IMPROVEMENT OF PVD COATINGS ADHESION BY IMPLEMENTED IN-SITU CLEANING IN THE ARC CATHODIC PVD PROCESS

S. Roggero – F G GRUPPO S.p.A. Caselette, Italy
C. Galamand – GaMa Concept, Royas, France
A. Messer – Alliance Concept – Cran-Gevrier, France

The in-situ cleaning of tool steel surfaces by different type of etching in the arc cathodic PVD process have been studied. The study has been carried out after implementing the coating equipment with an extra etching step named PB technology in order to obtain an excellent surface preparation even in case of particular toothed cutting tools as broaches or complex geometry like plastic moulds. The PB technology is included at the beginning of the coating process during the heating phase. The new PB technology improves the existing etching technology and avoid the use of Metal Ion Etching. As a second major advantage, the PB technology is a soft process using parameters allowing the etching of complex geometries without having the risk of arcing on the tools and parts to be coated. High speed steel has been chosen as substrate to be TiN – PVD coated. Substrates surfaces have been analysed after different etching modes using different microscope techniques. The influence of etching method has been also evaluated after the coating process, studying topographical features of the TiN coating surfaces as well as the coating adhesion to substrates

KEYWORDS: PVD COATING, ARC-PVD, SURFACE PREPARATION, ETCHING, PB TECHNOLOGY

INTRODUCTION

The adhesion of PVD hard coatings is one of the most important factor determining their performance in any industrial applications. When speaking about PVD coating adhesion both substrate properties, such as microstructure, roughness and cleanliness, and PVD parameters as temperature and bias voltage, must be considered.
Thus, substrate pre-treatment and coating technique have a key role in the production of any highly adherent coatings.
To achieve optimal PVD coating adhesion, particular attention must be paid to cleaning and preparation of substrate in order to obtain a tool surface free from contaminants and undesirable oxides. Cleaning process is the first treatment of surfaces prior to introduce them in the coating equipment but it may not be sufficient. For this reasons, a common practice is to perform etching processes directly in the coating chamber. In the arc cathodic PVD process the in-situ cleaning of tool steel surfaces by heating and metal ion etching (MIE) can remove contaminants generated from the previous cleaning process but bombardment with metal ions can produce a very thin layer of cathode material on the substrate surfaces. Moreover, metal ion etching can lead to contamination of substrate surfaces with droplets of the cathode material, reducing the adhesion of the subsequent coating. [1]. Another disadvantage of MIE technique is related to the risk of softening effects if performing a strong and intensive etching on small tools.
Normally, PVD coating equipment allow to coat different type of tools from geometrical and size point of view in a single job batch. During the metal ion etching phase, flat surfaces and sharp edges are subjected to different ion flux density as the electric filed is localized in a different way, resulting in non-uniform ion etching within the batch and also on different parts of the same tool.
In this paper, the in-situ cleaning of tool steel surfaces by metal ion etching and a new type of etching step named PB technology in the arc cathodic PVD process have been compared. The study has been carried out after implementing the coating equipment with an extra etching step named PB technology in order to obtain an excellent surface preparation even in case of particular toothed cutting tools as broaches or complex geometry like plastic moulds. The PB technology is included at the beginning of the coating process during the heating phase and it uses a low-pressure Argon plasma. The new PB technology improves the existing etching technology and avoid the use of Metal Ion Etching. As a second major advantage, the PB technology is a soft process using parameters allowing the etching of complex geometries without having the

risk of arcing on the tools and parts to be coated.

MATERIALS AND METHODS

The industrial arc cathodic PVD equipment AC2500 was used to perform the in-situ cleaning of tool steel surfaces by different type of etching. All experiments were performed on high-speed steel samples in the form of discs by simulating the normally use loading of the machine. For each test, two samples were introduced in the coating chamber in order to evaluate the process uniformity over the entire two meters length of the machine. Prior to the etching process, all samples were first polished to a surface roughness (S_a) of around 0.01 µm then cleaned using ethyl alcohol and an ultrasonic cleaning unit.
At the beginning of each test, samples were first heated to 430℃ and then in situ-cleaned by metal io n etching (MIE) in Ar atmosphere or by the PB technology. All MIE processes were performed using a bias voltage of 750 V and Ar flow of 50 sccm while in case of PB processes a bias voltage of 350 V and Ar flow of 120 sccm had been used. Different etching repetitions (3x, 5x, 7x, 9x) had been performed for each etching method.
In both cases, the coating step wasn't performed.
Surface analysis based on the use of the profilometer DektaktXT Bruker and the Keyence microscope had been carried out in the Advanced Laboratory of Alliance Concept on each sample before and after etching process in order to obtain information on roughness and topographic features of substrates.
The morphology and the possible presence of droplets and contamination of titanium cathode material was studied using the scanning electron microscope combined with Energy Dispersive Spectrometry (SEM-EDS) LEO 1430 in the Metallurgical Laboratory of F G GRUPPO Caselette.
After having found the best operating conditions, a production batch of HSS end mills was TiN – PVD coated. Before the coating process, topography and surface roughness of a reference end mill were evaluated using the profilometer DektaktXT Bruker and the Keyence microscope.
Prior to the coating process, all substrates were cleaned using a fully automatic ultrasonic cleaning unit. The end mills, together with two test samples, were mounted on a carousel system that enabled up to three-fold rotation in order to produce a high uniform coating on the entire tool surface. In the coating chamber, end mills were first heated to 430℃ and then in-situ cleaned by the PB technology (350 V bias voltage and 120 sccm Ar flow, 5 repetitions). After that, 2 µm thickness of TiN coating was deposited on tools surfaces.
Surface studies of the coated reference end mill as well as the coated samples were performed as described above. A Calotest and a Rockwell test were also carried out on the test samples in order to measure the effective thickness of the TiN layer and to have a preliminary evaluation of coating adhesion respectively.

RESULTS

The etching process removes native oxides and contaminants from substrate surface influencing the subsequent adhesion of the coating and its growth. Depending on the etching method, current density, plasma homogeneity and other parameters, as well as substrate shape and its rotation, different results can be obtained.
In this paper, the efficiency of the etching technique has been studied by making a 2D image (Fig. 1) and measuring the surface roughness Sa of each test samples (Tab. 1). Substrate irregularities induced by metal ion etching and by etching with PB technology after the same number of repetitions are compared.

Tab. 1 – Surface roughness Sa [µm]

	3x	**5x**	**7x**	**9x**
MIE	0.086	0.126	0.110	0.098
PB Technology	0.020	0.019	0.022	0.026

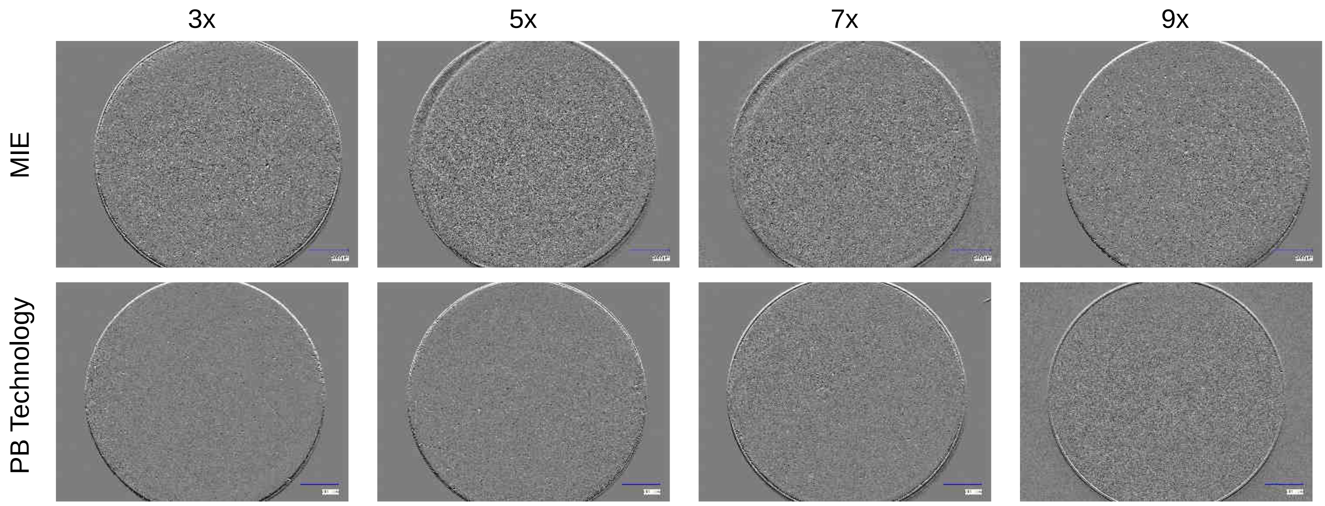


Fig.1 – Top-view Keyence VHX microscope images of surface topography after metal ion etching (MIE) and etching with PB technology after different etching repetition (x20, high contrast, coaxial light)

In all cases, the roughness increases a little if compared to the reference value of 0.01 µm and no significant changes take place on surfaces after 5 etching loops regardless of etching method. However, the roughness value S_a increases significantly after metal ion etching technique while scratches and bumps seem to be smoothed on surfaces etched with PB technology.
The top-view SEM images of surface samples after 5 MIE etching loops and 5 PB technology loops are given in Fig. 2.

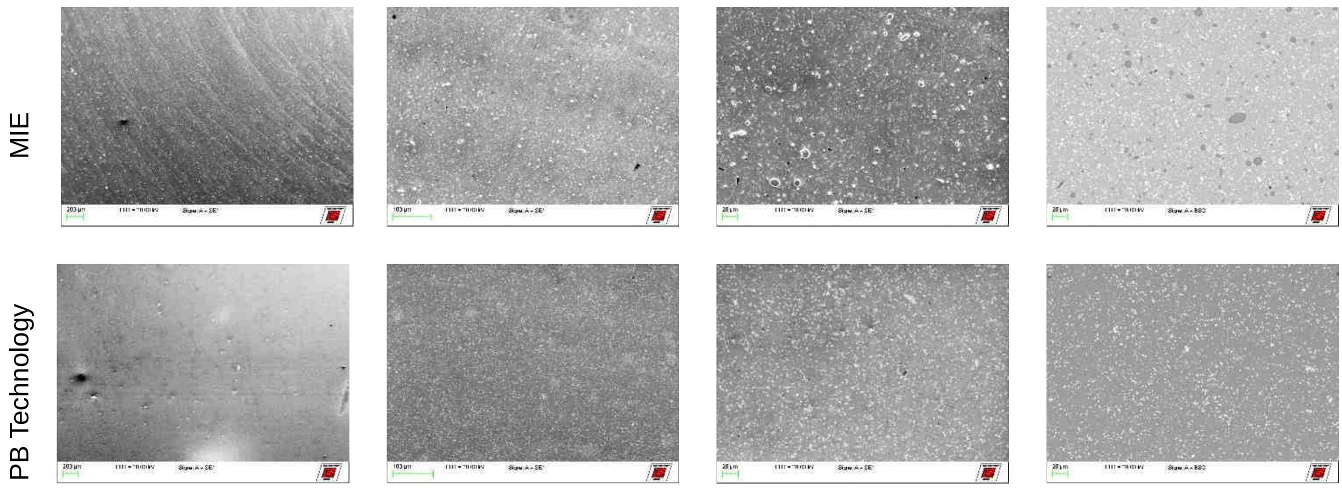


Fig.2 – Top-view SEM images of sample surface after metal ion etching (MIE) and etching with PB technology after 5 etching repetition

SEM images show considerable differences between MIE etched surface and PB technology etched surface. Etching with PB technology produce highly uniform surfaces if compared to those produced by MIE. Moreover, no microparticles of the cathode material are observed. Contamination of substrate surfaces with droplets of the cathode material is a typical phenomena of the metal ion etching as shown in previous SEM images related to MIE etched substrates in which Titanium droplet are clearly visible.

Since best results from surface point of view have been obtained after 5 etching loops in both cases, a HSS end mills batch have been TiN – PVD coated using the PB technology etching method. The coating thickness as well as the adhesion class have been evaluated on a test sample specifically inserted in the end mills coating batch. The Calo test and the Rockwell Test techniques have been respectively used. The measured coating thickness is 2.2 µm while the adhesion class is HF1 according to VDI 3198 (Fig. 3).

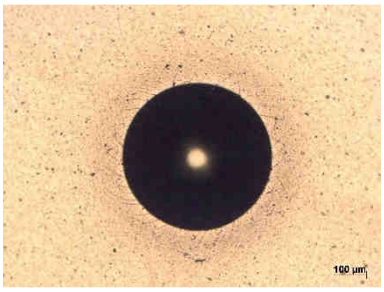
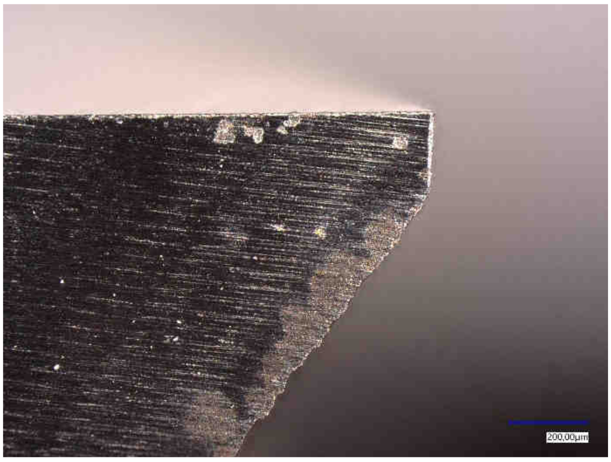


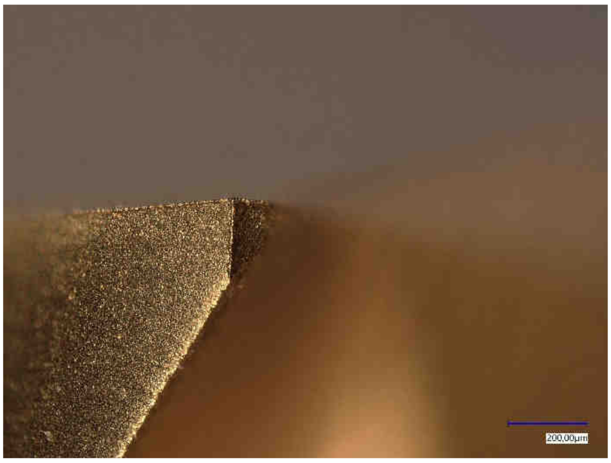
Fig.3 – Rockwell indentation on test sample, only fine cracks at the of imprint and no delamination (HF1 adhesion class)

The PB etched end mill edges have been observed before and after the coating process using the Keyence VHX 167 microscope (Fig. 4).

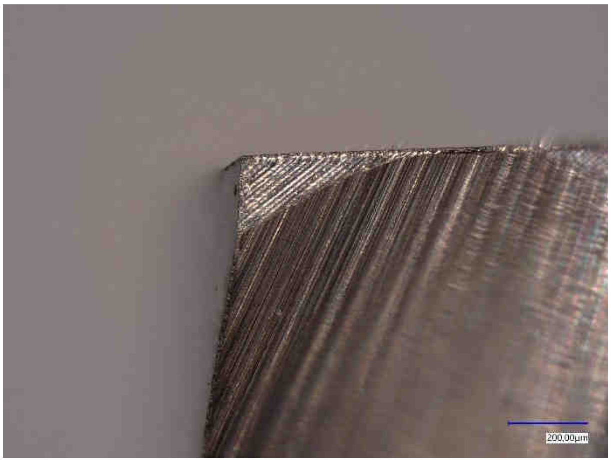
Before coating | **TiN – PVD coated**



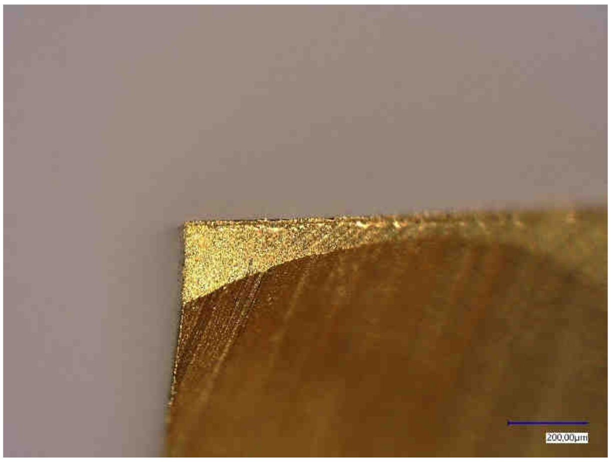
(a)



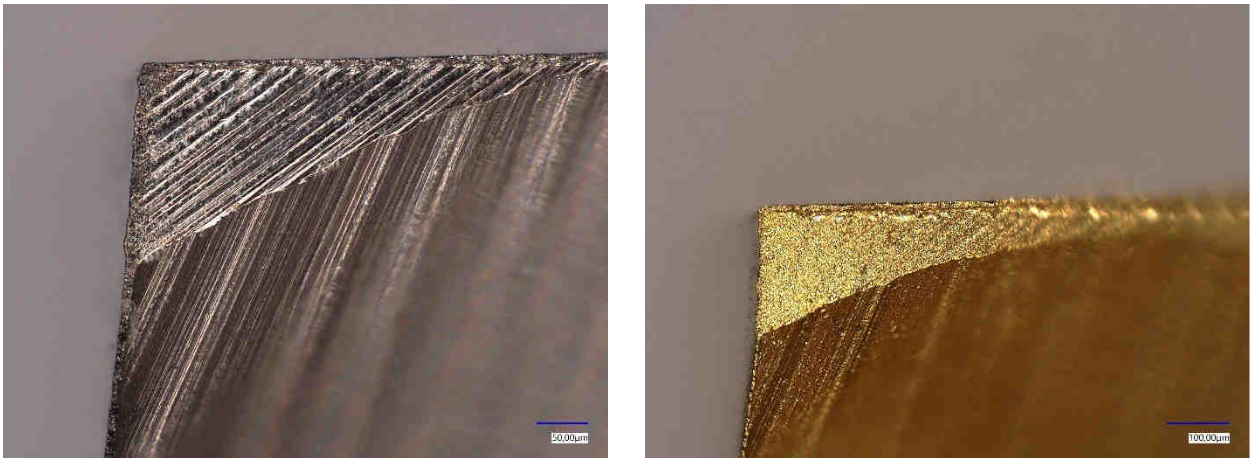
(b)



(c)



(d)



(e) (f)

Fig.4 – (a,b) rake face, (c, d, e, f) flank face of PB- etched uncoated and TiN – PVD coated end mill

On cutting tools, such as end mills, during the MIE etching step, the applied negative bias leads to non-uniform electric field and non-uniform plasma density over different parts of tool surface [2]. In particular, the ion etching effect is stronger at the sharp edges than on flat surfaces with subsequent variation in surface topography.
Using the PB technology as etching method, the creation of a low pressure plasma allows a more uniform and controlled etching over the entire tool surfaces can be obtained, avoiding rounding effect of the cutting edge, as shown in Fig. 4. The more uniform etching is reflected on subsequent coating properties with less differences between sharp edges and flats surfaces, even on the same tool.
In general, all surface irregularities related to previous surface machining operations are transferred to the final PVD coating. Moreover, others defects, such as droplets, can be introduced after the coating process itself.
As mentioned above, etching with PB technology avoids the formation of droplets of the cathode material during the etching phase. That means that the number and the size of TiN droplets on tool surface after coating deposition is lower than that obtained in case of etching with MIE using the same coating parameters (Fig. 5).

MIE + TiN – PVD coating **Etching with PB technology + TiN – PVD coating**

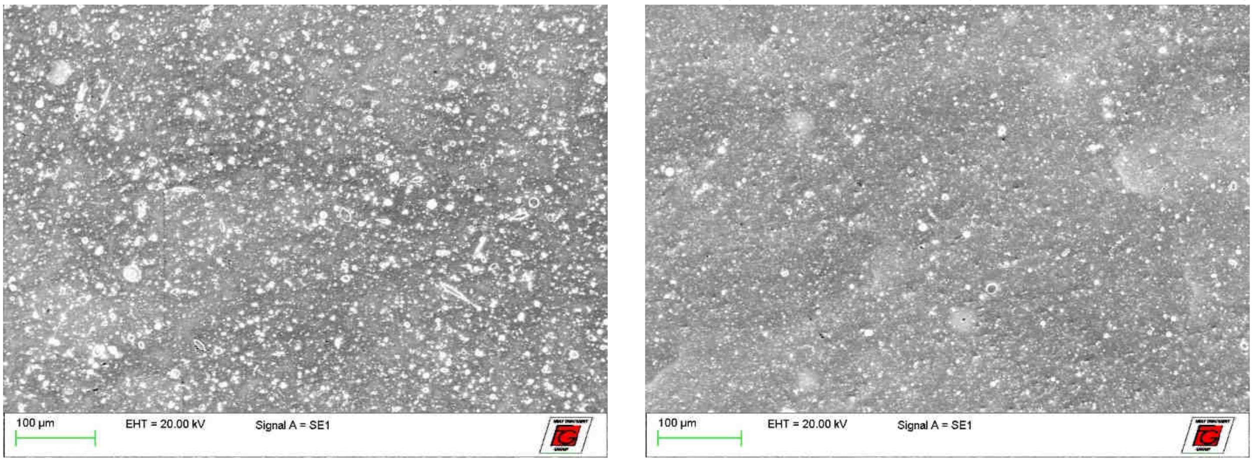


Fig.5 – SEM images of TiN – PVD coated surfaces after Metal Ion Etching (left) and after etching with PB technology (right)

CONCLUSION

In this study, we analyzed the surface quality of HSS samples and HSS end mills after two different type of etching in several batch-type in a cathodic arc evaporation deposition system. We showed that using the

new PB technology as etching method, a more uniform and smoother surface can be obtained compared to that produced by the normally used metal ion etching. We found that the surface roughness of initial samples S_a increased up to ten times in case of MIE and only up to two times in case of PB technology.
In fact, an important advantage of the PB technology etching method, is the absence on the substrate surface of droplets of metal sputtered by the cathode. The PB technology uses softer process parameters (i.e. bias voltage and current) avoiding the risk of arcing on complex geometry tools and part to be coated. The creation of a low pressure plasma leads to a more uniform etching over the surface, minimizing differences between flat surfaces and sharp edges. We demonstrated that even in case of complex cutting tools, such as end mills, the PB etching efficiency was reached uniformly around the main flank and on the rake face.
Since the coating surface is a replication of the substrate surface, surface irregularities such as pure metal droplets or cavities introduced by the etching method are transferred to the coating surface so that they can be observed on coated surfaces, as shown by SEM images of TiN – PVD coated surfaces after Metal Ion Etching and after etching with PB technology. It is known that non-homogeneity caused by the presence of droplets, apart from increasing surface roughness, can increase the friction and the thermal load of the cutting edges leading to premature wear of cutting tools during mechanical operations. The formation of a more uniform substrate surface related to the use of PB technology as etching method results in very good quality coating minimizing the risk of premature failure of the coating as well as of the cutting tool itself.

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