



Microstructure evolution of laser remelted Al_2O_3 –13 wt.% TiO_2 coatings



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ABSTRACT

Laser remelting of plasma sprayed Al_2O_3 – TiO_2 coatings has been extensively investigated. However, little work on the microstructural evolution of laser-remelted Al_2O_3 – TiO_2 coatings has been reported. This study presents a detailed microstructural investigation of laser-remelted Al_2O_3 – TiO_2 coatings. Nanostructured feedstock has been reconstituted from nano-sized starting powders. A compound process of plasma spraying and laser remelting has been applied to fabricate the Al_2O_3 – TiO_2 coatings on titanium alloy. The evolution of microstructures of laser-remelted Al_2O_3 – TiO_2 coatings was investigated by X-ray diffractometer (XRD), scanning electron microscope (SEM), energy-dispersive spectrometer (EDS) and transmission electron microscope (TEM). Microstructural observations revealed microstructure correlation throughout the compound process. It was found the α - Al_2O_3 in feedstock was transformed to γ - Al_2O_3 after plasma spraying, which was completely transformed into stable α - Al_2O_3 phase after laser remelting. Bi-modal microstructure features are found for the laser-remelted coatings. For nanostructured coating, the net structure of its remelted coating resembles that of its feedstock and as-sprayed coating. For conventional coating, the strip-like structure of its remelted coating also resembles that of its feedstock and as-sprayed coating.

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1. Introduction

Titanium has many desirable characteristics in terms of high temperature properties, excellent strength to weight ratio and good corrosion resistance. It is therefore attractive for various structural applications in aerospace, aviation and marine engineering [1]. However, titanium is susceptible to wear and abrasion because of its low hardness [2,3]. Surface properties are critical for the long-term stable services of titanium components. Therefore, for the improvement of its low hardness, a variety of surface treatments and coatings have been developed for titanium and its alloys, such as carburizing [4], nitriding [5], physical vapor deposition [6] and spraying [7]. Among various methods used for this purpose, plasma spraying has been a versatile and widely used method [8]. There is a vast range of materials that can be sprayed onto titanium substrates to reduce wear.

Ceramic coatings, such as WC, ZrO_2 , Al_2O_3 and TiO_2 , possess excellent wear resistance and corrosion resistance, which are suitable for serving as the protective coatings for titanium substrates [9,10]. Plasma sprayed Al_2O_3 – TiO_2 coatings, in particular, the nanostructured Al_2O_3 – TiO_2 coatings, have been extensively applied as

surface coatings for metal substrates in many fields owing to their superior properties [11,12]. Plasma spraying is a process in which feedstock materials are thermally melted into liquid droplets and coated to the substrate surface at a high speed. The individual particles stick and condense at the surface and the coating is formed by a continuous build-up of successive layers of melted liquid droplets and hard particles. Owing to its particular forming process, plasma sprayed coatings generally have the characteristics of high porosity and relatively low bonding strength with the substrate, which degrades the lifetime of the coatings [13]. Post surface treatment by laser is effective to reduce the voids in as-sprayed coatings. Rapid laser treatment with high energy density produces pore-free, homogeneous and fully dense hard top-coat [14]. Furthermore, it generally remelts the whole thickness of the as-sprayed coatings and thus produces a strong metallurgical bonding to the substrate, which is beneficial to increase the service life of the coatings.

Plasma spraying and laser remelting is an effective way to fabricate high-performance hard ceramic coatings. During the compound process, different microstructures of the coatings could be achieved at different stages of the process. Thus, the in-depth understanding of the microstructure evolution is vital for the formation of the final structure and the properties of the coatings. Plasma spraying of nanostructured Al_2O_3 – TiO_2 coatings has been

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extensively investigated in the last decade [11,12]. Laser remelting of the as-sprayed nanostructured Al_2O_3 - TiO_2 coatings has also received considerable attention in the past several years [15–17]. However, little work on the microstructural evolution of laser-remelted Al_2O_3 - TiO_2 coatings has been reported. In the present study, a detailed microstructural investigation of laser-remelted Al_2O_3 - TiO_2 coatings is presented, focusing on the effects of feedstock preparation, plasma spraying and laser remelting process on the microstructure of the laser-remelted coatings. The microstructure evolution study throughout the process is beneficial for the fabrication of the protective ceramic coatings with homogeneous microstructure for various applications.

2. Experimental procedures

2.1. Substrate and feedstock powders

A Ti-6Al-4V titanium alloy is used as the substrate for the present study. The chemical composition (wt.%) of the as-received substrate is listed as follows: 6.0 Al, 4.3 V, 0.3 Fe, 0.1 Si, 0.1 C, 0.15 O, and the balance is Ti. The feedstock used for plasma spraying is nanostructured reconstituted powders derived from nanoparticles. The raw nanoparticles consisted of Al_2O_3 (δ and γ phases, 99.9% purity, Degussa Co., Ltd., Germany) with particle size of 20–45 nm, TiO_2 (anatase, 99.9% purity, Nanjing High Technology Nano Materials Co., Ltd., China) with particle size of 20–50 nm, ZrO_2 (tetragonal phase, 99.9% purity, Cug-Nano Materials Manufacturing Co., Ltd., China) with particle size of 20–50 nm and CeO_2 (cubic phase, 99.9% purity, Rare Chem. Co., Ltd., China) with particle size of 20–40 nm. Al_2O_3 and TiO_2 were the main compositions with a mass ratio of 87:13. ZrO_2 and CeO_2 were used as additives and their weight content was about 5%, respectively. All the nanoparticles were mixed by wet ball milling. An emulsion of polyvinyl alcohol (PVA) was added into the slurry as a binder addition. The reconstitution process of the feedstock also included spray drying, agglomerates sintering and plasma treatment. During the plasma treatment, the feedstock powders were treated in a plasma flame to improve their density and sphericity. Meanwhile, a commercially available micron-sized Metco 130 powder (Sulzer Metco Co., Ltd., USA), with a chemical composition of 87 wt.% Al_2O_3 and 13 wt.% TiO_2 , was also used for plasma spraying and laser remelting to fabricate a conventional Al_2O_3 -13 wt.% TiO_2 coating for comparisons. The scanning electron micrographs of the as-prepared nanostructured Al_2O_3 -13 wt.% TiO_2 feedstock and commercial Metco 130 powders are shown in Fig. 1. As indicated in Fig. 1a, the as-prepared nanostructured feedstock presents a regular spherical morphology with smooth surface. Fig. 1b shows an irregularly angular morphology of the Metco 130 powders with a particle size of 20–80 μm .

2.2. Plasma spraying and post laser remelting

Atmospheric plasma spraying was used to deposit the as-sprayed coatings on ultrasonic-cleaned and freshly grit-blasted Ti-6Al-4V titanium alloy substrates. The as-sprayed coatings were deposited by a Metco 9 M plasma spraying system (Sulzer Metco, USA). A mixture of Ar and H_2 was used as the plasma gas and Ar was used as the powder carrier gas. The parameters of plasma spraying were as follows: (a) voltage 65 V, (b) current 600 A, (c) primary Ar gas pressure 690 kPa, (d) secondary H_2 gas pressure 380 kPa, (e) Ar gas flow rate 2000 l/h, (f) powder feed rate 1000–1500 g/h, and (g) spray distance 100 mm. The thickness of the as-sprayed coatings was in the range of 200–300 μm .

The as-sprayed coatings were subsequently remelted using a continuous wave CO_2 laser (DL-HL-T5000, China) operating at a wavelength of 10.6 μm with a maximum output power of 5 kW. During laser remelting, a high-energy laser beam is focused onto the as-sprayed coatings to form a molten pool. The laser beam was

defocused to a spot of 3.5 mm in diameter at the surface of the coatings. The laser output power used in this study ranged from 600 to 1200 W. The laser scanning velocity was in the range of 600–1400 mm/min. A CNC system is used to control the movement of the samples on a workbench to ensure high process accuracy for the laser-remelted Al_2O_3 -13 wt.% TiO_2 coatings (hereon referred to as LRmC). The LRmC for the conventional coating system is referred to as C-LRmC, and that for the nanostructured coating system is referred to as N-LRmC.

2.3. Characterization

The phase constituents and microstructure of the feedstock and coatings before and after laser remelting were examined by an X-ray diffractometer (XRD, D/max- γB , Japan, with $\text{Cu K}\alpha$ radiation ($\lambda = 0.15418$ nm)), a scanning electron microscope (SEM, Quanta 200, FEI, USA) equipped with X-ray energy-dispersive spectroscopy (EDS) and a transmission electron microscope (TEM, Philips-CM12, Holland). For the preparation of TEM test samples, the coatings were cut off from the substrate and polished to a thickness around 50 μm by abrasive paper, followed by ion-beam milling.

3. Results and discussion

3.1. Phase constituents

In the present study, the conventional micron-sized coatings were derived from commercially available Metco 130 powders, while the nanostructured coatings were derived from the reconstituted powders consisting of nanoscale Al_2O_3 , TiO_2 powders and modified additives of ZrO_2 and CeO_2 . As-sprayed coatings and LRmC were obtained by plasma spraying and laser remelting, respectively. For the nanostructured coatings, the reconstitution process, plasma spraying and the final laser remelting have comprehensive effects on the microstructure and properties of the coatings. During the compound process, the microstructure of the feedstock and coatings before and after laser remelting shows certain similarities and apparent evolution regularity, in spite of their own characteristics at different stages.

The phase constituents of the feedstock, as-sprayed coatings and laser-remelted coatings were examined by XRD analysis. The XRD results indicate that the phase composition of the feedstock and coatings varies at different stages of the compound process. Table 1 shows the phase constituents of feedstock, as-sprayed coatings and LRmC. As presented in the table, for the conventional coating system, part of α - Al_2O_3 of the Metco 130 feedstock was transformed to γ - Al_2O_3 after plasma spraying. After laser remelting, the metastable γ - Al_2O_3 phase was completely transformed into stable α - Al_2O_3 phase. For the nanostructured coating system, the similar transformation was found. In addition, Al_2TiO_5 and Ti_3Al phase were also found in LRmC.

During plasma spraying process, the transformation of α - Al_2O_3 to γ - Al_2O_3 phase is mainly ascribed to that the latter has a lower free energy of nucleation [18]. When the feedstock is melted by high-temperature plasma flame, it nucleates and grows to γ - Al_2O_3 phase under a rapid cooling rate. However, the γ - Al_2O_3 phase

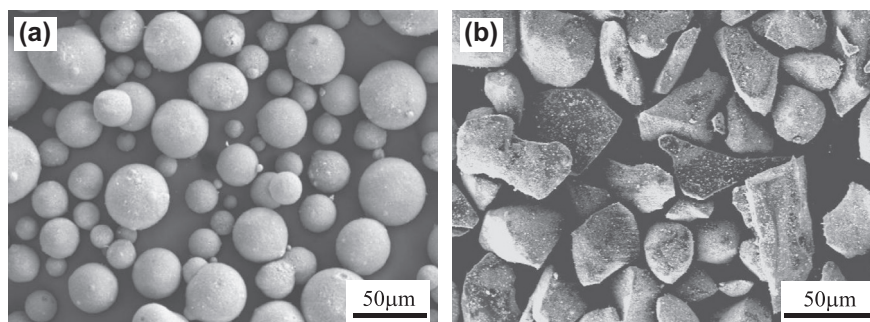


Fig. 1. SEM micrographs of Al_2O_3 -13 wt.% TiO_2 feedstock powders. (a) As-prepared nanostructured reconstituted powders, and (b) as-received Metco 130 powders.

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