



## Short communication

## Combining thermo-diffusing titanium and plasma nitriding to modify C61900 Cu–Al alloy

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## ABSTRACT

To improve the mechanical properties of C61900 copper–aluminum alloy, a multi-phases coating was fabricated by the combination of thermo-diffusing Ti film and plasma nitriding. The results showed that a smooth coating with fine and uniform grains could be achieved, and the 9 μm thick multi-phases coating, containing Ti–Cu–Al intermetallics (CuTi<sub>2</sub>, CuTi, AlCu<sub>2</sub>Ti) and titanium nitride (Ti<sub>2</sub>N) formed on the nitrided surfaces. The surface hardness of the coating was obviously improved to be 630 HV<sub>0.01</sub>, and the cross-sectional hardness decreased gradually to that of C61900 Cu–Al alloy. Moreover, the wear rate of the coated specimen decreased significantly, dropping 95.5% compared with the untreated Cu–Al alloy.

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C61900 copper–aluminum (Cu–Al) alloys are important structural materials due to their good thermal conductivity and excellent wear resistance, which are often used to produce gears, bearings, bushings, etc. For the excellent performance at hardness, wear resistance and corrosive resistance, TiN shows great advantage as protective coating material to further improve the high hardness, low friction and good wear resistance of Cu alloys [1,2]. However, the higher modulus and hardness of TiN resulted in dramatic change at the interface between TiN coating and the substrate, which made the coating easily shedding. To solve this problem, the preparation of the intermediate layer consisting of Cu based intermetallics or using Cu based intermetallics as secondary phases in TiN layer could be an effective method [3,4].

Among the varied technologies to enhance the surface mechanical properties, such as equal channel angular extrusion (ECAE) [5], laser surface alloying [6], heat treatment [7,8], and special elements addition [9], etc, thermochemical diffusion process is a significant surface modification process that has been widely used in modern manufacturing technologies [10]. Nitriding is one of the thermochemical treatment processes, which involves deposition and diffusion of nitrogen into metallic materials and can improve

surface hardness, wear and corrosion resistance significantly [11–13]. Cathodic cage plasma nitriding (CCPN) is an innovative technique that can reduce the edge effect [14,15]. Unfortunately, only a limited number of investigations have been reported to improve the properties of Cu alloys through doping titanium and nitrogen into the surface of copper alloys [16,17]. Especially for C61900 Cu–Al alloy containing higher content of Al element, the above method would cause obvious change of the component and structure, thus resulting in great change in performance.

Based on the above discussion, plasma nitriding of Ti coated C61900 Cu–Al alloy was conducted to obtain a multi-phases surface modification layer in the present work. Both the microstructure and mechanical properties of the coating were investigated, compared with the untreated material surface.

The chemical composition of C61900 Cu–Al alloy used in this work is listed in Table 1. Solution treatment of the alloy is performed under 850 °C for 1 h, and then quenched in water. The specimens were machined into Φ19 mm × 5 mm as substrate. Before the experiment, they were manually ground using silicon carbide papers from 240 to 2000 grade, then polished to mirror with diamond polishing agent. Finally, the substrated were degreased in acetone, ethanol and dried with hair dryer.

Deposition of Ti film was conducted by a closed field unbalanced magnetron sputtering ion plating system (Teer, UDP450/4) with high quality Ti target (99.999%). The base pressure of the chamber

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**Table 1**  
The composition of Cu–Al alloy C61900 (Wt.%).

Al	C	Si	Pb	P	S	Fe	Mn	Sn	Zn	Cu
8.95	0.01	0.06	0.02	0.05	0.06	3.87	0.35	0.09	0.08	Balance

is  $3 \times 10^{-5}$  Torr. Prior to deposition, substrates were etched by Ar plasma sputtering at a bias of  $-400$  V for 40 min. Then the substrate bias was changed to  $-70$  V, and the target power was set as 2.8 kW for deposition. The depositing time is 5 h, and the achieved thickness of Ti film on the substrate is about 6.3  $\mu\text{m}$ .

Plasma treatment was carried out in a cathodic cage plasma nitriding (CCPN) system, which consists of a plasma nitriding unit (LDMC-30, 30 kW) and an auxiliary heating device made of two layers stainless steel mesh. Specific illustration of the nitriding system could be found in our previous study [16]. Ti coated specimens were hanging in the center of the mesh, with the specimens and mesh being connected with cathode. Before the application of a glow discharge, the chamber was evacuated to be below 10 Pa. At the beginning of plasma treatment, only hydrogen gas with a flux of 0.1 L/min was imported to the CCPN system, and the temperature in the stainless steel mesh was raised to 650 °C, which was held for 2 h to prompt element diffusion between Ti film and the substrate before plasma nitriding. Then, the mixed gas of nitrogen (0.1 L/min) and hydrogen (0.1 L/min) was introduced, and the specimens were hold at 650 °C for 2 h for nitriding. Then, the specimens were cooled down slowly inside the vacuum furnace.

Surface morphology and elemental distributions of the fabricated coating and worn surface after tribo test were characterized by scanning electron microscope (SEM, FEI QUANTA 200 F), equipped with EDS. The phase compositions of the coating were determined using X-ray diffraction (XRD, D/max-rB) with  $\text{CuK}\alpha$  radiation in the range of 20–100 ° at 40 kV and 30 mA with 0.05° interval step. Three repeated indentations at the same depth into the surface were performed to measure the cross-sectional hardness a HV-1000 Vickers microhardness tester under a load of 10 g for 15 s. Tribological properties of the samples were evaluated using a ball-on-disk tribometer (POD-1) against GCr15 balls (5 mm in diameter). Unlubricated wear tests were conducted under a constant load of 4 N, sliding time of 1800 s and sliding speed of 0.1 m/s at the room temperature. The wear volume was calculated according to ASTM G99 by using [18]:

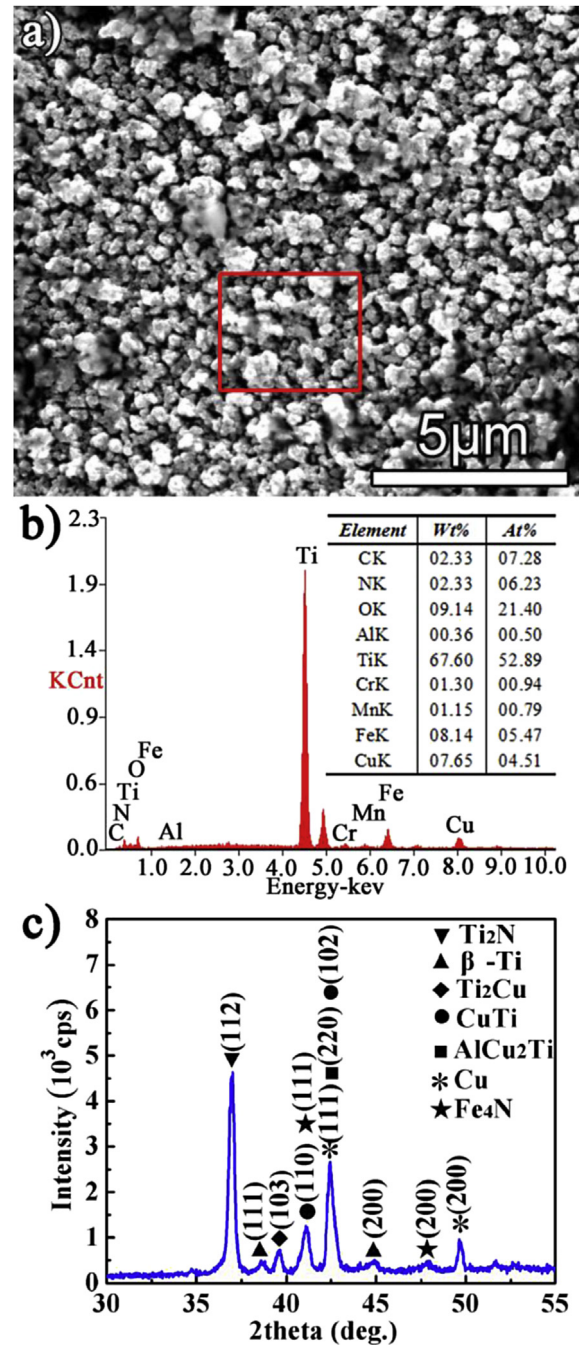
$$\Delta V = 2\pi R \left[ r^2 \arcsin(w/2r) - (w/4)\sqrt{4r^2 - w^2} \right] \quad (1)$$

where  $R$  (mm) and  $r$  (mm) is the radius of the wear track and GCr15 ball, respectively, and  $w$  (mm) is the average width of the worn track. Wear rate,  $\eta$ , was calculated by the following equation:

$$\eta = \Delta V / FS \quad (2)$$

where  $F$  (N) is the normal contact load and  $S$  (m) is the total sliding distance.

Fig. 1(a) shows the typical surface morphology of the fabricated multi-phases coating. It can be seen that the smooth surface with fine and uniform grains could be achieved by the combination of thermo-diffusing Ti film and plasma nitriding. The EDS result of the circled area in Fig. 1(a) indicates that a slight oxidation occurred attributed to the relative low degree vacuum (10 Pa). The Mn, Cr and part of Fe atoms came from the stainless steel net which worked as auxiliary heating device in the plasma nitriding unit [19], and the Cu and Al atoms should be caused by diffusion from the substrate. The XRD pattern of the coated surface, as is shown in Fig. 1(c), indicates that the Ti-coated specimen after plasma



**Fig. 1.** (a) Surface morphology, (b) Element composition, and (c) XRD spectra of the multi-phases coating.

nitriding consists of Ti–Cu–Al intermetallics ( $\text{CuTi}_2$ ,  $\text{CuTi}$ ,  $\text{AlCu}_2\text{Ti}$ ) and titanium nitride ( $\text{Ti}_2\text{N}$ ).

Fig. 2(a) shows the cross-sectional microstructure of C61900 Cu–Al alloy modified at 650 °C. It can be seen that a continuous and uniform coating was formed, the thickness of which is about 9  $\mu\text{m}$ . Gradual distribution of elements could be easily found in Fig. 2(b). During the stage of plasma treatment, the inter-diffusion between Ti film and Al–Cu substrate was stimulated to produce intermetallics ( $\text{CuTi}_2$ ,  $\text{CuTi}$ ,  $\text{AlCu}_2\text{Ti}$ ). Then, nitrogen was introduced to the furnace for nitriding treatment for 2 h, resulting in the formation of nitrides ( $\text{Ti}_2\text{N}$ ). The element distribution of the modified layer indicates that about 1  $\mu\text{m}$  thick  $\text{Ti}_2\text{N}$  formed at the outmost

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