Multilayered and nanolayered hard nitride thin films for a better yield in micro machining.

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Abstract

TiN/AlTiN, TiN/CrN and CrN/AlTiN multilayer coatings have been deposited by the cathodic arc evaporation technique. The period is in the range 7 – 200 nm for a total thickness of 3 µm. The period’s control of the nanostructure during deposition is achieved, and is realized by a simple geometrical calculation and, posteriorly, by X-ray diffraction and transmission electron microscopy on cross-sections. Microstructure of the as-deposited coatings has been investigated by means of X-ray diffraction and transmission electron microscopy in connection with the decrease of the period \( \lambda \). For lower periods (multilayered coatings), the fcc structures, which derive from each nitride are observed while only the superlattice structure is found for nanoscale layered films (nanolayered coatings). Microstructure evolution with the period (defined as the sum of two elementary layers) is investigated for the three systems and the differences are comment. Mechanical and tribological properties are strongly dependent on coating structure. The best mechanical properties were obtained with TiN/AlTiN nanolayers with 7 nm periods and superlattice structure [1]. These coatings induced low main cutting force and low flank wear during Inconel 718 turning. Time life of superlattice TiN-AlTiN coated cutting tool is increased comparatively to CVD coated and AlTiN coated cutting tools actually used in production.

Keywords: arc evaporation, superlattice, TEM, X-Ray Diffraction

1. Introduction

The demand for PVD hard coatings exhibiting properties such as high wear and oxidation resistance has grown enormously during the last decades particularly for mechanical applications in severe environment as machining. Evolvement of both machining techniques and materials to be machined led to the development of multilayered coatings and, recently, to super-hard superlattice structure [2, 3, 4]. It is well established that the bond structure in transition metal nitrides is responsible for high hardness, high wear resistance and chemical inertness. Moreover, AITiN is well known for its high oxidation resistance enabling improved performances in hard material machining [5, 6, 7]. Therefore, AlTiN based superlattice coatings, which have been already extensively investigated [3, 8, 9], are good candidates for cutting difficult-to-machine materials such as nickel based alloys.

The purpose of this study was to control multilayer or superlattice coatings deposition on cemented carbide cutting tools in an industrially sized arc-PVD system. Morphology and micro-structure of TiN-(Al,Ti)N, TiN-CrN and CrN-(Al,Ti)N as deposited coatings with different periods \( \lambda \) were also investigated in order to be correlated with tribological behaviours and cutting performances. The main goal was to establish the relationships between deposition conditions, micro-structure, hardness and wear/friction measurements and, ultimately, the Ni based alloys cutting performances.

2. Experimental details

All the coatings were deposited by cathodic arc evaporation in an IMD 700 Plassys system equipped with 4 random arc BMI sources (100 mm in diameter) and with a threefold rotating substrate holder [10]. AITiN, TiN and CrN were elaborated from AlTiN (60:40 at.%), pure Ti and pure Cr targets. Hardened M2 HSS and glass substrates were cleaned in a heated alkaline solution degreased in acetone and rinsed in alcohol prior to deposition. A first ion etching is performed at 3 Pa in pure argon (bias: - 600 V) during 30 min prior to a metal etching atmosphere, arc intensity and substrate bias voltage are 1Pa, 100 A and – 150 V. Deposition temperature is kept constant at 400 °C.

TiN or AITiN are deposited as top layer for TiN/CrN or TiN/AlTiN and CrN/AlTiN coatings respectively and the interlayer is a thin pure metal (Cr or Ti) film. This interface, which has a intermediate hardness, allows accommodation between the substrate (cemented carbide, WC-Co) and the hard coating and thus favours adhesion. Two face to face pairs of cathodes with Ti/AITi; Cr/AITi and Ti/Cr targets are used to deposit multilayered coatings with different periods from bilayered film to multilayered coatings (12 to 52 layers) and then to the superlattice structure (figure 1).

Fig. 1. Scheme of the different coatings architectures. The total thickness is 3 µm.

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The total thickness is kept constant at 3 μm. The period \( \lambda \) is controlled via the speed of the turntable (700 mm in diameter) of the threefold rotation substrate holder. In this study, a two rotations mode is used to coat cemented carbide inserts. For nanolayered coatings deposition, the four targets are evaporated simultaneously and the speed of turntable rotation is in the range 1.5 to 4 rpm. For multilayered coatings, the targets are evaporated alternatively and the period is controlled via evaporation duration of each material. Interface between each elementary layer of multilayered coatings is a mixture of the two elements in order to ensure a good cohesion.

X-ray diffraction is performed by means of a D8 advance Bruker type goniometer (\( \lambda \), CuK\( \alpha \) = 1.5418 Angstrom) in the \( \theta / 2\theta \) mode. Scanning electron Microscopy observations are made in a Philips XL30.

Coatings have been observed by conventional transmission electron microscopy (TEM) on a Jeol-2000FX microscope (200 kV). The cross-section TEM samples have been prepared by mechanical grinding and polishing (down to 30 μm) followed by Ar\(^+\) ion milling on a Fischione 1010 polisher (accelerating voltage of 4 kV with ion current of 5 mA).

Coated tools were compared to the tools actually used in production. First, instrumented tool holder was used in order to select precisely which new coating gave the best results by measuring cutting force and flank wear. In a second step, the best coating selected from previous tests was compared to the cutting tools of the market by analyzing tool wear and measuring life time.

3. Results and discussion

3.1 Microstructure

3.1.1 X-ray diffraction

TiN-(Al,Ti)N, TiN-CrN and CrN-(Al,Ti)N systems tend to lead to superlattice microstructure when the two materials are modulated with small periods because they have the same face-centered cubic NaCl structure with similar lattice constant.

Figure 2 and 3 show X-ray diffraction spectra from TiN/CrN and TiN/AlTiN systems respectively for different periods.

![Fig. 2](image)

**Fig. 2.** X-Ray Diffraction patterns of TiN/CrN coatings deposited on HSS substrates with various periods: substrate (a); \( \lambda = 230 \) nm (b); \( \lambda = 32 \) nm (c); \( \lambda = 13 \) nm (d); \( \lambda = 9 \) nm (e); \( \lambda = 6.5 \) nm (f).

For higher periods, the coatings are mixtures of the two fcc phases derived from the two materials whereas X-ray diffraction spectra reveal the superlattice micro-structure for lower periods. Coatings are single-phased fcc below 10 nm for both system and the (111) reflection is the average of the (111) reflections positions of the two nitrides. Nevertheless, since the difference between the lattice constants is higher for the TiN/CrN system (0.424 and 0.414 nm for TiN and CrN respectively) than for the TiN/AlTiN system (0.424 and 0.419 nm for TiN and AlTiN – 60/40 at.% respectively), the growth of a superlattice phase is clear for TiN/AlTiN with a 41 nm period (figure 3b) whereas it is not the case for the coating TiN/CrN with a 13 nm period (figure 2d). All the microstructures in the CrN/AlTiN system were found to be fcc NaCl single phased whatever the period. Nevertheless, it was impossible to resolve each nitride phases reflections.

![Fig. 3](image)

**Fig. 3** X-Ray Diffraction patterns of TiN/AlTiN coatings deposited on HSS substrates with various periods: \( \lambda = 225 \) nm (a); \( \lambda = 41 \) nm (b); \( \lambda = 15 \) nm (c); \( \lambda = 7 \) nm (d).

Preferential orientations in these nano-scale multilayer coatings are similar for both AlTiN based systems: (111) plane is parallel to the substrate surface. According to D.B. Lewis and al. [11], if the strain energy predominates as in the case of thick coatings columnar growth, (111) preferential orientation is expected because this plane has the lowest packing density (compared to (100) and (220) planes) and the lowest strain energy. Orientation in TiN/CrN films is not clear because of the low crystallinity in this system.

X-ray diffraction data can also be used to calculate nanolayered films period [12, 13, 14, 15, 16, 17, 18, 19], when it is performed at low scan rates (5 sec per 0.02° in this study). Spectra for different nanolayered TiN/AlTiN periods reveal satellite peaks on both sides of the (111) reflection (figure 4). The position of the \( m \)th order satellite peak is given by:

\[
\sin \theta_\pm = \sin \theta_B \pm \frac{m \lambda}{2 \lambda}
\]

Where \( \theta_B \) is the main Bragg reflection position, \( \lambda \) is the bilayer period, \( I \) the X-ray wavelength and \( \theta_\pm \) the position of \( m \)th negative or positive satellite peak. Calculated values and X-ray diffraction measurements were found to be similar.
3.1.2 Transition Electron Microscopy

Bright field micrographs of TiN/AlTiN nanolayered films are shown figure 5. A very good correlation between measured periods on micrographs and calculated values from X-ray diffraction spectra was found. The superlattice microstructure consists of columnar grains in the growth direction through interfaces. The grains growth starts from the TiN interlayer underneath (figure 5a) and the electrons diffraction pattern in microdiffraction mode (figure 5b) confirms the oriented growth of the superlattice microstructure: the [111] direction is perpendicular to the substrate surface. Furthermore, observation of the central spot of electron diffraction patterns shows satellite spots associated with the principal modulation as the satellite peaks in X-ray diffraction.

3.2 Cutting tests

Standard cutting tests (cutting force and flank wear measurements) were carried out in order to evaluate the performance of each coating. Coatings elaborated in this study are in a first step compared each others. In a second step the best coated tool is compared with the tool actually used in production.

Figure 6 shows the positions of each coated tool on a diagram cutting force; flank wear. What ever the tool used as substrate (890 or H13A), uncoated tools have the higher couple (flank wear; cutting force). Cutting force and flank wear are much linked because an increase of cutting force is mainly due to wear on the tool (mainly flank wear in this case). It is very interesting to note that TiN single layer allows a decrease of flank wear (by reducing wear rate) but have not a big influence on cutting force. This could be mainly due to its tribological behaviour (high friction coefficient) [1].

For both roughing and finishing steps, nanolayers TiN/AlTiN seem to be the best coatings: the couples (flank wear; cutting force) are very low for these coated tools and as a consequence, this coating give the largest cutting tools life time among all coated tools elaborated in this study. The association of a very high hardness and very good tribological performances [1] (even at high temperature with AlTiN based coatings) could explain these good results.

Then, nanolayer TiN/AlTiN coated tool is compared to commercial coating solutions and tool used in production (figure 7). Multilayer A, B and C are commercial solutions: TiN/TiAlN multilayer coatings. Multilayers B & C seem to be efficient comparatively to uncoated solutions: a lower cutting force is observed for roughing step (figure 7) and a lower flank wear than uncoated tools. Multilayer A seems to have a worst behaviour than uncoated solution because of to strong etching step during the elaboration process. As a consequence, the interface between WC-Co substrate and coating is brittle and strong failures on the tool edge are observed during machining.
Nanolayer TiN/AlTiN elaborated in this study is the best solution of coating for roughing and finishing step (see Figure 6 for finishing steps): this coating induces the lowest cutting force on the tool and limits built-up edge phenomenon. This coating is also very efficient to avoid strong abrasive wear (low flank wear observed in roughing steps). High temperature resistance as well as good tribological behaviours of AlTiN coating prevent sticking phenomena on rake face. As a consequence, this coating gave the best result in terms of cutting tool life time but also in terms of machining quality.

4. Conclusion

Polycrystalline TiN/AlTiN; TiN/CrN and CrN/AlTiN multilayer coatings have been deposited with different periods by the cathodic arc evaporation technique in an industrially sized reactor. A simple method is used to predict the nanolayered films period in the case of coatings deposited on cemented carbide insert which are on a twofold rotation substrate holder. The predictions are in good agreement with the X-ray measurements and TEM observations. For higher periods, coatings are a mixture of both fcc nitride phases whereas for low periods, coatings are fcc superlattice single phased. Except for CrN/TiN nanolayered films which have a low crystallinity, the other superlattice films grow with the (111) plane parallel to the substrate surface. Chromium is better than titanium for the metallic ions etching in terms of coating surface roughness. Defects density and surface roughness are also higher for AlTiN based species than for CrN/TiN films. Superlattice coatings based on AlTiN seems to be good candidate for superalloys machining by limiting abrasive wear and avoiding built up edge phenomenon frequently encountered for this type of machining. Cutting tools life time was drastically increased comparatively to CVD coated commercial solutions by applying superlattice TiN-AlTiN coatings.

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References