Mechanical properties and microstructural evolution of TiN coatings alloyed with Al and Si

Li Chen [a,*], Yong Du [a], She. Q. Wang [a,b], Ai. J. Wang [a], H.H. Xu [a]

[a] State Key Laboratory of Powder Metallurgy, Central South University, Changsha, Hunan 410083, China
[b] Zhuzhou Cemented Carbide Cutting Tools Co. Ltd, Zhuzhou, Hunan 412007, China

ARTICLE INFO

ABSTRACT

TiN coatings have been widely used as hard coating on cutting tools, but the poor oxidation resistance at temperatures above 500 °C created an interest in ternary coatings, e.g., Ti–Al–N and Ti–Si–N due to their improved hardness and high-temperature oxidation resistance. Here, we present a comparative research on the Ti–X–N (X = Al or Si) coatings deposited onto cemented carbide substrates by cathodic arc evaporation. The incorporation of Al and Si into TiN coating results in an increase in hardness, thermal stability and cutting performance. Compared to TiSiN coated inserts, TiAlN coated inserts show similar performance during continuous turning of stainless steel at the cutting speed of 160 m/min, but worse performance at the higher cutting speed of 200 m/min. However, during milling of steel, TiAlN coated inserts perform better than TiSiN coated inserts.

Crown Copyright © 2008 Published by Elsevier B.V. All rights reserved.

1. Introduction

TiN coatings, which have severed for 30 years in industrial applications, have still been studied extensively today due to relatively high hardness and wear resistance [1–3]. However, at temperatures higher than 500 °C it starts to oxidize into TiO_2 resulting in crack formation and delamination of the brittle oxide layer deteriorating mechanical and tribological properties [2,3]. In the recent years, a lot of efforts were done by alloying TiN based coatings with several elements acting in different ways [4–8]. Among them, the ternary coatings by incorporation of Al and Si into TiN attract more attention for cutting applications [7–12]. Ti–Al–N coatings become the most widely used coating in cutting tool due to the better oxidation resistance, higher hardness and improved thermal stability as compared to TiN. And a further increase in hardness can be acquired by Ti–Si–N coatings via formation of nanocomposite structure, where amorphous (a) Si_3N_4 boundary layers encapsulate nanocrystalline (nc) TiN grains [10–15]. The purpose of this work is to investigate the effect of incorporation of Al and Si into TiN coatings. We concentrate on the mechanical properties and microstructural evolution of the coatings.

2. Experimental details

2.1. Coating deposition

Ti–N, Ti–Al–N and Ti–Si–N coatings were deposited onto powder metallurgically prepared CNMG120408 style (WC-6 wt.% Co) and SEET12T3 style cemented carbide (WC-10 wt.% Co) substrates by a commercial cathodic arc evaporation system (Balzers Oerlikon Rapid Cooling System, RCS) from the Ti, $Ti_{0.50}Al_{0.50}$ or $Ti_{0.94}Si_{0.06}$ targets. Prior to the deposition with a twofold substrate-rotation fixture in N_2 (99.99% purity) atmosphere at ~2 Pa, −100 V DC substrate bias and 550 °C, the substrates were cleaned by an Argon-ion-etching process.

2.2. Isothermal annealing

Isothermal annealing of the coated samples has been performed in vacuum furnace (COD533R) at 0.1 mPa. Heated from room temperature with a heating rate of (RT) 5 K/min, each sample was annealed at 700, 900, and 1100 °C for 2 h. And then the annealing samples cooled down inside the furnace with the heater switched off.

2.3. Characterization

The elemental compositions of the coatings were determined using electron probe microanalysis (EMPA) (JXA-8800R, JEOL). Structural investigations were conducted by X-ray diffraction (XRD)

---

* Corresponding author. Fax: +867318710855.
E-mail address: chenli_927@126.com (L. Chen).

with Cu Kα radiation using a Bruker D8 in Bragg/Brentano mode. The grain size was determined by the single method for analysis of XRD line broadening using a Pseudo-Voigt profile function [16]. The hardness of the coatings was obtained by nanoindentation with a Fischerscope H100VP after the Oliver and Pharr method [17]. According to the experimental results based on the large-load (30 mN) penetration test, a smaller penetration load of 8 mN was chosen to measure the mechanical properties of the coatings. The bonding structure of the Ti–Si–N coatings was characterized by XPS using a RBD upgraded PHI-5000C ESCA system (PerkinElmer) with Mg Kα radiation ($h\nu$ = 1253.6 eV) or Al Kα radiation ($h\nu$ = 1486.6 eV). In general, the X-ray anode was run at 250 W and the high voltage was kept at 14.0 kV with a detection angle at 54°. The pass energy was fixed at 23.5, 46.95 or 93.90 eV to ensure sufficient resolution and sensitivity. The base pressure of the analyzer chamber was about 5×10^{-8} Pa. The sample was directly pressed to a self-supported disk (10 mm × 10 mm) and mounted on a sample holder then transferred into the analyzer chamber. The whole spectra (0–1100 (1200) eV) and the narrow spectra of all the elements with much high resolution were both recorded by using RBD 147 interface (RBD Enterprises, USA) through the AugerScan 3.21 software. Binding energies were calibrated by using the containment carbon (C 1s = 284.6 eV). The data analysis was carried out by using the RBD AugerScan 3.21 software provided by RBD Enterprises or XPS-Peak4.1 provided by Raymund W.M. Kwok (The Chinese University of Hongkong, China). Scratch tests were performed using a scratch tester (MS-T3000 scratch) with a Rockwell C diamond indenter. More details on the characterization experiments are described in Ref. [6].

2.4. Cutting tests

Continuous dry turning of stainless steel (1Cr18Ni9Ti) with CNMG120408-EM style inserts (WC-6 wt.% Co) was conducted with a cutting speed (v_c) of 160 and 200 m/min, a depth of cut (a_p) of 1.0 mm and a feed rate (f) of 0.2 mm per revolution (mm/r). Dry face milling of steel (42CrMo) with SEET12T3-DM style inserts (WC-10 wt.% Co) was performed with v_c = 320 m/s, a_p = 2.0 mm and f = 0.15 mm/r. The criterion for the tool lifetime is when the flank wear lands exceed 0.3 mm.
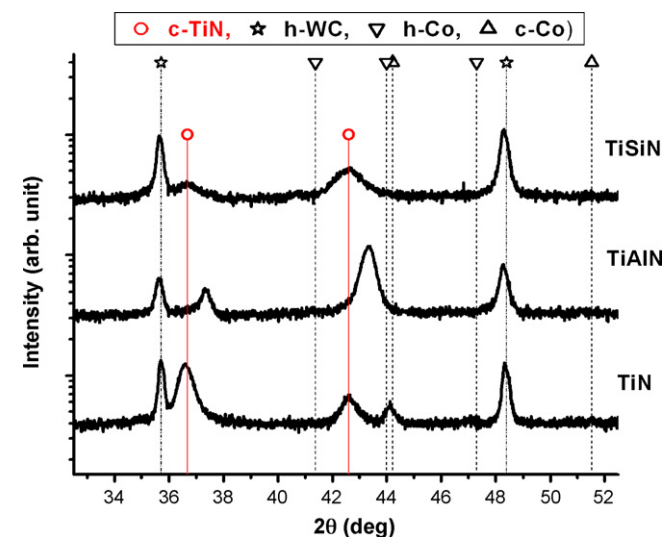
3. Results and discussion

EPMA measurements show that the nitride coatings have an elemental composition of $Ti_{0.52}Al_{0.48}N$ and $Ti_{0.94}Si_{0.06}N$. Hence, the atomic ratios in the coating correspond to that of the targets. Fig. 1 shows the XRD patterns of as-deposited three coatings. Analysis of the XRD results reveals that all coatings as-deposited exhibit a single-phase NaCl structure. The diffraction peaks of TiAlN coating, which shows a preferred orientation in crystal plane of (2 0 0), shifts to higher angle owing to lattice constant decreases arising from the replacement of titanium atoms in the TiN lattice by aluminium. And as for the TiSiN coating, no signals from crystal Si_xN_y or from titanium silicide could be observed. This result implied that Si is present in an amorphous phase of either compound or simple substance. With the addition of Al and Si, the decreased grain size is observed, which is shown from the reduced intensity of XRD peaks as well as peak broadening. Determination of the average crystallite size using broadening of the XRD peaks indicates an average crystallite size of approximately 24.7 nm with TiN coating, ~18.6 nm with TiAlN coating and ~10 nm with TiSiN coating, respectively. In general, the crystallite size using XRD is smaller than the actual crystallite size due to the solution strain and residual stress [18].
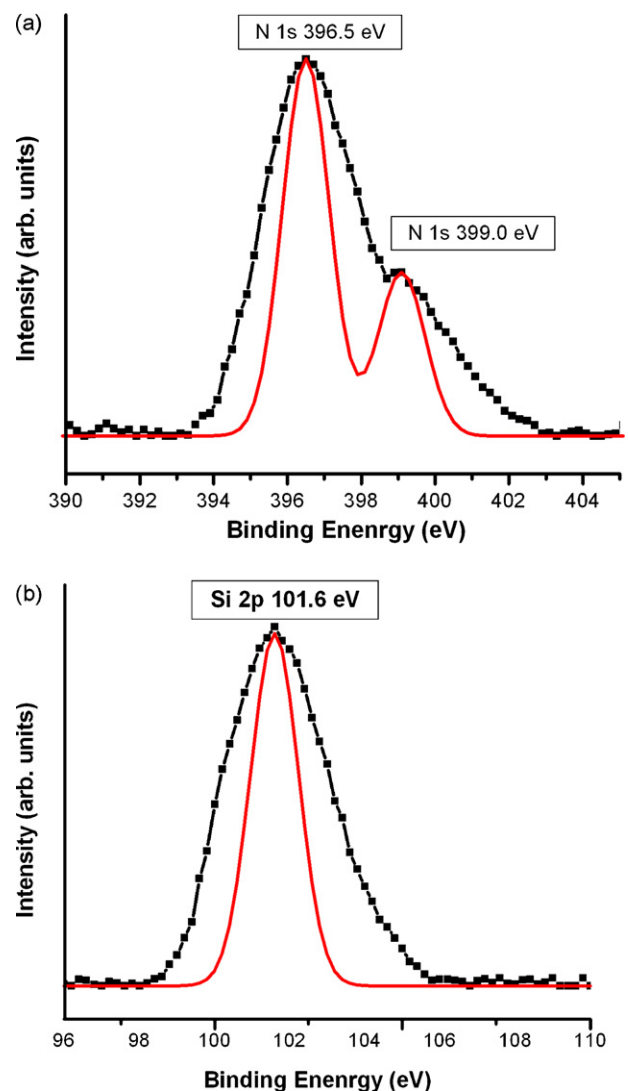

Fig. 2. XPS spectra of N 1s (a) and Si 2p (b) peaks for the Ti–Si–N coating.

Fig. 1. XRD patterns of Ti–X–N coatings.