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TECHNICAL PAPER

Improvement of coating adhesion on cemented carbide tools by plasma etching

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Abstract Surface treatments are one of the main factors to control adhesion between coating and substrate on a cutting tool. Poor coating adhesion on the substrate accelerates the wear progress and decreases tool lifetime due to the pull-out and release of hard abrasive particles between the tool rake or flank face and the workpiece. Mechanical and chemical substrate treatments are used in order to improve the coating adhesion. This study evaluated and compared the chemical and plasma etching effectiveness in the improvement of substrate-coating adhesion, and consequently, tool life of PVD-coated cemented carbide tools. The plasma etching was performed in plasma reactors in which the cations produced collide with the samples and remove from the surface atoms or molecules modifying the topography. In the chemical etching, acid and alkaline solutions were used to remove tool surface material, changing its initial roughness and chemical composition. After these surface treatments, the samples were PVD coated with (Ti,Al)N. To ascertain the effectiveness of the surface treatment, Rockwell B indentation and machining experiments were performed on treated and untreated tools. Tool topographies were analyzed by atomic force microscopy (AFM) and flank wear lands were evaluated by scanning electronic microscopy (SEM). The plasma-treated tool showed better performance in the indentation and turning tests. Therefore, the chemical etching-treated tool showed the highest roughness, but the coating adhesion was poor due to chemical changes on substrate surface. Furthermore, good anchoring is not influenced only by roughness, it also depends on the substrate surface chemical nature.

Keywords Plasma etching · Chemical etching · Coated tools · Coating adhesion

1 Introduction

In manufacturing industry, productivity increases the demands on cutting processes that can be addressed, among others, through the application of coated tools. Coated cemented carbide is a material with several applications, mainly in cutting tools for machining of different metallic alloys. In addition, the interaction between substrate and coating materials is an essential factor for increasing tool lifetime. According to Bouzakis et al. [1], the most extensively investigated coating system is (Ti,Al)N, due to the ease of deposition parameters and material contents manipulation, as well as its potential to increase the cutting tool performance.

Adhesion between coating and substrate depends significantly on surface treatments [1, 2]. The tool's surface characteristics can be modified by different types of surface treatment, which improves adhesion and, consequently, cutting performance. Therefore, coating adhesion is strongly influenced by tool's surface characteristics, as texture features and roughness. Another relevant characteristic related to the cemented carbide is the presence of cobalt in its composition. Cemented carbides contain cobalt as a binder, which provides additional toughness to the tool, although it is harmful to coating adhesion [2–4].

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Several treatments have been employed to change tool substrates before coating application, increasing the interaction between coating and tool surface. They can be classified into chemical processes, mechanical treatments, plasma methods and laser ablation.

Water peening, grinding, micro-blasting were studied as surface treatments, and, the first treatment has improved coating adhesion values of TiN and (Ti,Al)N on cemented carbide tools [5–8]. For these coatings and tools, micro-blasted and water-peened substrates have shown improved interface strength in comparison to ground carbides, and consequently, superior wear resistance [8]. Additionally, water peening showed efficiency to improve diamond coating adhesion on the tool's surfaces [9]. These mechanical treatments are applied to cemented carbide substrates and promotion of Co removal from the surface because it is softer than other phases (WC, TiC and TaC) as well the its roughness increase [2].

In addition to mechanical treatments, laser texturing and plasma etching can modify the tool's surfaces. The first treatment was studied by Neves et al. [2, 11] and Arroyo et al. [10], who investigated the influence of laser treatment on the surface morphology, surface structure and coatings cemented carbide adhesion. According to these researches, laser treatment produced a surface with adhesion strength comparable to commercial tools pretreated by micro-sandblasting. Laser of short pulses and high repetition rate generates a surface texturing that improves coating adhesion [12]. This treatment practically does not cause thermal damage of adjacent zone, but cobalt vaporizes due to energy absorption of tools surface and its high vapor pressures. In the second treatment, surface modification involves excited species generated by the interaction of the plasma with the solid substrate [13–16]. This process promoted a physical and/or chemical first few molecular layers modification on the surface without changing the properties of the bulk material [13, 14]. Barshilla et al. [13] verified that the pretreatment with in situ Ar + H_2 plasma is an effective method for removing oxide/contaminant layers from mild steel substrate surface and it also induced sufficient surface roughness, which is favorable for mechanical interlocking of the PVD coatings.

An efficient treatment to eliminate or reduce Co binder negative effect on the tool's surfaces is chemical etching [17–19]. Selective etching of Co with various acids offers a wide range of different pretreatment methods. This treatment consists in the immersion of tool into chemical solutions, such as Murakami's and Caro's reagents. Time of treatment should be optimized in order to improve the coating adhesion. Lei et al. [20] verified that 10 min of pretreatment with Murakami's reagent exhibits better NCD (nanocrystalline diamond) coating adhesion in drilling tests. Etched zone thickness is important, because remaining porosity in the cemented carbide results in reduced layer adhesion [1]. Haubner et al. [4] proposed a combination of etching method in which a Murakami solution is used to attack the WC and then the remaining cobalt structure is treated with Caro's acid (H_2SO_4 with H_2O_2). Chemical etching is widely used for diamond coatings.

In this work, plasma and chemical etching were used to prepare the substrate surface of cemented carbide inserted aiming the (Ti,Al)N deposition by PVD. The main objective was to verify if plasma etching was suitable as methods of substrate surface preparation and to compare with widely used treatment, chemical etching. The coating adhesion was evaluated by the Rockwell B indentation and machining tests.

2 Materials and methods

Cemented carbide inserts substrates were used to carry out two techniques of surface treatment (chemical and plasma etchings). The surface treatments were applied before the coating deposition, with the aim to evaluate their influence in adhesion of physical vapor deposition (PVD-(Ti,Al)N) hard thin film.

Commercial uncoated inserts code CCMT 09 T3 04-KF (ISO equivalent K20 grade), without chip-breaker, were employed due to its geometry to make easy the surface treatment. According to tool supplier [21], ISO K20 grade is suitable for machining of gray, nodular and compacted cast irons and, in specific cases, of titanium- or nickel-based super alloys. Atomic force microscopy (AFM) and micro-hardness tests evaluations were performed in order to characterize tool's surfaces. The equipments were an AFM Shimadzu, model SPM 9600; and Shimadzu Micro Hardness Tester, model HMV-G20, with a square pyramidal diamond indenter and equipped with an automatic indentation reading function using a built-in CCD camera. The images, with scan 30 × 30 μm (area of 900 μm^2), were recorded in contact mode; the feedback electronics and the corresponding software were used to keep the V-shaped cantilever at constant deflection measuring the sample's topography. Topography results showed average roughness of 0.16, 1.5 and 0.2 μm in R_a, R_z and R_q scale, respectively. Concerning the inserts hardness, results presented average values of 1750 HV and a standard deviation of 175 HV.

Chemical etching used acid and alkaline solutions with proposal of reacting to main elements of the cemented carbides inserts aiming to superficial modifications. Therefore, acid solution reacts oxidizing the cobalt, and alkaline solution promotes attack on tungsten carbide grains. After some preliminary trails, two solutions were established for the process: (a) alkaline solution—Murakami (10 g of $K_3(Fe(CN)_6)$ + 10 g of KOH + 100 ml H_2O)

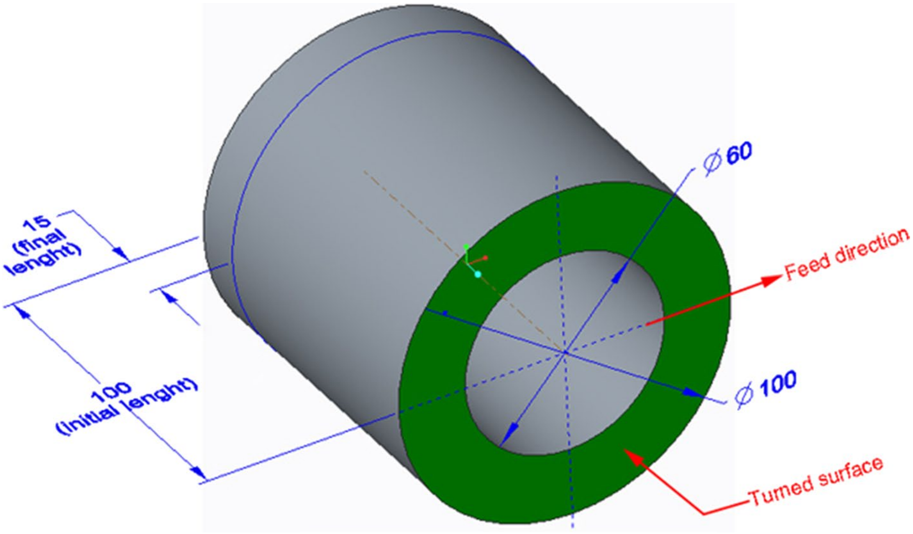


Fig. 1 Details of geometry and dimensions of workpiece

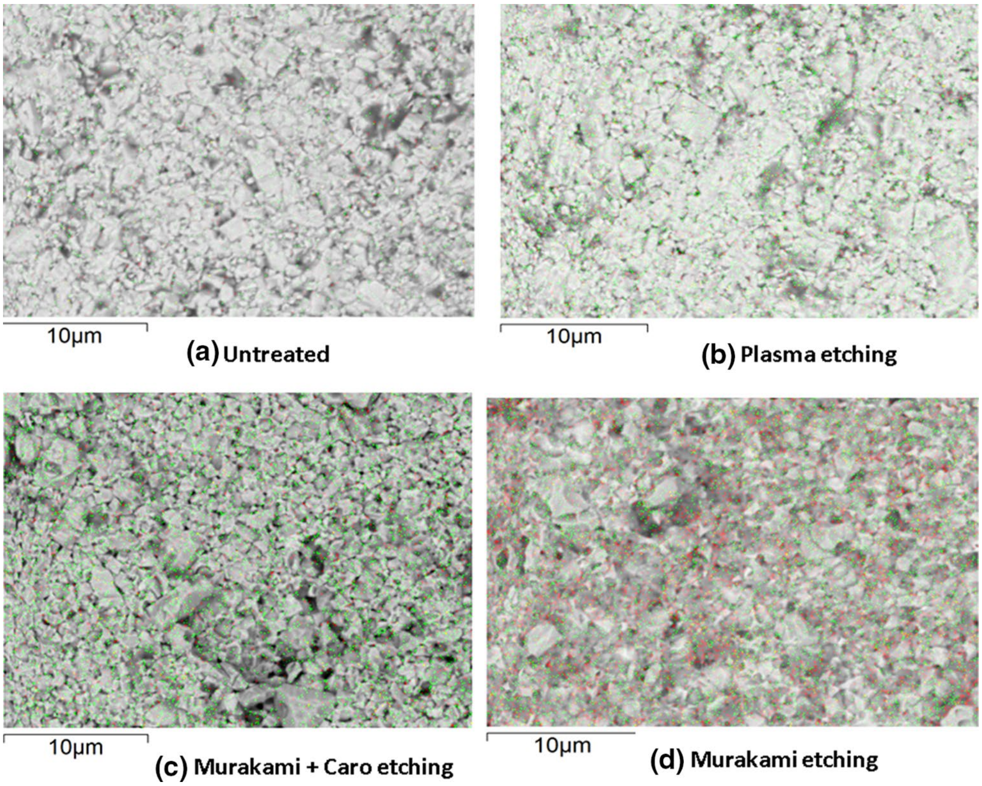
for 15 min, followed by Caro's acid solution (3.5 % vol. H_2SO_4 + 96.5 % vol. H_2O_2) for 20 s; (b) only alkaline solution—Murakami (10 g of $K_3(Fe(CN)_6)$ + 10 g of KOH + 100 ml H_2O) for 15 min. After the chemical etching, cemented carbide inserts were ultrasonically cleaned in acetone for 5 min and dried in hot air.

Plasma etching treatment occurred in a cylindrical vacuum chamber (diameter of 300 mm and length of 400 mm), designed and manufactured in the university, based on stainless steel, in which an electron beam of inert gas bombarding the insert surfaces. Vacuum chamber is supported by DC voltage source using 1500 V and 2A of maximum voltage and current, respectively. This process was conducted at a pressure of 60 Pa for 30 min with the samples were introduced making them form a part of the cathode discharge, being heated only by ion bombardment. The substrate temperatures were maintained at 200 °C during the treatment and cathode voltage of 620 V was applied for maintaining a discharge current about 60 mA. The working gas was composed of 50 % Ar (5 sccm) and 50 % of H_2 (5 sccm). Due to bombardment, particles from cemented carbide insert were removed and the superficial conditions modified.

Physical vapor deposition of (Ti,Al)N coating took place in a 5 station adjustable planetary Eifeler Furnace, model Alpha 40 (BodyCote Brasimet Brazil) using an atmosphere with N_2, Ar, CH_4 and H_2. All tools were coated at the same time in order to ensure same coating conditions. According to the information provided by the company, the coatings are multilayer, with a chromium intermediate layer. Also, deposited layer thickness was evaluated using optical inspection of a spherical cap using Calotest equipment coupled to the optical inspection software. Results showed an average layer thickness of 4.4 µm for all coated surfaces. After coating deposition, new AFM analyses were performed on tool surfaces.

Two methods were defined in order to evaluate (Ti,Al) N PVD film adhesion on the treated substrate: indentation and machining tests. Ollendorf et al. [22] stated that appropriate techniques to study adhesion of thin hard coatings are scratch test, bending test, impact test, cavitation test and Rockwell indenter test. In the last technique, a conventional Rockwell hardness test is performed and the damage pattern of the hard coating, around the indent, is evaluated microscopically at a defined magnification. This technique is related as an easy use in an industrial environment. Therefore, indentation test used Rockwell B Panambra durometer, model RASN 810, with preload of 10 kgf, load of 100 kgf and indenters diameter of 1/16 in. The main objective was identifying coating peeling on the treated substrate by chemical and plasma etchings.

Machining tests were carried out in a Romi conventional lathe, model 520, using an orthogonal facing test to assess tool's lifetime. A constant feed (f), depth of cut (a_p) and rotation speed of 0.2 mm/rev, 1 mm and 560 rpm, respectively, were selected during all facing tests. Workpiece material was a tubular high-chromium content gray cast iron (0.5 % wt. Cr and hardness of 230 HB). The initial workpiece dimensions were: external diameter of 100 mm, bore diameter of 60 mm and length of 100 mm (Fig. 1). During the tests, in each workpiece, the length was faced to final dimension of 15 mm (85 mm facing per workpiece). Common criteria for tool life using sintered carbide tools (average width of flank wear of $VB_B = 0.3$ mm), based on ISO-3685 (1993) [23], was performed in optical stereo microscopy with a built-in digital camera and image software. Tool wear measurements were carried out every 3 turned faces. After tool life tests, cutting edges were observed using Hitachi scanning electron microscopy (SEM), model TM3000, equipped with energy dispersive X-ray spectrometer (EDS) in an attempt to understand the wear mechanisms.



(a) Untreated (b) Plasma etching (c) Murakami + Caro etching (d) Murakami etching

Fig. 2 SEM images of rake faces evidencing Co (*red points*) and W (*green points*) content

One experiment consisted of successive facing passes on surface (from smaller to larger diameter, using a constant rotation speed, which ranged from 105 to 175 m/min for 560 rpm, up to the moment the tool reached the end of its life. Each experiment was carried out three times. All experiments used dry cutting.

3 Results and discussion

Surface treatments aim at the modification on the cemented carbide surfaces. They can be chemical or mechanical and affect the tool's surface topography. It will be discussed in this section tool's surface modification promoted chemical and plasma etching and their influence on coating adhesion.

3.1 Substrate surface characterization

Substrate surface characterization involved two aspects: chemical and topographic. The first was analyzed by SEM/EDS. These analyses allowed evaluating changes on the surface morphology as well as changes in chemical composition. The second aspect was determined, using AFM 3D images, focusing on the roughness changes.

3.1.1 Chemical aspects of tool's substrates

Treated surfaces are shown in Fig. 2. The presence and distribution of cobalt (Co) and tungsten (W) atoms was identified in order to evaluate as each treatment influenced chemical composition, aiding in comprehension of coating adhesion. The Co was represented by red points while W for green ones on the SEM surface image. In order to corroborate with EDS image analyses, Fig. 2 presents a quantitative analysis of Co and W perceptual on the insert surface.

SEM analyses of untreated cemented carbide substrate revealed a homogeneous grain structure (Fig. 2a). Average Co concentration on the substrate surface was 3.0 % and its concentration was homogeneous along the insert surface.

Chemical etching can dissolve or select etch the binder phase. Analyzing Fig. 2c, d, Murakami + Caro's and Murakami etching, respectively, it was possible to notice the effect of two chemical solutions on the cemented carbide surface. When tool surface was etched by Murakami solution (Fig. 2d), Co content increased to around 22 % (more red points on surface), indicating that WC grains were attacked. However, different chemical composition is found for Murakami + Caro etching, in which the surface showed a decrease of Co content (0.8 %).These

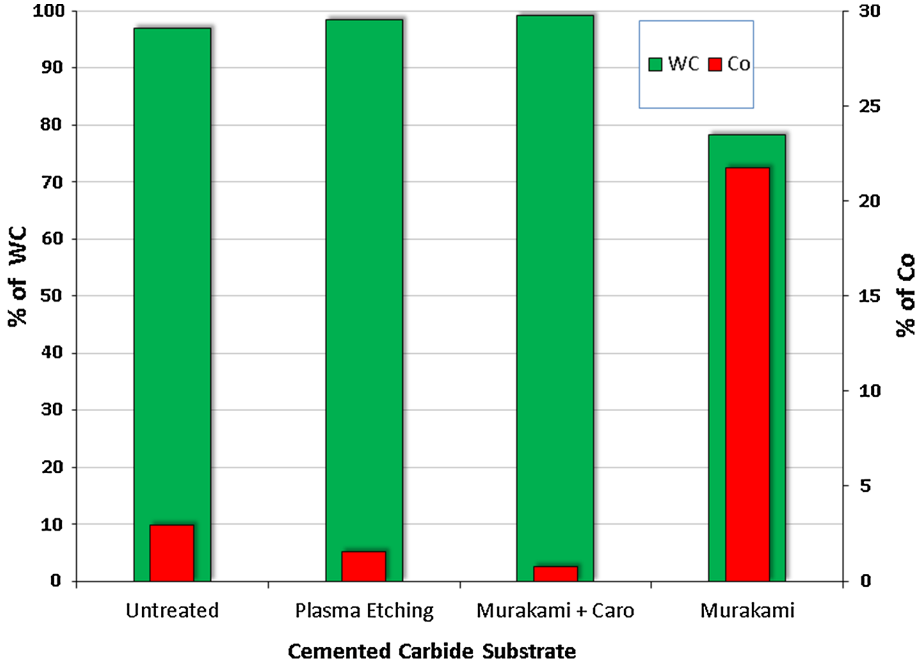


Fig. 3 Influence of surface treatment on surface composition

results agree with Polini [3] statement "Murakami's reagent attacks WC grains, thus roughening the substrate surface. Caro's acid oxidizes the binder to soluble Co^{2+} compounds, thus reducing the surface Co concentration".

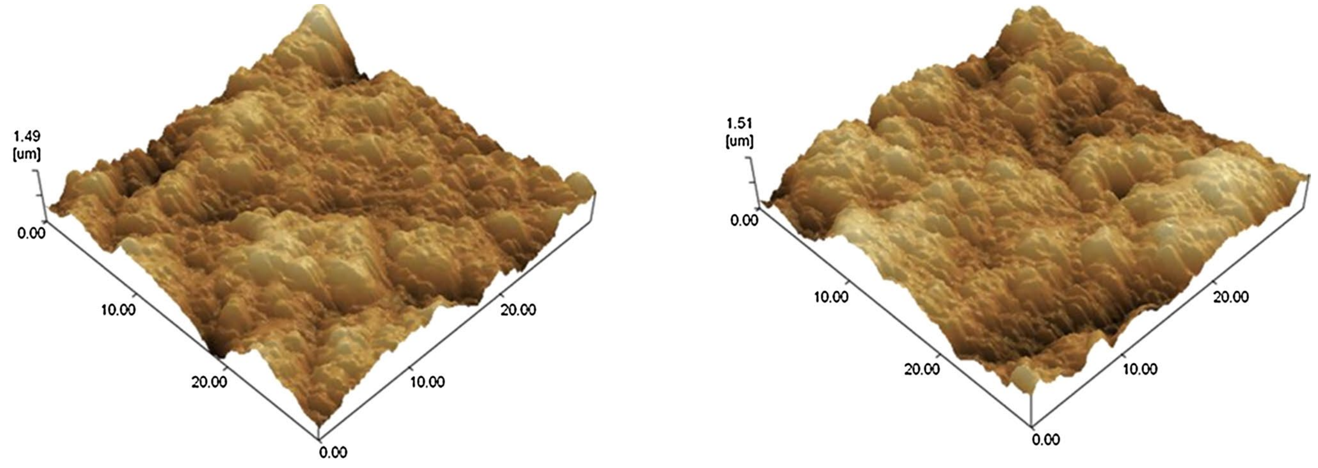
The SEM of plasma-etched surface and changes in chemical composition can be observed in Fig. 2b. Co content decreased in comparison with untreated surface to 54 % (see Fig. 3). This process employs ablation, in which the positive ions bombard the surface. The process can dislodge atoms from the surface [15]. An explanation for this case is given by Co vaporization due to energy absorption. According to Korner et al. [24], the plasma chemical cleaning process based on the argon–hydrogen discharge can be used for "soft" cleaning of different materials.

3.1.2 Topography of the substrate surfaces

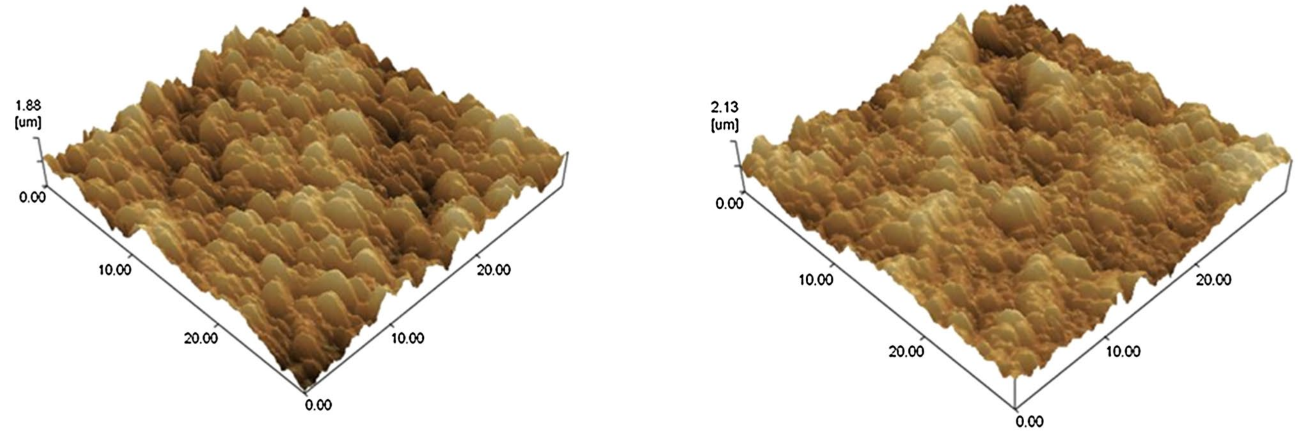
Figure 4 depicts the surface 3D images obtained by AFM corresponding to the untreated WC–Co (a) surface, after plasma etching (b), combined etching (c) and Murakami etching (d). Treated surfaces using Murakami's reagent displayed an increased surface roughness parameter, as observed in Table 1, of R_a equal to 0.225 μm with a partial removal of the original surface features. More peaks and valleys were formed after the Murakami attack due to removal of WC grains, increasing peak densities. Besides, the peaks height and depth valley are more notable, it can be verified by R_p and R_v values or R_z (Table 1). The combined etching (Fig. 4c) reveals a uniform surface with a R_a value of 0.251 μm, the increase in roughness for this treatment is attributed to the initial attack of WC gains by Murakami's solution and after that the acid oxidizes the cobalt binder to soluble Co^{2+} compounds that are removed in ultrasonically wash [17]. In addition to that, the washing may leach weakly bonded WC grains from the surface generating a new surface topography.

From the AFM image of Fig. 4b, it is visible that plasma pretreatment leads to considerable amount of etching on the substrate. It also can be seen that Ar plus H_2-treated substrate developed a rough surface which was higher when compared to untreated tool and lower than chemical-etched substrate (Table 1).

AFM analyses showed an increasing trend in R_a, R_z and R_q values after the treatment surface in comparison with untreated surfaces for all studied treatments, as summarized in Table 1. These results indicate that all of them are suitable to change the surface topography, but chemical etching promotes high increment in roughness values, for all parameters analyzed. A complementary evaluation of the surface topography can be done by ratio between R_p and R_z. R_p parameter is obtained from average peak height in relation to the central line in five consecutive readings; while R_z is calculated by the sum of maximum peak ones (R_p) and maximum valley depths (R_v), in a sampling length. The R_p/R_z ratio is especially important in assessing surface shape, since a ratio greater than 0.5 indicates sharp peaks, whereas values lower than 0.5 indicate a surface with rounded peaks [25]. Based on the ratio (R_p/R_z), the chemical etching besides the high increment in roughness values these treatments produces surface with more rounded peaks can be verified. This fact can be easily seen in AFM 3D images (Fig. 4c, d).



(a) Untreated **(b) Plasma Etching**



(c) Murakami+Caro etching **(d) Murakami etching**

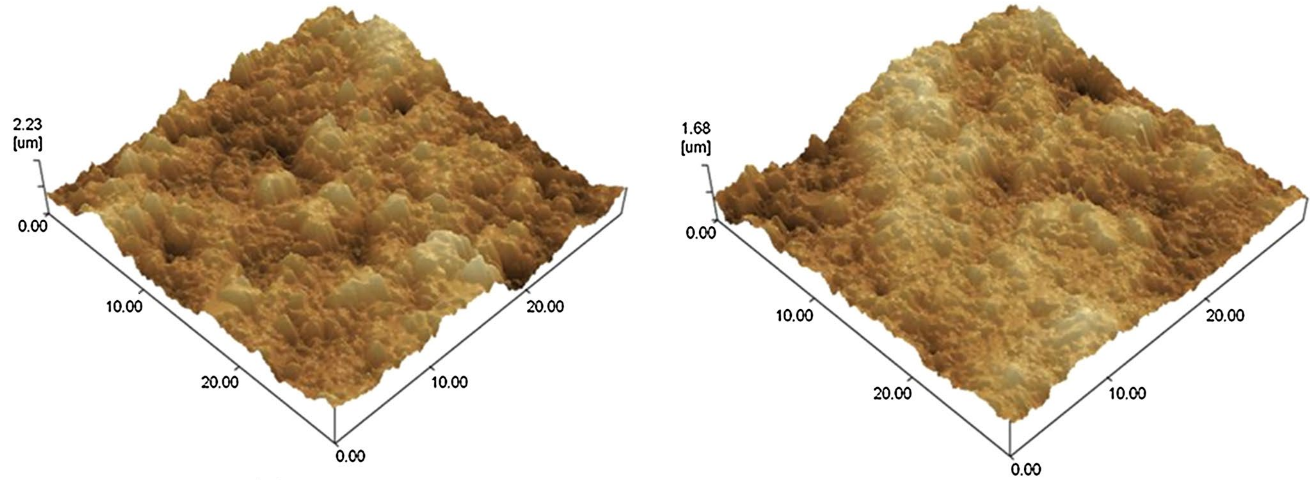
Fig. 4 AFM images of tool surface after treatments

Table 1 Different roughness parameters obtained after surface treatment

Treatment	R_a	R_q	R_z	R_p	R_v	R_p/R_z
Murakami	0.225	0.280	1.800	0.907	0.897	0.504
Murakami + Caro	0.252	0.309	1.990	0.898	1.099	0.451
Plasma	0.211	0.260	1.510	0.729	0.780	1.070
Untreated	0.164	0.208	1.490	0.778	0.714	0.918

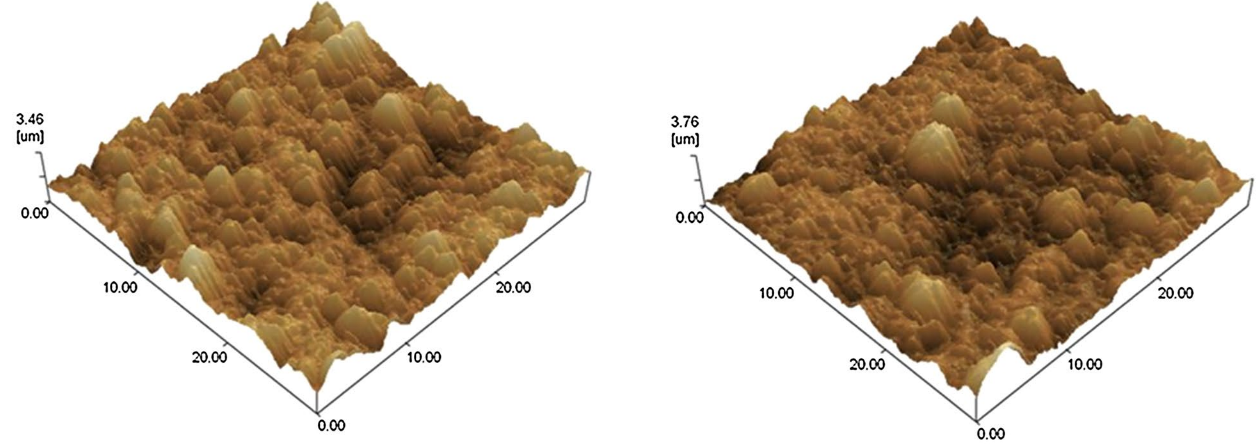
Three-dimensional AFM images of coating inserts after surface treatment are shown in Fig. 5. Based on these images, it is possible to analyze the final topography as well as the coating characteristics; also, the Table 2 shows the roughness values to corroborate the coating deposition analysis. As mentioned above, peaks density and distance between peaks and valleys in chemical-etched surface are higher than plasma-etched and untreated (Fig. 5b, a) insert surfaces, this fact promotes a preferential deposition, in which the coating deposits on the peaks of substrate surface. This deposition type produces coatings with columnar structure, with free spaces between the columns that determines the growth of film less dense with high roughness (Fig. 5c, d). Coating columnar growth is associated to the geometrical shadowing of peaks on the substrate surface that makes the atoms deposition in the valleys of substrate surface difficult [26]. This observation is confirmed by R_p and R_v values in Table 2, where R_p increase is higher than R_v increase, proving the preferential deposition on the peaks.

AFM analyses showed increasing trend in R_a, R_z and R_q values after the coating deposition, except for plasma-etched surface, as summarized in Table 2. However, peak



(a) Untreated

(b) Plasma Etching



(c) Murakami+Caro etching

(d) Murakami etching

Fig. 5 AFM images of TiNAl coating tools

Table 2 Roughness parameters obtained after coating surface

Treatment	R_a	R_q	R_z	R_p	R_v	R_p/R_z
Murakami	0.385	0.501	3.751	2.397	1.354	0.639
Murakami + Caro	0.369	0.476	3.441	1.897	1.543	0.551
Plasma	0.190	0.236	1.676	0.768	0.909	0.458
Untreated	0.240	0.303	2.206	1.164	1.042	0.528

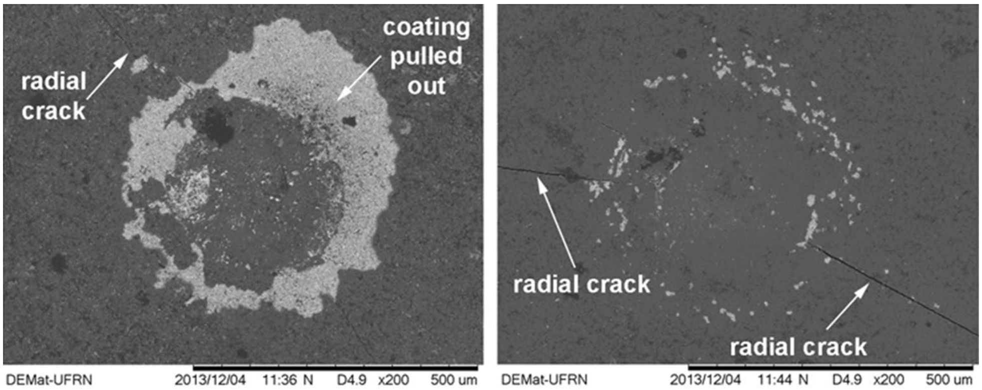
aspects changed in relation to surface before the coating. As it can be seen in Table 2 the R_p/R_z ration increased to chemical etching indicating the presence of waveforms, the opposite was observed to plasma and untreated surfaces that had high values of R_p/R_z ration showing a surface with more sharp peaks.

3.2 Coating adhesion characterization

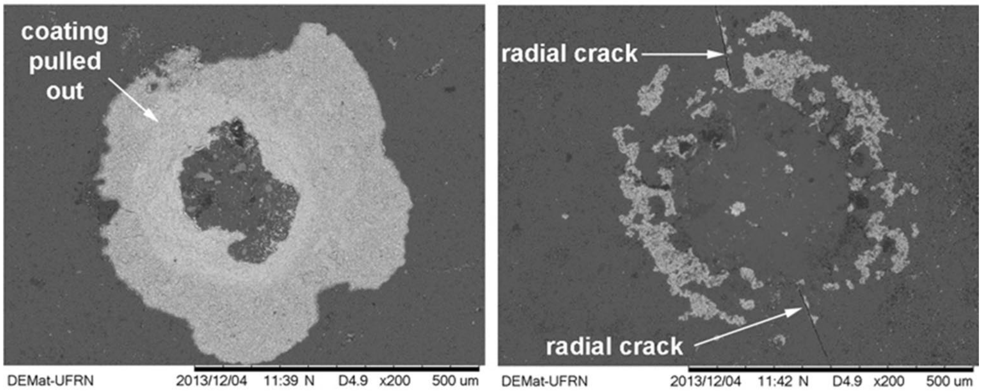
Adhesion of (Ti,Al)N coatings was measured by the indentation method and turning process. Rockwell B indentation test and conventional turning were carried out. Besides it, a comparison between both methods responses allowed to check if the indentation test, widely used to measure coating adhesion, corresponds to machining requirements.

3.2.1 Rockwell indentation test

The results of Rockwell indentation on the coating film deposited on the cemented carbide substrate are shown in Fig. 6.



(a) untreated + TiAlN **(b)** plasma etching



(c) murakami + caro etching **(d)** murakami etching

Fig. 6 SEM imprint images of Rockwell indentation on coating for substrate treatments

Based on a qualitative analysis of Fig. 6, while checking Rockwell's imprints, there is a clear influence of the substrate treatment in coating adhesion. Figure 6c, concerning Murakami plus Caro etching treatment, shows a large pulled out area and without radial cracks, while Fig. 6b, d, concerning Murakami and plasma etching treatment, respectively, shows a small pulled out area. However, in these cases, there are radial cracks. Finally, Fig. 6a, concerning untreated substrate, shows an intermediate pulled out area when compared to the two previous cases.

Vidakis et al. [27] stated that extended pulled out area (or delamination) at the vicinity of the imprint indicates a poor interfacial adhesion; on the other hand, radial cracks coupled with small pulled out area indicate a strongly adherent coating but also brittle ones. Furthermore, Jorgensen et al. [28] described large pulled out areas are related to substrate hardness. Thus, a hypothesis to explain the different imprint images is the significant change in the substrate properties by chemical and plasma treatments. In the worst pulled out case, according to Fig. 6c, it occurred with the application of Murakami plus Caro solutions, then superficial tungsten carbide grains were not strongly anchored on the substrate as a function of cobalt oxidizing by the acid solution. In this particular instance, (Ti,Al)N coating was deposited on a low stiffness substrate, which made the coating peeling out an easy task due to the stress caused by the indenter. An evidence of the statement is the only Murakami chemical etching treatment (without cobalt oxidation, see Fig. 6d), which pulled out a smaller area and radial cracks appeared. Chemical etching only in tungsten carbide altered the topography (see item topography surface), and coating adhesion, according to the indentation tests, was more suitable than the previous one.

Plasma etching treatment provided the better coating adhesion to the substrate with regard to peeling out. As observed in Fig. 6b, a restricted area pulled out and radial cracks appeared. In this sense, it is proposed that plasma etching removed the cobalt in such a way not to weaken the anchoring of the tungsten carbide grains, in addition to changing the topography with increasing roughness values. These actions make the substrate more suitable for coating adhesion. Finally, adhesion of coating on the untreated surface promotes an intermediate peeling out area, because there is a high presence of cobalt on the surface, which reduces coating adhesion.

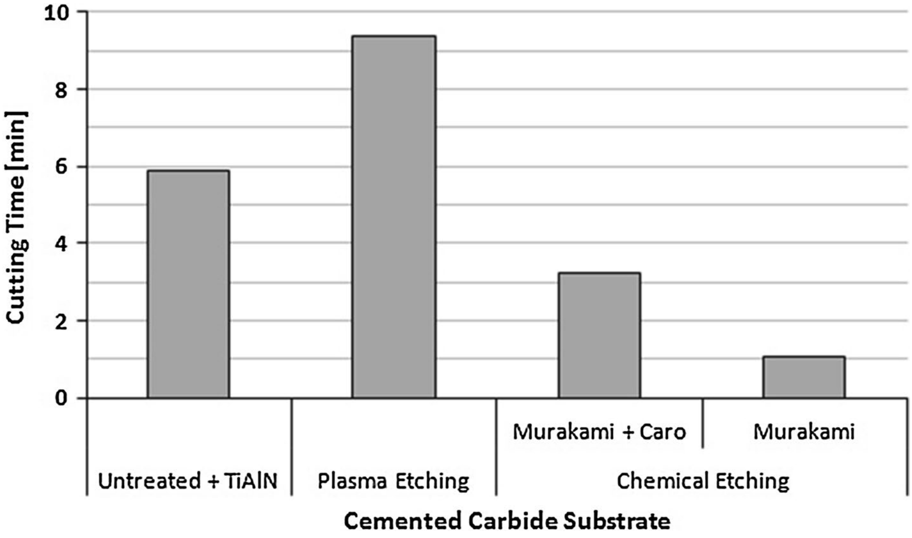


Fig. 7 Tool life for all substrate treatment

Rockwell indentation tests are reasonable indicators of coating adhesion on the substrate, rapidly done and suitable for the industrial environment. However, coatings applied to cutting tools are exposed to different tribological demands depending on the operation. For example, milling promotes thermal and mechanical shocks as well as high shear rates. Drilling causes a cutting speed variation along the cutting edge, which drives a tension gradient between coating and substrate. However, in turning, higher temperatures and shear rates on the rake face in addition to strong contact on flank face with the workpiece require maximum coating adhesion to substrate. This is the reason why turning tools use coating based on chemical vapor deposition (CVD) technique. In order to promote a wider assessment, turning tests were carried out to understanding the surface treatment influence in coating adhesion.

3.2.2 Machining tests

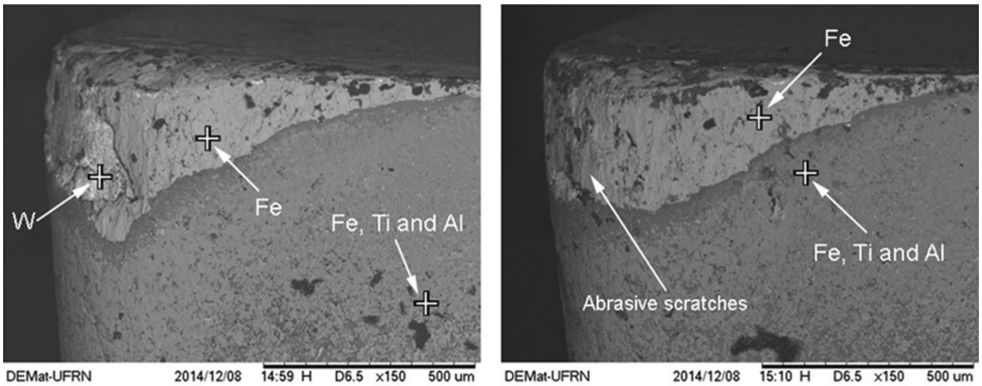
The results of tool life in all experiments are shown in Fig. 7, based on the cutting time, considering average width of flank wear of $VB_B = 0.3$ mm.

Despite the cutting conditions used in these experiments, considering the presence of high chromium content in the workpiece bulk microstructure, catastrophic failures were not observed on all cutting edges. This is a strong evidence of proper stiffness in the lathe setup and workpiece geometry for machining tests.

When adopting as reference, for cutting tool life performance, the untreated substrate plus (Ti,Al)N coating (this has no modification before coating) two characteristics are defined: plasma etching increased average cutting time to 59 %; while chemical etching (Murakami + Caro and only Murakami) decrease average cutting time to 45 and 81 %, respectively, according to Fig. 7. These results clearly showed that chemical etching on the substrate is not suitable for the purpose of increasing cutting time in turning operations. On the other hand, plasma etching showed to be an excellent alternative to increase tool lifetime. Besides, substrate and coating processes can be carried out in the same equipment, which promotes a clean and more efficient process.

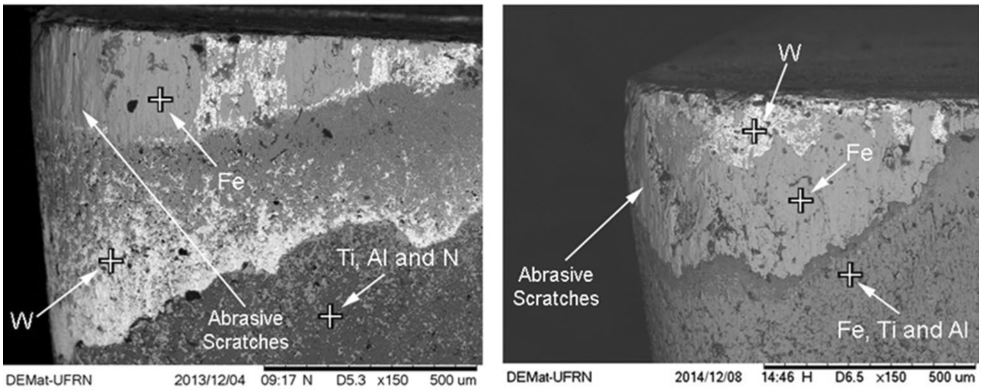
An important point is the correlation between the indentation and the machining tests. Indentations on substrates with different chemical etching promoted different pulled out areas when adopted by the reference untreated substrate. Murakami plus Caro etching promoted the largest pulled out area, while only Murakami etching promoted the second smallest pulled out area. However, in both cases of chemical etching, cutting time decreased when compared to the untreated substrate lifetime. Notwithstanding, plasma etching also promoted a smaller pulled out area, when it was compared to untreated substrate, and increased the cutting time strongly. This is evidence that the indentation test has limitations for corresponding with all turning tests. However, it has become clear that the results presented in Figs. 6 and 7 have shown good agreement for Murakami plus Caro etching (the largest pulled out area) and plasma etching (the smallest pulled out area). Jorgensen et al. [28] stated that Rockwell test as an adhesion method for diamond-coated cemented carbide reveals good qualitative correlations to tool cutting time performance in milling and drilling operations. However, for turning operations this correlation has limitations. Ollendorf et al. [22] explained the application that only one test method can lead to a false evaluation, and several test methods are recommended to evaluate the coating adhesion.

In order to promote a better wear mechanisms understanding on the cutting edges at the end of lifetime, Fig. 8



(a) untreated + TiAlN

(b) plasma etching



(c) murakami + caro etching

(d) murakami etching

Fig. 8 Flank face of worn cutting edges for substrate treatments

shows SEM images supported by EDS analyses of worn flank face.

An overall analysis of the worn cutting edges highlights shallow abrasive scratches on the flank in the cutting direction. This wear topography is a common characteristic of an abrasion mechanism. In the same way, presence of edge chipping, notch wear cutting, edge plastic deformation and crater wear could not be observed, which points out adequate system stiffness and turning parameters definition. On the other hand, based on EDS analyses, large iron (Fe) adhesions from the turned material (gray cast iron) are identified on all flank faces.

Chemical tool inertness is a property that changed over the cutting time, i.e., with progression of flank wear. Iron (Fe) adhesions only took place in regions where the coating was removed and the substrate had been exposed. Although apparently adhesions from the turned material could protect the cutting edge of the abrasive wear, they have a negative side effect: as they are a sum of several layers, their growth leads to instability and breaking, which carry fragments from substrate together, accelerating the flank wear. An evidence of this fact is a region in which it identifies the element tungsten (W), in Fig. 8a. As all boundaries have iron (Fe) adhesion, a hypothesis is that the central region pulled out and carried together a substrate fragment. This wear mechanism is known in literature as “stick and slip” [29]. There was no adhesion on coating, which has enormous chemical inertness against turned material; therefore, it is assumed that this property, after coating removed, changed over time. Thus, methods for achieving better adherence and maintenance of coating on substrate are critical for machining tools.

Figure 8a, b, c shows similar flank wear topography: curved wear region with a strong presence of tungsten (W) and iron (Fe) elements—out during the cutting time

EDS analyses—and the wear contour (the darker region) with presence of iron (Fe—from the turned material) and titanium and aluminum (Ti and Al—from coating). However, Fig. 8c show different flank wear topography: a transition region between the wear land and coating. In addition, iron (Fe) element was not identified in coating area, as in previous cases. An explanation for this phenomenon is that in a test with substrate treatment of Murakami plus Caro etching a large region was pulled out by turning shearing force. Based on optical images carried out during the cutting time, the region was considered as abrasive wear and interrupted the test. This is the reason for the absence of iron (Fe) in the intermediate region and also on the coating. Indentation tests evidenced restricted adhesion to the substrate with this chemical process.

Substrate treatment based on Murakami etching caused major qualitative difference between the indentation results and cutting time results. Indentation images showed a small pulled out region, which in theory, evidence a proper coating adhesion. Machining tests confirmed that this substrate treatment provided a lower cutting time, but the images of the worn cutting edge exhibit a similar topography to the other cutting conditions. An explanation for the fact is the coating pull-out took place in smaller sizes regions, however, faster. The feature that led to the difference in the tests is attributed to the load type: while in the indentation test, there is only compressive stress; in cutting test, the predominant load in flank face is shear. In front of coating with intermediate adhesion on substrate, this evidence could be the critical point.

Plasma etching promoted consistent results between indentation and cutting time. This feature is assigned to the topographic modification and proper cobalt removal from the surface. Due to plasma etching, in the machining test, cutting time was increased to 59 % when compared to untreated substrate plus (Ti,Al)N coating (reference condition). Increasing the cutting time was the main objective of this research. The parameter influence of plasma etching (voltage, current, exposure time and chamber atmosphere), proper cobalt removal, topographical changes on the treated surface and induction of residual stresses (tensile or compressive) are factors for technical optimization. Detailed studies of the mentioned factors influence are the second-phase aims of this research project.

4 Conclusions

Based on the results obtained in the experiments of this work, it can be concluded:

Plasma etching increased surface roughness with a light reduction of cobalt content promoting good adhesion proven by Rockwell B and machining tests.

Although chemical etching treatments changed the topography significantly, they did not provide good coating adhesion on the substrate.

Cobalt content influenced the coating adhesion, high Co content like found after Murakami etching and low Co content observed after Murakami and Caro etching did not promote a good adhesion.

Indentation test is relatively consistent with the machining tests; in 50 % of cases of good adhesion in the Rockwell adhesion test B are not confirmed with the tool life tests.

Considering the surface treatments studied, the plasma etching is suitable for PVD (Ti,Al)N coating deposition on cemented carbide tool increasing the cutting time to 59 %.

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