In order to improve the comprehensive performance of titanium alloy, the mechanical ball milling method was used to optimize the preparation of composite powder by different ball milling time, rotation speed and SiCp content. SiCp/TC11 composites were prepared by selective laser melting (SLM) technology. The results show that the milling speed, time and SiC content have an effect on the morphology, size and distribution of the composite powder. Compared with TC11, the hardness of the composite prepared by SLM process is 426.723 HV, which is increased by 15.10%, and the enhancement effect of SiCp is successfully realized. The research results provide a theoretical basis for obtaining high-quality aerospace titanium alloy parts.

Key words: TC11 composite material, SiCp, SLM, X-ray research, mechanical property

INTRODUCTION

Titanium matrix composites have gradually become the focus of research in recent years. Titanium matrix composites can combine the high strength and plasticity of matrix titanium alloy with the advantages of high modulus and high wear resistance of reinforcement, so as to make up for the shortcomings of titanium alloy, improve performance and broaden the application in aerospace field[1]. Selective laser melting technology is a rapid prototyping technology of metal powder. The technology is based on the forming concept of ‘discrete+accumulation’. The high-energy laser beam melts the automatically laid powder layer by layer according to the path specified by the layered software, and finally forms a nearly completely dense metal part. It has the advantages of fast forming speed, short production cycle, complex shape parts and low cost. In recent years, many scholars have done a lot of research on SLM manufacturing titanium matrix composites. Attar et al.[2] in-situ synthesized TiB reinforced titanium matrix composites by selective laser melting technology. After testing, it was found that the grain was significantly refined, and the microhardness, compressive strength and wear resistance were significantly improved. Tang et al.[3] added 3 wt.% TiC to Ti6Al4V and formed it by SLM. It was found that there were a large number of granular and strip eutectic TiC phases, with microhardness of 487 HV and ultimate tensile strength of 1565 Mpa. Pan et al.[4] prepared TiBw/Ti composites by SLM, and observed unique nanomesh TiB whiskers in the microstructure, and the strength of the composite reached 851 Mpa.

The preparation of composite powder is a key step in SLM forming composite materials, because the uniformity of composite powder mixing, particle size, distribution, adhesion, agglomeration and content have a great influence on the next forming process, and ultimately affect the mechanical properties of the material. High-energy ball milling is a common preparation method for composite powders. Ball milling can disperse the agglomeration of reinforcements, but it also reduces the sphericity of matrix powders and has a negative impact on the fluidity of composite powders. Therefore, the optimization of ball milling parameters is the premise of good formability of SLM. In this paper, nano-silicon carbide particles were used as reinforcements and TC11 titanium alloy was used as matrix. Composite powders were prepared by adjusting planetary ball milling process and different contents of reinforcements. Titanium matrix composites were prepared by selective laser melting process. Compared with TC11 sample, the mechanical properties are improved, which provides a theoretical and technical basis for obtaining titanium matrix composites with excellent comprehensive properties.

EXPERIMENTAL MATERIALS AND METHODS

The SiCp/TC11 nanocomposites prepared in this paper are composed of spherical titanium alloy powder with particle size of 15-53 μm and polygonal silicon carbide powder with average particle size of 500 nm. It can be seen from Figure 1 (a), (b) that TC11 powder generally presents a nearly regular spherical shape, smooth surface, no other impurities, and has good wet-
tability, SiCp powder presents irregular polygon, which is conducive to adhesion. Figure 1 (c),(d) shows that the main component of TC11 is Ti, the main components of SiC are Si and C.

The experimental scheme of this experiment is as follows: First, appropriate amount of TC11 and SiCp powder was weighed on an electronic balance and put into a ceramic pot to get mixed powder. Secondly, the agate ball is added into the ceramic pot, and different ball milling process parameters are used on the ball mill to obtain the mixed powder after ball grinding. Scanning electron microscopy (SEM) and X-ray diffraction (XRD) were used to observe the microstructure and phase analysis. Finally, appropriate printing parameters were selected to print 8 × 8 × 8 mm samples. The density and microhardness of the composites were measured.

RESULTS AND ANALYSIS

In order to explore the influence of ball milling parameters on the powder, 0.5 % SiCp was selected for mixed powder preparation. The powder and agate ball were weighed and placed in a ball mill tank according to the mass ratio of 1:4, and the ball milling speed and time were changed.

Figure 2 shows the micro-morphology of the powder at different rotation speeds and times. It can be seen from the figure that SiCp successfully adhered to the matrix after 8 hours of ball milling, and no SiCp aggregates were found in the titanium powder, indicating that the reinforcement was well dispersed in the titanium powder. However, compared with the original microscopic morphology of TC11, different ball milling speeds have a great influence on the morphology of the powder. As the speed increases from 100 r/min to 300 r/ min, the powder begins to undergo severe plastic deformation from the ‘spherical-cake-flaky’, the powder surface breaks, and many fine, broken and irregular particles are also visible. As the ball milling time gradually increased from 4 h to 8 h, the surface of the powder changed from smooth to rough. The analysis shows that with the increase of rotational speed, the ball mill changes from low energy state to high energy state. In unit time, the impact and contact frequency between powder and powder, powder and ball are obviously improved, which leads to the plastic deformation of titanium powder particles with larger particle size due to external forces such as extrusion, friction and collision. At the same time, some small, fragmented and irregular parts are dropped. With the increase of ball milling time, the number of collisions between powders increases, the hardness of SiC particles is larger, and it is an irregular triangle. During ball milling, SiC particles constantly collide with the surface of titanium powder, which makes the smooth surface rough, scratches and pits appear. When conditions permit, SiC particles are inserted into the surface of titanium powder and successfully attached. Figure 3 is the XRD diffraction pattern at different rotational speeds. Compared with the pattern without ball milling, there is no new diffraction peak, which also shows that there is no reaction between the powders during the ball milling process and no other substances are generated.

Figure 4 is the morphology of the mixed powder with different content. It can be observed from the diagram that with the continuous increase of SiCp content, the surface of the powder changes from flat to rough, and there are many pits and scratches. This is because during the ball milling process, with the increase of the content, the contact of titanium powder with SiC particles becomes more common, and the hard SiC particles continue to collide in one contact, rubbing the surface
of the titanium powder, making the surface rough. After increasing to 1%, the agglomeration of SiCp appears on the surface of the titanium powder.

Figure 5 is the XRD pattern of the four groups of schemes. It can be seen from the diagram that the different SiC content also has a certain influence on the refinement of titanium powder. With the increase of SiC content, the diffraction peak of titanium gradually widened. According to the Scherrer formula, the widened diffraction peak will reduce the grain size, that is, the refinement of particles. This is because the hard SiC constantly collides with the surface of the titanium powder, making the surface rough. In the process of ball milling, the friction between the titanium powder and the agate ball is increased, which is beneficial to the deformation and fragmentation of the powder. At the same time, the nano-scale SiC has a large specific surface area, has a large activity, and has a great polarization and attraction to the surrounding media. When the titanium powder has a crack due to external force, SiC can enter quickly, rub in the crack, accelerate the further expansion of the crack, and crush the titanium through continuous circulation, so as to achieve the purpose of refinement.

Figure 3 X-ray Diffraction(XRD) patterns of mixed powders with different milling speeds

Figure 4 SEM images of mixed powders with different SiCp contents

Analysis of mechanical properties

The ball milling parameters were selected as follows: speed 200 r/min, time 6 h, ball-to-powder ratio 4:1, SiCp content 0.5%. It can be seen from Figure 6 that the powder is spherical, SiCp is evenly distributed in the matrix, and the powder meets the SLM molding requirements.

The printing parameters of laser power of 170 W, scanning speed of 1 100 mm/s, spacing of 0.1 mm and forming layer thickness of 30 μm were selected. Strip printing strategy was adopted and the sample of $8 \times 8 \times 8$ mm was printed by rotating $67^\circ$ layer by layer. At the same time, TC11 powder printing sample was used as control test. The density of SiCp/TC11 and TC11 samples measured by drainage method reached more than
99.36%, which also showed that the forming quality of the samples under this printing parameter was good. The microhardness of the sample was measured by VH1102 micro Vickers hardness tester. The results are shown in Figure 7. Compared with the pure TC11 sample, the average hardness of the TC11 reinforced by SiCp increased by 15.10%. This is because after adding SiC to TC11, the interface reaction between SiC and TC11 occurs and a new phase is formed. The reaction equation is:

$$\text{SiC} + \text{Ti} \rightarrow \text{TiSiy} + \text{TiC}$$

The in-situ generated TiC and other hard intermetallic compounds provide more deformation resistance for the deformation of the material, which can effectively improve the bonding strength between the silicon carbide particles and the matrix, thereby improving the mechanical properties of the composite.

**CONCLUSIONS**

1. With the increase of ball milling speed and time, the powder will undergo plastic deformation and the surface becomes rough. No other substances were produced during the ball milling process. The addition of SiCp is beneficial to the refinement of the powder.

2. Under the ball milling parameters of 200 r/min, 6 h, ball-to-powder ratio of 4:1 and SiCp content of 0.5%, SiCp uniformly adhered to the TC11 matrix without agglomeration, and the composite powder had good sphericity.

3. Under the same SLM parameters, the SiCp/TC11 sample has a higher microhardness than the TC11 sample, which is increased by 15.8%. The reason for the increase is that TC11 and SiC in situ form hard metal compounds such as TiC.

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**REFERENCES**


**Note:** The responsible translator for English language is X.B.Lu, Ningbo, China