

Effect of Process Parameters and Heat Treatments on Properties of Cold Sprayed Copper Coatings

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Cold gas dynamic spraying or cold spray is specifically suitable to obtain high-conductivity copper coatings for a variety of applications. Copper coatings at different coating parameters were deposited and subjected to various post treatments. The effect of process parameters and the treatment conditions on coating properties such as electrical conductivity, porosity, microhardness etc., was studied. The as-coated specimens exhibited low conductivities and conductivity was found to improve with heat treatment. Treatments were carried out in vacuum at different temperatures and for different durations and conductivities close to bulk annealed copper were achieved. Good correlation was observed between the conductivity, porosity and hardness of the as-coated and heat-treated specimens. Similar correlations were observed between conductivity-porosity and hardness-porosity of the coatings and the relative influence of cold work and porosity on coating properties was determined.

Keywords cold spray, conductivity, copper, heat treatment, porosity

1. Introduction

Cold gas dynamic spraying or cold spray is a thermal spraying technique in which powder particles (typically 10–50 μm) are accelerated to velocities of the order of 600–1000 m/s by a supersonic jet of compressed gas and form coating layer by layer upon impact onto a target surface (Ref 1). Cold spray was developed in the mid 1980's at the Institute of Theoretical and Applied Mechanics of the Siberian Division of the Russian Academy of Science in Novosibirsk (Ref 2, 3). The process details have been elaborated elsewhere (Ref 3, 4). It has been employed to successfully deposit a variety of materials (Ref 5, 6). Unlike conventional thermal spraying processes, the cold spray process does not heat the powder particles significantly and thus provides an excellent opportunity to produce coatings with low-oxide content and low-thermal stresses (Ref 4, 7). Thus, cold spray is specifically suitable to obtain high-conductivity copper coatings for a variety of applications (Ref 8). However, proper selection of process parameters and appropriate post coating treatments are cardinal for exploiting the process capabilities fully so as to obtain the maximum possible electrical conductivity.

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Prior studies on the optimization and post treatment of copper coatings are very limited. Dykhuzien and Neiser (Ref 9) investigated the influence of gas inlet pressure and gas pre-heat temperature on deposition process and concluded that particle velocity is the most important parameter influencing the properties of cold sprayed coatings. McCune et al. (Ref 10) reported the microstructural features of copper coatings produced by two widely different types of starting powders along with preliminary annealing studies for improving the mechanical properties of the deposited layers and found the high-purity feedstock to yield better coating properties. Post processing of copper coatings to obtain equi-axed grains by inducing recrystallization and grain growth was also studied by McCune et al. (Ref 11). Borchers et al. (Ref 12) observed that the cold sprayed copper coatings, upon heat treatment, exhibit electrical conductivities close to that of bulk annealed copper without considerable decrease in hardness even after annealing at 600 °C due to the formation of persistent dislocation loops. Gartner et al. (Ref 13) compared the mechanical properties of cold sprayed and thermal sprayed copper coatings and found the mechanical properties of cold sprayed coatings to improve substantially with heat treatment unlike other thermal sprayed coatings, largely due to diffusion effects during annealing of cold sprayed coating in contrast to oxide rearrangements in thermal sprayed coatings. Stoltenhoff et al. (Ref 14) compared the microstructural features of cold sprayed copper coatings deposited by employing nitrogen and helium as process gases and HVOF coatings along with their subsequent behavior upon annealing and found that cold sprayed coatings exhibit superior electrical properties compared to HVOF coatings. The effect of annealing conditions on microstructure of cold sprayed copper coatings was studied by

Li et al. (Ref 15) and attributed the electrical conductivity improvement upon annealing to recrystallization.

The literature survey thus reveals that copper coatings with electrical conductivity values close to that of bulk copper can be obtained utilizing cold spray, provided, a post-coating annealing treatment is carried out. Nevertheless, a number of aspects pertaining to cold spray coating of copper require further understanding. Some of these aspects, which will be investigated in the present study, are indicated below.

- The relative influence of the cold spray process parameters (e.g.: gas pre-heat temperature, gas inlet pressure, powder feed rate and stand-off distance) on the properties of the resulting copper coatings like porosity, electrical conductivity (henceforth referred to as conductivity) and microhardness have not been systematically investigated.
- At a more basic level, are the properties of the coatings determined only by the velocity of the particle or does particle temperature related to gas temperature also play a role over and above its known effect on particle velocity?
- Is there any correlation between the as-coated properties and the properties after vacuum heat treatment? Alternatively, does the heat treatment alter the coating properties to such an extent that the as-coated properties and hence optimization of coating process parameters, are not relevant?
- What is the relative contribution of cold work and porosity to the electrical conductivity of cold spray copper coatings?

2. Experimental Procedure

2.1 Materials and Coating Deposition

Copper coatings were deposited using the in house facility for cold spraying. A De Laval nozzle with a rectangular exit was used for the present study. Compressed air was used as the process gas as well as the powder carrier gas. Commercially available electrolytic grade copper powder in the size range of 10-45 μm and having an oxygen content of 1400 ppm was used as the feedstock. SEM micrograph of the powder illustrating its dendritic morphology is presented in Fig. 1. Grit blasted commercial purity aluminum was used as the substrate. These specimens were subjected to thorough ultrasonic cleaning prior to coating deposition for better adhesion.

2.2 Parameter Selection

A three level Taguchi design of experiments (Ref 16) was selected for the present study. The parameters for each process variable were carefully chosen so as to include the maximum and minimum possible value for each of the process variable being studied along with a mean value. Modeling studies (Ref 17) have indicated the gas

pre-heat temperature and gas inlet pressure to be important parameters in determining the particle velocity at the exit of the nozzle during cold spraying. Earlier studies have also indicated that powder feed rate and stand off distance have some effect on coating properties (Ref 18). Hence, gas pre-heat temperature, gas inlet pressure, powder feed rate and stand off distance were chosen as the major process variables. Gun speed was maintained at a constant value of 16 mm/s for all the coatings.

A L9 experimental matrix as shown in Table 1 was chosen with the nomenclature and coating parameters for each experiment being specified in the table. The parameters chosen for each variable were based on the equipment capability and process window for practically achieving copper coatings as determined through preliminary experimentation so as to include the entire possible range. The gas pre-heat temperature was maintained at a pre-determined level using a PID controller, while the powder feed rate was varied by controlling the rpm of the motor connected to the feeder. Samples were generated using the chosen parameters in a random sequence to avoid any errors because of specific ordering in the experiments.

2.3 Characterization

X-ray diffraction studies of the powder were carried out using Bruker X-ray Diffractometer (Cu- K_{α} radiation, 40 kV, 40 mA, 0.02 deg/s scan rate). Conductivity of the coatings was measured using an eddy current based coating

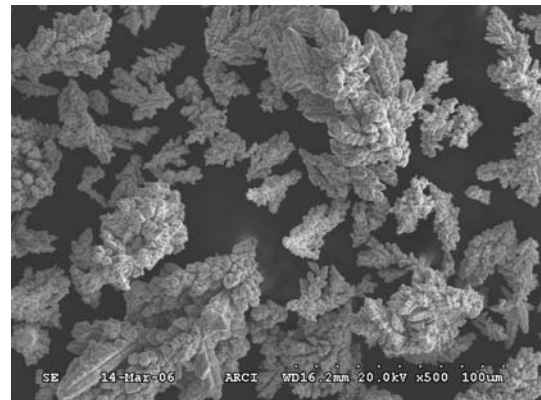


Fig. 1 SEM micrograph of copper powder

Table 1 Experimental matrix

Experiment no	Temperature, °C	Pressure, MPa	Feed rate, rpm	Stand off distance, mm
1	300 (A ₁)	1.4 (B ₁)	15 (C ₁)	5 (D ₁)
2	375 (A ₂)	1.8 (B ₂)	29 (C ₃)	5 (D ₁)
3	450 (A ₃)	1.4 (B ₁)	29 (C ₃)	15 (D ₂)
4	300 (A ₁)	1.8 (B ₂)	22 (C ₂)	15 (D ₂)
5	375 (A ₂)	2.2 (B ₃)	15 (C ₁)	15 (D ₂)
6	300 (A ₁)	2.2 (B ₃)	29 (C ₃)	25 (D ₃)
7	450 (A ₃)	1.8 (B ₂)	15 (C ₁)	25 (D ₃)
8	375 (A ₂)	1.4 (B ₁)	22 (C ₂)	25 (D ₃)
9	450 (A ₃)	2.2 (B ₃)	22 (C ₂)	5 (D ₁)

conductivity gauge (Sigma Test 2.069, Foerster). The specimens were polished before carrying out the conductivity measurements. Around 10 measurements were taken for each specimen at different locations in the coating and the values were found to be within $\pm 6\%$ of the reported mean value, which was used for further analysis. Coated specimens and heat treated specimens were sectioned and the cross sectional face was mounted using Bakelite for metallographic polishing. Coating porosity was measured using an Image Analyzer system (Image Pro Plus, Media Cyber Netics, USA) attached to an optical microscope. An aqueous solution of 5 g FeCl_3 , 10 mL HCl and 100 mL H_2O was used for etching the samples for grain size measurements. Grain size measurements were made using the intercept method. Coating hardness was measured using a Vickers microhardness tester (Leitz-112473,

Germany). At least 10 measurements were taken in a staggered manner on the coating cross section at a load of 100 g and the readings were within $\pm 5\%$ of the reported mean value, which was used for subsequent calculations.

2.4 Heat Treatment

The cold spray coatings were annealed in both vacuum and air at 300 and 450 °C to evaluate the extent to which the conductivity of the coating can be enhanced and thus decide the appropriate treatment atmosphere and temperature. In case of vacuum treatment, a vacuum level of 2×10^{-4} mbar was maintained. Identical heating rates were maintained for air and vacuum treatments. The results from such an exercise presented in Fig. 2, clearly indicates that vacuum annealing at 300 °C is sufficient to obtain conductivity values close to that of bulk copper, as reported by other investigators (Ref 15). No substantial improvement in conductivity is observed beyond 300 °C for vacuum treatment. On the other hand, even a 4 h annealing treatment at 450 °C in air, did not result in the required conductivity. Thus, it has been decided that all further annealing experiments will be carried out at 300 °C in vacuum for 1 h.

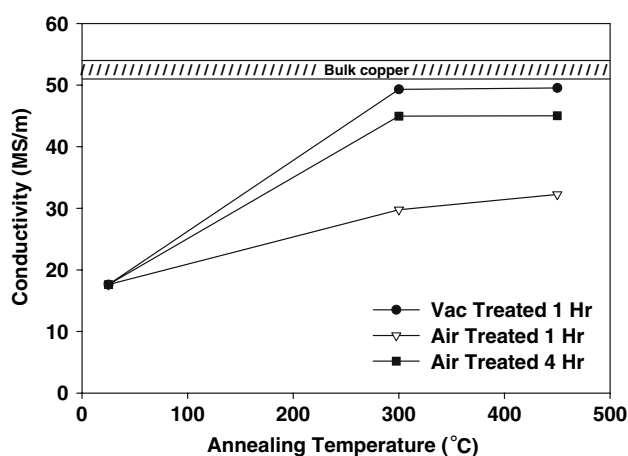


Fig. 2 Variation of conductivity with annealing temperature

3. Results

3.1 Microstructure and Phase Analysis

Figure 3(a, b) shows the typical microstructure of the as coated as well as the vacuum treated specimen for experiment number 7 (see Table 1). A close examination reveals a slight decrease in porosity levels after heat treatment. A similar trend was observed microstructurally for all the other experiments. Figure 3(c, d) shows the

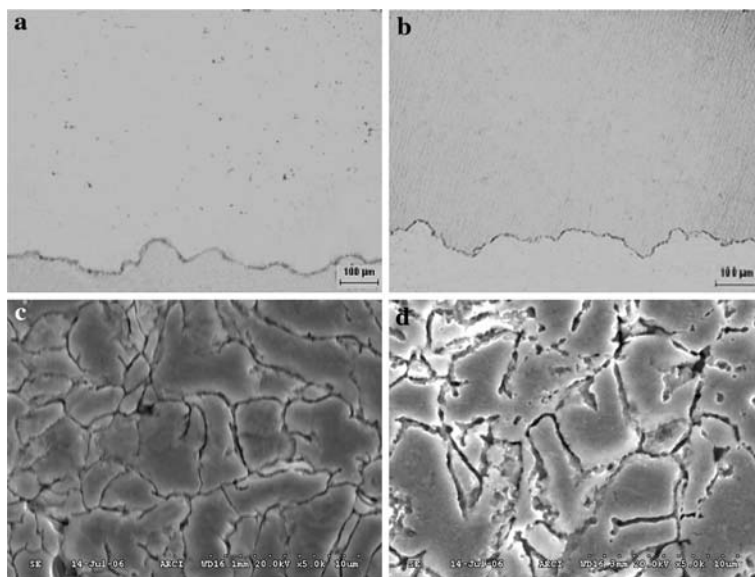


Fig. 3 Typical microstructure of the coating for experiment no 7 (a) as coated cross section; (b) vacuum treated (300 °C 1 h) cross section; (c) as coated and etched; and (d) vacuum treated (300 °C 1 h) and etched

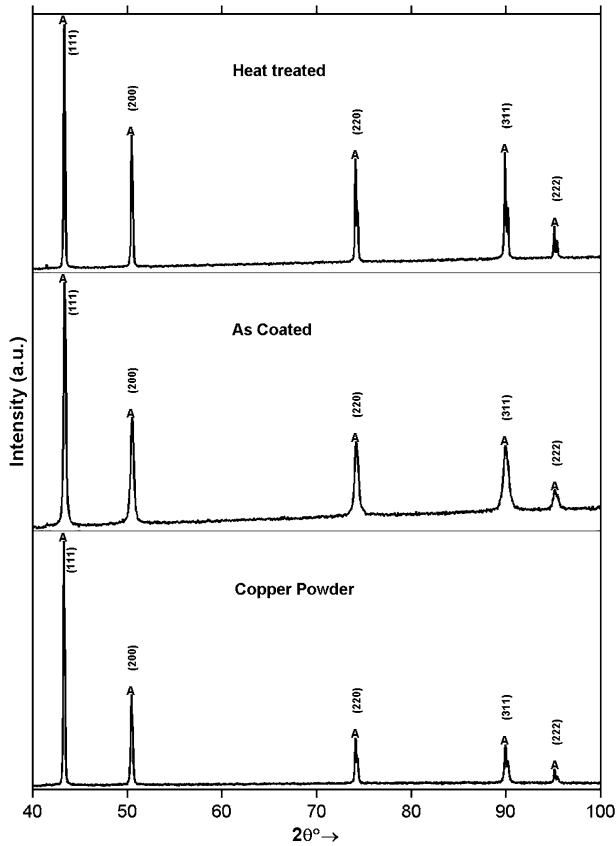


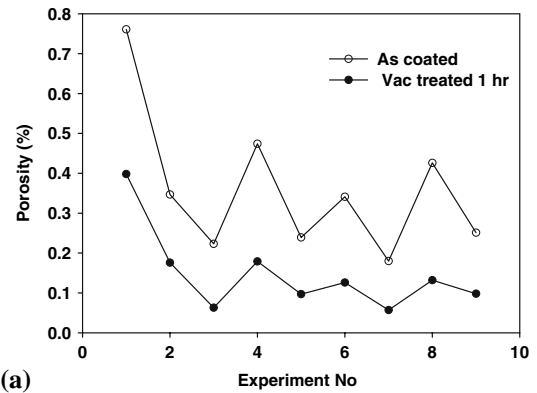
Fig. 4 XRD pattern of powder and coatings

microstructure of the etched specimen for experiment number 7. Substantial change in grain size was not observed for the treatment conditions employed in the present study, as also reported by other investigations (Ref 15). The grain size as measured by the intercept method for the as-coated specimen is $2.17 \pm 0.7 \mu\text{m}$ and showed only a slight increase to up to $3.0 \pm 0.6 \mu\text{m}$ for the specimen heat treated at 300°C in vacuum for 1 h. Similar trend was observed for the other specimens as well.

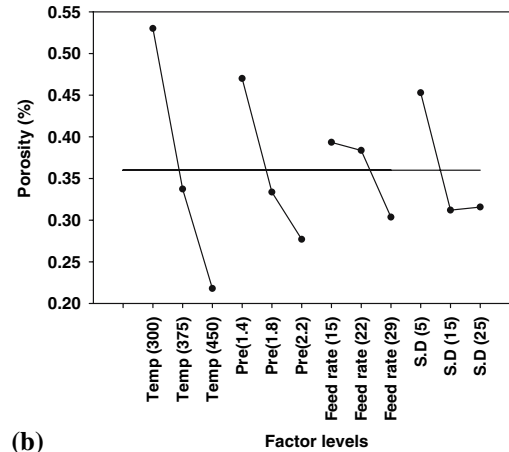
The X-ray diffraction patterns of the powder, as-coated specimen and vacuum treated specimen for experiment no 3 are shown in Fig. 4. Major differences in the patterns are not observed as expected. However, slight broadening of the peaks could be observed in the as-coated specimen. This could be attributed to the cold work experienced by the copper powder during the high-velocity impact onto the substrate. No evidence of any new phase, especially oxides of copper, could be found.

3.2 Effect of Process Parameters and Heat Treatment on Coating Properties

3.2.1 Porosity. Figure 5(a) shows the variation in coating porosity of the as coated as well as the heat treated specimens for all the nine experiments carried out as per Table 1. The porosity levels in the coatings are less than 1% even for the coatings deposited at the lowest gas pre-heat temperature (experiment no 1), clearly indicating



(a)



(b)

Fig. 5 (a) Porosity of copper coatings at various parameters and (b) factor effects plot for porosity of as coated specimens

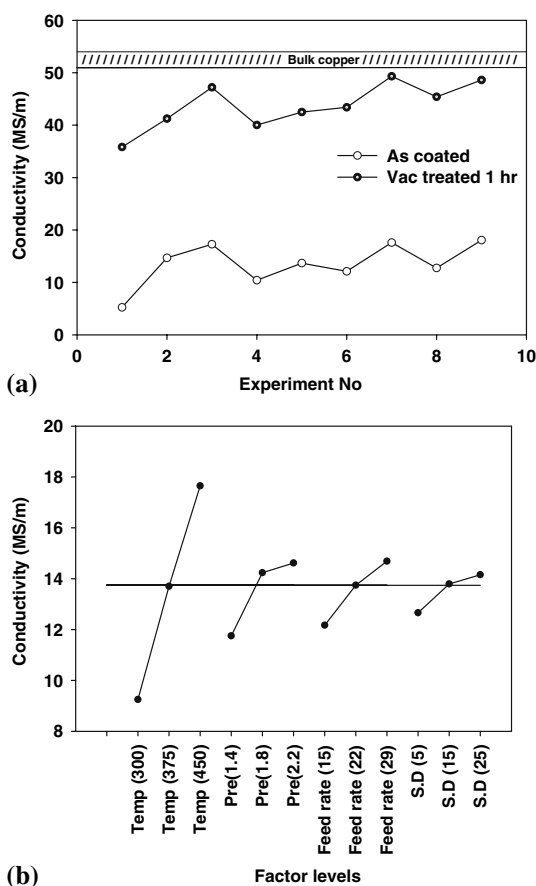
the capability of the cold spray process in achieving highly dense coatings. Porosity is found to decrease with vacuum treatment.

The factor effects plot for porosity of the as coated specimens is shown in Fig. 5(b). The plot clearly shows that the porosity decreases with increase in gas pre-heat temperature. The contribution of gas pre-heat temperature calculated based on Taguchi method (see Table 2) is overwhelmingly high compared to the other parameters. A very low stand off distance (5 mm) is found to yield high porosities. Porosity is also found to be slightly high at the highest stand off distance, while feed rate is found to have minimal influence. Similar trend was observed after heat treatment in vacuum for 1 h, where gas pre-heat temperature was again found to be the most dominating factor as shown in Table 2.

3.2.2 Conductivity. The variation of conductivity for all the nine experiments for as coated as well as, specimens heat treated at 300°C in vacuum for 1 h is as shown in Fig. 6(a). Conductivities of the as coated specimens are low. However, a substantial improvement in the conductivity is observed upon heat treatment and conductivities close to that of bulk annealed copper are achieved as confirmed by other studies (Ref 14, 15). Conductivity shows a dramatic increase with heat treatment under all the nine experimental conditions.

Table 2 Contribution of process parameters before and after heat treatment

Factor	Contribution, %					
	Conductivity		Microhardness		Porosity	
	As coated	Heat treated	As coated	Heat treated	As coated	Heat treated
Temperature	79.14	73.81	66.99	72.48	56.19	46.61
Pressure	10.88	4.11	2.53	0.42	23.04	14.93
Feed rate	7.26	4.60	26.00	11.29	5.68	7.45
Stand off dist	2.73	17.48	4.49	15.80	15.09	31.02

**Fig. 6** (a) Conductivity of copper coatings at various parameters and (b) factor effects plot for conductivity of as coated specimens

The factor effects plot for conductivity of the as coated specimens is shown in Fig. 6(b). The plot clearly shows that the conductivity increases with increase in gas pre-heat temperature. The coatings generated at the highest gas pre-heat temperature of 450 °C (Experiment nos: 3, 7, and 9) exhibit the highest conductivities. Similarly, the conductivities are high at higher gas inlet pressures. Also, the contribution of gas pre-heat temperature (see Table 2) calculated based on Taguchi method is again overwhelmingly high (79%) compared to the other parameters emphasizing the importance of gas pre-heat temperature in cold spray coating. Feed rate and stand off distance are found to have negligible effect on conductivity within the

present window. Similar contribution level for gas pre-heat temperature was observed after heat treatment as shown in Table 2. Level three for all the variables is found to be the local optimum parameter for coating conductivity before and after heat treatment. The global optimal value for gas pre-heat temperature and gas inlet pressure might be out of the window chosen for the present study. However, coating generation at higher gas pre-heat temperatures and higher gas inlet pressures may not be commercially viable and may also cause oxides to form resulting in lower conductivity.

3.2.3 Microhardness. The variation of coating microhardness for the as-coated and heat treated specimens from all the experiments is summarized in Fig. 7(a). These microhardness values are consistent with the results reported in earlier studies (Ref 12). The microhardness of the as-coated specimens is much higher than that of bulk copper and upon heat treatment, it decreases to around 85 HV_{0.1}.

The factor effects plot for microhardness of the as-coated specimens is shown in Fig. 7(b). Identical trend was observed in the heat treated specimens. The plot clearly shows that the microhardness increases with increase in gas pre-heat temperature. However, the coatings exhibited the maximum microhardness value at 1.8 MPa pressure and the microhardness decreased marginally at a higher pressure of 2.2 MPa. A very low stand off distance (5 mm) is found to be undesirable as observed in the case of conductivity. The contribution of gas pre-heat temperature (Table 2) is again overwhelmingly more before (66.99%) and after heat treatment (72.48%) compared to the other parameters. Unlike the earlier cases, feed rate is found to have reasonable effect on microhardness of the as-coated specimens.

4. Discussion

4.1 Particle Velocity and Gas Pre-Heat Temperature

Results presented in the last section clearly point to the fact that gas pre-heat temperature is the single most important process parameter affecting the properties of the cold spray copper coatings. It is well accepted that the velocity of the gas (air in the present case) at the nozzle exit, which in turn determines the velocity of the copper particles at the nozzle exit, is strongly influenced by gas pre-heat temperature. The question is whether the

observed influence of gas pre-heat temperature on coating properties can be explained on the basis of its influence on particle velocity per se or does gas pre-heat temperature have an additional influence through its effect on particle temperature.

As a first step towards answering the above question, the velocity of the copper particles at the nozzle exit has been computed using the empirical relationship formulated by Alkimov et al. (Ref 19), given below.

$$V_e = \frac{V_{ge}}{1 + 0.85 \sqrt{\frac{D}{X}} \sqrt{\frac{\rho_p V_{ge}^2}{P}}} \quad (\text{Eq 1})$$

where

$$V_{ge} = \sqrt{\gamma R T_e} \quad (\text{Eq 2})$$

and

$$T_e = \frac{T_0}{1 + \frac{\gamma-1}{2} M_e^2} \quad (\text{Eq 3})$$

In Eq 1-3, V_{ge} and V_e are the gas velocity and particle velocity at nozzle exit, respectively, D is the particle diameter ($D=22 \mu\text{m}$), X is the length of the diverging section of the nozzle ($X=102 \text{ mm}$), ρ_p is the powder

particle density ($\rho_p=8910 \text{ kg/m}^3$), P is the gas inlet pressure (experimental values in Pa), γ is the specific heat ratio ($\gamma=1.4$ for air), R is the gas constant and T_0 and T_e are the gas temperatures at the inlet and exit of the nozzle, respectively.

Earlier work by Dykhuizen and Neiser (Ref 9) has demonstrated that the particle velocity calculated using Eq. 1 is very close to the velocity predicted by detailed modeling studies. The velocity of the copper particles calculated using Eq. 1-3, for the nine experimental conditions utilized in the present study, are presented in Table 3. Using the data in Table 3, the variation of coating porosity, conductivity and microhardness with the particle velocity is presented in Fig. 8(a-c) for the as-coated condition. The numbers indicated against each data point in Fig. 8 represent the gas pre-heat temperature and gas inlet pressure associated with that particular experiment. It is clear from Fig. 8 that coating properties are largely determined by impact velocity, with microhardness and conductivity increasing with increasing particle velocity while the porosity obviously decreasing with increasing particle velocity. Higher particle velocity should induce greater deformation of the particle during its impact onto the substrate leading to higher hardness. Similarly, the greater deformation of the particles should lead to better bonding between the deformed particles leading to lower porosity. The increasing coating conductivity with increasing particle velocity also implies that the increase in conductivity due to better bonding and lower porosity more than offsets the decrease in conductivity due to increased deformation of the particle.

A very careful study of Fig. 8 also indicates that the data points pertaining to a gas pre-heat temperature of $450 \text{ }^\circ\text{C}$ result in improved properties (i.e., higher conductivity and hardness and lower porosity) in comparison to data points pertaining to a gas-inlet temperature of $300 \text{ }^\circ\text{C}$. If gas pre-heat temperature affected the coating properties only through its effect on impact velocity (as per Eq. 1-3), all the data points, irrespective of the gas pre-heat temperature, should have fallen randomly across the reference line. The observed additional effect of gas pre-heat temperature is obviously related to the higher temperature experienced by the particle with increasing gas pre-heat temperature during its flight through the nozzle. A higher particle temperature implies increased deformability and bonding leading to improved properties as observed experimentally.

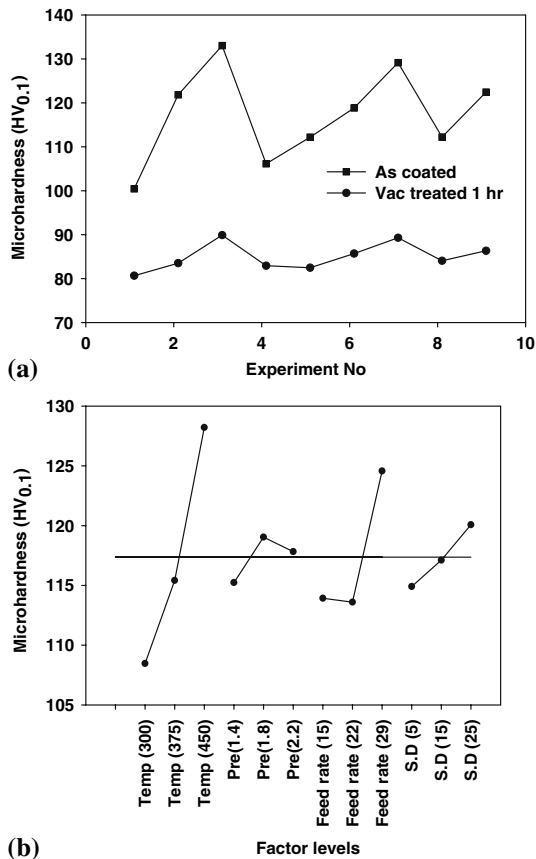


Fig. 7 (a) Microhardness of copper coatings at various parameters and (b) factor effects plot for microhardness of as coated specimens

Table 3 Particle velocity for different experiments

Experiment no	Temperature (T_0), $^\circ\text{C}$	Pressure (P), MPa	Velocity (V_e), m/s
1	300	1.4	455
2	375	1.8	498
3	450	1.4	484
4	300	1.8	481
5	375	2.2	520
6	300	2.2	501
7	450	1.8	513
8	375	1.4	471
9	450	2.2	537

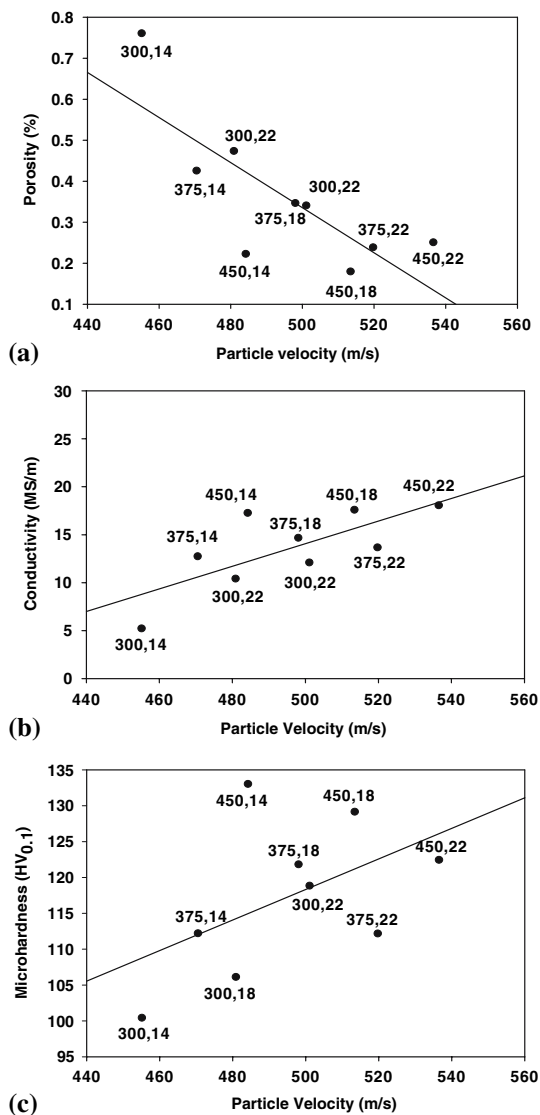


Fig. 8 Influence of particle velocity on coating (a) porosity; (b) conductivity; and (c) microhardness

In conclusion, it can be stated that the effect of gas pre-heat temperature on coating properties is largely manifested through its effect on particle velocity and its effect on coating properties through the effect on particle temperature though marginal cannot be neglected.

4.2 Relationship between the As-Coated Properties and the Coating Properties after Heat Treatment

In this section, we will look into the question as to whether the final coating properties after heat treatment have any relation to the as-coated coating properties.

In Fig. 9, the porosity, microhardness and conductivity of the coating after heat treatment (at 300 °C for 1 h) are compared with the properties prior to heat treatment. It is clear that even though the property values have changed

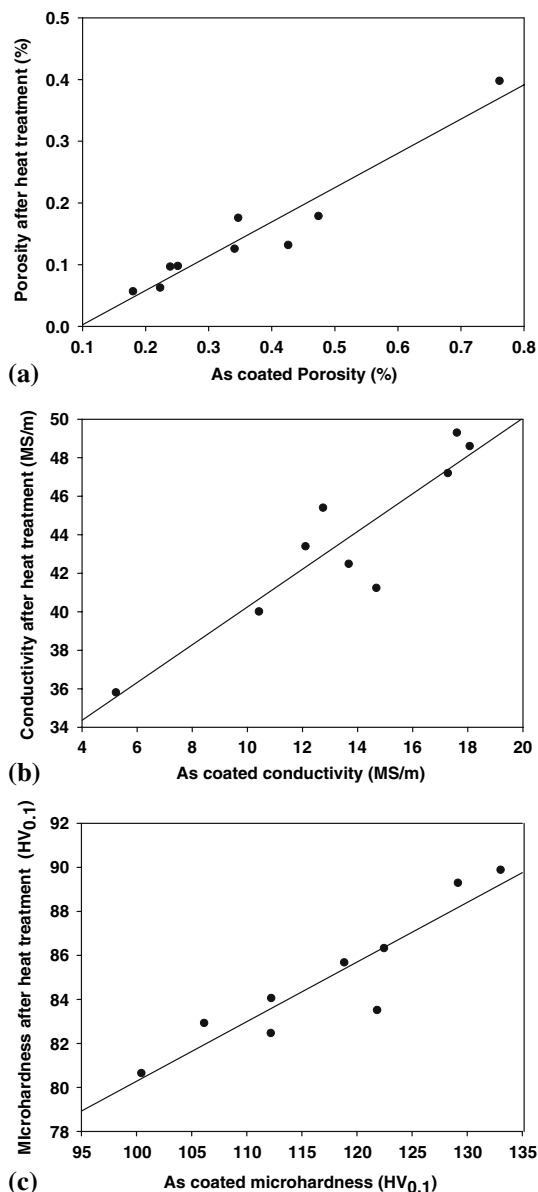


Fig. 9 Influence of heat treatment (300 °C 1 h) on coating (a) porosity; (b) conductivity; and (c) microhardness

substantially upon heat treatment, they still correlate extremely well with the as-coated properties prior to heat treatment. Thus, it can be concluded that the optimization of coating process parameters to obtain the best conductivity or hardness values (as done in the present study), is very important and necessary to get the best properties after heat treatment. Such a result, though somewhat surprising, can be rationalized on the basis that a combination of process parameters which result in a coating with poorly bonded particles and high porosity cannot be improved upon even after heat treatment. Heat treatment is more likely to anneal out the effect of cold work and reduce porosity only to a limited extent as our own experiments indicate.

4.3 Relative Contribution of Cold Work and Porosity to Coating Properties

4.3.1 Coating Conductivity. In order to evaluate the relative contributions of cold work and porosity to the conductivity of the copper coating, the variation of the conductivity of the copper coating in the as coated and heat treated conditions are compared as a function of porosity in Fig. 10(a). On the basis of the data in Fig. 10(a) and on the assumption that the conductivity of a fully annealed copper coating with no porosity, obtained by back extrapolation of the best-fit line pertaining to vacuum treated coating in Fig. 10(a) to zero porosity (calculated to be 49.2 (MS/m)), the relative contributions of cold work and porosity in decreasing the conductivity can be easily evaluated. The result, presented in Table 4, indicates that cold work is responsible for almost 88% of the decrease in conductivity with the porosity accounting

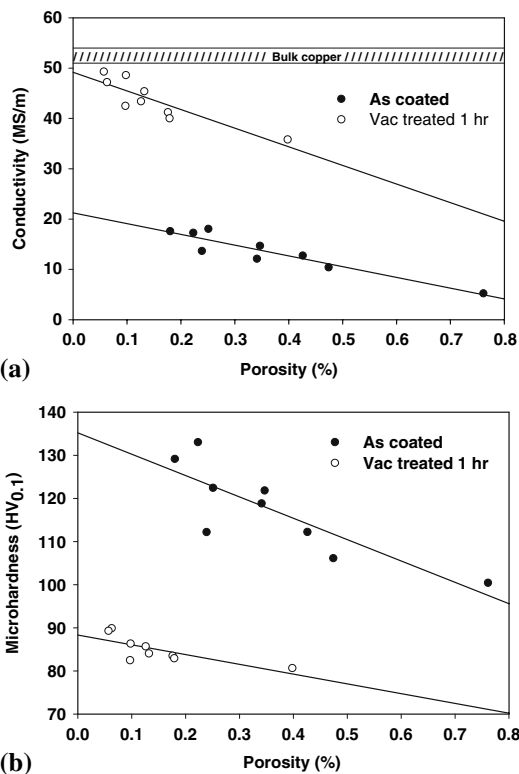


Fig. 10 Effect of coating porosity on (a) conductivity and (b) microhardness

for the balance, at a porosity level of 0.1%. In contrast, at a porosity level of 0.8%, almost 2/3rd of the decrease in conductivity is due to porosity and only 1/3rd is due to cold work (see Table 4). Such a dramatic effect of porosity is not only due to the fact that the porosity level has increased (from 0.1% to 0.8%) but also due to the fact that the coating process conditions which led to high porosity also induced the least cold work due to particle deformation since the particle velocities were the lowest under these conditions (see Table 3).

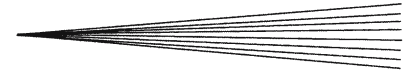
It is also obvious from Fig. 10(a) and Table 4 that the increase in porosity from 0.1% to 0.8% causes a dramatic decrease in conductivity of the coating. Such a dramatic decrease cannot be explained on the basis of existing models, which have looked at the influence of porosity in a bulk material on its conductivity (Ref 20). In fact these models predict that even a porosity level of 0.8% can decrease the conductivity of a material from its bulk value by only 0.5% as opposed to an experimentally observed 60% decrease in conductivity. The above discrepancy can be understood on the basis that cold spray coating process results in a layered structure with the porosity (mostly formed on the inter particle boundaries) being also layered and pore shape exhibiting elongated morphology. Under such circumstances, as shown by Nakamura et al. (Ref 21), the effect of porosity can be more substantial than dictated by their volume fraction. In addition, poor bonding between the deformed particles in the coating, though not counted as porosity can influence the coating conductivity.

4.3.2 Coating Hardness. The relative contribution of cold work and porosity on coating hardness has been evaluated using an approach similar to that adopted for coating conductivity, using the data from Fig. 10(b). Unlike the case of conductivity, porosity and cold work have opposing effects on microhardness, with porosity resulting in decrease in hardness while cold work resulting in increase in hardness. The effect of cold work on hardness is overwhelmingly high at low-porosity levels, as evident from a contribution of around 95% at 0.1% porosity (see Table 4). In contrast to the effect on conductivity, the contribution of cold work is significant even at high porosity levels (58% at 0.8% porosity). From such an overwhelming contribution of cold work it is obvious that the hardening due to cold working more than offsets the decrease in hardness due to porosity.

A porosity level of 0.8% resulted in around 20% decrease in hardness as against a 4% decrease predicted by existing models (Ref 22). As discussed earlier (Ref 21), this could be rationalized by the fact that some amount of

Table 4 Contribution of porosity and cold work to conductivity and microhardness

Porosity	Conductivity, MS/m		Contribution, %		Microhardness, HV _{0.1}		Contribution, %	
	As coated	Heat treated	Porosity	Cold work	As coated	Heat treated	Porosity	Cold work
0.1	19.1	45.5	12.3	87.7	130.2	86.0	4.9	95.1
0.3	14.8	38.1	32.3	67.7	120.3	81.5	14.9	85.1
0.5	10.5	30.7	47.9	52.1	110.4	77.0	25.3	74.7
0.8	4.1	19.6	65.7	34.3	95.6	70.2	41.6	58.4



energy during indentation would be utilized in bridging the gap between the layers of the coating.

5. Summary and Conclusions

The present work aims to study the effect of cold spray process parameters on coating properties using Taguchi—design of experiments approach, to ultimately optimize the process parameters for copper. Heat treatment was carried out to study the extent to which the as coated properties and thereby optimization of coating process is relevant vis-à-vis post coating heat treatment, in achieving superior properties.

- Gas pre-heat temperature is found to be the most dominant factor in influencing coating properties followed by gas inlet pressure and stand off distance; while, powder feed rate is found to have the least influence within the parameter window chosen for the present study.
- Electrical conductivity of the as coated specimens is found to be low whereas the hardness is high compared to bulk copper due to heavy cold working during coating formation. However, conductivities close to that of bulk annealed copper are achieved upon heat treatment with a decrease in hardness.
- The porosity levels achieved by cold spray are less than 1% and show a monotonic decrease upon heat treatment and the lowest porosity levels are achieved by vacuum treatment.
- Coating properties are not completely determined by particle velocity and gas pre-heat temperature is found to have an influence on coating properties over and above its known effect on particle velocity.
- The coating parameters have significant influence on post treatment coating properties as well, emphasizing the importance of parameter selection in obtaining better coatings.
- Electrical conductivity is found to be a strong function of porosity and shows a linear correlation with porosity for the as coated as well as the heat treated samples and similar linear correlation was observed between hardness and porosity.
- The contribution of cold work to conductivity and microhardness is overwhelmingly more at low porosities and the observed discrepancy in the effect of porosity on coating properties could be attributed to the layered structure of the coatings.
- It can be inferred from the present study that cold spray offers an excellent opportunity to obtain high-conductivity copper coatings by appropriate selection of process parameters and treatment conditions.

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References

1. A.P. Alkimov, A.N. Papyrin, V.F. Kosarev, N.J. Nesterovich, and M.M. Shuspanov, "Gas Dynamic Spraying Method for Applying a Coating," U.S. patent 5,302,414, Apr. 12, 1994
2. A.O. Tokarev, Structure of Aluminium Powder Coatings Prepared by Cold Gas Dynamic Spraying, *Met. Sci. Heat Treat.*, 1996, **38**(3-4), p 136-139
3. R.C. Dykhuizen, M.F. Smith, D.L. Gilmore, R.A. Neiser, X. Jiang, and S. Sampath, Impact of High Velocity Cold Spray Particles, *J. Thermal Spray Technol.*, 1999, **8**(4), p 559-564
4. H. Kreye and T. Stoltenhoff, Cold Spraying - A Study of Process and Coating Characteristics, *Thermal Spray: Surface Engineering via Applied Research*, C.C. Berndt, Ed. (Montreal, Quebec, Canada), ASM International, May 8-11, 2000, p 419-422
5. J. Karthikeyan and C.M. Kay, Cold Spray Technology: An Industrial Perspective, *Thermal Spray: 2003: Advancing the Science and Applying the Technology*, B.R. Marple and C. Moreau, Ed. (Orlando, FL), ASM International, May 5-8, 2003, p 117-121
6. T. SteenkisteVan and J.R. Smith, Evaluation of Coatings Produced via Kinetic and Cold Spray Processes, *J. Thermal Spray Technol.*, 2004, **13**(2), p 274-282
7. T. Stoltenhoff, H. Kreye, and H.J. Richter, An Analysis of the Cold Spray Process and its Coatings, *J. Thermal Spray Technol.*, 2002, **11**(4), p 542-550
8. J. Haynes and J. Karthikeyan, Cold Spray Application for Upper Stage Rocket Engine Design, *Thermal Spray: 2003: Advancing the Science and Applying the Technology*, B.R. Marple and C. Moreau, Ed. (Orlando, FL), ASM International, May 5-8, 2003, p 19-26
9. R.C. Dykhuizen and R.A. Neiser, Optimizing the Cold Spray Process, *Thermal Spray: 2003: Advancing the Science and Applying the Technology*, B.R. Marple and C. Moreau, Ed. (Orlando, FL), ASM International, May 5-8, 2003, p 79-83
10. R.C. McCune, W.T. Donion, O.O. Popoola, and E.L. Cartwright, Characterization of Copper Layers Produced by Cold Gas Dynamic Spraying, *J. Thermal Spray Technol.*, 2000, **9**(1), p 73-82
11. R.C. McCune, R.P. Cooper, and O.O. Popoola, Post-Processing of Cold Spray Deposits of Copper and Iron, *Thermal Spray: Surface Engineering via Applied Research*, C.C. Berndt, Ed. (Montreal, Quebec, Canada), ASM International, May 8-11, 2000, p 905-908
12. C. Borchers, F. Gartner, T. Stoltenhoff, and H. Kreye, Formation of Persistent Dislocation Loops by Ultra High Strain Rate Deformation During Cold Spraying, *Acta Mater.*, 2005, **53**(10), p 2991-3000
13. F. Gartner, T. Stoltenhoff, J. Voyer, H. Kreye, S. Riekehr, and M. Kocak, Mechanical Properties of Cold Sprayed and Thermally Sprayed Copper Coatings, *Surf. Coat. Technol.*, 2006, **200**, p 6770-6782
14. T. Stoltenhoff, C. Borchers, F. Gartner, and H. Kreye, Microstructures and Key Properties of Cold Sprayed and Thermally Sprayed Copper Coatings, *Surf. Coat. Technol.*, 2006, **200**, p 4947-4960
15. W.-Y. Li, C.-J. Li, and H. Liao, Effect of Annealing Treatment on the Microstructure and Properties of Cold Sprayed Cu Coating, *J. Thermal Spray Technol.*, 2006, **15**(2), p 206-211
16. G. Taguchi and S. Konishi, Taguchi Methods: Orthogonal Arrays and Linear Graphs, . . ., ASI press, Dearborn, MI, 1987
17. R.C. Dykhuizen and M.F. Smith, Gas Dynamic Principles of Cold Spray, *J. Thermal Spray Technol.*, 1998, **7**(2), p 205-212
18. J. Karthikeyan, C.M. Kay, J. Lindeman, R.S. Lima, and C.C. Berndt, Cold Spray Processing of Titanium Powder, *Thermal Spray: Surface Engineering via Applied Research*, C.C. Berndt, Ed. (Montreal, Quebec, Canada), ASM International, May 8-11, 2000, p 255-262

19. A.P. Alkimov, V.F. Kosarev, and S.V. Klinkov, The Features of Cold Spray Nozzle Design, *J. Thermal Spray Technol.*, 2001, **10**(2), p 375-381
20. E. Botcharova, J. Freudenberger, and L. Schultz, Mechanical and Electrical Properties of Mechanically Alloyed Nanocrystalline Cu-Nb Alloys, *Acta Mater.*, 2006, **54**(12), p 3333-3341
21. T. Nakamura, G. Qian, and C.C Berndt, Effect of Pores on Mechanical Properties of Plasma-Sprayed Ceramic Coatings, *J. Am. Ceram. Soc.*, 2000, **83**(3), p 578-584
22. J. Luo and R. Stevens, Porosity-dependence of Elastic Moduli and Hardness of 3Y-TZP Ceramics, *Ceram. Inter.*, 1999, **25**, p 281-286