P-09 laser ablation performance of double glow discharge sputter deposition Mo on titanium alloy surface

Shunqi Zhang (郑顺奇)[1], Gaohui Zhang (张高会)[2*], Guoqing Huang (黄国青)[2], Pinze Zhang (张平则)[3], Peng Xu (徐　鹏)[2], Gen Li (李　根)[2], and Mingzhou Yu (于明洲)[2]

[1]*Ningbo Branch of the China Academy of Ordnance Science, Ningbo* 315103, *China*
[2]*China Jiliang University, Research Institute of Surface Physics, Hangzhou 310018, China*
[3]*Nanjing University of Aeronautics and Astronautics, Nanjing 210016, China*
[*]*Corresponding author: ghzhang@cjlu.edu.cn*

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In order to improve the burn-resistant property of titanium (Ti) alloy, Ti-molybdenum (Mo) burn-resistant alloyed layer is coated onto the Ti6Al4V substrate with the double glow plasma surface alloying technology by permeating the Mo. The experiment is performed at a working temperature of 800 °C to keep the layer warm for 2.5 h under air pressure of 30 Pa. Mo and Ti6Al4V are used as the source electrode and cathode, respectively. The result of X-ray diffraction shows that the alloy phase of Ti and Mo is formed on the surface of Ti6Al4V substrate. The hardness of the alloyed layer on the surface is more than 800 HK. The friction result further indicates that wear rate can be reduced by three orders of magnitude. Laser ablation experiment is used to characterize the burn-resistant properties of the Ti alloy. The result indicates that the laser burn area of the treated Ti alloy is reduced to 1/12 of the untreated sample. Moreover, the burn-resistant properties improved greatly. Current experimental results clearly show that the laser ablation can be used to characterize the burn-resistant property of the material.

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The high specific strength of titanium (Ti) alloy has earned it the reputation of being "flight metal". The alloy has been used as a structural material in the field of aerospace engineering, in which it is commonly used to reduce the weight of the aircraft and increase the thrust-weight ratio of aero engine. It also is also widely used as compressor casing and rotor blade in advanced aero gas turbine engines[1−3]. However, recent developments in aircraft performance has increased the requirements on Ti alloys, such as greater resistance to high temperature, pressure and gas flow velocity, particularly for parts used in compressor that are subjected to high pressure and are exposed to extreme conditions. Ti alloys may be ignited and burnt under certain conditions with high temperature, pressure, and gas flow velocity. The combustion sensitivity of conventional Ti alloys increases in such severe conditions and these are prone to igniting into "Ti fire," which can limit its applications in aero engines[4−6]. Therefore, investigations on Ti alloy combustion and the explorations on fire-resistant Ti alloys to meet the requirements for high-performance aero engines have become significant issues in the aerospace engineering industry. Different series of burn-resistant Ti alloy can be made by adding different elements, such as alloy C, BTT-1, and BTT-3[3,7,8]. Although these can improve the burn-resistant properties, specific strength is reduced because the other alloy elements can enhance the specific gravity. Considering the fact that combustion always begin from the surface, surface treatment technology can be a good way to form a burn-resistant alloyed layer on the surface of these alloys. In this way, their burn-resistant performance can be improved and the advantage of high specific strength can be maintained at the same time.

"Ti fire" always accompanies heat generation induced by friction. For burn-resistant Ti alloy, a basic mechanical property is wear-resistance. Given that molybdenum (Mo) is a stable element in β state, it can be a good alloying element for burn-resistant Ti alloy due to the low burst temperature of Ti-Mo system[9]. Thus, a burn-resistant alloy layer can be formed on the surface by permeating Mo into the Ti6Al4V substrate.

The experiments were conducted in a double glow plasma furnace. Highly purified Mo in the shape of multilayer honeycomb and Ti alloy Ti6Al4V were used as source cathode and substrate, respectively. The furnace shell was taken as anode. Depend on bombardment and heat diffusion, the glow discharge and hollow cathode effect was used to allow the particle streams (composed of Mo atoms and Mo ions) to be absorbed into the surface of the speimen and form the alloy lager (Fig. 1).

The annealed Ti6Al4V was made into a ϕ 30×5 (mm) film specimen and had a chemical percentage composition wt.-% of 6.7% Al, 4.21% V, 0.07% Si, 0.10% Fe,

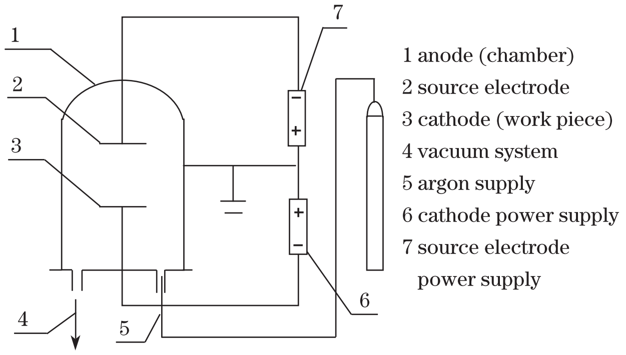

Fig. 1. Principle map of double glow discharge.

0.03% C, 0.14% O, 0.015% N, and 0.003% H. The remaining Ti was used in the current experiment. The following experimental conditions were adopted. The material was rinse polished (from 240 # to 1000 #) and then rinsed with ultrasonic wave, before returning it to the stove, with a back vacuum of 6×10^{-1}Pa. The space between the electrodes was 15 mm, source voltage was 860−900 V, cathode voltage was 350−550 V, working air pressure was 30 Pa, temperature was 880 °C, and holding time was 2.5 h.

X-ray diffraction X′Pert Pro type (PANalytical Company) was employed to analyze the composition and phase microstructures of the sample. Modelocked Ti sapphire Laser (Mai Tai HP) was used for the laser burning experiments. Both untreated and treated samples were cleaned with methanol in order to maintain the samples with the same absorbance. The operating parameters for the laser were set to the following conditions: average power of 2.60 W, pulse width of 100 fs, central wavelength of 780 nm, repetition rate of 80 MHz, and burning time of 60 s. The morphology of the sample was observed using an Olympus Metallurgical Microscope. The composition distribution of the alloying elements in the surface alloy layers produced by DG technology was analyzed through glow discharge spectroscopy (GDS). Micro-hardness was measured using SHIMAZDU HMV-2T hardness tester, and the wear properties were tested using ball-pin sliding wear machine.

The microstructure of the alloyed layer with molybdenum on the surface of Ti6Al4V is shown in Fig. 2. A single white alloyed layer composed of a single β phase with thickness of more than 30 μm formed on the surface of the Ti alloy because Mo has unlimited solubility in the β phase. The surface sediment layer is formed by part of the precipitable molybdenum under the alloying temperature (880 °C). The X-ray diffraction curve of the alloyed layer is illustrated in Fig. 3, thereby confirming that the structure of the surface layer is a single phase. The composition results of spectrum GDA750 GDS of the alloyed layer can be seen in Fig. 4. The composition of the alloyed layer shows obvious gradient change along the layer depth. The Mo content gradually decreased from the surface to the inner layer and it is very high near the surface. Moreover, the Ti gradually increased and reached normal levels at a depth of 25 μm. The gradually changing structure is helpful in forming a good transition for composition and hardness between the alloy layer and the substrate, allowing the creation of better adhesion between the substrate and alloyed layer. Moreover, it is worth noting that Al and V spread out from the substrate during the process, causing the content of the alloyed layer to become slightly less than the substrate. Due to these evaporable elements, the phenomenon of poor aluminium and vanadium occurs on the surface. This structure has some advantages in terms of its burn-resistant performance.

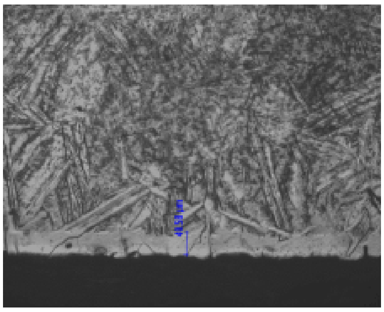

Fig. 2. Organization of Ti6Al4V after Mo alloying.

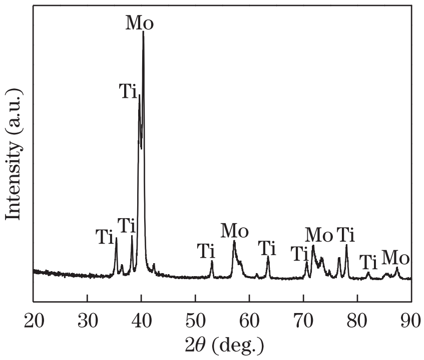

Fig. 3. XRD curve of the Mo layer.

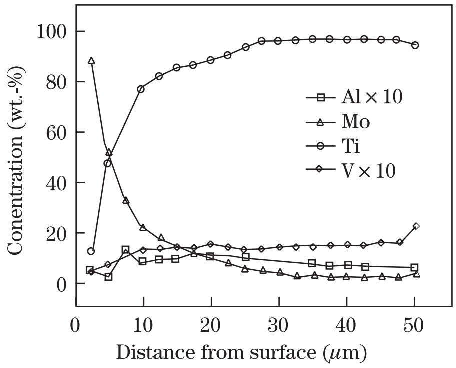

Fig. 4. Composition curve of Ti6Al4V after Mo alloying.

Hardness is a very important physical parameter characterizing the mechanical properties of the material. The hardness distribution curve is shown in Fig. 5. The surface hardness is over 800 HK, with the highest hardness value obtained on the surface of the alloyed layer due to solid solution strengthening. However, the hardness value decreased gradually with the layer depth as the content of the Mo decreased. The hardness value further decreased in the substrate as a result of the structural change from the β phase to the α phase. The gradient change in hardness is helpful in improving the coordination of deformation between the alloyed layer and the substrate.

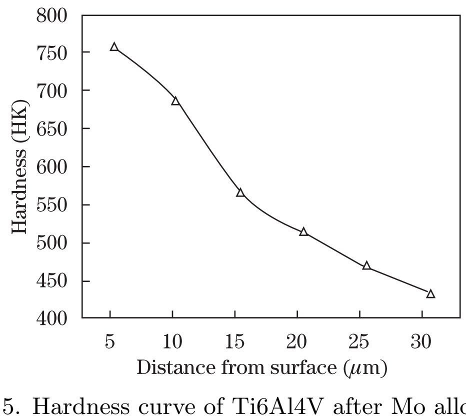

Fig. 5. Hardness curve of Ti6Al4V after Mo alloying.

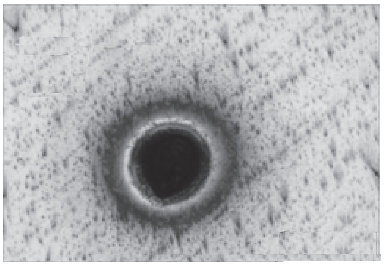

Fig. 6. Laser ablation surface morphology of untreat Ti6Al4V.

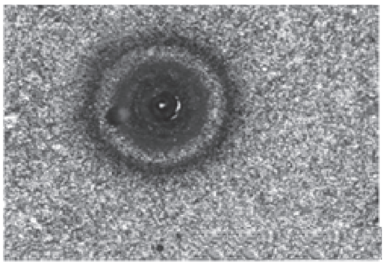

Fig. 7. Laser ablation surface morphology of Ti6Al4V after Mo alloying.

The abrasion test was conducted with a disk abrasion testing machine, which combined the abrasion of the nick morphology channel to calculate the specific wear rate. The specific wear rate of Ti alloy was 2.166×10^{-4} $mm^3 \cdot N^{-1} \cdot m^{-1}$ before infiltrating Mo. It turned to 4.548×10^{-7} $mm^3 \cdot N^{-1} \cdot m^{-1}$ after infiltrating Mo, before becoming lower than three orders of magnitude. Obviously, the abrasion loss of the Ti-Mo alloy layer is far below the substrate. Moreover, due to high hardness, its abrasion performance is better than that of the base material. Mo and β-Ti, which have the same crystal type, formed a replacement type of a continuous solid solution with a small degree of lattice contortion. Thus, the alloy both had a high intensity and plasticity.

Testing the burn-resistance performance remains a challenge. Many methods have presented both advantages and disadvantages, such as CO_2 laser beam, melted Ti drip, surface friction, spark, and plasma mechanical collision. In this letter, laser burning experiments with the mode-locked Ti-sapphire laser Mai Tai HP was used to test the burn-resistant performance of materials surface. In pulsed laser ablation, a pulsed laser beam with the power of a high peak value is focused on the material surface, causing the material surface to form a small local zone within the high temperature zone. Thus, in a short time, the material surface can be melted and evaporated rapidly, thereby resulting in the formation of plasma. The physical process of solid ablation using laser is very complex and includes energy coupling between electrons in the solid and laser radiation, absorption of laser energy by the solid caused by the transfer from laser energy to the lattice, ablation of material surface, and formation of plasma.

The sample surface was cleaned with non-water methanol to make the sample surface possess some degree of absorbance. Experimental conditions for the combustion experiment were as follows. Under normal temperature and pressure, the sample surface was subjected to 2.60-W laser power, 100-fs pulse width, 780-nm central wavelength, 80-MHz repetition rate, and 60-s laser ablation. Figures 6 and 7 show the sample surface morphologies of laser ablation before and after infiltration of Mo, respectively. The ablation marks are very characteristic. When a high-energy laser reaches the specimen surface, conductive electrons in the metal atom can absorb laser photon energy, which can then be transformed into the lattice kinetic energy in a very short time. This will cause the surface atoms to be activated and the metal surface melted with generated heat. Due to high concentration of heat, the temperature in melting area can reach evaporation temperature immediately. Thus, the metal atoms are melted and evaporated, and the ablation trace can be formed. Materials with poor fire-resistant properties have big ablation area and deep ablation depth. On the other hand, the ablation area is relatively small and ablation depth is shallow for the material with good fire-resistance properties. Moreover, the depth penetrated by laser is only 10−5 cm from the metal surface. Thus, only the surface can be heated with laser heating and the heat conducted from the surface to inner. Therefore, the ablation area on the surface of samples with laser ablation is also very shallow. One spreads from the center to the outside due to thermal diffusion, while the other has plenty of concentric circles because of the pulse laser, whose heat absorption is the pulse. After the infiltration of Mo, the marked diameter of the central ablation part decreased from 113.5 (when untreated) to 55.143 μm. In addition, the central ablation area decreased to 1/4 of the original size. The marks after the infiltration of Mo showed that there is a large transition zone from the central ablation zone to the outside. Analyzing the reasons, it should be the role of infiltrated Mo layer to delay the diffusion of combustion. The results of the experiments indicate that the infiltration of Mo plays an important role in burn-resistance.

In conclusion, after coating the alloy layer with a double glow discharge sputter deposition onto the Ti alloy surface with a thickness of over 50 μm, the conclusions listed below are reached. The hardness of the alloy layer decreases gradually from the surface to the depth, and the surface hardness is more than 800 HK. The abrasion performance of the Ti alloy that is permeated by Mo clearly improved, with the wear rate reduced by three orders of magnitude. The central ablation area is reduced to 1/4 of the original size, indicating the existence of burn-resistant properties.

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