



In situ synthesized TiC/Mo-based composites via laser powder bed fusion

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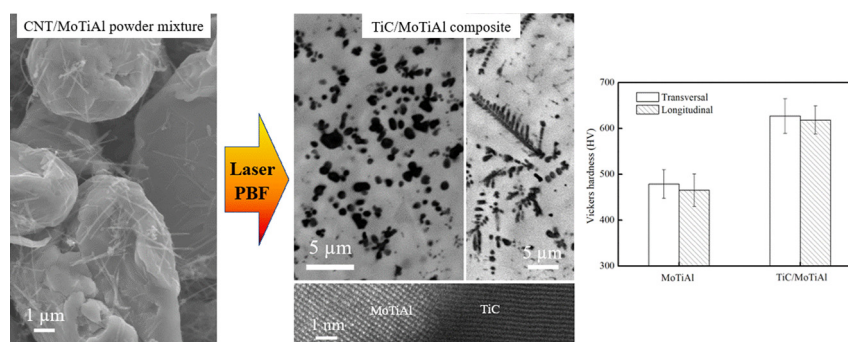
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HIGHLIGHTS

- A novel strategy utilizing carbon nanotubes was put forward to synthesize in situ TiC/Mo-based composites via laser powder bed fusion.
- Carbon nanotubes were well mixed with MoTiAl powders under electrostatic attraction during heteroagglomeration process.
- In situ TiC, possessing two unique structures, namely spherical and dendritic TiC, was uniformly dispersed in the matrix.
- The MoTiAl matrix underwent a morphological change from nearly columnar to fine equiaxed grains after incorporating TiC.

GRAPHICAL ABSTRACT



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ABSTRACT

A strategy utilizing carbon nanotubes (CNTs) was put forward to synthesize in situ TiC/Mo-based composites via laser powder bed fusion (laser PBF). The functionalized CNTs were dispersed with MoTiAl powders under electrostatic attraction by heteroagglomeration. During laser PBF, individual CNTs reacted with Ti elements and were completely transformed into monocrystalline TiC. Those TiC reinforcements were homogeneously dispersed and intimately contacted the matrix, giving rise to the morphological evolution of a MoTiAl matrix from the nearly columnar to fine equiaxed grains as well as improved mechanical performance. Our finding offers significant guidance for designing and producing advanced Mo-based composites in the application of heat-resistant materials.

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1. Introduction

The increasing demand for new ultra-high-temperature materials beyond the realm of Ni-based superalloys has generated significant interest in refractory intermetallics [1,2]. Owing to the high melting point and superior stiffness, Mo alloys possess promising potential in

the fields of aeronautics and astronautics [3]. Unfortunately, the application reliability of Mo alloys is seriously hindered by their insufficient elevated-temperature strength [2]. In this regard, stiff and fine ceramic particles were utilized to strengthen Mo alloys by impeding the dislocation movement [4,5]. Among various ceramic reinforcements, TiC is particularly attractive for its excellent properties, such as low density ($\sim 4.93 \text{ g/cm}^3$), high melting point ($\sim 3430 \text{ K}$), high Young's modulus ($\sim 440 \text{ GPa}$), and good thermal stability [6,7]. Yoshimi et al. [5,8] reported that incorporating TiC into a ternary MoSiB alloy resulted in higher mechanical strength and creep resistance at elevated

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Table 1
Characteristics of acid-treated CNTs and MoTiAl and 1.2 wt% CNT/MoTiAl powders.

Powders	Composition	Particle size	Zeta potential	Laser absorptivity ($\lambda = 1070$ nm)
CNTs	Fe < 0.3 wt%, C: Bal.	Diameter: 20–110 nm, length: 2–15 μ m	–51.3 mV	89.8%
MoTiAl	Ti ~ 33 wt%, Al ~ 13 wt%, Mo: Bal.	D ₁₀ ~ 5.1 μ m, D ₅₀ ~ 12.8 μ m, D ₉₀ ~ 24.9 μ m	Positive, weak	71.6%
1.2 wt% CNT/MoTiAl	–	D ₁₀ ~ 5.3 μ m, D ₅₀ ~ 13.0 μ m, D ₉₀ ~ 24.2 μ m	–	77.9%

temperatures via traditional casting. However, such traditional manufacturing techniques usually have some drawbacks, e.g., the insufficient densification rate and the aggregation and irregular microstructure of reinforcements, as well as the occurrence of interfacial cracking, being harmful for the mechanical performance of composites [8–10]. Furthermore, the traditional approaches involve highly time-, energy-, and material-consuming processing steps to achieve the final products [11]. Particularly, Mo alloys with poor ambient-temperature ductility are challenging to machine for complex structures.

Laser powder bed fusing (Laser PBF) is newly capable of fabricating three-dimensional products with almost uninhibited freedom of design [12]. The product is directly built using a high-energy laser beam to fuse and consolidate the loose powders selectively according to the corresponding computer-aided design models in a layer-by-layer manner [12,13]. Due to its flexibility in materials and processing, laser PBF offers new technological opportunities for producing high-performance metal matrix composites (MMCs) with tailored structures. In recent years, the laser-PBF processing of MMCs, including Al- [14–19], Ti- [20–24], Ni- [10,25–27], and Fe-based alloys [28–30], has been documented. In most cases, the reinforcements were directly added to the metallic powders by mechanical milling/blending, followed by laser PBF to create an ex situ composite. However, the poor wettability and interfacial strength between the reinforcements and metal usually cause microcracks and even premature failure of the composites [9]. In contrast, the in situ reinforcements synthesized by chemical reactions are extremely effective for composite strengthening, since they are fine and thermodynamically stable, having clean and compatible interfaces with the matrix [7,9]. AlMangour et al. [7] prepared in situ TiC/316L stainless steel composites with enhanced mechanical properties through the laser-PBF processing of 316L-Ti-graphite powder mixtures. Li et al. [22] found that the in situ TiB₂ reinforcement dramatically enhanced the nanohardness of TiB₂/TiAl composites via laser PBF. Unfortunately, to the best of our knowledge, the fabrication of in situ Mo-based composites by laser PBF has never been reported.

CNTs are considered to be ideal reinforcements for MMCs due to their low density, large aspect ratios, and excellent mechanical properties [31,32]. Recently, Chen et al. [33] synthesized high-strength Al₄C₃/Al composites via the in situ reaction of CNTs with Al during spark plasma sintering. Herein, an idea is put forward to develop high-performance Mo-based composites by transforming CNTs into TiC reinforcements during laser PBF, based on the following considerations: (i) after a surface modification, individual CNTs could wrap on the surface of metallic powders easily under electrostatic attraction without severe fabrication processing [34]; (ii) CNTs still possess excellent load-bearing capability in MMCs, even though the reaction is incomplete, e.g., Wang et al. [27] reported that incorporation of CNTs enhanced the mechanical properties of PBF-produced Inconel 625 parts; and (iii) compared with using large graphite sheets [7,35], using CNTs may allow the formation of fine TiC structures for enhanced strengthening. In this work, a TiC reinforcement was synthesized in situ via laser PBF; its microstructural characteristics and effect on the morphology and mechanical properties of Mo alloy were investigated.

2. Experimental

2.1. Raw materials

A simple Mo-33wt%Ti-13wt%Al solid solution was chosen as one example of Mo alloys in this work [36]. The starting MoTiAl powders having an irregular shape (Fig. S1 of Supporting information), were fabricated via a combination of arc melting, mechanical milling and sieving, and their size distribution of D₁₀, D₅₀ and D₉₀ were 5.1 μ m, 12.8 μ m and 24.9 μ m, respectively (Table 1). Pristine CNTs having a diameter of 20–110 nm and a length of 2–15 μ m were provided by Hodogaya Chemical Co., Ltd., Japan. The high-purity ethanol, sulfuric acid (H₂SO₄, 97 wt%) and nitric acid (HNO₃, 61 wt%) were obtained from Wako Industries, Japan.

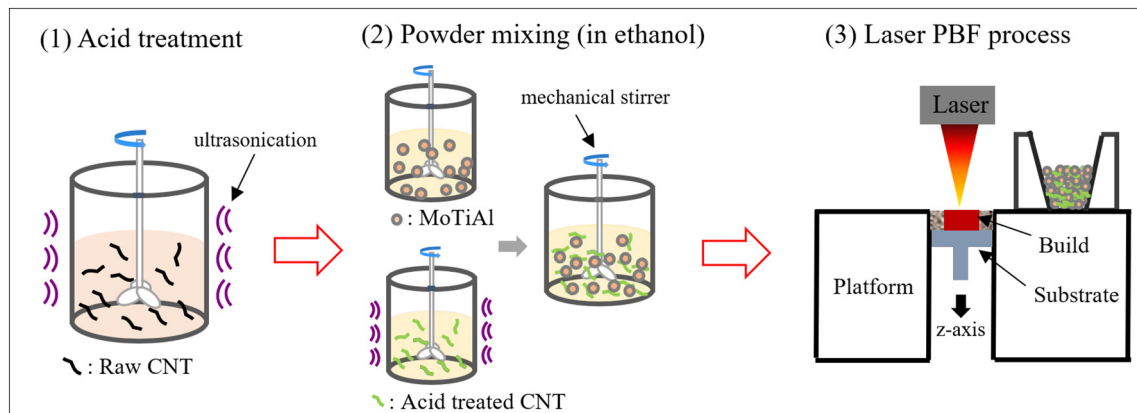


Fig. 1. Schematic illustration of the fabrication processes for Mo-based composites.

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