



# High temperatures tribological properties of Ta-Ag films deposited at various working pressures and sputtering powers

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## ARTICLE INFO

### Keywords:

Tantalum  
Magnetron sputtering  
Coating  
High temperature  
Friction and wear

## ABSTRACT

The tribological performances of Ta-Ag films are greatly influenced by their composition and depositing parameters. In this study, Ta-Ag films were deposited on carbon steel substrate by DC magnetron sputtering. To adjust microstructures of the films, the sputtering pressures were controlled between 0.2 Pa and 0.6 Pa, and the sputtering powers of silver target were varied from 10 W to 25 W. The result was shown that the  $\alpha$ -Ta phase (bcc) was obtained at lower pressure and higher sputtering power of Ag. And the contents of Ag in the films increase as the sputtering power of Ag increases. The high silver contents in the deposited films at 0.4 Pa (24.66 wt% and 31.10 wt% for Ta-Ag15 and Ta-Ag25 films, respectively) contribute to a low friction at RT, while the tribo-chemical reaction products on the frictional surface are responsible for the friction reduction at high temperatures. Low friction coefficient ( $\sim 0.2$ ) was presented for the Ta-Ag25 film when rubbing against Si<sub>3</sub>N<sub>4</sub> ball at RT, 200 °C and 700 °C.

## 1. Introduction

Protective coatings are of great use under severe environmental conditions, such as elevated temperatures, high working stress and controlled atmospheres. They have got considerable interest in versatile industrial applications. Besides, the protective coatings used for sliding conditions such as barrel or railgun are required to possess self-lubrication properties. Recently, tantalum films have been extensively studied owing to their high melting point, corrosion resistance, excellent ductility and strength at high temperatures [1–5].

The structure of the deposited Ta coating is sensitively influenced by the sputtering power of target, working pressure, and the base pressure [6,7]. Considerable efforts have been dedicated to the development of the depositing methods of tantalum thin films [8–11]. The phase transition between  $\alpha$ -Ta and  $\beta$ -Ta was controlled by adjusting the sputtering power, pressure, bias voltage, substrate temperature and substrate materials [12–16]. Otherwise, an  $\alpha$ -Ta coating with good corrosion resistance was grown on the  $\gamma$ -TiAl substrate by double-glow plasma surface metallurgy technique [17].

Besides the processing parameters, the structure of Ta can also be monitored by doping with other alloy or non-metal elements [18]. The Ta-Ag immiscible alloy films are developed as the protective coatings under the sliding condition and their tribological properties are concerned. Due to the fact that the supersaturated solid solution of Ta-Ag film is metastable, phase separation may occur in the immiscible alloy

driven by heat or plastic deformation [19]. Then the variation in composition on the frictional surface of Ta-Ag films leads to complex tribological behaviors.

At present, the tribological properties of hard coating at high temperature lubricated by AgTM<sub>x</sub>O<sub>y</sub> has become a research hotspot [20]. Stone et al. [21] tested the AgTaO<sub>3</sub> coating at 750 °C under different load. The AgTaO<sub>3</sub> coatings displayed a low CoF from 0.04 to 0.15. TaN-Ag coating [22] showed lower friction coefficient at 750 °C due to generation of AgTaO<sub>3</sub>, Ta<sub>2</sub>O<sub>5</sub>, and Ag. The tribo-chemical reactions with the oxygen in the environment may be involved in the tribological properties of tantalum films.

In this study, the Ta-Ag immiscible alloy films were deposited on steel substrates by DC magnetron sputtering. The microstructures of the Ta films were adjusted by incorporating the silver elements. The effects of sputtering pressure and power of Ag on the microstructure, mechanical and tribological properties of the Ta-Ag coatings were investigated.

## 2. Experimental method

### 2.1. Films preparation

Ta-Ag coatings were co-deposited on the 1045 carbon steel disc with the diameter of 40 mm and the thickness of 5 mm. The surfaces of mild carbon steel substrates were polished with 200, 400, 600 and 800-grit

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**Table 1**  
Sputtering deposition parameters for Ta-Ag film and their chemical composition.

| Sample | Target power Ta (W) | Target power Ag (W) | Working pressure (Pa) | Composition (wt%) |       |      |      |
|--------|---------------------|---------------------|-----------------------|-------------------|-------|------|------|
|        |                     |                     |                       | Ta                | Ag    | O    | Fe   |
| 1      | 100                 | 10                  | 0.2                   | 96.77             | 3.23  |      |      |
| 2      | 100                 | 10                  | 0.4                   | 87.05             | 8.46  | 3.67 | 0.82 |
| 3      | 100                 | 10                  | 0.6                   | 86.67             | 13.33 |      |      |
| 4      | 100                 | 15                  | 0.2                   | 87.38             | 12.62 |      |      |
| 5      | 100                 | 15                  | 0.4                   | 75.13             | 24.66 |      | 0.22 |
| 6      | 100                 | 15                  | 0.6                   | 84.01             | 15.99 |      |      |
| 7      | 100                 | 20                  | 0.2                   | 73.48             | 26.52 |      |      |
| 8      | 100                 | 20                  | 0.4                   | 62.00             | 31.31 | 6.35 | 0.33 |
| 9      | 100                 | 20                  | 0.6                   | 69.69             | 30.31 |      |      |
| 10     | 100                 | 25                  | 0.2                   | 71.57             | 28.43 |      | 0.54 |
| 11     | 100                 | 25                  | 0.4                   | 62.98             | 31.10 | 5.60 | 0.33 |
| 12     | 100                 | 25                  | 0.6                   | 71.12             | 28.06 |      | 0.82 |

SiC paper. The depositions of coatings were carried out by using a JGP-450 Magnetron Sputtering System. The vacuum chamber was draw to a pressure of below  $5.5 \times 10^{-3}$  Pa. Ta (99.95% purity) and Ag (99.97%

purity) plates with the diameter of 60 mm were used as sputtering targets.

In order to obtain the  $\alpha$ -phase Ta, the sputtering power of 100 W was selected for Ta, and sputtering power of Ag was varied from 10 W to 25 W to control the composition of silver in the Ta-Ag films. The target-to-substrate distance was kept at 110 mm during sputtering. All the sputtering parameters are shown in Table 1. The pure Ar gas (99.999%) was used for protecting atmosphere with the pressure of 0.2, 0.4 and 0.6 Pa (shown in Table 1) to identify the influence of working pressure on the structure of Ta-Ag films. All depositions were carried out within constant period of 90 min. In situ measurements with thermocouple yielded a substrate temperature of below 100 °C at a plasma power of 100 W.

2.2. Performance and characterization

The structures and morphologies of the films were detected by X-ray diffraction (XRD), scanning electron microscopy (SEM) and energy dispersive X-ray analysis (EDS). The XRD patterns were recorded on X-ray diffraction (Bruker D8 X-ray facility) using Cu  $K_{\alpha}$  radiation ( $\lambda = 0.154$  nm), which was carried out at a voltage of 40 kV and a current of 40 mA. The scanning angle ranged from 20° to 80° at a scanning speed of 10°/min with a 0.02° step size. The surfaces and

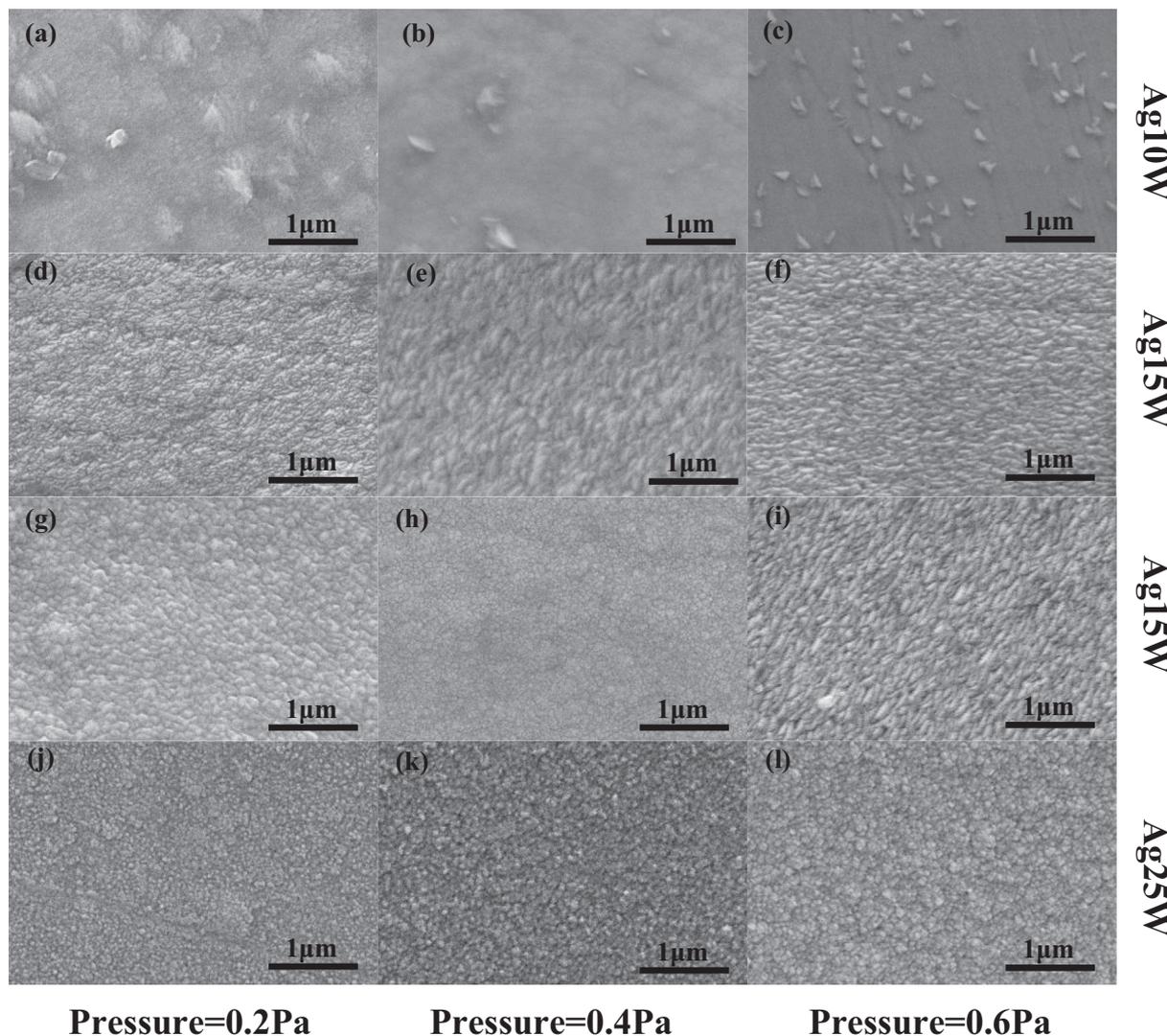


Fig. 1. Top-view SEM micrographs of Ta-Ag coatings with different working pressures and sputtering powers of silver.

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