Effects of Process Parameters on Microstructure and Hardness of Layers by Laser Cladding

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1. Introduction

Laser, as a special source of energy has a wide scope of applications and plays an important role in materials processing. Laser cladding is one of the emerging technologies and has multiple applications in manufacturing industry, ranging from surface repair and modification with special coating over a mechanical component to direct fabrication of three-dimensional functional metallic components. The advantages of laser cladding over the conventional processes of surface modification have been well documented, such as minimum dilution, minimal distortion of substrate, finer microstructure, higher wear and corrosion resistances, etc.1–5) In the die industry, it is commonly agreed that residual tool life can be successfully extended by timely repair of damaged surfaces by laser cladding.6,7) Laser cladding with powder feed is a multi-parameter process, such as laser power, laser beam profile, laser scanning speed, powder feeding rate, material properties, etc. The deposition track characteristics such as the geometry, dilution, microstructure, etc., would be influenced greatly by these parameters. It is of great importance to find the relationships among the process parameters and the microstructure and the properties of the cladding materials. In past decade, the effects of variant parameters have been extensively studied. Pinkerton and Li studied the influences of pulse frequency on the microstructure, surface roughness and hardness of fabricated 316L stainless steel.8) Capello et al. investigated the influences of operator skills, processing parameters and materials on clad shape in repair using laser cladding by wire.9) Li et al. demonstrated the effect of the specific energy on the cross-section shape of nickel alloy cladding by a single-track cladding experiment with different laser processing parameters and found an appropriate range for the specific energy in which the nickel alloy samples could be fabricated layer by layer with a uniform height.10) Li et al. reported the influences of the processing parameters on forming characterizations during laser rapid forming.11) These processing parameters are closely related, so would produce intricate effects on the properties of the cladding layers.

As a kind of low-cost material, medium carbon steel is widely used in industry. However, it does not offer special properties such as wear resistance, corrosion resistance or oxidation resistance, etc. Laser cladding is an efficient and cost-effective technology that endues the medium carbon steel a special surface layer without changing the main properties of the bulk material. Ni-base alloys are widely used to obtain wear and corrosion resistant coatings in laser cladding. In the present study, Investigations focused on the...
effects of the powder feeding rate and the scanning speed on the microstructure and microhardness of the laser cladding layers. A series of laser cladding layers of Ni35 on a medium carbon steel substrate by varying the powder feeding rate and the laser scanning speed were carried out. The phase composition, microstructure and microhardness of the cladding layers were investigated by X-ray diffractometer (XRD), energy dispersion spectroscopy (EDS), scanning electron microscope (SEM) and Vickers hardness tester, respectively.

2. Experimental Procedure

2.1. Materials

The 45 steel plate with a dimension of 200 mm×100 mm×10 mm was selected as the substrate for laser cladding treatment. The steel plate was ground with 300-grid SiC abrasive paper and cleaned with acetone and ethanol before the laser cladding. The particles of Ni35 alloy powder (feeding material) are 47–120 μm in size. In order to eliminate the moisture of the powder particles, the powders were dried in a drying oven for 24 h. The compositions of the substrate and the cladding powder are listed in Table 1.

2.2. Laser Cladding Process

The experimental system consists of a 5-kW continuous wave CO2 laser, a 3-axis CNC-machine and a coaxial powder feeder with a processing head. The principle of this technology is shown in Fig. 1. During the laser cladding, a high power laser beam is focused onto the substrate to create a molten pool, the metal powders are simultaneously injected into the focal zone by the powder delivering nozzles and then melted and rapidly solidified. A single layer can be deposited by the relative motion of the laser-substrate, namely, either by the worktable motion or by the robot movement on the substrate. An argon shielding gas at the flow rate of 200 L/h was blown coaxially to protect the molten pool from oxidation. Argon was also used as the delivery gas for the powder feeder. In order to investigate the effects of the processing parameters on the microstructure and the hardness, a series of the layers of 100 mm long were cladded on the substrate with different processing parameters which are listed in Table 2, where the laser power was 2 kW.

2.3. Microstructural Observation

Ethanol solution of 4% HNO3 was used to etch the transverse cross-section of the laser cladding layer. A scanning electron microscope (SEM) was used to observe the microstructure. X-Ray diffraction (XRD) was used to examine the phase composition of the cladding layer. The composition was measured by energy dispersion spectroscopy (EDS). The microhardness was measured by a Vickers tester using a load of 0.1 kg and loading time 15 s. The values of hardness were averaged by at least 5 points of measurements. The results of evaluations are presented in Chap. 3.

3. Results and Discussions

3.1. Microstructures of Laser Cladding Layers

Figure 2 is a representative microstructure from transverse cross-section of the cladding layer treated by a laser power of 2 kW and a laser scanning speed of 100 mm/min. Three distinct regions, namely, the cladding layer, the HAZ and the substrate, from the right to the left correspondingly, can be clearly found on the cross-sections of all the cladding layers. A thin white layer is observed at the interface between the layer and the substrate indicating a good metallurgical bonding between the cladding layer and the substrate. It can be found from Fig. 2 that in the same corrosion condition, the cladding layer has better corrosion-resistant than the substrate. In order to show the microstructure of the substrate, the time of corrosion is reduced which results in an unclear microstructure of the cladding layer. Figure 3 is the microstructures from the transverse cross-section of the cladding layer in different positions. A planar growth and cellular crystal near the bonding line, relatively coarse columnar dendrite away from the bonding line and fine dendrites near the surface of the layer are observed in the cladding layer. The microstructure is predominantly dendritic. The columnar dendrites grow parallel to the heat flux direction, leading to a preferential orientation. Compared with that of the substrate, the microstructure of the cladding layer shows a greatly refined structure, characteristic of the high cooling rates inherent to this process. Figures 4(a) and 4(b) are the large magnification micrographs of the interfaces of the HAZ and the cladding layer, the substrate and the HAZ, respectively. The HAZ can be clearly observed.
In fact, the solidification microstructure of the cladding layer essentially depends on the local solidification conditions. According to the rapid solidification theory, the growth morphologies of the rapidly solidified layer mainly depend on the temperature gradient ($G$) and the solidification rate ($R$) especially the parameter $G/R$. $G/R$ is the critical controlling parameter, which determines the feature of the solidification microstructure. During the laser cladding, the heat of the cladding layer mostly dissipates through the substrate and the surrounding atmosphere. Figure 5 is the schematic illustration that shows the effect of $G/R$ on the microstructure of the cladding layer. At the beginning of the solidification, since the liquid metal maintains contact with the solid substrate, the solidification rate is initially zero at the interface between the substrate and the melt, $G/R$ at the interface between the substrate and the melted zone is very high such as indicated by the region of the high $G/R$ as shown in Fig. 5. Therefore, the planar growth forms at the beginning of the solidification as shown in Fig. 2. With the propelling of the solid/liquid interface and heat accumulation, the solidification rate increases rapidly and the temperature gradient decreases, which results in the transformation of the crystal feature from the planar growth to the cellular and dendrite solidification such as indicated by the region of the low $G/R$ as shown in Fig. 5. The dendrite solidification appears after the cellular solidification in the cladding layer. This region is relatively wide as shown in Figs. 3(b) and 3(c). The cellular dendrite and the dendrite solidification all tend to grow perpendicularly to the interface because the heat mostly dissipates through the substrate and the temperature gradient and the heat flux density perpendicular to the interface are the largest. While near the top of the cladding layer, the dendrite become very fine and disorientated as shown in Fig. 3(a). This is possibly because that at the surface of the cladding layer, the heat mostly dissipates through the surrounding atmosphere and the temperature gradient ($G$) is not dominant any more.

### Table 2. The list of processing parameters.

<table>
<thead>
<tr>
<th>No.</th>
<th>Laser scanning speed (mm/min)</th>
<th>1°</th>
<th>2°</th>
<th>3°</th>
<th>4°</th>
<th>5°</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>100</td>
<td>100</td>
<td>100</td>
<td>100</td>
<td>150</td>
<td>200</td>
</tr>
<tr>
<td>2</td>
<td>Powder feeding rate (g/min)</td>
<td>6</td>
<td>6.5</td>
<td>7.0</td>
<td>6.5</td>
<td>6.5</td>
</tr>
</tbody>
</table>

**Fig. 2.** Microstructure on the transverse cross-section of the laser cladding layer ($P=2\ kW$, $V_s=100\ mm/min$, $V_f=6.5\ g/min$).

**Fig. 3.** Microstructures on the transverse cross-section of the laser cladding layer in different positions. (a) top (b) middle (c) bottom ($P=2\ kW$, $V_s=100\ mm/min$, $V_f=6.5\ g/min$).

**Fig. 4.** Microstructures on the transverse cross-section of the sample. (a) the interface between the HAZ and the cladding layer (b) the interface between the substrate and the HAZ ($P=2\ kW$, $V_s=100\ mm/min$, $V_f=6.5\ g/min$).

**Fig. 5.** Schematic illustration of the formation of the cladding layer and crystallographic analysis.
Figure 6 is the magnified microstructure of the cladding layer. The results of EDX analysis reveal that the composition of the dendrites (area E) is 85.14 wt% Ni, 6.43 wt% Fe, 6.09 wt% Cr and 2.34 wt% Si. The compositions of the interdendritic are shown in Table 3. The interdendritic morphologies are different due to different compositions. Figure 7 is the energy spectrum analysis results on the cladding layer in different areas. The block area (area F) contains relatively less Fe, the parallel club-shaped area (area G) is relatively rich in Si and the petal-shaped area (area H) is rich in Ni and poor in Si. The results of the XRD analysis are shown in Fig. 8. The results of the XRD and EDX analyses indicate the existence of \(\gamma\)-nickel, Ni\(_3\)B and Cr\(_3\)Si phases in the composite layer, and the fcc \(\gamma\)-nickel peak are most prominent. The phases between the \(\gamma\)-nickel dendrites are eutectic phases.

EDS analysis of the cladding layer in different positions has been also carried out, as shown in Fig. 2. Three points on area A of the region near the interface of the cladding layer, area B of the region far from the interface of the cladding layer, area C of the region near the interface of the substrate and area D of the region far from the interface of the substrate are, respectively, tested and the average values of the compositions have been presented. The results of the EDX analyses in Table 4 show the existence of diffusion. By composition contrast, it indicates that at the interface of the cladding layer and the substrate, slight element diffusion happens. With the increase of the distance from the interface to the cladding layer, there is a slight reduction in Fe content and similarly with the decrease of the distance from the interface to the substrate, there is a slight increase in Ni content. The difference of compositions between the bonding interface and the cladding layer indicates the existence of dilution, namely the cladding layer is diluted. This phenomenon can be attributed to the inherent characters of the process. Since the laser cladding is a process of the rapid heating and solidification. In this process, the cladding powders and the substrate are heated simultaneously. The slight diffusion will happen and lead to a good metallurgi-

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**Table 3.** Elemental compositions in different areas by EDX analysis.

<table>
<thead>
<tr>
<th>Element</th>
<th>Si</th>
<th>Cr</th>
<th>Fe</th>
<th>Ni</th>
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<tr>
<td>E</td>
<td>2.34</td>
<td>6.09</td>
<td>6.43</td>
<td>85.14</td>
</tr>
<tr>
<td>F</td>
<td>5.55</td>
<td>4.89</td>
<td>4.96</td>
<td>84.6</td>
</tr>
<tr>
<td>G</td>
<td>7.41</td>
<td>5.87</td>
<td>6.27</td>
<td>80.45</td>
</tr>
<tr>
<td>H</td>
<td>0.82</td>
<td>5.67</td>
<td>6.34</td>
<td>87.17</td>
</tr>
</tbody>
</table>

**Table 4.** EDX results for the major elements in different positions in Fig. 2.

<table>
<thead>
<tr>
<th>Element</th>
<th>Si</th>
<th>Cr</th>
<th>Fe</th>
<th>Ni</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>3.06</td>
<td>6.40</td>
<td>10.58</td>
<td>75.35</td>
</tr>
<tr>
<td>B</td>
<td>2.84</td>
<td>5.61</td>
<td>13.73</td>
<td>73.32</td>
</tr>
<tr>
<td>C</td>
<td>-</td>
<td>-</td>
<td>77.77</td>
<td>1.95</td>
</tr>
<tr>
<td>D</td>
<td>-</td>
<td>-</td>
<td>80.72</td>
<td>-</td>
</tr>
</tbody>
</table>
The effects of the laser scanning speed on the microstructure are shown in Figs. 9(a), 9(b) and 9(c). When the laser scanning speed is 100 mm/min, coarse dendrites are observed in the cladding layer. When the laser scanning speed is increased to 150 mm/min, the coarse dendrites are relatively fine. When laser scanning speed is further increased to 200 mm/min, the dendrites are finer. According to Ref. 13), the ratio of the temperature gradient to the solidification rate can be estimated according to the following equation:

\[
\frac{G}{R} = \frac{2\pi K(T - T_0)^2}{\eta PV_f \cos \theta}
\]

where \( G \) is the temperature gradient, \( R \) is the solidification rate, \( V_s \) is the laser scanning speed, \( T \) is the liquidus temperature of the alloy, \( T_0 \) is the preheated temperature of the substrate, \( \eta \) is the laser absorption coefficient, \( P \) is the laser power and \( K \) is the thermal conductivity of the material. The solidification is initiated at the clad/substrate interface and orients towards the surface of the clad region (following the direction of the heat flux). For the given values of other parameters, as \( V_s \) increases, \( G/R \) decreases. So, for low laser scanning speed, the growth of the \( \gamma \)-nickel is characterized by coarse columnar dendrites, the phases between the \( \gamma \)-nickel dendrites are eutectic phases. As the laser scanning speed increases, the \( \gamma \)-nickel dendrites become fine. With further increasing laser scanning speed, the growth of \( \gamma \)-nickel presents the cellular crystals and eutectics. In a word, with the increase of the laser scanning speed, the microstructures of the cladding layers are refined significantly.

Similarly, the powder feeding rate also has an impact on the microstructure of the cladding layer. The effects of the powder feeding rate on the microstructure are shown in Figs. 9(c), 9(d) and 9(e). As the powder feeding rate increases, the grain sizes of the cladding layers become fine accordingly. The effects can be analyzed by specific energy as follows. The specific energy per mass of the cladding material can be calculated by Eq. (2),

\[
E_v = \frac{P}{\eta V_f}
\]

where \( E_v \) is the specific energy per mass of the cladding material, \( P \) is the laser power, \( \varepsilon \) is the effectively utilized efficiency of powders in laser cladding and \( V_f \) is the powder feeding rate. It is obvious that under the circumstances that \( P \) and \( \varepsilon \) kept invariable, an increase in the powder feeding rate might lead to a decrease of \( E_v \). Thus, the average temperature of the particles of the cladding powders decreases accordingly. At the same time, the temperature of the substrate decreases because the energy which melts the substrate mostly comes from that of permeated from the cladding powders. Both the decreased temperature of the particles of the cladding powders and the decreased temperature of the substrate result in an increase of the cooling rate of the cladding layer. So this provides higher temperature gradient and cooling rate, which results in an increase of nuclear efficiency and more fine microstructures of the cladding layer.

Actually, under given powder feeding rate and other technological parameters, when the laser scanning speed is in-

<table>
<thead>
<tr>
<th>( V_s ) (mm/min)</th>
<th>( V_f ) (g/min)</th>
<th>Macro-morphology</th>
<th>Microstructure</th>
</tr>
</thead>
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<tr>
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<td>7.0</td>
<td></td>
<td><img src="image" alt="Macro-morphology" /></td>
<td><img src="image" alt="Microstructure" /></td>
</tr>
</tbody>
</table>

**Figure 9.** The macro-morphology and the corresponding microstructure from traverse cross-section built with different technological parameters. \( V_s \) and \( V_f \) refer to the laser scanning speed and the powder feeding rate, respectively. The laser power \( P \) is 2 kW for all the specimens.
creased, the duration of the laser beam interaction on the powder and the substrate becomes shorter, which results in less heat input of the laser cladding. After certain a threshold of the laser scanning speed, energy available will be insufficient to fuse the powder to the substrate, and an incomplete fusion appears. A relatively weak bonding between the layer and the substrate will be formed. This phenomenon is undesired. By adjusting the processing parameters, this phenomenon will be eliminated. Similarly, under a given laser scanning speed, the powder feeding rate also exists a threshold. When the powder feeding rate is higher, the powder flux shields the laser beam. The energy which melts the substrate mostly comes from that of permeated from the cladding powders is so small that it causes insufficient substrate melting, the metallurgical bonding between the cladding layer and the substrate can’t also achieved. The laser cladding process doesn’t realize. In a word, on condition that other technological parameters hold immutable, a given laser scanning speed corresponds with a critical powder feeding rate and a given powder feeding rate also corresponds with a critical laser scanning speed. Furthermore, the critical powder feeding rate decreases accompanying the increase of the laser scanning speed at the same technological condition. Similarly, the critical laser scanning speed decreases with the increase of the powder feeding rate. In practice, the adjustment of the powder feeding rate should limit within the critical powder feeding rate at a given scanning speed and the adjustment of the scanning speed should limit within the critical scanning speed at a given powder feeding rate. A good matching of the scanning speed and the powder feeding rate is necessary in laser cladding.

3.2. The Microhardness of Cladding Layers

The microhardness is an important index to evaluate the material properties. It is possible to understand the mechanical properties of the cladding layer by means of the microhardness across the layer, at least on a rough scale. The microhardness measurement across the transverse cross-section of the sample was carried out using microhardness tester. Figure 10 exhibits the effects of the laser scanning speed and the powder feeding rate on microhardness distribution of the transverse section of the sample. Horizontal ordinate represents the vertical distance from the substrate to the measuring point of the sample and vertical ordinate represents the Vickers hardness of the measuring point. From Fig. 10, it can be observed that with the increasing of the distance, the hardness profiles all present increasing trend. It is apparent that the microhardness of the laser cladding layer is much higher than that of the substrate and the HAZ is significantly hardened. The reason for increase in microhardness is due to the higher cooling rate and the fine microstructure obtained in laser cladding. It could be clearly seen that the average microhardness of the cladding layer for high laser scanning speed is higher than that of the cladding layer for low laser scanning speed and with increasing laser powder feeding rate, the average microhardness of the cladding layers increases. This is because that with increasing laser scanning speed, the ratio of the temperature gradient to the solidification rate decreases, microstructure characteristic transits from the coarse columnar dendrites to the fine dendrites and cellular crystals. The finer the microstructure is, the more the crystal boundary area is. Since the crystal tropism at side of the crystal boundary are completely different and ruleless and crystal boundary is disorganized region of atom arrangement, when the plasticity distortion goes through the crystal boundary from one crystal grain to another, it is very difficulty to go through owing to high resistance of the crystal boundary. The hardness is related to the resisting distortion ability. Consequently, the hardness increases as results of the microhardness. Similarly, with an increasing laser powder feeding rate, the heating temperature of the powder particles decreases and the cooling rate increases which resulted in the finer microstructure. So the hardness of the cladding layer increases. Figure 10 also shows that the microhardness profile of the cladding layer decreases slightly near the fusion boundary and indicates the existence of a narrow region of the dilution in the cladding layer. At the same time, the microhardness in the HAZ zone is higher than that of the substrate and lower than that of the region near the fusion boundary. On the other hand, the hardness at the top of the cladding layer is higher caused by the rapid
melt and solidification of the materials. Of cause, the state of the dendrite, the distribution of the solid solution and the region of the carbide all affect the uniform of the hardness. Actually, this continuous transition of the microhardness is helpful to the property of the cladding layer. Firstly, the highest hardness region guarantees the wearability of the cladding layer. Secondly, the region of less hardness transition near the fusion boundary in the cladding layer and in the HAZ could help to decrease the inner stress and provide the powerful support for the highest hardness region at the outermost. At the same time, the transition region of less microhardness could help to enhance the bonding intensity between the cladding layer and the substrate.

From Fig. 10, it could be seen that the profiles of hardness all present two obvious turning points. The first corresponds to the hardness of the margin of dilution region and the second corresponds to the hardness of the end of HAZ. The distance from the first turning point to the broken line (corresponds to the interface) shows the range of dilution of the cladding layer and the distance from the broken line to the second turning point shows the region of the HAZ. From the joining line of the first kinds of turning points and the change of the position of the second kinds of turning points, the effect rules of the laser scanning and the powder feeding rate on the range of dilution and the region of the HAZ can be intuitively derived. Under given laser scanning speed and other technological parameters, with an increase of powder feeding rate, the range of dilution and the region of the HAZ decrease. This is because that the energy melting the substrate mostly comes from that of permeated from the cladding powders. With the increase of powder feeding rate, the energy of laser beam permeated from the cladding powders attenuates. The higher the powder feeding rate is, the more the attenuation is. So the energy of laser beam irradiating the surface of the substrate decreases which results in the decrease of the range of dilution depending on the molten quantity of the substrate. At the same time, the region of the HAZ decreases due to the lower energy input led higher cooling rate. With the increase of laser scanning speed, the range of dilution and the region of the HAZ decrease. This is because that by increasing the laser scanning speed, the duration of the laser beam interaction on the substrate decreases, which results in the decrease of the heat input of the laser cladding. So the molten quantity of the substrate decrease which results in the decrease of the range of the dilution and the region of the HAZ. Indeed, the real dilution region is near the bonding interface. This method that the joining line of the first kinds of turning points of the hardness profile facilitates the study on the question of the bonding interface, for the control of the dilution rate and range and the microstructure and properties of the cladding layer.\(^{16}\)

4. Conclusions

(1) As the laser scanning speed increases, the microstructure characteristic of the cladding layer transits from the coarse dendrites to the fine dendrites resulting from the decrease of the ratio of the temperature gradient to the solidification rate.

(2) With increasing powder feeding rate, the microstructure gets refined resulting from the decrease of the specific energy per mass of powder and the increase of cooling rate of the cladding layer.

(3) Hardness distributions across the transverse cross-section of the samples show that as the laser scanning speed and the laser powder feeding rate increase, the microhardness of the cladding layer increases. This is attributed to the finer microstructure which results in the more crystal boundaries and thus higher ability in resisting distortion.

(4) From the joining line of the first and the second kinds of turning points of the hardness distribution profile, the effect rules of the laser scanning speed and the powder feeding rate on the range of dilution and the region of the HAZ can be derived. Adopting the joining line of the first kinds of turning points of the hardness profiles to indicate the range of dilution is a facilitative method.

Acknowledgements

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