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Influence of Ceramic Powder Size on Process of Cermet Coating Formation by Cold Spray

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Influence of the ceramic particle size on the process of formation of cermet coatings by cold spray is experimentally studied. A specially developed nozzle with separate injection of ceramic and metal powders into the gas stream is used in the experiments. The results obtained demonstrate that fine ceramic powders (Al_2O_3, SiC) produce a strong activation effect on the process of spraying soft metal (Al, Cu) and increase deposition efficiency of the metal component of the mixture compared to the pure metal spraying. At the same time, coarse ceramic powder produces a strong erosion effect that considerably reduces coating mass growth and deposition efficiency of the metal component. It is experimentally shown that the addition of fine hard powder to soft metals as Al and Cu allows to significantly reduce the "critical" temperature (the minimum gas stagnation temperature at which a nonzero particle deposition is observed) for spraying these metals.

Keywords cermets, cold spray, composite coating, particle size

1. Introduction

Cold spray is a worldwide emerging method of surface engineering and refurbishing for direct manufacturing applications. The principle of the method is that, under certain conditions, 10- to 100-μm-sized particles are capable to fix to the target surface at their impact on it and, thus, to form a coating (Ref 1-5). The mechanism of cold spray is mainly based on plasticity of materials. Therefore, only plastic materials such as metals can form coatings. Deposition of metal particles it still achieved only at certain, so-called critical relationships between particle parameters, namely size, velocity, and temperature. In case of a deviation from the critical parameters, plastic metals do not coat the surface but provoke its erosion as a result of a high-speed impact (Ref 2-5).

Nonplastic materials (ceramics, oxides, etc.) in their pure state produce no coating but erode the surface. However, the possibility to deposit preliminary prepared cermet mixtures is reported in numerous experimental studies. In this case, a metal coating with ceramic inclusions can be formed (Ref 5-9).

Different properties of the cold spray cermet coatings such as adhesion, wear resistance, corrosion resistance, etc., were studied (Ref 5-9). For example, the addition of Al_2O_3 to Al powder is reported to enhance the coating-to-substrate adhesion. Hard ceramic particles create microasperities that favor the bonding of the incoming Al particles and also increase the contact area between the coating and the substrate. 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 8).

It is important to note that the process of coating formation from cermet mixtures essentially differs from that employing pure metal. For example, it is known that the deposition coefficient of cermet mixtures depends strongly on the mass ratio between components and, under certain conditions, can considerably exceed that of pure metals (Ref 6, 8). Currently, this effect is basically explained by the fact that ceramic particles due to a high-speed impact pin and roughen the bombarded surface, thus enhancing the metal particles fixing to it; the number of fixed metal particles augments and, therefore, the deposition coefficient increases. Optimal deposition efficiency was found for a mass fraction of about 30% of alumina in the initial powder (Ref 6, 8).

Despite of advancements in the state of the art of the deposition of cermet coatings, further experimental and theoretical research in this field is required.

The purpose of the present study is to assess the influence of the ceramic particle size on the process of cermet coating formation. To do this, kinetics of coating mass growth and deposition coefficients of mixtures of soft metals (copper and aluminium) with ceramic powders (aluminium oxide and silicon carbide) of various fractions are analyzed.

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Another object of this study is to find "critical" thermal parameters (the minimum gas stagnation temperature at which a nonzero particle deposition is observed) for different cermet mixtures and to compare with "critical" parameters for pure metal. This research could contribute to a better understanding of regularities of cold-spraying cermets.

2. Nozzle Optimization

The cold spray prior art in the field of cermet coatings proposes two methods of powder injection into the gas stream. In the first method, cermet mixture is fed into the subsonic converging part of the nozzle (Ref 4, 10). In this case, the mixture is carried by the gas through the nozzle critical section (throat) and further on through its supersonic part (Fig. 1a). The drawback of this method is that abrasive particles erode the nozzle walls in the throat. It is known that even a small change in the throat area affects strongly the balance of gas-stream parameters leading to their non-uniform transversal distribution and, hence, worsens spraying results. This is why even the smallest erosion of the nozzle throat makes the nozzle unusable.

In the second method, cermet mixture is injected into the supersonic part on the nozzle beyond the critical section (Fig. 1b) (Ref 5-8). In this case, it is possible to avoid erosion of the nozzle walls in the throat. However, this method has another drawback. It is known that for achieving a high deposition coefficient under powder injection in the supersonic part, stagnation temperature of the carrier gas should be much higher than if the same powder was fed into the subsonic part. The reason is that when injected in the supersonic part particles do not pass through the high-temperature subsonic one and therefore their temperature at the nozzle outlet is insufficient for successful spraying. The absence of the zone of intensive heating is then compensated by applying higher stagnation temperatures of the carrier gas in order to heat particles up to the necessary temperature in the supersonic part. Such a method is effective only for easy-sprayable materials like aluminium, zinc, nickel, copper, but complicated for hard-sprayable ones (Ref 5).

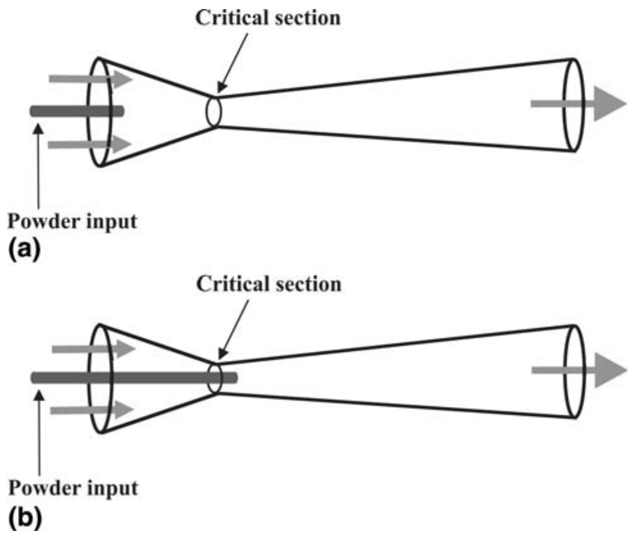

Fig. 1 Nozzles with the different location of the powder injection: (a) the subsonic and (b) supersonic part

An important point to note is that spaying preliminary prepared cermet mixtures does not allow varying the composition ratio within the spraying process and, hence, to spray multilayered and gradient coatings in a single step process.

Taking into consideration the above-mentioned conditions, it seems expedient to separate locations of the metal and ceramic powders injection into the stream. Metal powder should be injected at the most appropriate point in the subsonic part for an effective heating of particles. As for ceramic particles, their temperature is not important for the process and, thus, they can be injected in the supersonic part that will allow to avoid erosion of the nozzle throat.

A specially calculated nozzle was manufactured for experiments with separate injection of the components of cermet mixtures into the gas stream (Ref 11). Its schematic view is shown in Fig. 2.

The key parameters of the nozzle are presented in Table 1. One can see from the figure and the table that metal powder is injected into the converging subsonic area, and the ceramic component into the supersonic one 55 mm beyond the throat.

Some results obtained by using a nozzle with two points of powder injection are presented in Ref 12, 13. Here, it is only emphasized that this type of nozzle can be used for spraying not only metal-ceramic composites but also different metal-metal mixtures.

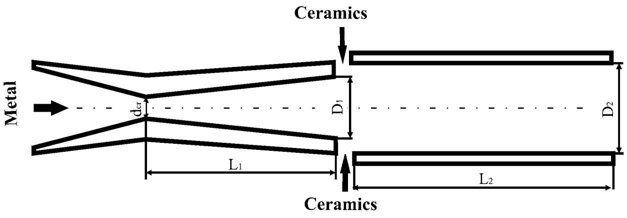

Fig. 2 Nozzle with two points of powder injection into the gas stream

Table 1 Nozzle parameters

Parameter	Value
Diameter of the throat d_{cr}	3 mm
Diameter at the outlet of the diverging part D_1	5.5 mm
Diameter of the barrel D_2	6.5 mm
Length of the diverging part L_1	55 mm
Length of the barrel L_2	70 mm
Exit Mach number M_{exit}	~2.5

3. Experimental

3.1 Equipment

The experiments were performed on cold spray equipment schematically shown in Fig. 3. This equipment allowed varying the gas stagnation pressure from 1.0 up to 2.5 MPa, and the gas stagnation temperature from 300 up to 850 K. The nozzle and the heater were mounted on a robot ABB 4400. Ceramic and metal powders were injected by means of two independent feeders. Powder was fed into the converging subsonic part (powder feeder 1) by a domestic high-pressure powder feeder. A commercial low-pressure powder feeder Oechio (powder feeder 2) was used to feed powder into the supersonic part in the low-pressure area between the diverging part and the barrel with a constant cross section. The powder mass flow rate was calculated by weighing the powder injected into a closed volume after 10, 30, and 60 s time intervals. Nitrogen was used as a career gas with 5 m^3/h flow rate. Particle size analysis was performed by an optical granulomorphometer ALPAGA 500 NANO, which is a real-time optical sieving system, and the CALLISTO image analysis software. The ceramic content in the coatings was evaluated by optical microscope image analysis based on the calculation of the metal and ceramic particles area percentage in coating cross sections (Carl Zeiss Axio Scope microscope with Axiovision 4.7 image analysis software).

3.2 Materials

Aluminium and copper powders were employed as metal components. Ceramic components were two fractions of aluminium oxide and two fractions of silicon carbide. 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, etc. Figures 4-9 represent SEM photos of the employed metal and ceramic powders and their size distribution histograms.

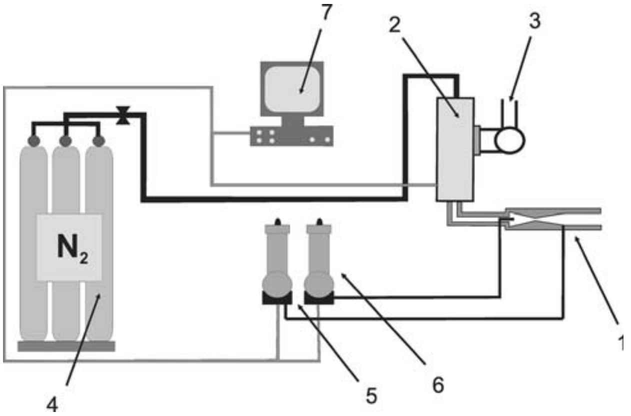

Fig. 3 Schematic view of the experimental device: 1, nozzle; 2, gas heater; 3, robot; 4, carrier gas; 5, powder feeder for the first point of injection; 6, powder feeder for the second point of injection; 7, control panel

The substrate material was aluminium, acetone-processed before spraying.

3.3 Scheme of Experiments

The main objectives of the experiments were:

1. To test a new nozzle design with two points of powder injection with the aim to spray metal-ceramics mixtures.
2. To reveal some regularities of the metal-ceramic coating formation process. In particular, to explore the influence of the ceramic particles size and gas stagnation temperature on the coating growth. Research with high ceramic content mixtures was conducted in order to find out the influence of the ceramic powder erosion on the coating formation process.
3. To find the approximate ''critical'' temperature of the gas flow (the minimum gas stagnation temperature at which a nonzero particle deposition is observed) for different metal-ceramic mixtures and to compare them with the ''critical'' temperature for pure metal.

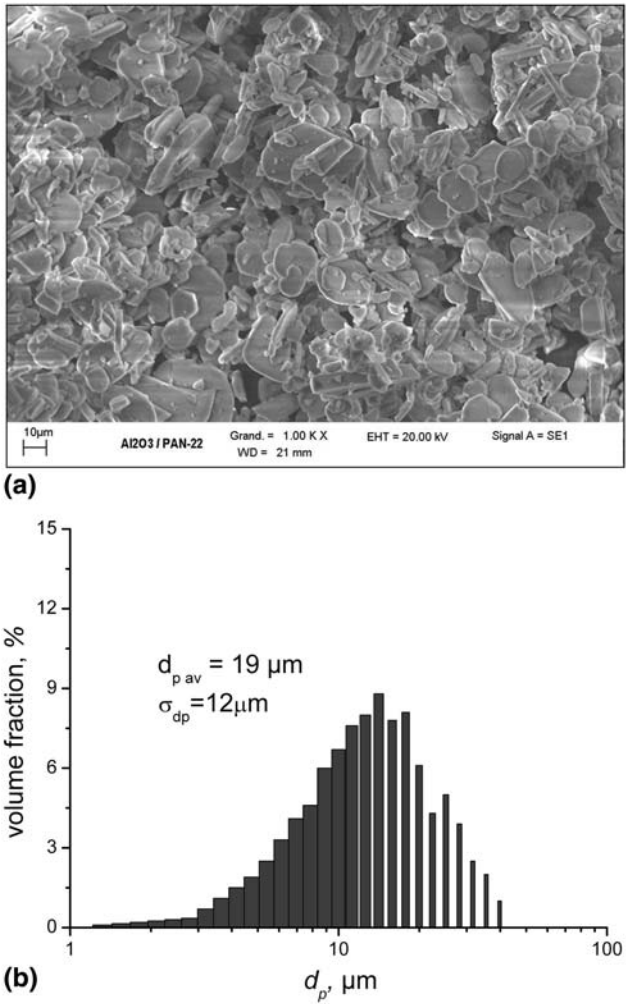

Fig. 4 Al_2O_3-83: SEM photos of powder (a) and powder size distribution (b)

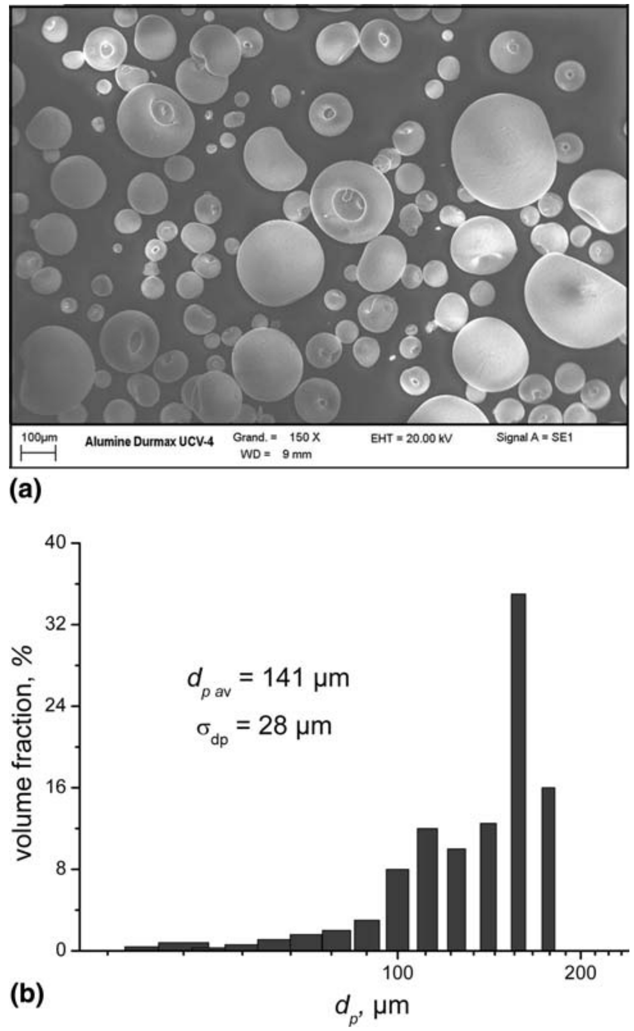

Fig. 5 Al_2O_3-141: SEM photos of powder (a) and powder size distribution (b)

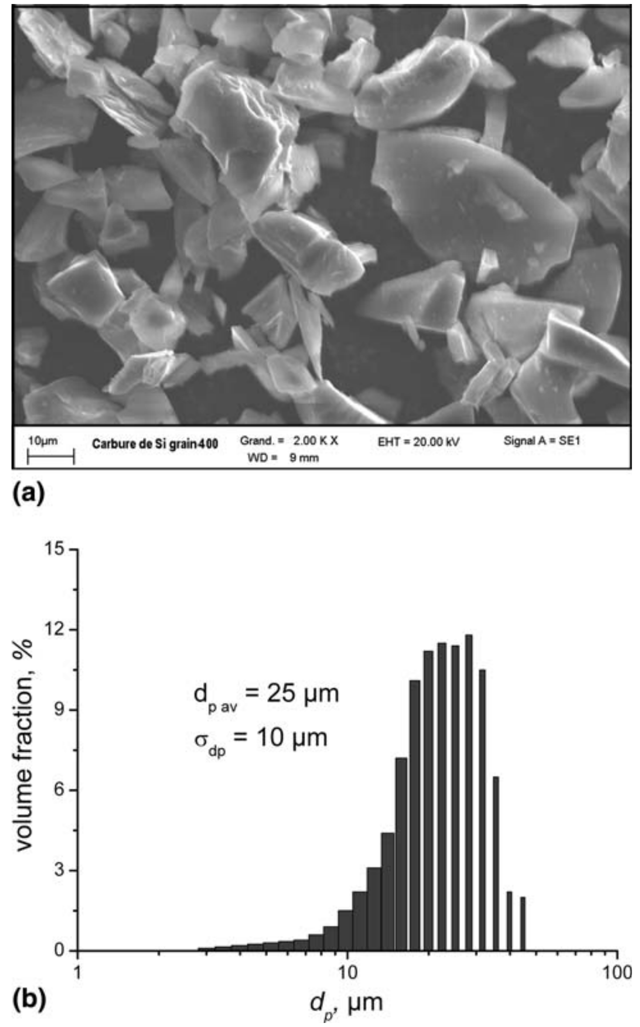

Fig. 6 SiC-25: SEM photos of powder (a) and powder size distribution (b)

The sequence of experiments was as follows. First, aluminium powder was injected at the first point with 0.1 g/s feed rate. The spraying angle was 90°, the nozzle speed was 7 mm/s, and the spraying time was 35 s. This series of experiments was carried out for four values of gas stagnation temperature: 373, 423, 473, and 503 K. The gas pressure was fixed at 1.6 MPa. Four samples with aluminium coating were obtained. Further on, the same parameters were used in a series of experiments with simultaneous injection of aluminium powder with 0.1 g/s feed rate at the first point and ceramic powder at the second point. Four types of ceramics were sprayed at feed rate 0.5 g/s. Thus, the mass composition of the sprayed mixtures were 16.5%Al-83.5% ceramics. Overall four types of aluminium/ceramic mixtures were sprayed. At last, the ''critical'' temperatures for pure Al and mixtures were determined.

The same scheme was used to spray pure copper and copper/ceramics mixtures. Copper was injected at 0.3 g/s feed rate. The gas stagnation temperatures were 473, 573, 643, and 698 K, and the gas pressure was 1.6 MPa. The ceramics feed rate was also 0.5 g/s yielding the mass compositions of 37.5%Cu-62.5% ceramics, respectively. Overall four types of copper/ceramic mixtures were sprayed. At last, the ''critical'' temperatures for copper and mixtures also were determined.

Ten types of powders employed in the experiments (pure copper, pure aluminium and eight types of mixtures) are listed and classified in Table 2.

4. Experimental Results

The coating mass dm could be measured experimentally as $dm = m_{\text{after}} - m_{\text{before}}$ where m_{before} and m_{after} are the substrate mass before and after spraying, respectively. In the case of spraying pure metals, the deposition efficiency $k_{\text{eff pure metal}}$ is evaluated as the coating mass dm divided by the total mass of powder impinged on the

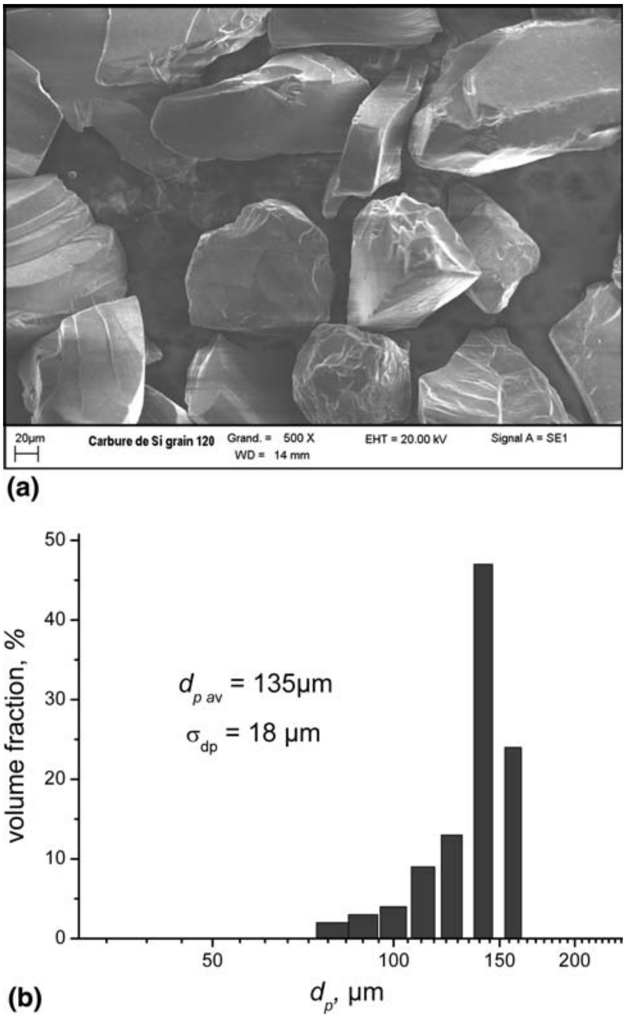

Fig. 7 SiC-135: SEM photos of powder (a) and powder size distribution (b)

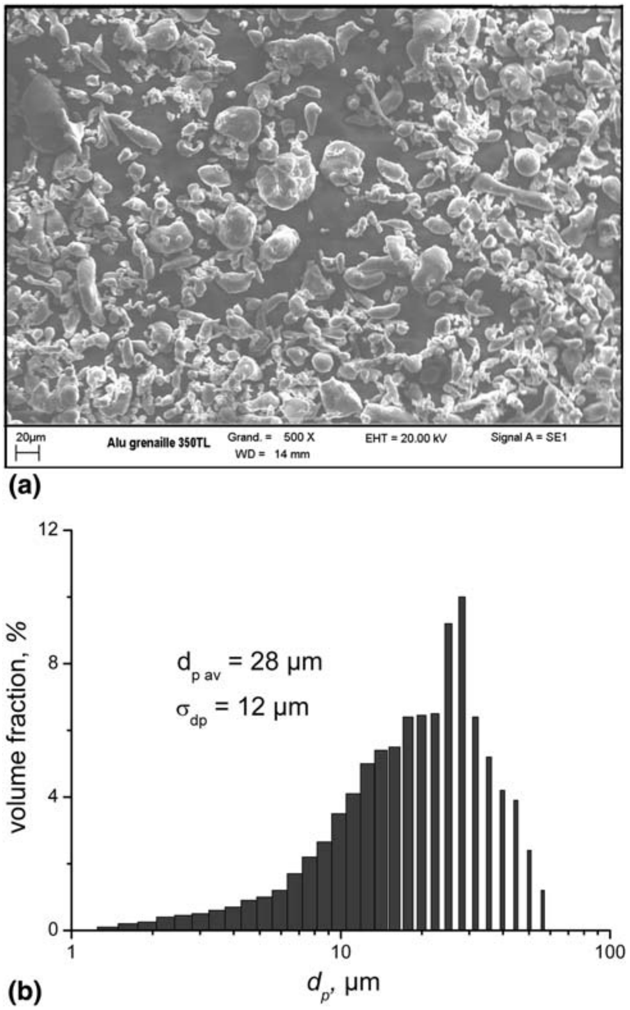

Fig. 8 Al: SEM photos of powder (a) and powder size distribution (b)

substrate m_{powder}. In the case of spraying mixtures, the overall deposition efficiency can be written as

$$k_{\text{eff mix}} = \frac{dm}{m_{\text{powder}}} = \frac{dm_{\text{metal component}} + dm_{\text{ceramics component}} - dm_{\text{erosion}}}{m_{\text{metal powder}} + m_{\text{ceramics powder}}}$$

where $dm_{\text{metal component}}$ and $dm_{\text{ceramic component}}$ are the coating mass growth resulting from the metal particle and ceramic particle deposition, respectively, dm_{erosion} is the coating mass loss because of the abrasive-induced erosion, $m_{\text{metal powder}}$ and $m_{\text{ceramics powder}}$ are the mass of all metal powder and all ceramic powder impinged on the substrate, respectively. Deposition efficiency of the metal component in the mixture can be calculated by the formula:

$$k_{\text{eff metal component}} = \frac{dm_{\text{metal component}} - dm_{\text{erosion}}}{m_{\text{metal powder}}}$$

The graphs in Fig. 10 and 11 represent the coating mass growth dm and the overall deposition coefficient $k_{\text{eff mix}}$ for aluminium/ceramic mixtures and $k_{\text{eff pure metal}}$ for pure aluminium versus gas stagnation temperature.

As can be seen from the plot, dm for mixtures 1 (Al + SiC-25) and 3 (Al + Al_2O_3-19) reaches its maxima and essentially exceeds dm obtained for pure metal over all the gas stagnation temperature range. The overall deposition coefficient of these mixtures is higher than that of pure metal at small gas stagnation temperatures; however, the latter grows quickly with the gas stagnation temperature and, finally, exceeds the overall deposition coefficient of mixtures 1 (Al + SiC-25) and 3 (Al + Al_2O_3-19).

Coating mass growth dm for mixture 2 (Al + SiC-135) is also above that of pure metal. However, the overall deposition coefficient is lower for these mixture compared to pure aluminium all over the gas stagnation temperature range. In the case of mixture 4 (Al + Al_2O_3-141), both coating mass growth and deposition coefficient are considerably lower than in the case of pure aluminium.

Deposition efficiency of pure aluminium exceeds the overall deposition efficiency of the mixtures at gas stagnation temperatures 473 and 503 K. However, taking into account the results of the coating mass growth, it is interesting to compare the deposition efficiency of pure aluminium and the deposition efficiency of the aluminium component in the mixture $k_{\text{eff metal component}}$.

The ceramic content in the aluminium-ceramics coatings sprayed at 473 and 503 K was evaluated by analyzing the coating image. It is found that the ceramic mass content in the sprayed coatings lays in the range 12-18% for mixtures 1 (Al + SiC-25) and 3 (Al + Al_2O_3-19), and

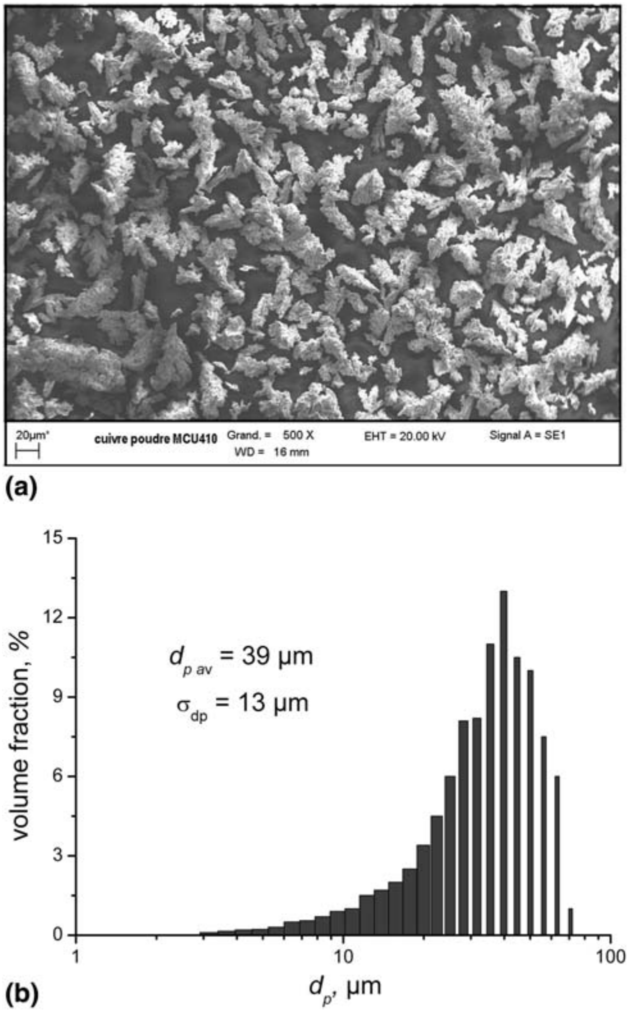

Fig. 9 Cu: SEM photos of powder (a) and powder size distribution (b)

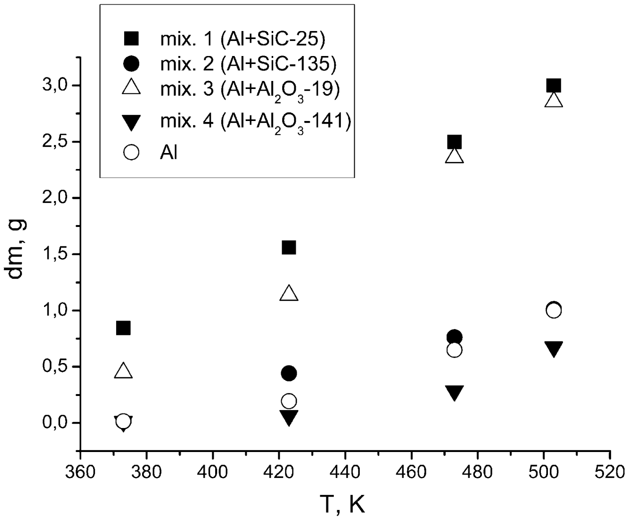

Fig. 10 Coating mass growth *dm* vs. gas stagnation temperature *T* for aluminium and aluminum/ceramic mixtures 1-4

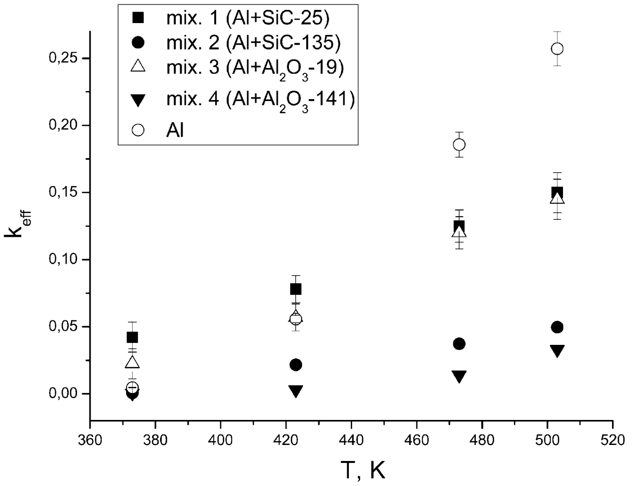

Fig. 11 Overall deposition efficiency coefficient k_{eff} vs. gas stagnation temperature *T* for aluminum and aluminum/ceramic mixtures 1-4

Table 2 The powders employed in the experiments

Name of powder	Types and feeding rate of components	Obtained mass composition
Pure Al	0.1 g/s Al	100% Al
Pure Cu	0.3 g/s Cu	100% Cu
Mixture 1	0.1 g/s Al + 0.5 g/s SiC-25	16.5%Al-83.5%SiC-25
Mixture 2	0.1 g/s Al + 0.5 g/s SiC-135	16.5%Al-83.5%SiC-135
Mixture 3	0.1 g/s Al + 0.5 g/s Al_2O_3-19	16.5%Al-83.5%Al_2O_3-19
Mixture 4	0.1 g/s Al + 0.5 g/s Al_2O_3-141	16.5%Al-83.5%Al_2O_3-141
Mixture 5	0.3 g/s Cu + 0.5 g/s SiC-25	37.5%Cu-62.5%SiC-25
Mixture 6	0.3 g/s Cu + 0.5 g/s SiC-135	37.5%Cu-62.5%SiC-135
Mixture 7	0.3 g/s Cu + 0.5 g/s Al_2O_3-19	37.5%Cu-62.5%Al_2O_3-19
Mixture 8	0.3 g/s Cu + 0.5 g/s Al_2O_3-141	37.5%Cu-62.5%Al_2O_3-141

10-15% for mixtures 2 (Al + SiC-135) and 4 (Al + Al_2O_3-141). Similar results were observed in Ref 8. It should be noted that large (more than 50-70 µm) ceramic particles are not present in the coating produced from coarse powders containing particles up to 100 µm and larger. Figure 12 shows typical microstructures of composite coatings obtained from mixtures with coarse ceramic powders. Taking into account the ceramic mass content in the coatings, the deposition efficiency of the metal component in the mixtures $k_{\text{eff metal component}}$ were calculated. The graph in Fig. 13 represents $k_{\text{eff metal component}}$ versus gas stagnation temperature for mixtures 1-4 and pure Al. As can be seen from the plot, the deposition efficiency of the metal component $k_{\text{eff metal component}}$ in the mixtures with fine ceramics is much greater than that of pure aluminum. On the contrary, the deposition efficiency of the metal component $k_{\text{eff metal component}}$ in the mixtures with coarse ceramics is smaller than that of pure metal.

The graphs in Fig. 14 and 15 represent coating mass growth and deposition coefficient versus gas stagnation temperature under spraying pure copper and copper/ceramic mixtures.

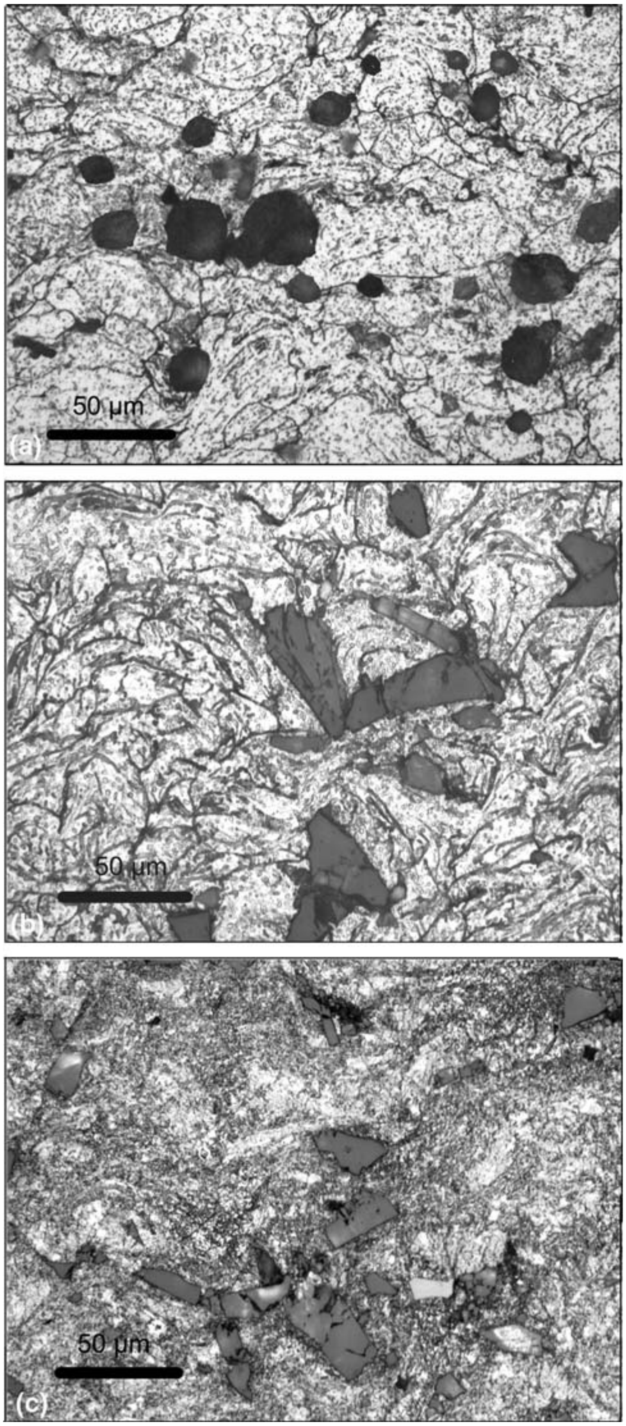

Fig. 12 Optic microscope images of sprayed mixtures: (a) mixture 4 (Al + Al_2O_3-141); (b) mixture 2 (Al + SiC-135); (c) mixture 6 (Cu + SiC-135)

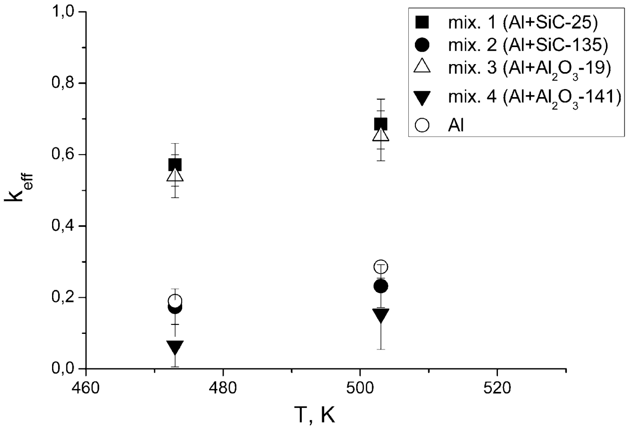

Fig. 13 Deposition efficiency of metal component $k_{\text{eff metal component}}$ vs. gas stagnation temperature T for aluminum and aluminum/ceramic mixtures 1-4

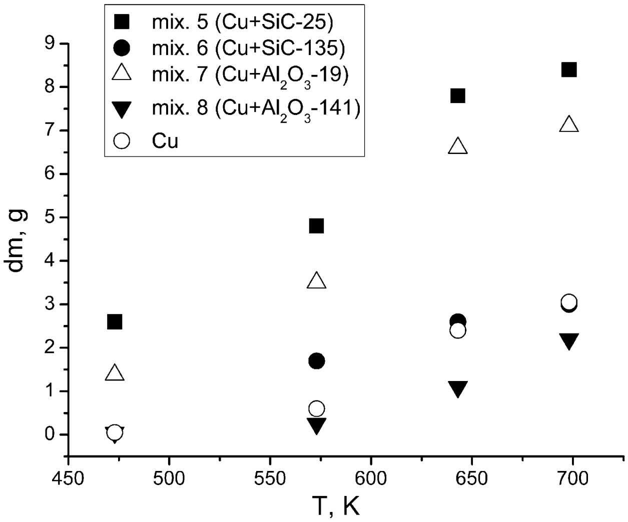

Fig. 14 Coating mass growth dm vs. gas stagnation temperature T for copper and copper/ceramic mixtures 5-8

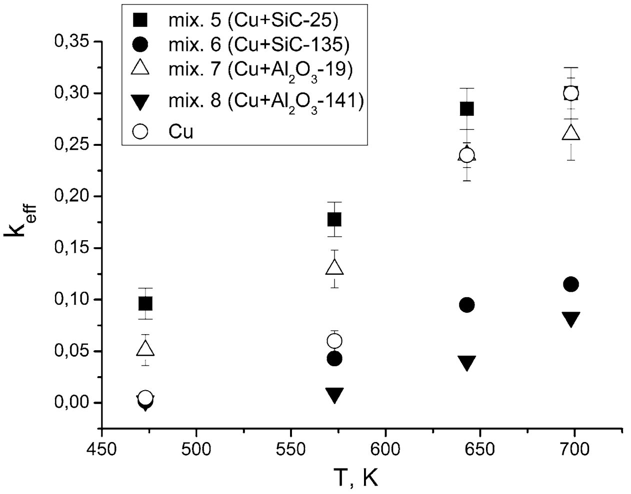

Fig. 15 Overall deposition efficiency coefficient k_{eff} vs. gas stagnation temperature T for copper and copper/ceramic mixtures 5-8

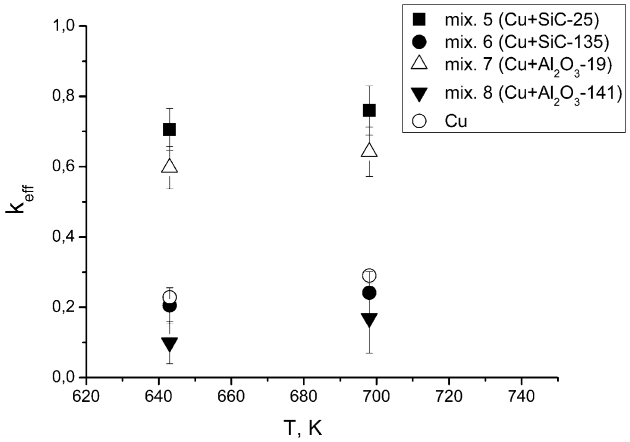

Fig. 16 Deposition efficiency of metal component $k_{eff\ metal\ component}$ vs. gas stagnation temperature T for copper and copper/ceramic mixtures 5-8

In this case, similarly to the experiments with aluminium, *dm* is maximal for mixtures of metal with fine fractions of ceramic particles, namely mixtures 5 (Cu + SiC-25) and 7 (Cu + Al_2O_3-19). The graphs show as well that *dm* is considerably lower for mixtures with larger ceramic particles compared to mixtures with fine ceramics, and *dm* and $k_{eff\ mix}$ are the lowest for mixtures with aluminium oxide Al_2O_3-141 (mixture 8).

The ceramic content in the copper-ceramics coatings sprayed at 643 and 698 K was evaluated. It is found that the ceramic mass content in the sprayed coatings lays in the range 12-18% for mixtures 5 and 7, and 10-15% for 6 and 8. The graph in Fig. 16 represents $k_{eff\ metal\ component}$ versus gas stagnation temperature for mixtures 5-8 and pure Cu. As can be seen from the figure, the deposition efficiency of the copper component $k_{eff\ metal\ component}$ in the mixtures strongly depends on the ceramic particles size.

Table 3 The "critical" temperatures of sprayed mixtures

Composite	T_{cr1} for pure metal, K	T_{cr2} for mixture, K	ΔT, K
Al + fine Al_2O_3 or SiC (mixtures 1 and 3)	~420	~360	~60
Al + coarse Al_2O_3 or SiC (mixtures 2 and 4)	~420	~420	~0
Cu + fine $Al_2\ O_3$ or SiC (mixtures 5 and 7)	~520	~420	~100
Cu + coarse $Al_2\ O_3$ or SiC (mixtures 6 and 8)	~520	~520	~0

"Critical" temperatures experimentally established for the gas flow are presented in Table 3. It was found that metal mixtures with fine ceramics have lower values of "critical" temperature than pure metals. Mixtures with coarse ceramics have approximately the same values of "critical" temperature as pure metals.

5. Discussion

Taking into account the above results, one can conclude that the addition of fine ceramic powder in metal powder significantly increases the deposition efficiency of the metal component in the mixture $k_{eff\ metal\ component}$ compared to the deposition efficiency of pure metal $k_{eff\ pure\ metal}$. At the same time, the overall deposition efficiency coefficient of this mixture $k_{eff\ mix}$ can be much lower than the deposition efficiency coefficient of pure metal. Thus, fine ceramic particles act as "catalysts" that increases the coating mass due to an increase of the volume of adhered metal. Along with this, the "catalyst" itself remains in the coating in rather small amounts that strongly reduces the overall deposition coefficient of the mixture $k_{eff\ mix}$. The nature of such "catalytic" effect of ceramics might be related to roughening, cleaning, and mechanoactivating of the surface resulting from the high-speed impact of ceramic particles. These results are consistent with previous studies (Ref 6, 8).

A very important conclusion to be drawn from the experimental results is that, because of the activation effect, the addition of fine hard powder (Al_2O_3, SiC) to soft metals such as Al and Cu allows to significantly reduce "critical" temperature T_{cr} for these metals (60-100 K). The effect of decrease in T_{cr} observed in spraying metals with a hard phase can be important from the practical point of view and should be taken into account!

A crucial point to note is that the above considerations on the strong activation effect of the ceramic additive are true only for mixtures with fine ceramics. Experiments on deposition of mixtures of metals with large ceramics and fine ceramics gave greatly different results. In mixtures of metals with SiC-135, activation is insignificant over all the gas stagnation temperature range. In the case of spraying mixtures of metals with Al_2O_3-141 powder, a negative effect of the ceramic additive on the coating growth was observed at any gas stagnation temperature, that is, *dm* of these mixtures was lower compared to pure metal. Let us try to explain the reason of such a significant difference between the results of deposition of mixtures of metals with large and fine fractions of ceramic powders, and to find out why the activation effect is absent in the case of the presence of large ceramic particles.

It is obvious that the cold spray deposition of metals and especially mixtures of metals with any ceramics involves the competition of two antagonistic processes: coating formation and surface erosion (Ref 13). On the one hand, a ceramic additive increases the coating mass due to its activation effect on metal particles described above, but, on the other hand, reduces the coating mass due to erosion and its knocking out material at high-speed impact.

It was experimentally demonstrated that fine ceramic particles increases the value of $dm_{\text{metal component}}$ due to the activation effect. But the erosion effect of fine ceramic particles is evidently not too strong. It may be explained by the fact that erosion is mainly a volumetric effect, and its value is proportional to the mass (volume) of the particle. At the same time, the mass (volume) of a particle is proportional to r^3. Accordingly, the erosion effect strengthens sharply with the size of ceramic particles and begins to increase the value of dm_{erosion} and, finally, to decrease dm.

It is possible to suggest that the substrate relief induced by the high-speed impact of fine ceramic particles favors the metal particle deposition as it contains a great number of micropeaks.

Another point of importance is that ceramics used in the experiments had a large enough particle size distribution. For example, Al_2O_3-141 powder contained a rather great number of fine particles which can produce an activation effect and, to some extent, smooth erosion caused by the large particles.

Besides, the question of what the essential difference between the results of spraying mixtures 4 and 8 containing Al_2O_3-141 and mixtures 2 and 6 containing SiC-135 remains open. Probably, more intensive erosion in the case of Al_2O_3 is connected to the fact that particles having various shapes have various impact speeds. In any case, these questions require further research.

6. Conclusions

The proposed nozzle with separate injection of components into the gas stream was experimentally validated for cermet coatings. The focus of further research should be on optimization of the nozzle design in terms of gas dynamics that would offer a considerable advance in understanding the process of the cermet coating formation and allow to improve spraying technique.

It is experimentally shown that when spraying soft metals (Al, Cu) with fine ceramic powders the "critical" temperature of the metals decreases compared to pure metals.

The experiments demonstrated the importance of the ceramic particle size for the process of cermet coating formation. Ceramic powders of fine fractions produce a strong activation effect on the spraying process and enhance the coating growth, while ceramic powders of large fractions considerably hinder coating growth.

The complexity of metal-ceramic mixture spraying is that not only spraying parameters of metal must be taken into account but those of ceramics as well, namely type of ceramics, particle size, shape, feed rate, velocity and temperature, etc. This deserves further experimental and theoretical research.

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