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## Original Research

## Nanoindentation response analysis of TiN-Cu coating deposited by magnetron sputtering

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## ABSTRACT

To investigate the change of the mechanical properties of soft metals doped PVD (Physical Vapor Deposition) coatings after the migration of soft metal to the surface, TiN-Cu coating was deposited on Si (100) by magnetron sputtering. The microstructure and mechanical properties at room temperature and after vacuum heat treatment at 300 °C were investigated. The results showed that the grains were clustered and the microstructure was porous for TiN-Cu coating at room temperature, while many micro- and nano- sized Cu particles were observed on the surface after vacuum heat treatment at 300 °C. The elastic properties of the TiN-Cu coating after vacuum heat treatment at 300 °C degraded compared with that at room temperature. The hardness and elasticity modulus of TiN-Cu coating kept constant (3.7 GPa and 125.0 GPa, respectively) with the increase of nano-indentation depth, while the hardness and elasticity modulus of TiN-Cu coating after vacuum heat treatment at 300 °C increased gradually.

## 1. Introduction

In the last decades, PVD-TiN coatings have attracted increasing attention due to the superior properties, such as high hardness, good thermal stability and excellent tribological properties [1–4]. Recently, self-lubricating PVD coatings have been developed to improve the tribological performance in a wide temperature range [5–12]. These coatings usually consist of hard matrix and solid lubricant phases, and low friction at different temperatures could be obtained with the intelligent adjustment effect of the solid lubricant phases. Among them, the soft metals such as Ag or Cu are the effective and promising solid lubricant phases for lubrication in a wide temperature range. Aouadi et al. [9] prepared VN/Ag nanocomposite coatings and investigated the friction property from 25 to 1000 °C. They found that a low friction coefficient (0.15–0.35) was obtained in this temperature range. Yu et al. [10] deposited W<sub>2</sub>N-Cu films and studied the tribological properties from 25 to 600 °C. Their results showed that the friction coefficient varied from 0.3 to 0.4 in the test temperature range. More recently, Bondarev et al. [12] prepared TiNbCN-Ag coatings and investigated the wide-temperature tribological behavior. They found that the friction coefficient was below 0.45 in the temperature range of 25–700 °C.

As reported by these above-mentioned studies, it can be found that the reduced friction coefficients of the coatings containing soft metal in a wide temperature range were attributed to the migration of soft metal

to the coating surface. The change of the element distribution would also result in the change of mechanical properties. However, up to now, few studies have been done to investigate the mechanical properties of the coatings containing soft metal phases after the migration. Nanoindentation has been widely used to explore the nano-mechanical properties of coatings [13], due to its high sensitivity of obtaining the mechanical properties on a small scale. In this work, TiN-Cu coating was deposited on Si substrate by magnetron sputtering. To better understand how the mechanical properties change after the migration of soft Cu to the surface, the microstructure and nano-mechanical properties of TiN-Cu coating at room temperature and after vacuum heat treatment at 300 °C were investigated.

## 2. Experimental details

TiN-Cu coating was deposited on Si (100) substrate by magnetron sputtering (MSP-300B) in N<sub>2</sub>-Ar mixed atmosphere. The Si substrate was ultrasonically cleaned in alcohol for 10 min. The power of Ti target and Cu target was 150 W and 30 W, respectively. The flow rate of N<sub>2</sub> and Ar was 5 sccm and 10 sccm, respectively. The deposition time was 2.5 h. The heat treatment was conducted in a vacuum furnace (CHY-1200) at 300 °C for 0.5 h with a heating rate of 10 °C/min, and then the samples were cooled down to room temperature in the furnace.

The composition of TiN-Cu coating was measured by Energy

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Dispersive Spectrometer (EDS-EDAX, Genesis XM2). And the crystalline structure of the TiN-Cu coating was measured by X-ray diffraction (XRD-BRUKER, D8 Advanced) equipped with a Cu K $\alpha$  source, working

at 40 kV and 40 mA. The morphologies of the cross section and top-view of the TiN-Cu coating were observed by field emission scanning electron microscopy (SEM, Hitachi S-4800). The surface roughness of

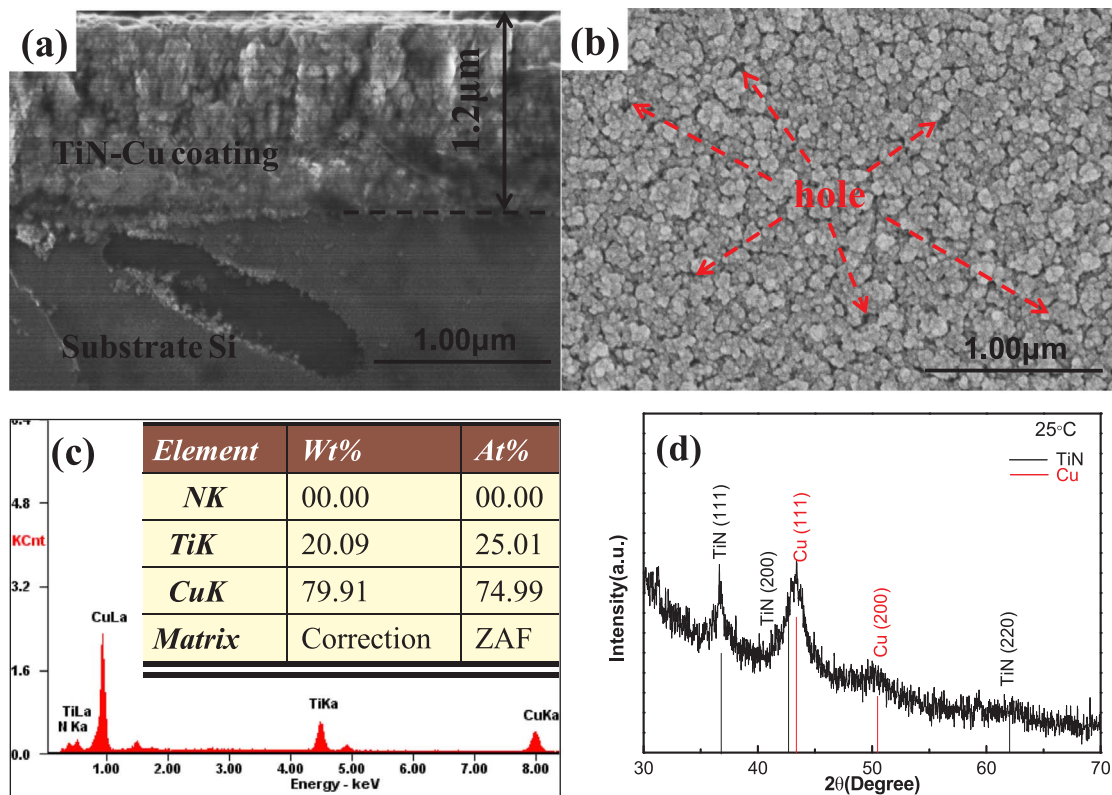


Fig. 1. Cross-sectional, top-view SEM micrographs (a, b), EDS (c) and XRD pattern (d) of TiN-Cu coating at room temperature.

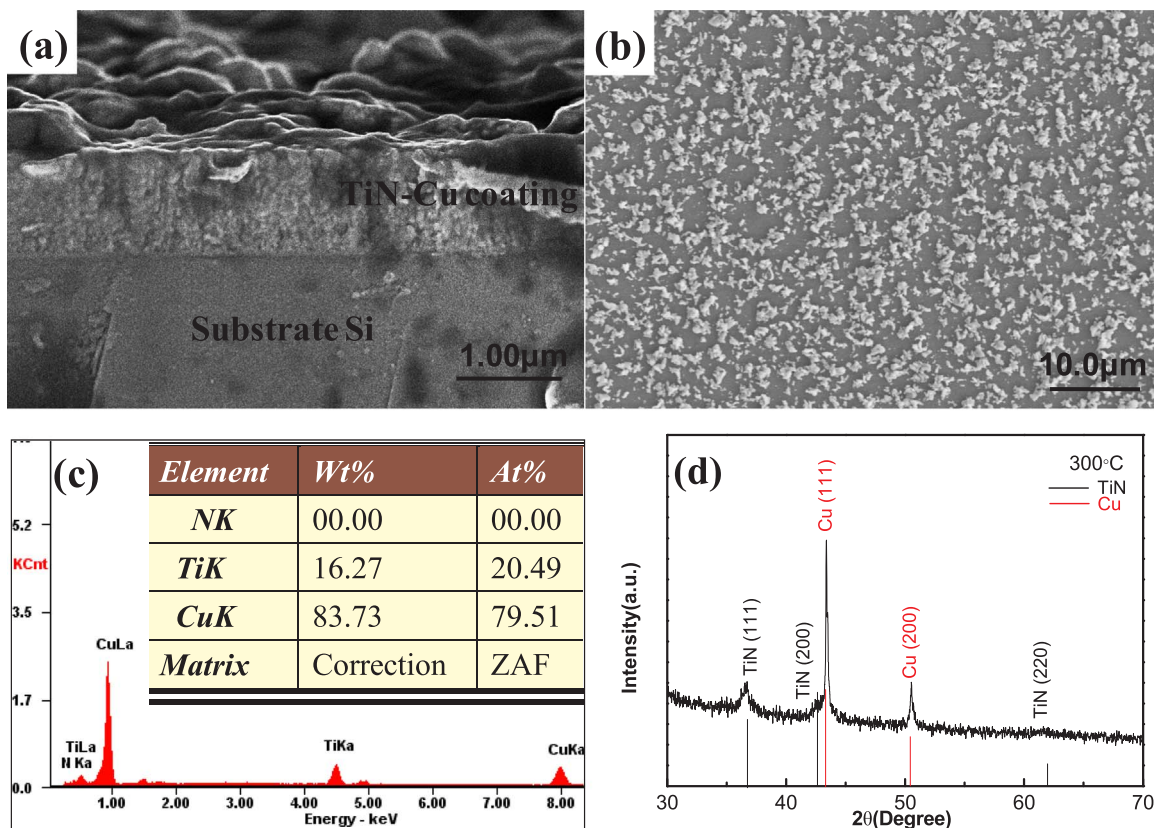


Fig. 2. Cross-sectional, top-view SEM micrographs (a, b), EDS (c) and XRD pattern (d) of TiN-Cu coating after vacuum heat treatment at 300 °C.

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