

Effect of layer thickness on thermal properties of multilayer thin films produced by PVD

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ABSTRACT

Cr/CrN/CrAlN, CrN/CrAlN and Cr/CrN thin layers were deposited by PVD (Physical Vapor Deposition). The multilayers were obtained from the combined deposition of different layers Cr, CrN and CrAlN thick films on on AISI4140 steel and silicon substrates at 200 °C, and evaluated with respect to fundamental properties such as structure and thermal properties. Cr, CrN and CrAlN single layers were also prepared for comparison purposes. The structural and morphological properties of PVD layers were characterized by X-ray diffraction (XRD) and scanning electron microscopy (SEM) coupled with EDS + WDS micro-analyses, stresses were determined by the Newton's rings methods using the Stoney's equation and surface hardening and hardness profiles were evaluated by micro hardness measurements. The XRD data and HRTEM showed that both the Cr/CrN, CrN/CrAlN and Cr/CrN/CrAlN multilayer coatings exhibited B1NaCl structure with a prominent reflection along (200) plane, and CrAlN sub-layer microstructures composed of nanocrystallites uniformly embedded in an amorphous matrix. The innovation of this work was to use the thickness of three different coating types to determine the thermal properties. Furthermore, an empirical equation was developed for the thermal properties variations with temperature of AISI4140 steel coated with different multilayer coatings. The thermal conductivity of CrAlN single layered was lower than the multilayer and the bulk material AISI4140. Moreover, the influences of structure and composition of the multilayer coatings on the thermal properties are discussed.

The thermal conductivity of nanoscale thin film is remarkably lower than that of bulk materials because of its various size effects.

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1. Introduction

Thin films based on carbides and nitrides of transition metals have been very successful in improving the performance of cutting tools. Although TiN is the most widely used in industrial applications, CrN has been gaining importance in recent years due to its superior corrosion and wear resistance, friction behavior and low internal stress [1–3]. However, there are an increasing number of applications where properties of single layer coatings are not sufficient. Recent research is focused on the development of nanolayered multilayer coatings in order to meet the requirements in terms of hardness, thermal stability, coefficient of friction, corrosion resistance and internal stress [4–11]. Nanolayered

multilayer coatings are composed of two or three different layers at the nanometer scale. The bilayers of the multilayers can be metals, carbides, nitrides or oxides of different materials or a combination of one layer made of nitride, carbide or oxide of one metal and the second layer made of another metal. A variety of multilayer systems such as TiN/CrN [4,5], TiAlN/CrN [6,7], TiN/NbN [8–11], etc., have been studied extensively. However, there are very few reports on the multilayer coatings based on CrN and CrAlN [12]. CrN films have been used for cutting tools, molding dyes and machine parts. But the oxidation resistance of CrN is limited up to 800 °C [13]. For hard protective coatings, thermal stability is a main property as they are exposed to high temperatures during the cutting process. The addition of Al to CrN system permits to work at higher temperatures where the oxidation occurs [14]. CrAlN coatings have been reported to be stable up to a temperature of 900 °C depending upon the Al content in the coatings [15]. CrAlN coatings also exhibit higher hardness and a lower friction coefficient compared to CrN coatings [16,17]. The structural and thermal properties have been

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studied by X-ray diffraction (XRD), photo-thermal deflection (PTD) technique [18–20].

2. Experimental details

The substrate material used in the present study was commercial AISI 4140 steel with the composition presented in Table 1. The substrates were hardened and tempered to a hardness of 4.5 HV_{0.05}. They were ground, polished and cleaned with trichloroethylene, acetone and alcohol in an ultrasonic cleaner.

2.1. Coatings deposition and characterizations

The multilayers were deposited on mechanically polished steel (AISI4140), with a surface roughness $R_a = 0.2 \mu\text{m}$ and silicon $10 \times 10 \text{ mm}^2$ (for internal stress and thickness measurements) SiO_2 $10 \times 10 \text{ mm}^2$ substrates by using a dual RF magnetron sputtering system (NORDIKO type 3500-13, 56 MHz) equipped with two targets of high purity (Cr of 99.995% and Al of 99.999%) as shown in Fig. 1, the working pressure is 0.4 μbar . Before deposition, the substrates and the targets were ultrasonically cleaned and etched in pure argon plasma for 5 min. The deposition conditions such as target power, bias voltage, and deposition time are given in Table 2. The deposition temperature was around 200 °C. Very high purity nitrogen (N_2 of 99.9999%) was introduced into the vacuum chamber as the reactive gas. The residual pressure was 10^{-7} mbar. In both cases, the targets were 101.6 mm of diameter and 3 mm thick. The substrate/target distance was 80 mm.

The Cr/CrN or Cr/CrN/CrAlN multilayers can be defined as graded coatings. Actually, the CrN or CrAlN layers were achieved after decreasing the argon concentration inside the deposition chamber from 100% to 80%, and increasing the nitrogen content to 20%.

The morphology, thickness and the composition of the coatings were determined by Scanning Electron Microscopic observations (SEM) and EDS + WDS microanalyses (Jeol JSM-5900 LV). XRD analyses (SIEMENS D500, $\text{Co-K}(\text{radiation})$) permitted the determination of the crystalline orientations of the layers.

The residual stresses (σ) were determined by interferometer (Newton's rings method [21]) by the measurement of the curvature radius (R_0 , R) of the Si (100) specimens before and after deposition and calculated with the Stoney's equation [21,22]:

$$\sigma = \frac{1}{6} \left(\frac{E_s}{(1 - \nu_s)} \right) \left(\frac{t_s^2}{t_f} \right) \left(\frac{1}{R} - \frac{1}{R_0} \right) \quad (1)$$

where E_s and ν_s are Young's modulus (181 GPa) and Poisson's ratio (0.28) of silicon (100), respectively [17]. The $(E_s/(1 - \nu_s))$ term is the biaxial modulus of the Si (100) substrate ($E = 1.805 \times 10^{11} \text{ N/mm}^2$). Both t_s and t_f are the thicknesses of the Si substrate and of the layer, respectively, while R_0 and R are the curvature radius before and after deposition, respectively.

2.2. Photo-thermal deflection (PTD)

The thermal properties are determined by photo-thermal deflection (PTD) [23,24] which is a non-destructive technique. In

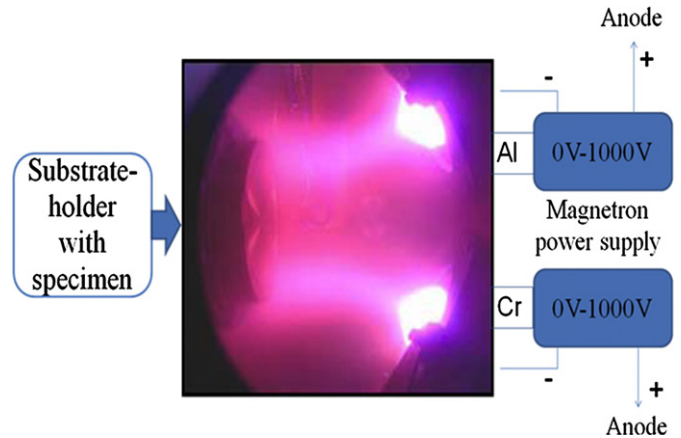


Fig. 1. Plasmas operated in the magnetron.

this work we used this technique in order to determine the local thermal properties of a stacking formed with succession of thin layers described above (Section 2.1). We proceeded by determining the thermal conductivity and thermal diffusivity of a single layer of Cr deposited on the substrate then, thermal properties of the bilayers of Cr/CrN and multilayer Cr/CrN/CrAlN are determined using a mathematical model and an appropriate experimental setup.

2.2.1. Experimental setup

The PTD setup developed in our laboratory as shown in Fig. 2. It has been applied here to study the photo-thermal signal; the sample is heated by a light from a 100 W power halogen lamp with power modulated thanks to a mechanical chopper at a variable frequency. A He–Ne laser probe beam of diameter $d = 100 \mu\text{m}$, skimming the sample surface at a distance z , is deflected. This deflection can be detected by a four quadrant photo-detector and converted into an electrical signal which is measured by a lock-in amplifier (EG&G5210). Through the intermediary of the interfaces of the mechanical chopper and the Look-in amplifier, a microcomputer will set the desired modulation frequency and read the values of the amplitude and phase of the photo-thermal signal and then draw their variations according to the square root modulation frequency.

2.2.2. Theoretical model

The PTD technique consists in heating an absorbing sample using a modulated light pump beam. The optical absorption of the sample will generate a thermal wave that will propagate into the sample and in the surrounding fluid medium, inducing a temperature gradient, and as a consequence, a refractive index gradient in the fluid. A laser probe beam skimming the sample surface and crossing the region with inhomogeneous refractive index gradient is deflected. Its deflection ψ may be related to the thermal properties of the sample and to the temperature T_0 at the sample surface.

Table 2
Deposition conditions.

Coatings	Time of deposition (min)	Bias voltages (-V)		Target power (KW)	Gas mixture (4 μbar)	
		Al	Cr		Ar (%)	N_2 (%)
Cr	60	0	-900	4	100	0
CrN	60	0	0	//	80	20
CrAlN	60	-300	-900	//	//	//
Cr/CrN	51	//	//	//	//	//
CrN/CrAlN	105	//	//	//	//	//
Cr/CrN/CrAlN	99	//	//	//	//	//

Table 1
Chemical composition of AISI4140 steel.

C	0.410
Mn	0.770
Si	0.280
S	0.026
Cr	1.020
P	0.019
Mo	0.160

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