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### Surface performance and physical properties of nanoscale ceramics reinforced laser alloying composite coatings

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# 1. Introduction

LA ceramics reinforced metal matrix composites (MMCs) is an available surface modification technique, which was able to improve the wear resistance of titanium alloys. As the most promising method, nano-particles are expected to play an important role in strengthening the light metal surface [1,2]. Moreover, there has been a growing interest in the laser alloying of glassy alloys onto light alloys recently, with the aim of improving the wear resistance of titanium alloys [3]. The results so far show that when the substrate is coated with a layer of amorphous material, it becomes more resistance to some lose efficacy. Lasers have provided the important opportunities in realization of nano/amorphous-manufacturing. The laser surface engineering (LSE) have been widely used for improving the mechanical, chemical or tribological properties of the metal parts [4]. LA is belong to LSE, which is a promising approach to produce the amorphous-nanocrystalline coatings on metals.

Nickel-base self-fluxing alloys are mainly used in the chemical industry, glass mold industry based, etc., when wear resistance is required [5]. Moreover, the wear resistance of the modified surface was improved by virtue of the hardness of TiN and Ti(CN) present [6,7]. LA of the Ni60–Si<sub>3</sub>N<sub>4</sub> pre-placed powders on a TA7 titanium alloy substrate can form NC, such as TiN and Ti(CN) reinforced composite coating, which improved the wear resistance of substrate surface. It was confirmed that during LA process, Si<sub>3</sub>N<sub>4</sub>

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#### ABSTRACT

 $Si_3N_4$ -Ni60 is firstly used to improve the wear resistance of a TA7 (Ti-5Al-2.5Sn) titanium alloy surface by mean of a laser alloying (LA) technique. The synthesis of the hard composite coating on a TA7 titanium alloy by LA of Ni60-Si\_3N\_4 pre-placed powders was investigated by means of scanning electron microscope (SEM), energy dispersive spectrometer (EDS) and high resolution transmission electron microscope (HRTEM). Experimental results indicated that a number of the interdendritic lamellar eutectics, nanoscale ceramics (NC) and the amorphous phases were produced in such LA coating. The production of NC led the free energy to enhance, which increased the density of point defect, leading to the formation of hypersaturated state of point defect, causing lattice distortions. Compared with a TA7 alloy substrate, an improvement of the wear resistance was obtained for such LA composite coating.

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in the coating can be dissolved due to a high temperature generated by an impingement of the laser beam, resulting in delivering Si and N into the molten pool. Si and N were able to react with other elements in the molten pool, leading to the formations of the hard compounds and NC, improving the wear resistance of the composite coating. This paper will discuss the microstructures, physical/wear properties of the Si<sub>3</sub>N<sub>4</sub>–Ni60 LA composite coating on a TA7 titanium alloy, which provides the theoretical and experimental to promote the application of LA technique in surface modification of titanium alloys.

#### 2. Experimental

The materials used in this experiment: TA7 titanium alloy samples (10 mm  $\times$  10 mm  $\times$  35 mm or 10 mm  $\times$  10 mm  $\times$  9 mm) and the thickness of the pre-placed layer was 0.6 mm, which were polished with SiC grit paper prior to the coating operation, and chemical compositions of the TA7 alloy in this study: 5.00Al, 2.50Sn, 0.5Fe, 0.08C, 0.05N, 0.015H, 0.2O and balance Ti; alloy powders of Ni60 ( $\geq$ 99.5% purity, 100–200  $\mu$ m), and Si<sub>3</sub>N<sub>4</sub>  $(\geq 99.5\% \text{ purity}, 50-200 \,\mu\text{m})$  were used for LA, the chemical compositions of Ni60 (wt.%): 0.8C, 15Cr, 3B, 4Si, 10Fe and balance Ni. Compositions (wt.%) of the pre-placed powders: 85Ni60-15Si<sub>3</sub>N<sub>4</sub>. And the water glass was used to be the binder. A 5.0 kW continuous wave CO<sub>2</sub> laser (TJ-HL-T5000), spot size of 4 mm, was employed to melt the surface of sample. Three-track lap coating was formed on substrate, and the lap rate was approximately 25% in order to uniformly cover a large area. During LA process, the surface oxidation was prevented by inert gas (Ar) with a flow







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rate of 35 L/min, the parameters of LA process: laser power of 0.9 kW, scanning speed of 3–9 mm/s.

The wear volume losses were measured after 40 min. Wear resistance of LA composite coating was tested by a WMM-W1 disc wear tester. Rotational speed of the wear tester was 465 r/min. The linear velocity of the friction surface was 0.88 m/s. Microstructural morphology of such composite coating was analyzed by means of a Titan 80–300 high resolution transmission electron microscope (HRTEM) and a LEO 1525 scanning electron microscope (SEM), and the spot distributions were measured using an energy dispersive spectrometer (EDS). A HV-1000 microsclerometer was used to test the micro-hardness distribution of the composite coating.

#### 3. Experimental results and analysis

#### 3.1. SEM, TEM, HRTEM and EDS analysis

As shown in Fig. 1a, a metallurgical combination was obtained between a Si<sub>3</sub>N<sub>4</sub>-Ni60 LA composite coating and the TA7 substrate. It was also noted that a fine microstructure of the coating was achieved, and the block/stick shape precipitates were dispersed uniformly in the coating matrix. In fact, due to a dilution effect, lot of the elements, such as Ti, Al, Mo and Zr entered into the molten pool from the substrate. Mo is an effective strengthening element for Zr. In Zr-rich compositions the  $\alpha$  (hexagonal closepacked, hcp) + Mo<sub>2</sub>Zr phase region exists at lower temperatures while the  $\beta$  (body-centered cubic, bcc) and  $\beta$ +Mo<sub>2</sub>Zr are stable at high temperatures [8]. As shown in Fig. 1b, the middle-coating had a uniform and dense microstructures consisting of the primary dendrites and minor amount of interdendritic eutectic-like structure. Owing to a rapid cooling rate of molten pool, a small amount of elements, such as Si, B and Mo had no time to be precipitated from the liquid and solution in  $\gamma$ -Ni to form super-solution, which caused a solution strengthening. Moreover, the dendritics were also produced in the middle-coating. It was considered that during LA process, the different locations of the molten pool absorbed the different energy from laser beam, i.e. the middle-coating absorbed more energy than that of the bottom-coating. Thus, the ceramics, such as TiN and Ti(CN) in the middle-coating had enough time to grow up, forming the dendritics.

As shown in Fig. 1c, the fishbones/block shape precipitates were produced in middle-coating, which retarded the growth of themselves, favoring the formation of a fine microstructure. It is known that lot of B are included in Ni60, so TiB was formed during LA process. And the formation of the TiB stick-shape precipitates are mainly attributed to the lattice characteristics of TiB (see Fig. 1d), which may act as the heterogeneous nucleation of the matrix phase [9]. In addition, during LA process, Mo reacted with a portion of Si, C, Ti and Al in laser molten pool, leading to the formations of the hard compounds [10,11], which also increased the wear resistance and the micro-hardness.

TEM image and the corresponding HRTEM image showed that lot of TiN phases are existence in such coating, which corresponded its (111) crystal plane (see Fig. 2a and b). Moreover, it was noted that the the acicular martensites were produced (see Fig. 2c). The fact that the martensites transformation completed without the diffusion, and the acicular martensites were produced in the heat-affected zone [12]. TEM image and the corresponding selected area diffraction pattern (SADP) image showed that the amorphous phases were existence in test location (see Fig. 2d). LA is one of the surface amorphization technologies due to its rapid heating and cooling that inhibites long-range diffusion and avoids crystallization, and there are also series of the amorphous alloying with high glass forming ability in Fe and Si base alloy systems [13–16]. Thus, due to the action of Fe and Si, lot of the amorphous phases can be produced, which was beneficial in improving the wear resistance of such coating [17].

As shown in Fig. 3a, lot of NC are produced in the coating. The productions of NC are mainly ascribed to the rapid cooling process of LA molten pool, lot of ceramics did not have enough time to



Fig. 1. SEM images of the composite coating: (a) the bonding location and (b and c) LA location.

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