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Physical and Mechanical Properties of CrAlN and CrSiN Ternary Systems for Wood Machining Applications

Yacine Benlatreche, Corinne Nouveau,* Hamid Aknouche, Luc Imhoff, Nicolas Martin, Joseph Gavaille, Christophe Rousselot, Jean-Yves Rauch, David Pilloud

Nowadays, almost all the cutting tools in metal machining are protected with a surface treatment. Nevertheless, this is not the case in wood machining where no tools are protected, except by thermal treatments, and so they present a previous wear because of the use of steel or carbide materials in milling, sawing, routing, etc. During these processes, the tools are particularly exposed to abrasive and shock wear. To enhance their wear resistance, one solution is to protect them with hard coatings. The present study deals with the development of ternary systems (CrAlN and CrSiN) obtained by two PVD different magnetron sputtering systems ['A' (laboratory where CrAlN layers have been obtained) and 'B' (laboratory where CrSiN layers have been obtained) in the following text] on carbide WC–Co tools used in second transformation of wood to be compared to the binary CrN one. CrAlN and CrSiN films were deposited with different Al and Si contents, respectively, in order to check the effect of the additive element (Al or Si) on the different properties of the Cr–N system. The different coatings were characterized by SEM and EDS for thickness measurements, morphology and composition analyses, respectively, by nanoindentation for hardness and Young's modulus measurements and by pin-on-disc to determine their friction coefficient. Routing of medium density fibreboard (MDF) was realized employing untreated or modified carbide WC–Co tools in order to compare their wear resistance. We observed that the Al and the Si addition improved the hardness and the Young's modulus of the Cr–N system ('A': 29 and 410 GPa, respectively, 'B': 18 and 280 GPa, respectively). Indeed, the hardness values are 15–36 GPa for CrAlN and 15–24 GPa for CrSiN coatings. Besides, the Young's modulus values are 331–520 GPa for CrAlN and 260–320 GPa for CrSiN coatings. The friction coefficient of the CrAlN layers varied between 0.6 and 0.7 and it increased slightly with the Al content. For the CrSiN coatings, the friction coefficient was lower and about 0.4. In both cases, the CrN layers 'A' and 'B' presented similar friction coefficient than CrAlN and CrSiN, respectively. During the routing of MDF, the CrN 'A' coating has a similar wear behaviour than the optimized CrAlN one (5 at.% of Al) while the optimized CrSiN coating (1.2 at.% of Si) showed a better behaviour against wear than the CrN 'B' one. The wear resistance of CrAlN- and CrSiN-coated carbide tools decreased when the Al and Si contents increased.

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Table 1. Deposition conditions and properties of the CrN 'A' and CrAlN coatings.

Coating	Al bias V	Cr bias V	Al at.%	Thickness μm	Hardness GPa	Young's modulus GPa	Friction coefficient
CrN 'A'	-0	-900	0	1.76	29	410	0.55
CrAlN	-300		5	2.1	26	410	0.6
	-500		13	2.5	26	410	0.65
	-700		28	2.7	23	380	0.7
	-900		30	3	35	460	0.65
		-700	44	2	15	331	—
		-500	51	2.1	36	520	—

Introduction

In the last decade, the improvement of the service life and the wear resistance of cutting tools used in first and second transformation of wood became an important research topic.^[1,2] Actually, compared to the tools used in metal machining, wood machining requires special attention due to the lack of cooling agents during the cutting process as well as the sensitivity of the wood material to thermal and mechanical factors. Various surface treatments based on the deposition of hard coatings like diamond or a-DLC,^[3] chromium and binary systems like VC,^[4] TiN and CrN coatings^[5] have already been developed in order to use them in wood machining.

Furthermore, it has been shown that the addition of a third element as Al, Mo, C, V and W to binary systems as CrN and TiN in order to enhance their intrinsic properties has been studied intensively.^[6,7] It has been reported that the addition of Al in CrN and TiN films modified their structural, mechanical and tribological properties.^[8] Besides, Lee et al.^[9] and Kim et al.^[10] showed that the addition of Si in CrN films increased the hardness above 24 GPa and decreased the friction coefficient up to 0.2.

In the present work, CrAlN and CrSiN films with different Al and Si contents have been deposited by two different magnetron sputtering techniques in view to first study the effect of Al and Si contents on physicochemical, mechanical

and tribological properties of the Cr-N system. Second, the optimized coatings were deposited on carbide tools for routing of MDF to improve their wear resistance.

Experimental Part

Coatings Deposition

CrN (called 'A') and CrAlN coatings were deposited using an RF dual magnetron sputtering system (NORDIKO type 3500-13.56 MHz). These films were deposited using two pure targets Cr (99.995%) and Al (99.999%) (101.6 mm of diameter and 3 mm thick). The targets/substrates distance was 90 mm. Our objective was to check the effect of Al content on CrAlN properties. Table 1 summarizes the deposition conditions and the properties of the CrN 'A' and CrAlN layers. The working pressure was 0.4 Pa, the N₂/Ar ratio was 20:80 and the deposition time was 90 min.

In the second part of this study, CrN (called 'B') and CrSiN films were deposited using DC/RF dual magnetron sputtering system (AC450). In order to study the Si content effect on the CrSiN properties, two targets (50.8 mm of diameter) of Cr (99.99%) and Si (99.99%) were used. A DC and RF (13.56 MHz) generator were used to polarize the Cr and Si targets, respectively. The Cr target/substrates distance was 80 mm while the Si target/substrates was only 70 mm. Table 2 summarizes the deposition conditions and the properties of the CrSiN films. The DC applied voltage on the Cr target was -380 V, the working pressure was 0.4 Pa, the N₂/Ar ratio was 40:60 and the deposition time was 105 min.

Table 2. Deposition conditions and properties of the CrN 'B' and CrSiN films.

Coating	Si bias V	Si at.%	Thickness μm	Hardness GPa	Young's modulus GPa	Friction coefficient
CrN 'B'	-0	0	0.59	18	280	0.43
CrSiN	-183	1.2	0.77	16.5	260	0.44
	-256	5	0.55	26	350	0.35
	-317	7.5	0.65	28	470	0.3
	-440	14.3	0.68	24	320	0.4

To determine the physicochemical, mechanical and tribological properties of the layers, different kinds of substrates were employed. The CrN 'A' and CrAlN films were deposited on silicon $10 \times 10 \text{ mm}^2$ (for thickness measurements), on SiO_2 samples $10 \times 10 \text{ mm}^2$ [for nanoindentation tests: NHT from CSM Instruments with a Berkovitch indenter, sinus mode employed (1 Hz of frequency and 1 mN of amplitude), max load 10 mN, load and unload rate $5 \text{ mN} \cdot \text{min}^{-1}$], on 90CrMoV8 steel ('La Forézienne-MFLS' trademark, France) samples $20 \times 20 \text{ mm}^2$ frequently used in wood machining to realize pin-on-disc tests (applied load of 5 N, 100Cr6 ball of 5 mm diameter with an alternative friction each 8 s and a test duration of 10 min). The CrN 'A' and CrAlN coatings' thickness and compositions were determined by SEM (JEOL JSM-5900 LV) equipped with an energy dispersive spectrometry (EDS).

The CrN 'B' and CrSiN films' thickness was measured with a 2D profilometer (Dektak 3030). Hardness measurement was performed using a CSM Nano Hardness Tester with a Berkovich tip in a dynamical mode analysis (maximum load reached of 10 mN, the sinus amplitude was fixed at 1 mN with a frequency of 1 Hz). Wear tests were realized by using a conventional pin-on-disc tribometer (X85WMoCrV6542 steel as substrate and alumina ball (5 mm of diameter) as counterpart, with a normal load of 25 N). Before deposition, all the targets and the samples were etched in Ar plasma by RF and DC discharges, respectively.

Routing of MDF

In order to study the addition of Al on the CrN 'A' (CrAlN layers) and the addition of Si on the CrN 'B' (CrSiN layers) effect on their wear resistance, cemented tungsten carbide inserts (WC-2% Co) (50 mm long, 12 mm width and 1.5 mm thick, with a cutting edge's angle of 55°) were coated on both side. The experiments were performed using a three-axis industrial RECORD1 SCM S.p.A CNC router. The tool-holder, the routing procedure and the WC-Co tool's geometry are detailed in a previous study.^[11]

The wood material employed was medium density fibreboard (MDF); $670 \text{ kg} \cdot \text{m}^{-3}$ of volume-density; we chose this material because of its abrasive properties. Indeed the MDF panels contain in addition to wood fibre a formaldehyde adhesive, silicon and other abrasive elements. The MDF platelets were 600 mm long, 200 mm width and 19 mm thick. For each machining test, the cutting conditions were 2 mm as radial engagement and 5 mm as axial engagement along the platelet length. These conditions give 170 m of linear cutting distance. The final distance of MDF cut was 1.530 m, which requires nine tests. The cutting rate was $38 \text{ m} \cdot \text{s}^{-1}$, the rotation rate was 18 000 rpm and the feed rate was $15 \text{ m} \cdot \text{s}^{-1}$.

The wear quantification was realized at the end of each cutting test and evaluated by measuring the nose width (NW),^[12] using an optical binocular (LEICA MZ12) equipped with a digital camera (Sony 3CCD IRISS) and the 'Lida' software. This method has already been used in previous studies.^[13,14] Several measurements were made along the cutting edge and an average wear value was calculated.

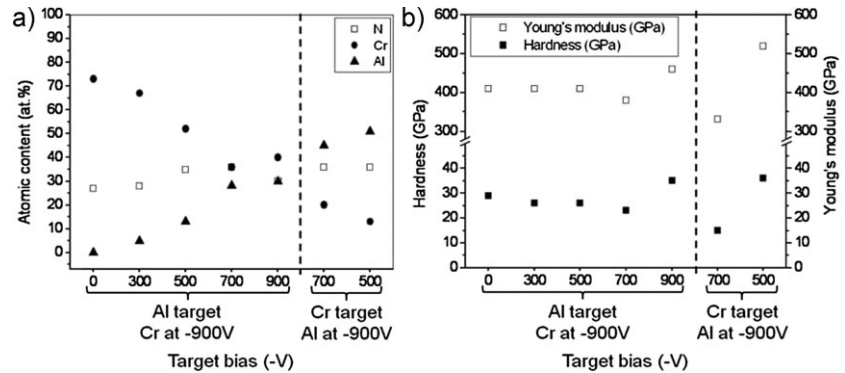


Figure 1. CrN 'A' and CrAlN composition (a), hardness and Young's modulus (b) as a function of Al and Cr applied voltages.

Results and Discussion

CrN 'A' and CrAlN Films

Figure 1(a) presents the CrN 'A' and CrAlN coatings composition as a function of Cr and Al target bias. The Al content in CrAlN coatings vary between 0 and 51 at.%. It is noteworthy that Al content increases with the Al target bias and the maximum value was about 30 at.% obtained at -900 V . When we vary the Cr target bias, we obtained 51 at.% of Al for -500 V . This can be explained by the lower sputtering rate of Al in comparison to the Cr one.^[15]

The hardness and Young's modulus of the CrN 'A' and CrAlN coatings as a function of Cr and Al target bias are represented in Figure 1(b). They varied between 15 and 36 GPa and 331 and 520 GPa, respectively. The CrN coating showed a high hardness and a high Young's modulus of around 29 and 410 GPa, respectively. These values are higher than those of the conventional Cr-N system (18 and 220 GPa, respectively) obtained by Ding et al.^[16] The hardness and Young's modulus decrease slightly for the small Al content before increasing up to 35 and 460 GPa, respectively, at -900 V of bias on both Cr and Al targets. Similar values have been already obtained in previous studies.^[16,17] The maximum values of hardness and Young's modulus were obtained for 30 and 51 at.% of Al content, this can be explained by the formation of a solid solution due to the substitution of Cr atoms by Al ones.^[16] The smallest values of hardness and Young's modulus were 15 and 331 GPa, respectively, these values were obtained for 44 at.% of Al content. Indeed, this Al content has been obtained with a bias of -700 V on the Cr target and -900 V on the Al one. So, when we change the Cr bias from -900 to -700 V , the energy of the incident atoms on the substrate is lower, and the defects' number created during deposition decreases. As a consequence the hardness also decreases because it depends on this defects' proportion. We also have two hardening effects, one is the solid solution formation and the second is the number of created defects during the deposition process.

The friction coefficient of CrN 'A' coating is about 0.55, it increases and vary between 0.6 and 0.7 for CrAlN films (Table 1). Similar values of the friction coefficient have been obtained in previous studies.^[16] According to Bobzin et al.,^[18] the AlN films have a higher friction coefficient than CrN and CrAlN ones, so we can suppose that the Al addition is responsible for the formation of AlN phase which could explain the high values of friction coefficient of CrAlN coatings.

CrN 'B' and CrSiN Films

Table 2 presents the CrN 'B' and CrSiN properties for different bias (0, -183, -256, -317 and -440 V) on the Si target in order to vary its contents. The Cr target bias was fixed at -380 V. Figure 2(a) shows the composition of CrN 'B' and CrSiN coatings as a function of the Si target bias. We observe that the Si content increases slightly with the Si target bias, indeed it varied between 1.2 and 14.3 at.% when the Si target bias increased from -182 to -440 V. This slight increase in Si content can be explained by the Si sputtering rate which is lower (three times) than that of Cr.^[15] We can also observe the presence of oxygen which indicates that the deposition atmosphere is pollute.

Figure 2(b) represents the hardness and the Young's modulus of CrN 'B' and CrSiN coatings as a function of the Si target bias. The hardness and Young's modulus of CrN 'B' coating are about 18 and 280 GPa, respectively. The CrSiN layers present high hardness and Young's modulus. They varied between 16.5 and 28 GPa and 260 and 470 GPa, respectively. Same results have been obtained in previous studies.^[9,10] The hardness and Young's modulus increase from 16.5 to 28 GPa and 260 to 470 GPa, respectively, when the Si content increases from 1.2 to 7.5 at.% then they decrease to 24 and to 320 GPa, respectively for 14.3 at.% of Si content. Lee et al.^[9] reported the presence of two hardening regions as a function of Si content in CrSiN films deposited by magnetron sputtering. Hardening at low Si content was attributed to solid solution formation, while that at higher Si content could be explained by the formation of a

nanocomposite but this needs to be verified by further works. Moreover, Park et al.^[19] found an increase in the hardness of the CrSiN films prepared by a hybrid process combining arc deposition of Cr and magnetron sputtering of Si, indicating the highest hardness value for 9 at.% of Si content. Besides, Kang et al.^[20] also explained the increase of the hardness by the formation of a solid solution due to the dissolution of Si atoms and they explained the hardness decreasing for the highest values of Si content (up to 9 at.%) by the increase in volume fraction of amorphous Si₃N₄ phase.

The friction coefficient of the CrN 'B' and CrSiN films is about 0.3–0.44. As a comparison, Park et al.^[19] and Shin et al.^[21] showed that the friction coefficient decreased from 0.3 to 0.2 when the Si content increased from 9.3 to 12.5 at.% during pin-on-disc tests (steel ball). Shin et al.^[21] explained the decrease in friction coefficient with the increase of Si contents by the formation of a smoother surface due to codeposition of amorphous phase and by a tribo-chemical reaction, which often takes place in many ceramics, e.g. Si₃N₄ reacts with H₂O to produce SiO₂ or Si(OH)₂ tribo-layer. These products of SiO₂ and Si(OH)₂ are known as self-lubricating layers.

Routing of MDF With Untreated and Modified Carbide Tools

In the following, we present the routing of MDF results obtained with CrN 'A' and 'B', all the CrAlN [Figure 3(a)] and all the CrSiN [Figure 3(b)] layers.

Figure 3(a) shows the variation of the nose width as a function of the cutting length of MDF and the Al content in the CrN 'A' and CrAlN layers. We observe that the CrN 'A' coating presented a similar behaviour as the CrAlN coating obtained with 5 at.% of Al. The nose width increases with the Al content. Indeed, the CrAlN-coated tool deposited with the lower Al content (5 at.%) presents the best wear resistance.

In the case of CrN 'B' and CrSiN (just the coatings with 1.2 and with 14.3 at.% of Si were tested) coated carbide tools, as the CrAlN layers, the nose width increases with the Si content. Indeed, the optimal CrSiN layer is the one obtained with 1.2 at.% of Si which protects better the WC tool than the CrN 'B'.

In this work, we studied the effect of Al and Si contents on the Cr–N system properties. It seems that the addition of Al and Si improves the hardness and Young's modulus of the Cr–N system but not the friction coefficient. Only the addition of Si permitted to increase the wear resistance during MDF routing. MDF routing is a specific process

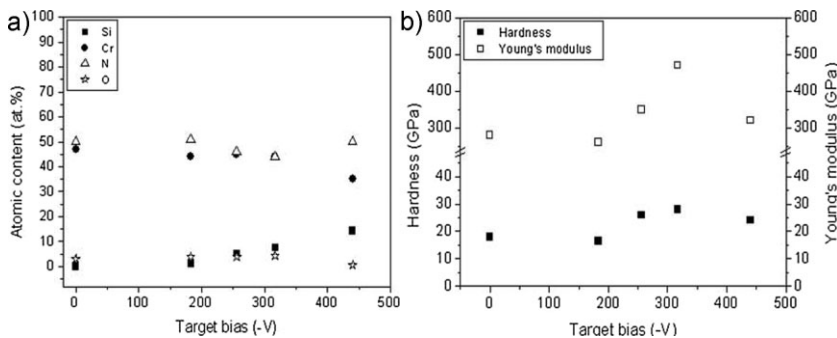


Figure 2. CrN 'B' and CrSiN composition (a), hardness and Young's modulus (b) as a function of Si applied voltage.

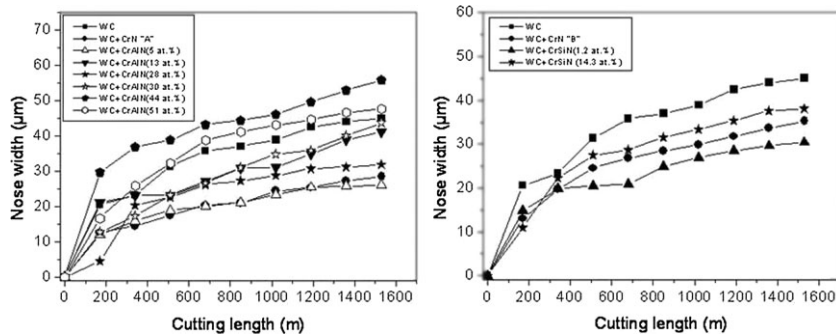


Figure 3. Nose width variation of: (a) CrN 'A' and CrAlN- (b) CrN 'B' and CrSiN-coated WC-Co tools as a function of cutting length and Al or Si contents.

completely different from metal cutting. Actually, the composite material (MDF) is not hard enough to cause the wear of WC tools. The high vibration force resulting during the machining of this material causes the detachment of WC grains from the tool edge. So, regarding the specificity of this process, we cannot conclude concerning the relation between the friction coefficient and the wear resistance during MDF routing of CrN 'A' or 'B', CrAlN and CrSiN-coated tools.

Conclusion

CrAlN and CrSiN layers were synthesized by magnetron sputtering but with two different systems. The first part of this work was devoted to study the effect of Al and Si contents on hardness, Young's modulus and friction coefficient of the Cr-N system. In the second part, we studied the improvement of the wear resistance of WC tools using CrN obtained with two different PVD techniques ('A' and 'B'), CrAlN and CrSiN coatings during routing of MDF.

From the above results we can conclude that:

- The addition of Al and Si permitted to enhance the hardness and Young's modulus of the Cr-N system. Two phenomena can be responsible for the hardness and Young's modulus improvement when we vary the Al or Si content; the first one is the solid solution formation by substitution of Cr atoms by Al or Si ones, while the second one is the defects' number created as a result of the atomic bombardment.
- The friction coefficient increases with Al and Si addition.
- During the routing of MDF, CrN 'A' protects the WC tools similarly than CrAlN coatings obtained with 5 at.% of Al and the wear resistance decreases when the Al content increases.
- During the routing of MDF, CrSiN obtained with the lower at% of Si protects the WC tools better than CrN 'B' coatings.

- The wear resistance decreases when Si content increases.

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