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In-situ synthesis of the one-dimensional Ag wires reinforced composites film by a novel active screen plasma process: nanostructure and excellent adhesion resistance

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Abstract: A novel active screen (AS) plasma process is developed for the *in-situ* growth of one-dimensional Ag wires and the deposition of reinforced composite coating thereof. Silver wires embedded in the matrix were deposited with Ag nanoparticles, stainless steel nanoparticles and carbon during the AS plasma sputtering process in the ambient of methane and hydrogen. This provided a relatively low-temperature processing route for one-step synthesis of abrasion resistant coating on metallic substrate. Microstructure evolution during sliding with ductile materials proven the ultra-low friction deriving from the molecular ordering of carbon and the high-strength structural holding by the Ag wires.

Key words: composite coating; sputtering; silver wires; active screen; tribological.

1. Introduction

Novel composite coatings, which have unique structures and superior mechanical, thermal and electrical properties, have attracted a lot of interest due to their wide range of applications in the field of protection, catalytic, energy storage, and biomedical etc. Various deposition techniques have been developed to deposit coatings with novel architectures [1], which are commonly divided into the following types: multicomponent [2], multilayered [3], nanostructured [4], superlattice [5], gradient [6] and adaptive [7] structures. However, few works have been done on the 1D wires

reinforced composite coating structure, which may offer some unique properties refer to the conventional fibers reinforced bulk composite materials [8].

Carbon composite film has good anti-adhesive properties against sliding of adhesive materials. Due to the unique microstructure of wires reinforced composite coating, the toughness of the composite coating can be enhanced by suppress the fractures of matrix without detrimentally influence the overall anti-adhesive properties. It is known that aluminum alloy is a ductile material exhibiting an adhesion-prevalent wear behavior known to accelerate by abrasion under severe contacting stress. This causes high shear stress on the interface and possibly excessive metal transfer and damage on both counterparts. Thus, it is expected that the wires reinforced structure can provide a potential way to decrease the adhesion of ductile materials.

In this study, we firstly present a 1D Ag wires reinforced composite coating structure deposited by a novel active screen (AS) co-alloying process. The aim of the development is to produce composite coatings with tailored microstructures and controllable toughness and tribological properties. Therefore, tribological properties of the synthetic composite coating and its abrasion resistance to the ductile aluminium were explored with expectation to lower the energy dissipation of shear stress under severe wear conditions.

2. Experimental methods

The deposition was carried out in an adapted DC Klöckner Ionon 40 kVA plasma furnace. The schematic diagram of synthesis process is illustrated in Fig. 1. Samples were kept at a floating potential (i.e. zero bias) using ceramic insulators. A distance of 20 mm separated the samples and the stainless-steel (AS) lid. The lid and the silver plate were also spaced 6 mm apart to induce a hollow cathode effect. The coating was deposited on the substrates of AISI 316 SS grinded by the sandpaper (1200 grit). During deposition, low pressure plasma of 1mbar was formed at the cathode (worktable) using a gas mixture of 1.5% CH₄ and 98.5% H₂ and test pieces were heat to the target temperature in the plasma atmosphere. When the temperature reached 420°C, the pressure increased to 3mbar for the deposition of 20 hours.

Fig. 1. Synthesis of Ag wires reinforced composite coating by AS plasma deposition

A self-aligned conformal pin-on-disk CSM Tribometer was utilised to assess the tribological property of the composite coating. The composite coating was pushed against a flat-end pin made of aluminium. Severe adhesive wearing conditions of 5 kg heavy load and 500 cycles were applied to test the adhesion-abrasion resistance of the composite coating under dry condition.

3. Results and discussions

Morphologies of the Ag wire reinforced composite coating were analysed by SEM (JEOL 7000). Fig. 2 (a)&(b) shows the surface morphology of the Ag wires reinforced composite coating, and the Ag wires can be seen randomly embedded in the coating. The average length and diameter of Ag wires are around 5 μm and 500 nm. These Ag wires are relatively curled which are not as straight as the Ag wires grown by any solution based method [9]. Fig. 3 (c) shows the cross-section view of the composite coating with a thickness around 2 μm .

The crystallinities of the composite coating and the uncoated SS were detected by XRD which are shown in Fig. 2 (d). Typical textures (111), (200) and (220) of the γ -phase for the face-centered cubic (fcc) structured austenitic SS were detected. For the composite coating, the γ -phase patterns were also detected which verified that no reactions between SS nanoparticles and the carbon precursor were happened. Besides, strong textures of the pure Ag phase detected indicates that no Ag compounds were formed.

Fig. 2 (a)&(b) surface morphology of the composite coating; (c) cross-sectional view of the composite coating; (d) XRD results of SS and the composite coating.

It is known that Ag is a solid lubricant and hence could be used to reduce adhesion and friction. Therefore, reciprocating sliding friction tests have been carried out against an Al counterpart pin. Fig. 3(a) shows the friction coefficients of the Ag/C composite coating compared to that of uncoated SS substrate under dry sliding condition. After a couple of initial strokes, the friction coefficients of polished SS started to rise from 0.15 to 0.45 and then fluctuated largely throughout the test. This is a clear indication of stick-slip caused by the strong adhesion between SS and Al. In addition, the friction heat generated by the high friction would reduce the strength of aluminium alloys and hence increase adhesion further, thus leading to excessive materials transfer. This adhesion behaviour of ductile materials reflects the similar trends obtained from the previous friction coefficient analysis [1].

However, the interface friction coefficient was greatly reduced when the metal was coated with the Ag wires impregnated composite coating. The friction coefficient was 0.17 in the first 5 cycles and it slowly climbed to 0.25 after 25-50 strokes which are believed to be caused by the slow run-in between aluminium and the coating. Then, the friction coefficients of the composite coating against Al remained low throughout the test.

Fig. 3(c) shows the wear surface of the Ag wire reinforced composite coating, the original features of the silver wires and silver particles are still clearly visible and no transfer of aluminium onto the coating surface which is verified by the EDX results from the wear track shown in Table 1. Some horizontal scratches in the sliding direction appeared on the composite coating surface after 500 strokes of the reciprocating sliding test. Fig. 3(d)&(e) show the cross-section of the composite coating and the wear track area which were prepared by focused Ion beam (FIB), and the TEM sample was cut from the wear track area (Fig.3 (f)), which implies that Ag nanoparticles could aggregate to be large Ag particles to form Ag wires.

Fig. 3 (a) friction coefficient of Al pin against SS and Ag/C coating surfaces; (b) illustration of film growing and sampling orientations, (b)&(c) wear morphology of Ag/C coating with scratches and plastic deformed Ag wires; cross-sectional views of (d) as-treated coating and (e) wear track, (f) TEM image showing cross-section of Ag wire reinforced composite coating.

Table 1 Elemental distribution on the surface of frictional counterparts

	C	O	Mg	Al	Si	Cr	Fe	Ni	Ag
Al pin	8.23	3.06	0.53	84.02	3.66	0.00	0.51	-	0.00
Al wear area	43.87	4.94	0.35	44.53	1.73	0.63	2.84	-	1.11
Ag/C	23.56	1.87	-	0.00	0.25	8.61	26.29	3.57	34.78
Wear track	17.14	3.02	-	0.00	0.25	8.69	26.94	3.44	39.73

Beside the Ag wires, Ag nanoparticles were also distributed in the composite coating as matrix which was verified by the TEM results. The Ag nanoparticles and the carbon phase are benefitting to form a self-lubricant layer for friction reduction [10, 11]. According to the previous results and discussion, aluminium is very ductile and adhesive to the metallic surface under these conditions [12]. However, as shown by the

abrasion test, the deposited Ag/C coating has low shear tension when sliding with aluminium. The adhesion-free sliding of ductile aluminium on the Ag/C coating is opposite to the adhesive feature of soft aluminium to steels and cast iron in most studies [2]. Since the conformal sliding test was carried out using the same alloy and conformal configurations of pin-on-disc, the outstanding adhesion resistance must attribute to the Ag/C coating. Together with the finding of a small amount of C, Ag, Fe on the Al pin, it is assumed that the improvement of tribological performance could be attributed to the consumption of carbon and formation of barrier layers between the Al and coating. This also occurs with the carbon transformation from disordered- to ordered- graphite as evidenced by the Raman analysis of I_D/I_G ratio before and after sliding (1.13, 0.76). Although the total amount of Ag does not vary significantly, the imbedded Ag wires act as strengthen phase and prevent the coating from fast worn-out under sliding high-stress. Two dry-sliding surfaces were separated by the Ag and C composite solid lubricants thus this affects the coating's metal transfer resistance to a great extent.

5. Conclusions

In summary, a novel Ag wires reinforced composite coating has been developed through the ASPC approach. The deposited Ag/C functional coating has been shown great tribological property especially in preventing the adhesion of ductile aluminium alloy. Due to the low interface energy between silver/metals interface and carbon/metals interface, this composite coating can be promising in providing superlubricity with

exceptionally low energy dissipation for high-performance and long-life machine operations in industrial applications.

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