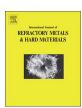
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## International Journal of Refractory Metals & Hard Materials

journal homepage: www.elsevier.com/locate/IJRMHM



# Study on the adhesion and tribological behavior of PVD TiAlN coatings with a multi-scale textured substrate surface



Kedong Zhang<sup>a</sup>, Jianxin Deng<sup>b,\*</sup>, Xuhong Guo<sup>a</sup>, Lining Sun<sup>a</sup>, Shuting Lei<sup>c</sup>

- a Research Institute of Advanced Manufacturing Technology, School of Mechanical and Electric Engineering, Soochow University, Suzhou 215021, PR China
- b Department of Mechanical Engineering, Key Laboratory of High Efficiency and Clean Mechanical Manufacture, Shandong University, Ministry of Education, PR China
- <sup>c</sup> Department of Industrial and Manufacturing Systems Engineering, Kansas State University, Manhattan, KS 66506, United States

#### ARTICLE INFO

Keywords: Multi-scale textures TiAlN coatings Adhesive strength Tribological properties

#### ABSTRACT

In this paper, we investigated the effects of multi-scale textures on improving the adhesion of PVD TiAlN coating-carbide substrate composites, which is crucial for tribological applications. The substrate pre-treatments were conducted using a nanosecond laser and a femtosecond laser to produce three types of surface textures: (i) micro-scale textures, (ii) nano-scale textures and (iii) micro/nano-scale textures. Characterization tests such as variable depth scratch tests and ball-on-disk wear tests were performed to evaluate the coating adhesiveness as well as the tribological behavior of the PVD TiAlN coatings deposited on the multi-scale textured substrates. The results revealed that the adhesion of three textured TiAlN coatings was greatly improved over that of the untextured one and that anti-adhesive wear properties of TiAlN coatings against AISI 316 stainless steel were significantly improved by using laser substrate pre-treatment. Moreover, the type of surface textures had a profound effect on the adhesive strength and tribological properties of the pre-treated samples. In the experiment, the micro/nano-scale texture was the most effective in providing a mechanical locking of the coatings on the substrate, a matching chemical property between substrate and coating materials, and a high active substrate surface, thus increasing the critical load of the coatings.

#### 1. Introduction

In recent decades, surface coatings fabricated by physical vapor deposition (PVD) have been extensively dedicated to improving the durability of various kinds of engineering materials and are now widely utilized in numerous technical fields [1]. In particular, TiAlN coated cemented carbide has long been a subject of interest due to its high hardness, excellent abrasion resistance, and high thermal stability. This enables PVD TiAlN coatings to be a material with a broad range of applications, especially as cutting tools for machining difficult-to-machine materials under harsh environments [2, 3]. In order to fully achieve the advantages of TiAlN coatings, a good coating/substrate interfacial adhesion is crucial. Since, the coating and carbide substrate is a layered system, the mechanical properties of the substrate and the graded properties between the coating and substrate will strongly determine its performance [4, 5]. However, the carbide substrate without surface pre-treatment usually exhibits high plasticity and insufficient toughness. Despite the high hardness, the coatings that are subject to high mechanical stresses and high temperature adhesive wear during machining will sag and collapse rapidly [4, 6, 7]. In addition, the TiAlN

coatings are also characterized by a high friction coefficient during the tribo-tests, which could cause a high wear rate [8].

To solve the problems described above, numerous methods such as adjustments of deposition conditions [9-11], modification of chemical composition [12–14], selective coating architectures [15, 16] and pretreatments of substrates [17-19] have been explored to optimize coating performance. Among these experimental techniques reported in the literature, the substrate pre-treatments exhibit many attractive features and are used for the modification of the upper substrate region and include various sequential processing steps. While mechanical pretreatments such as sandblasting are primarily responsible for cleaning the substrate surface (the elimination of possible oxide layers) and adjusting the surface topography, structural changes of the substrate material are achieved by means of thermal- or plasma chemical processes such as laser surface texturing (LST) [20, 21]. Surface textures are widely applied in mechanical joints and biomedical chips to improve the properties of lubricated surfaces, resulting in improvements in load capacity, wear resistance, the coefficient of friction, etc. Recently, LST presents competitive advantages for substrate precoating preparation, which can provide a high flexibility of the obtainable

<sup>\*</sup> Corresponding author at: Department of Mechanical Engineering, Shandong University, No. 17923 Jingshi Road, 250061 Jinan, Shandong Province, PR China. E-mail address: jxdeng@sdu.edu.cn (J. Deng).

structure geometries with high precision, simplification of the fabrication setup or direct-write, and contactless etching nature with minimal thermal impact on adjacent zones [22, 23]. Considerable research studies have been carried out to examine the effects of LST on the coating performance in some coating technologies, such as thermal spraying, painting, and electroless plating [20, 24, 25], indicating that laser texturing can induce a modification of the substrate surface to improve the adhesive strength of the coatings. Kromer et al. [20] demonstrated that laser surface texturing performed with an optimized laser-hole volume produced a much higher adhesion value for plasma sprayed Ni-Al coatings on 2017 aluminum alloy substrate, measured by the pull-off set and LASer Adhesion Test, compared to that performed with the conventional pre-treatment. The accuracy of laser texturing is achieved by controlling the laser and scanner parameters that influence the quality of the surface to be subsequently coated by thermal spraying. Thus, Lamraoui et al. [24] characterized and optimized two parameters of the texturing process (number of shots per hole and laser power) to get the best adhesion for the thermal sprayed Ni-Al coatings. It has been shown that a first parameter optimization produces an adhesion value higher than that generally observed with conventional pre-treatments. For the electroless coating, laser surface texturing was also applied to study its effect on the adhesive strength of the coatings. Here, Zheng et al. [25] utilized the electroless nickel plating technique to coat alumina surfaces that were either chemically coarsened or laser textured with different pit depths. Their results demonstrated that LST can improve the adhesive performance of electroless plated nickel coating due to the continuous and uniform coating on the surface, which provides effective mechanical anchors between the coatings and substrate.

Nevertheless, research on the application of laser surface texturing to improve PVD coating performance is limited; the topic related to the impact of multi-scale textures to PVD coatings has not been studied systematically and little is known about the combined effectiveness of different laser pretreatments as many reports focus on micro- or nanoscale textures. Furthermore, to fully understand how laser pre-treatment modifies the performance of the coatings and to provide more technical support to the viability of using this technology on different tribosystems, it is essential that the coatings are analyzed in friction and wear tests as well as in materials testing to characterize their properties such as morphology, microstructure, and adhesiveness. In this paper, efforts were made to improve the performance of PVD TiAlN coatings by employing a few types of surface textures on WC/Co substrate prior to coating deposition. Micro-scale, nano-scale, and micro/nano-scale textured substrates produced with a nanosecond laser and a femtosecond laser were considered. Characterization tests such as variable depth scratch tests and ball-on-disk wear tests were performed to evaluate the adhesive strength as well as the tribological behavior of the PVD TiAlN coating. Meanwhile, surface characteristics including nano-hardness, elastic module, wettability, and phase constitutions were investigated using the analytical tools, and the possible mechanisms for the effects of surface texture were discussed.

#### 2. Experimental details

#### 2.1. Specimen preparation

In this study, mirror polished YG6 (WC + 6%Co)-cemented carbide substrate with an average grain size of  $2-3\,\mu m$  and dimensions of

16 mm × 16 mm × 5 mm was used. The composition and properties of the as-received cemented carbide material are listed in Table 1. Three types of surface textures were fabricated prior to TiAlN coating deposition: (i) micro-scale textures fabricated by a nanosecond laser, (ii) nano-scale textures fabricated by a femtosecond laser, and (iii) micro/nano-scale textures fabricated by both lasers. For comparison, a conventional TiAlN coated sample without texture was also prepared. Thus, four different samples were fabricated: (i) a conventional TiAlN coated sample (named CCT), (ii) a micro-scale textured TiAlN coated sample (named MCT), (iii) a nano-scale textured TiAlN coated sample (named NCT), (iv) a micro/nano-scale textured TiAlN coated sample (named MNCT).

For the laser pre-treatment, two different pulsed lasers in the near IR region of the spectrum with different pulse durations were employed in this experiment to produce different types of surface textures on the carbide substrate: (i) an Nd:YAG nanosecond laser with a wavelength of 1064 nm and pulse duration of 10 ns and (ii) a regenerative amplified Ti:sapphire femtosecond laser operating at an 800 nm wavelength, 120 fs pulse duration, and 500 Hz repetition rate. The technical details about the femtosecond laser processing can be found in [26]. Based on authors' previous studies [26, 27, 28], the processing parameters of the Nd:YAG nanosecond laser for the regular micro-scale textures produced on the cemented carbide surface were a pump current of 8.6 A, frequency of 5000 Hz, and scanning speed of 20 mm/s; the optimal processing parameters of the femtosecond laser for the nano-scale textures were  $2.5\,\mu J$  pulse energy, a  $800\,\mu m/s$  scanning speed, and a 1 overscan. And the micro-scale textures generated on the substrate surfaces were parallel grooves with a period of ~200 µm. For the fabrication of the micro/nano-scale textured surface, the nanosecond laser was used first to create micro-scale grooves. The femtosecond laser was used to fabricate nano-scale grooves on the micro-scale textured substrate.

Immediately after laser irradiation, TiAlN coatings were deposited on the textured samples using cathode arc-evaporation without employing any adhesive layer. Before the deposition process began, the base pressure of the chamber was pumped to below  $7.0 \times 10^{-3}\,\mathrm{Pa}$ . For the deposition of the TiAlN coatings, pure Ti targets and TiAl composite targets with nitrogen gas (N<sub>2</sub>) were introduced into the chamber as a reactive atmosphere to obtain the coatings. Details of the deposition process can be found in our previous study [28]. All the deposition parameters are listed in Table 2.

#### 2.2. Testing and characterization

Several levels of characterizations were implemented in this work. A scanning electron microscope (SEM, QUANTA FEG 250, FEI Inc., USA) in combination with an energy dispersive X-ray spectroscopy (EDS, Oxford Instruments, UK), and white light interferometer (Wyko NT9300, Veeco Inc., USA) were used to compare the surface structure and topography of the different samples. Investigation of the samples' structural information was carried out by X-ray diffraction (XRD) using a D8 ADVANCE (Bruker AXS) diffractometer with Cu  $K_{\alpha}$  radiation.

Additionally, mechanical properties such as coating hardness and elastic modulus were determined by a Micro Combi Tester (CSM Instruments). For measurements of coating/substrate system hardness and elastic modulus, a spherical indenter with a radius of  $5\,\mu m$  was used as a more uniform stress distribution under the indenter tip allows a more accurate measurement of the hardness and elastic modulus. The relatively high maximum load of  $5000\,m N$ , which led to the indentation

Table 1
Properties of the cemented carbide materials.

Composition (wt%)	Density (g/cm <sup>3</sup> )	Hardness (GPa)	Flexural strength (MPa)	Fracture toughness (MPa·m <sup>1/2</sup> )	Thermal conductivity (W/(m·K))	Thermal expansion coefficient $(10^{-6}/K)$
WC + 6%Co	14.6	16.0	2300	14.8	75.4	4.51

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