Study on microstructure and mechanical properties of 4Cr5MoSiV1 steel manufactured by selective laser melting

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Abstract. The 4Cr5MoSiV1 steel samples were manufactured by selective laser melting technique. The effects of laser line energy density on microstructure, microhardness and wear resistance were analyzed. The relationship between microstructure and mechanical properties was studied. The results show that when the laser line energy density is too high, the grain size of the sample is larger, and there is a small amount of granular second phase precipitates in the sample. When the laser line energy density is too low, the grain size of the sample is small, and the grain shape is irregular, and there are obvious pores. When the laser line energy density is 633J•m⁻¹, the microstructures of the samples are uniform in size and regular in shape. The highest microhardness is 682.3HV, and the lowest wear rate is 0.425×10⁻¹⁰kg•N⁻¹•m⁻¹.

1. Introduction

Mold is the basic equipment of modern industry, mainly used for mass production of parts and components in industrial products. At present, traditional machining and EDM are mainly used in mold processing, which have many problems such as low processing efficiency, poor precision and poor complex manufacturing capacity. 4Cr5MoSiV1 die steel belongs to chromium hot work die steel, which has high strength, toughness, hardening and fatigue resistance. It is widely used in casting, forging and hot extrusion die production[1]. The microstructure of the traditional cast 4Cr5MoSiV1 die steel is composed of coarse dendrite and carbide, and macrosegregation exists. The material is mainly subjected to high temperature, high pressure and corrosion, impact, friction, etc., prone to wear, corrosion, thermal crack, plastic deformation and mechanical fatigue and other failure forms, which seriously affect its service life[2].

Selective laser melting (SLM) technology, based on the principle of discrete/stacking, uses high-energy laser beam to realize point-line-plane-body machining process, which is especially suitable for parts with complex structures. It is one of the most promising metal additive manufacturing technologies[3-5]. At present, this technology has been applied in some high-end injection molds, showing obvious advantages in uniformity, cooling efficiency, manufacturing cycle, manufacturing cost and so on. In the manufacture of complex molds using SLM technology, the molded parts must have excellent mechanical properties, such as high hardness and wear resistance, etc., which are closely related to the density and microstructure of the molded parts.

In order to optimize the process parameters of SLM forming 4Cr5MoSiV1 steel and improve the mechanical properties of the manufactured parts, the influence of process parameters on the microstructure, microhardness and wear resistance of the manufactured parts was firstly studied in this
paper, and then the evolution mechanism of microstructure, wear mechanism and the influence mechanism of microstructure on mechanical properties of the manufactured parts were discussed. Thus, it provides the basis for improving the mechanical property optimization of SLM manufactured 4Cr5MoSiV1 steel, and expands the application field of SLM manufactured 4Cr5MoSiV1 steel.

2. Experimental conditions and methods

2.1 Experimental equipment
DiMetal-50 SLM manufacturing equipment was used in the experiment. The equipment was equipped with a 200W SPI fiber laser with a laser wavelength of 1070nm and a spot diameter of 70μm. The maximum size of the parts manufactured in this equipment is 50mm×50mm×50mm. During the molding process, high purity AR gas is filled for protection, and the oxygen content of the molding chamber is less than 0.1%.

2.2 Experimental materials
The 4Cr5MoSiV1 die steel powder produced by air atomization was used in the experiment. The chemical composition of the powder is shown in Table 1, and the particle size of the powder is distributed in the range of 15-45μm. Before the experiment, the powder was processed by vacuum drying oven.

| Element | C  | V  | Mo | Ni | Si | Cr  | Mn | P  | S  | Fe   |
|---------|----|----|----|----|----|-----|----|----|----|------|
| Content | 0.39 | 1.05 | 1.27 | 0.23 | 0.87 | 5.24 | 0.48 | 0.019 | 0.01 | Balance |

2.3 Experimental method
The block sample and wear sample of 4Cr5MoSiV1 steel were manufactured by DiMetal-50 SLM manufacturing equipment. The size of block sample was 10mm×10mm×5mm, and the size of wear sample was 20mm×20mm×2mm. The laser scanning process parameters of 4Cr5MoSiV1 steel specimen manufactured by SLM are as follows: Laser power P=190W; Powder layer thickness H=25μm; Scanning spacing S=70μm; The scanning mode is S-type orthogonal. Scanning speed V=200mm/s, 300mm/s, 400mm/s, 500mm/s. The laser line energy density is defined as η(η=P/V), and the corresponding η is 950J•m⁻¹, 633J•m⁻¹, 475J•m⁻¹, and 380J•m⁻¹, respectively. The sample is cut from the substrate by wire cutting, and then cleaned by ultrasonic wave with acetone. Then the block sample and the worn sample are polished and polished. Then the nitric acid alcohol corrosion solution of 4% is selected to corrode the surface of the block sample. The weight loss and friction coefficient of the samples were measured by HT-1000 ball-disc friction and wear testing machine at room temperature. In the friction and wear experiment, the grinding material is 45 steel, the friction radius is 5mm, the experimental load is 1000g, the rotational speed is 300r/min, and the time is 30min. Then HXS-1000AK microhardness tester was used to measure the microhardness of the block sample surface. The experimental loading load was 200g and the loading time was 15s. Finally, the Ultra55 field emission scanning electron microscopy was used to observe the microstructure of the block samples, the wear mark morphology of the wear samples and the fracture morphology of the tensile samples.

3. Results and discussion

3.1 Microstructure analysis
SLM is a metallurgical process of rapid melting/solidification. The extremely fast cooling rate makes the microstructure of SLM molded parts significantly different from that of traditional manufacturing parts. The microstructure of SLM molded samples is shown in Figure 1. As can be seen from Figure 1, the microscopic structure of the sample is relatively uniform cellular structure. When the laser line energy density is 950J•m⁻¹, the grain size of the sample is larger, and there is a small amount of
granular second phase precipitates in the sample. When the laser line energy density is 633J•m\(^{-1}\), the grain shape of the sample is regular, and the grain size and distribution are uniform. When the laser line energy density is 475J•m\(^{-1}\), the grain size of the sample decreases and the grain distribution is not uniform. When the laser line energy density is 380J•m\(^{-1}\), the grain size of the sample decreases obviously, and the grain shape is irregular, and there are obvious pores.

In the solidification process of liquid molten pool, the heat at the boundary of molten pool is mainly conducted through solidified metal. Because the grain growth direction is opposite to the direction of heat flow and has the largest temperature gradient along the direction perpendicular to the pool boundary, the grain tends to grow along the direction perpendicular to the pool boundary. The laser beam with Gaussian energy distribution causes the driving force of fluid flow in the molten pool, which mainly includes the buoyancy force and the shear stress caused by the surface tension gradient\(^6\). As shown in Figure 2, the density \(\rho\) of the liquid metal decreases with the increase of temperature \(T\), so the liquid metal decreases along the edge of the molten pool and rises along the axis of the molten pool. The shear stress caused by the surface tension gradient is shown in Figure 3. During SLM process, a temperature gradient is generated on the surface of the molten pool, and the surface tension \(\gamma\) of the liquid metal decreases with the increase of the temperature \(T\), which causes the liquid metal to flow from the center of the molten pool surface to the boundary of the molten pool and return below the molten pool surface. Because the driving force of fluid flow can cause the fragmentation of directionally grown grains and the formation of nuclei needed for new grains, the cellular structure is formed in the central region of the molten pool.

In the process of laser beam melting metal powder layer by layer, the solidified metal will be subjected to the complex thermal action of continuous action and periodic change. When the laser beam melts the current material, part of the heat absorbed by the material will be quickly transferred to the solidified channel and the underlying metal, thereby changing the temperature distribution of the solidified metal. This process is called subsequent thermal cycle\(^7\). The subsequent thermal cycle plays a tempering role on the solidified channel. When the laser line energy density is too high, the carbon element in the supersaturated solid solution precipitates in the form of carbide, forming granular second phase precipitates. When the energy density of the laser line is low, the energy absorbed by the metal powder is insufficient, resulting in a relatively small amount of liquid phase and a low temperature of liquid phase. The lower liquid temperature increases the dynamic viscosity of the liquid, reduces the wettability and fluidity of the melt, and easily leads to the pore defects in the sample.
3.2 Microhardness analysis

The average microhardness of samples with different laser line energy densities is shown in Figure 4. As can be seen from Figure 4, with the increase of laser line energy density, the microhardness of the sample shows a trend of increasing at first and then decreasing. When the laser line energy density is 633J•m\(^{-1}\), the microhardness value of the sample is the highest, which is 682.3HV. When the laser line energy density is 380J•m\(^{-1}\), the pore defects of the sample are more and the grain size is not uniform, so the microhardness value is low, which is 586.7HV. When the laser line energy density is 950J•m\(^{-1}\), the grain size of the sample is larger, and there is a second phase precipitates, the solid solution strengthening and fine grain strengthening effect is reduced, so the microhardness is low, 666.2HV.

3.3 Wear resistance analysis

In the experiment, an electronic balance with an accuracy of 0.01mg was used to measure the wear amount of the sample, and then the wear resistance was evaluated by calculating the wear rate\(^{[8]}\), namely:

\[
\omega = \frac{\Delta m}{F \cdot S}
\]

Where: \(\omega\) is the wear rate; \(\Delta m\) is the wear quantity; \(F\) is the experimental load, \(F=10N\); \(S\) is the gliding distance, \(S=282.6m\).

The wear rates of samples under different laser line energy densities are shown in Table 2.

| Sample | Linear energy density \(\eta\) / J•m\(^{-1}\) | Wear loss weight \(\Delta m\) / mg | Wear rate \(\omega\) / \(10^{-10}\) kg•N\(^{-1}\)•m\(^{-1}\) |
|--------|------------------------------------------|----------------------------------|---------------------------------|
| 1      | 950                                      | 0.26                             | 0.920                           |
| 2      | 633                                      | 0.12                             | 0.425                           |
| 3      | 475                                      | 0.39                             | 1.380                           |
| 4      | 380                                      | 0.51                             | 1.804                           |

As can be seen from Table 2, with the decrease of laser line energy density, the wear of the sample decreases first and then increases. When the laser line energy density is 633J•m\(^{-1}\), the wear rate of the...
sample is the lowest, which is $0.425 \times 10^{-10} \text{kg} \cdot \text{N}^{-1} \cdot \text{m}^{-1}$. When the laser line energy density is $380 \text{J} \cdot \text{m}^{-1}$, the wear rate of the sample is the highest, and the wear rate is $1.804 \times 10^{-10} \text{kg} \cdot \text{N}^{-1} \cdot \text{m}^{-1}$.

Figure 5 shows the wear trace morphology of samples at different forming angles. The results of EDS analysis show that oxidation phenomenon exists in the friction process of the sample, and there are obvious pits on the surface of the sample, and the color is dark. This is because a large amount of frictional heat generated in the grinding process raises the temperature of the local area of the sample, leading to the formation of an oxide layer on the surface after oxidation, and the oxide layer is prone to fatigue under the action of repeated friction rolling shear stress. After a certain number of loading and unloading cycles, part of the oxide layer on the surface of the sample falls off and finally forms pits.

As can be seen from Figure 5, the surface of the worn sample is characterized by adhesion pits, adhesion tracks, oxide layer, oxide layer shedding zone and a small amount of furrows. This shows that the wear mechanism of the wear samples is mainly adhesive wear and oxidation wear, accompanied by a small amount of abrasive wear. With the decrease of laser line energy density, the wear degree of the sample decreases first and then increases, which corresponds to the test results of wear rate. When the laser line energy density is low, the oxide layer and the oxide layer shedding area increase, the area increases, the adhesion track and the adhesion pit increase, and the furrow is obvious.

Figure 5. Wear morphologies of samples
(a) $\eta = 950 \text{ J} \cdot \text{m}^{-1}$; (b) $\eta = 633 \text{ J} \cdot \text{m}^{-1}$; (c) $\eta = 475 \text{ J} \cdot \text{m}^{-1}$; (d) $\eta = 380 \text{ J} \cdot \text{m}^{-1}$

4. Conclusion
(1) The microstructure of 4Cr5MoSiV1 steel sample manufactured by SLM is cellular. When the laser line energy density is $633 \text{J} \cdot \text{m}^{-1}$, the grain shape, grain size and distribution of the sample are uniform. When the laser line energy density is $950 \text{J} \cdot \text{m}^{-1}$, the grain size of the sample is large, and there are granular second phase precipitates. When the laser line energy density is $380 \text{J} \cdot \text{m}^{-1}$, the grain shape of the sample is irregular and there are pore defects.

(2) With the increase of laser line energy density, the microhardness of the sample increases firstly and then decreases. When the laser line energy density is $633 \text{J} \cdot \text{m}^{-1}$, the microhardness of the sample is the highest, which is 682.3HV.

(3) The wear mechanism of SLM manufactured 4Cr5MoSiV1 steel specimen is mainly adhesive wear and oxidation wear, accompanied by a small amount of abrasive wear. When the laser line energy density is $633 \text{J} \cdot \text{m}^{-1}$, the wear of the sample is the lowest, which is $0.425 \times 10^{-10} \text{kg} \cdot \text{N}^{-1} \cdot \text{m}^{-1}$.

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