Indentation size effect on the mechanical properties of supersonic plasma sprayed NiCr-Cr₃C₂ coating

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Abstract: In this paper, NiCr-Cr₃C₂ coating was deposited onto AISI 1045 steel substrate by employing supersonic plasma spraying technique. The surface and cross-section morphologies of the NiCr-Cr₃C₂ coating were observed by SEM. The phase constituents of the coating were identified by XRD. The SEM images showed that the coating was very dense, and it had a low porosity structure. The XRD pattern implied that the coating was mainly composed of NiCr, Cr₃C₂ and Cr₇C₂. Nanoindentation tests were performed on the coating surface by a Nano indenter. The widely used Oliver–Pharr method was adopted to extract the mechanical properties of the coating. The hardness exhibited a strong peak-load-dependence, i.e. indentation size effect; the elastic modulus was not affected by indentation size effect.

Key words: nanoindentation; mechanical property; indentation size effect

Hardness and elastic modulus are basic mechanical properties, and they are often used to characterize the performance of coatings. They can strongly influence the wear behavior of coatings, and thus it is very important to determine the hardness and elastic modulus of the coatings for characterization purposes.

Depth-sensing indentation technique (DSI), sometimes referred to as nanoindentation, has become an important tool for probing the mechanical behavior of materials at a nanometric scale. The mechanical properties can be determined by analyzing the indentation load–displacement data without the need to image the hardness impression.

NiCr-Cr₃C₂ coatings deposited by plasma or other thermal spraying methods are often applied in engineering due to their good tribological properties, corrosion and oxidation resistance [1-3]. In the present investigation, the hardness and elastic modulus of supersonic plasma sprayed NiCr-Cr₃C₂ coating were measured by nanoindentation technique under different peak loads. Moreover, the indentation size effect on the mechanical properties of the NiCr-Cr₃C₂ coating was discussed.

1 Experiment

The NiCr-Cr₃C₂ coating was deposited onto AISI 1045 steel substrate of 15×15×8 mm³ by employing supersonic plasma spraying technique. The NiCr-Cr₃C₂ powders with a composition of 5 wt.% Cr, 1.5-1.9 wt.% B, 20 wt.% Ni, 3-5 wt.% Si, 3-5 wt.% Fe, and 63.1-67.5 wt.% Cr₃C₂ were used in this work, as shown in Fig.1. In addition, Ni-Al powders were coated as a bond coat in order to increase the adhesion between the coating and substrate. A HEPJet spraying system was used to deposit the NiCr-Cr₃C₂ and Ni-Al coatings. The spraying parameters are listed in Table 1. A coating with thickness of 200 μm was obtained.

Nanoindentation tests were performed on the coating surface using a Nano Indenter® XP (MTS Corporation, U.S.A.) with a diamond Berkovich indenter. Prior to indentation, the geometry of the Berkovich indenter was calibrated on standard fused quartz specimen. Load control was adopted under 20 mN, 50 mN, 100 mN, 200 mN and 300 mN peak loads. An array of 3×3 was performed at each peak load. The mechanical...
Table 1 Supersonic plasma spraying parameters

<table>
<thead>
<tr>
<th>Spraying distance /mm</th>
<th>Spraying voltage /V</th>
<th>Spraying current /A</th>
<th>Ar Pressure /MPa</th>
<th>Ar Flow rate /m³·h⁻¹</th>
</tr>
</thead>
<tbody>
<tr>
<td>100</td>
<td>140</td>
<td>400</td>
<td>1.1</td>
<td>4.0</td>
</tr>
<tr>
<td>H₂ Pressure /MPa</td>
<td>H₂ Flow Rate /m³·h⁻¹</td>
<td>N₂ Pressure /MPa</td>
<td>N₂ Flow rate /m³·h⁻¹</td>
<td>Powder feed rate /g·min⁻¹</td>
</tr>
<tr>
<td>1.0</td>
<td>0.20</td>
<td>0.7</td>
<td>0.60</td>
<td>40</td>
</tr>
</tbody>
</table>

properties of the NiCr-Cr₃C₂ coating at each peak load were averaged over the nine measurements.

The hardness and elastic modulus were measured based on the method of Oliver and Pharr \[4\]. The hardness \(H\) and elastic modulus \(E\) were calculated by the following equations:

\[
H = \frac{P_{\text{max}}}{A}
\]

\[
E = \frac{S\sqrt{1-\nu^2}}{2\beta A}
\]

\[
\frac{1}{E_e} = \frac{1-\nu^2}{E} + \frac{1-\nu_i^2}{E_i}
\]

Where \(P_{\text{max}}\) is the peak load, \(A\) is the projected contact area, \(S\) is the elastic contact stiffness, \(\beta\) is a correction factor depending on the type of indenter, and \(\beta=1.034\) for the Berkovich indenter used in this study. \(E\) is the effective elastic modulus, \(\nu\) and \(\nu_i\) and \(E\), \(E_i\) are the Poisson’s ratio and elastic modulus of the measured material and indenter, respectively. For the diamond indenter, \(E_i=1141\) GPa, \(\nu=0.07\). In this study, the Poisson’s ratio of the NiCr-Cr₃C₂ coating is assumed to be 0.3.

The surface and cross-section morphologies of the NiCr-Cr₃C₂ coating were observed by scanning electron microscopy (SEM). The phase constituents of the coating were identified by XRD.

2 Results and Discussion

2.1 Characterization

Fig.2 depicts the cross-sectional SEM micrograph of the NiCr-Cr₃C₂ coating. The NiCr-Cr₃C₂ coating is very dense, and it shows a low porosity structure. The coating thickness is about 200 μm. The area A is hard phase Cr₃C₂; the area B is alloy phase NiCr. It is obvious that the hard phase is distributed dispersively in the alloy phase. Fig.3 shows the XRD pattern of the coating. The diffraction peaks of the coating are mainly composed of NiCr, Cr₃C₂ and Cr₃C. The appearance of Cr₃C was due to the carbon loss of Cr₃C₂ during the high-temperature spraying process.

2.2 Investigation of mechanical properties

Fig.4 shows load-depth curves for various peak loads. It can be seen that the loading curves for various peak loads trace each other very well, which exhibits the repeatability of the experiments \[5\]. This resulted in indentation depths between 350 and 1650 nm, corresponding to the load range between 20 and 300 mN. The indentation depth should not exceed 10–25% of the coating thickness, otherwise the results will be affected by the properties of the substrate \[6\]. In this study, the maximum indentation depth is 1.65 μm and coating thickness is 200 μm. Since the ratio of indentation depth to coating thickness is very low, the mechanical properties of the coating will not be affected by those of the substrate.

Fig.5 shows the hardnesses of the NiCr-Cr₃C₂ coating at various peak loads. Each of the data point represents an average of measurements from nine tests, and the error bars represent the standard deviation. At lower peak loads, the hardness has a large scatter due to the effect of surface roughness. When the applied load and depth increase, surface roughness effect decreases. It is clear that the hardness exhibits a significant indentation size effect, i.e. as the peak load increases, the hardness decreases distinctly. There are several explanations for the indentation size effect, and we have summarized the reasons in our earlier publication \[7\], including the Meyer’s law, strain gradient plasticity theory, surface pile-up or sinking-in, and the indenter tip correction.
The indentation size effect has been observed extensively for most materials [8-10]. This effect obviously has a significant influence on the application of the hardness to material evaluation, which will make confusion when the peak-load-dependent hardness is used to evaluate the material properties. Therefore, it is significative to compare the hardness of one material with that of another at the same peak load.

The elastic modulus of the NiCr-Cr$_3$C$_2$ coating was calculated with Eqs. (2) and (3), and the results are shown in Fig.6 as a function of the peak load. Compared with the variation of the hardness, the variation law of the elastic modulus shows very different. The modulus does not exhibit obvious peak-load-dependence. For elastic modulus belongs to the elastic property of materials and depends on the binding force between atoms, it is not affected by indentation size effect.

3 Conclusions

The nanohardness and elastic modulus of a supersonic plasma sprayed NiCr-Cr$_3$C$_2$ coating were extracted by the widely adopted Oliver-Pharr method. The variations of the hardness and modulus with peak load were investigated and the following conclusions were obtained:

1) The hardness decreases with increasing peak load, from 9.54 GPa at 20 mN to 6.37 GPa at 500 mN, and therefore exhibits a strong peak-load-dependence. This phenomenon will make confusion when the hardness is used to evaluate the material properties. It makes sense to compare the hardness of one material with that of another at the same peak load.

2) The variation law of the elastic modulus with the peak load is different from that of the hardness. The modulus does not exhibit obvious peak-load-dependence. For elastic modulus belongs to the elastic property of materials and depends on the binding force between atoms, it is not affected by indentation size effect.

References

1 Suarez M, Bellayer S, Traisnel M et al. Surface & Coatings Technology[J], 2008, 202: 4566