Microstructure and interfacial metallurgical bonding of 1Cr17Ni2/carbon steel extreme high-speed laser cladding coating

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Received: 14 October 2020 / Revised: 13 November 2020 / Accepted: 20 November 2020 / Published online: 4 January 2021
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Abstract
The extreme high-speed laser cladding (EHLA) was employed to fabricate a 1Cr17Ni2 coating. The solidification behavior, phase transformation, and interfacial metallurgical bonding of the coating were systematically investigated. The results showed that the major phase transformation during solidification was liquid to γ-Fe. The large temperature gradient of melting pool and slow grow rate of γ-Fe grain contributed to the fine columnar prior austenite grain (PAG) in coating. The largest thermal conductivity of [0 0 1] crystal direction determined the preferential [0 0 1] orientation of PAG perpendicular to the liquid-solid interface. A thin γ-Fe layer (approximately 5 μm) was observed between coating and substrate. The Bain relationship between interfacial γ-Fe layer and substrate and the K-S relationship between interfacial γ-Fe layer and coating contributed to the reliable metallurgical bonding between coating and substrate. The shear test revealed the high shear strength (approximately 92% of that of substrate) and weaker plastic deformation ability of the interface.

Keywords Extreme high-speed laser cladding · Columnar grain · Interfacial metallurgical bonding · Bain relationship · K-S relationship

1 Introduction
Laser cladding is an effective way to protect parts via a stronger surface. With the help of laser cladding, the Fe-based [1–3], Ni-based [4–6], and Co-based [7–9] coating could be fabricated on the surface of parts. In addition, the outfield-assisted laser cladding process (e.g., electromagnetic field and ultrasonic) has been also employed to modify the microstructure and mechanical properties of coatings [10,11]. Recently, the new extreme high-speed laser cladding (EHLA) process has showed a great prospect as its huge advantages.

EHLA was a new method to fabricate thin coating. It has the advantages of high efficiency, low heat input, and low dilution [12–14]. Regarding traditional laser cladding method, the laser beam melts the substrate first. Then, the alloy powder is sent to the melting pool [12,15,16]. The schematic of the cross section of the EHLA feeding system is shown in Fig. 1a. The powder is sent to the focus point with the help of high-speed Ar gas as shown in Fig. 1b [12]. The focus point is exposed in the laser beam as shown in Fig. 1a. After being heated by the laser beam, the powder becomes liquid alloy and falls on the surface of substrate as shown in Fig. 1a. Generally, a liquid piller with a height of 1–2 mm will generate between the focus point and substrate.

As there is extreme high cladding speed, the coating always has an ultra-high cooling speed. The ultra-high cooling speed induces a remarkable non-equilibrium phase transition and a fine gran size. However, the non-equilibrium phase transition and the refine behavior of the EHLA coating have not been discussed until now. In addition, the interfacial metallurgical bonding theoretically generates between the coating and substrate. Interface widely exists in composites [17–20], joints [21–23], and coatings [24,25]. Regarding EHLA coating, the interfacial metallurgical bonding is hard to be observed using an optical microscope (OM) or a scanning electron microscope (SEM) due to its ultra-low dilution. The existence of interfacial metallurgical bonding is extensively doubted.
This investigation was aimed to reveal the solidification, phase transformation behavior, and interfacial metallurgical bonding of 1Cr17Ni2 EHLA coating on carbon steel. The 1Cr17Ni2 coating has a microstructure of martensite, which has the advantages of high hardness and excellent wear resistance. The microstructure of coating was characterized using electron backscattered diffraction (EBSD). Based on the reconstruction of prior austenite grain (PAG), the solidification of liquid alloy was discussed. In order to show the bonding mechanism between coating and substrate, the interfacial metallurgical behavior was characterized based on element and crystal analysis. Furthermore, the shear test of the interface was conducted to reveal the bonding property between coating and substrate.

2 Materials and experiments

The 1Cr17Ni2 powder with an average diameter of 30 μm and carbon steel pipe with a thickness of 50 mm were employed. The chemical compositions of the powder and steel pipe are shown in Table 1. The EHLA process with a power of 4.0 kW, a cladding speed of 50 m/min, and a feed powder speed of 30 g/min was used. The schematic of cladding process is shown in Fig. 2a–d. The macro cross-sectional morphology of EHLA coating is shown in Fig. 2e. The coating has a thickness of approximately 533 μm. In addition, there was no remarkable dilution from the optical image of cross section (in Fig. 2e).

Fig. 2 a–e Schematic of the EHLA process. d Schematic of the cross section of coating. e 1Cr17Ni2/carbon steel coating

Table 1 Chemical composition of powder and steel pipe (wt.%)

| Region          | Cr  | Ni  | C  | Mn | Si |
|-----------------|-----|-----|----|----|----|
| Powder/coating  | 17  | 2   | 0.1|    |    |
| Substrate       | 0.45| 0.5 | 0.2|    |    |

The microstructure of the coating and interface was characterized using the EBSD method. The data was analyzed using the Channel 5 software. The grain element distribution of the interface was characterized using EDS. The shear test was conducted with the help of fixture. The fractured surface was observed using SEM.

3 Results and discussion

3.1 Microstructure of EHLA coating

The microstructure of coating was characterized using EBSD. The orientation image is shown in Fig. 3a. The phase distribution image is shown in Fig. 3b. The red region and blue region were retained austenite (RA) and martensite, respectively. In this coating, the fraction of RA was approximately 0.8 vol.%. According to the grain orientation and grain boundary in Fig. 3, six PAGs were distinguished and indicated in Fig. 4a. It was observed that the PAGs were fine columnar grain with a width of approximately 8 μm. The {0 0 1} pole figure of the martensite in six PAGs is revealed in Fig. 4b. The blue point in every pole figure represents the orientation of martensite inner every PAG. Based on the K-S relationship of PAG and martensite, the orientation of {0 0 1} crystal planes of every PAG was indicated using red circle.

The pole figure of {0 0 1} crystal plane of PAGs is shown in Fig. 5a. According to the {0 0 1} pole figure, six pole points from six PAGs gathered as indicated in Fig. 5a. The schematic of crystal orientation of PAGs was illustrated in Fig. 5b. It was concluded that the direction of one of the {0 0 1} crystal planes was paralleled to the major axis of austenite grain in Fig. 4a.

Fig. 5 {0 0 1} orientation distribution of PAGs. a Pole figure of {0 0 1}. b Schematic of grain orientation of PAGs

The equilibrium phase diagram of the coating is shown in Fig. 6a. Generally, the δ-Fe phase precipitates from the liquid
first at 1500 °C during the cooling stage. At 1450 °C, the liquid completely becomes the δ-Fe phase. At 1350 °C, the δ-Fe phase starts transforming into the γ-Fe phase. However, the solidification of the coating was rapid for the EHLA process. It contributed to a non-equilibrium phase transformation of coating; the precipitation of the δ-Fe phase from liquid was inhibited. The γ-Fe phase directly precipitated from the liquid. Therefore, the schematic of the non-equilibrium phase diagram is shown in Fig. 6b. A small amount of the δ-Fe phase could still precipitate from liquid and transformed into the γ-Fe phase as the temperature decreases. Major γ-Fe directly came from the liquid along with the moving of the liquid-solid interface.

Two main parameters (the growth rate \( R \) and the temperature gradient \( G \)) determined the growth of γ-Fe grain. According to the research by Kou [26], the \( G/R \) ratio determines the solidification mode, while the \( G^*R \) value dominates the size of solidification structure as shown in Fig. 6c. For the EHLA process, the liquid metal falls on the surface of the steel pipe and forms a melting pool. There was a larger temperature gradient from the liquid melting pool to solidified cladding metal. According to the columnar grain of PAGs, the \( G/R \) ratio is located in the region of “cellular” in Fig. 6. Moreover, the grain size of PAGs was only approximately 8 μm. The fine structure revealed a large \( G^*R \) value. Therefore, the EHLA process with large \( G/R \) ratio and \( G^*R \) value contributed to the generation of fine columnar grain of PAGs as shown in Fig. 6c.

For γ-Fe grain, the crystal direction of [0 0 1] has the largest thermal conductivity. Therefore, the columnar γ-Fe grain has a [0 0 1] direction perpendicular to the liquid-solid interface which has the largest growth rate. In other words, the growth of γ-Fe grain with other orientation was inhibited.

As the continuous cooling of coating, the γ-Fe was retained to a lower temperature. When the temperature reached to martensite staring (Ms) temperature, the martensite transformation occurred. The γ-Fe gradually transformed into fine martensite. The Ms temperature of the coating was calculated based on Eq. (1) [27].

\[
\text{Ms} = 539 - 423C - 30.4Mn - 17.7Ni - 12.1Cr - 7.5Mo + 10Co - 7.5Si
\]

(1)

The results showed that the martensite transformation of coating occurred at approximately 286 °C. As the cooling rate of the EHLA coating is high, γ-Fe could not completely transform into martensite. A small amount of γ-Fe was retained to room temperature and contributed to the RA with a volume fraction of 0.8%.
3.2 Microstructure of interface

The microstructure of interface is shown in Fig. 7a, b. According to the phase distribution image (in Fig. 7b), a γ-Fe layer with a thickness of approximately 5 μm was observed. The SEM image of the interface is shown in Fig. 7c. The element distribution is measured using EDS and is shown in Fig. 7d. A transition layer with a thickness of approximately 5 μm was observed. The transition layer was precisely the γ-Fe layer in Fig. 7b. According to the Cr fraction and the calculation method in [27,28], the chemical compositions of the γ-Fe layer were calculated and are listed in Table 2.

Based on the chemical composition of coating and transition layer, the phase diagrams (in Fig. 8) were calculated using JMatPro software. According to Fig. 6a, the maximum fraction of γ-Fe in coating was approximately 88 vol.%. Regarding the transition layer, the maximum fraction of γ-Fe increased to 100 vol.% as shown in Fig. 8. It contributed to a higher retaining tendency of γ-Fe. In addition, the super high cooling rate of the interface also contributed to the retention of γ-Fe. Therefore, the γ-Fe in the transition layer was completely retained to the room temperature.

Based on the above results and discussion, the metallurgical bonding between coating and substrate relied on the γ-Fe transition layer. However, why could the γ-Fe phase bond the coating and substrate together?

3.3 Crystallographic relationship of interface

The crystallographic relationship between the interface and substrate was analyzed based on the pole figure of region 1 (as shown in Fig. 9a, b). The pole figures of γ-Fe of the transition layer and α-Fe of the substrate are shown in Fig. 9c, d. It can be observed that the {1 0 0} pole figure of γ-Fe in Fig. 9d originated from the {1 0 0} pole figure of α-Fe in Fig. 9c after rotating 45° around the pole indicated in Fig. 9c. The parallel relationship between the {1 1 1} pole figure of α-Fe and the {1 1 0} pole figure of γ-Fe was observed. It meant that there was a Bain relationship between the γ-Fe grain of the transition layer and α-Fe of the substrate [29]. The lattice relationship of the γ-Fe and α-Fe which has a Bain relationship is shown in Fig. 9e, f. The other γ-Fe grain in the transition layer was also found to have a Bain relationship with the neighboring α-Fe grain in the substrate.

The crystallographic relationship between the interface and martensite in coating is shown in Fig. 9g, h. According to the relationship of pole figure in Fig. 9g, h, the parallel K-S relationship was also observed between the martensite and γ-Fe in the transition layer [27,30]. It meant that there was a parallel relationship between the prior γ-Fe of martensite and γ-Fe in the transition layer was observed. In other words, the prior γ-Fe in coating and γ-Fe in the transition layer belonged to one γ-Fe grain when the temperature was higher than Ms temperature. After the temperature was below Ms temperature, the γ-Fe in coating transformed into martensite. Therefore, the martensite in coating had a K-S relationship with the γ-Fe in the transition layer.

Based on the above results and discussion, the metallurgical bonding of the transition layer/substrate and coating/transition layer relied on the Bain relationship and K-S relationship, respectively, in crystallography.

3.4 Shearing properties of interface

The bonding strength of the coating/substrate was analyzed using shear test. The stress-strain curves of interfacial sample and substrate are shown in Fig. 10a. The sample is shown in
Fig. 10b, c. It was observed that the maximum load of substrate and interfacial sample was approximately 467 MPa and 429 MPa, respectively. The interfacial sample also has a smaller displacement (approximately 0.51 mm). It was concluded that the shear strength of interfacial sample reached to approximately 92% of that of substrate.

The fractured surfaces of the two shear samples are shown in Fig. 10d–g. The EDS result showed that the marked region in Fig. 10f had a Cr fraction of approximately 11 wt.%. It meant that the fracture of interfacial sample preciously occurred in the transition layer. According to the micro morphology in Fig. 10e, g, dimples were observed on the fractured surface of substrate. The brittle shear fracture was observed on the fractured surface of interfacial sample.

Based on the above results and discussion, there was a strong bonding strength between the coating and substrate with the effect of interfacial γ-Fe. The shear test revealed that the shear strength of the interface reached to approximately 92% of that of substrate. However, the plastic deformation ability of interface was weaker than that of substrate.

4 Conclusions

Based on the above results and discussion, the following conclusions were derived:

1. The sound 1Cr17Ni2 EHLA coating with a thickness of approximately 533 μm was successfully fabricated on steel pipe. The coating was consisted of martensite and a small amount of RA (0.8 vol.%).
2. The high cooling speed resulted in the direct transformation from liquid to γ-Fe phase. The larger temperature gradient of melting pool and slow growth rate of PAG determined the generation of fine columnar PAG in coating. The largest thermal conductivity of [0 0 1] crystal direction contributed to the preferential [0 0 1] orientation of PAG perpendicular to the liquid-solid interface.
3. The transformation from PAG to martensite occurred at approximately 286 °C. The high cooling rate of the coating contributed to the retention of a small amount of γ-Fe (0.8 vol.%) in coating.
At the γ-Fe transition layer (approximately 5 μm) was observed between the coating and substrate. The expanded γ-Fe phase region and super-high cooling rate of the transition layer contributed to the complete retention of the γ-Fe phase.

The γ-Fe layer connected the coating and substrate via the special crystal orientation. There was a Bain relationship between the interfacial γ-Fe grain and α-Fe grain in substrate. There was a K-S relationship between the interfacial γ-Fe grain and martensite in coating. The two crystallographic relationships that originated from the interfacial γ-Fe layer contributed to the bonding between coating and substrate.

There was a strong bonding strength between the coating and substrate with the effect of interfacial γ-Fe. The shear test revealed that the shear strength of the interface reached to approximately 92% of that
of substrate. However, the plastic deformation ability of interface was weakened.

Acknowledgments All authors are particularly grateful to the medical staff who are fighting against COVID-19 for providing us with a safe research environment.

Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interest.

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