

Microstructure, mechanical properties and cutting performance of $\text{Cr}_{1-y}\text{Ta}_y\text{N}$ single layer and $\text{Ti}_{1-x}\text{Al}_x\text{N}/\text{Cr}_{1-y}\text{Ta}_y\text{N}$ multilayer coatings

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ABSTRACT

$\text{Cr}_{1-y}\text{Ta}_y\text{N}$ single-layer and $\text{Ti}_{1-x}\text{Al}_x\text{N}/\text{Cr}_{1-y}\text{Ta}_y\text{N}$ multilayer hard coatings were deposited on cemented carbide substrates using magnetron sputter deposition and cathodic arc evaporation. For the sputtered coatings a bias voltage of -50 V was applied, while arc evaporated coatings were deposited at three different bias voltages of -40 , -60 and -80 V . The coatings were grown from powder metallurgically produced CrTa targets with a composition of 75 at% Cr and 25 at% Ta, resulting in a Cr/Ta ratio of 71/29 in the sputtered coatings and 74/26 in the arc evaporated coatings, independent of the applied bias voltage. All coatings are characterized by a single-phase face centered cubic structure. The sputtered single- and multilayer coatings exhibited a hardness of ~ 22.2 and $\sim 25.2\text{ GPa}$, respectively. For the arc evaporated coatings, a hardness of ~ 22.7 could be determined for the multilayer, while for the single-layers the hardness increased from ~ 21.6 to $\sim 24.3\text{ GPa}$ with increasing bias voltage. Ball-on-disk tests against alumina at room temperature, 500 and 700°C yielded wear rates comparable to conventional $\text{Ti}_{1-x}\text{Al}_x\text{N}$ coatings, but significantly lower friction coefficients. The performed cutting tests also showed promising results, indicating the high application potential of $\text{Cr}_{1-x}\text{Ta}_x\text{N}$ -based hard coatings.

1. Introduction

The continuing trend of the machining industry for higher productivity and cost efficiency results in further increasing cutting velocities and feed rates and simultaneous demand for longer tool lifetimes. This calls for continuous improvement of the applied hard wear protective coatings. Physical vapor deposited (PVD) $\text{Cr}_{1-x}\text{Al}_x\text{N}$ and $\text{Ti}_{1-x}\text{Al}_x\text{N}$ coatings are frequently used to reduce tool wear [1,2]. A frequently used strategy for their further improvement is based on the addition of alloying elements to form quaternary compounds or nanocomposites [1]. A possible but challenging alternative is the quest for novel materials to be applied as hard coating. Since CrN exhibits high hardness and good oxidation resistance [1,3–5] and TaN combines high hardness and wear resistance with chemical inertness and excellent diffusion barrier properties [6–8], $\text{Cr}_{1-y}\text{Ta}_y\text{N}$ might represent a promising candidate for a novel wear resistant hard coating with superior properties. Only few studies are available discussing the microstructure, mechanical properties and thermal stability of sputtered $\text{Cr}_{1-y}\text{Ta}_y\text{N}$ coatings [9–13]. However, up to our knowledge no publications on cathodic arc evaporated $\text{Cr}_{1-y}\text{Ta}_y\text{N}$ coatings exist. Further, no literature

on the tribological properties and application behavior of both, sputtered and arc evaporated $\text{Cr}_{1-y}\text{Ta}_y\text{N}$ coatings can be found.

Thus within the present work, $\text{Cr}_{1-y}\text{Ta}_y\text{N}$ single-layer and $\text{Ti}_{1-x}\text{Al}_x\text{N}/\text{Cr}_{1-y}\text{Ta}_y\text{N}$ multilayer coatings were synthesized by sputter deposition as well as cathodic arc evaporation. The chemical composition and microstructure of the coatings was investigated using energy dispersive X-ray spectroscopy and X-ray diffraction, respectively. Nanoindentation was applied to determine the coating hardness. Information on the tribological properties of the sputter deposited coatings was obtained using ball-on-disk test, while selected arc evaporated coatings were evaluated in milling tests.

2. Experimental methods

$\text{Cr}_{1-y}\text{Ta}_y\text{N}$ single-layer and $\text{Ti}_{1-x}\text{Al}_x\text{N}/\text{Cr}_{1-y}\text{Ta}_y\text{N}$ multilayer coatings were deposited using an industrial scale CemeCon CC800/9 MLT deposition system, equipped with four powder metallurgically produced compound targets: two TiAl (40 at% Ti, 60 at% Al) and two CrTa targets (75 at% Cr, 25 at% Ta). The coatings were grown on ground and polished cemented carbide substrates in SNUN 120312EN (according to

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ISO 1832) and disk geometry mounted onto the substrate carousel operating in twofold rotation. The composition of the SNUN substrates was 92 wt% tungsten carbide, 6 wt% cobalt and 2 wt% mixed carbide. Prior to deposition, an Ar^+ ion etching step for substrate cleaning was implemented [14]. During deposition, the magnetrons were bipolarly pulsed at a frequency of 55 kHz and a duty cycle of 50% in constant power mode set to 6 kW per target. For the $\text{Cr}_{1-y}\text{Ta}_y\text{N}$ single-layer coating, only the cathode pair with the CrTa targets was activated, while in case of the $\text{Ti}_{1-x}\text{Al}_x\text{N}/\text{Cr}_{1-y}\text{Ta}_y\text{N}$ multilayer coating first a $\text{Ti}_{1-x}\text{Al}_x\text{N}$ base-layer was deposited, where only the cathode pair with the TiAl targets was used, followed by the multilayer, where all four cathodes were activated simultaneously. The asymmetrically bipolarly pulsed d.c. bias voltage was set to -50 V at a frequency of 350 kHz and 1 μs reversal time. During deposition, the Ar flow was held constant at 180 sccm, while the N_2 flow was controlled to maintain a total pressure of 6.2×10^{-3} mbar. The substrate temperature was set to 550°C and the deposition time to 95 min for the single-layer and to 260 min for the multilayer coating (180 min for the $\text{Ti}_{1-x}\text{Al}_x\text{N}$ base-layer and 80 min for the $\text{Ti}_{1-x}\text{Al}_x\text{N}/\text{Cr}_{1-y}\text{Ta}_y\text{N}$ multilayer). The coating thicknesses were, as determined by ball cratering, $\sim 2.4 \mu\text{m}$ for the single-layer and $\sim 4.8 \mu\text{m}$ for the multilayer (1.8 μm for the $\text{Ti}_{1-x}\text{Al}_x\text{N}$ base-layer and 3.0 μm for the $\text{Ti}_{1-x}\text{Al}_x\text{N}/\text{Cr}_{1-y}\text{Ta}_y\text{N}$ multilayer). Considering the rotation speed of the carousel (1 rpm) and the respective deposition rates, sublayer thicknesses of ~ 25 nm for the $\text{Cr}_{1-y}\text{Ta}_y\text{N}$ and ~ 10 nm for the $\text{Ti}_{1-x}\text{Al}_x\text{N}$ were calculated for the multilayer.

The arc evaporated coatings were synthesized in an industrial scale Oerlikon Balzers INNOVA deposition system. Powder metallurgically produced compound TiAl (33 at% Ti, 67 at% Al) and CrTa (75 at% Cr, 25 at% Ta) targets were used to deposit the coatings. The deposition runs were conducted in a pure N_2 atmosphere at a pressure of 3.3×10^{-2} mbar and a substrate temperature of 450°C . The coatings were grown on cemented carbide cutting inserts in SNUN 120312EN (ground and polished) and XDKT 11T308SR-F50 (surface finish according to industrial standards for these inserts) geometry (according to ISO 1832) and disk geometry. The composition of the SNUN substrates was the same as given above and the XDKT substrates consisted of 87.5 wt% tungsten carbide, 10.5 wt% cobalt and 2 wt% mixed carbide. The substrates were mounted on a carousel allowing for two-fold (disk geometry) and three-fold rotation (SNUN and XDKT geometry). Prior to deposition, the substrates were sputter-etched in a pure Ar plasma. A series of single- and multilayer coatings were grown using bias voltages of -40 , -60 and -80 V for single-layer coatings and -70 V for the multilayer coating. For the $\text{Cr}_{1-y}\text{Ta}_y\text{N}$ single-layer coating, only two CrTa cathodes were used, while in case of the $\text{Ti}_{1-x}\text{Al}_x\text{N}/\text{Cr}_{1-y}\text{Ta}_y\text{N}$ multilayer coating first a $\text{Ti}_{1-x}\text{Al}_x\text{N}$ base-layer was deposited, where four TiAl cathodes were used, followed by the multilayer structure, where all 6 cathodes were activated simultaneously. For the $\text{Ti}_{1-x}\text{Al}_x\text{N}$ base-layer, the deposition procedure described in refs [15,16] was applied. The deposition time was adjusted to reach a thickness of $\sim 3.2 \mu\text{m}$ for the single-layer coatings and $\sim 3 \mu\text{m}$ for the multilayer coating (1.7 μm for the $\text{Ti}_{1-x}\text{Al}_x\text{N}$ base-layer and 1.3 μm for the $\text{Ti}_{1-x}\text{Al}_x\text{N}/\text{Cr}_{1-y}\text{Ta}_y\text{N}$ multilayer). This corresponds to a growth rate per cathode of $\sim 0.5 \mu\text{m}/\text{h}$ for the single-layer $\text{Cr}_{1-y}\text{Ta}_y\text{N}$ coatings. The applied substrate rotation speed yields a bilayer period of 30 to 35 nm for the multilayer

arrangement as determined on the substrate disks.

The chemical composition of the coatings was determined by energy dispersive X-ray spectroscopy (EDX) using an Oxford Instruments INCA extension in a Zeiss EVO 50 scanning electron microscope (SEM). In addition, a field emission gun SEM (Zeiss FEG Ultra Plus) was used for micrographs of fracture cross-sections of the coatings. Information on the microstructure was obtained by X-ray diffraction (XRD) applying a Bruker-AXS D8 Advance diffractometer in grazing incidence (GI) geometry (incidence angle 2°). The residual stresses were determined using a Rigaku SmartLab X-ray diffractometer with $\text{Cu-K}\alpha$ radiation and parallel beam optics applying the $\sin^2\psi$ method on the (200) peaks [17]. The diffractometer was equipped with an Eulerian cradle, a scintillation counter detector and a Soller collimator with an acceptance angle of 5° in the diffracted beam. A CSM nanoindenter equipped with a Berkovich diamond tip was used for the determination of hardness and Young's modulus. The tests were conducted at a load of 30 mN. A minimum of 9 indents were averaged to achieve statistically relevant results. The tribological properties of the sputtered coatings were investigated applying a CSM ball-on-disk tribometer at room temperature (RT), 500 and 700°C . A wear track radius of 5 mm, a normal load of 5 N and a sliding distance of 300 m were chosen. As counterparts Al_2O_3 balls with a diameter of 6 mm were used. The resulting wear tracks were examined using a Veeco Wyko NT1000 white light interferometer. The arc evaporated $\text{Ti}_{1-x}\text{Al}_x\text{N}/\text{Cr}_{1-y}\text{Ta}_y\text{N}$ multilayer coatings and a single-layer $\text{Ti}_{1-x}\text{Al}_x\text{N}$ reference coating (similar to the $\text{Ti}_{1-x}\text{Al}_x\text{N}$ coating used as base-layer of the multilayer system, compare ref. [18]) were evaluated in milling operation using a Ceratizit C211.25.R.03-B-32 shoulder milling system with a working diameter of 25 mm equipped with one cutting insert (XDKT 11T308SR-F50 geometry) against X5CrNiCuNb17-4-4 (DIN 1.4548) steel as work piece material. Two inserts of each coating were tested to get minimum statistics. All tested inserts had a comparable coating thickness. The cutting parameters were: cutting speed $v_c = 120$ m/min, feed per tooth $f_z = 0.15$ mm, engagement $a_e = 20$ mm and maximum depth of cut $a_{p,\text{max}} = 3$ mm. The lifetime criterion for this test was a flank wear of 0.3 mm. All cutting tests were carried out without coolant. Wear marks after cutting periods of 9 min were investigated by light optical microscopy.

3. Results and discussion

In Fig. 1a and b, SEM micrographs of fracture cross-sections of the sputtered single-layer coating and the arc evaporated single-layer coating deposited at -60 V bias voltage are presented. The sputtered coating consists of a fine columnar structure, while the arc evaporated coating is much coarser, also showing droplets typical for arc evaporated coatings [19,20]. The micrograph of the arc evaporated coating is representative for all coatings grown at different bias voltages. The elemental composition of the sputtered $\text{Cr}_{1-y}\text{Ta}_y\text{N}$ was 34 at% Cr, 14 at% Ta and 52 at% N, corresponding to a Cr/Ta ratio of 71/29 and thus a higher Ta content compared to the target. This can be explained by gas scattering and resputtering effects during coating growth, based on the different atomic masses of Cr and Ta [21–23]. Furthermore, selective poisoning of the target surface and thus variations in the sputter yield might also affect the Cr/Ta ratio of the coating [24]. For the arc

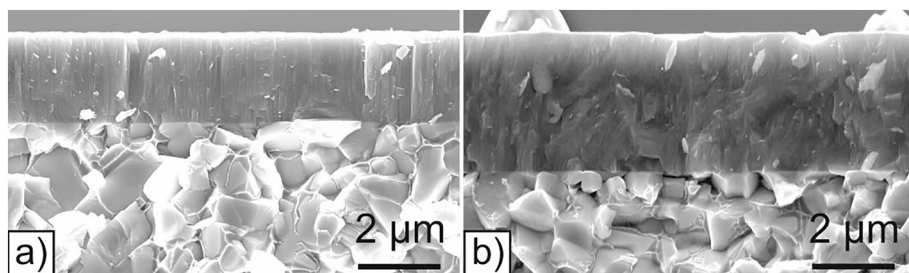


Fig. 1. SEM micrograph of fracture cross-sections of $\text{Cr}_{1-y}\text{Ta}_y\text{N}$ single-layer coatings synthesized by a) sputter deposition and b) arc evaporation (bias voltage: -60 V).

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