case of mixtures with coarse ceramic particles, volumetric content of ceramics is less than 1% (practically indistinguishable against a background of the coating porosity), and all of them are smaller than 50 µm.

Figure 2(c) shows that Cu + SiC-25 coatings have a layerlike structure that is observed in cross sections parallel to the nozzle moving direction as well as in cross sections perpendicular to the nozzle moving direction. Formation of these layers from small ceramic particles could be explained by the unsymmetrical injection of ceramics under configuration C. It is also important to note that the injection ceramic particle jet is smaller than the main supersonic jet. Consequently, one can suggest that ceramic particles injected on the periphery of the free jet did not have enough time to fill all the jet area before impact with substrate. Therefore, concentration of ceramic particles in the jet is not uniform, which explains the formation of such layerlike structure. Besides, it is possible to suggest that the concentration of the ceramic particles in the jet is not uniform because the ceramic particle injection jet only partially penetrates the supersonic jet.

However, it is clear that the ceramic content in the coating depends on the method of ceramic component injection.

3.2 Ceramic Particle Velocity

The ceramic particle velocity before impact was measured for all the spraying configurations; the results are presented in Fig. 3(a) and (b). The curves present the ceramic particle impact velocity versus ceramic particle size. It is clear that velocity of the ceramic particles strongly depends on their size and the location of the injection zone. The difference in velocity of the ceramic particles of the same size injected in the prechamber of the MOC nozzle (configuration A) and in the supersonic part of the ejector nozzle (configuration B) does not exceed 100 m/s. Injection of the ceramic particles in the free jet of the MOC nozzle (configuration C) decreases their impact velocity dramatically. Figure 3 shows that velocity of a ceramic particle larger than 100 μm is less than 200 m/s.

Taking into account these results, it is possible to conclude that, in general, the content of ceramic particles in the coating correlates with their velocity and size.

It should be noted that the metal particle velocities in configurations A and B differed from those in configuration C. Results of PTV measurements show that in the case of MOC nozzle (configurations A and B) mean velocities of aluminum and copper particles were 540 and 515 m/s, respectively, whereas in the case of ITAM ejector nozzle mean velocities of aluminum and copper were 520 and 490 m/s. Therefore, it is necessary to emphasize again that the present experiments permit only evaluation of the basic trends of the influence of ceramic particle velocity on coating formation process.

3.3 Coating Microhardness

Approximately 30 measurements for each sample were performed to calculate the mean value of microhardness. Measurements of the layerlike coatings were carried out in the layers with the maximum ceramic particle concentration. The mean microhardness of the coatings is presented in Fig. 4. The corresponding content of ceramics is also marked on each column of the histograms. Microhardness of pure Al and Cu sprayed by the MOC nozzle and the ejector nozzle was practically the same: 35 $HV_{0.1}$ for aluminum and 65 $HV_{0.1}$ for copper.

It is obvious that microhardness of the deposited coatings depends on the ceramic content. The coatings

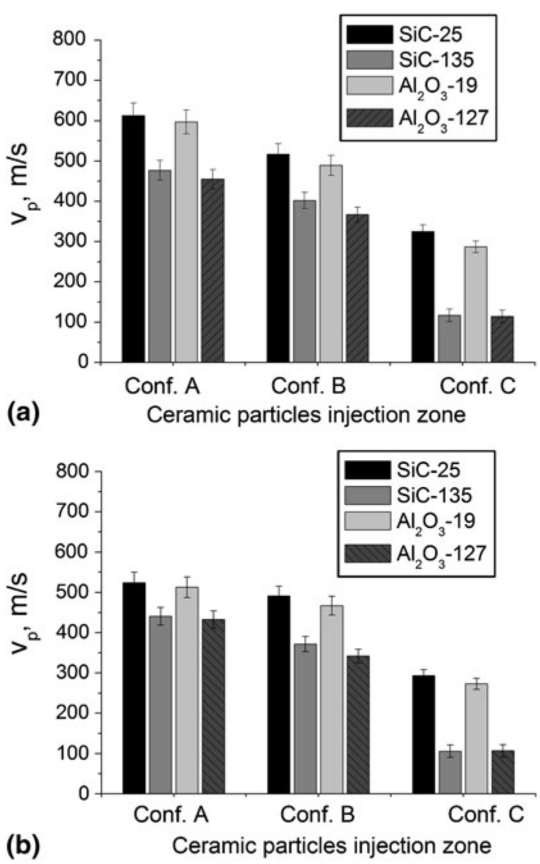

Fig. 3 Measured mean ceramic particle velocity for different spray configurations. (a) Gas stagnation temperature 700 K. (b) Gas stagnation temperature 500 K

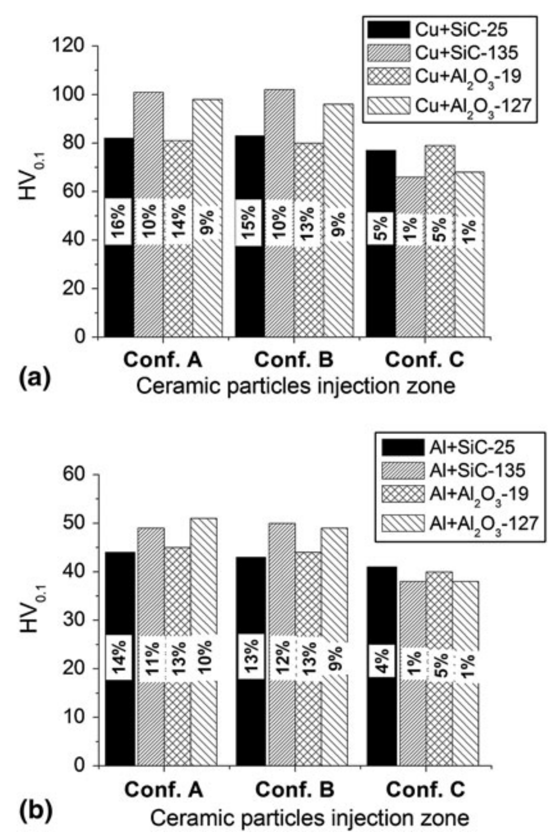

Fig. 4 Microhardness of the coatings sprayed at different configurations. (a) Copper-ceramic, gas stagnation temperature 700 K. (b) Aluminum-ceramic, gas stagnation temperature 500 K

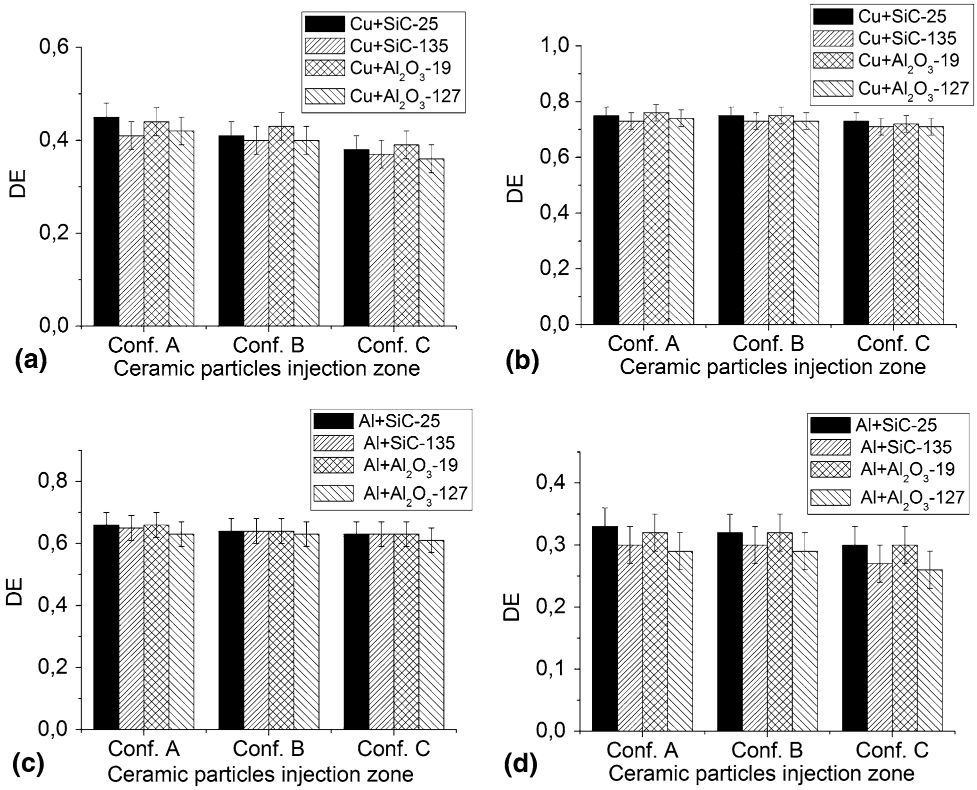

Fig. 5 Deposition efficiency of (a) copper-ceramic mixtures, (b) copper component sprayed in mixture with ceramics, (c) aluminum-ceramic mixtures, and (d) aluminum component sprayed in mixture with ceramics

deposited under configurations A and B have the maximum ceramic content and, as consequence, the maximum microhardness. The coatings deposited under configuration C from mixtures with fine ceramics have almost the same microhardness as those deposited under configurations A and B. Spraying mixtures with coarse ceramics under configuration C allows deposition of coatings with the same microhardness as for pure metal coatings.

It also should be noted that the microhardness of the cermet coatings sprayed with coarse ceramic powders at configurations A and B is slightly higher than microhardness of those sprayed with fine ceramics at a close percentage of the ceramic content.

3.4 Deposition Efficiency

In previous studies, it was indicated that the addition of ceramic particles in a metal powder allows increasing the deposition efficiency of the mixture and the deposition efficiency of the metal component in the mixture (Ref 7, 8). Deposition efficiencies of the sprayed cermet mixtures are presented in Fig. 5(a)-(c). Deposition efficiency (DE) was measured as the coating mass m_c divided by the total mass of powder impinged on the substrate m_p: $DE = m_c/m_p$.

Deposition efficiency of the pure copper powder sprayed by the MOC nozzle and by the ejector nozzle was ~75%; deposition efficiency of the pure aluminum powder was ~65% (MOC nozzle) and ~60% (ejector nozzle).

It is clear from the figure that the deposition efficiencies of the metal-ceramic mixtures are lower than the deposition efficiency of the pure metal. At the same time, deposition efficiencies of the metal component in the mixtures are close to deposition efficiency of the pure copper and aluminum powders. Taking into account the results of the present experiments, it is possible to conclude that, under the given spray conditions, the activation effect of ceramics was not detected.

4. Discussion

Experimental results have shown that velocity of the ceramic particles influences the process of their embedding in the coating during cermet mixture spraying. It was found that ~300 m/s velocity is enough for a certain amount of small SiC and Al_2O_3 particles (10-30 μm) to penetrate into the coating. Further, the metal particles cover the ceramic particles already penetrated into the coating and, therefore, create a metal coating with separate ceramic inclusions. Large ceramic particles (40-70 μm) accelerated to a velocity of 350-500 m/s also penetrate into the coating.

Velocity about 100 m/s is not enough for stable penetration of the large ceramic particles (50-100 μm) and, as a result, the deposited coatings do not have many ceramic inclusions. One can suggest that, for ceramic particles of a given size, there exists a minimum velocity necessary for their penetration into the coating.

It is known that, under certain spraying parameters, the addition of ceramic particles to metal powder permits the increase of the deposition efficiency of the metal from 5 to 10% up to 50 to 60% (Ref 7, 8). In the present study, it was found that the deposition efficiency of the metal component of the mixtures Al + ceramics and Cu + ceramics is practically the same as that of the pure aluminum and copper sprayed at the same conditions (DE about 60-65%). Based on the results obtained, it is possible to conclude that if the spraying parameters are enough to reach high deposition efficiency of the pure metal powder, the addition of ceramic powder does not play a role in deposition efficiency. Activation effect of ceramic powder occurs if the spraying parameters are insufficient for deposition of pure metal with high deposition efficiency. Consequently, the influence of the ceramic particle velocity on the effectiveness of the activation produced by ceramic particles should be explored in future research at low spray parameters, that is, insufficient for effective deposition of metal without stimulation by ceramics.

5. Conclusion

The experiments demonstrated the importance of the ceramic particle velocity for the process of cermet coating formation. Stable cermet coating formation is possible if the ceramic particles are accelerated to a minimum optimal level: ceramic particles accelerated to a high velocity penetrate into the coating, while low-velocity ceramic particles rebound from its surface. It was concluded that, in order to reach the velocity that enables them to penetrate into the substrate, coarse ceramics must be provided with a longer acceleration path than fine ceramics.

Cermet coating microhardness depends on the ceramic content in the coatings and, therefore, depends on the ceramic particle impact velocity. The cermet coatings sprayed from the mixtures with coarse ceramics have a slightly higher microhardness than those sprayed from the mixtures with fine ceramics.

At the tested spray parameters, the influence of the ceramic component on the deposition efficiency of the metal component is negligible: the deposition efficiencies of the pure copper and aluminum compared with the copper/aluminum component of the cermet mixture were practically the same.

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