



Effect of Zr on structure and properties of Ti–Al–N coatings with varied bias

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ABSTRACT

Ti–Al–N coatings exhibit considerable mechanical and thermal properties, which make them promising candidates for advanced machining and other high temperature application. Here, the effect of Zr addition on the structure, mechanical and thermal properties of Ti–Al–N coatings deposited by industrial cathodic arc evaporation system is studied. Furthermore, we researched the machining application of Ti–Al–Zr–N coated inserts with varied bias from -50 to -150 V. Addition of Zr leads to a structural transition from cubic to mixed cubic–hexagonal, and an increased hardness from ~ 31.2 to 33.1 GPa. The high hardness value of ~ 35.8 GPa for Ti–Al–Zr–N coating after annealing at 1000 °C indicates the improved thermal stability. Oxidation experiments yield fully oxidized Ti–Al–N coating for 16 h at 850 °C, whereas Ti–Al–Zr–N coating only forms of a layered oxide scale ~ 1.0 μm . Therefore, an increased machining performance in continuous turning is obtained by Ti–Al–Zr–N coated insert. Increasing of deposition bias favors the growth of cubic structure, and thereby results in a structure transition from a mixed cubic–hexagonal structure to finally a single phase cubic structure for bias of -150 V. With consideration of its structure, hardness, defect densities, stress and cohesion with substrate, Ti–Al–Zr–N coated inserts with bias of -100 V behaved the best performance in both continuous turning and milling.

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1. Introduction

Al-containing cubic transition metal nitride (Me–Al–N) based hard coatings, where Al substitutes for Me in the MeN based lattice, are widely used to reduce tool wear due to their good tribological and high thermal properties [1–7]. The crystal structure, mechanical and thermal properties of Me–Al–N coatings are scaled with the Al content [5–10]. Increasing Al content, while keeping cubic phase, results in an increased mechanical properties and oxidation resistance [5–10]. However, the mechanical properties of Me–Al–N coatings decrease for Al contents exceeding the maximum solubility in the cubic phase, where a mixed cubic–NaCl and wurtzite–ZnS (w) structure is formed [5–10]. Among them, cubic Ti–Al–N coating is industrially well applied as it covers the above properties [7,8,11], and even exhibits self-hardening during elevated temperature, know as age-hardening [11,12].

Depending on different loading conditions, application temperatures of these wear coatings are different ranging from low temperatures for deep drawing dies to above 1000 °C for specific dry-cutting tools. Ti–Al–N coatings with the oxidation resistance below ~ 900 °C [8] can not meet the need of these specific machining above 1000 °C.

Several attempts have been made to improve application-related properties of Ti–Al–N by doping and alloying of fourth alloying elements [13–19]. An improvement on the mechanical and thermal properties has been observed e.g. for Ta, Nb, Zr, and Si alloying [13–18], while e.g. V alloying substantially lowers friction and wear of Ti–Al–N [19]. Our previous works on sputtered quaternary Ti–Al–Zr–N with different Zr content shows that alloying with Zr promotes the formation of cubic domains but retards the formation of stable wurtzite AlN during thermal annealing, and thereby leads to high hardness values of ~ 40 GPa over a broad temperature range of 700 – 1100 °C [15]. Furthermore, an improved oxidation resistance is obtained by incorporation of Zr, where Zr assists the formation of a dense oxide scale, and thus retards its growth rate [15]. In particular, $\text{Ti}_{0.40}\text{Al}_{0.55}\text{Zr}_{0.05}\text{N}$ which contains only 5 at.% Zr exhibits the best mechanical and thermal properties [15]. Nevertheless, the industrial application of Ti–Al–Zr–N coating has not yet been explored.

The purpose of this work is to investigate the machining application of Ti–Al–Zr–N coated inserts followed the $\text{Ti}_{0.40}\text{Al}_{0.55}\text{Zr}_{0.05}\text{N}$ composition according to our previous work. Consequently, we deposited Ti–Al–Zr–N coatings with varied bias by industrial cathodic arc evaporation system using $\text{Ti}_{0.40}\text{Al}_{0.55}\text{Zr}_{0.05}$ target in order to research the influence of bias on Ti–Al–Zr–N coatings. Additionally, a comparative research on mechanical and thermal properties of Ti–Al–N and Ti–Al–Zr–N coatings has been done in order to verify our previous work [15].

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2. Experimental details

2.1. Coating deposition

Ti–Al–N and Ti–Al–Zr–N coatings were deposited by a commercial cathodic arc evaporation system (Balzers Oerlikon Rapid Cooling System, RCS) with 6 cathodic arc sources for two Ti targets and four $\text{Ti}_{0.50}\text{Al}_{0.50}$ ($\text{Ti}_{0.40}\text{Al}_{0.55}\text{Zr}_{0.05}$) targets (99.99% purity). More details on the deposition system and growth conditions used are described in Ref. [20]. Different substrates were used for the individual investigations: powder metallurgically prepared cemented carbide inserts of CNMG120408 style (WC-6 wt.% Co) for X-ray diffraction and hardness measurements of as deposited and annealed coatings; cemented carbide inserts of CNMG120408 style (WC-6 wt.% Co) and SEET12T3 style (WC-10 wt.% Co) for cutting measurements; low alloyed steel for structural evolution and polycrystalline Al_2O_3 plates ($10 \times 10 \times 1 \text{ mm}^3$) for oxidation resistance measurements. Before loading the deposition chamber, the substrates were ultra-sonically cleaned in acetone and ethylene. Prior to the deposition with a two fold substrate-rotation fixture in N_2 (99.99% purity) atmosphere at $\sim 2 \text{ Pa}$, 180 A target current, 550 °C and varied substrate bias (-100 V DC for Ti–Al–N coatings and varied bias from -50 to -100 to -150 V DC for Ti–Al–Zr–N coatings), the substrates were cleaned by an Argon-ion-etching process with bias of 180 V. The coatings deposited onto cemented carbide, low alloyed steel and Al_2O_3 plates are realized by four cathodic arc sources equipped with $\text{Ti}_{0.50}\text{Al}_{0.50}$ ($\text{Ti}_{0.40}\text{Al}_{0.55}\text{Zr}_{0.05}$) targets without the TiN interlayer in order to avoid its interference during subsequent analysis. However, for coatings deposited onto cemented carbide inserts to cutting tests, two cathodes are equipped with Ti targets (99.99% purity) for the deposition of a $\sim 30 \text{ nm}$ thin TiN interlayer followed by a thickness of $\sim 3 \mu\text{m}$ Ti–Al–N (Ti–Al–Zr–N) coatings according to the calculated deposition rate by the previous experiments, where the TiN interlayer is used to improve the adhesion strength with substrate. Details on the deposition substrate bias, interlayer, substrates and corresponding analysis methods are presented in Table 1.

2.2. Isothermal annealing and oxidation

Isothermal annealing of the coated cemented carbide substrates has been performed in vacuum furnace (COD533R) at 0.1 mPa. Heated from room temperature with a heating rate of (RT) 5 K/min, coated cemented carbide substrates were annealed at 700, 900, 1000 and 1100 °C for 2 h, respectively. And then the annealed samples cooled down inside the furnace with the heater switched off. For isothermal oxidation experiments coated polycrystalline Al_2O_3 plates were isothermally oxidized at 850 °C for 16 h, 900 °C for 8 h and 950 °C for 8 h in a conventional tube furnace, respectively, and then the thickness of oxidized layer was observed by scanning electron microscopy (SEM).

Table 1
Deposition substrate bias, interlayer, substrates and analysis methods.

Coatings	Bias (V)	TiN interlayer	Substrates	Analysis methods
Ti–Al–N	100	With	Cemented carbide	XRD, adhesion strength, cutting tests
		Without	Low alloyed steel Cemented carbide Al_2O_3	XRD (structural evolution) SEM (surface), Hardness SEM (oxidation)
Ti–Al–Zr–N	50–150	With	Cemented carbide	XRD, adhesion strength, cutting tests
		Without	Low alloyed steel Cemented carbide Al_2O_3	XRD (structural evolution) SEM (surface), hardness SEM (oxidation)

2.3. Characterization

The elemental composition of the coatings was determined using electron probe microanalysis electron probe micro-analyzer (EPMA) (JXA-8800R, JEOL). The error of measurements for the metal atoms is below 2 at%. The surface and oxidized fracture morphology of the coatings were studied using scanning electron microscope (SEM) (LEO1525, Germany). Phase identification and structural investigations of the coatings (onto cemented carbide and after removal from their low alloy steel substrates) in their as deposited state were conducted by XRD with $\text{CuK}\alpha$ radiation using a Bruker D8 diffractometer in Bragg/Brentano mode at 40 mA and 40 KV. Prior to these measurements, the coatings were removed from their low alloyed steel substrates by chemical etching in 10 mol% nitric acid, in order to avoid substrate interference. The hardness of the coatings as deposited and annealed at 700, 900, 1000 and 1100 °C for 2 h in vacuum was obtained by nanoindentation with a Fischerscope H100VP after the Oliver and Pharr method [21]. 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 to keep the indentation depth below 10% of the coating thickness.

2.4. Cutting tests

Continuous dry turning of stainless steel (1Cr18Ni9Ti) with CNMG120408-EM style cemented carbides 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 stainless steel with SEET12T3-DM style inserts (WC-10 wt.% Co) was performed with $v_c = 80 \text{ m/min}$, $a_p = 1.0 \text{ mm}$ and $f = 0.2 \text{ mm/r}$. The criterion for the tool life-time is when the flank wear lands exceed 0.3 mm.

3. Results and discussion

Elemental analysis by EPMA reveals that the Ti–Al–N and Ti–Al–Zr–N coatings are stoichiometric with N/metal ratios of 1 ± 0.02 and compositions of $\text{Ti}_{0.52}\text{Al}_{0.48}\text{N}$ with -100 V substrate bias, $\text{Ti}_{0.45}\text{Al}_{0.49}\text{Zr}_{0.06}\text{N}$ with substrate bias from -50 to -150 V . Varied bias has not obvious effect on the composition of Ti–Al–Zr–N. Fig. 1 exhibits the surface morphology of Ti–Al–N and Ti–Al–Zr–N coatings with varied bias. Compared to Ti–Al–N coating (Fig. 1a), Ti–Al–Zr–N coating with the same bias of -100 V (Fig. 1c) shows more metallic macro-particles. Increasing bias of Ti–Al–Zr–N coatings from -50 to -150 V results in a decrease in metallic macro-particles, see Fig. 1b, c and d, which is in agreement with Ref. [22]. Fig. 2 shows the XRD patterns of as-deposited coatings onto cemented carbide inserts (Fig. 2a) and powdered freestanding coating samples (Fig. 2b). Analysis of the XRD results shows that the $\text{Ti}_{0.50}\text{Al}_{0.50}\text{N}$ coating reveal a single phase cubic structure. Addition of Zr into Ti–Al–N coating, while keeping the substrate bias of -100 V , results in a slight structure transition into a mixed cubic–hexagonal structure, where the weak peak of w-AlN at $\sim 33.2^\circ$ is observed, see Fig 2. b. Incorporation of Zr with big atomic radius into Ti–Al–N results in a shift of the XRD peak positions to lower 2θ angles. Additionally, an increase in peak broadening indicates a reduction in grain size and/or an increase in micro-stresses with incorporation Zr into Ti–Al–N coating. Decreasing substrate bias from -100 to -50 V promotes hexagonal phase at the expense of cubic phases, which is in agreement with the previous reports [22,23], see Fig. 2. High bias voltages lead to a large local fluctuations of Ti and Al in Ti–Al–N, and thus favors the growth of cubic phase [23]. Here, our evaporated Ti–Al–Zr–N coatings exhibit the reduced metastable solubility limit for AlN, compared to the previous sputtered Ti–Al–Zr–N coatings [15]. Consequently, depending on the defect structure and coating orientation, different values for the maximum AlN solubility in the cubic

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