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CrVN/TiN nanoscale multilayer coatings deposited by DC unbalanced magnetron sputtering

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ABSTRACT

The growing interest in development coatings with enhanced properties opened the door to the research and development of nanoscale multilayer coatings. Some of the most interesting properties of these multilayer coatings when compared to monolayers are hardness higher than 40 GPa, friction coefficients < 0.3, improved adhesion and increased heat and corrosion resistance.

In this work, CrVN/TiN nanoscale multilayer coatings were deposited onto H13 steel substrates by DC unbalanced magnetron sputtering. For the deposition of the multilayer coatings, two targets of Cr-V (70–30 wt%) and Ti (99.5 wt%) confronted opposite each other were used. In order to vary the bilayer period, a microcontroller was used to control the rotation and residence time of the substrates in front each target, thereby obtaining multilayer periods of 15 nm, 30 nm and 43 nm. CrVN and TiN monolayer coatings were also deposited.

X-ray diffraction (XRD) showed a FCC crystal structure for all coatings. SEM images revealed a columnar structure for CrVN and a cauliflower-like structure for TiN, similar to that obtained for the multilayer coating. All coatings showed a Hardness and Young's modulus around 19 GPa and 270 GPa, respectively. The CrVN/TiN multilayer with a period of 15 nm showed the lowest friction coefficient (0.31) and the lowest wear rate $(2.23 \times 10^{-9} \text{ kg/Nm})$ of all coatings, representing a reduction of 36% compared to the friction coefficient of H13 steel and a reduction in the wear rate of three orders of magnitude compared to the TiN. Finally, it was possible to identify three stages in the wear mechanism in the multilayer coatings, the lowest period, molecule-to-molecule adhesive wear occurred, followed by a rupture in the Ti–N and Cr–N bonds when the period increased to 30 nm, while with the highest period of 43 nm, oxides of titanium, chromium and vanadium were formed.

1. Introduction

Modern industrial requirements create significant challenges for protective coatings. Hard coatings, with good tribological performance, high thermal stability and resistance to oxidation and corrosion are needed for different applications in various areas of engineering.

TiN protective coatings have been widely used in industrial applications because of their high hardness, satisfactory tribological behavior and good adherence to most substrates [1]. CrN coatings have lower hardness but better resistance to corrosion than TiN coatings, and have also been of great use in manufacturing applications. The combination of these two coatings to obtain multilayer TiN/CrN coatings at nanometric scale has led to an improvement of up to 250% in mechanical and tribological properties when compared to monolayer coatings [2–4]. The use of multilayer coatings reduces residual stress to a large extent and inhibits the proliferation of cracks across the coating. In addition, multilayer coatings have a reduced grain size. Zhou et al. link the increased hardness and endurance of multilayer coatings to Keohler Hall-Petch effects [5], whereby the enhancement of these properties is attributed to the increased volumetric density resulting from refined grain size, which increases the resistance to dislocation movements [6,7].

The incorporation of elements such as carbon, silicon and vanadium has made it possible to obtain ternary coatings with superior performance to binary coatings. In particular, the self-lubricating properties of coatings containing vanadium have proven to be of great interest [8,9]. Authors such as B. Deng et al. report a striking reduction from 0.6. to 0.25 in the friction coefficient of TiN coatings implanted with vanadium ions. Equally, the wear rate of TiN coatings decreases by an order of magnitude when they are implanted with densities of

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4×10^{21} vanadium ions/m² [10].

To date, no evidence of CrVN/TiN coatings with modulation periods in the nanometric order has been reported in the literature. The use of monolayer hard coatings is becoming less and less because multilayer coatings exhibited better mechanical and tribological properties than those of their individual constituents. Based on the above, this work focuses on the development and study of nanometric CrVN/TiN coatings that combine the known properties of these two alloys in order to obtain coatings with improved mechanical and tribological properties for potential use in different industrial applications.

2. Experimental procedure

2.1. Coating deposition

AISI H13 steel substrates of 1.6 cm diameter and 0.3 cm thickness were used for the deposition of the coatings. The substrates were subjected to a heat treatment cycle consisting of an austenitization at 1298 °K, quenching in oil and tempering at 753 °K to obtain an average hardness of 60 HRC. The steel samples were polished using SiC emery paper with a grit size of 300, 600, 1000, 1500 and 2000, and subsequently polished with diamond paste to a mirror finish until an average roughness of Ra = 50 nm was obtained. Thereafter, the substrates underwent ultrasonic cleaning in a solution of Ethanol:Acetone of 3:1 for 0.25 h. Afterward, monolayer coatings of CrVN and TiN and multilayer of CrVN/TiN were deposited by unbalanced magnetron sputtering deposition technique. This was performed in a self-built vacuum chamber with dimensions of 0.55 m \times 0.6 m \times 0.8 m.

Prior to the procedure, the vacuum chamber was evacuated to a background pressure below 10^{-3} Pa. After this, cleaning was performed by ionic bombardment of the targets using an argon flow rate of 40 sccm, pressure of 3 Pa, temperature of 523 °K, power of 0.7 KW and 0.3 KW applied to the CrV and Ti targets, respectively.

Once the cleaning of the targets was completed, the substrates were cleaned in the same way, with a bias voltage of -700 V applied to the substrates. The monolayer and multilayer coatings were both carried out in an atmosphere of 13.5% nitrogen (7 sccm) as a reactive gas and argon (45 sccm) as working gas. A CrV (70–30 wt%) target with 99.9 wt % of purity and Ti target with 99.5 wt% of purity were used, with dimensions of 0.5 m \times 0.1 m \times 0.01 m. The distance between the substrate and the targets was of 8 cm, while the distance between both targets was approximately 65 cm. The coatings were deposited at a temperature of 523 °K, pressure of 0.45 Pa, bias voltage of -70 V and power of 1.2 KW. Fig. 1a shows the schematic representation of the substrates was the in front of Ti target, therefore the first layer deposited was TiN and the last one was CrVN in all the multilayer

structures.

An ARDUINO UNO system (Fig. 1b) was incorporated, that made it possible to program and control the time spent by the substrates in front of each of the targets by rotating them, thereby achieving the deposition of the multilayer coatings with controlled period thickness.

The deposition rates of CrVN and TiN during the development of the monolayer coatings were 44 nm/min and 22 nm/min respectively. Three deposition times were selected (15, 30 and 40 s) to obtain approximate bilayer periods of 15, 30 and 43 nm in accordance with the deposition rates previously determined. Based on the previous data, the aim was to obtain 125, 61 and 40 bilayer coatings in each of the evaluation times, with a total thickness of each multilayer coatings of 1800 nm approximately.

2.2. Characterization of the coatings

The morphology and microstructure of the coatings were studied using a JEOL JSM-6490LV Scanning Electron Microscope. The thickness was determined using a BRUKER DEKTAK XT model profilometer. The grain size and roughness of the coatings were determined using an AFM MFP-3D Infinity (Asylum Research) machine in AC Mode. The tips used were Silicon-Al coated (AC240TS-R3). The nominal spring constant was 2.5 N/m and the resonance frequency was 73.52 kHz. The crystallinity of the coatings was evaluated using an EMPYREAN diffractometer in θ -2 θ mode with a Cu anode; wavelength of $\lambda = 0.1540598$ nm, scan range of 20° to 65° and 0.02° step, at 45 kV and 40 mA.

X'PERT HIGH SCORE ANALYSIS software and XRD diffractograms of the multilayers were used to calculate the Full Width at Half Maximum (FWHM) of the peaks (200), applying a Gaussian adjustment. After this the crystallite size was determined through the Scherrer equation. The measurement values of the FWHM of the diffraction (200) were 1.21293°, 1.0411° and 0.9242° for the $\Lambda = 15$ nm, $\Lambda = 30$ nm, $\Lambda = 43$ nm coatings respectively. The Scherrer equation relates the FWHM and the crystallite size in accordance with Eq. (1) [11]:

$$\beta = \frac{(k).(\lambda)}{(FWHM).(Cos\theta)}$$
(1)

Where β is the crystallite size; *k* is the crystal form factor, the value of which can be between 0.9 and 1, λ is the wavelength of the radiation used; FWHM is the width at the average height of the diffraction peak of the sample and θ is the angle of the diffraction peak.

A HORIBA JOBIN YVON (Labram HR model) high-resolution confocal Raman micro-spectroscope with a helium-neon laser with 633 nm wavelength and 17 mW power was used to evaluate the chemical composition of the coatings. The tribological properties of the coatings were determined with a ball-on-disc type tribometer, using as counter



Fig. 1. Schematic representation of a) configuration of deposited multilayer coatings and b) arrangement of targets and substrates in the sputtering chamber.

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