Mechanical properties assessment of additive manufacturing products by non-destructive testing technology

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Abstract
WC reinforced Ni-based composite coatings were obtained on FV520B substrate by plasma cladding with mixed Ni60-WC powder. Acoustic emission was employed to detect the wear behaviors under the condition of dry rotary sliding friction at 600°C and 800°C. The wear morphology of the coating was observed by SEM. The results showed the weight loss at 800°C was less than that at 600°C, but the plastic deformation region at 800°C was larger. The residual stress was tested by X-ray diffraction. The existence of second phase was the reason of residual stress release. The elastic modulus was measured by the oscilloscope. The elastic modulus and Vickers hardness for composite coatings were enhanced by the addition of WC. The more WC content, the greater the elastic modulus.

Keywords: Plasma cladding, Wear resistance, Acoustic emission, Residual stress, Elastic modulus

1. Introduction
Plasma cladding as a typical kind of surface strengthening and repair technology, is an important part of surface engineering, which can be used to repair the important components of aircraft engine, such as the high temperature compressor blades. Different from the traditional surface modification techniques, it has its own characteristics such as the coating and the substrate are in a good metallurgical bond status and the shape of coating can be easily controlled, etc. [1, 2]. However, further industrial applications are limited because of low mechanical properties which resulted by the thermal stress cracking. How to assess the mechanical properties in the cladding layer needs to be explored [3].

With the progress of high-energy beam cladding powder system, Ti-based, Ni-based, Co-based and Fe-based self-fluxing alloy powder have been developed, which are widely used in mechanical parts’ surface strengthening and repairing [4, 5]. They have impressive performance of high temperature strength [6], fatigue properties [7, 8] as well as fracture toughness [9, 10] and they also have good oxidation resistance and corrosion resistance [11]. As a result, they can be worked at the condition of over 600°C for a long term [12]. But with the development of aircraft engine, especially in the environment of overload and high temperature, the conventional self-fluxing alloy powder cannot meet the working conditions. Researchers develop a new method by adding hard phase to the self-fluxing alloy powder to meet the required conditions [13-16]. In the mixed powder, the hard-phase should be fully taken account of wettability and matching degree with self-fluxing alloy powder, in case of cracking tendency of the coatings. Compared with three kinds of powder, Ni-based powder has well overall performance and popular price. Although Ni-based composite cladding layers’ micro-structure and micro-hardness have been researched a lot before [17-19], the cladding layers’ cracking tendency has not been fully explored. Baranov et al. [20] identified the range of energy and frequency of acoustic activities which were generated from cracks propagation and debris formation stages in the sliding contacts. They also found that roughness, hardness, sliding velocity and friction mode were the factors of influencing the amplitude of the acoustic
emission signals. Furthermore, such extracting the AE features in the frequency may explain the friction behaviors more accurately. Hase et al. [21] used the Fast Fourier Transform (FFT) to distinguish adhesive wear and abrasive wear. In their research, adhesive wear was characterized by a frequency peak at 1.1 MHz with the effect of transfer particles and quantities of wear elements, while the frequency of abrasive wear was in the range of 250 kHz -1 MHz with the effect of cutting and plastic deformation. Chang et al. [22] explored the influence factors of signal frequency, and found that the AE activity with the range of 200–400 kHz are refer to friction surface rather than the crack length. However, further research is limited because of the existence second phases resulting in the uncertainty of the cracks.

Based on the above background, studying and clarifying the effect of WC content on the structure and properties of coating at high temperature, which is of great significance on designing and applying the composite coating. In this paper, our purpose is to find out WC content’s effect on high-temperature mechanical properties of Ni-based coating and provide a theoretical basis for future engineering applications.

2. Experimental methods

FV520B steel (C: 0.02-0.07 wt-%, Mn: 0.3-1.0 wt-%, Si: 0.15-0.7 wt-%, Cr:13.0-14.5 wt-%, Ni:5.0-6.0 wt-%, Mo:1.30-1.80 wt-%, Nb: 0.25-0.45 wt-%, Cu: 1.30-1.80 wt-%, S: <0.025 wt-%, balance Fe) was used as substrate material with a dimension of 100mm×100mm×10mm, the sample surface was polished by abrasive paper and degreased by anhydrous ethanol before cladding. Three kinds of Ni-based alloy powder with 0 wt-%, 10 wt-% and 30 wt-% WC were mixed uniformly by the planetary ball mill and dried 120°C for 2h by the drier. And the particle diameters of the powder are 45-75um.

This plasma cladding involved synchronous feeding the mixed powder into the molten pool of substrate. The equipment’s working power was about 3kW. The plasma beam scanning speed was 3mm·s⁻¹. And the powder feeding rate was 4-6g/min. The layer’s overlap was set at 40-60% in each track. Argon was used as the shielding gas during the operation with a flow rate of 6L·min⁻¹.

Dry reciprocating sliding wear of the coatings were carried out by HT-1000 friction and wear apparatus at 600°C and 800°C. The applied load was 20N and the applied time was 20min. The sliding frequency was 10Hz and the sliding radius was 3mm. The worn surface morphologies and the elements of oxidation product were tested by SEM and EDS, respectively. The AE-win software and PCI-2 data acquisition system were used to record the AE events with the sampling rate of 1MHz during the wear experiments. One piezoelectric sensor was pasted on the sample. The signals were amplified by a 40dB preamplifier and the threshold was 45dB. X-ray diffraction with a DX-2700 diffract meter was used (using Cu Kα radiation) to analysis the type of phases and the residual stress. The working voltage and current were 40kV and 40mA, respectively. The step speed was 5 deg·min⁻¹. The samples used for SEM observation were ground by abrasive paper and cleaned by anhydrous ethanol. The observation of the second phase in the coatings were tested by the Philips Quanta J200 SEM. The measure of Elastic modulus was carried out by means of ultrasonic, using TPP0500 oscilloscope to record the respective speed of transverse wave and longitudinal wave in the coatings. Vickers hardness was tested by HVS-1000 digital micro-hardness tester, the applied load was 3N and the applied time was 10s. Each layer was tested three times and all the test points were in the middle of the layers. The last value of the hardness was depended by the average of three values.
3. Results and discussion

3.1 Wear behaviors and AE results

3.1.1 Effects of WC content on the wear resistance of the coatings

Table 1 shows the weight loss of coatings with different WC contents at 600°C and 800°C. Under the conditions of above, weight loss decreases with the increase of WC content. And the weight loss at 600°C is more than that at 800°C of the same WC content.

<table>
<thead>
<tr>
<th>Temperature</th>
<th>0 wt-%WC</th>
<th>10 wt-%WC</th>
<th>30 wt-%WC</th>
</tr>
</thead>
<tbody>
<tr>
<td>600°C</td>
<td>19.5</td>
<td>11.6</td>
<td>8.8</td>
</tr>
<tr>
<td>800°C</td>
<td>18.1</td>
<td>8.2</td>
<td>5.9</td>
</tr>
</tbody>
</table>

To elucidate the wear mechanisms of the composite coatings, Fig. 1 demonstrates the worn surfaces of the coatings by SEM. The worn surface of coatings exhibits several deep and wide continuous furrows with noticeable plastic deformation. Compared with the wear morphology of coatings at 600°C, there are more plastic deformation area and delamination was more obvious on the surface of coatings at 800°C. This indicates that the coatings’ plastic deformation at 800°C is much easier than that at 600°C. In the condition of the same temperature, the plastic deformation area of coatings with 30 wt-% WC is smallest and 0 wt-% WC is the largest. This makes clear that WC could protect the coatings from the wear of grinding ball. In addition, there are a lot of uneven particles adhered on the coatings’ surface. The local high stress and high experimental temperature are the reasons which make the adhesion effect between grinding ball and coating happens. With the sliding of the grinding ball, the adhesion and fracture happens alternately between the contact points at high temperature [23]. When the wear debris adhered on the grinding ball meets the block, wear debris will be attached to the surface of the coating. Therefore the more WC content in the coatings, the more particles on the wear surface.

These results reveal that the wear mechanisms of composite coatings at high temperature are mainly in the form of adhesive wear and oxidation wear.Comparatively speaking, when the experimental temperature is 800°C, the coatings plastic deformation region is larger and oxidation degree is more serious than those at 600°C. And WC particles can improve the wear resistance of the coatings effectively.
In order to ascertain the elemental composition of the wear surface at high temperature, an EDS study is carried out at different areas and the corresponding results are shown in Table 2. Four different regions can be observed in Fig. 1 and labeled S1, S2, S3 and S4, respectively. As shown in figure, all the regions contains oxygen which indicates that oxidation occurred when friction and wear at high temperature. And in the condition of the same WC content, oxygen content of coatings at 800°C is higher than that at 600°C. It makes clear that the phenomenon of oxidation is more serious at 800°C. Additionally, a small amount of tungsten appears in Region S3 and the content of tungsten in Region S4 is only 18.08%, which indicates that part of the grinding ball is ground to the surface and a large number of WC particles are ground off. And the amount of WC which are ground off by the ball is much greater than that ground to the surface. Therefore, serious oxidation and materials of grinding ball transferred to the coatings are the reasons of that the weight loss at 800°C is less than that at 600°C.
Table 2 Elements analysis of Area S1, S2, S3 and S4. (wt-%)

<table>
<thead>
<tr>
<th></th>
<th>O</th>
<th>Si</th>
<th>Cr</th>
<th>Fe</th>
<th>Ni</th>
<th>W</th>
</tr>
</thead>
<tbody>
<tr>
<td>S1</td>
<td>8.49</td>
<td>3.03</td>
<td>19.76</td>
<td>13.44</td>
<td>55.28</td>
<td>0</td>
</tr>
<tr>
<td>S2</td>
<td>14.21</td>
<td>2.65</td>
<td>11.45</td>
<td>12.69</td>
<td>31.05</td>
<td>27.95</td>
</tr>
<tr>
<td>S3</td>
<td>15.28</td>
<td>3.84</td>
<td>15.22</td>
<td>14.45</td>
<td>47.15</td>
<td>4.07</td>
</tr>
<tr>
<td>S4</td>
<td>20.29</td>
<td>2.32</td>
<td>9.13</td>
<td>21.20</td>
<td>28.97</td>
<td>18.08</td>
</tr>
</tbody>
</table>

3.1.2 AE signal processing

The recorded AE signals are non-stationary and appear as transient signals with undefined waveform. As can be seen from Fig. 2, an analysis of the time series of friction coefficient and AE counts reveals some features in these curves, which can be used to estimate the characters of the friction process. The friction coefficient exhibits insignificant variations at the initial stage, which is evidence for a stationary regime of friction. Then, the character of friction behaviors changes, which is manifested by an increase of the friction coefficient and the appearance of its aperiodic oscillations. It is found that these oscillations were related to the adhesion interaction of conjugated surfaces and increased wear of both the steel disk and counter-body. Changes in the friction coefficient were accompanied by variations of the AE counts. Out investigations showed that changes in the intensity of acoustic signals are correlated with variations in the AE counts, which correspond to the moments of fracture of the material surface layer in the contact zone. It can be suggested that the fracture of both the sample surface and counter-body are accompanied by the generation of the corresponding acoustic signal. However, these events cannot be reliably separated using only data on the sound intensity and/or AE counts.

Another interesting observation is the accompanying periodic variation of parameters of the acoustic signal. This correlation between the parameters of friction and acoustic emission can be explained by a periodic change in the real area of contact in the course of friction. As the contact area increases (due to matching of the conjugated surfaces as a result of their plastic deformation), both the friction coefficient and acoustic emission increase, while the avalanche fracture of the surface layer (leading to decrease in the contact area) results in a decrease in the friction coefficient and acoustic emission. If this explanation is correct, the observed behaviours are indirectly indicative of the predominating adhesion component in the friction force.

Fig. 2 Time series of the AE counts and friction coefficient measured at 600°C
(a) 0 wt-% WC; (b) 10 wt-% WC; (c) 30 wt-% WC
3.2 Effects of WC content on the residual stress of the coatings

According to the residual stress calculation formula of two-point method, as is shown by equation (1).

\[ \sigma_{\psi} = 2K \Delta 2\theta_{1,2} \]  

where \( K \) and \( \Delta 2\theta_{1,2} \) are the stress constant and the ordinate difference between two points of relation curve, respectively. According to Bragg's law, if the optical path difference between two adjacent planes of the reflection line is an integer multiple of the wavelength, the reflected rays will be strengthened and the diffraction phenomenon will be obtained. The value of residual stress depends on the shift distance of the diffraction peak. In this experiment, the value of \( K \) was -601MPa/deg. Based on the value of \( K \), the residual stress of coatings with different WC content are -630MPa, -749MPa and -418MPa, respectively.

The influence factors of residual stress include the heat effect and the phase transformation effect [24]. The heat effect is determined by the difference of thermal expansion coefficient between the different phases during fast heating or cooling process [25]. While the phase transformation is mainly determined by the temperature. Owing to the technological parameters of the coatings are the same, the heat effect is the main influence factors which affects the value of residual stress. During the progress of heating or cooling, the difference of expansion coefficient makes the tiny gaps between the second phase and the binder phase occurred. Due to the difference of surface shape and surface area, the size of gaps are different [26]. And the residual stress will be released because of the gaps [27].

Fig. 3 shows the SEM of coatings and Table 3 illustrates EDS of the second phase. The black phase in Fig. 3 (a) is mainly made up of Cr, Ni, Fe and C. We could also know the white phase in Fig. 3 (b), (c) mainly contains W, Cr, Ni, Fe and C, and W accounts for the majority. To figure out the type of second phase in the coatings, the cross section of coatings are tested by XRD and shown in Fig. 4. Based on the XRD and EDS experiments, it can be known that the main second phase of coating with no WC is Cr3C2, and it also contains a small amount of Ni-Cr-Fe-C. The second phase of coating with WC mainly comprised WC and W2C, and there are also a little of Ni-Cr-Fe-C. It also can be known from the EDS experiments that all the coatings don’t comprise nitrides and oxides because of the protection of the high pure argon. In conclusion, the reaction between WC and Ni-based powder leads to the change of the second phases’ shape and color. Comparatively speaking from the Fig. 3, it can be known that the coating with 10 wt-% WC has the least and most regular second phase. As a result, the release of residual stress is the least, so the absolute value of residual compressive stress is the highest and cracks of coating can be inhibited effectively.

| Table 3 EDS of the second phase in coatings, (wt-%) |
|-----------------|-----|-----|-----|-----|-----|
|                | C   | W   | Cr  | Fe  | Ni  |
| 0 wt-% WC      | 3.40| 0   | 73.64| 9.67| 13.30|
| 10 wt-% WC     | 3.52| 51.09| 26.68| 10.46| 7.30 |
| 30 wt-% WC     | 6.41| 57.49| 10.88| 6.16 | 15.40|
3.3 Effects of WC content on the elastic modulus of the coatings

Elastic modulus as an engineering indicator of mechanical properties, reflecting the spring-back properties of materials. It has been found from the literature [28] that, a relatively lower value of elastic modulus will be particularly beneficial for the coating, especially if the elastic modulus of the coating material can closely match that of the substrate material. Table 4 illustrates the values of transverse wave velocity, longitudinal wave velocity, density, Poisson's ratio and elastic modulus of three kinds of coatings. Among of them, longitudinal wave velocity and transverse wave velocity are tested by the oscilloscope. And the density is measured by means of drainage. The Poisson's ratio and elastic modulus are calculated by equation (2) and equation (3).
\[ \nu = \frac{1-2\left(\frac{V_T}{V_L}\right)^2}{2-2\left(\frac{V_T}{V_L}\right)^2} \]  
\[ E = \frac{V_L^2 \rho (1+\nu)(1-2\nu)}{1-\nu^2} \]

where \( V_L \) is the longitudinal wave velocity, \( V_T \) is the transverse wave velocity, \( \rho \) is the density, \( \nu \) is the Poisson's ratio and \( E \) is the elastic modulus. It can be known that the more WC, the greater the elastic modulus. And the coating with no WC has a better match with the substrate materials.

Fig. 5 shows the variation of Vickers hardness and elastic modulus of the coatings with the increase of WC content. The plots indicate that the coating with 30 wt-% WC processes the highest hardness (~648.2HV) and the greatest value of elastic modulus (~247.59 GPa), as compared to those with 0 wt-% and 10 wt-% WC. And the tendency of Vickers hardness and elastic modulus are almost the same which indicates the fine WC particles distribute in coatings and form meta-stable phases with Ni60 powder. Therefore, it is clear that WC not only increases the value of Vickers hardness, but also increases the value of elastic modulus.

<table>
<thead>
<tr>
<th>Physical properties of FV520B substrate and coatings.</th>
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<tbody>
<tr>
<td>FV520B</td>
</tr>
<tr>
<td>( V_L/\text{mm} \ \text{us}^{-1} )</td>
</tr>
<tr>
<td>( V_T/\text{mm} \ \text{us}^{-1} )</td>
</tr>
<tr>
<td>( \rho/\text{g cm}^{-3} )</td>
</tr>
<tr>
<td>( \nu )</td>
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<tr>
<td>( E/\text{GPa} )</td>
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</table>

4. Conclusions

Plasma cladding Ni-based composite coatings on FV520B substrate. The coatings with no WC mainly comprises irregular black phase Cr₃C₂, while the coatings with WC have the white blocky WC or W₂C particles. The content of WC in the coatings has a great influence on the properties of coatings.

(1) The friction and wear properties of the coatings are improved at high temperature with WC content increased. The weight loss at 800°C is less than that at 600°C, but the plastic deformation area of coatings at 800°C is larger. The wear mechanisms of composite coatings at 600°C and 800°C are mainly adhesive wear and oxidation wear. Changes in the friction
coefficient were accompanied by variations of the AE counts. AE signals could distinguish the stages of friction behaviors.

(2) There exists the difference of thermal expansion coefficient between the second phase and the binder phase, there will be tiny gaps between the second phase and the binder phase during the heating and cooling process. The residual stress of coatings is determined by the number and shape of second phase. The second phase in the coatings with 10 wt-% WC is the least and the most uniform. The release of stress is the least, so the absolute value of residual compressive stress is the highest.

(3) The addition of WC not only increases the value of Vickers hardness, but also increases the value of elastic modulus. The dissolution of fine WC particles in coatings and the formation of meta-stable phases lead to the increase of the value of elastic modulus. The coating with no WC has a better match with the substrate materials.

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