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Investigation of modelling and stress distribution of a coating/substrate system after an indentation test

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ABSTRACT

A monolayer TiAlSiN coating and multilayer TiAlSiN nano-coating were deposited on an ISO P30 cemented carbide substrate, and their mechanical properties and fracture mechanisms were investigated by indentation and scratch tests. Mechanical models of indentation were proposed and optimized according to the results of numerical simulations, and it was found that these models could contribute to the quantitative analysis of stresses. The tensile stresses at the middle of the two adjacent corners along the indentation edge of the multilayer TiAlSiN nano-coating were found to be lower than those of the monolayer TiAlSiN coating, which could decrease the possibility of fracturing the coating. The crack initiation and propagation in the cross-sections of the coatings were observed using a dual-beam focused ion beam (FIB) machine to slot-cut the indentations, and the mechanisms of the crack propagation were analyzed. These cracks propagated perpendicularly to the interface in the monolayer TiAlSiN coating so the fractures could be mainly caused by tensile stress. With the multilayer TiAlSiN nano-coating, the orientation of the cracks observed in the same area were inclined at an angle rather than perpendicular to the interface, possibly due to the combination of tensile stress and shear stress at the interface between layers.

1. Introduction

Thin films and coatings play important roles in many applications, such as microelectronic functions [1,2], energy devices [3,4], sensors and actuators [5,6], and coating technologies. In manufacturing, coating prolongs a tool's life by improving its wear/corrosion resistance [7,8] and high-temperature oxidation resistance [9,10], which is essential for the high-speed cutting, dry cutting [11,12], and machining of difficult-to-machine materials [13]. The successful performance and reliability of coatings are often limited by their brittleness and their adhesion to the substrate of the cutting tool. Many researchers have investigated the cracks within the coating or at the interface of the coating/substrate, and evaluated the interfacial adhesion properties of the coating/substrate system. Interfacial adhesion properties play crucial roles in the reliability of a coated cutting tool. If the coating or interface fails due to substantial plastic deformation, cracking, or interfacial delamination, the tool may deteriorate and fail quickly.

The most important feature of a coated cutting tool is its high hardness, so failures are more likely to occur at the interface or in the coating. Many experimental techniques have been developed to investigate the fracture properties of coating structures and coating/substrate systems. For coating fracture testing, the indentation depth was much less than the coating thickness and the nanoindentations could be used. Swain and Menčik [14] proposed various responses on nanoindentation load-displacement curves when the fracture occurred during indentations with different combinations of coatings and substrates. The load-displacement curves may be disturbed (pop-in events) when a fracture has occurred within the coating, and the critical load for the pop-in events could be used to determine the fracture toughness of a coating. Another method for determining the fracture properties of coatings is to measure the crack length corresponding to the nanoindentation. Harding et al. [15] and Li and Bhushan [16] investigated the fracture toughness of coatings using a nanoindentation with a cube-corner indenter. It was reported that the cracking threshold loads could be substantially reduced and a larger stress concentration leading to fracture could occur when employing this sharper indenter. Thus, the fracture toughness could be calculated by the measured crack length, and the fracture process of the coating was analyzed in detail.

However, the applied loads used in standard nanoindentation could be too low to generate measurable cracks at the interface of the coating/substrate. Therefore, for coating/substrate system fracture testing, the indentation depth was larger than the coating thickness, and the indenter penetrated deeply into the substrate. Marshall and Evans [17] and Rossington et al. [18] proposed a mechanical analysis using indentation to investigate the interfacial adhesion properties. This was

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Table 1
Chemical composition of ISO P30 cemented carbide. (%).

WC	TiC	TaC + NbC	Co
75	7	8	10

based on the configuration of a coating that was loaded on a substrate by a Vickers indenter, which left a permanent impression. Under the influence of the elastic/plastic residual stress, radial cracks and penny-shaped lateral cracks were generated during the indentation. The lateral crack system that propagated along the interface was important for the analysis of the interfacial properties. In addition, delamination behavior is caused by plastically-induced stresses in the coating, and takes place during unloading of the indentation. Spalling may also accompany delamination in the presence of large coating stresses. Drory and Hutchinson [19] developed an analysis to characterize the adhesion properties and delamination behavior of a brittle coating on a ductile substrate with the indentation test under much greater loads. They presented mechanical solutions for assessing interfacial toughness from the applied load, delamination radius, and coating/substrate material properties. Nekkanty and Walter [20] and Suresha et al. [21] investigated contact-induced damage to the coating/substrate system by indentations at large loads. Picture-frame crack patterns were observed on the coating surface, and the composite hardness of the coating/substrate system was determined for a broad range of indentation depths. From the cross-sectional FIB images of a coating, it was observed that edge cracks and columnar shear cracks were generated, and the propagation mechanisms of these two damage modes were quite different.

As described above, the interfacial properties and crack propagation in the subsurface of a coating/substrate system have been extensively studied by indentation tests, and the crack initiation and propagation mechanisms of the coating surface were investigated in our previous study [22]. However, the fracture mechanisms and quantitative analysis of the stresses in the cross-sections of the coatings with different indentation depths have not been fully addressed. In addition, the indentation depths in most of the prior indentation tests were less than 10% of the coating thickness. The indenter only induced a slight deformation in the coating, and interfacial delamination could not be achieved. In this study, a Vickers indenter was used to perform sequential load-indentation tests at loads of 1–20 N for a monolayer TiAlSiN coating and multilayer TiAlSiN nano-coating. The surface morphology and chemical composition of both coatings were analyzed with a scanning electron microscope (SEM) and energy dispersive spectrometer (EDS). The crack initiation and propagation in the cross-sections of the coating were prepared using a dual-beam focused ion beam (FIB) machine to slot-cut the indentations. The objective of the present work is to investigate the cross-sectional fracture mechanisms, including crack initiation and propagation within the coating or at the interface. Also, mechanical models of the indentation were proposed and optimized according to the results of numerical simulations, which could contribute to quantitative analysis of the stresses.

2. Material and experimental methods

2.1. Coating process and specimen preparation

The coatings were engineered as single-layer and multilayer nanocomposite materials using the cathodic arc physical vapor deposition (PVD) method. The monolayer TiAlSiN coating and multilayer TiAlSiN nano-coating were deposited on a polished ISO P30 cemented carbide substrate using a cathodic arc PVD system. The substrate samples were cleaned in an ultrasonic cleaner with acetone and alcohol for 30 min. A rotational substrate holder was located in the vacuum chamber. The rotational speed was fixed at 2 rpm. Before deposition, the sputtering chamber was pumped down from atmospheric pressure to a base pressure of 1×10^{-3} Pa using a combination of rotary pump and turbo molecular pump. Substrates were cleaned again by ion bombardment using a bias voltage of -800 V under an Ar atmosphere of 1 Pa for 15 min. Titanium and AlSi alloy targets were used as cathodic arc sources to deposit the TiAlSiN coatings. A substrate bias voltage of −80 V and N_2 pressure of 2.5 Pa were used. The cathode current applied to Ti and AlSi targets was 100 A to control the composition of the deposited TiAlSiN coatings. Substrate samples were heated by a radiant heater arranged inside the chamber, and the temperature of the sample during the deposition was controlled within the range of 400–430 °C [7]. As for the multilayer nanocomposite coating, numerous layer parameters (elements, atomic structure, crystallinity, crystalline defects, and morphology) were modified via the process parameters, and the main layer characteristics could be controlled. The thickness of the deposited monolayer TiAlSiN coating and multilayer TiAlSiN nano-coating was controlled at 2 μm, and the multilayer TiAlSiN nano-coating was designed at about 100 layers.

The chemical composition and typical mechanical and thermal characteristics of the ISO P30 cemented carbide are listed in Tables 1 and 2. The chemical elements of both coatings were analyzed by a scanning electron microscope with an additional EDS. The monolayer TiAlSiN coating exhibited concentrations (wt.%) of Ti, Al, and Si of 20.97, 7.59, and 1.22, respectively, and the corresponding figures for the multilayer TiAiSiN nano-coating were 29.71, 7.86, and 1.92. To obtain a clear indentation and accurate measurement results, the coatings were polished using a W0.5 diamond-polishing agent to wipe off the droplet phase and reduce the surface-finish roughness. The final procedure of specimen processing was to ultrasonically clean the coatings with acetone and ethanol (20 min each) and dry them in a hot-air stream. The cross-sectional profiles of the two coatings after polishing are shown in Fig. 1. The thickness of the monolayer TiAlSiN coating was about (1.72 ± 0.07) μm, and the thickness of the multilayer TiAlSiN nano-coating was about (1.76 ± 0.10) μm. The surface roughness of the coatings was quantified using a Zygo NV7300-3D Optical Profiler (objective: × 10; zoom: × 2; scan length: 10 μm; averages of scan number: 10; horizontal resolution: 1 μm; vertical resolution: 0.1 nm). The surface roughnesses of the monolayer TiAlSiN coating and multilayer TiAlSiN nano-coating were 59.34 nm and 64.05 nm, respectively.

2.2. Nanoindentation tests

Nanoindentation of coatings was performed with a sharp Berkovich diamond indenter and a NanoTest Vantage (Micro Materials Ltd., Wrexham, UK). Indentations were performed in load-control mode to a maximum load of 30 mN, at a loading rate of 1 mN/s, with a 10 s dwell period at peak load. The indenter was then withdrawn at the same rate as during the loading. The contact depth was nearly 10% of the total coating thickness, and a low indentation depth could avoid the substrate influence. The experiments performed on each coating were repeated 30 times, and the average values of hardness and Young's modulus of

Table 2
Typical mechanical and thermal characteristics of ISO P30 cemented carbide: hardness HV [kgf/mm^2], flexural strength σ_{RS} [N/mm^2], compressive strength σ_{CS} [N/mm^2], relative brittleness B_{tm}, fracture toughness K_{IC} [MN/m$^{3/2}$], elasticity module E, coefficient of thermal expansion α_{et}, Poisson's ratio ν_t, thermal conductivity k_t [W/(m·K)], specific heat C_p [J/(kg·K)], density ρ [kg/m^3].

HV	σ_{RS}	σ_{CS}	B_{tm}	K_{IC}	$E{\cdot}10^3$	$\alpha_{et}{\cdot}10^{-6}$	ν_t	k_t	C_p	$\rho{\cdot}10^3$
1500	1950	4750	2.45	8.5	530	6.2	0.23	50	165	12.5