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Reviewed Paper

Tailoring Commercially Pure Titanium Using Mo_2C During Selective Laser Melting

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Abstract

Commercially pure Ti (CP Ti), Ti alloys and Ti composites have applications in a wide range of industries. With the merits of the emerging additive manufacturing technique, e.g. layer-wise fabrication and rapid solidification rate, the fabrication of Ti composites with tailorable microstructure and hence controllable properties is possible. In this work, a Ti composite was fabricated through selective laser melting (SLM) of CP Ti with Mo_2C, consisting of a matrix mainly from α-Ti and a small amount of β-Ti which could potentially embed very fine existing particles. The influence of SLM on the phase formation and microstructure of the fabricated Ti composite was investigated. The results showed that a tailorable microstructure of the Ti composite can be achieved via SLM. This work provides fundamental and important information on the fabrication of Ti composites with controllable microstructure through SLM of CP Ti with ceramic particle additions.

Introduction

Commercially pure titanium (CP Ti) is an important engineering material which has various applications in industry, especially in chemical, petrochemical processing and biomedical sectors, due to its excellent corrosion resistance, fabricability and durability in service [1]. However, the drawbacks of CP Ti such as low strength, low hardness and poor wear property severely impede its wider applications where mechanical properties are equally important as environmental resistance [1]. To accommodate the high demand for CP Ti, great efforts have been devoted to improving the mechanical properties while maintaining a superior environmental resistance through design and fabrication of titanium matrix composites (TMCs) or fabrication of CP Ti with small grain size [1]. However, the fabrication of TMCs through conventional approaches is time- and energy-consuming due to the high strength and hardness of TMCs. In the meantime, the increased level of mechanical properties which can be achieved through the fabrication of CP Ti with small grain size is limited [1].

Selective laser melting (SLM), as a laser-based powder bed additive manufacturing technique, enables the coupling of fabricating complex geometric metal components with local tailoring of microstructures in the fabricated parts. A variety of metallic materials including Al, Ti, Ni, steel and even amorphous and high entropy alloys with tailorable microstructures have been successfully fabricated using SLM recently [2-10]. Fine microstructures with improved

mechanical properties are obtained [11-13]. Aside from this, SLM features a high temperature rise depending on the materials being processed and parameters used during SLM. This provides a potential way of promoting melting of the secondary particles and their reaction with the metal matrix. As a result, the fabrication of metal matrix composite materials with addition of secondary ceramic or metal particles using SLM has also been reported [14-18]. Additionally, it is known that at least two melt flow patterns are triggered by the laser in the melt pool: damped capillary oscillations and thermocapillary flows [19]. This means that it is possible for the laser to alter the chemical homogeneity (distribution of the constituent elements) of the melt alloy due to the formation of liquid oscillations or capillary waves within the melt pool. These all together render SLM a promising technique to tailor metallic materials in a site-specific manner through the introduction of a small amount of secondary particles and by controlling the processing parameters in SLM.

In this study, 1 vol.% of Mo_2C particles were introduced into a CP Ti matrix by mechanical mixing and the mixed powder was processed using SLM. Different SLM processing parameters including laser power, scanning speed and scanning strategy were used to produce fully dense and crack-free TMCs with tailorable microstructures which were characterised using X-ray diffraction (XRD) and scanning electron microscopy (SEM). The results showed that tailorable microstructure of the TMCs can be achieved by controlling the SLM processing parameters. This study provides fundamental and important information not only on the fabrication of TMCs with controllable microstructure through SLM of CP Ti with ceramic particle additions but also on local tailoring of metallic materials by introduction of a small amount of secondary particles.

Experimental procedures

Commercially pure titanium powder (CP Ti, Grade 1, LPW, UK) with a powder particle size from 15 to 45 µm and molybdenum carbide powder (Mo_2C, Changsha Langfeng, China) with an average powder particle size ~3.5 µm were used. 1 vol.% Mo_2C powder was added to the CP Ti powder by mixing on a multidirectional mixer for 4 h to ensure a homogeneous distribution. The laser reflectivity of the CP Ti powder, Mo_2C powder and the mixed powder were measured using diffuse reflectance spectroscopy (DRS). DRS was carried out in the 250 to 2250 nm wavelength range using an UV-Visible-NIR Lambda 950 Perkin Elmer spectrometer equipped with a 150 mm diameter integrating sphere coated with Spectralon with 1 nm spectral resolution. A Spectralon reference was used to measure the 100% reflectance and internal attenuators were used to determine zero reflectance in order to remove background and noise. The powders were placed in a quartz cuvette, sealed, and mounted on a Teflon sample holder for the DRS measurement. Titanium matrix composite (TMC) specimens were produced on an in-house built selective laser melting machine equipped with a fibre laser, with a wavelength of 1.06 µm and maximum power of 300 W on the part bed. An inert, high purity (≥99.99%) argon gas atmosphere flow was used during the whole process to minimise oxidation. Various SLM processing parameters including laser power (50 and 250 W), scanning speed (225~325 mm/s at 50 W and 1400~1800 mm/s at 250 W) and scanning strategy (bidirectional and island scanning patterns) were used to fabricate fully dense and crack-free specimens. The powder layer thickness was fixed at 30 µm, the scan spacing at 60 µm, without pre-heating. The microstructure of the powder materials and SLMed TMCs was characterised using a FEI-Nova

NanoSEM 450 scanning electron microscope (SEM). The phase formation in the powder and SLMed TMCs were characterised using X-ray diffraction (XRD, Siemens D500 powder diffractometer, Cu Kα source, operated at 40 kV and 40 mA with a step size of 0.02° and scanning speed 2°/min. All XRD scans were done on the plane parallel to the SLM building direction). The relative density of the TMCs was measured using the Archimedes method.

Results and discussion

The morphology of the CP Ti, Mo_2C and mixed powder is shown in Figure 1. CP Ti shows a typical spherical morphology of gas-atomised powder while Mo_2C shows an irregular morphology. After 4 h mechanical mixing the majority of the Mo_2C powder was homogeneously distributed and mainly attached to the surface of the CP Ti powder particles, although some isolated Mo_2C powder particles can be seen. Because only 1 vol.% of Mo_2C powder was added to CP Ti, the mixed powder maintained a good flowability.

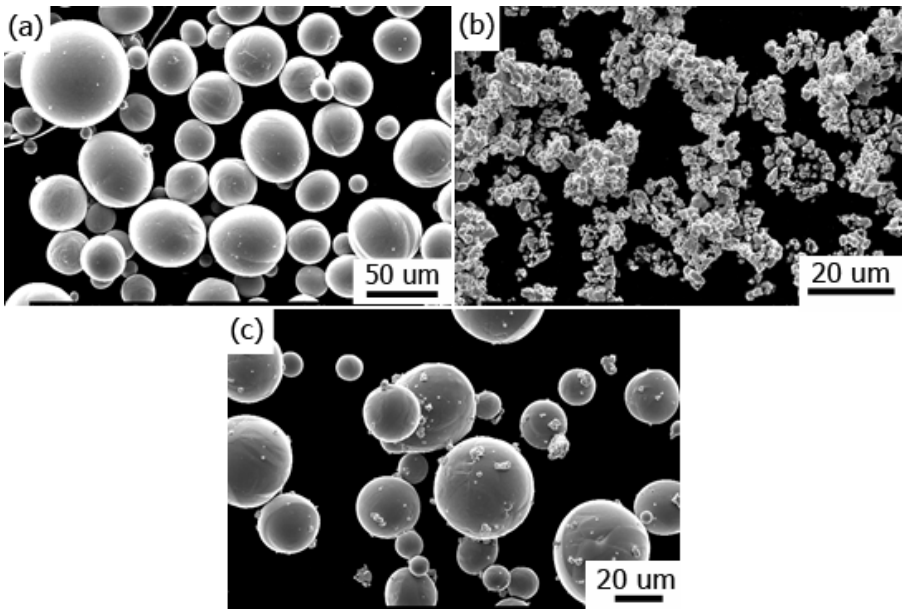

Figure 1 Morphology of the (a) CP Ti, (b) Mo_2C and (c) mixed powder.

Since the laser reflectivity of the powder particles has an important and direct influence on the laser energy being absorbed by the materials, which will further affect not only the temperature profile during the SLM process but also the resultant microstructures of the fabricated materials. The laser reflectivity of the CP Ti, Mo_2C and mixed powder is measured and given in Figure 2. The laser reflectivity of Mo_2C powder and CP Ti powder at 1.06 μm laser wavelength was measured to be ~22% and ~30%, respectively. As a result of the addition of Mo_2C powder with lower laser reflectivity, the laser reflectivity of the TMC powder was lower (~27%) than that of CP Ti powder. This indicates that even a small amount of addition of Mo_2C (1 vol.%) can result in an improved laser absorptivity.

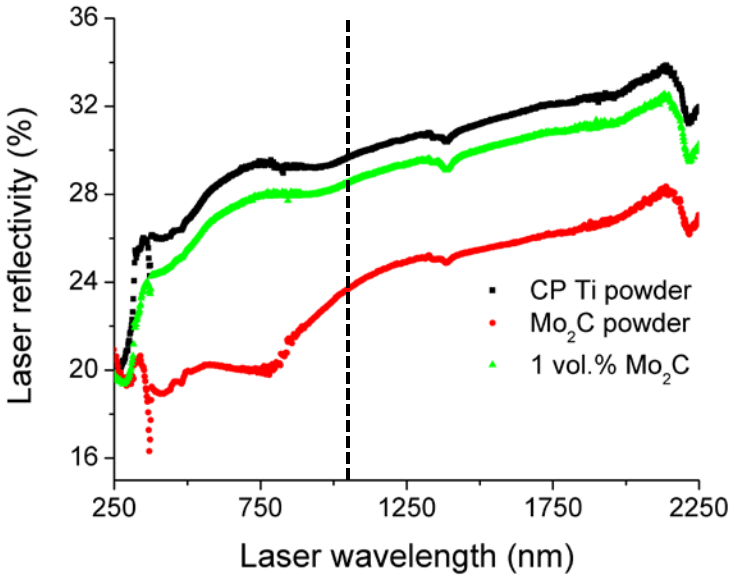

Figure 2 Laser reflectivity of the CP Ti, Mo2C and mixed powder. The black dotted line indicates 1.06 μm laser wavelength used in this study.

Based on our previous studies on SLM of CP Ti [20], different processing parameters were explored and fully dense TMCs (relative density ≥99%) were achieved at both low and high laser power, as shown in Figure 3a. However, when using a bidirectional scanning strategy, cracks were formed in the TMCs no matter which laser power and scanning speed were used. These cracks were also found to be aligned perpendicular to the building direction, as shown in Figure 3b. This is probably caused by the built-up residual stresses and weak bonding between the layers during SLM. By using an island scanning strategy, fully dense TMCs without cracks can be obtained. Therefore, the scanning strategy plays an important role in eliminating the cracks in the SLMed TMCs.

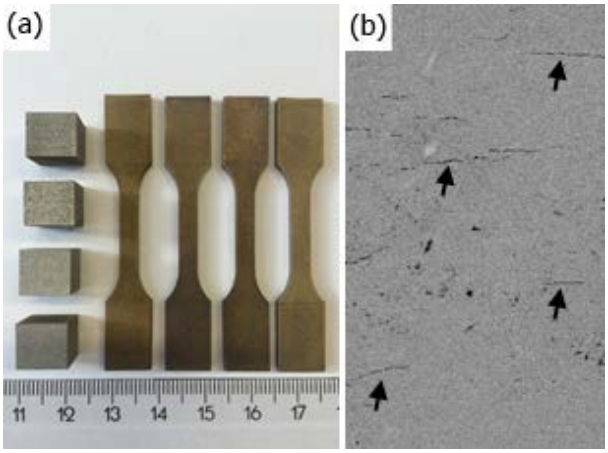

Figure 3 (a) SLMed TMC cubes and tensile bars; (b) typical microstructure of the SLMed TMCs showing cracks (black arrows) perpendicular to the building direction.

The phase formation in the CP Ti and Mo_2C powder as well as the SLMed TMCs is shown in Figure 4. Both CP Ti and Mo_2C powder have a hexagonal close packed (*hcp*) crystal structure. Only α-Ti was detected in the CP Ti powder while no β-Ti (body centred cubic, *bcc* Ti) was observed. In all fully dense and crack-free SLMed TMCs, along with the peaks from α-Ti a tiny peak corresponding to β-Ti appeared, signifying the formation of a small amount of β-Ti in the TMCs. In addition, the relative peak intensity in the SLMed TMCs and the CP Ti remains identical. This implies that no strong crystallographic texture was formed after SLM.

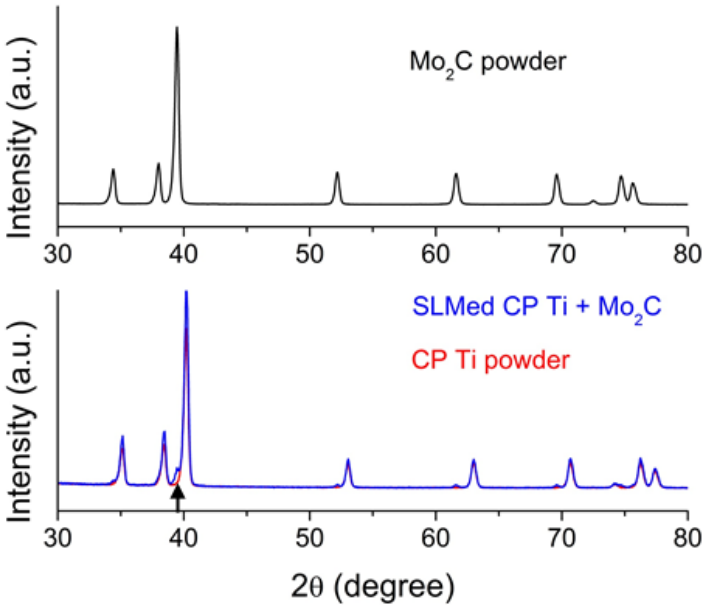

Figure 4 XRD patterns of the CP Ti, Mo_2C powder and the SLMed TMCs. The black arrow indicates the β-Ti peak.

The microstructure of the SLMed TMCs fabricated using two different sets of processing parameters, i.e., one with high and one with low laser power, is shown in the back-scattered SEM images in Figures 5a and 5b. Both high laser and low laser power fabricated TMCs exhibit a heterogeneous microstructure in which three distinct regions can be observed. According to our recent study (not shown here), the bright and grey regions contain a higher and lower content of Mo respectively, while no Mo was found in the dark region. The regions with Mo are confirmed to be β-Ti while the region without Mo is α-Ti. This implies that the Mo_2C decomposed during SLM, releasing Mo and C into the laser melt zones. Upon solidification, Mo as a well-known β-Ti stabiliser dissolved into the Ti matrix and formed β-Ti. C on the other hand could be dissolved in Ti matrix or potentially form TiC particles and more future work will be carried out to uncover this. Meanwhile, some pure Mo and Mo_2C powder particles were also observed embedded in the TMC matrix, especially in the TMCs fabricated at low laser power. This indicates that the temperature rise in some local area is not high enough to trigger the reaction between the Mo_2C and CP Ti. In addition, the α-Ti and β-Ti show a different distribution in high and low laser power fabricated TMCs. In the high laser power fabricated TMCs, β-Ti distributes mainly at the edge around the melt pool, while α-Ti is located inside the melt pool, forming a nest-like microstructure. In the low laser power fabricated TMCs, both α-Ti and β-Ti distribute randomly and the α-Ti shows a highly localised distribution in some areas. Higher magnification

microstructures, shown in Figures 5c and 5d, reveal that the size of the α-Ti differs apparently in the TMCs fabricated using high laser power and low laser power. More α-Ti with much smaller size can be seen in the TMCs fabricated using low laser power compared to high laser power. This is probably due to the peak temperature difference in the melt pool and the resultant different melt pool behaviour as well as the different local thermal profiles in the TMCs fabricated using high and low laser power.

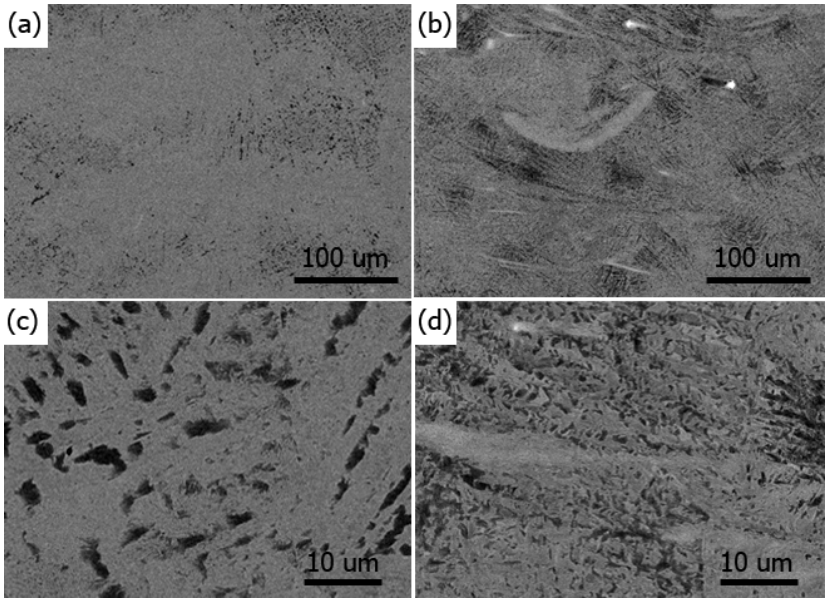

Figure 5 Microstructures of the SLMed TMCs fabricated at (a) high laser power and (b) low laser power. (c) and (d) are high magnifications of (a) and (b), respectively.

Conclusion

Different SLM processing parameters were used to fabricate fully dense crack-free TMCs from 1 vol.% Mo_2C doped CP Ti. The microstructure of the TMCs can be tailored using different processing parameters. Both high and low laser power can result in a heterogeneous microstructure consisting of three distinct regions with different content of Mo. The Mo enriched region was confirmed to be β-Ti while the region without Mo is α-Ti. The underlying mechanism is the reaction between Mo_2C and CP Ti during SLM, which is promoted by the higher temperature rise in Mo_2C. The distribution of α-Ti and β-Ti and the grain size of α-Ti were also different in the TMCs fabricated using high and low laser power. This is probably due to a peak temperature difference and concomitant different melt pool behaviour as well as the different local thermal profiles. This study provides fundamental and important information not only on the fabrication of TMCs with controllable microstructure through SLM of CP Ti with ceramic particle additions but also on local tailoring of metallic materials by introduction of a small amount of secondary particles.

Acknowledgements

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