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RECENT DEVELOPMENTS IN IMPROVING TRIBOLOGICAL PERFORMANCE OF TC4 TITANIUM ALLOY VIA DOUBLE GLOW PLASMA SURFACE ALLOYING IN CHINA: A LITERATURE REVIEW

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Abstract. Double glow plasma surface alloying (DGPSA) process is an effective approach to providing satisfactory surface protection and enhancement of metallic materials. DGPSA can accomplish surface alloying by both non-metallic elements and solid metallic elements as alloying elements to create a strong/gradient metallurgical bonding between the coating and the substrate. This review begins with the technological principle and processing research of DGPSA. The studies and applications of the DGPSA for improving tribological performance of TC4 titanium alloy in China are reviewed in the sight of alloying element selections.

1. GENERAL BACKGROUND

Titanium is the fourth most abundant structural metal in the world and is present in the earth's crust at a level of about 0.6 percent. Titanium and its alloys have been developed rapidly since the pure metal first became commercially available about sixty years ago [1,2]. Due to their promising merits of high strength to weight ratio, high yield strength and toughness, good corrosion resistance as well as exceptional biocompatibility, titanium alloys exhibit an ever increasing interest in the fields ranging from civilian goods to military equipments [3–7]. Many kinds of titanium alloys have been designed and developed for different purposes in recent years.

Up to now, Ti6Al4V (referred to TC4 in the following) is still the most frequently and successfully used titanium alloy which occupies about one half of the total world production of titanium alloys [8]. However TC4 cannot meet all of the engineering requirements, e.g. it is seldom operated in tribological-related engineering conditions by its drawbacks of low surface hardness values, high coefficient of friction and poor abrasive wear resistance [9–11]. It has been well accepted that failure of material in engineering, e.g. wear or corrosion is mainly determined by the surface properties of the material rather than by bulk properties. Hence the surface treatment is an attractive and suitable way to solve the aforementioned problems [12]. Meanwhile surface performances of materials can be improved selectively using appropriate surface treatment techniques while the desirable bulk attributes of the materials are retained, and proper surface treatment can expand the applications of materials in different fields [13]. The surface treatment can also make a favourable compromise between the cost and the performance of engineering components. Therefore a variety of surface techniques, such as surface alloying [14,15], micro arc oxidation (MAO) [16,17], plasma electrolytic oxidation (PEO) [18,19], physical vapour deposition (PVD) [20,21], chemical vapour deposition (CVD) [22,23], ion implantation [24,25],

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thermal/cold spraying [26,27], thermal oxidation [28,29], laser surface treatment [30,31], electron beam irradiation [32,33], and electro spark deposition (ESD) [34], also several duplex treatments have been conducted to enhance the corrosion resistance and tribological property of TC4 by obtaining a chemical stable (mechanical isolation or passivation) and hard (wear resistant or friction reduction) surface. Each of the mentioned techniques has achieved certain success under different service conditions; actually the techniques also hold their own advantages and disadvantages [35–39].

Surface alloying can improve the surface hardness, enhance the wear and corrosion resistance, as well as obtain a strong metallurgical bond between the alloying layer and the substrate with less alloying elements than bulk alloying. Amongst the surface alloying techniques, double glow plasma surface alloying (referred to as DGPSA hereafter) which is also called as Xu-Tec/Xu-Loy process, was invented by Prof. Zhong Xu from Taiyuan University of Technology (Taiyuan University of Technology, Taiyuan, P. R. China) in 1980 and since then covered by US and other international patents [40]. Actually the DGPSA was developed based on the plasma nitriding and sputtering techniques, while the DGPSA is able to accomplish surface alloying by both non-metallic elements (NME) and solid metallic elements (ME) as alloying elements. A great number of studies that concerning the DGPSA technology have been conducted to refine the composition or microstructure in the near surface and enhance the surface properties, such as corrosion resistance, wear resistance and resistance to high temperature oxidation of Ti-based materials under different conditions in the past three decades.

In this review, the technological principle and the processing research of DGPSA were briefly introduced at first, and the applications of DGPSA process for improving the tribological performance of Ti6Al4V titanium alloy were reviewed and summarized.

2. TECHNOLOGICAL PRINCIPLE AND PROCESSING RESEARCH OF DGPSA

As mentioned above that the DGPSA was developed based on the plasma nitriding and sputtering techniques, a chamber equipped with a complete vacuum system is necessary. Fig. 1 suggests the schematic diagram of DGPSA apparatus. In the vacuum chamber, the work piece and the so called

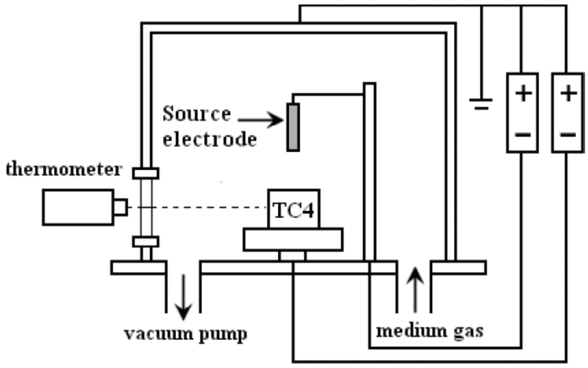


Fig. 1. Schematic diagram of DGPSA apparatus.

source electrode (made up of one or more desired alloying elements) are two negatively charged members and the vacuum bell jar which is earthed is the anode. As shown in Fig. 1, when the two power supplies are put through and reach certain voltage values, both cathode and source electrode are enveloped with glow discharge under the argon atmosphere. One glow discharge heats the work piece to be alloyed. While the second glow discharge bombards the source electrode to sputtering the desired alloying elements. The phenomenon is called as the “double glow discharge”. The charged ions or particles which are bombarded from the source electrode migrate to and firstly diffuse into then deposit onto the surface of the work piece under the influence of an electric field, and then obtaining an alloyed surface. The thickness values of the surface alloying layers vary from several micrometers to 500 μm, with alloying elements in a concentration of few percent to 90% or more [41]. Prof. Zhong Xu and his team in Taiyuan University of Technology (Taiyuan, P. R. China) discovered this phenomenon during the plasma nitriding trials in 1980s, and then developed and named the so called “DGPSA” process [40]. DGPSA holds many advantages, several most important features are listed as follows: (1) resource and precious metal element conservation, (2) free of pollution, (3) controlled alloy composition on the surface, (4) wide range of alloying elements, (5) gradient distribution of the composition/property, (6) holding a metallurgical bond between alloying layer and substrate [40–42].

Since the double glow discharge phenomenon was discovered, great deals of studies on DGPSA processing with the purposes of obtaining higher-performance alloyed surface were conducted. However the research work can be mainly grouped into the following three aspects: investigation of process parameters; placement between the work piece and

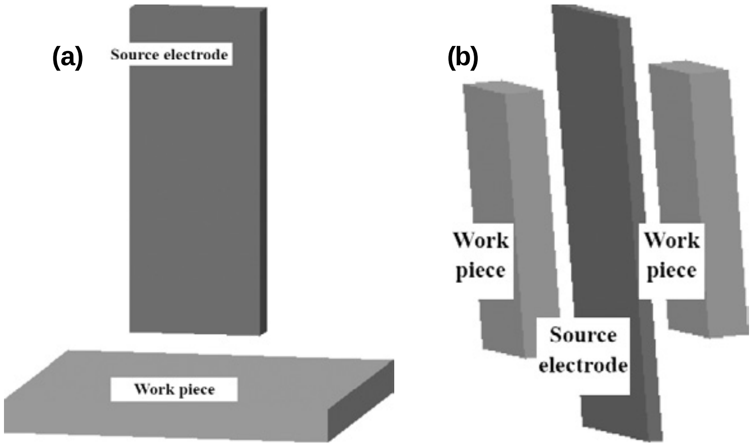


Fig. 2. Placements between the work piece and the source electrode.

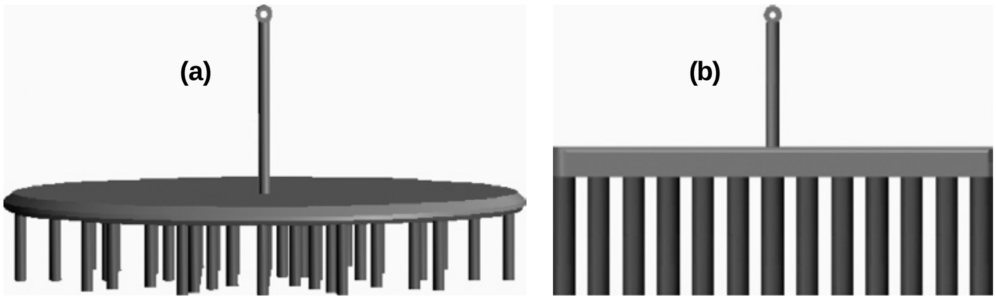


Fig. 3. Special shapes of the existed source electrode.

the source electrode; shape of the source electrode [43–48]. In respect of the investigation of process parameters, related parameters contain: bias voltage of work piece, bias voltage of source electrode, distance between the work piece and the source electrode, gas pressure, process temperature and soaking time. By adjusting the bias voltages of work piece and source electrode, the glow discharge, heating of work piece and sputtering of source electrode are all realized. Only when the distance between the work piece and the source electrode, as well as gas pressure is in proper range can form continuous and uniform coating. The formation of DGPSA coating is basically determined by diffusion, therefore process temperature and soak time can affect the thickness of the obtained coating. With respect to the placement between the work piece and the source electrode, two types of placements are shown in Fig. 2. The two types of placements could accomplish DGPSA for work pieces with different sizes. As to the shape of the source electrode, besides the normal source electrode as given in Fig. 2, Fig. 3 exhibits the available special shapes. Designing the source electrode with special shapes aimed at improving the sputtering of source electrode.

3. APPLICATION OF DGPSA FOR IMPROVING TRIBOLOGICAL PERFORMANCE OF TC4

A great amount of research has confirmed that the tribological performance of materials depends very much on the properties of their surfaces that are affected by two major factors: one is the nature and composition of the surface; and the other is the degree of hardness on the surface. According to the literature, the improved tribological performance of TC4 that achieved by DGPSA is due to the change in surface hardness and surface composition, and then the DGPSA treated TC4 can indicate excellent friction-reducing effect and/or promising wear resistance. Meanwhile obtaining a strong metallurgical bond between the coating and the substrate benefits the tribological performance. Up to now the tribological performance of TC4 after the DGPSA treatment has been estimated by different authors, as presented in the references. At present, there are two modes to achieve surface alloy via DGPSA which can improve the tribological performance of TC4 in various degree. One is single element alloying by DGPSA, alloying with single NME or ME; the other is binary element alloying by DGPSA,

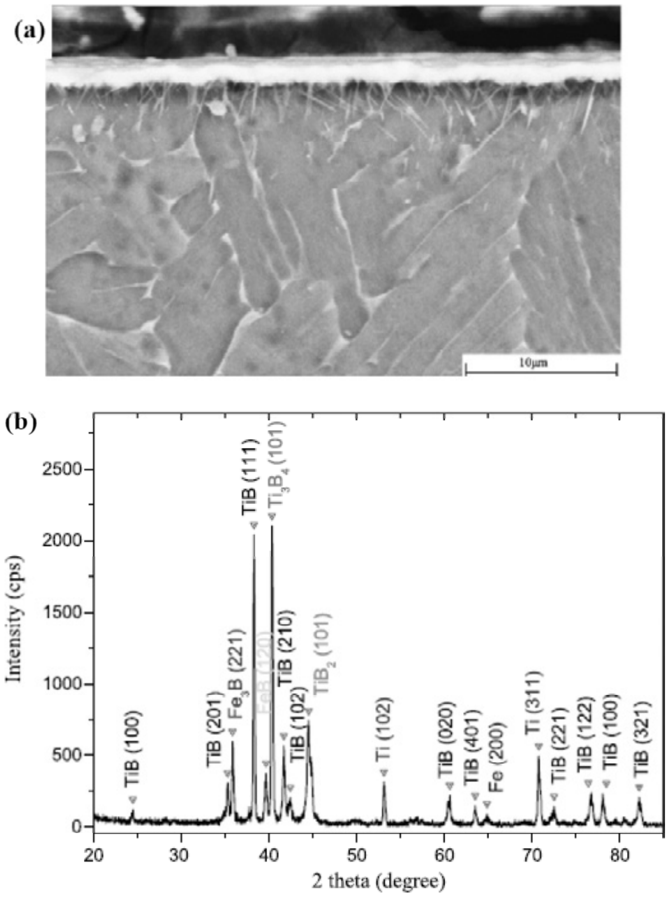


Fig. 4. Cross-section SEM image (a) and XRD spectrum (b) of the borided TC4.

existing NME + NME, NME + ME and ME + ME. The NMEs contain boron (B), carbon (C), and nitrogen (N), while the MEs cover a series of elements: aluminium (Al), chromium (Cr), Copper (Cu), molybdenum (Mo), niobium (Nb), nickel (Ni), tungsten (W), and zirconium (Zr). Also minor research of ME + ME + NME: W-Mo-N alloying has been conducted. The positive role of the mentioned elements lie in the following aspects: formation of hard phases in the coating or in the near surface of TC4, receive a hard surface by obtaining intermetallic compounds, solid solution or dispersion strengthening on the near-surface of TC4. The present work reviews the applications of DGPSA process for improving tribological performances of TC4 that are mainly centred on single element and binary element alloying. While the tribological performances of TC4 substrates and DGPSA treated TC4 samples were comparably estimated via various conditions in laboratory including dry friction in air at different temperature and corrosive-wear in different solutions. This work is hoped to create database and provide reference information for practical application of DGPSA on TC4 or other metallic materials.

3.1. Single element alloying by DGPSA

3.1.1. Alloying with non-metallic elements

From an alloying element selection of view, boron (B), carbon (C), and nitrogen (N) are the main alloying elements of DGPSA on TC4 at present. Boriding on metal materials, as a diffusion technique, is attracting more and more attention because the borides formed during the treatment exhibit excellent wear resistance in many tribological systems. Qin et al. produced a boride layer on TC4 by DGPSA, and the boride layer was mainly composed of TiB with minor TiB_2 and Ti_3B_4 (Fig. 4). Compared to TC4 substrate, the borided TC4 presented significant improvement of wear resistance under dry sliding conditions. Meanwhile the boride layer exhibited excellent toughness and bonding strength [49,50]. As Ti is a kind of strong carbide forming element, it is realizable to obtain Ti-carbide layer on TC4 by employ of DGPSA. Zhang et al. prepared carburized layers using netlike highly pure graphite as the source electrode. Hard TiC phase was formed in

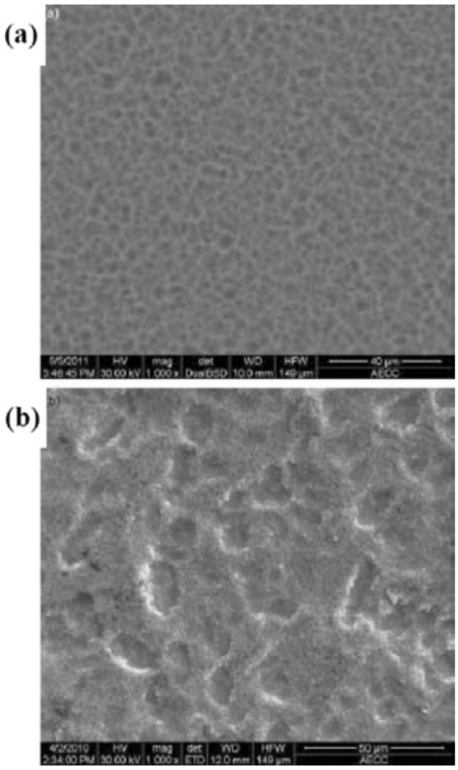


Fig. 5. SEM image of surface morphologies of Nitrogen-DGPSA (a) and CPN layers.

the carburized layer, and the carburized TC4 showed a hard and high wear resistant surface [51]. The improvement in the friction and wear performance of the carburized specimen was attributed to the changes in the composition, microstructure, and hardness by the carburizing. Nitrogen has a high solubility in α-Ti so it can significantly strengthen the surface layer [52]. The nitriding process leads commonly to the formation of titanium nitrides within the matrix Ti (α)-N solid solution. Tang et al. compared the characteristics and properties of Ti-N layers on TC4 that obtained by conventional DC plasma nitriding (CPN) and nitrogen-DGPSA, respectively [53]. It was indicated that the surface roughness of the DGPSA layer was about 1 order less than that of the CPN layer (Fig. 5). Furthermore, the nitrogen-DGPSA treated surface possessed much higher wear resistance than the CPN layer at higher loads, and increased the wear resistance of Ti6Al4V alloy by about 2 orders was attributed to the higher surface hardness. This is because the double glow discharge effect in nitrogen-DGPSA strengthened the density of the active nitrogen atoms and improved the nitriding efficiency. Hu conducted Nitrogen-DGPSA on TC4 and invested the tribological properties of the formed nitriding layer by ball-on-disc tests against GCr15 steel ball and corundum ball [54]. The continuous nitriding layer was composed of Ti_2N, TiN and N-solid solution in α-Ti. The N-modified TC4 with higher surface hardness showed lower friction coefficients and wear loss, the obtained nitriding layer had significantly improved the tribological performance of TC4.

3.1.2. Alloying with metallic elements

There are several metals, such as aluminium (Al), chromium (Cr), copper (Cu), molybdenum (Mo), niobium (Nb), nickel (Ni), and zirconium (Zr) have been selected as alloying elements for DGPSA on TC4. Duan observed that the aluminium-DGPSA coating which consisting of an Al deposition layer and an Al_3Ti compound layer, could enhance the surface hardness and tribological property of TC4 under dry sliding against GCr15 ball in air [55]. Zhang et al. reported that when alloying element Cr was induced into the TC4 substrate by DGPSA, Ti (α)-Cr solid solution and minor Cr_2Ti were detected on the subsurface. The formed chromizing coating could improve the wear resistance of TC4 at elevated temperature and avoided the ignition and burn of titanium alloy [56–58]. Zhang et al. found that Cu-DGPSA coating on TC4 showed excellent wear re-

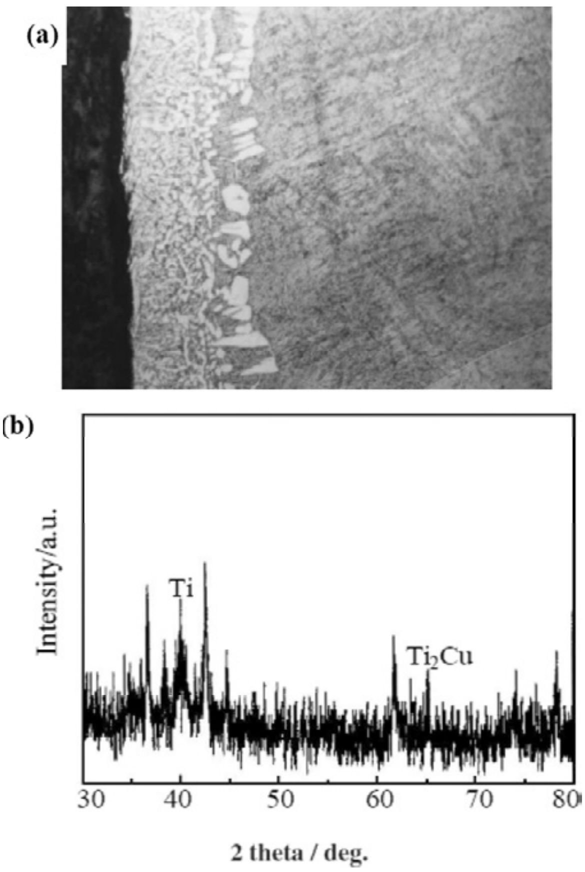


Fig. 6. Cross-section SEM image (a) and XRD spectrum (b) of the Cu-alloyed layer on TC4.

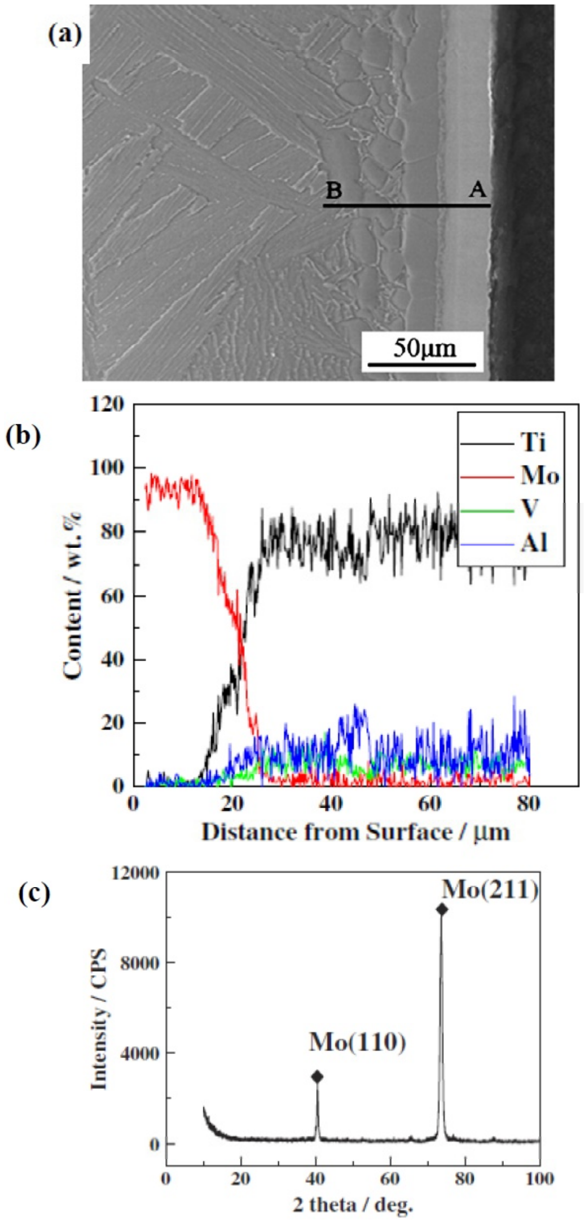


Fig. 7. Cross-section SEM image (a), EDS composition profile (c) and XRD spectrum (c) of the Mo-alloyed layer on TC4.

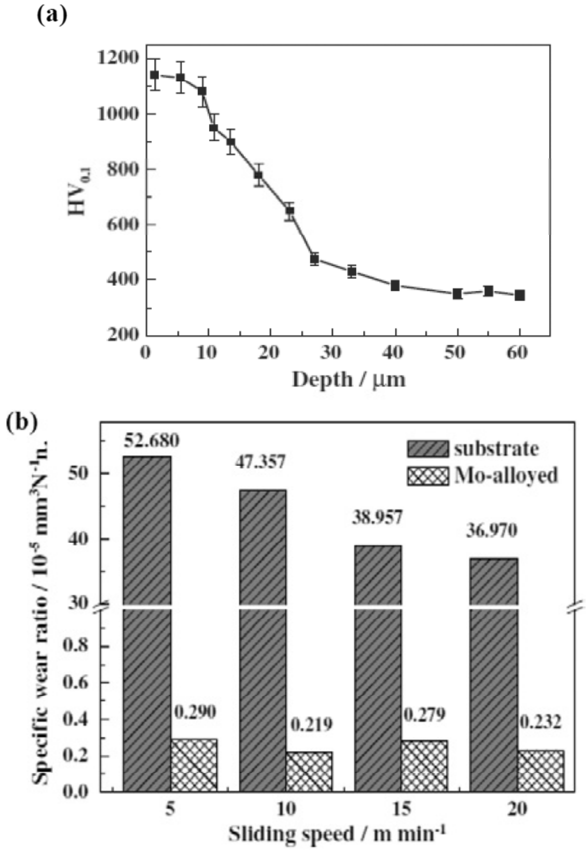


Fig. 8. Micro-hardness as a function of the depth (a) and wear ratios after rubbing in different velocities (b) of the Mo-alloyed layer on TC4.

sistance at high temperature. The obtained continuous copperizing alloyed layer was composed of Ti-Cu solid solution and Ti_2Cu intermetallic (Fig. 6). Meanwhile the alloyed layer indicated promising burn-resistance. Both effects of the copperizing alloyed layer mentioned above was attributed to the evolvement of Cu in the form of liquid phase from the coatingÿwhich could both play roles of liquid lubrication and endothermic effect [59]. As Mo possesses infinite solubility in Ti (b), Mo-DGPSA treatments and tribological behaviors of the titanium alloys have been studied by different authors. Liang et al. obtained a uniform and compact molybdenizing coating on TC4 and investigated the tribological behaviors of the Mo-DGPSA treated TC4 by ball-on-disc rubbing experiments under different sliding speeds [60]. The results indicated that the coating was composed of a deposition layer and a diffusion layer (Fig. 7 and Fig. 8). The received coating exhibited gradient distributions in cross-sectional composition and hardness values which are beneficial to enhance the tribological behavior of the Mo-DGPSA treated TC4. While the Mo-alloyed layers showed lower mass losses in tribological tests, as expected. The wear resistance of the specimens with molybdenized layer was significantly improved due to the much higher surface hardness and higher bonding strength (Figs. 9 and 10). Qin et al. fabricated Ti-Mo alloyed layer on TC4 using Mo-DGPSA, it was found that the Ti-Mo alloyed layer exhibited good wear resistance in sliding and its wear rate is 100 times less than that of TC4 substrate [61]. Zhang et al. found that that the ratio wear rate of molybdenized TC4 decreased to 1/500 of the substrate when sliding against GCr15 steel ball, promising wear resistance of the TC4 benefited from the high hardness on the surface [62]. By employ of

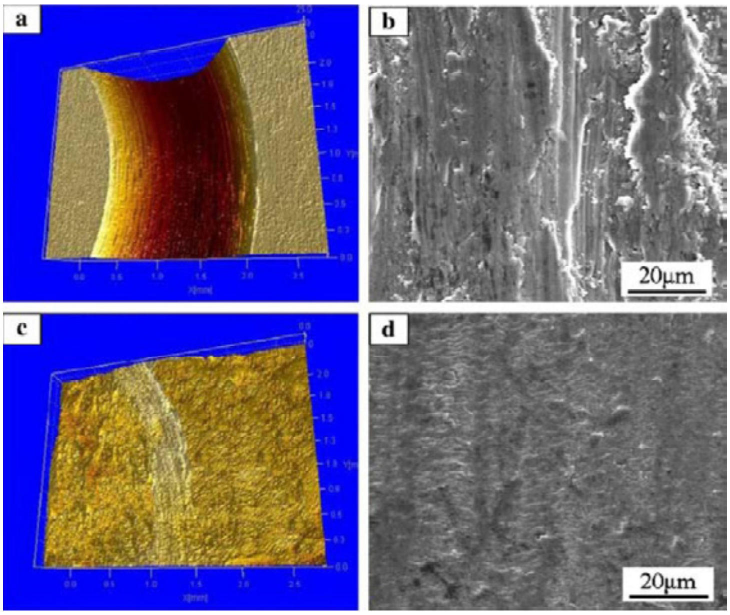


Fig. 9. 3D surface profilometry and SEM images of the wear traces after rubbing in 10 m/min: (a), (b) TC4; (c), (d) Mo-alloyed layer.

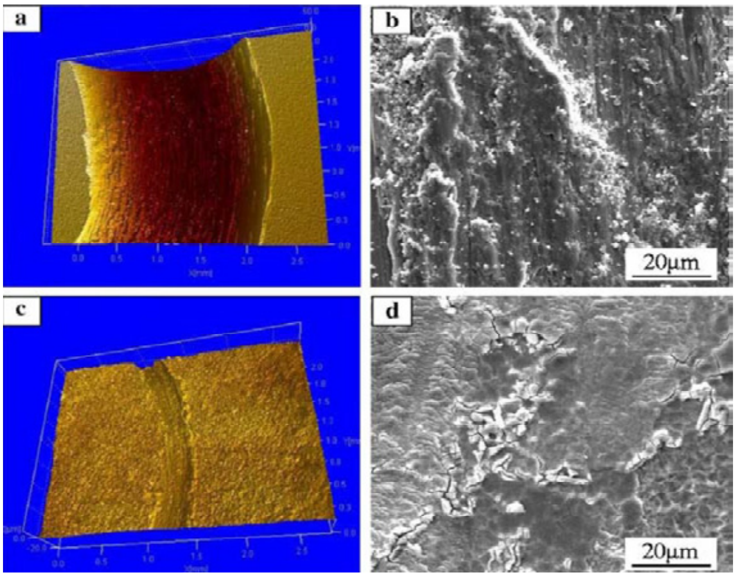


Fig. 10. 3D surface profilometry and SEM images of the wear traces after rubbing in 20 m/min: (a), (b) TC4; (c), (d) Mo-alloyed layer.

orthogonal experiment design, Ben obtained optimum parameters of Mo-DGPSA: temperature: 900~950 °C, source voltage: -900~-950 V, cathode voltage: -400~-450 V, distance between source electrode and work-piece: 15 mm, working pressure: 30~35 Pa, diffusing time: 3 h. Mo-DGPSA treatment dramatically enhanced the wear resistance of TC4 [63]. Tang et al. investigated the tribological behavior of double-glow discharge Mo layers on TC4 in aviation kerosene environment [64]. It was found that the polished Mo modified layers could both reduce the friction coefficient and enhance the wear resistance of the TC4. An et al. also produced a hard and wear-resistant Mo modified layer on TC4, whose wear trace was far narrower than that of raw TC4 [65]. He obtained Nb-coating and studied the effect of Nb-DGPSA treatment on the surface property of TC4 alloy. The results showed that the uniform/continuous Ti-Nb alloying layer could increase the surface hardness and wear resistance, as well as oxidation resistance in air of TC4 (Fig. 11) [66]. Wang et al. achieved a sufficiently dense and defect-free TiNi layer by DGPSA with nickel (Fig. 12). The maximum value of surface Ni content is nearly 90%, and the concentration of Ni presented a gradient distribution. The Ni modified layer is mainly composed

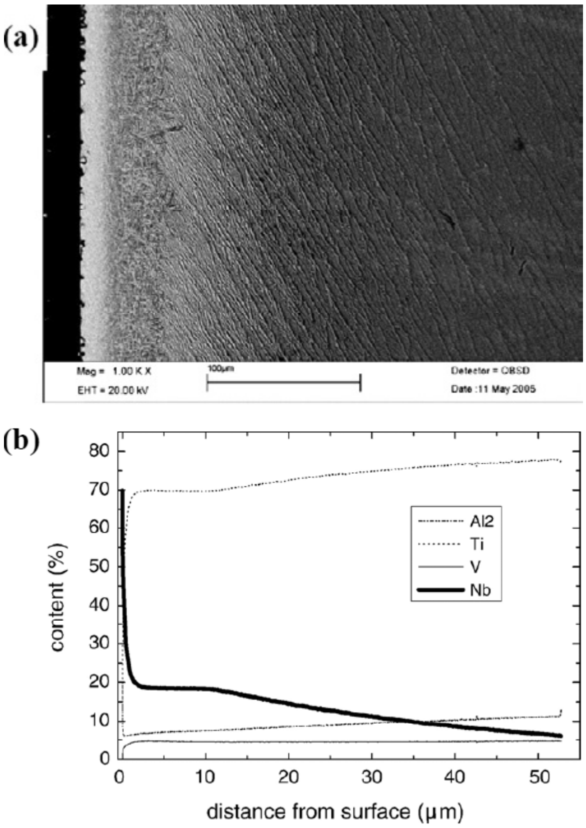


Fig. 11. Cross-section SEM image (a) and GDS composition profile of the Nb-alloyed layer on TC4.

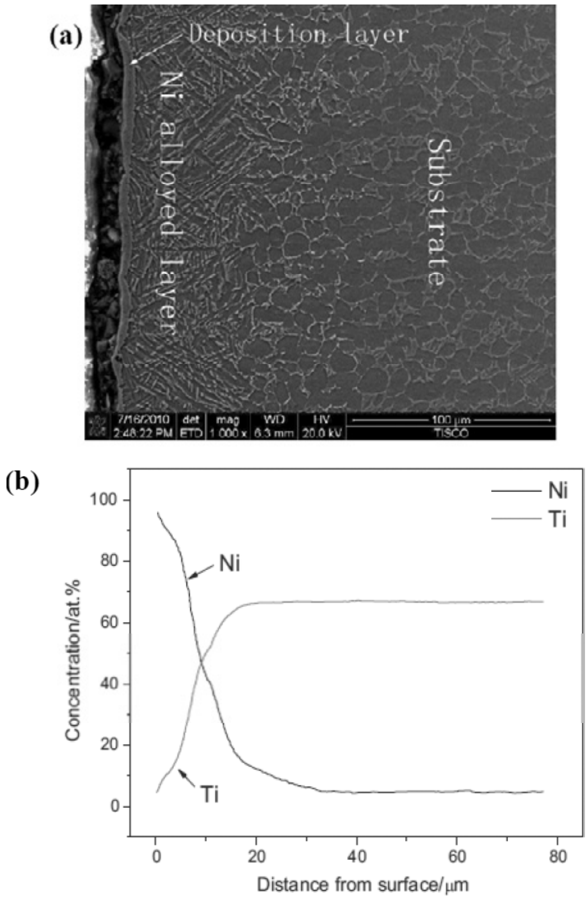


Fig. 12. Cross-section SEM image (a) and GDS composition profile of the Ni-alloyed layer on TC4.

of TiNi, Ti_2Ni, and Ti phases. The maximum microhardness of the Ni modified layer was about 677 $HV_{0.025}$, which is increased about two-fold of microharness value of the substrate due to solid solution and dispersion strengthening. The TiNi layer exhibited better tribological performance than that of the raw TC4 sample due to the high surface hardness of intermetallic precipitation of Ti_2Ni, and excellent ductility of TiNi phase (Fig. 13) [67,68]. Li et al. prepared homogeneous and compact Zr alloying layer using DGPSA process on TC4 (Fig. 14). Micro hardness values of the Zr-alloying layer along the thickness presented a gradient distribution. Solid solution strengthening resulted in the improvement in hardness and wear resistance of Zr surface alloyed TC4 alloy [69].

3.2. Binary elements alloying by DGPSA

3.2.1. Alloying with metallic + non-metallic elements

Fan prepared Mo-N co-alloying layer on TC4 alloy by DGPSA, it was found that the co-alloying layer demonstrated better friction reduction and higher anti-wear property than that of TC4 against corundum ball in Hanks' solution [70]. Similar to Fan's results, Liang investigated the tribological behaviors of Mo-N modified layers. The results showed that Mo-N surface-modified layers exhibit lower friction coefficients and lower wear rates in comparison to untreated TC4 samples at room temperature and 300 °C. However when the tribological tests were conducted at 500 °C, the Mo-N surface-modification layer exhibited higher friction coefficient and lower wear rate as compared with untreated TC4 surface. The satisfactory performance of Mo-N modified layer was attributed to its high chemical stability, surface hardness and strong bonding strength between the layer and the substrate [71]. He obtained Nb-C co-alloying layer on TC4 by subsequent carburizing after Nb-DGPSA, hard phases in the Nb-C layer had ensured the surface of TC4 with higher hardness and load bearing capacity, as well as better tribological performance [66]. Li and Tang formed Zr-N modified layers on

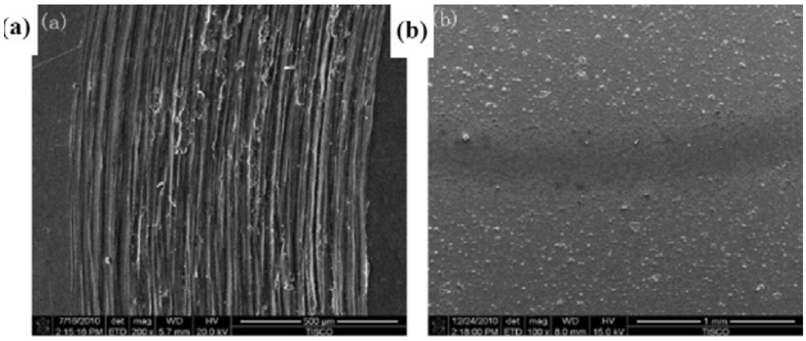


Fig. 13. SEM images of worn surfaces: (a) TC4; (b) Ni modified layer.

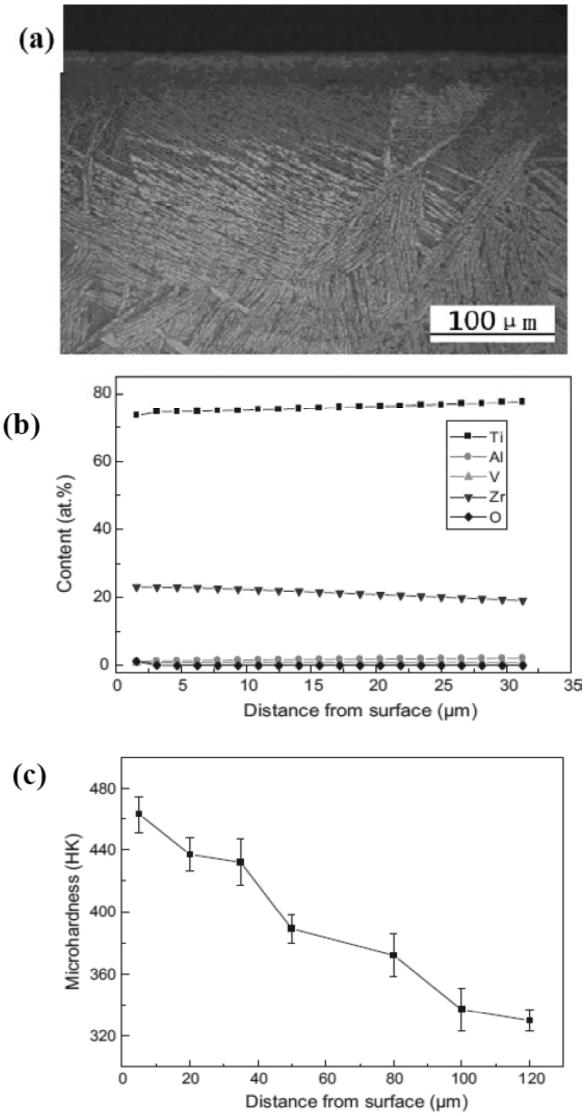


Fig. 14. Cross-section OM image (a), GDS composition profile (c) and microhardness distribution (c) of the Zr-alloyed layer on TC4.

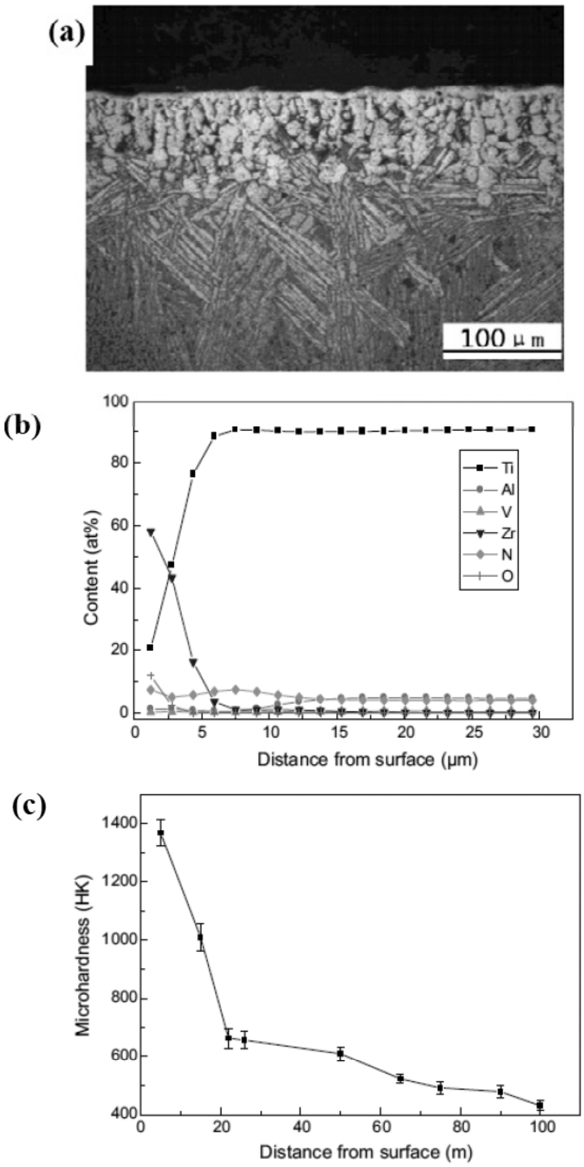


Fig. 15. Cross-section OM image (a), GDS composition profile (c) and microhardness distribution (c) of the Zr-N alloyed layer on TC4.

TC4 alloy by DGPSA process (Fig. 15). As shown in Fig. 15, the Zr-N layers is continuous and compact, the surface microhardness of TC4 alloy were significantly increased, meanwhile the wear resistance of TC4 at room temperature and elevated temperature were significantly improved [69,72].

3.2.2. Alloying with metallic + metallic elements

Qin employed DGPSA to synthesis Mo-Cr coating on TC4 alloy; the obtained coating significantly im-

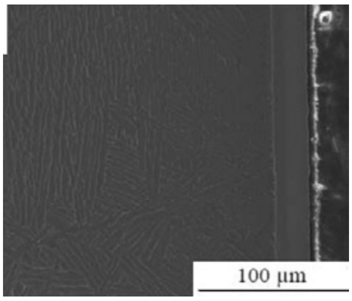


Fig. 16. Cross-section SEM image W-Mo alloyed layer on TC4.

proved both the surface hardness and wear resistance of the TC4 [73]. Wang et al. developed Ni-Ti co-diffusion coating on TC4 by employ of DGPSA using a NiTi shape memory alloy source electrode, the coating exhibited good tribological performance when sliding against GCr15 and Si_3N_4 balls in air. Meanwhile the corrosive-wear resistance on the surface of TC4 in NaCl and HCl solution was greatly enhanced by Ni-Ti co-diffusion coating [74,75]. Zhang et al. prepared W-Mo DGPSA-coating on TC4 and

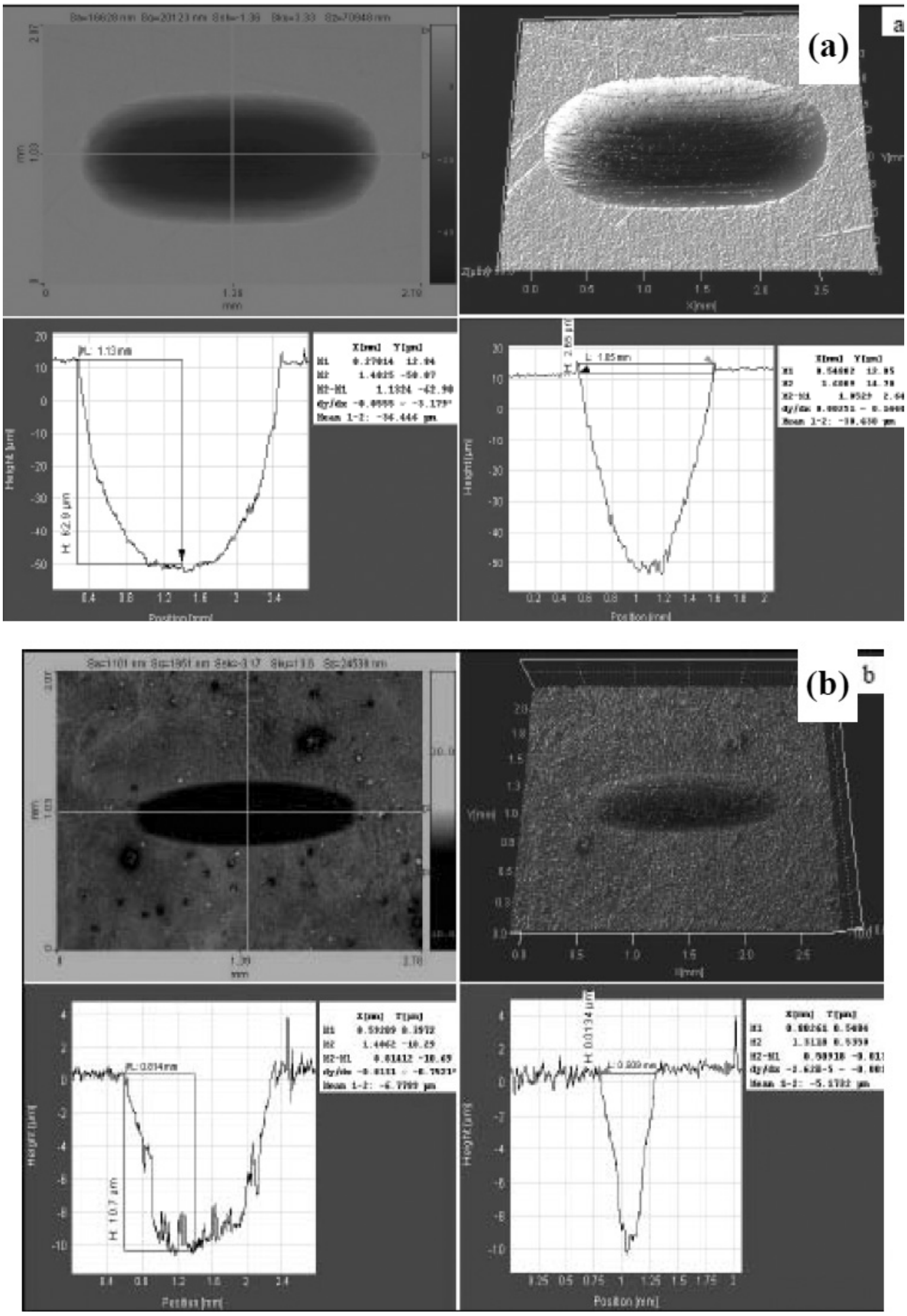
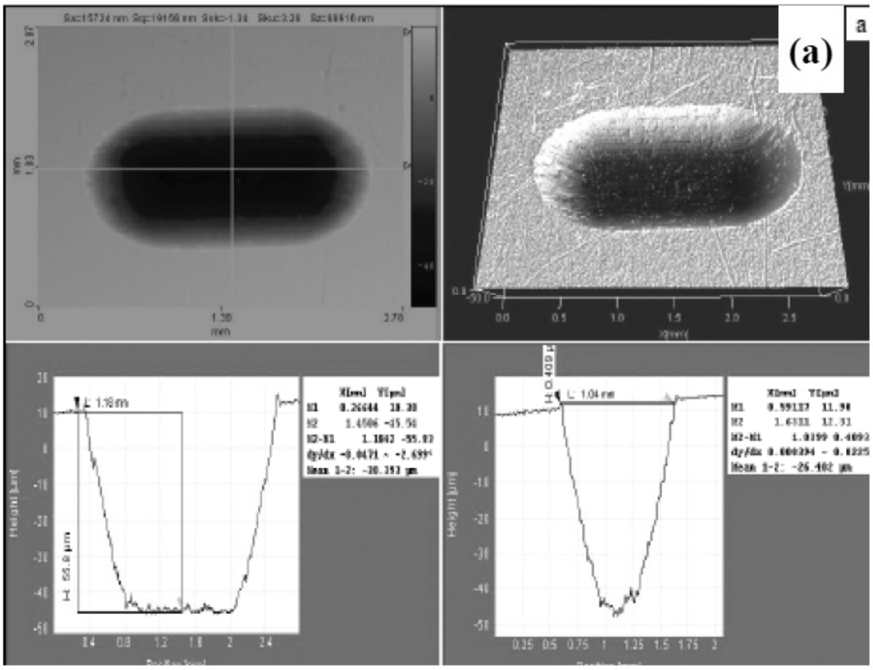


Fig. 17. Scratch morphologies and 3D profiles of TC4 (a) and W-Mo (b) co-penetrated layer in 5% NaCl solution.



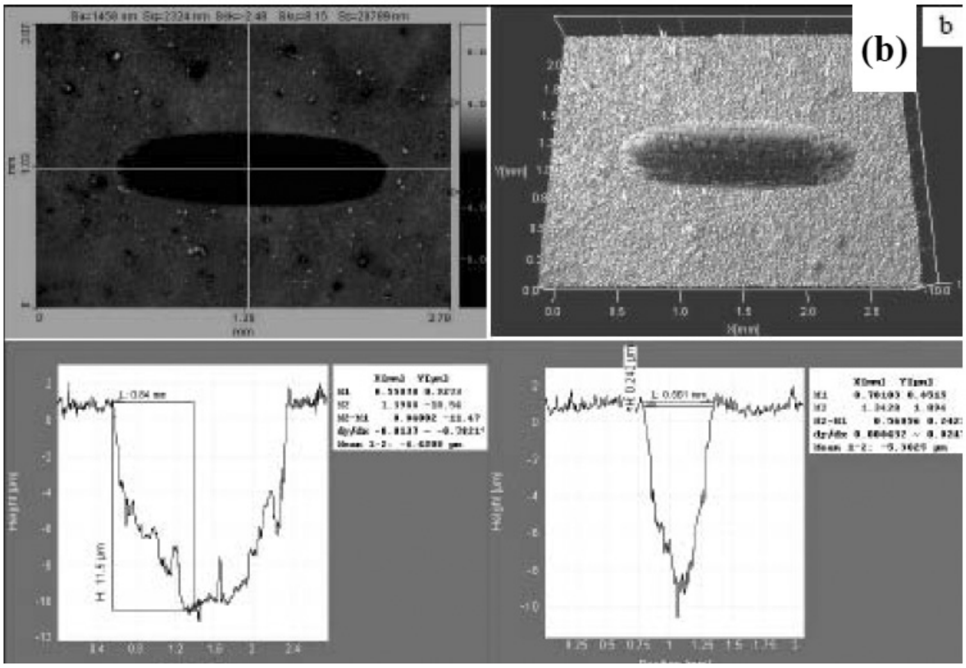


Fig. 18. Scratch morphologies and 3D profiles of TC4 (a) and W-Mo (b) co-penetrated layer in 5% H_2SO_4 solution.

systematically compared the tribological performance of the treated/untreated TC4 in 5 wt.% NaCl and 5 wt.% H_2SO_4 solutions [76]. Uniform and compact W-Mo DGPSA-coating combined well with the substrate (Fig. 16). Seen from Fig. 17, the alloying layer exhibited improved wear morphology and an obviously decreased wearing volume in 5 wt.% NaCl as compared with TC4 substrate. While similar findings had been found when the tribological tests were conducted in 5 wt.% H_2SO_4 solutions, as shown in Fig. 18. All these results indicated that W-Mo DGPSA-coating could enhance the corrosive-wear resistance of the TC4 alloy. In Sun's research achievements, the W-Mo DGPSA-coating exhibited excellent wear resistance in corrosive medium and at elevated temperature [77].

3.2.3. Alloying with non-metallic + non-metallic elements

Zhang utilized DGPSA to fabricate C-N co-diffusion layer on TC4 alloy, and the obtained layer illustrated higher surface hardness and better wear resistance than those of the TC4 substrate under the same testing condition [78]. Wu systematically studied the tribological behaviors of C-N DGPSA modified coatings on TC4 under different sliding rates, loadings and counterparts [79]. It was found that the C-N modified coatings always presented better tribological performance than that of the TC4 substrate.

3.3. Multi-elements alloying by DGPSA

Tang et al. obtained W-Mo and W-Mo-N coatings on Ti6Al4V alloy using DGPSA. The surface hardness values of the W-Mo-N and W-Mo coatings were 25.3 GPa and 14.2 GPa, which is 7-fold and 3.9-fold harder than the TC4 substrate, respectively. The wear and corrosion resistance of TC4 were significantly improved by W-Mo and W-Mo-N coatings. Meanwhile the W-Mo and W-Mo-N coatings showed better wear resistance in NaCl solution than that of in ambient air, which was benefited from the lubrication effect of the NaCl solution and the excellent corrosion resistance of the modified coatings [80].

4. SUMMARY

Ti6Al4V frequently has been used for engineering components by offering several advantages of low density, low modulus elasticity, excellent corrosion resistance, and biocompatibility. However, poor tribological performance has restricted its widely applications. DGPSA process which can provide a metallurgical bonding between the alloyed layer and the substrate can achieve surface alloying by applying a series of non-metallic and solid metallic elements. An overview on the studies and applications of the DGPSA for improving tribological performance of Ti6Al4V titanium alloy has been presented. The improved tribological performance under different testing conditions and various practical applications was mainly realized by obtaining a hard and friction reduction surface with high chemical stability on Ti6Al4V after various DGPSA treatments with great success. Formation of hard phases or intermetallic compounds, solid solution or dispersion strengthening in the coating or in the near surface of Ti6Al4V had contributed a great to the improved surface properties.

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