INVESTIGATION OF MICROWAVE SINTERING ON HIGH VELOCITY PARTICLE CONSOLIDATION COATINGS

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ABSTRACT

The effects of microwave sintering on the properties of various coatings deposited by High Velocity Particles Consolidation (HVPC) process (also known as cold spray) were investigated to see whether microwave sintering could be a viable post process to improve HVPC coatings. The coating materials studied were pure nickel, pure cobalt, and 316L stainless steel, deposited on a steel substrate. After microwave sintering, the coating materials were assessed in terms of hardness, microstructure, porosity, and interfacial diffusion. The sintered nickel and stainless steel samples showed relatively large reduction in hardness when compared to as sprayed samples as a result of stress relief and recrystallization, which was confirmed by the microstructure images. Sintered cobalt samples showed slight change in hardness due to the presence of a metastable phase. Grain coarsening was also observed as the sintering temperature increases. Porosity in the coatings was determined by the aid of image analyzing software. The lower temperature sintered samples showed higher porosity percentage than the as sprayed sample, which would be a result from pore coalescence and incomplete sintering; however, the porosity tended to decrease as the sintering temperature increases. Energy Dispersive Spectroscopy (EDS) results showed that there was inter-diffusion occurring across the coating/substrate interface in the sintered samples, which is desired to improve the coating adhesion.

KEYWORDS: coating, cold spray, high velocity particle consolidation, microwave, sintering, nickel, stainless steel, cobalt

1. INTRODUCTION

High Velocity Particle Consolidation (HVPC) or cold spray process is a novel thermal spray technique, which uses high velocity rather than high temperature to deposit coatings. In HVPC process, coating particles are accelerated by supersonic gas jet toward the substrate. When the particles impinge on the substrate surface, they plastically deform and adhere to the substrate surface. This process can produce a dense coating layer with less thermal effects in the coating and substrate than conventional thermal spray processes. However, the coatings obtained from this process are subjected to high residual stress and near-zero ductility condition; as a result, a subsequent heat treatment is required for some coating materials to retrieve ductility and remove stresses.1) Microwave sintering of metallic materials was proved possible in 1999.2) Owing to its rapid heating rate, microwave heating consumes less energy, provides short processing time, and eliminates undesirable excessive temperature effects to the material during heating and cooling cycles. Therefore, the materials processed in microwave furnaces exhibit superior quality with high energy efficiency and better economics.2) Due to these benefits, microwave sintering was selected in the work as a possible post process to improve the HVPC coatings. In this study, the effect of microwave sintering to the properties of HVPC coatings was investigated and evaluated in terms of hardness, microstructure, porosity, and interfacial diffusion.
2. EXPERIMENTAL

2.1 Materials and coating deposition

Three metallic coatings, nickel, cobalt, and 316L stainless steel, were studied in this work. The coatings were deposited on 4140 steel substrates by the HVPC facilities at the Applied Research Laboratory of the Pennsylvania State University. The precursor metallic powders have their median sizes in range of 10-15 microns. The coating thickness was around 500-600 microns.

2.2 Microwave sintering

The coated samples were sintered using 2.45 GHz microwave furnaces at the Materials Research Institute (MRI) of the Pennsylvania State University. The sintering conditions were selected based on the conventional sintering data and presented in Table 1. Microwave susceptors, graphite coated on alumina rods and tubes, were used to improve the heating profile since the coatings were relatively dense and poorly coupled with the microwave field. The temperatures were measured periodically by an optical pyrometer and the microwave power was adjusted accordingly to maximize the heating rate. It is found that all coated samples can be heated rapidly in microwave furnaces with the use of susceptors. After the target temperature and the holding time were achieved, the microwave power was shut off and the sample was allowed to cool naturally.

<table>
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<th>No.</th>
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<tr>
<td>1</td>
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<td>30</td>
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</tr>
<tr>
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<td>Ni</td>
<td>1225</td>
<td>30</td>
<td>Vacuum</td>
</tr>
<tr>
<td>4</td>
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<td>1050</td>
<td>30</td>
<td>Vacuum</td>
</tr>
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<td>316L</td>
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<td>5%H₂-N₂</td>
</tr>
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<td>Co</td>
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<tr>
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<td>Co</td>
<td>1250</td>
<td>30</td>
<td>5%H₂-N₂</td>
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</tbody>
</table>

2.3 Characterization

Porosity and microstructure of the coatings were observed by optical microscopes. Image analyzing software, ImageJ and Clemex, were used to measure percentage of porosity from microstructure images. Vickers microhardness indentors were used to determine the hardness of the coatings at a load of 300 g. The interdiffusion between the coating materials and the steel substrate was observed by energy dispersive spectroscope attached with scanning electron microscope.

3. RESULTS AND DISCUSSION

3.1 Porosity

Figure 1 shows porosity percentage of the coatings in as sprayed and as sintered conditions. By sintering nickel and 316L stainless steel coatings at relatively low temperatures, the percentage of porosity in the coatings increases tremendously when compared to the as-sprayed samples. This should be due to incomplete sintering and pore coalescence, leading to the presence of a larger pore which is more visible and detectable by the image analyzing software. However, the porosity percentage of the sintered coating decreases as the sintering temperature increases. Thus, the sintering parameters should be adjusted, such as increasing sintering time and temperature, to obtain...
optimum quality coating. In cobalt coating, sintering at temperatures above 1225°C provides denser coating than the as-sprayed condition.

Fig. 1. Percentage of porosity in the coatings obtained at various sintering conditions (thick horizontal line indicates the value for as-sprayed sample) (a) Ni, (b) 316L stainless steel, (c) Co.

3.2 Hardness and microstructures

Figure 2 shows hardness data of the coatings obtained at various microwave sintering conditions and compared with the data of as-sprayed samples. After sintering, hardness of the nickel decreases substantially to a typical range of annealed pure nickel. Some reduction in hardness was also found in sintered 316L stainless steel coatings. The high hardness of the coatings in as sprayed condition is a contribution from high degree of cold work introduced into the coating by HVPC process. Sintering temperatures relieved the residual stress inside the coating and annealed the coatings as confirmed by the microstructure images in figure 3 (a)-(b). Grain coarsening was also observed as the sintering temperature increases. In the cobalt coating, the hardness of the sintered samples changes slightly when compared with the as-sprayed sample. This could be a result from the presence of a metastable phase (α-fcc), which was not completely transformed to the stable ε phase (hcp) after cooling to room temperature. This is due to the sluggish nature of this kind of phase transformation. The fcc phase has low stacking fault energy, and can be transformed to the hcp phase by an application of external stress; as a result, the presence of metastable fcc phase and the interaction among readily formed stacking faults contribute to the high hardness. Furthermore, the sintered cobalt coatings have relatively small grain size. According to Romanski, the surface oxide of the initial cobalt powder trapped in the coating could impede the grain growth. The microstructure of cobalt coating is shown in figure 3 (c).

Fig. 2. Hardness of coatings from various sintering conditions (thick horizontal line indicates the values for as-sprayed sample) (a) Ni, (b) 316L stainless steel (c) Co.
3.3 Interface diffusion

Diffusion of elements between the coating and the steel substrate were observed in every sintered sample by the EDS line analysis across the coating/substrate interface. Figure 4 compares the concentration profiles across the interface obtained from nickel coated samples in as sprayed, as sintered at 1175ºC, and as sintered at 1225ºC conditions. The diffusion length increases as the sintering temperature increases. The development of the inter-diffusion layer is desired to improve the coating adhesion.

4. CONCLUSIONS

Microwave sintering shows a promising future as a process to improve density and adhesion of the cobalt coating deposited by HVPC process on steel substrate. For the nickel and 316L stainless steel coatings, microwave sintering parameters are needed to be adjusted to obtain higher density with the expense of reduction in coating hardness.

5. REFERENCES