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1. An oxide-dispersion-strengthened (ODS) layer was formed on China Low Activation Martensitic (CLAM) steel by laser cladding.
2. The effects of Y_2O_3 content on microstructure, micro-hardness of cladding zone and tensile properties were studied.
3. The cladding zone shows a higher micro-hardness and tensile strength due to surface treatment to form a dispersed oxide layer.

Microstructure and Properties of the Laser Cladding ODS Layers on CLAM Steel

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Abstract

In this study, oxide dispersion strengthened (ODS) layers with different Y_2O_3 addition were cladded on China Low Activation Martensitic (CLAM) steel to improve its mechanical strength, micro-hardness and high-temperature behavior. The results show that fine surface morphologies can be observed in cladding zones. In cladding layers, the Fe, Cr, W, Y, Ti elements was discriminated by the scanning electron microscope (SEM) with energy-dispersive spectrometer (EDS) and the most stable Y–Ti oxide complexes $Y_2Ti_2O_7$ phase were analyzed by X-ray diffraction (XRD). The micro-hardness of ODS layers are higher than that of CLAM steel. The CLAM steel with 2wt.% Y_2O_3 addition ODS layer possesses an average tensile strength of 840MPa which is 166MPa higher than that of CLAM steel. It is suggested that the dispersion strengthening of oxide particles in ODS layer contributed to the increased strength.

Keywords: CLAM steel; Laser cladding; Oxide dispersion strengthened layer; Microstructure; Tensile strength

1. Introduction

China Low Activation Martensitic (CLAM) steel is a kind of reduced activation ferritic/martensitic (RAFM) steel which is chosen as candidate for structural materials in future fusion reactor applications considering their excellent swelling resistance [1-3]. The ton-scale CLAM steel can be produced by casting and rolling [4]. However, CLAM steels exhibit a low strength during the long-term thermal exposure in operation temperature around 550°C [5]. Oxide dispersion strengthened (ODS) steel was developed to improve its high temperature properties. The Y–Ti–O rich nano-scale clusters play key roles in ODS steels by pinning dislocation which can improve its thermal stability behavior [6-7]. ODS steels are fabricated by mechanical alloying, hot extrusion and spark plasma sintering (SPS) generally [8]. However, it is difficult to produce ODS steel by casting, because the oxide particles will agglomerate in the casting process, which results in the decrease of mechanical properties [9]. Recently, some researches about ODS coating on different substrate to improve the substrate properties have been done. Y.-H. Koo et al [10] had coated Y_2O_3 layer on Zircaloy-4 by laser beam scanning to increase its strength. With the formation of Y_2O_3 coating, the tensile strength increase by 20% at room temperature. Fu et al [11] investigated the effect of La_2O_3 content in ODS cladding layer on 316L steel. The corrosion resistance and the micro-hardness of ODS coated 316L can be improved when the addition amount of La_2O_3 is 0.4%, too much La_2O_3 cause inclusion and other defects which is unfavorable to the improvement of the performance. Laser cladding is a dominating and popular surface technology to fabricate coatings on substrate which has more advantages in large scale samples [12].

In this paper, ODS layers was cladded on the CLAM steel surface by laser cladding to increase its mechanical property and operating temperature. The effects of Y_2O_3 addition on

the microstructure, micro-hardness, tensile strength of the ODS coated CLAM steel were investigated.

2. Experimental procedures

The CLAM steel (Fe-8.93Cr-1.43W-0.48Mn-0.19V-0.1Ta-0.09C-0.05Si) was used as the substrate material with a thickness of 1.5mm. The chemical composition of ODS layers are shown in Table 1. The pre-deposit powders are prepared by elemental blended. The micromorphology and XRD results of powders are shown in Fig.1. Y_2O_3 exist in the XRD results of 2wt.% Y_2O_3 and 5wt.% Y_2O_3 addition ODS powders. No peak of Y_2O_3 was detected in XRD results of 1wt.% Y_2O_3 addition powders due to its low content. The powders are mainly spherical and the average diameter of the powders is about 5 μ m. In order to obtain clean substrates, the CLAM steel was sanded with metallographic sand paper, cleaned with acetone solution, alcohol successively and then dried. To form a pre-deposit layer, ODS powders were added in an agate bowl, mixed with distilled water and Carboxyl Methyl Cellulose (CMC). The mass ratio of ODS powders to water to CMC is 50:25:1. The ODS powders were wet-coated on the CLAM steel by a doctor blade and dried in an oven at 80 °C for 40 min to form a 200 μ m thick paste. In order to ensure the reliability and reproducibility of the test results, three specimens of every parameter were tested for analysis.

Table 1 Chemical composition (wt. %) of pre-deposit powders

Specimen	Fe	Cr	W	Ti	Y_2O_3
1	88.7	9	2	0.3	0
2	87.7	9	2	0.3	1
3	85.7	9	2	0.3	2
4	83.7	9	2	0.3	5

Fig.1. The XRD result and micromorphology of ODS powders

A 1000W pulsed Nd:YAG laser system was used for laser cladding. According to previous study and our great deal of tests, the laser power of 150W, 170W and 190W were chosen to discuss in this article. The scanning speed is 3.5 mm/s. The diameter of laser spot is 0.6mm, pulsed frequency is 25HZ, pulse width is 2ms, overlap rate is 50%, the defocusing distance was 0.5 mm. 12L/min Ar gas continuously blow on the samples' surfaces to prevent oxidation.

After laser cladding, metallographic samples were cut perpendicular to the cladding direction. The samples were grinded on emery papers and polished, then etched in Vilella's reagent for 35 second. Optical microscope (OM, HT630CN), scanning electron microscope (SEM, JSM-6490LV), energy dispersive X-ray spectroscopy (EDS, Oxford Inca Energy350) and X-ray diffraction (XRD, D/MAX2500V) were used to analyze microstructure, element distribution and phases in the cladding zone. Vicker's micro-hardness was measured by Warnway VTS401 at the cladding zone from top to substrate on depth direction of every laser track with a load of 50 g and a holding time of 10 seconds. Tensile strength was evaluated on CMT5150 Universal tester with a constant displacement rate of 1mm/min. The sample size

and macroscopic morphology of samples after tensile test are shown in Fig.2.

Fig.2. Tensile sample size and appearance of ODS CLAM specimens

3. Results and analysis

3.1 Surface and cross-sectional morphologies

Fig.3 shows the top surface and cross-sectional morphologies of cladding zone with 2wt.% Y_2O_3 addition under different laser power. The fine ripple patterns on the surface of cladding zone were observed. With the increase of laser power, cladding zone present incremental tendency in thickness. The certain depth of cladding zone is important to ensure the impact on the performance of CLAM steel [10]. At the power of 150W, the top surface of cladding zone shows relatively smooth while the cross-sectional morphologies indicate that the depth is very shallow. Too shallow depth of ODS layer make it difficult to affect its mechanical property and high-temperature strength of CLAM steel. At the power of 190W, the cladding zone exhibit a deeper depth while the spatters bring out a large number of ODS

powders pointed out with an arrow in Fig.3. Too high laser energy leads to many powders burning out. The top surface of cladding zone is relatively smooth with little burning loss at the power of 170W. The cross-sectional morphologies of cladding zone shows metallurgical bonding between ODS layer and CLAM steel. Take these two factors into account, laser power of 170W was selected in our subsequent experiments.

Fig.3. Surface and cross-sectional morphologies of cladding zone with different powers:
(a)150 W;(b)170 W;(c)190 W.

In order to ensure the good metallurgical combination of cladding material and substrate, a certain amount of matrix must be melted in the process of laser cladding, and the melted matrix will dilute the cladding layer. Proper dilution rate is a key to ensure the effective bonding of cladding and matrix [13]. The schematic diagram of dilution rate was shown in Fig.4. The area of deposit (A_d) and molten area (A_m) under different laser powers were calculated by Image Pro-plus. The dilution rate can be approximately calculated by the following equation:

$$D_c = \frac{A_m}{A_d + A_m} \quad (1)$$

According to Fig3, with the laser power of 150W, the dilution ratio is 39.7%. With the laser power of 170W, the dilution ratio is 66.1%. The dilution ratio is 70.1% with the laser power of 190W. The results show that the dilution rate increases with laser power increase. At too low dilution rate, the pre-deposit powders may not be completely integrated with the substrate. Too high dilution might result in the loss of pre-deposit powders effect. In our subsequent experiments, the laser power of 170W was chosen to study other effects on clad layer.

Fig.4 Schematic diagram of dilution rate

3.2. Microstructure analysis of samples after laser cladding

Fig.5 and Fig.6 show the cross-sectional microstructures of ODS clad CLAM steel with different Y_2O_3 addition in different magnification. Owing to different coefficient of thermal expansion between coating layer and substrate, residual stress may arise. If the residual stress exceed to the yield strength of the metal, the crack will occur [14]. The powders shown in table 1 possess similar thermal expansion and excellent wettability with CLAM steel due to their similar chemical composition [15-16]. The cross section morphology between coating layer and substrate in Fig.5 indicates fine metallurgical bonding with few pores and cracks. Three areas with different microstructure can be observed, the cladding

zone, heat affected zone, and substrate. As shown in Fig.5 and Fig.6, the microstructure of cladding zone and heat affected zone (HAZ) is mainly composed of equiax crystal and columnar crystals. The cladding process is a high temperature gradient and high speed solidification process, the liquid metal in the bottom of cladding zone is always close to the solid substrate which possesses highest cooling velocity. As shown in Fig.6a, the microstructure of center cladding zone with Fe-Cr-W-Ti powders is mainly composed of equiaxed grain. The average grain size in cladding zone calculated by Image Pro-plus is $50.9\mu\text{m}^2$. Microstructures of cladding layers with 1wt.%, 2wt.%, 5wt.% Y_2O_3 addition were shown in Fig.6b, c, d respectively. In Fig.6b, with 1wt.% Y_2O_3 addition, the number of columnar crystals decreased significantly and the equiaxed grain was refined. The average grain size in center cladding zone with 1wt.% Y_2O_3 is $19.2\mu\text{m}^2$. When the addition of Y_2O_3 is 2 wt.%, the grain size of the cladding zone was further refined, and the average grain size in center cladding zone decreased to $4.5\mu\text{m}^2$. The melting point of Y_2O_3 and other yttrium oxide is higher than that of Fe, Cr, W and Ti. The solidification process of particles containing rare earth is earlier than that of Fe, Cr, W and Ti. These pre-solidified or unmelted particles provide sufficient nucleation centers. The rare earth oxide has a dragging effect on the grain boundary, which greatly inhibits the grain growth and results in the refinement of the microstructure [17]. Oxide dispersed phase can hinder the movement of dislocation and grain boundary, slow down the diffusion rate of metal atoms which leads to strengthening effect [18]. The content of Y_2O_3 can make great difference to mechanical properties and oxidation resistance of ODS alloys at high temperature [19]. Excessive yttrium oxide may cause the accumulation of yttrium oxide and reduce the effect of grain refinement [20]. While Y_2O_3 addition increases to 5wt.%, defects such as slag inclusion, segregation and grain growth

occurred in the cladding zone, and the average grain size in center cladding zone is $7.5\mu\text{m}^2$. S. Ohtsuka et al found that 1.5wt.% Y_2O_3 ODS alloys decreased the density of oxide particles in equiaxed crystal and did harm to the scale and number of Y–Ti–O oxide, leading to the decrease of strength [20].

Fig.5. The microstructure of cross-section with different Y_2O_3 addition (a) 0wt.% Y_2O_3 (b) 1wt.% Y_2O_3 (c) 2wt.% Y_2O_3 (d) 5wt.% Y_2O_3

Fig.6. The microstructure of cross-section with different Y_2O_3 addition (a) 0wt.% Y_2O_3 (b) 1wt.% Y_2O_3 (c) 2wt.% Y_2O_3 (d) 5wt.% Y_2O_3

The sample with 2wt.% Y_2O_3 addition was chosen to analyze what elements and phases existent in the top surface and cross-section. The SEM-EDS analysis results in Fig.7 indicate the presence of different composition on the top surface and cross-section. During laser cladding, part of the powder is metallurgically bonded with the substrate and small part of powders splash out. Fig.7a showed the existence of Fe, Cr, Y, and Ti elements on the top surface. In the interval of two laser cladding beads, Fe, Y, Ti and Cr elements accumulation were observed due to the impact force of laser. Relatively few Fe, Y, Ti and Cr elements was observed at the center area of every single bead because the powders in these areas react metallurgically with the matrix and mixed into the matrix. The cross-sectional microstructures were shown in Fig.7b. It is found that cross-section zone contains Fe, Y, Ti and Cr elements, which indicates that the pre-deposit powders and substrates are mixed in the cladding layer. During laser cladding process, laser energy transfers to CLAM steel through pre-deposit ODS

powders by convection and conduction to form molten pool. Thus, metallurgical bonding between the ODS powders and substrate has taken place [21].

Fig.7. The SEM-EDS mapping (a) top surface result (b) cross-sectional result

3.3 XRD analysis

Fig.8 show X-ray diffraction patterns of the cladded layer top surface with 2 wt.% Y_2O_3 and 5wt.% Y_2O_3 addition. It can be observed from Fig.8 that XRD results reveal the presence of $Y_2Ti_2O_7$, $FeCr_2O_4$ and bcc Fe-Cr solid solution. During laser cladding, the preplaced coating powders were fully mixed into the substrate and decomposed into Y and O atoms which combined with Ti atoms to form $Y_2Ti_2O_7$ [22]. $Y_2Ti_2O_7$ phases can inhibit the dislocation movement, improve the strength under loading and improve radiation resistance of the materials, and also play a role in grain refinement [23]. As shown in Fig.8, $FeCr_2O_4$ was also found. A small quantity of $FeCr_2O_4$ can increase the density of the ODS layer and

improve the oxidation resistance of the substrate according to Zhou et al [24]. The diffraction peak of some phase may not be detected by XRD due to their low content in multiphase mixture. It is suggested that peak of Y_2O_3 and $Y_2Ti_2O_5$ was not found in Fig.8 in our experiment due to their possible low content [25].

Fig.8. X-ray diffraction spectra of cladding layers (a) 2wt.% Y_2O_3 addition (b) 5wt.% Y_2O_3 addition

3.4. Hardness distribution

Fig.9. Hardness distributes from top to substrate on depth direction with different Y_2O_3

addition

The average Vickers hardness distributions from top to substrate on depth direction of five tracks were shown in Fig.9. The point interval of the hardness test was 50 μ m. As shown in the Fig.9, the hardness of cladding zones with different Y₂O₃ addition are all higher than that of substrates. High cooling rate during solidification of the cladding zone, different composition and the difference in microstructure will account for the change. The average hardness of substrate was 202 HV and the cladding zone with Fe-Cr-W-Ti powders has the average hardness of 321 HV. In the cladding zone with Fe-Cr-W-Ti powders, the microstructure is mainly composed of columnar crystals and Fe-Cr solid solution and other harder compounds may exist. The cladding zone with different Y₂O₃ addition 1wt.%, 2wt.%, 5wt.% Y₂O₃ exhibit average micro-hardness of 282Hv, 313Hv, 304Hv, respectively. With Y₂O₃ addition, the hardness increase contributes to the mutual effects of forming harder compounds, grain refinement hardening and dispersion hardening of Y₂O₃. With 2wt.% Y₂O₃ addition, the grains size is the finest in the cladding zone which the average hardness is the highest. Excessive addition of Y₂O₃ may cause aggregation of yttrium oxide, slag inclusion. When Y₂O₃ addition increases to 5wt.%, the grains of the cladding region become coarser, and the effects of grain refinement strengthening and oxide dispersion strengthening are less obvious, and the average hardness is lower than that of 2wt.% Y₂O₃ addition.

3.5. Tensile property

Fig.10.The tensile stress and elongation of CLAM steel with different ODS layer

The tensile strength of CLAM steel can be improved effectively by forming ODS layers on the surface. Fig.10 shows the tensile strength and elongation of CLAM steel with different ODS layers. The CLAM steel and laser-beam-scanned CLAM steel was tested as contrast. The tensile strength and elongation of CLAM steel were about 674 MPa and 14.8%. The strength of CLAM steel was enhanced to 697 MPa and elongation was decreased to 14.4% after scanned by laser beam. Rapid cooling after laser scanning and the hardening laser scanned zone in the scanned joint account for the strength increase and elongation decrease. When the CLAM steel was scanned with Fe-Cr-W-Ti powders, the tensile strength was 713MPa while with much lower ductility. Fig.11 shows the fracture morphology of Fe-Cr-W-Ti layer. The cladding zone displayed a characteristic of typical brittle fracture without dimple at the surface layer. Under loading, once the load exceeds the tensile strength

of the cladding zone, cracks will occur in the cladding zone. As the load continues, the crack may spread quickly to the substrate. The tensile strength of ODS clad samples with 1wt.%, 2wt.%, 5wt.% Y_2O_3 addition exhibited 780MPa, 840MPa and 792MPa respectively, which are higher than that of CLAM steel and laser scanned sample. The sample with 2 wt.% Y_2O_3 addition showed a highest tensile strength of 840MPa and elongation of 13.4%. The increased strength of ODS layer and the ductility mainly provided by the substrate contribute to an excellent mechanical property of clad CLAM steel. The ODS layer showed brittle fracture while the substrate is ductile, which was shown in Fig.12. The fracture morphologies of the sample scanned by laser beam with 2wt.% Y_2O_3 addition were shown Fig.12b. The results indicate that the increased strength of clad samples was mainly caused by the oxide dispersion strengthening in ODS layers. The oxide particles can interfere the dislocation movement under loading, which leads to the strength increase in the ODS samples [26]. Due to the lower ductility of ODS layer, the fracture occurred initially in the ODS layer and then extended to the interior CLAM steel substrate during the tensile test. Similar situation occurs in the sample with 5wt.% Y_2O_3 addition. Since oxide particles are stable at high temperatures, it is inferred that ODS clad CLAM steel will possess relatively higher tensile strength than that of CLAM steel itself at high temperatures. Summarize the above results, the tensile strength of the ODS clad CLAM steel with 2wt.% Y_2O_3 addition reaches the highest value with moderate elongation in our experiments, which show that the appropriate Y_2O_3 addition of ODS layer should be around 2wt.%.

Fig.11. Microstructures on the fracture surface of the CLAM steel with Fe-Cr-W-Ti powders

Fig.12. Microstructures on the fracture surface of the CLAM steel with 2wt.% Y_2O_3 content ODS powders

4. Conclusion

The ODS layer with different Y_2O_3 content was added to CLAM steel by laser cladding. The microstructure and properties of the laser cladded ODS layers on CLAM steel have been investigated and the following conclusions are summarized.

- (1) ODS layer nearly without pores and cracks were formed on CLAM steel at the laser power of 170W and scanning speed of 3.5mm/s.
- (2) The cladding zone exist Fe, Y, Ti and Cr elements. The most stable Y–Ti oxide complexes $Y_2Ti_2O_7$ was observed on the top surface of cladding zone. The micro-hardness of all the coating layers are higher than that of substrate.
- (3) Different Y_2O_3 addition in ODS powders significantly affects the microstructure of cladding zone and the properties of ODS cladde CLAM steel. The tensile strength of CLAM steels with different ODS layers are all higher than that of CLAM steel itself. The appropriate Y_2O_3 addition of ODS layer should be around 2wt.% to reach the optimum properties of ODS cladde CLAM steel in our experiments.

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