Effect of nano TiC on microstructure and microhardness of composite additive manufacturing 316L stainless steel

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Abstract
The lower surface hardness limits the further application of 316L stainless steel. In this study, selective laser melting (SLM)/laser metal deposition (LMD) composite additive manufacturing technology was used to prepare five kinds of 316L-nano-TiC cermet strengthening layers on the surface of 316L stainless steel, and to study the effect of nano-TiC particle content on the microstructure and the influence of microhardness. Use Laser microscope, scanning electron microscope (SEM), X-ray diffractometer (XRD) to analyze the structure, element distribution and phase composition of the strengthening layer. The hardness of the strengthened layer was analyzed using a Vickers microhardness tester. The study found that the composite SLM/LMD formed samples changed continuously from LMD forming to SLM forming, showing good metallurgical bonding. Diffusion of TiC particles was observed in the SLM strengthening layer, and TiC phase was detected in the strengthening layer. Compared with the 316L matrix, the microhardness of the strengthened layer is significantly improved. When 50 wt% TiC is added, the average hardness of the strengthened layer is 1.9 times that of the 316L matrix, and the highest is 408.9 HV. The results of this study show that the strengthening layer manufactured by composite additive materials can effectively improve the hardness of the 316L stainless steel matrix. As the content of nano-TiC in the preset powder increases, the microhardness of the strengthening layer first increases and then decreases, and the hardness of the 50 wt% TiC strengthening layer is the highest. There are distributed nano-TiC particles in the structure of the strengthening layer, and the distribution of nano-TiC particles in the 50 wt% TiC strengthening layer is more uniform than other samples. This research provides a new reference for the strengthening of 316L stainless steel through SLM/LMD composite additive manufacturing technology and the addition of nano-TiC particles.

1. Introduction
The Mo content in 316L stainless steel makes the steel has excellent resistance to pitting corrosion, and has a widerange of applications in chemical machinery, biomedicine and other industries [1]. However, the low-strength, low-surface hardness 316L stainless steel is easy to be destroyed undercertain high-strength and complex working conditions, which limits its use in equipment with high performance requirements [2, 3]. Therefore, the surface of 316L steel needs to be strengthened to improve its surface properties.

In order to meet the needs of metal alloys with multiple composite properties, metal matrix composites (MMCs) have received great attention due to their simultaneous improvements in hardness, strength, wear resistance and corrosion resistance [4]. The metal matrix composite material strengthening layer is a composite strengthening layer manufactured artificially by using metal or metal alloy as the matrix and using one or more metal or non-metal strengthening phases. Cermet reinforced layer is a common high-performance metal matrix composite reinforced layer. The metal matrix composite ceramic reinforced layer has some advantages of both metal and ceramics, such as the excellent mechanical properties of metal and the good wear resistance of...
ceramics [5, 6]. This composite reinforced layer has the characteristics of good comprehensive performance, such as stable structure, wear resistance, high hardness, etc., which can expand the use range of metal materials. In the traditional manufacturing process, metal matrix composites are manufactured by casting [7, 8] and powder metallurgy [9, 10]. These processing processes are complicated and time-consuming, and are prone to coarse grains and uneven distribution. Condition. In contrast, composite materials produced by SLM/LMD have finer grains due to the fast cooling rate. In addition, the melting zone during SLM/LMD processing is very small, which can prevent particle separation, and can be a potential method for manufacturing metal matrix composites with dispersed ceramic particles [11–13]. Although the current process of manufacturing metal matrix composites is becoming more and more mature, the existing research mainly focuses on the influence of laser power, laser scanning speed, laser remelting and other processes on the structure and performance of the strengthening layer, while the research on the original powder ratio less. Therefore, a detailed study on the proportion of composite powder content is of great value for clarifying the strengthening mechanism of the strengthening layer and expanding its use range.

In recent years, there have been many experiments on SLM/LMD preparation of metal-based composite strengthening layers [14–16]. Studies have found that the added ceramic elements can change the surface properties of metals. Among them, there are several typical ceramic reinforcement phases, such as carbonization. Compounds and borides [17, 18] have been used to develop stainless steel matrix composites. The advantage of these reinforcing particles is that they can effectively participate in the grain refinement of metal matrix composites and improve the surface quality and mechanical properties of the material. Wengang Zhai [19] used SLM technology and added 1% and 3% TiC micron particles to make the printed 316L-1%TiC and 316L-3%TiC yield strengths of 660 MPa and 832 MPa. Analysis shows that this strengthening comes from grain refinement and second phase strengthening. Zhao Z et al [20] added micron-sized TiC particles to 316L for SLM processing, and found that the microhardness and wear resistance increased with the addition of TiC particles. AlMangour et al [21] manufactured TiB2/316L composite material through SLM, which exhibits higher yield strength and ductility than pure iron at room temperature and high temperature. Zhao S et al [22] studied the effects of different TiC mass fraction, particle size and processing parameters on TiC/316L composites. Wilson et al [23] manufactured Inconel690/TiC gradient composites by laser metal deposition and found that the addition of TiC enhanced the hardness and wear resistance, and when the content of TiC exceeded 30%, the microstructure was refined. Recently, graphene has attracted the attention of researchers as a reinforcing material. Han Y et al [24] studied the performance of low-content nano-graphene reinforced 316L stainless steel, and found that the strength was increased by 17.9% compared with ordinary 316L, and the ductility was not affected. Mandal et al [25] used laser additive manufacturing technology and found that graphene-enhanced 316L composite material has self-lubricating properties, high wear resistance and low friction coefficient. Among the commonly used reinforcing phases, TiC is a very stable interstitial compound with high melting point, high hardness, high elastic modulus, high chemical stability, good wear and corrosion resistance, and it has good chemical compatibility with stainless steel [26, 27]. If nano TiC particles are used as the reinforcing phase, it is possible to prepare a 316L-TiC strengthening layer on the surface of 316L stainless steel. SLM technology is mostly used for additive manufacturing of a single metal material, and it is more difficult to additively manufacture two materials. So this study adopts the SLM/LMD composite additive manufacturing method.

In this study, nano TiC particles were added as a strengthening phase, and selective laser melting (SLM)/laser metal deposition (LMD) composite additive manufacturing technology was used to manufacture 316L-TiC strengthening layers with different TiC content on the surface of 316L stainless steel. To study the effect of TiC particle addition on the microstructure and microhardness of the strengthening layer.

2. Experimental details

2.1. Powder preparation and composite additive manufacturing process

The base material of this test is 316L stainless steel, and its chemical composition (in mass fraction) is: 16.8% Cr, 12.7% Ni, 2.5% Mo, 1.5% Mn, 0.1% Si, 0.03% C, iron balance. The strengthening layer material is 316L-TiC mixed powder, which contains 0, 10, 30, 50,70 wt.% of nano-TiC particles. To ensure that the nano TiC particles are evenly dispersed in the 316L powder, the 316L powder and the TiC powder are placed in a ball mill and mixed for 30 min. The composite additive manufacturing method is to perform SLM forming on the LMD forming 316L matrix, and both SLM and LMD forming adopt the filling and contour scanning strategy. The size of the 316L substrate formed by LMD is 30 mm × 10 mm × 10 mm, and the mixed 316L-TiC powders are spread on the 316L substrate respectively, and the height and width of the powder layer are 1 mm and 3.5 mm, respectively. The Raycus RFL-A2000D fiber laser is used for laser deposition processing, and the movement is executed by the ROKAE XB16 six-axis robot. In the laser additive manufacturing process, high-purity argon (99.99%) is used as a protective gas to avoid oxygen contamination. The Manufacturing process is shown in...
On the basis of a series of preliminary experiments, five samples with different TiC content were produced in this experiment with a laser power of 1200W and a scanning speed of 1 mm s$^{-1}$.

Use a wire cutting machine to cut the metallographic sample whose surface is perpendicular to the scanning direction, first use 360#, 1000#, 1500#, 2000#, 2500# water-resistant sandpaper for treatment, until the surface of the metallographic sample is flat and the direction of the small scratches is the same. Afterwards, 1.5 $\mu$m and 0.5 $\mu$m diamond polishing agents are used for polishing. The polishing disc is turned perpendicular to the direction of the last scratch, and the direction of the sample is changed from time to time until the surface of the metallographic part is as smooth as a mirror, and no obvious scratches can be seen under the metallographic microscope. After polishing, it is corroded in ferric chloride and hydrochloric acid etching solution for 1 min. After corrosion, the sample is ultrasonically cleaned.

2.2. Microstructure characterization and Microhardness measurements
A Rigaku Ultima IV X-ray diffractometer (XRD) was used to characterize the phase of the strengthening layer. The X-ray source was CuKα, the wavelength was 0.154 nm, the working voltage was 40 kV, the current was 30 mA, and the diffraction angle 2θ range was 30°-90°, the test rate is 10°/min, and the step size is 0.02°.
A scanning electron microscope (Nova Nano SEM450) and an energy dispersive X-ray spectrometer (EDS, Oxford Swift 3000) were used to observe the morphology and composition of the strengthening layer. Use HXD-1000TMC/LCD type Vickers microhardness tester to test the microhardness of the sample. The test method is as follows: Measure 10 points at intervals of 1 mm from top to bottom along the cross-section in the strengthened layer. The working load is 300 g and the loading time is 10s.

3. Results and analysis

3.1. Phase analysis

Figure 3 shows the XRD patterns of the 316L-nano TiC strengthening layer with different contents. It is found that the diffraction peaks of the (Fe, Ni) solid solution of all samples are the strongest, which proves that there are a large number of (Fe, Ni) solid solution phases in the strengthening layer. This is because the atomic radii of Fe and Ni are 1.27 Å and 1.24 Å, respectively, and the electronegativity is 1.83 and 1.9, respectively. The two have similar electronegativity and atomic radii, and they are easily combined into phases in the laser molten pool [28].

![Figure 3. XRD pattern of 316L-nano TiC strengthening layer (a) 316L; (b) 316L-10TiC; (c) 316L-30TiC; (d) 316L-50TiC; (e) 316L-70TiC.](image-url)
From figures 3(b)–(d), it can be seen that the strongest peak is still (Fe, Ni) solid solution, and TiC phase is also present after adding TiC particles. And as the content of TiC increases, the intensity of diffraction peaks also increases. It can be seen from figure 3 that, except for the (Fe, Ni) solid solution phase, TiC enhanced phase and ferrite, there are no other obvious diffraction peaks in the XRD diffraction pattern, indicating that there are no other obvious new phases.

During the solidification process, TiC particles are dispersed into the strengthening layer, leading to the precipitation of the ferrite phase (α-Fe). One of the factors is that the coefficient of thermal expansion of TiC is lower than that of the 316 L matrix, which will introduce more tensile stress into the matrix, thereby promoting the formation of α-Fe. Another important factor is that the cooling rate of the composite molten pool will increase with the increase of TiC content. Studies have shown that during solidification, the degree of peak shift
is positively correlated with the cooling rate of the molten pool. When the TiC content increased from 10% to 30%, the peak value of TiC was found to shift in the 316L-10TiC and 316L-30TiC samples, as shown in figures 3(b) and (c). In order to further understand this phenomenon, EDS surface scanning was performed on

Figure 4. (Continued.)
the cross-sections of different strengthening layers, as shown in figure 4. This phenomenon can also be explained by figure 4, where the EDS results show the distribution of TiC in the matrix, and most of the TiC has diffused in the matrix. However, agglomeration of TiC can also be seen. Although it has been mixed by a ball mill for 30 min, agglomeration of a small amount of TiC particles inevitably occurs. At the same time, due to the relatively large mass of TiC, it is difficult for such agglomerated TiC particles to completely diffuse in the matrix under ultra-high cooling rates.

In addition, in the 316L-50TiC and 316L-70TiC samples, the diffraction peak intensity of the TiC phase is significantly higher than that of the 316L-10TiC and 316L-30TiC samples. It shows that the 316L-50TiC and 316L-70TiC samples contain a higher volume fraction of TiC phase. This is mainly because the TiC content in the powder becomes higher and the laser scanning speed is lower, which makes the temperature of the laser molten pool higher, the convection in the molten pool is stronger, and the TiC particles have a higher dilution rate in the molten pool.

In summary, the 316L-TiC strengthening layer is mainly composed of (Fe, Ni) phase, TiC phase and ferrite. The formation of these three phases depends on the composition of the strengthening layer and the laser process. After adding TiC particles to the mixed powder, a variety of pre-crystallization, eutectic or pseudo-eutectic phases can be produced during the cooling process of the molten pool. Studies have found that higher laser input energy makes the (Fe, Ni) phase in a liquid phase reaction state [29], and since the melting point of TiC is 3140°C, which is higher than the temperature of 316L, the TiC phase does not undergo melting and recrystallization. Therefore, during the cooling process of the laser molten pool, the (Fe, Ni) phase crystallizes preferentially, and the fine TiC particles are distributed at the grain boundaries, which will hinder the growth of the crystal grains, especially the growth of dendrites, and cause the alloy phase at the same time.

3.2. Microstructure analysis

Figure 5 shows the cross-sectional morphology of the 316L-10TiC strengthened layer. After adding 10wt% TiC, TiC particles were observed in the micrograph of the strengthening layer, and the structure morphology was clear. After adding a higher content of TiC, it is found that the metal structure is not as clear as the 316L-10TiC sample. Therefore, only 316-10TiC strengthened layer was selected for morphological analysis. In the SLM process, the grain size is affected by the temperature gradient and the solidification rate. The bottom structure is
planar or cellular, the middle structure is cellular or dendritic, and the top structure is equiaxed. Figure 5(a) shows the top structure, which can be found to be basically equiaxed grains. This is because the temperature gradient at the top is the smallest and the solidification rate is the largest, so the component undercooling is relatively large. It is greater than the supercooling required for crystal nucleation, so crystals will form equiaxed crystals on the top of the structure. Figures 5(b) and (c) show the middle part of the structure. It can be found that most of them are dendrites. This is because as the crystal expands towards the middle part of the strengthening layer, the temperature gradient at the solid-liquid interface decreases, the solidification rate increases, and the crystal grains The growth mode is transformed into cell crystals or dendrites. Figure 5(d) shows the bottom structure. It can be found that the boundary between the strengthening layer and the matrix is flat or cellular, with coarser crystal grains. This is because the ratio of the temperature gradient to the solidification rate is large, so the component undercooling is small. The crystal grows up sharply. The upper-middle structure of the strengthening layer is smaller than the bottom structure and arranged tightly, which can improve the performance of the organization to a certain extent. At the same time, new dendrites were found to grow in different directions at the interface, and some dendrites continued to grow along the direction of the previous dendrite.

Figure 6 shows the SEM images of the 316L, 316L-10TiC, 316L-30TiC, and 316L-50TiC strengthened layers after corrosion. The regular epitaxial structure can be clearly observed, which is due to rapid solidification and Marangoni effect [30]. Figure 6(b) shows a typical honeycomb structure, which has been reported by Shakerin [31] and Salman [32] et al. The honeycomb grain structure is believed to directly affect the strength and ductility [33, 34], and it can be shown that the 316L matrix has no significant difference in microscopic surface morphology and phase. No significant changes in the morphology of TiC particles were observed in the SEM images. Although studies have shown that Fe-based alloys can dissolve TiC [35], this only occurs when the molten Fe-based alloy and TiC are kept for a few minutes. Due to the rapid cooling, there is not enough time for dissolution during the SLM. This makes a lot of TiC particles uniformly distributed in the strengthening layer, which will form dispersion strengthening. The fine TiC particles are distributed at the grain boundaries, which will hinder the growth of crystal grains, especially the growth of dendrites, thereby refining the grains and forming fine grain strengthening.
In order to study the distribution of elements inside the grains of the strengthening layer and near the grain boundaries, EDS surface scanning was performed on the cross section of the strengthening layer, as shown in figures 4(a)–(e). It is observed that various component elements are distributed in the strengthening layer. It is
proved that in the SLM process, the various Ni, Cr compounds and other carbides generated are distributed in each crystal grain under stable diffusion conditions. There is obvious element aggregation, which has a greater effect on improving the hardness of the strengthened layer. Table 1 shows the specific element data of the EDS surface scan of the strengthening layer. It can be seen that during the composite additive manufacturing process, some elements on the surface of the strengthening layer have recrystallized. Among them, Fe element diffuses from the matrix to the strengthening layer, causing its content to increase rapidly. Comparing figures 4(a) and (b), the energy spectrum intensities of Fe, Ni and Cr increase with the addition of TiC particles, which means that more of them are precipitated near the grain boundaries. From figure 4(d) and table 1, it can be seen that the EDS peak of Ti element in the gray phase is the highest, and its corresponding atomic number fraction is 28.79%, while the peaks of Ni, Cr, and Fe elements are relatively low, and their corresponding atomic number fractions are respectively 2.91%, 6.49% and 18.40%. It can be seen from table 1 that the atomic number fraction of C element in the gray phase is 43.41%, while the Fe element content is very low. Therefore, it can be inferred that the gray phase is the TiC phase. From the element content in figure 4(b) and table 1, it can be seen that the content of Ti and C elements in the white surface is lower, while the content of Ni and Fe elements increases, indicating that the white surface is mainly [FeNi] alloy phase and contains part of Mo, Cr element, play the role of binding phase. In addition, there is still a higher content of Cr in the white appearance, which may be caused by the process of matrix melting and recrystallization.

Therefore, it can be found that as the content of nano-TiC increases, the strengthening layer structure appears obvious Ti element aggregation. When 30 wt% TiC is added, the aggregation phenomenon occurs; when 50wt% TiC particles are added, the Ti element aggregation phenomenon is more serious than that of the 30 wt% TiC sample. When 70wt% TiC is added, the Ti element content is reduced. This phenomenon can be explained by figure 7. Figure 7 is a schematic diagram of the formation mechanism of the microstructure of different strengthening layers. First, the honeycomb structure is a direct result of rapid solidification, and similar
images of honeycomb structures were reported in early research on composite materials [36]. Sub-micron honeycomb structures are also frequently observed in other SLM processes, such as FeCrCoMnNi [37], 304 [38] and nickel alloys [39]. Secondly, the accumulation of these elements at the grain boundaries will produce more pinning points, preventing the diffusion of the grain boundaries and effectively inhibiting the growth of grains. In addition, the SLM process is a process with a high cooling rate, which can reach 10^7 K/s. At such a high cooling rate, a very fine structure can be produced. At the same time, the high thermal conductivity of nano-TiC ceramics may lead to a higher temperature gradient at the solidification interface, which will also increase the cooling rate of the molten pool.

### 3.3. Hardness

Figure 8 shows the hardness distribution diagrams at the cross-sections of different strengthening layers. It can be seen from the figure that the cross-sectional hardness distribution of the strengthened layer has the same evolution trend, as the cross-sectional depth increases, the hardness gradually decreases, and there is an obvious sharp drop in the hardness in the heat-affected zone. According to the hardness data, the average hardness of the 316L reinforced layer without TiC particles is 273.6 HV. After adding 10 wt% TiC particles, the average hardness increases to 318.4 HV. After adding 30wt% TiC particles, the average hardness increased to 345.8 HV; after adding 50wt% TiC particles, the average hardness increased to 394.1 HV, of which the highest hardness value was 408.9 HV. After adding 70wt% TiC particles, the average hardness is reduced to 367.4 HV. The average hardness value of the 316L substrate is 208.8 HV. It can be seen from the above data that the hardness of the strengthened layer with 50wt% TiC increased the most, and the hardness value is about 1.9 times that of the base material. When 70wt% TiC is added, the hardness value is reduced. This is because the TiC content is high, and the laser beam energy cannot make the metal phase completely cover the nano TiC, and part of the TiC does not enter the strengthening layer, resulting in its microhardness lower than 316L-50TiC. It can be seen that the addition of nano-TiC can significantly increase the microhardness of 316L, which indicates that the aggregation and uniform distribution of nano-TiC particles significantly increase the hardness.

In the measurement, it is found that the hardness value of the top of the strengthening layer is significantly higher than that of the middle bottom layer. This is mainly due to the fact that the equiaxed crystals produced at the top during the recrystallization process are smaller in size than the dendrites of the middle layer and the plane crystals at the bottom. Orderly arrangement, resulting in higher hardness value. According to the longitudinal...
comparison, the hardness increases with the increase of TiC content, which can be attributed to the change of the microstructure caused by the addition of TiC particles. The contribution of TiC to hardness is mainly attributed to two points: (1) The addition of TiC phase increases the content of the dispersed high-hard ceramic phase in the strengthening layer [40]; (2) The added TiC phase becomes the nucleation point of the (Fe, Ni) alloy phase, which promotes the refinement of the alloy phase. Based on the Hall-Petch formula (1), (2) [41], it can be seen that the refinement of the alloy phase can further increase the hardness of the strengthened layer.

\[ \sigma_y = \sigma_0 + kd^{\frac{1}{2}} \]  
\[ H = 3\sigma_y \]  

In the formula: \( \sigma_y \) is the yield strength, \( \sigma_0 \) is the single crystal yield strength, \( d \) is the grain size, and \( k \) is a constant.

### 3.4. The influence of nano-TiC on the microstructure of the strengthening layer

The effect of nano-TiC on the 316L strengthening layer can be explained by the formation mechanism of the microstructure. As shown in figure 7, when the laser beam is irradiated on the powder, the powder melts rapidly. Due to the high energy input and light weight, 316L powder first melts and decomposes, and then migrates and diffuses in the pool under the combined action of surface capillary force and Marangoni. In the molten pool, Fe preferentially combines with Ni to form the (Fe, Ni) phase, because its chemical affinity for Ni is stronger than other elements in 316L. The (Fe, Ni) phase will first nucleate and grow during the solidification process. It can be seen from figures 7(b) and (c) that when 30 wt% and 50 wt% TiC are added, TiC particles aggregate, and 316L-30TiC has very obvious TiC aggregation. When 50 wt% TiC is added, TiC particles are basically uniformly distributed in the strengthening layer, which can be explained by the Ti element distribution diagram in figures 4(c) and (d). When 70 wt% TiC is added, due to the high TiC content, the molten metal phase cannot completely cover the nano-TiC, and partial melting occurs, resulting in a part of TiC not entering the strengthening layer, which reduces the performance of the strengthening layer.

### 4. Conclusion

(1) Using SLM/LMD composite additive manufacturing technology, 316L powder and nano TiC powder are used as raw materials to manufacture nano TiC reinforced 316L stainless steel-based strengthening layer, which shows a good metallurgical bond with the substrate.

(2) The phase composition of the strengthening layer is TiC, (Fe, Ni) and ferrite phases, and various elements are uniformly distributed on the surface of the strengthening layer, and no obvious aggregation occurs. Elements such as Ti, Fe and Ni are in the matrix 316L and strengthened. Obviously spread between layers of tissue. From top to bottom, the microstructure of the strengthening layer is: equiaxed crystal, dendritic crystal, and cellular crystal. There are differences in the structure of different areas of the strengthening layer. The dendrites along the surface to the interface of the strengthening layer gradually decrease, and the content of the hard phase nano-TiC particles decreases.

(3) Because the densely distributed nano-TiC particles in the structure can strengthen the 316L matrix, and the nano-TiC phase acts as a strengthening phase, the 316L-TiC strengthening layer can effectively improve the hardness of 316L stainless steel. When adding 10 wt% TiC, scattered TiC particles are found in the structure, with an average hardness of 318.4 HV; adding 30 wt% TiC, TiC particles appear agglomeration, with an average hardness of 345.8 HV; adding 50 wt% TiC, the TiC particles in the strengthening layer are basically evenly distributed, the average hardness reaches 394.1 HV, which is about 1.9 times that of the base material 316L, and the highest value reaches 408.9 HV. Adding 70 wt% TiC, because part of TiC does not enter the strengthening layer, the average hardness is reduced to 367.4 HV. Therefore, adding 50 wt% TiC can increase the hardness of the 316L-TiC strengthened layer the most.

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### Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).
Conflicts of interest

The authors declare that they have no conflicts of interest to report regarding the present study.

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