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Influence of Spray Materials and Their Surface Oxidation on the Critical Velocity in Cold Spraying

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The critical velocity is an important parameter in cold spraying, which determines the deposition efficiency under a given spray condition. The critical velocity depends not only on materials types, but also on particle temperature and oxidation conditions. In the present paper, three types of materials including copper, 316L stainless steel, Monel alloy were used to deposit coatings by cold spraying. The critical velocities of spray materials were determined using a novel measurement method. The oxygen content in the three powders was changed by isothermal oxidation at ambient atmosphere. The effect of oxygen content on the critical velocity was examined. It was found that the critical velocity in cold spray was significantly influenced by particle oxidation condition besides materials properties. The critical velocity of Cu particles changed from about 300 m/s to over 610 m/s with the change of oxygen content in powder. It is evident that the materials properties influence the critical velocity more remarkable at low oxygen content than at high oxygen content. The results suggest that with a severely oxidized powder the critical velocity tends to be dominated by oxide on the powder surface rather than materials properties.

Keywords coating, cold spray, critical velocity, oxidation

1. Introduction

Cold spraying is a new emerging coating technology by which spray particles in a solid state are deposited via plastic deformation on a substrate at a high velocity and a temperature that is much lower than the melting point of the starting powder (Ref 1). During the last two decades, research and development on cold spray process and technology have been much concerned, which leads to significant progresses on both coating process and technologies (Ref 2-5). With such progresses, most metals and their alloys can be deposited by cold spraying (Ref 2-4), and cermets including nanostructured WC-Co (Ref 6) can also be deposited, and fine ceramics particles can be deposited into a substrate to form a coating under vacuum conditions (Ref 7).

The most important parameter of a spray material particle in cold spray process is its critical velocities (Ref 1, 8-10). It becomes known that three different critical velocities exist for a given material and a substrate. One critical velocity is for the particle deposition on a substrate, which possibly changes significantly with the combinations of spray materials with substrates. The other critical velocity is for a continuous build-up of a coating, which corresponds to the critical velocity for a spray material impacting on the substrate of the same compositions as the powder. The third one is the up-limit velocity for particle deposition, over which a strong erosion is generated (Ref 11).

Therefore, only particles having a velocity between the critical velocity for coating build-up and the up-limit velocity contribute coating build-up. With most spray materials the up-limit velocity is higher than 1000 m/s (Ref 11). Therefore, practically the critical velocity is an essential parameter for coating build-up. Only the particles having reached a velocity larger than the critical velocity can be deposited to produce coating. The particle with a velocity lower than the critical velocity may lead to the erosion of substrate. Therefore, the critical velocity and particle velocity prior to impact determine the deposition efficiency under a given spray condition. It was confirmed that the critical velocity depends mainly on spray materials type (Ref 1, 12) and substrate (Ref 12-14), particle temperature and oxidation conditions (Ref 9, 15). It was also argued that the critical velocity is influenced by particle size (Ref 11). Copper has been used as a typical

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material for cold spraying by many investigators. Accordingly, the critical velocity of copper particles has been reported by several investigators (Ref 1, 9-18). However, there was a large discrepancy among the critical velocities for copper particle deposition obtained by different investigators, for example, 470-500 m/s (Ref 12, 16), 550-570 m/s (Ref 13) and 640 m/s (Ref 10) can be found in different reports. From our previous study, this large discrepancy can be attributed to the difference in oxygen content of the copper powder (Ref 9). Therefore, it is necessary to investigate the influence of oxygen content in spray powder on the critical velocity for particle deposition.

The estimation of the critical velocity for coating built-up can be estimated through direct experiment determination and numerical simulation estimation. Based on the numerical simulation of plastic deformation behavior involved in high-velocity particle impact, Assadi et al. (Ref 8) proposed a theoretical approach to estimate the critical velocity through utilizing the lowest velocity causing the adiabatic shear instability of spray particle and summarized a simple formula related the critical velocity to material properties and temperature. This numerical approach has been employed by several other groups and similar results were obtained (Ref 19, 20). However, our previous simulation revealed significant effect of meshing size during numerical simulation on the critical velocity (Ref 9). Therefore, such effect should be taken into consideration when the numerical simulation approach is employed to estimate the exact critical velocity. On the other hand, with experimental testing, different approaches were employed by different investigators (Ref 1, 9, 10, 21). Owing to a wide particle size distribution of a practical spray material and subsequent wide particle velocity distribution, it is difficult to measure accurately the critical velocity through onset of particle deposition. It is only possible to set experimental condition to obtain a particle velocity distribution in which particle velocity is inversely proportional to particle size, which can be measured properly, to make reliable measurement of the critical velocity along with particle size distribution as suggested by Gärtner et al. (21).

In the present paper, a novel experimental method was used to measure the critical velocity based on the theoretical relationship between relative deposition efficiency and critical velocity at different spray angles (Ref 22). Different materials including copper, 316L stainless steel and Monel alloy in different oxidation conditions were used to deposit coatings by cold spray process to reveal the effect of oxidization conditions of powders on the critical velocity.

2. Materials and Experiments

2.1 Materials

Copper, 316L stainless steel and Monel alloy powders were used in different oxygen content levels. Each powder was employed in three to four different oxygen content levels determined by the oxygen determinator (RO-316, LECO, USA). The powder was received at the lowest oxygen content level and then oxidized in ambient atmosphere at high temperature to different oxygen contents shown in Table 1. The powders used were produced by gas atomization process and exhibited a spherical morphology as shown in Fig. 1. The particle size distribution of each powder was measured quantitatively by laser diffraction sizer (MASTERSIZER 2000, Malvern, UK).

Table 1 Particle size and oxygen content levels of materials used

Materials	Mean particle size, μm	Oxygen content level, wt.%
Cu	56.2	0.02, 0.14, 0.38
316L	9.6	0.086, 0.166, 0.195
Monel alloy	20.8	0.016, 0.085, 0.108

2.2 Test Procedures

2.2.1 Determination of Particles Parameters. The method used to measure the critical velocity has been described in detailed in a previous paper (Ref 9). The following is a simple description of the measuring procedures. The size distribution was fitted by the modified Rosin-Rammler formula (Eq 1) using experimental data to obtain the relation between the cumulative mass fraction and particle diameter (Ref 9).

$$f_m = \left\{1 - \exp\left[-\left(\frac{d_p - d_{\min}}{d_0}\right)^m\right]\right\} \cdot \left\{1 - \exp\left[-\left(\frac{d_{\max} - d_{\min}}{d_0}\right)^m\right]\right\}^{-1} \cdot 100\% \qquad \text{(Eq 1)}$$

where $d_{\max}$ and $d_{\min}$ are the maximum and minimum cut-off diameters of the particles, which means that the size of all particles ranges from $d_{\min}$ to $d_{\max}$. f_m is the mass fraction of all powders having a diameter less than the particle size d_p, m and d_0 are constants.

The comparison of the fitted relation with the observed one is shown in Fig. 2 for three powders. It was convinced that the modified Rosin-Rammler formula represented well the size distribution of powders used in this study. As a result, all parameters including $d_{\max}$, $d_{\min}$, m and d_0 can be obtained before test.

2.2.2 Relationship Between Particle Velocity and Particle Size. According to our previous numerical simulation results (Ref 23), the particle velocity (V_p) can be expressed empirically by the following equation in the case that gas velocity is larger than the highest particle velocity as far as the maximum particle velocity is less than that of accelerating gas.

$$V_p = \frac{k}{d_p^n} \qquad \text{(Eq 2)}$$

where n and k are constants. Those two constants were determined by simulation using spraying condition prior deposition.

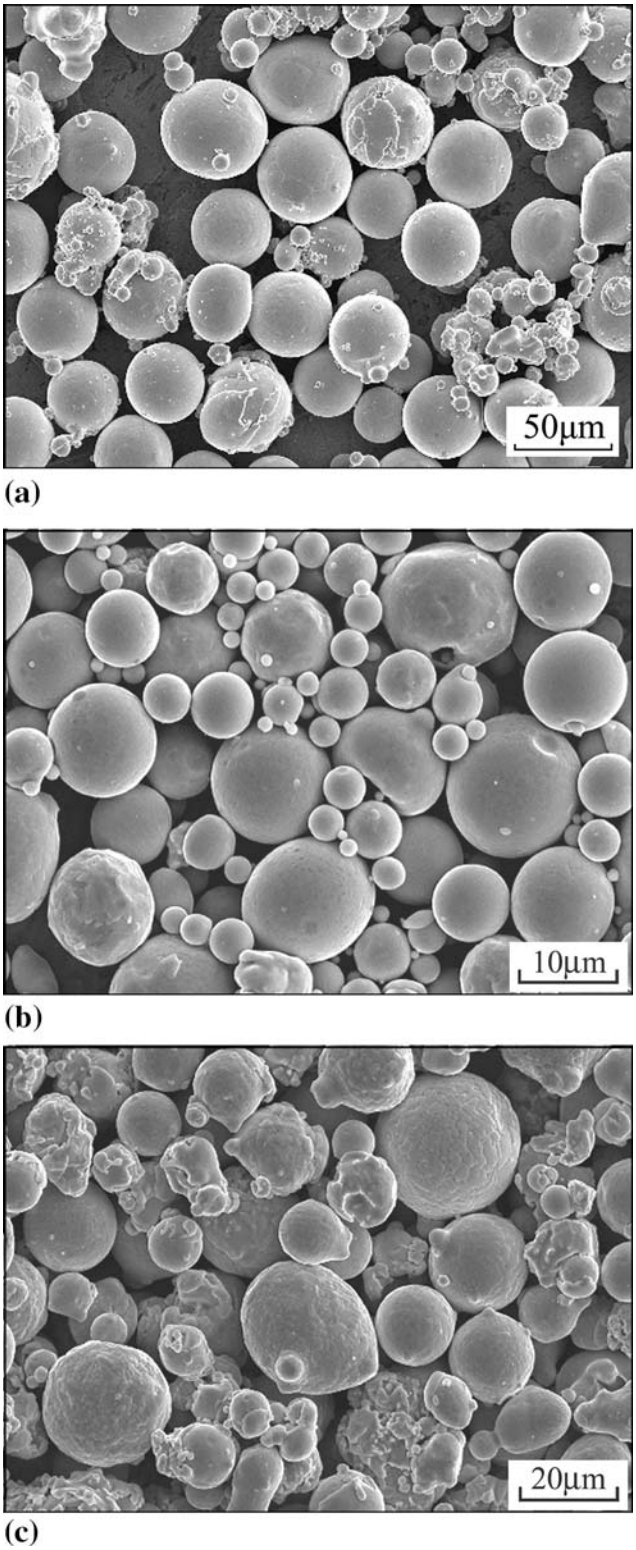

Fig. 1 Morphology of three different powders: (a) copper, (b) 316L stainless steel, and (c) Monel alloy

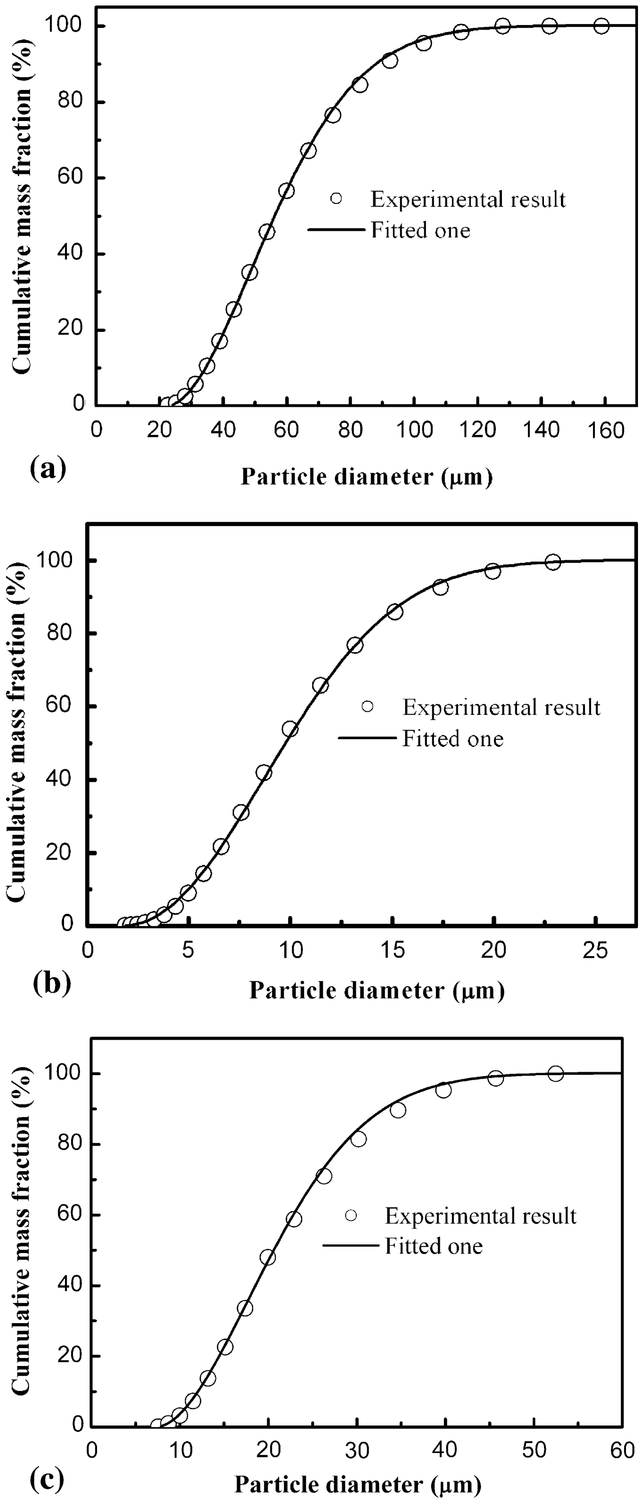

Fig. 2 Particle size distribution of three powders in comparison of the observed (circle points) with fitted one (solid lines): (a) copper, (b) 316L stainless steel, and (c) Monel alloy

2.2.3 Coating Deposition. Cold spray deposition was carried out using a cold spray system developed in Xi'an Jiaotong University. A converging/diverging de Laval nozzle of a conical shape was adopted. The throat diameter was 2 mm. Selection of this spray distance ensured the validity of Eq 2. The expansion ratio of the nozzle was

9 and the downstream length from the throat to the exit was 100 mm. Cu and stainless steel plate in the dimensions of 45 × 15 × 3 mm was used as substrates being grit-blasted with 24-mesh alumina grits prior to spray. The substrate was fixed at a specially designed fixture which was used to measure the relative deposition efficiency of spray particle at different angles with respective to normal impact (Ref 22) as shown in Fig. 3. The standoff distance was 20 mm from the nozzle exit to the centre of substrate surface. During deposition, the spray gun was manipulated by a robot and traversed at a speed of 80 mm/s across the substrate and all specimens at different angles were prepared in the same one pass. The processing parameters of cold spraying are shown in Table 2. The weight gain of each specimen after deposition was measured using a balance with the precision of 0.1 mg. The relative deposition efficiency was evaluated by the ratio of the weight gains of the specimens placed at different tilting angles to that of the specimen sprayed at the normal angle. As a result, the experimental relationship between the relative deposition efficiency and spray angle was obtained. Because in the theoretical relationship between the relative deposition efficiency and spray angle which was given in Ref 9, only one parameter, i.e., critical velocity, is unknown except those parameters in Eq 1 and 2, the critical velocity was determined through fitting the theoretical relation with experimental data as described more detailed elsewhere (Ref 9).

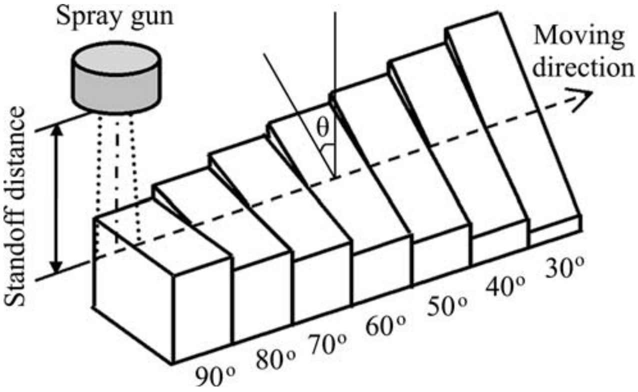

Fig. 3 Schematic diagram of the substrate fixture for spraying at different angles

Table 2 Processing parameters in cold spraying

Materials	Accelerating gas Type	Pressure, MPa	Temperature, °C
Cu	N_2	2.0	265
316L	He	1.5	495
Monel alloy	He	2.0	530

3. Results

The measurement yielded a critical velocity of 610 m/s for copper powder of an oxygen content of 0.38 wt.%. Gilmore et al. (Ref 10) obtained a critical velocity of 640 m/s for Cu powder containing an oxygen content of 0.336 wt.%. With copper powder of oxygen content 0.14 wt.%, a critical velocity of 550 m/s was obtained. Stoltenhoff et al. (Ref 13) reported a critical velocity of 570 m/s for copper powder of oxygen content of 0.1 wt.%. In a recent report by the same group author, a critical velocity of 550 m/s was given for copper powder of oxygen content of 0.2 wt.% (Ref 18). With a copper powder of oxygen content of 0.1 wt.%, Van Steenkiste et al. (Ref 12) reported a critical velocity of 470 m/s. In comparing those reported data with the data measured in this study, therefore, it is clear that the result obtained in this study agreed well with those reported (Ref 10, 12, 18). All those results clearly indicate that oxygen content influences significantly the critical velocity. Using a copper powder of 0.02 wt.% oxygen content, a critical velocity of 310 m/s was obtained in the study. This result is much lower than those reported. Figure 4 shows the effect of oxygen content in copper powder on the critical velocity. It was clearly found that the critical velocity in cold spray was significantly influenced by particle oxidation condition. With the increase of oxygen content in copper powder from 0.02 to 0.14 wt.% the critical velocity was rapidly increased from 310 to 550 m/s.

With stainless steel powder and nickel-based Monel alloy, it was also found that the oxygen content influenced significantly the critical velocity for particle deposition in cold spraying. Figure 5 shows the test results in terms of effect of oxygen content in 316L stainless steel powder on the critical velocity. The critical velocity of 316L stainless steel powder of an oxygen content of 0.086 wt.% was 582 m/s. With the increase of the oxygen content in 316L powder to 0.166 wt.% through isothermal oxidation, the

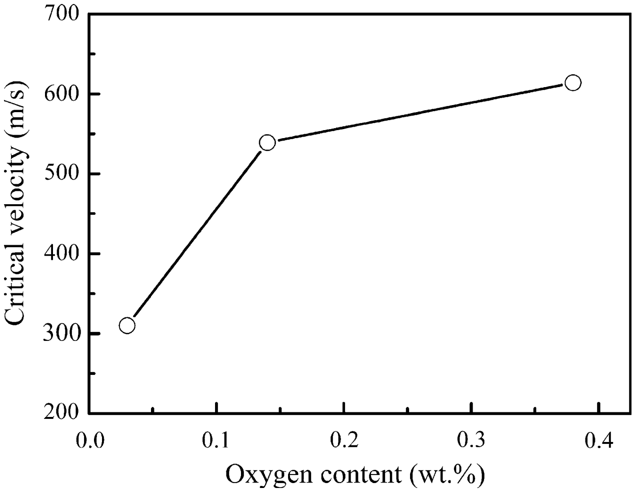

Fig. 4 Effect of oxygen content of copper powder on its critical velocity for deposition in cold spraying

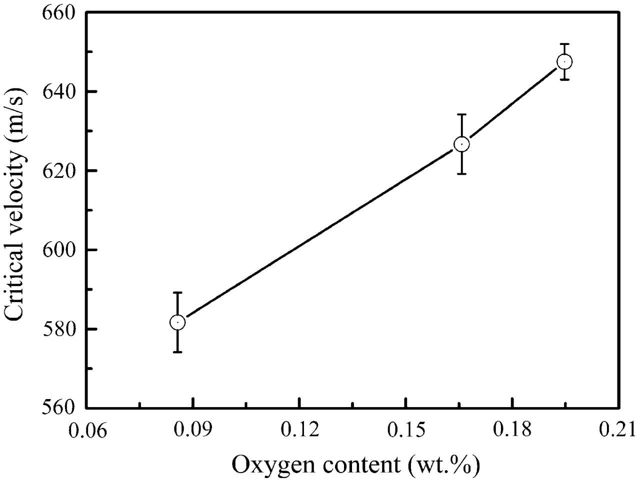

Fig. 5 Effect of oxygen content of 316L stainless steel powder on its critical velocity for deposition in cold spraying

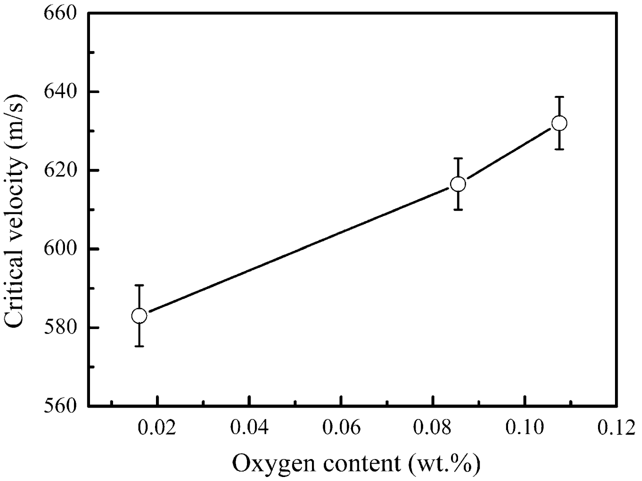

Fig. 6 Effect of oxygen content of Monel alloy powder on its critical velocity for deposition in cold spraying

critical velocity was increased to 627 m/s. As powder oxygen content was further increased to 0.195 wt.%, the critical velocity reached 648 m/s.

The effect of oxygen content in nickel-based Monel alloy on the critical velocity is shown in Fig. 6. It can be clearly seen that with powder oxygen content increasing from 0.016 to 0.108 wt.% the critical velocity was increased from 583 to 632 m/s. The results clearly indicated that a high oxygen content in powder results in a high critical velocity.

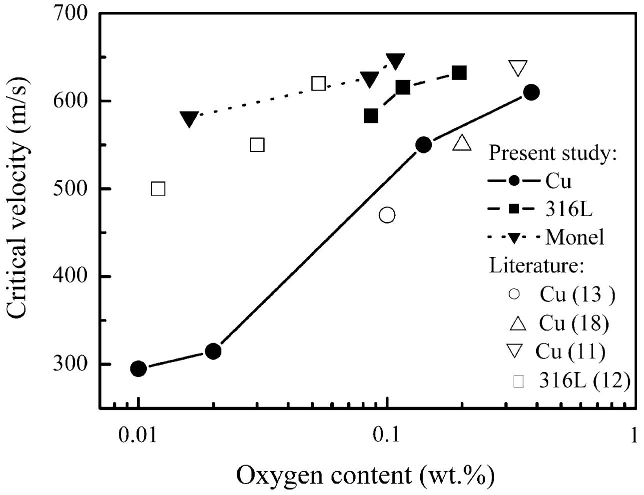

Fig. 7 Effect of oxygen content on the critical velocity of different spray materials. The numbers in the parenthesis represent the references

4. Discussion

Figure 7 summarizes all data in terms of effect of oxygen content on the critical velocity for three materials used in the present study. For comparison, some data reported in the literature for copper (Ref 10, 12, 18) and 316L stainless steel were also plotted (Ref 11). With those data of the critical velocity reported for 316L stainless steel in the paper (Ref 11), the oxygen contents of the same spray powders reported in the paper (Ref 24) were adopted.

Despite high critical velocity reported for copper in the literature, a low critical velocity of about 300 m/s was obtained for copper of low oxygen content. Schmidt et al. (Ref 11) recently reported a critical velocity of about 250 m/s for copper ball of 20 mm in diameter, determined through copper ball impact on copper substrate. It can be considered that such a result confirmed the fact that a low critical velocity of about 300 m/s for pure copper is reasonable. The dependency of the critical velocity on materials is evident in particular when the oxide content of metal powder is low. However, it is also evident that with the increase of oxygen content in powder the critical velocity of different materials tends to drop into a narrow range compared with those at low oxygen content. This fact means that when metal alloy powders are oxidized severely the difference in the critical velocity resulting from materials type becomes less significant.

Under the same oxidation conditions, the oxygen content of the powder of the identical chemical compositions will be increased with the decrease of particle size owing to the large specific surface area of small powder (Ref 25, 26). Based on the effect of the oxidation on the critical velocity shown above, it seems reasonable that the critical velocity of spray powder increases with the decrease of particle size as reported by Schmidt et al. (Ref 11). However, our previous experiment showed that under the same oxidation condition, the critical velocity is influenced little by particle size (Ref 9). The comparison of the critical velocity of about 300 m/s obtained for copper powder of a mean diameter of 20 μm (Ref 9) with a critical velocity of about 250 m/s for a Cu ball in a diameter of 20 mm (Ref 11) provides reasonable evidence for limited influence of particle size on the critical velocity.

Temples et al. (Ref 25) characterized the oxygen contents and oxide thickness of three types of MCrAlY powders in different particle size ranges. The powders were produced by inert gas atomization. It was shown that the oxygen content increases with the decrease of particle size. However, their AES analysis results revealed that the oxide thickness on the surface of individual powders is the same in a particle size range from −90/+75 μm to −10 μm. Therefore, the independency of the critical velocity of spray powder on particle size implies that the influence of oxide content on the critical velocity obtained in the study represents the effect of oxide scale thickness rather than the simple oxygen or oxide content. The examination of cross-sectional microstructure of the stainless steel powders and Monel powders before and after oxidation showed no difference. The analysis of chemical compositions within powders both in the as-received state and oxidized state by EDS also exhibited the comparable results without the trace of oxygen within the resolution of EDS analysis. Therefore, further analysis of the oxide scale thickness becomes necessary.

The present results indicate that with the same powder the progress of the oxidation leads to the increase of the critical velocity and consequently the decrease of deposition efficiency during spraying. It was also clear that at high oxygen content the critical velocity of three types of powders tended to become close despite the significant difference in the critical velocity at low oxygen content. Those results suggest that when metal powder is oxidized to certain degree the critical velocity will be dominated by oxide scale thickness rather than the materials themselves. This is possibly attributed to the fact that the bonding formation between cold-sprayed particles and the previously deposited particles surface requires to break and extrude the oxide scale from the interface on impact. At a high oxygen content being equal to a thick oxide scale, the same order of impact energy is required to break the oxide scales on particle surface of different materials. The detailed modeling and experimental investigations are further necessary to clarify the effect of thick oxide scale on the critical velocity and particle deposition behavior.

The critical velocity determines particle deposition behavior and deposition efficiency together with particle velocity distribution. For a certain spray condition, the deposition efficiency will be increased with the decrease of the critical velocity. Therefore, it is necessary to use metallic materials of low oxygen content to achieve high deposition efficiency. The metallic materials generally tend to be oxidized gradually as they are in stock in ambient atmosphere. Subsequently, the oxygen content and consequently critical velocity will be increased with stocking time of powder. This means that with the same lot of metal powder the coating deposition behavior will be changed with the time in stock. As a result, the consistency in coating deposition cannot be achieved owing to the gradual oxidation of metal powder with time. Therefore, the present result also revealed the importance to monitor the oxygen content of metal powder for cold spraying.

5. Conclusions

The critical velocity in cold spray was measured for three metal alloys at different oxidizing conditions. The results showed that the critical velocity was significantly influenced by particle oxidation condition besides materials properties. With copper powder the critical velocity changed from about 310 m/s at an oxygen content of 0.02 wt.% to 610 m/s at an oxygen content of 0.38 wt.%. With nickel-based Monel alloy, the critical velocity was increased from 583 to 632 m/s as the oxygen content was changed from 0.016 to 0.108 wt.%. It is evident that the materials properties influence the critical velocity more remarkably at a low oxygen content level than at a high oxygen content level. The results suggest that with a severely oxidized powder the critical velocity tends to be dominated by oxide on the powder surface.

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