JTTEE5 2:179-186

Optimizing the Vacuum Plasma Spray Deposition of Metal, Ceramic, and Cermet Coatings Using Designed Experiments

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The vacuum plasma spray (VPS) deposition of metal, ceramic, and cermet coatings has been investigated using designed statistical experiments. Processing conditions that were considered likely to have a significant influence on the melting characteristics of the precursor powders and hence deposition efficiency were incorporated into full and fractional factorial experimental designs. The processing of an alumina powder was very sensitive to variations in the deposition conditions, particularly the injection velocity of the powder into the plasma flame, the plasma gas composition, and the power supplied to the gun. Using a combination of full and fractional factorial experimental designs, it was possible to rapidly identify the important spraying variables and adjust these to produce a deposition efficiency approaching 80%. The deposition of a nickel-base alloy metal powder was less sensitive to processing conditions. Generally, however, a high degree of particle melting was achieved for a wide range of spray conditions. Preliminary experiments performed using a tungsten carbide/cobalt cermet powder indicated that spray efficiency was not sensitive to deposition conditions. However, microstructural analysis revealed considerable variations in the degree of tungsten carbide dissolution. The structure and properties of the optimized coatings produced in the factorial experiments are also discussed.

1. Introduction

OPTIMIZATION of vacuum plasma spraying requires the ability to identify parameters that have a significant influence on the structure and properties developed within a deposit and to establish the combination of parameter settings that yield the maximum improvement in the characteristics desired of the coating or free-standing artefact. The development of a plasma sprayed coating depends on many parameters. The effect of these parameters and the complex interactions among them are not sufficiently understood to allow "optimum" conditions to be derived without some form of empirical evaluation of the process. The classical one-factor-at-a-time approach, which is often used to investigate the effect of process parameters in relatively simple processes,[1] is not adequate for analysis of the VPS process. This is because a prohibitively large number of spray trials would be required, and it would be impossible to quantify the effect of the complex parameter interactions.

Many of the limitations of the one-factor-at-a-time approach can be overcome by using statistical experimental design methods. There are a number of techniques based on statistical principles that are capable of identifying the effect of process parameters and their interactions; these include Taguchi methods,[2] evolutionary operation,[3] central composite designed experiments,[4] and full and fractional factorial experiments.[5] The effectiveness of each technique depends on the objectives of the experiment (i.e., higher efficiency or reduced variability), the availability of pre-existing knowledge of the process, and on the environment under which the information must be obtained (e.g., production or development).

Taguchi methods[2] use highly saturated experiments (i.e., only a limited amount of information can be obtained on preselected interaction effects). Therefore, they are more suited to the analysis of processes in which the effect of parameters are already partially quantified and for which the important interaction effects are known, for example the optimization of a process in a production environment. Major advantages of using Taguchi methods include the ability to investigate the effects

Keywords: design of experiments, spray efficiency (deposition efficiency), statistical experiments, Taguchi methods, vacuum plasma spray

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that controllable parameters have on the variability of a process and the possibility of establishing parameters that reduce this variability (robust product design), again more relevant to a production process. Evolutionary operation (EVOP)[3] is another method of investigating the effects of process parameters in a production environment. Like Taguchi methods, it requires some background knowledge of the important process parameters and interactions. However, EVOP involves performing a series of experiments in sequence and thus is more flexible than the single-design approach of Taguchi. The levels of parameters investigated using EVOP are close to the standard operating parameters, and only small adjustments are made after each cycle of experiments. Thus, although the optimization process may involve a reduction in performance in certain trials, the variations are small enough to allow the production process to continue. The analysis of processes that are operated in a research environment do not involve the same constraints as the investigation of a production process. Usually, a wider variation in the process conditions can be incorporated into an experiment because it is not important that a certain level of quality be maintained. In addition, the researcher is often more interested in the effects of parameters and in identifying optimum conditions than in analysis of variability. Therefore, experiments based on the use of factorial designs are one of the most efficient methods of investigating these processes.[5]

Two-level full and fractional factorial experiments have been used to investigate the melting behavior of metal, ceramic, and cermet powders during vacuum plasma spraying. Spray efficiency (also called deposition efficiency) was chosen as the response (that is, the property to be optimized) because it is closely related to particle melting, which influences interparticle cohesion and porosity and hence has a major effect on the strength, corrosion behavior, and wear properties of a coating.

2. Experimental

Plasma spraying was performed using an industrial VPS system (Plasma-Technik Ltd., Switzerland). Powder was supplied by a Twin 10-V powder feed unit to an F4V gun attached to a five-axis robot. Metal and cermet coatings were deposited onto grit-blasted mild steel (BS 970,070M20), and alumina coatings were deposited onto commercial-purity copper substrates. The effect of substrate material was not investigated; therefore, the results are representative of a particular substrate/coating composite. A surface area of 2500 mm^2 was coated to reduce the errors introduced by edge effects and thus allow the spray efficiency to be calculated with sufficient accuracy. Efficiency was calculated from the rate of powder delivery to the gun, the mass of coating applied to a sample, and the impingement time of the spray stream on the substrate. The latter was determined from the gun velocity and the geometry of the raster motion of the gun over the sample.

The optimization process involved analysis of parameters that were considered likely to have a significant influence on particle melting, but excluded those that involved major equipment modifications. The quality of the precursor powder was not considered. The parameters that have a significant influence on the plasma spray process are well documented and include (1) flow rates of the plasma forming gases, which affect the temperature and velocity profile of the plasma flame;[6] (2) powder carrier gas flow rate, which controls the velocity and trajectory of the particles in the plasma flame;[7] (3) spray distance, which affects the dwell time of the particle in the plasma;[7] (4) gun current, which affects the heat input into the flame;[8] and (5) chamber pressure, which controls the velocity and temperature profile of the flame[9] and indirectly controls the substrate temperature.

The parameters above were investigated using a combination of full and fractional factorial experimental designs. If the results from an initial series of trials indicated that parameter effects were significant and the spray efficiencies were low, then a second experiment was constructed with parameter levels adjusted in the direction suggested by the analysis of the first factorial experiment.

The results from the full and fractional factorial experiments were analyzed using a commercial statistical analysis package (Statgraphics, Statistical Graphics Corporation, USA). The effects determined from the experiments were plotted on normal probability paper in ascending order. In such plots, effects that comprise only random variations should lie on an approximately straight line passing through 50 cumulative percent when the effect = 0. Effects that are significant will not conform to this linearity. Points lying on a straight line were used to estimate the residual standard deviation, RSD, using the relationship:

$$\text{RSD} = \sqrt{\frac{n}{4}\,\frac{\Sigma(\text{relevant effects estimates})^2}{\text{number of effects}}} \quad [1]$$

where n is the total number of test runs.

This RSD value was then used to obtain 95% confidence intervals. All factor effects larger than this interval were considered significant. The confidence interval was given by:

$$95\% \text{ confidence interval} = \pm\frac{\text{RSD} \times 2 \times t}{\sqrt{n}} \quad [2]$$

where t is the Student t-value for a confidence interval of 95% for a double-sided significance test with n degrees of freedom.

3. VPS Processing of Alumina

3.1 *Initial Factorial*

The processing of alumina by VPS was investigated using UA 500# alumina (Universal Abrasives Ltd., England). This powder had a nominal particle size range of 5 to 25 μm and a morphology typical of a fused and crushed material. In the initial factorial experiment, it was important to investigate as many of the major processing variables as possible. Six factors were identified—gun current, flow rates of the plasma gases supplied to the gun (argon and hydrogen), flow rate of the powder carrier gas, chamber pressure, and spray distance.

A full factorial experiment with 6 factors requires 64 spray trials. It was impractical to produce such a large number, and therefore, the experiment was reduced to 16 trials using a quarter 2^6 fractional factorial design. The defining equation chosen was

$$I = ABCE = BCDF = ADEF \quad [3]$$

where *I*, the constant effect, is the overall average response calculated from the 16 spray trials, and *A* to *F* are the processing factors listed in Table 1.

The design constructed using this defining equation has all of the main effects confounded, with only high-order interactions involving three or more factors. This is important, because high-order interactions are generally negligible, and thus, the effects estimates calculated can be attributed solely to the main effects.

The high and low levels of the six factors investigated were chosen to give a wide variation in each parameter, as this increases the likelihood of distinguishing significant effects from the variability arising from random errors. The levels chosen are summarized in Table 1. The design matrix constructed using the defining equation given in Eq 3 is described in Table 2, with the spray efficiencies calculated from the weight gain measurements. Spray efficiencies ranging from 6.9 to 45.2% were obtained, with a mean of 22%. The effects estimates calculated from the efficiencies are listed in Table 2 and the aliased factors are listed in Table 3.

A normal probability plot of these effects is shown in Fig. 1. The constant effect, *I*, is an average of all the trials rather than the effect of changing a factor or a combination of factors between two values and therefore has not been included in Fig. 1. The points lying on or close to a straight line were assumed to be due to random process fluctuations and measurement errors. Therefore, the residual standard deviation was calculated using these data; a value of 6.0 was obtained. Using this value in Eq 2, with 12 degrees of freedom yielded a confidence interval of ±3.3. Effects greater than this can be considered significant, i.e., *A*, *B*, and *F*, as shown in Fig. 2. This check also confirmed that the remaining factors and interactions were insignificant.

Chamber pressure, factor *E*, was not found to be significant, but its effect was close to the 95% confidence interval of ±3.3; therefore, this factor was included in the second factorial design. All of the aliased effects that contained only interaction effects and no main effects were found to be insignificant. Therefore, it was assumed that high-order interactions (those involving three or more factors) were also insignificant. This is an important assumption because if high-order interactions are significant then the effects estimates could not be attributed to the main factors alone, and additional experiments, e.g., full factorial experiments, would be required.

3.2 *Second Factorial*

The sign of the significant effects estimates obtained from the initial factorial experiment indicated that the direction these

Table 1 Levels of factors investigated in initial experiment with Universal Abrasives 500# alumina powder

Factors	Levels
A, gun current	600 and 750 A
B, argon plasma gas flow rate	40 and 50 SLPM
C, hydrogen plasma gas flow rate	8 and 12 SLPM
D, spray distance	220 and 320 mm
E, chamber pressure	90 and 140 mbar
F, carrier gas flow rate	2 and 3 SLPM

Note: SLPM ≡ standard liters per minute.

Table 2 Quarter 2^6 fractional factorial design and efficiency results obtained for Universal Abrasives 500# alumina

Trial No.	Factor *A*	*B*	*C*	*D*	*E*	*F*	Spray efficiency, %
1	–	–	–	–	–	–	12.0
2	+	–	–	–	+	–	26.4
3	–	+	–	–	+	+	19.4
4	+	+	–	–	–	+	22.9
5	–	–	+	–	+	+	38.0
6	+	–	+	–	–	+	45.2
7	–	+	+	–	–	–	8.5
8	+	+	+	–	+	–	18.1
9	–	–	–	+	–	+	16.9
10	+	–	–	+	+	+	44.8
11	–	+	–	+	+	–	6.9
12	+	+	–	+	–	–	10.4
13	–	–	+	+	+	–	15.3
14	+	–	+	+	–	–	20.2
15	–	+	+	+	–	+	14.8
16	+	+	+	+	+	+	33.1

Table 3 Aliased factors and effects estimates calculated from data in Table 2 for Universal Abrasives 500# alumina

Aliased effects	Effects estimates
Average (*I*) + *ABCE* + *BCDF* + *ADEF*	22.1
A + *BCE* + *ABCDF* + *DEF*	11.2
B + *ACE* + *CDF* + *ABDEF*	–10.6
AB + *CE* + *ACDF* + *BDEF*	–2.4
C + *ABE* + *BDF* + *ACDEF*	4.2
AC + *BE* + *ABDF* + *CDEF*	–1.2
BC + *AE* + *DF* + *ABCDEF*	–0.5
E + *ABC* + *ADF* + *BCDEF*	6.4
D + *ABCDE* + BCF + *AEF*	–3.5
AD + *BCDE* + *ABCF* + *EF*	2.5
BD + *ACDE* + *CF* + *ABEF*	2.5
ABD + *CDE* + *ACF* + *BEF*	–0.3
CD + *ABDE* + *BF* + *ACEF*	–3.1
ACD + *BDE* + *ABF* + *CEF*	–0.9
F + *BCD* + *ADE* + *ABCEF*	14.7
ABCD + *DE* + *AF* + *BCEF*	3.1

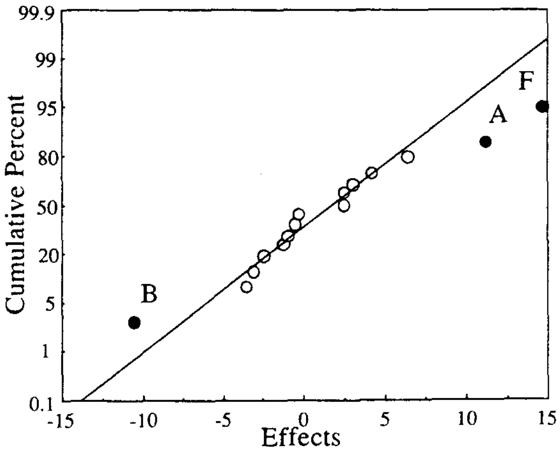

Fig. 1 Normal probability plot of the effects estimates listed in Table 3 for Universal abrasives 500# alumina powder. Closed circles represent significant factors, i.e., those greater than the 95% confidence interval of ±6.6. *A*, gun current; *B*, flow rate of argon plasma gas; and *F*, flow rate of the powder carrier gas.

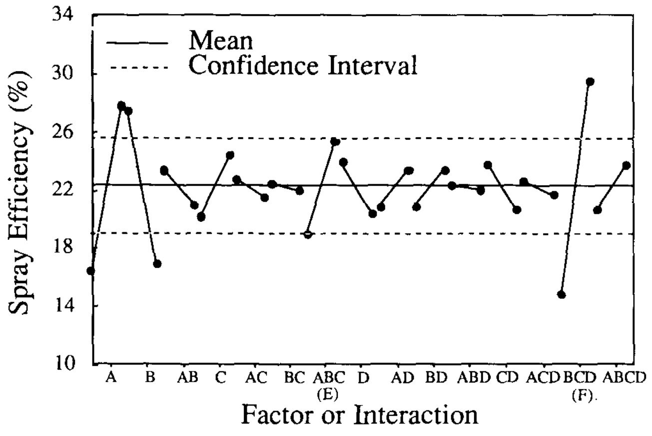

Fig. 2 Response of spray efficiency to variations in the level of main and interaction effects. The average value is the mean of all spray trials. \ represents an increase in efficiency with an increase in the level of a factor and / represents a decrease in efficiency with an increase in the level of a factor. Factors *E* and *F* are shown in parentheses because they are confounded with interactions *ABC* and *BCD*, respectively.

Table 4 Levels of factors investigated in the second factorial experiment

Factor	Levels
A, argon plasma gas flow rate	35 and 40 SLPM
B, hydrogen plasma gas flow rate	8 and 12 SLPM
C, spray distance	220 and 320 mm
D, chamber pressure	120 and 160 mbar

factors needed to be adjusted to increase spray efficiency, e.g., a positive effect implies the factor should be increased. A second factorial experimental design was constructed with factor levels adjusted according to the sign of the significant effects estimates determined previously. It was considered impracticable to increase the gun current or powder carrier gas flow rate beyond that used in the first experiment, and hence, these were held constant at 750 A and 3.5 SLPM, respectively. The removal of these two factors allowed a full factorial experiment to be performed with the remaining four factors. Therefore, the flow rate of hydrogen plasma gas and spray distance were incorporated in the second experiment, even though they were not found to be significant in the first experiment. The levels of the factors used in the design are given in Table 4. The factors were incorporated into a 2^4 design, shown in Table 5, together with the spray efficiencies calculated from weight gain measurements.

The average response increased to 66.9%, a significant improvement on the initial experiment. This suggests that the assumption made in the earlier experiment was correct, i.e., the high-order interaction effects were insignificant. The effects estimates calculated using the data from Table 5 are summarized in Table 6.

Overall, there was a reduction in the size of the effects estimates compared with the average response. This was expected, given that the optimum spraying conditions were being approached (most industrial processes become less sensitive to the process variables as optimum conditions are approached). The effects estimates were used to construct a normal probability plot (Fig. 3). Three effects appeared significant—*A*, argon flow rate; *C*, spray distance; and *D*, chamber pressure. An RSD of ±0.9 was calculated using the 12 effects estimates that were considered insignificant (lying on or close to the straight line in Fig. 3). The 95% confidence interval calculated using this deviation was ±0.9. This confirmed that only factors *A*, *C*, and *D* were significant (see Fig. 4). As in the first experiment none of the interaction effects are significant.

Table 5 Design matrix for a full 2^4 factorial experiment and spray efficiencies obtained for 500# Universal Abrasives powder

Factor				Spray
A	*B*	*C*	*D*	efficiency, %
−	−	−	−	67.5
+	−	−	−	59.4
−	+	−	−	68.6
+	+	−	−	62.5
−	−	+	−	66.2
+	−	+	−	57.1
−	+	+	−	66.8
+	+	+	−	59.8
−	−	−	+	79.1
+	−	−	+	69.2
−	+	−	+	75.7
+	+	−	+	68.4
−	−	+	+	69.7
+	−	+	+	64.0
−	+	+	+	71.8

Table 6 Effects estimates calculated from the data in Table 5

Factor/interaction	Effect estimates
Average (*I*)	66.9
A	−7.5
B	0.8
AB	0.7
C	−3.7
AC	0.4
BC	0.8
ABC	−0.4
D	6.9
AD	0.1
BD	−1.1
ABD	−0.3
CD	−1.7
ACD	0.8

3.3 *Discussion of Alumina Results*

The substantial increase in spray efficiency observed in the second series of trials (Table 5) clearly shows that an optimization methodology based on factorial experimental design can be used to improve the processing of ceramic materials in the VPS system. The success of this approach is due to two main factors: (1) processing variables (gun conditions, powder injection, and gun manipulation, etc.) can be controlled to a sufficient degree to minimize process variability and thus allow significant effects to be identified; and (2) several of the basic process variables have a major influence on the degree of particle melting. The improvement in spray efficiencies obtained in the second factorial experiment indicates that the assumptions about the ef-

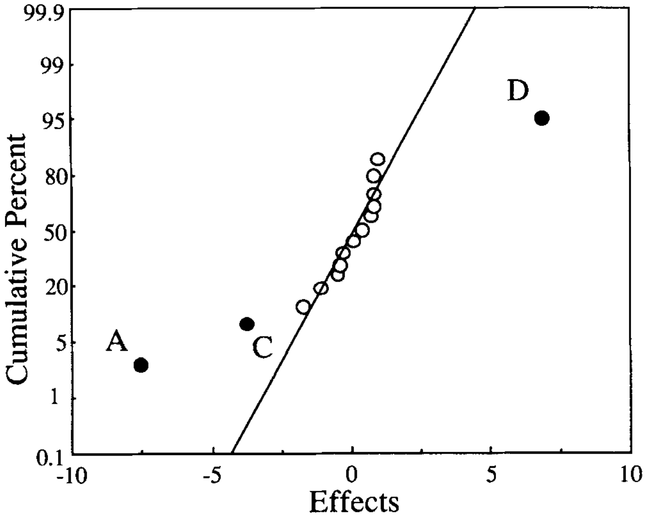

Fig. 3 Probability plot of the effects listed in Table 6. *A*, argon flow rate; *C*, spray distance; and *D*, chamber pressure.

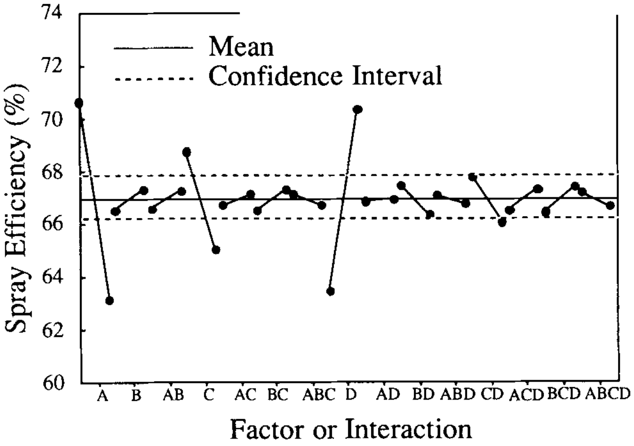

Fig. 4 Response of spray efficiency to variations in the level of main effects and interactions. The average response (—) is the mean of all of the spray trials, and the confidence interval (- -) was calculated using Eq 2.

fect of factors inferred from the first experiment were valid and not due to random variations. Therefore, it was not considered necessary to replicate any of the experiments. The significant effect of chamber pressure in the second experiment and its insignificance in the first experiment indicate that some of the interactions involving this parameter are important. Because none was identified in either experiment, it suggests that the interaction effect is small, i.e., less than the 95% confidence interval for the first experiment.

By comparing the effects estimates from the two factorial experiments, it is possible to draw general conclusions on the influence of process variables on coating development. For example, increasing the pressure within the chamber during spraying and reducing the flow rate of the argon plasma forming gas improved the spray efficiency. Both of these factors would tend to reduce the velocity of the plasma flame,[6,9] which suggests that poor efficiency is due in part to insufficient dwell time of the alumina particles within the plasma flame. One of the most significant factors in both experiments was the carrier gas flow rate. This is not surprising, because the flow rate has a strong influence on the velocity of the particles as they are injected into the plasma and hence their trajectory close to the hottest part of the flame. This explanation is an over simplification, because the parameters often influence the spray process in a complex manner; for example, chamber pressure affects the trajectory of the powder particles into the plasma as well as the temperature and velocity profile of the flame. Although the spray efficiencies obtained in the second factorial experiment were high, the size of the factor effects indicated that further improvements could be achieved. However, an efficiency of ~80% was considered to be adequate for coating production, and the optimization process was terminated. It is important to note that the optimum conditions identified are only relevant to the deposition of the Universal abrasives 500# alumina powder. A similar, optimization process would need to be performed if a different powder size range or morphology was considered. However, it is likely that the same factors would be significant.

The structure of the coatings produced using the efficient plasma spraying conditions has been assessed using a combination of X-ray diffraction and metallographic examination. Polished cross sections of the coatings exhibited relatively small amounts of porosity compared with standard air plasma sprayed alumina coatings. All of the deposits were predominately γ alumina. Quantitative X-ray analysis indicated that approximately 3.5% of the coating consisted of α alumina, which was attributed to the entrapment of particles that had not been fully molten in the spray stream.[10] Dry erosion tests were performed on VPS alumina coatings sprayed using optimum conditions. The steady-state wear rates calculated from weight loss measurements indicated that the wear resistance of the VPS coatings was comparable with that of bulk sintered alumina.[11] In similar tests performed on APS deposits sprayed using commercial parameters, the steady-state wear rates were approximately two orders of magnitude greater.[12]

4. VPS Processing of a Nickel-Base Alloy

The spray deposition of the nickel-base alloy powder Nybynic 625 (61% Ni, 21.1% Cr, 8.5% Mo, 4.5% Fe, and 3.5% Nb; Anval Nyby AB, Sweden) was investigated using a full 2^4 experimental design. The experiment was performed using two sets of eight spray trials (two separate chamber pump-down cycles). A blocking design[5] was used to allow the effect due to the two pump-down cycles to be quantified. The blocking effect was aliased with the highest order interaction, *ABCD*. The deposition of a metal powder was expected to be less sensitive to the plasma spraying conditions than a high-melting-point ceramic material, and therefore, the range of each factor was relatively wide, as shown in Table 7. The experimental design described in Table 5 was used for the evaluation. Spray trials were randomized to reduce the effect of time-related variations in each set of eight trials. The spray efficiencies calculated from weight gain measurements ranged from 63.1 to 89.4%, with an average response of 74.9%. The magnitude of the effects was very small,

Table 7 Parameters in the full 2^4 factorial experiment to investigate deposition of Nybynic 625

Factor	Levels
A, gun current	550 and 700 A
B, chamber pressure	80 and 160 mbar
C, hydrogen plasma gas flow rate	6 and 9 SLPM
D, carrier gas flow rate	1.5 and 3 SLPM

Table 8 Effects estimates calculated from spray efficiency data for Nybynic 625 powder

Factors/interactions	Effects estimates
Average (*I*)	74.9
A	2.1
B	−1.6
AB	0.9
C	−1.6
AC	4.1
BC	2.1
ABC	0.8
D	3.9
AD	1.9
BD	−4.5
ABD	4.2
CD	4.4
ACD	3.6
BCD	−0.1
ABCD (block variable)	−11.9

confirming the assumption that particle melting is relatively insensitive to processing conditions.

The only significant effect was the high-order interaction *ABCD* (see Table 8); however, this interaction was confounded with the block variable and thus is more likely to be due to the effect of performing two sets of spray trials. These results suggest that, for metal powders, variations in particle trajectory, acceleration and deceleration rates, dwell time, and flame temperature and enthalpy do not have a significant influence on the degree of particle melting.

A limited investigation has been made of the properties of coatings produced using optimized spraying conditions. Polished cross sections of the coatings contained very low levels of porosity and a small quantity of particles that had not been fully melted in the plasma flame. This structure provided excellent corrosion-resistant properties; for example, in tests performed on 500-µm thick coatings in artificial seawater, the rate of corrosion was comparable to wrought Inconel 625.[13]

5. VPS Processing of a WC/Co Powder

Table 9 Parameters in a full 2^3 factorial experiment to investigate the deposition of a WC-12%Co powder

Factors	Levels
A, chamber pressure	160 and 180 mbar
B, argon plasma gas flow rate	30 and 35 SLPM
C, helium plasma gas flow rate	30 and 45 SLPM

Table 10 Effects estimates calculated from weight gain measurements for WC-12%Co powder

Factor or interaction	Effects estimates
Average	1.655
A	0.053
B	−0.121
AB	0.095
C	0.313
AC	−0.089
BC	0.099
ABC	−0.027

Tungsten carbide/cobalt cermet coatings are widely used for applications that require low-temperature wear resistance. Cermet properties are a function of the degree of inter-splat contact, porosity level, the ratio of metal-to-ceramic in the precursor powder and the degree of carbide dissolution into the cobalt matrix during spraying. A factorial experiment was performed to investigate the effect of processing conditions on particle melting of a WC-12% Co powder (Metco 72F-NS, Metco Ltd, Chobham, England), see Table 9.

The degree of particle melting was assessed from weight gain measurements. The weight gained by substrates after spraying (normalized to remove the effect of coating area) was used to calculate the effects estimates (see Table 10).

The weight gain measurements varied by ±20% about the mean value. This small variation, combined with the lack of any significant effects, suggests that the deposition efficiency of the WC/Co material is relatively insensitive to VPS conditions. Metallographic examination of the coated samples revealed relatively high porosity levels ranging from 10 to 15%. The pores were not distributed evenly throughout the coating, which suggested there was a time-related effect that influenced the deposition process, such as turbulence in the plasma flame or pulsing of the powder in the feed system. A high percentage of the WC ceramic was retained within the sprayed coating, typically 60 to 70 vol%. This compares with ~10% observed in air plasma sprayed deposits. Preliminary trials using an argon/hydrogen plasma flame produced WC/Co coatings with lower levels of porosity, but with appreciable dissolution of the carbide into the matrix. Further optimization of this material will require a detailed analysis of the effects of plasma gas composition and the incorporation of efficiency, porosity, and carbide retention as response parameters.

6. Conclusions

The VPS process can be investigated efficiently using methods based on factorial experimental designs. Statistical experiments may be used to identify parameters that have a significant influence on particle melting and spray efficiency. If the deposition of a coating is influenced by the level of one or more parameters, or by the interaction of several parameters, then the factorial analysis can be used to indicate optimum conditions. In

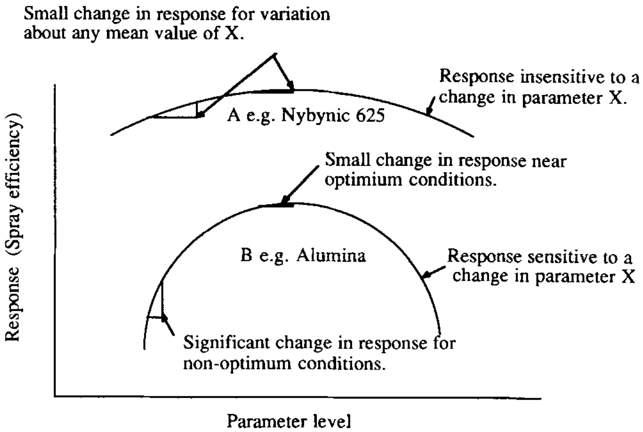

Fig. 5 Types of behavior observed in factorial experiments performed using metal and ceramic precursor powders.

the analyses performed in this work, two distinct types of behavior were observed, as shown in Fig. 5. First, the processing of alumina was found to be highly sensitive to the deposition conditions (represented by curve B in Fig. 5). Thus, a series of factorial experiments could be used to rapidly optimize the process variables. The processing of metal and cermet materials was found to be relatively insensitive to processing conditions, and the high deposition efficiencies obtained tend to suggest that this is because the metal particles or metal in cermet particles can be melted easily under a wide range of processing conditions (curve A in Fig. 5).

Acknowledgments

This work was partly funded by the Commission of European Communities under BRITE project 1203. The authors would like to thank The Materials Characterization Service of AEA Industrial Technology for preparation of metallographic specimens and for performing the X-ray analyses.

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