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Optimization of Cold Spray Nozzles Based on the Response Surface Methodology

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Abstract. Spraying technical parameters are important factors that affect spraying efficiency. Most studies on spraying technical parameters use single-factor methods to study the speed of spray particles, and few scholars have studied the joint influence of multiple factors. This article uses gas temperature, particle size, and gas pressure as independent variables, and the independent variables interact. The design-expert method was used to establish a linear regression equation model of the velocity of sprayed Al and Cu particles at the Laval exit and the velocity before deposition with the substrate, and the response surface analysis method was used to predict the optimal spraying parameters of Al and Cu particles. The study found the contribution rate of three factors to particle velocity: the prediction of particle velocity at the exit of the Laval nozzle and before deposition with the substrate was realized; the error between the predicted value of particle velocity and the actual value obtained by simulation is less than 1.6 %, indicating that the speed linear regression equation established is effective and reliable in predicting the simulation results; the optimal spraying parameters and particle speeds of Al and Cu particles were obtained through response surface analysis.

Keywords: cold spray, multi-factorial experiment, regression analysis, design optimization.

1 Introduction

Cold spray technology is a solid-state deposition technology based on aerodynamic principles. High-pressure gas carries particles through the Laval nozzle, forming a supersonic two-phase flow. The particles are deposited on the surface of the substrate through plastic flow deformation to form a protective coating. In cold spray technology, many parameters affect the velocity of particles in the nozzle, which are roughly divided into structural and technical parameters; technical parameters are mainly parameters of the Laval nozzle structure. Tan summarized the technical parameters and their impact on cold spray technology [1, 2].

The technical parameters mainly include gas temperature and pressure, particle temperature and size, spraying distance, and angle [3]. Currently, most researchers use a single technical parameter factor as an independent variable to study the law of cold spraying technology in the spraying process [4, 5].

2 Literature Review

The research works [6–8] studied the deposition of Cu particles on the substrate at the incident angle. The results show that as the incident angle increases, the depth of the pits gradually decreases, and the bonding strength between the particles and the substrate gradually weakens. When the incident angle exceeds a critical value, the particles will not be embedded in the substrate but detach.

The research works [9–12] proved that preheating treatment of particles or sprayed substrates can enhance the bonding strength, improve the deposition efficiency, and reduce the critical speed. The influence of the shape of particles on the traction coefficient was studied in [13–15].

Plastic deformation and temperature of particles after the deposition of Al alloys of different sizes and shapes were studied in [16]. The distribution changes and the spraying effect is better if particles with similar particle size and shape are selected. It is significant to study the laws of the cold spray process under the joint influence of multiple factors of technical parameters.

Ashokkumar et al. [17] used the response surface (RSM) methodology to study the effects of temperature, spraying distance, and feed rate on the porosity of the layer. Silvello et al. [18] studied the influence of temperature and pressure on the density of the gas used and the influence of simple parameters on the particle properties with the particle velocity and coating. Hu et al. [19] studied the effects of nozzle length, spraying distance, and deposition speed.

In the spraying process, the factor that affects the particle velocity is not a single factor but the result of the coupling of many factors; among them, gas temperature, particle size, and gas pressure are the three most important factors that affect cold spraying, and are also the factors that many scholars currently influencing factors of joint research; this article uses gas temperature T_G , particle size D_p , and gas pressure P_s as independent variables.

The three independent variables interact in pairs, and the design of spraying Al and Cu particles in Laval is established through the design-expert approach. Linear regression equation model of velocity V_e at the exit and velocity V_p before deposition on the substrate, using the RSM method to predict the optimal spraying parameters and speeds of Al and Cu particles, comparing the predicted values with the actual values, and verifying the impact of RSM on cold spraying technical parameters feasibility and accuracy of optimization. The RSM combines mathematical and statistical methods and is often used to find optimal parameters in multi-factor systems.

3 Research Methodology

3.1 Laval nozzle's parameter design

The Laval nozzle is divided into a convergence section, a throat section, and an expansion section (Figure 1).

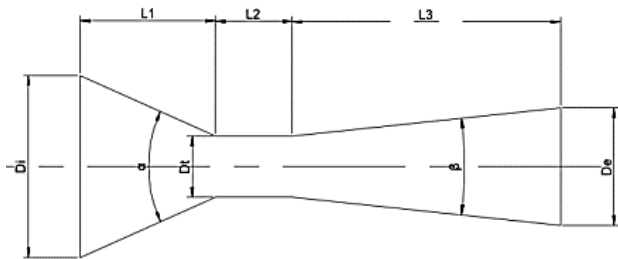


Figure 1 – The design scheme of the Laval nozzle

The main parameters of the Laval nozzle length of convergent section: L_1 – length of the throat, m; L_2 – length of the cylindrical part, m; L_3 – length of the expansion

section, m; D_i – inlet diameter, m; D_t – throat diameter, m; D_e – outlet diameter, m; α – inlet convergence angle, rad; β – outlet diffusion angle, rad.

The main design parameter of the convergence section is the entrance convergence angle, and its selection range is 30–60°; the outlet diffusion angle of the expansion section is 10–12°. In order to reduce energy loss, the radius of curvature R of the throat is equal to the radius of the throat, m.

The inlet pressure is 0.8–1.2 MPa, and the gas temperature is between 400–800 °C. The accelerating medium is air, the adiabatic index of air $\gamma = 1.4$, and the gas constant for air $R = 287.1$ J/(kg·K).

The inlet velocity of sprayed powder material equals 25 m/s. The particle entrance is located above the throat, and the diameter of the entrance is large.

The technical parameters of the Laval nozzle are as follows: $D_i = 3.5$ mm, $D_t = 11.5$ mm, $D_e = 6.1$ mm; $D_p = 1.0$ mm; $L_1 = 15$ mm, $L_2 = 5$ mm, and $L_3 = 65$ mm.

Two types of particle material (Al and Cu) are used as spraying materials.

Table 1 shows that increased gas temperature effectively reduces the particles' deposition rate on the substrate.

Table 1 – The velocity ranges of particles deposition, m/s

Air temperature, °C	Material	
	Al	Cu
424	327–651	447–905
526	314–647	449–883
632	301–638	437–872

The temperature is also an essential factor affecting deposition efficiency. The influence of gas temperature on critical velocity was shown in [20]. Also, spraying Al particles with a distance of 30 mm and Cu particles with a distance of 15 mm were shown in [1, 21].

Using the experimental design method, T_G , D_p , and P_s are selected as the key test factors, and the velocity of the sprayed particles at the Laval nozzle exit and the sprayed particles deposit the velocity in front of the substrate is used as the target, and -1, 0, and +1 are used to represent the numerical simulation factor levels, as shown in Table 2, which are the design parameters for the spraying materials of Al and Cu particle. The density of Cu is greater than that of Al, so a higher gas pressure is required for Cu, and the particle size of Cu particle should not be too large.

Table 2 – Design parameters of sprayed Al and Cu particle

Level	Factors					
	T_G , °C		D_p , μm		P_s , MPa	
	Al	Cu	Al	Cu	Al	Cu
-1	450	600	10	10	0.8	1.4
0	550	700	20	15	1.0	1.5
+1	650	800	30	20	1.2	1.6

3.2 Experimental results data

Design-Expert DX10 data analysis processes and analyzes the numerical simulation results. The experimental arrangement and results are summarized in

Table 3, where the following input (X) and output (Y) factors are designated: X_1 – gas temperature (T_G); X_2 – particles' diameter (D_p); X_3 – gas pressure (P_s); $Y_1 = V_e$ – particles velocity at the Laval nozzle outlet, m/s; $Y_2 = V_p$ – particles velocity before deposition, m/s.

Table 3 – Experimental results data for different materials of particles

No.	High and low-level code			Output parameters, m/s			
				Al		Cu	
	X_1	X_2	X_3	Y_1	Y_2	Y_1	Y_2
1	-1	0	-1	465.4	454.7	459.4	460.8
2	+1	+1	0	418.2	404.9	479.4	480.5
3	0	0	0	503.7	495.4	478.5	478.8
4	+1	0	+1	563.8	551.0	508.0	508.6
5	+1	0	-1	563.8	551.0	494.6	495.6
6	0	+1	-1	441.8	428.3	441.1	443.0
7	0	-1	-1	612.4	550.0	545.5	543.8
8	-1	0	+1	498.5	482.8	462.8	460.6
9	0	0	0	503.7	495.4	478.5	478.8
10	-1	+1	0	456.0	465.7	425.7	428.8
11	0	0	0	503.7	495.4	478.5	478.8
12	0	0	0	503.7	495.4	478.5	478.8
13	0	-1	+1	652.0	588.2	551.8	552.4
14	0	+1	+1	442.1	428.4	446.8	446.1
15	0	0	0	503.7	495.4	478.5	478.8
16	-1	-1	0	588.3	551.8	525.5	525.3
17	+1	-1	0	622.7	583.6	568.2	568.3

3.3 Linear regression analysis

The regression equation for outlet velocity V_e of Al particles at the exit of the Laval nozzle is as follows:

$$Y_{Al} = 503.66 + 20.04 \cdot X_1 - 89.68 \cdot X_2 + 9.11 \cdot X_3 - 18.06 \cdot X_1 \cdot X_2 - 8.27 \cdot X_1 \cdot X_3 - 9.83 \cdot X_2 \cdot X_3 + 1.72 \cdot X_1^2 + 15.90 \cdot X_2^2 + 17.53 \cdot X_3^2 \quad (1)$$

Table 4 shows the variance analysis of the Al particle at the exit velocity of the Laval nozzle.

It can be seen from Table 5 that the value of the model $P = 0.0008$, the model regression equation is significant, and the value of the lack of fit $F = 2.19$ (greater than 0.05) is not significant. The correction value of the model is 0.9771 (greater than 0.8). These values indicate that the equation fits well with the simulation, the correlation between the three factors and the experimental indicators is significant, and the fit degree is acceptable. Overall, the model is suitable for predicting the velocity of Al particles at the exit of the Laval nozzle.

Table 4 – Variance analysis of Al particle at the outlet velocity V_e

Source	Sum of squares	df	Mean square	F value	P-value (probability > F)	Significance
Model	72720.1	9	8080.01	15.1	0.0008	significant
X_1	3212.41	1	3212.41	6.02	0.0439	–
X_2	64336.4	1	64336.4	121	< 0.0001	–
X_3	664.480	1	664.480	1.24	0.3014	–
$X_1 \cdot X_2$	1304.29	1	1304.29	2.44	0.1620	–
$X_1 \cdot X_3$	273.570	1	273.570	0.51	0.4973	–
$X_2 \cdot X_3$	385.320	1	386.320	0.72	0.4231	–
X_1^2	12.4600	1	12.4600	0.02	0.8829	–
X_2^2	1064.80	1	1064.80	1.99	0.2008	–
X_3^2	1293.16	1	1293.16	2.42	0.1636	–
Residual	3737.41	7	533.920	–	–	–
Lack of fit	3737.41	3	1245.80	2.19	0.2337	not significant
Pure error	0.00000	4	0.00000	–	–	–
Cor total	76457.5	16	–	–	–	–

The regression equation for outlet velocity V_e of Cu particles at the exit of the Laval nozzle is as follows:

$$Y_{Cu1} = 479.11 + 20.18 \cdot X_1 - 48.13 \cdot X_2 + 3.60 \cdot X_3 + 6.60 \cdot X_1 \cdot X_2 + 2.49 \cdot X_1 \cdot X_3 - 0.16 \cdot X_2 \cdot X_3 - 0.14 \cdot X_1^2 + 16.61 \cdot X_2^2 + 1.40 \cdot X_3^2 \quad (2)$$

It can be seen from Table 5 that the value of the model $P = 0.0001$, the model regression equation is significant; the value of the lack of fit $F = 2.41$ (greater than 0.05) is not significant. The correction value of the model is 0.9477 (greater than 0.8), indicating that the equation fits appropriately.

Table 5 – Variance analysis of Cu particle at the outlet velocity V_e

Source	Sum of squares	df	Mean square	F value	Q-value (probability > F)	Significance
Model	25277.5	9	2808.61	122.9	< 0.0001	significant
X_1	3401.41	1	3401.41	144.8	< 0.0001	–
X_2	17075.9	1	17075.9	747.0	< 0.0001	–
X_3	103.610	1	103.610	4.530	0.0708	–
$X_1 \cdot X_2$	190.410	1	190.410	8.330	0.0234	–
$X_1 \cdot X_3$	24.8000	1	24.8000	1.080	0.3322	–
$X_2 \cdot X_3$	0.0990	1	0.09900	4341	0.9493	–
X_1^2	0.1500	1	0.15000	6590	0.9376	–
X_2^2	1093.3	1	1093.26	47.83	0.0002	–
X_3^2	8.0400	1	8.04000	0.350	0.5718	–
Residual	160.010	7	22.8600	–	–	–
Lack of fit	160.010	3	53.3400	2.410	0.1962	not significant
Pure error	0.00000	4	0.00000	–	–	–
Cor total	25437.5	16	–	–	–	–

The regression equation for outlet velocity V_p of particles before cooling is as follows:

$$Y_2 = 495.37 + 16.93 \cdot X_1 - 68.28 \cdot X_2 + 8.28 \cdot X_3 - 23.13 \cdot X_1 \cdot X_2 - 7.01 \cdot X_1 \cdot X_3 - 9.51 \cdot X_2 \cdot X_3 + 8.63 \cdot X_1^2 - 2.49 \cdot X_2^2 + 5.86 \cdot X_3^2 \quad (3)$$

Table 6 shows the variance analysis of the velocity V_p before the sprayed Al particle deposited on the substrate. The P -value indicates that the regression equation of the model is significant. The value of lack of fit F is more than 0.05, which is insignificant, indicating that the model fits appropriately and the simulation error is small. Overall, the model is suitable.

Table 6 – Variance analysis of particle at the outlet velocity V_p

Source	Sum of squares	df	Mean square	F value	R-value (probability > F)	Significance
Model	24637.9	9	2737.55	103.77	<0.0001	significant
X_1	3394.64	1	3394.64	128.68	<0.0001	–
X_2	16442.8	1	16442.8	623.31	<0.0001	–
X_3	74.7300	1	74.7300	2.83	0.1362	–
$X_1 \cdot X_2$	168.590	1	168.590	6.39	0.0393	–
$X_1 \cdot X_3$	43.1600	1	43.1600	1.64	0.2416	–
$X_2 \cdot X_3$	7.65000	1	7.65000	0.29	0.6070	–
X_1^2	0.15000	1	0.15000	5852	0.9412	–
X_2^2	1121.81	1	1121.81	42.53	0.0003	–
X_3^2	3.22000	1	3.22000	0.12	0.7370	–
Residual	184.660	7	26.3800	–	–	–
Lack of fit	184.660	3	61.5500	4.83	0.0081	not significant
Pure error	0.00000	4	0.00000	–	–	–
Cor total	28422.6	16	–	–	–	–

The contribution rate of experimental factors to experimental indicators is summarized in Table 7.

Table 7 – Contribution rate of factors to experimental indicators

Test index	Contribution rate of experimental factors			Contribution ranking
	X_1	X_2	X_3	
Y_{Al1}	6.02	120.50	1.24	$X_2 > X_3 > X_1$
Y_{Al2}	3.39	55.18	0.81	$X_2 > X_1 > X_3$
Y_{Cu1}	148.8	747.0	4.53	$X_2 > X_1 > X_3$
Y_{Cu2}	103.8	128.7	623	$X_3 > X_2 > X_1$

According to the F -value, the contribution rate of the three influencing factors on the velocity V_e of the Al and Cu particles and the velocity V_p of Cu particles before deposition on the substrate can be judged.

4 Results

4.1 An impact of factors on the velocities

Using the design-expert approach, make the RSM diagram of the interaction between the influencing factors

on the test indicators, and then find out the law of the interaction between the influencing factors on the test indicators.

Figure 2 shows the influence of three factors on the exit velocity of the Laval nozzle for Al particles.

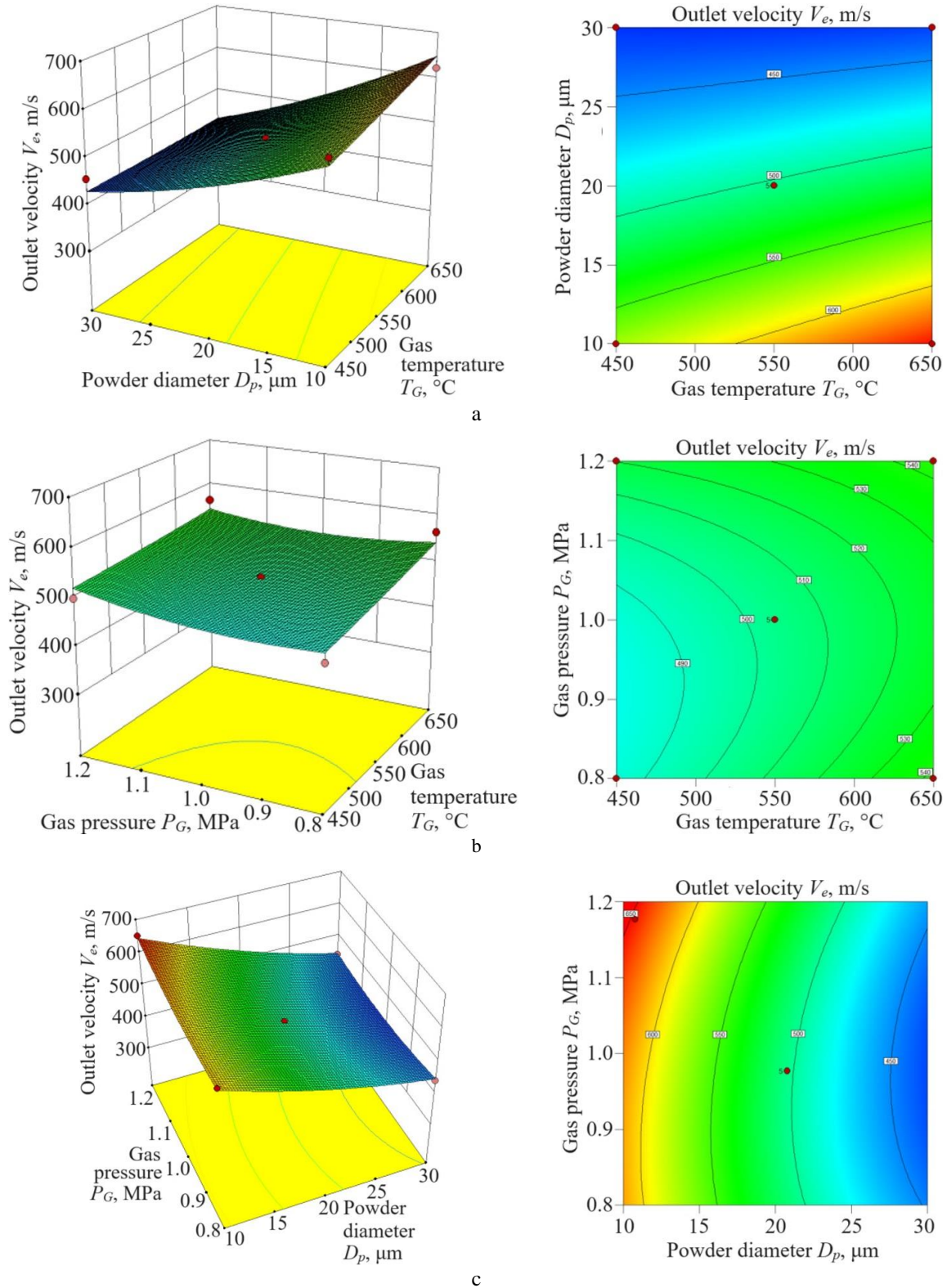


Figure 2 – The influence of three factors on the velocity of Al particles at the exit of the Laval nozzle: a – between D_p and T_G ; b – between P_s and T_G ; c – between P_s and D_p

Combining the analysis of the contribution rate of the experimental factors to the experimental indicators in Table 7, the contribution rate of the Al diameter is more significant. The design point gas temperature is 550 °C, and the Al particle diameter is 20 μm.

Figure 2 b shows the contribution of gas pressure for the design point: the gas temperature is 550 °C, and the gas pressure is 1.0 MPa.

From Figure 2 c, the contribution rate of the Al particle diameter is more significant. The design point is that the Al particle diameter is also 20 μm, and the gas pressure is 1.0 MPa. Among them, the red point in the contour map is the design point, the optimal solution. Simultaneously, the outlet velocity for the corresponding Al particle $V_e = 503.7$ m/s.

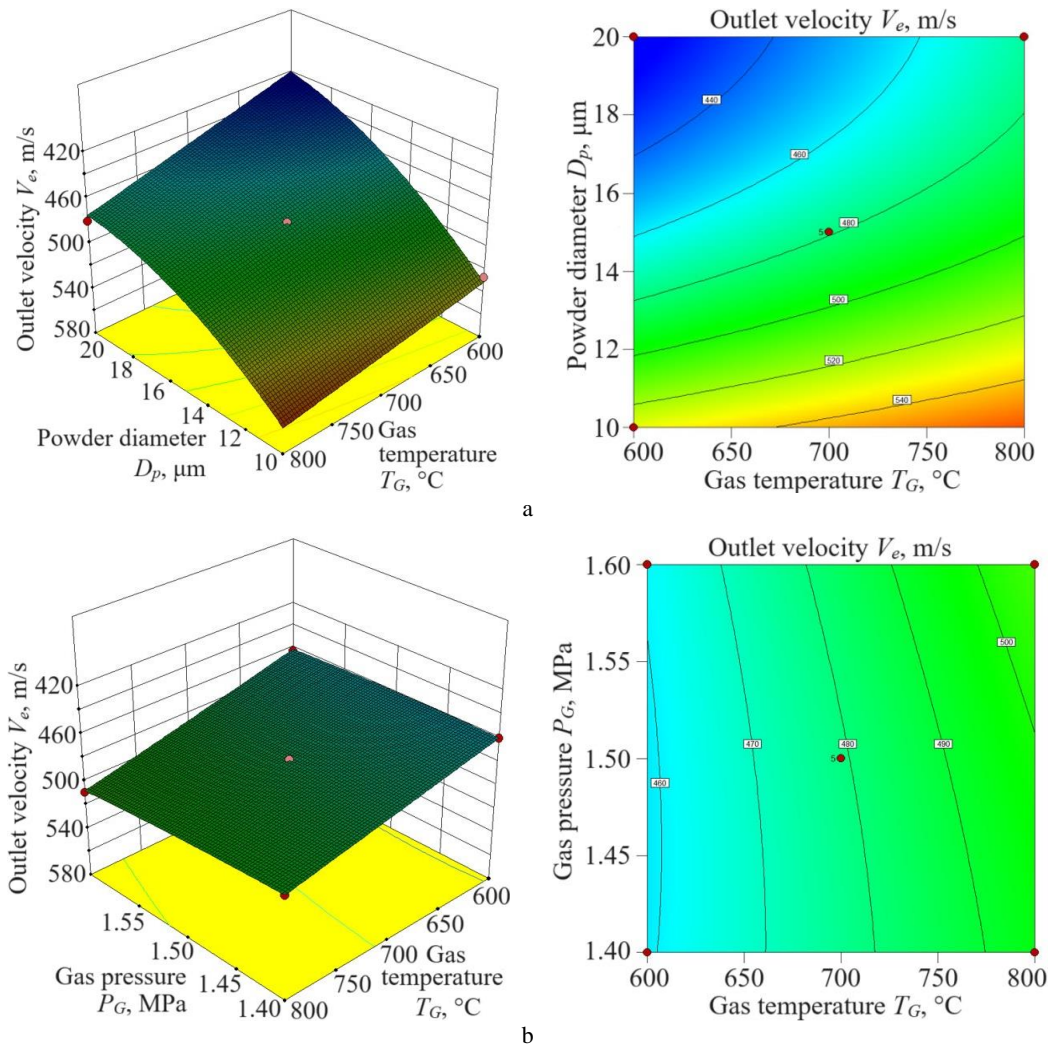
Figure 3 shows the influence of three factors on the exit velocity of the Cu particle Laval nozzle, combined with the analysis of the contribution rate of the experimental factors in Table 7 to the experimental indicators.

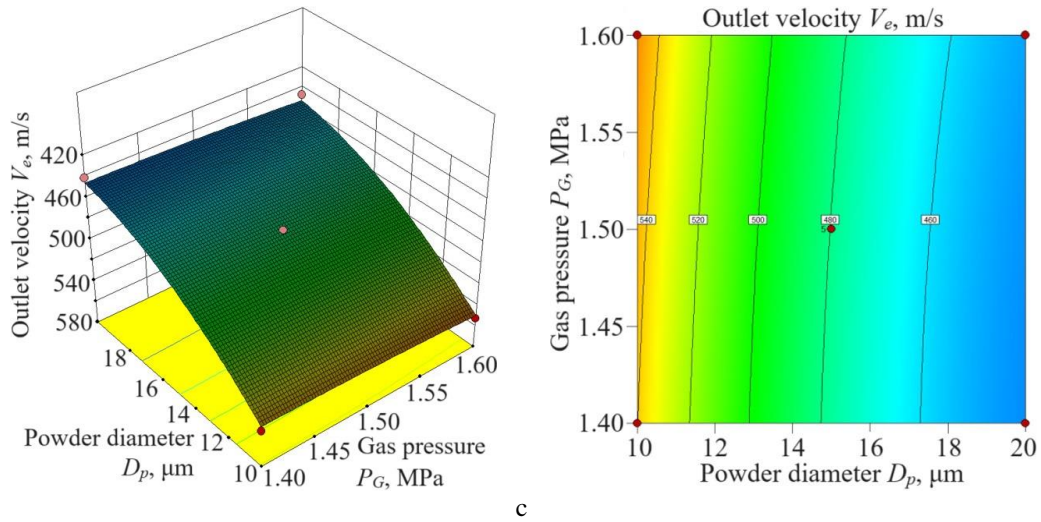
Figure 3 a shows that the contribution rate of the diameter for Cu particles is more significant. The gas temperature of the design point is 700 °C, and the diameter of Cu particles is 15 μm.

As shown in Figure 3 b, the contribution rate of the gas temperature is more significant. The design point is the temperature of 700 °C and the gas pressure of 1.5 MPa.

It can also be seen from Figure 2 c that the contribution rate of the Cu particle diameter is more significant; the design point is that the diameter of the Cu particle is 15 μm, and the gas pressure is 1.5 MPa. In the case of the optimal solution, the Cu particle is sprayed with the outlet velocity $V_e = 479.1$ m/s.

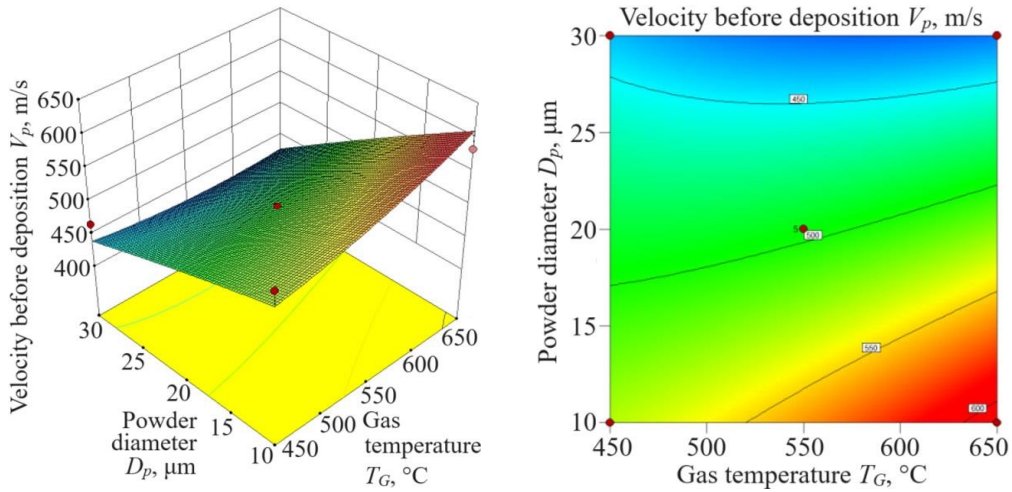
Figure 4 shows the influence of three factors on the velocity V_p before the Al particles were deposited on the substrate.



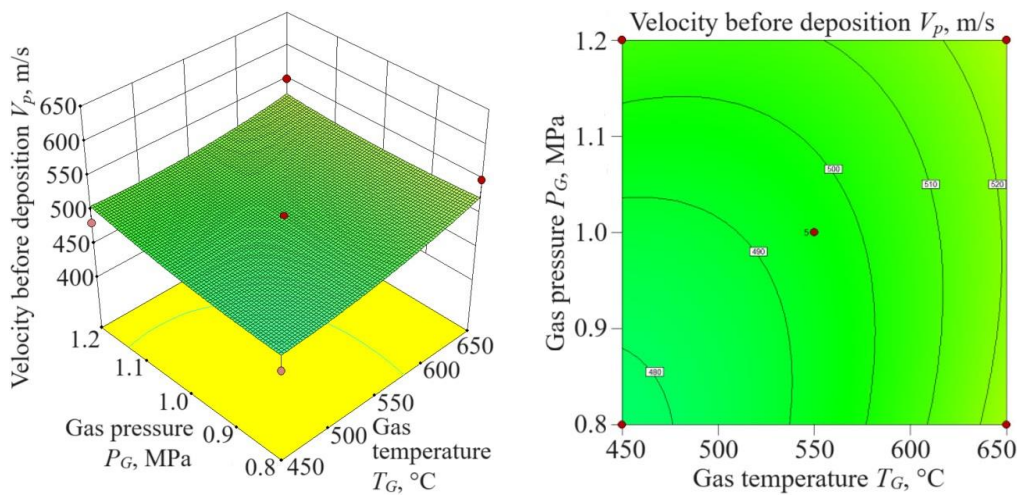


c

Figure 3 – The influence of three factors on the velocity of Cu particles at the exit of the Laval nozzle: a – between D_p and T_G ; b – between P_G and T_G ; c – between P_G and D_p



a



b

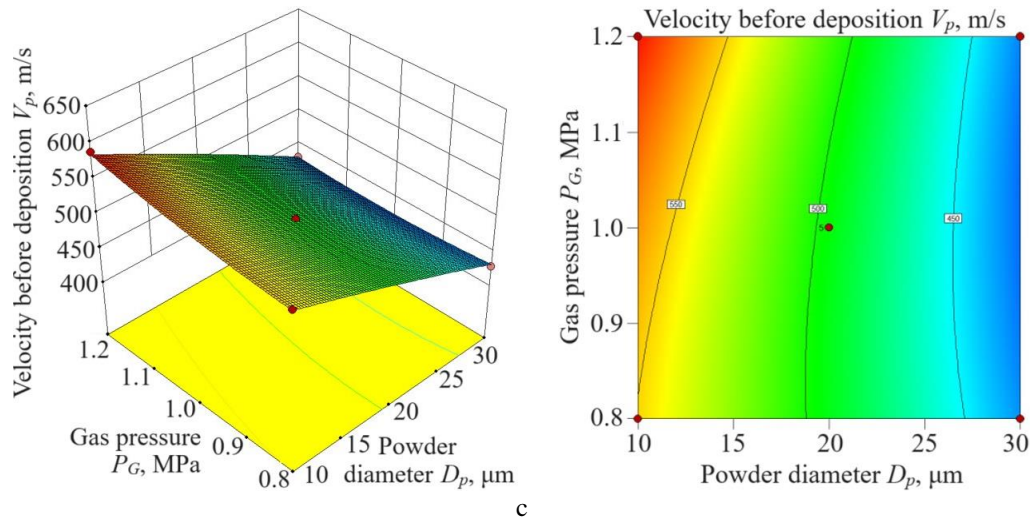


Figure 4 – The influence of three factors on the velocity V_p of Al particles deposited on the substrate: a – between D_p and T_G ; b – between P_s and T_G ; c – between P_s and D_p

Combining Table 7 is the analysis of the contribution rate of the experimental factors to the experimental indicators.

It can be seen from Figure 4 a that the contribution rate of the diameter of the Al particle is more significant; the design point gas temperature is 550 °C, and the diameter of the Al particle is 20 μm .

From Figure 2 b, the contribution rate of the gas temperature is even greater. The design point is that the gas temperature is 550 °C, and the gas pressure is 1.0 MPa.

Figure 2 c shows that the contribution rate of the Al particle diameter is more significant, and the design point is that the Al particle diameter is 20 μm , and the gas pressure is 1.0 MPa.

In the case of the optimal solution, the velocity V_p before the Al particle deposited on the substrate can reach 495.4 m/s.

Comparative analysis of the optimal solution for Al particles for the Laval nozzle's outlet velocity is 503.7 m/s, indicating that the Al particle is in a stage of decreasing velocity from leaving the Laval nozzle exit to the substrate surface, so it can be used as a reference for studying the

spraying distance. Spraying distance is also an essential factor affecting spraying efficiency [22].

Figure 5 shows the influence of three factors on the velocity V_p before Cu particles deposited on the substrate.

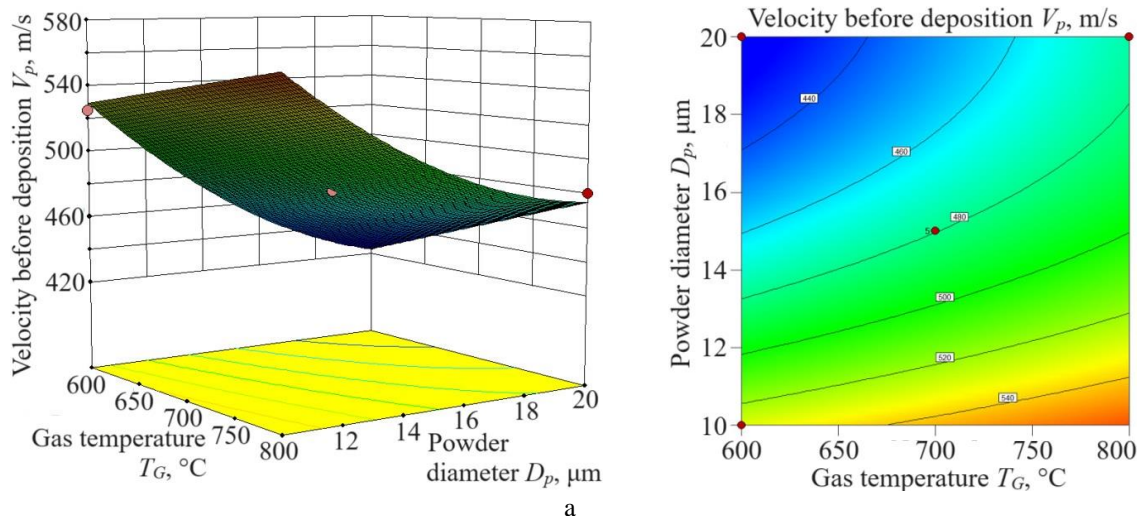
Combining Table 7 is the analysis of the contribution rate of the experimental factors to the experimental indicators.

It can be seen from Figure 5 a that the contribution rate of the diameter of the Cu particle is more significant; the design point gas temperature is 700 °C, and the diameter of Cu particles is 15 μm .

From Figure 5 b, it can be seen that the contribution rate of the gas temperature is more apparent. The design point is that the gas temperature is 700 °C, and the gas pressure is 1.5 MPa.

From Figure 5 c, the contribution rate of the diameter of Cu particles is more significant. The design point is that the diameter of the Al particle is 15 μm , and the gas pressure is 1.5 MPa. The velocity V_p can reach 479.6 m/s.

Comparative analysis of the optimal solution for Cu particle Laval nozzle outlet velocity is 479.1 m/s, indicating that the Cu particle is accelerated.



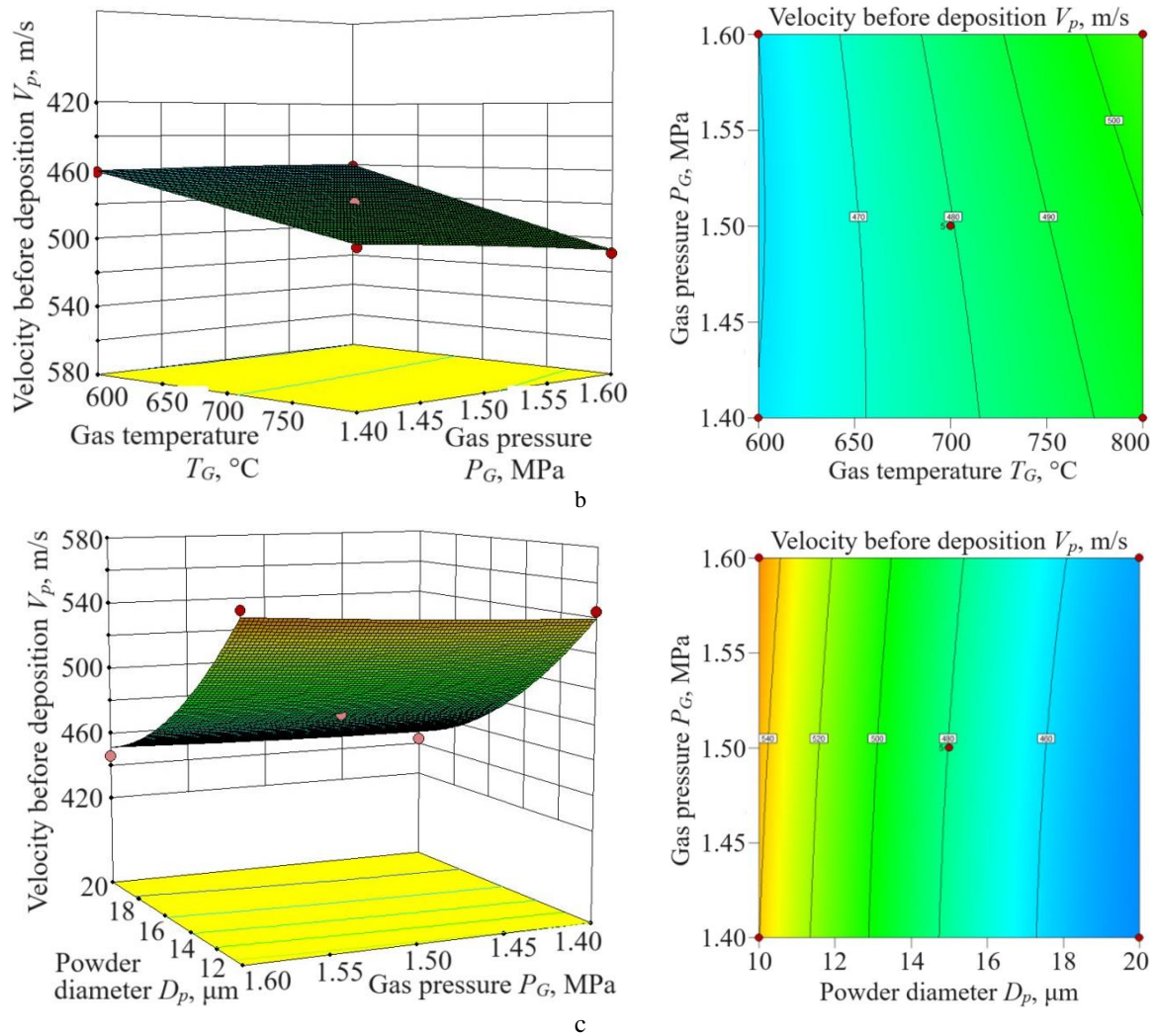


Figure 5 – The influence of three factors on the velocity V_p of Cu particles deposited on the substrate: a – between D_p and T_G ; b – between P_s and T_G ; c – between P_s and D_p

4.2 Design optimization of cold spray nozzles

Therefore, it can increase the spraying distance for Cu particles to obtain the velocity V_p before Cu particles are deposited on the substrate.

After combining the contribution rate of the experimental factors to the simulation indicators in Table 7, the velocity V_p of Cu particles before they are deposited on the substrate is analyzed.

The influence of gas temperature is more significant than that of gas pressure, mainly because the density of Cu particle is large, so it increases within a specific range. The gas temperature is the best solution.

Table 8 shows the optimized parameters' predicted and actual values and errors.

Figure 6 shows the particles' velocity distribution after optimized spraying parameters.

Table 8 – The optimized parameter velocity predicted and actual values and errors

Material	Factor			Predicted value, m/s		Actual value, m/s		Error, %	
	T_G , °C	D_p , μm	P_s , MPa	V_e	V_p	V_e	V_p	δ_e	δ_p
Al	550	20	1	503.66	495.37	502.54	494.5	1.6	0.2
Cu	700	15	1.5	479.12	479.64	478.41	478.83	0.1	0.2

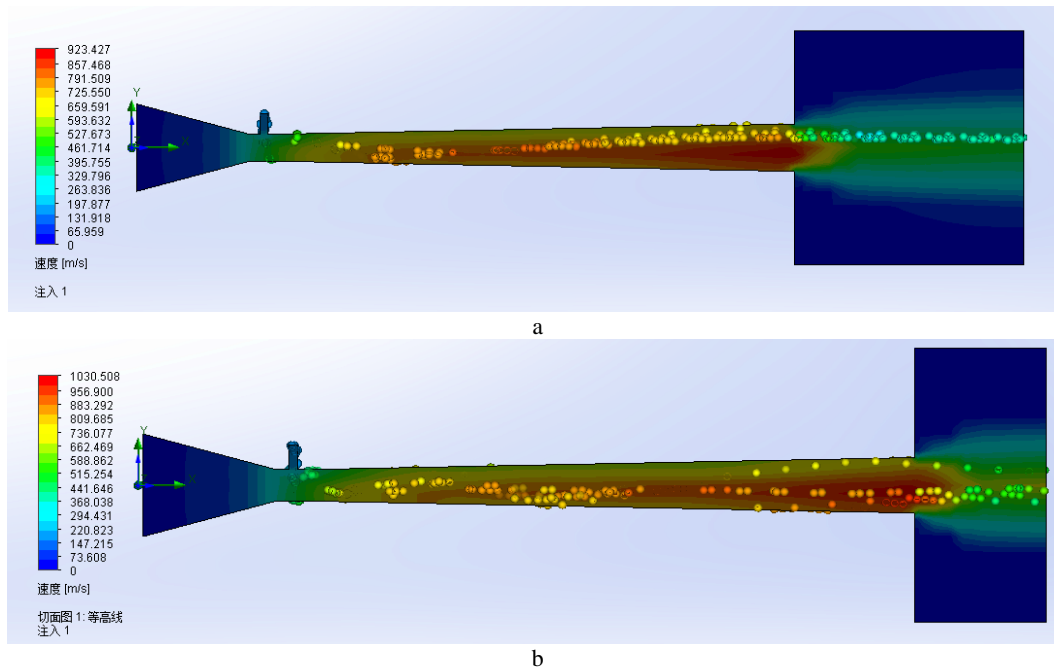


Figure 6 – Velocity distribution of Al (a) and Cu (b) particles after optimization of spraying parameters

5 Discussion

Optimal spraying parameters obtained through the design-expert optimization were obtained after the predicted value was compared with the experimental values.

The error between the predicted value and the actual value of the Al particle velocity at the Laval exit is 1.6 %. The error between the predicted value and the actual value of the velocity before being deposited on the substrate is 0.2 %.

The error between the predicted value and the actual value of the Cu particle velocity at the Laval exit is only 0.1 %. The error between the predicted value and the actual value of the Al particle velocity before it is deposited on the substrate is only 0.2 %.

Overall, the regression equation for the outlet velocity V_e at the Laval nozzle's exit and particle velocity V_p before depositing with the substrate are reliable. Therefore, the results can be effectively used by the regression prediction approach.

6 Conclusions

Al particles' optimal spraying technical parameters are $T_G = 550\text{ }^\circ\text{C}$, $D_p = 20\text{ }\mu\text{m}$, and $P_s = 1.0\text{ MPa}$. The velocity of Al particles at the nozzle outlet $V_e = 502.5\text{ m/s}$. The velocity of Al particles before reaching the substrate $V_p = 494.5\text{ m/s}$. The contribution rate of the Al particle velocity at the Laval exit is as follows: $(D_p) > (P_s) > (T_G)$. The contribution rate of the Al particle velocity before depositing with the substrate is as follows: $(D_p) > (T_G) > (P_s)$.

Cu particles' optimal spraying technical parameters are $T_G = 700\text{ }^\circ\text{C}$, $D_p = 15\text{ }\mu\text{m}$, and $P_s = 1.5\text{ MPa}$. The velocity of Al particles at the nozzle outlet $V_e = 478.4\text{ m/s}$. The velocity of Al particles before reaching the substrate $V_p = 478.8\text{ m/s}$. The contribution rate of the Cu particle velocity at the Laval exit is as follows: $(D_p) > (T_G) > (P_s)$. The contribution rate of the Cu particle velocity before depositing with the substrate is as follows: $(P_s) > (D_p) > (T_G)$.

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