



DEGREE PROJECT IN THE FIELD OF TECHNOLOGY  
MATERIALS DESIGN AND ENGINEERING  
AND THE MAIN FIELD OF STUDY  
MATERIALS SCIENCE AND ENGINEERING,  
SECOND CYCLE, 30 CREDITS  
*STOCKHOLM, SWEDEN 2016*

# **Quality evaluation of PVD coatings on cutting tools by micro-blasting**

**VICTOR LUNDBERG**

## **Abstract**

Sandvik Coromant in Gimo is in need of a good quality evaluation method of adhesion for PVD layers; since the existing testing method is located in Sandvik Coromant's other location in Västberga. Different micro-blasting methods were investigated in this thesis and the results show that some of the methods could potentially be used for evaluation of adhesion, more specifically the wet blasting methods, M1 and M2. The results also show that the investigated geometry received varying adhesion quality when lower etching bias in the PVD process was used and when different sides was pointing upwards in the PVD furnace. Further investigation will have to be made in order to fully implement micro-blasting as a testing method in production.

## **Sammanfattning**

Sandvik Coromant i Gimo är i behov av en utvärderingsmetod av god kvalitet för vidhäftning för PVD-skikt; eftersom den befintliga testmetoden finns på Sandvik Coromants andra plats i Västberga. Olika mikrobästringar metoder har undersökts i denna avhandling och resultaten visar att några av metoderna potentiellt kan användas för utvärdering av vidhäftning, mer specifikt våtblästringar metoderna M1 och M2. Resultaten visar också att den undersökta geometrin fick varierande vidhäftningskvalitet när lägre etsnings-förspänning i PVD-processen användes och när olika sidor pekade uppåt i PVD-ugnen. Ytterligare utredning kommer att behöva göras för att fullt ut implementera mikrobästring som en testmetod i produktionen.

## Table of Contents

Abstract.....	2
1 Introduction.....	5
1.1 Background.....	5
1.2 Purpose of study .....	6
1.3 Literature study .....	6
1.3.1 Processing and testing of inserts.....	6
1.3.2 Tribology of ceramics and metals.....	9
1.3.3 Abrasive and erosive wear .....	9
2 Theory .....	13
2.1 Blasting process in general .....	13
2.2 Light Optical Microscopy.....	14
2.3 Scanning Electron Microscopy .....	15
2.4 Edge radius and PER measurements .....	15
3 Test screening.....	16
3.1 Experimental .....	16
3.2 Results and discussion from test screening.....	17
3.2.1 Testing with M1 .....	18
3.2.2 Testing with M2.....	20
3.2.3 Testing with M3.....	22
3.3 Conclusions test screening.....	23
4 Experimental procedure .....	24
4.1 Different amount of pre-treatment blasting .....	24
4.2 Effect of etching in the PVD process. ....	27
4.3 Directional dependence in PVD furnace .....	28
5 Results.....	29
5.1 Wear analysis in SEM .....	29
5.2 Main tests.....	31
5.2.1 Influence of different amounts of pre-treatment on the surface structure before testing... 31	
5.2.2 Influence of etching bias in the PVD process on coating adhesion .....	33
5.2.3 Evaluation of different amount of pre-treatments effect on adhesion .....	35
5.2.4 Adhesion evaluation between the different testing methods .....	36
5.2.5 Varying etching bias in the PVD process .....	38
5.2.6 Directional dependence in PVD furnace.....	39
6 Discussion .....	41
7 Conclusions.....	44
8 Future work .....	44

9	Acknowledgments .....	45
10	References.....	46
	Appendix .....	47
	Appendix 1 .....	47
	Appendix 2.....	48
	Appendix 3.....	51

# 1 Introduction

## 1.1 Background

With 8000 employees represented in 130 countries, Sandvik Coromant is the world leading supplier of tools, tooling solutions, and know-how to the metalworking industry. Their main costumers are the major automotive, aerospace and energy industries. Extensive investments within research and development, together with constant communication with the costumer, have helped create new productivity standards and unique innovations that have driven the development forward to where they are today. The facility in Gimo is a production center of cutting tools with 1500 employees. [1]

One of the things that are constantly under evaluation is how the lifetime of cutting tools can be increased. One way to address this is by applying a hard coating layer, which reduces the friction and increases the wear resistance, on the tool. The two most common methods to deposit this coating are Chemical Vapor Deposition (CVD) and Physical Vapor Deposition (PVD). In CVD, gas-phase reactants are introduced into the reaction chamber. CVD generally requires high process temperatures, since the reactions that occur in the chamber often are thermally activated, although other activation techniques, such as plasma, photo-activation or electrochemical, exist [2]. Because of the high process temperatures, it is common that diffusion takes place between coating and substrate and also growth of interstitial phases. The smooth transition of composition caused by diffusion is often beneficial for the adhesion between substrate and coating whereas interstitial phases are not. The CVD process is good in order to create coatings in many different materials with very good adhesion between coating and substrate [3].

There are two different PVD processes, traditional thermal vaporizing and sputtering, but the basic principle for both is that the coating material is vaporized and transported in the shape of ions, atoms or molecules to the substrate. For PVD processes the pressures are reduced to very low levels and lower temperatures than CVD are required. This is an advantage since the higher temperature in CVD can change the substrates microstructure, shape and properties. The difference between the vaporizing method and the sputtering method is how the atoms from the coating material are released. For the sputtering method the coating atoms are released by argon ions that are accelerated towards the target by an electric field, impacting the surface and knocking target atoms, molecules etc. out of the surface. The free coating atoms are then accelerated by the same field through the chamber until they land on the substrate surface and a coating is made. There are different methods of vaporizing the coating material such as vacuum evaporation, ion plating, and arc-evaporation, of which electron beam and arc-evaporation is used in production at Sandvik Coromant in Gimo. The arc-evaporation method uses an electric arc between a cathode material and an anode material or the chamber wall to create a fast, local, evaporation. The evaporation is very intense and contains a high number of ions which is later attached to an, in most cases, negatively charged substrate surface. By introducing a reactive gas into the vacuum chamber the coating material can react with the gas to form for example an oxide or nitride coating. Since the attraction between the ions and substrate surface is very strong, the substrate is bombarded with ions which clean and heat the surface without initially creating any coating. This cleaning of the surface before coating is called etching. This bombardment continues during coating although the coating

ratio is much higher than the removal rate at this point. This makes it possible to create a very thin and even coating with good adhesion to the substrate. One problem with arc-evaporation is that the local heating with the arc can lead to creation of small droplets of molten cathode material which is incorporated into the coating, forming an uneven surface. This uneven surface is unwanted and can cause stresses in the coating and therefore make it weaker [3]. Only PVD coatings produced by arc-evaporation will be investigated in this thesis.

## **1.2 Purpose of study**

The main purpose of this thesis is to evaluate blasting with different types of media, machines and machine settings as a method for quality control of PVD coatings on cutting tools. This method should function as a complementary test or a replacement to the already existing evaluation method. The method used today uses three extra threading inserts that are included in the PVD process. If there is a small failure in the process, but unknown if it influences the quality, these inserts will be sent to Sandvik Coromant in Västberga for functional evaluation in a turning machine. This costs a lot of time and money, why a testing method in Gimo could save a lot of money for the company.

An even bigger advantage is that the old method only finds problems with the PVD process. The suggested method could also be used to test individual orders in the process. This could be used for example to find poor adhesion due to insufficient cleaning before coating something that is not done with the today used method.

The first part of the thesis consists of a literature study. This is followed by the second part with experimental testing by blasting at different pressures, angles and with different blasting media.

## **1.3 Literature study**

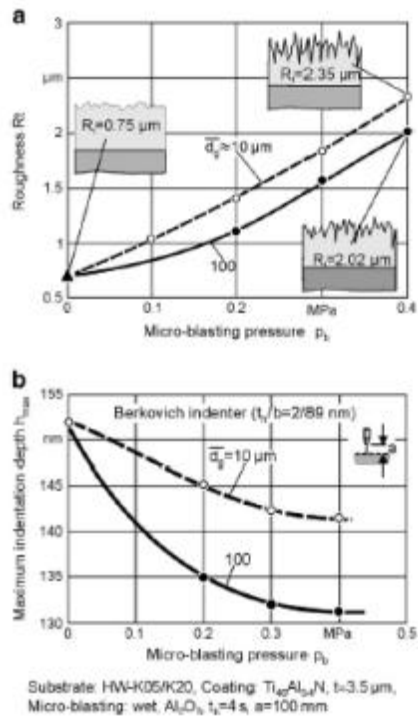
As part of this master thesis, a literature study about the micro-blasting process and the adhesion between substrate and PVD coatings was made. Different test methods for adhesion testing of hard coatings from literature were investigated. The production usage of micro-blasting process is also discussed.

### **1.3.1 Processing and testing of inserts**

There are different processing methods in production of cutting tools today, used to achieve correct dimensions of the tools, desired cutting edge quality, and to produce an acceptable surface finish. When used for material removal, these methods can be categorized into two different types of wear; abrasive and erosive wear [4]. Grinding is a typical application of abrasive wear, where a material with sharp edges is used to scratch a softer surface in order to change the geometry and/or surface finish. Another method in production that can be categorized as abrasive wear is the brushing process. In the brushing process, a hard brush is used for removing impurities of the surface and creating the correct edge geometry. An example of an erosive wear method that is used in production is micro-blasting. During micro-blasting, the material is bombarded with hard particles that are either gas- or liquid-borne. These particles rupture the surface and create different kinds of wear depending on the angle of impact, hardness of the particles and if the material is ductile or brittle. Micro-blasting is used for cleaning and polishing the surface before coating [4], [5]. This method will be used in this thesis for quality testing of PVD-films.

Adhesion testing of hard coatings is a very important and difficult task, utilized in order to guarantee the quality of the cutting tools. In fact, it is even difficult to find a proper definition of an acceptable adhesion between coating and substrate. There are two different kinds of adhesion, basic and practical adhesion [6]. Basic adhesion is broader, since it is seen as a summation of all interactions at the interface between the substrate and the film, or as the work needed to completely separate them from each other. Practical adhesion depends on the basic adhesion but it refers to the failure of wear protection of the hard coating which depends on several different parameters such as fracture toughness of both substrate and film, size distribution of defects etc. This makes the practical adhesion more interesting when studying hard coatings. Earlier studies have been made in order to find a suitable adhesion test that can be used for hard coatings. [6]. There are several different methods of testing adhesion that has been investigated, but not all is suitable for every application. In [6], the effect on adhesion of different pre-sputtering timespans before coating was tested. The investigation showed that comparing the seven test methods, no corresponding coherent results of testing adhesion was showing on TiN films. However, some of the methods showed promising results, and some of them are already used in industry today. One of the methods is the scratch test, where a Rockwell-shaped diamond indenting tip is loaded with a force and dragged along the surface, causing abrasive wear of the coating, while simultaneously calculating the friction work needed. This method is widely used in industry today in order to evaluate the quality of coatings [6]. Another method that is easy to use is the Rockwell test, which is a regular hardness test with evaluation of the damage pattern of the coatings. The study also showed that the laser-acoustic method is a promising non-destructive method for evaluation of coating quality. It uses surface acoustic waves in order to measure the Young's modulus,  $E$ , of thin films down to 100 nm in thickness.

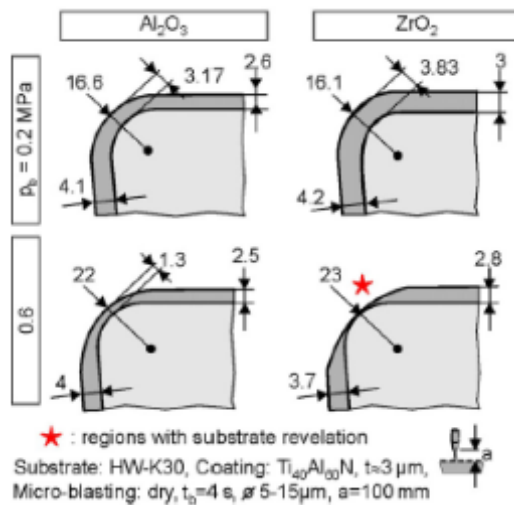
Wear resistance and cutting performance is obviously of great importance in advancing the life of cutting tools. Studies have shown that these properties can be improved by micro-blasting the coatings [7]. When micro-blasting is used, compressive stresses are induced in the film, which increases the coating's hardness as well as the brittleness. This can cause the coatings to fracture. Tests have been made in order to find the most optimum grains and corresponding process parameters in micro-blasting for improving the wear resistance and cutting performance [7] [8]. Two different investigations were made, one with dry micro-blasting with different qualities of abrasive  $\text{Al}_2\text{O}_3$  grains and one with wet micro-blasting with two different grain types; abrasive  $\text{Al}_2\text{O}_3$  and spherical  $\text{ZrO}_2$ . In both cases, pressure and grain sizes were varied in order to find the most suitable grain. The abrasion mechanisms after blasting were investigated by testing the coating's roughness. Other tests such as nano-indentation, nano-impact, and Energy-Dispersive-X-ray microanalyses, EDX, were made in order to investigate the hardness, brittleness, cutting edge geometry and thickness decrease. When using wet micro-blasting with  $\text{Al}_2\text{O}_3$  grains with different sizes, it showed that the roughness increased and nano-indentation depth decreased with increased pressure. Furthermore, the roughness and nano-indentation depth was lower when using coarser grains. This means that the surface is rougher but less hard after micro-blasting with finer grains on TiAlN coatings, which can be seen in Figure 1 below [7].



**Figure 1: The roughness as a function of blasting pressure for two different grain sizes (a) and the hardness as a function of blasting pressure for two different grain sizes (b) [7].**

Another factor that was affected by different blasting pressure and grain sizes was the cutting edge radius, where higher pressure and coarser particles resulted in a larger radius and also thinner coating, where the latter can result in substrate revelation and reduced lifetime of the cutting tool. When looking at different kinds of particles in dry micro-blasting, it showed that using  $\text{Al}_2\text{O}_3$  with sharp-edged particles resulted in coating material removal by abrasion, which did not occur when using spherical  $\text{ZrO}_2$  particles where the coating material deformation is higher. This resulted in rougher surface but a less hard material when using  $\text{Al}_2\text{O}_3$  than  $\text{ZrO}_2$ . The study showed that an increased micro-blasting pressure can instead of enhancing the coating properties, increase the brittleness of the coating and cause substrate revelation if the pressure is too high, as shown in Figure 2. Also, the same relationship for pressure vs. indentation depth and roughness was shown in this study as for wet micro-blasting [8]. Both studies proved that the cutting performance and tool life can be improved by micro-blasting at a certain pressure depending on the blasting particles and coating type.





**Figure 2: Coating thickness for different blasting pressures and grains. [7]**

### 1.3.2 Tribology of ceramics and metals

The main reason, why the inserts are coated with a ceramic material is to improve the tribological properties of the inserts. The tribological function of ceramics is much more dependent by low surface roughness than metals, why it is crucial to process a surface to be as smooth as possible in order to decrease the wear of the insert. Ceramics can achieve smaller grains than metals by processing with for example tribochemical polishing, and ultrafine grains have been shown to increase the toughness of the material [4].

There are different wear mechanisms depending on force, temperature, and wear rate. Wear of ceramics can be divided into two regimes; high level and low level, which can be used in sliding contact, abrasive, and erosive wear. In low level, the wear is done tribochemically or through plastic deformation in micro scale, whereas in high level, the wear is considered brittle. The limit where wear goes from low to high level is different depending on the wear mechanism. In sliding contact, the limit is at a certain value of contact pressure multiplied by the sliding speed. In abrasive and erosive wear, the load of single abrasive and erosive elements is instead considered. Sliding contact or plastic adhesive wear is rather unusual for ceramics but can occur at high contact temperatures on cutting tools during metal cutting. Long parts of the material are then sheared off. [4]

### 1.3.3 Abrasive and erosive wear

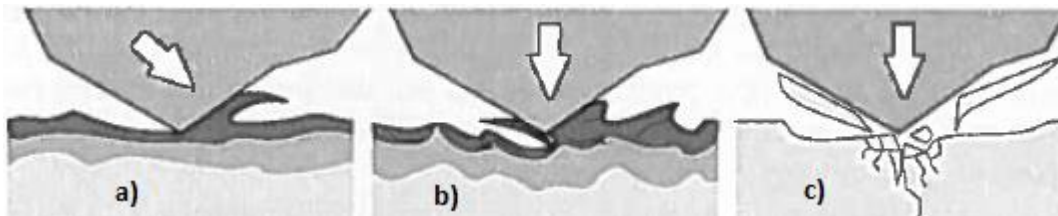
Abrasive and erosive wear is more common when studying ceramics, where abrasive wear is when a material with hard edges scratches a softer surface, and erosive wear is when a material is bombarded by hard particles. Abrasive wear is, as mentioned above, common in production of inserts in processes like brushing and grinding whereas erosive wear is more commonly encountered in different blasting processes. Abrasive wear can be further divided into two main mechanisms; two-body abrasion and three-body abrasion, shown in Figure 3 below. Two-body abrasion is characterized by that the abrasive elements represents a solid surface and three-body abrasion they are better represented by free particles that gets rubbed between two surfaces. The surface of a material worn by two-body abrasion is more clearly

striped than the surface worn by three-body abrasion, and if the same abrasive particles are used in both cases, two-body abrasion can be about ten times faster.



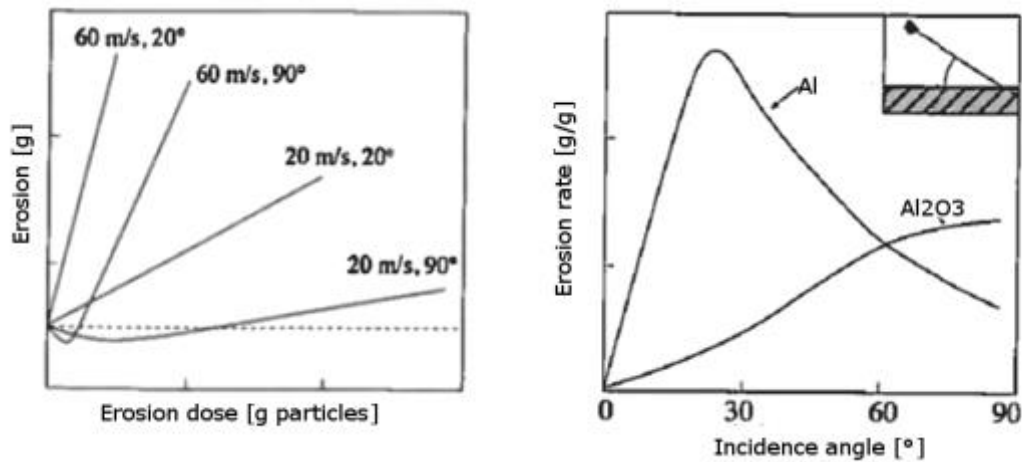
**Figure 3: Principle of a) two-body abrasion, and b) three-body abrasion. [4]**

There are three different types of erosive wear; chip separation erosion, fatigue erosion, and flake erosion, where the first two is more common for ductile materials and the last one is mainly for brittle materials. Chip separation erosion occurs when a chip is separated from a surface, which can happen on a polished surface but benefits from a rougher surface. Fatigue erosion is when the same point of the material is bombarded repeatedly, resulting in deformation of the material beneath the surface. Failure of the material occurs when a critical strain is reached. The third and last wear mechanism, flake erosion, is caused by single particles resulting in crack propagation. The crack propagation can cooperate with earlier cracks and separate large particles away from the actual point of impact.



**Figure 4: Erosive wear mechanisms: a) Chip separation erosion, b) fatigue erosion, and c) flake erosion. [4]**

In order to prevent these wear mechanisms, the material will have to be both tough and hard which can be difficult to manage, at least for metals where most metals and alloys have almost the same erosion resistance. During erosion of metals and other ductile materials, the wear does not start until after a period of incubation. This is explained by that a certain amount of particle impacts is needed to initiate material wear. Factors that affect the degree of erosive wear are; velocity, angle of impact, and type of particles [4]. Erosion is in general strongly dependent on the angle of impact. Ductile and brittle materials show different critical angles of impact, where ductile material exhibit maximum erosion at 30 degrees whereas brittle materials' maximum is located at 90 degrees. The angle of impact for maximum erosion is increasing gradually with decreased ductility.



**Figure 5: The figure to the left shows the relationship between erosion and amount of incoming erosive particles for different velocities and angles. The figure to the right shows the relationship between erosion rate and angle of impact on two different substrates. Adapted from [4].**

The velocity of the particles,  $v$  also affects the erosion rate,  $w$  strongly. The relationship can be described by the formula,  $w \propto v^s$ , where  $s$  is an experimental constant calculated from the proportion between erosion rate and the particles' momentum, normally in the range 2.3-3. It has also been shown that large particles give a slightly higher erosion rate than small particles, which is shown in Figure 6. If the particles are too small and/or the velocities are too low, the erosion rate goes towards zero, since the particles start following the gas flow around the specimen. Another effect that is caused by smaller particles or lower velocity is that wear of brittle materials can occur in a rate usually associated with ductile materials. The opposite will occur if a ductile material is impacted with high velocities and/or larger particles. Erosion rate is also dependent of the particles' hardness and angularity, exhibiting an increasing erosion rate with increasing particle hardness,  $H_p$ , for  $H_p < 1.25 \cdot H_m$ , where  $H_m$  is the hardness of the eroded material. Erosive wear can also occur when  $H_p < H_m$ . Further increased hardness of the particles, beyond  $1.25H_m$ , does not affect the erosion wear. Hardness and angularity of the particles is connected since harder particles appear more angular. This means that sharper particles result in higher erosion rate than spherical particles [4].

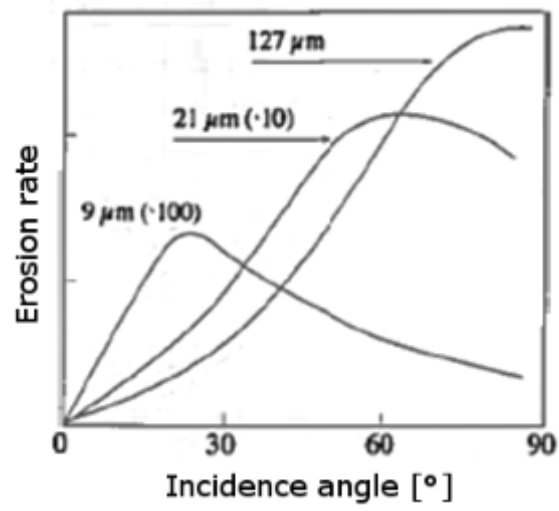


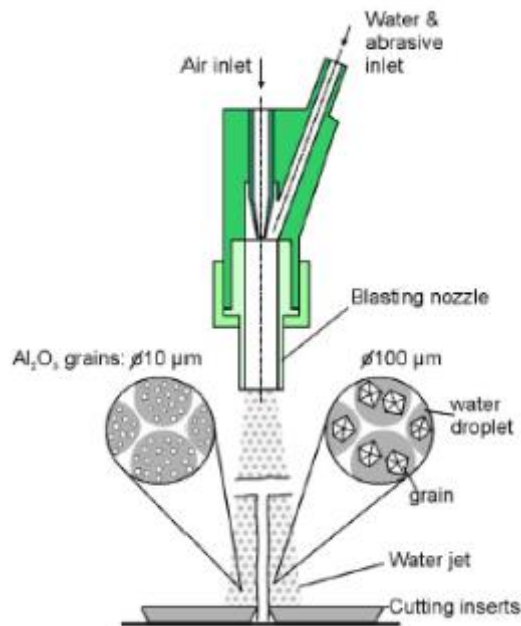
Figure 6: Relationship between erosion rate on the y-axis and angle of impact on x-axis on glass for different particle sizes. The values for the smaller particle sizes have been amplified to fit in the graph. Adapted from [4]

## 2 Theory

### 2.1 Blasting process in general

Blasting is a common process used in production of cutting tools as both pre-treatment and post-treatment for different purposes. The basic principle is that hard ceramic particles are sprayed at a material surface at a certain pressure and time in order to affect surface characteristics such as, roughness, hardness, brittleness and the cutting edge geometry [9]. Also, as mentioned in the literature study, different types and sizes of particles can be used in order to affect the surface differently. Production in Gimo uses two different types of machines for blasting; dry and wet blasting. In the dry blasting process, only coarse particles that are softer than the coating are used in a pre-treatment process to create large edge radii. The most flexible, and most used, process is the wet blasting process. The ceramic particles are mixed 30 vol. % with water, in pre-treatment process and 20 vol. % particles in post-treatment process which is used for CVD coatings. The resulting water-particle slurry is then pressurized with compressed air to achieve the appropriate processing conditions. There are different wet blasting machines for pre-treatment and post-treatment in production, where the main differences are how many blasting guns that are used and at which angle the particles impact the surface. In the eight-gun configuration used for pre-treatment, the incidence angle is 52 degrees, and in the three-gun post-treatment configuration, 90 degrees. Pre-treatment is made in order to clean the surface and process the best surface characteristics. The main purpose of the post-treatment process is to create residual compressive stresses in the coating to increase the toughness, but also to achieve the correct aesthetics. Depending on how much surface wear is wanted, different grain sizes and pressures are used in the pre-treatment wet blasting process, where coarser particles result in higher wear rates. Another factor that affects the amount and type of wear is the particle type, where both shape and hardness influence the result. As mentioned earlier  $\text{ZrO}_2$  particles were used in [8]. But other types of particles can also be used, which differs in size and hardness.

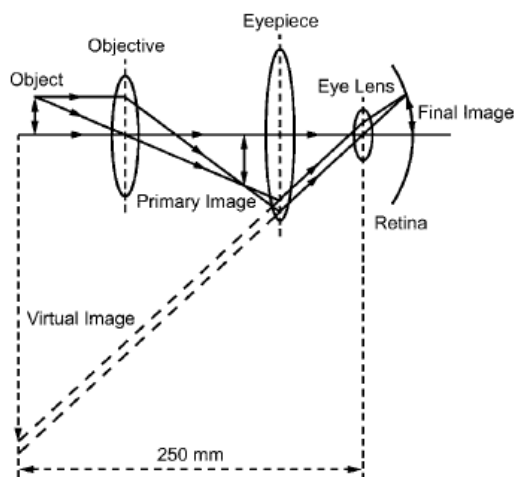
The basics of the wet blasting process are that ceramic particles are mixed with water to form a slurry that is sprayed at the material using pressurized air. Inserts are placed on a rotating plate in the machine with the guns dispensing the media moving over the plate linearly to create as even wear as possible. Depending on which generation of the machine is used, a protective screen could be inserted over the plates to make sure the inserts don't fall off. This is not needed in the latest generation of machines which create a more evenly distributed wear since the protective screen can't prevent the particles from hitting the entire surface. There are a total of eight guns in the machines which use 52 degrees incidence angle resulting in wear all over the inserts. Different blasting pressure and time can be used to create different surface characteristics [10]. All six blasting methods used in production in Gimo will be evaluated in this thesis and are listed in section 3.1 below. Basic principle of a blasting process with 90 degree gun is shown in Figure 7.



**Figure 7: Principle of wet micro-blasting with 90 degrees blasting gun [7].**

## 2.2 Light Optical Microscopy

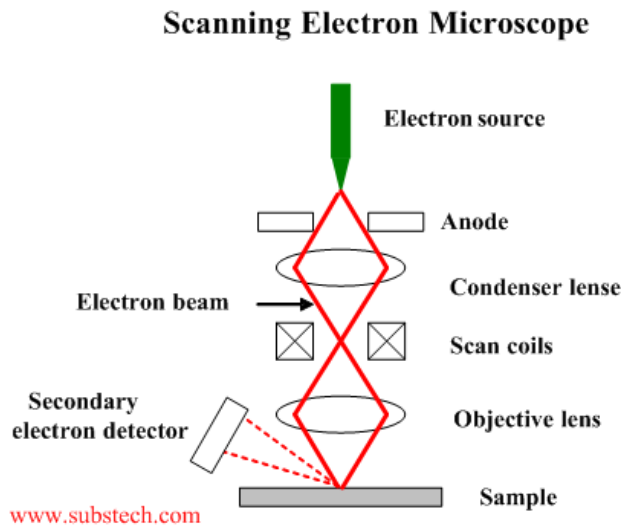
Light Optical Microscopy, LOM, was used to get a rough picture of the inserts before and after blasting tests. The main principle of the LOM is to project a high-resolution image by focusing light from a light source onto an object to increase the magnification. The light is focused by lenses onto a certain point of the specimen that is to be examined. Basic principle of image formation in a light optical microscope is shown in Figure 8 below. [11]



**Figure 8: Image formation in Light Optical microscope. [11]**

### 2.3 Scanning Electron Microscopy

Scanning Electron Microscopy, SEM, is a commonly used technique for high-resolution surface imaging. It uses electrons to obtain images with magnifications between 10 to 100.000 times. The electrons are accelerated by a potential difference and controlled by electromagnetic lenses in order to focus and scan the electron beam over a small section of the specimen surface. When the electrons collide with the material they will be exposed to a series of collisions with the material's atoms, causing some electrons to backscatter while some are attenuated. A detector then gathers the backscattered or secondary electrons to generate an image of the surface of the material. The resolution of a SEM is much better than the LOM since it uses electrons instead of light [12]. A schematic picture of the components is shown in Figure 9 below.



**Figure 9: Schematic picture of components i Scanning Electron Microscopy. [13]**

### 2.4 Edge radius and PER measurements

As mentioned earlier, there are different pre-treatment processes in order to obtain the correct edge and producing an acceptable surface finish. Examples of these are; brushing, grinding, dry and wet micro-blasting etc. A correct edge radius can improve the performance of the insert by increasing the strength of the cutting edge to reduce the risk of edge chipping and increasing the tools life. If a too sharp edge is used, it is possible that it will break during processing [5]. In production at Gimo a machine called PER is used in order to measure the edge radius of inserts. The measurement technique in PER is an optical measurement using laser.

In PER, three inserts at a time can be measured. These are placed in a fixture to hold them in a certain position. A laser is then used to focus on the edge to be measured and one to three measuring points are used depending on which geometry to be measured. The program then measures the width and height of the insert's cutting edge, which together gives the edge radius. [14]

### 3 Test screening

As part of this study a test screening was made in order to examine which blasting method that is most appropriate for further investigations. The inserts used for the test screening had been reported with one bad side because of flaking and one better side, making them appropriate for evaluation of the testing methods.

#### 3.1 Experimental

Six different blasting methods differing in size and hardness of blasting media particles and angle of impact were investigated. Henceforth, these will be denoted M1-M6 in the report. The six blasting methods that were examined are presented in Table 1:

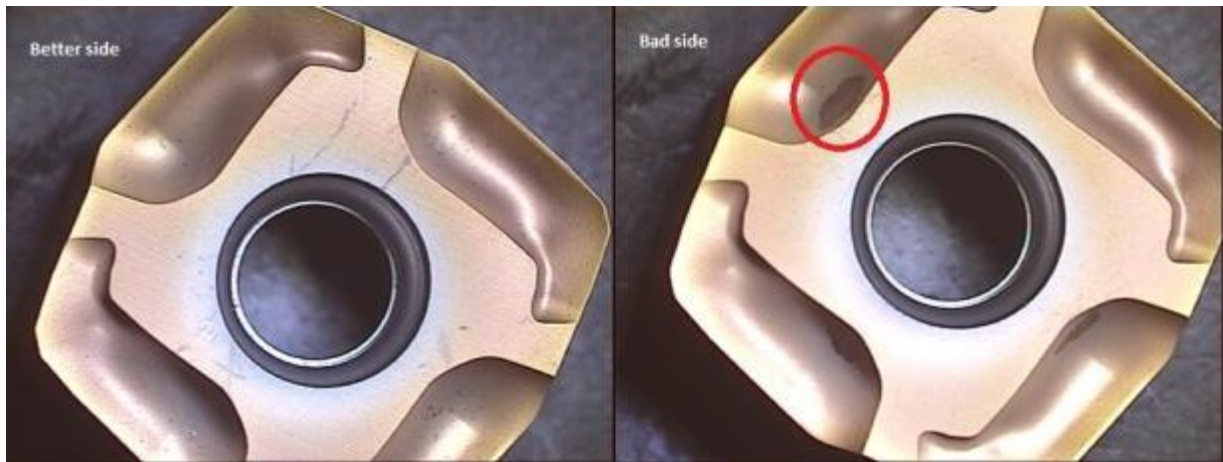
**Table 1: Listing of the different testing methods used in the test screening.**

Method	Mesh	Mean size of particles [μm]	Incidence angle	Dry/wet	Comment
M1	360	22.8 ± 1.5	52	Wet	Harder than coatings
M2	500	12.8 ± 1	52	Wet	Smallest particles
M3	220	55	90	Wet	Different incidence angle
M4	150	89	52	Wet	Coarsest wet particles
M5	120	109	52	Dry	Only dry process and coarsest particles
M6	220	55	52	Wet	Usually uses protective screen, but not in this study.

The particle sizes of the blasting media, together with the mesh sizes adhere to FEPA standards, which use a sieve where the particles pass through. If only 50 % of the particles pass, the average particle size is reached.

All these methods were tested with different evaluation matrices, varying time and pressure according to production standards. The matrices are summarized in Table 2 below. Five different pressures and three different times were used with six inserts in each test. Three of the inserts were tested on the bad side and three on the better side to evaluate the effect of blasting on the different quality that was known to be on the different sides. The turned side of most inserts was flaking even before blasting as can be seen within the red circle in Figure 10 below.





**Figure 10: The difference between the sides of the inserts where the bad side has flaked before blasting.**

The test parameters for the different methods are shown in Table 2 below, where the pressures are given in bar and the time factor is calculated from the cycle time of the machine where a higher time factor means longer cycle time.

**Table 2: Test parameters for testing methods M1-M6**

<u>Test method</u>	<u>Pressures [bar]</u>	<u>Time factors</u>
<b>M1</b>	1.6, 1.8, 2.3, 2.8, 3.2	60, 120 and 200
<b>M2</b>	1.6, 1.8, 2.3, 3.2, 3.7	60, 120 and 200
<b>M3</b>	1.6, 2.0, 2.2, 2.4, 2.8	60, 120 and 200
<b>M4</b>	1.6, 1.8, 2.3, 2.8, 3.2	60, 120 and 200
<b>M5</b>	0.6	1 = approximately 11 min
<b>M6</b>	1.6	60

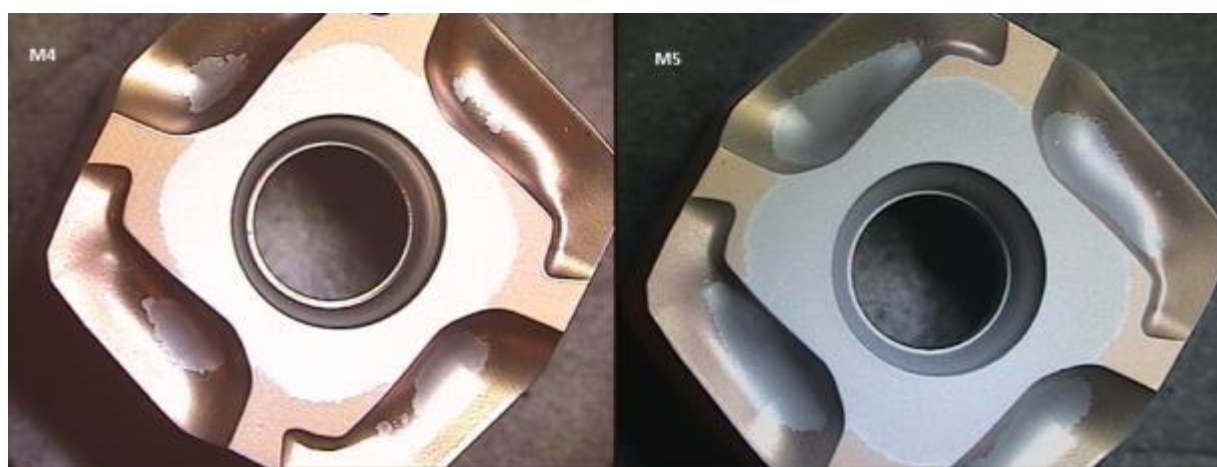
Analysis was made using LOM on all inserts, whereas some inserts were analyzed in SEM.

### **3.2 Results and discussion from test screening**

By analyzing the images from the different tests, it is clear that the coating on one side of the inserts, the bad side, have worse adhesion than the other since it is flaking even before testing. The bad side of the inserts was flaking at the lowest pressure and time for all the methods evaluated, whereas the better side only exhibited flaking at the lowest pressures when subjected to the M4 and M5 methods. However three of the methods, M1, M2 and M3 did not show flaking when using the lowest pressure, this leads to the assumption that blasting of

coated inserts is a promising method in order to investigate the quality of the adhesion of PVD coatings.

As mentioned, not all testing methods showed promising results. For example, as mentioned test method M4 was too strong resulting in an eroded PVD coating even at the lowest pressure and time also on the good side. This makes this method inappropriate for quality testing of PVD coatings and further investigations will not be made using M4. This was also the case using test method M5. The wear at very low pressure, 0.6 bar, was so severe that the PVD coating on both sides was worn off. Figure 11 shows the better side of two inserts tested with M4 and M5 respectively, both inserts showed flaking.



**Figure 11: Comparison using the lowest blasting pressure with M4 and M5 on the better side. The coating has flaked in all chip breakers for both inserts. The inserts were blasted with 1.6 bar pressure and time factor 60 with M4 and a pressure of 0.6 bar and time program 1 with M5.**

Usually when blasting with M6, a covering metal net is used to ensure that the inserts stay in place. But this is not wanted in this test since the effect of the screen cannot be predicted due to the partial shadowing effect the screen has on the inserts. Still, one test was made without protective net but then the inserts instantly flew off the carrier. This leaves three methods, M1, M2 and M3.

### 3.2.1 Testing with M1

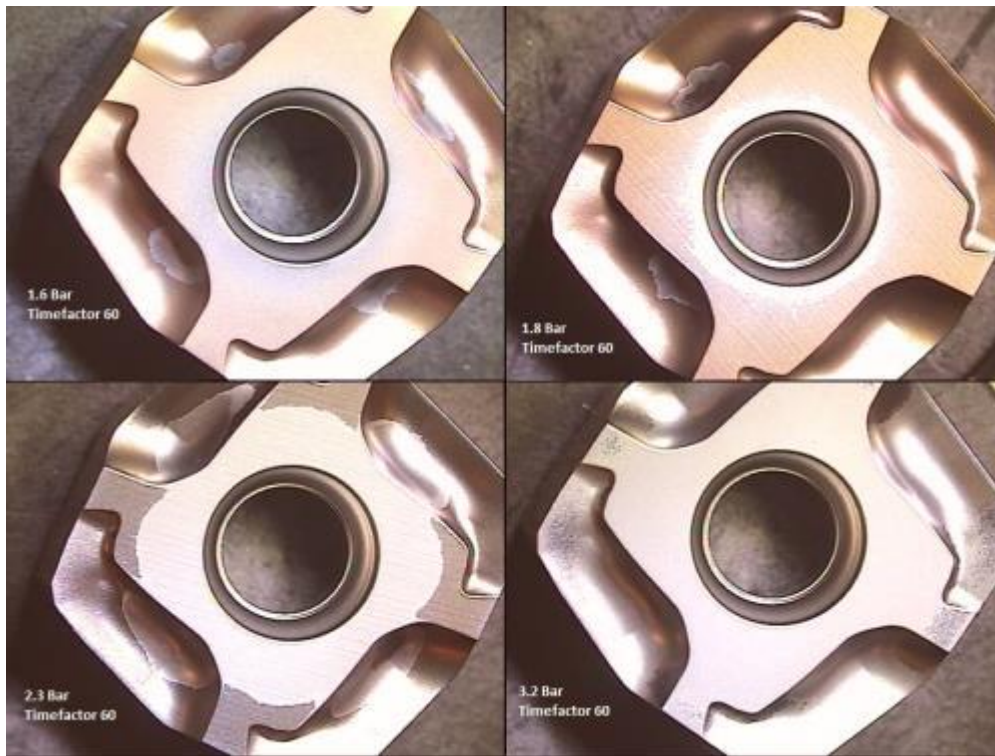
As mentioned earlier, blasting using M1 is done using particles that are harder than the PVD coating, which strongly suggests that wear of the coating would take place. However, in contrast with M4 and M5, the coating wasn't worn to the substrate at low pressures on the better side of the insert. As can be seen in Table 3, the inserts do not flake on the better side until a pressure of 2.8 bar is used. The red boxes in the table mean that flaking has occurred, whereas the white boxes mean that no flaking occurred. The fact that the particles in M1 are harder than the coating and abrasively wear the coating can be seen in Figure 12, since the color of the coating is worn off before it starts to flake. Moreover it can be seen in Figure 13 that the bad side of the inserts showed flaking at all pressures. These results make method M1 promising for further investigation since there is a process window where the coating is not flaked or worn off on the better side but flakes off on the bad side of the insert.

**Table 3: Result matrix of inserts blasted with M1 on both sides of the inserts. Red boxes mean that the coating was either worn down to the substrate or that flaking in the chip breaker occurred.**

<u>M1</u>	Time 60		Time 120		Time 200	
Side of insert:	Better	Bad	Better	Bad	Better	Bad
1.6 Bar						
1.8 Bar						
2.3 Bar						
2.8 Bar						
3.2 Bar						



**Figure 12: Better side of inserts tested with M1. The coating is worn down to the substrate before it flakes which can be seen in the lower picture to the left.**



**Figure 13: Bad side of inserts tested with M1. They flake in the chip breaker at the lowest evaluated pressure, and this occurs before the coating is worn down to the substrate.**

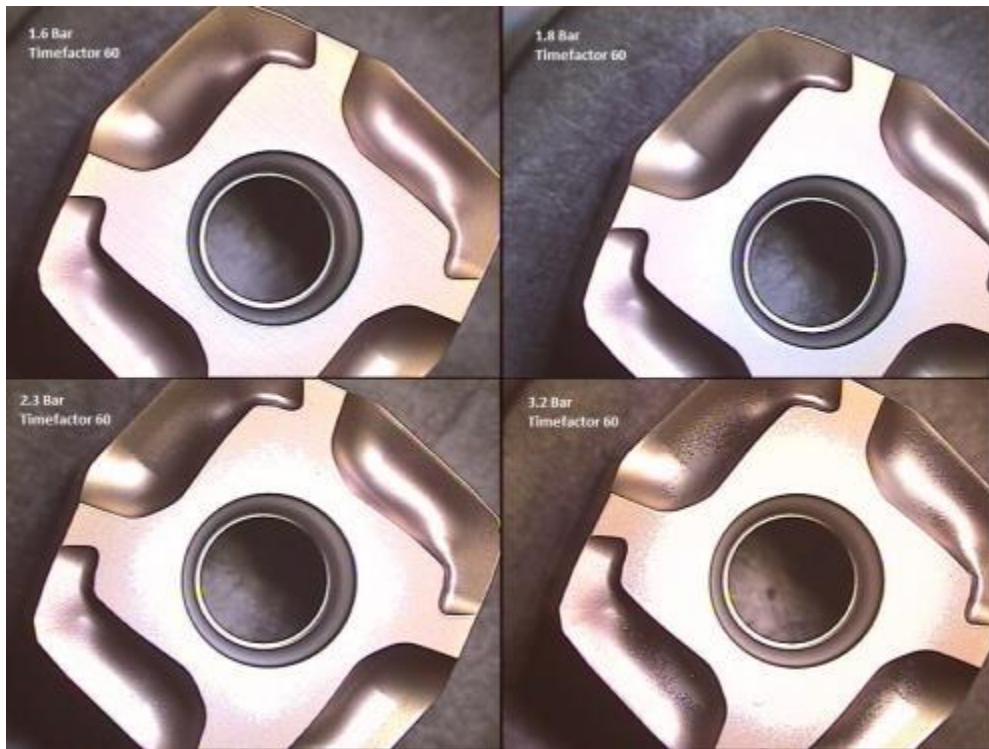
### 3.2.2 Testing with M2

Blasting with M2 showed similar results as those for M1 which is presented in Table 4. But because of the softer and smaller particles, the wear is slightly different since the coloring layer wasn't worn off before flaking. Otherwise, the flaking occurred at approximately the same pressure as seen for M1 on the better side and right away on the bad side, which can be seen in Figure 14 and Figure 15 below. This makes this method just as promising as M1 for further studies.

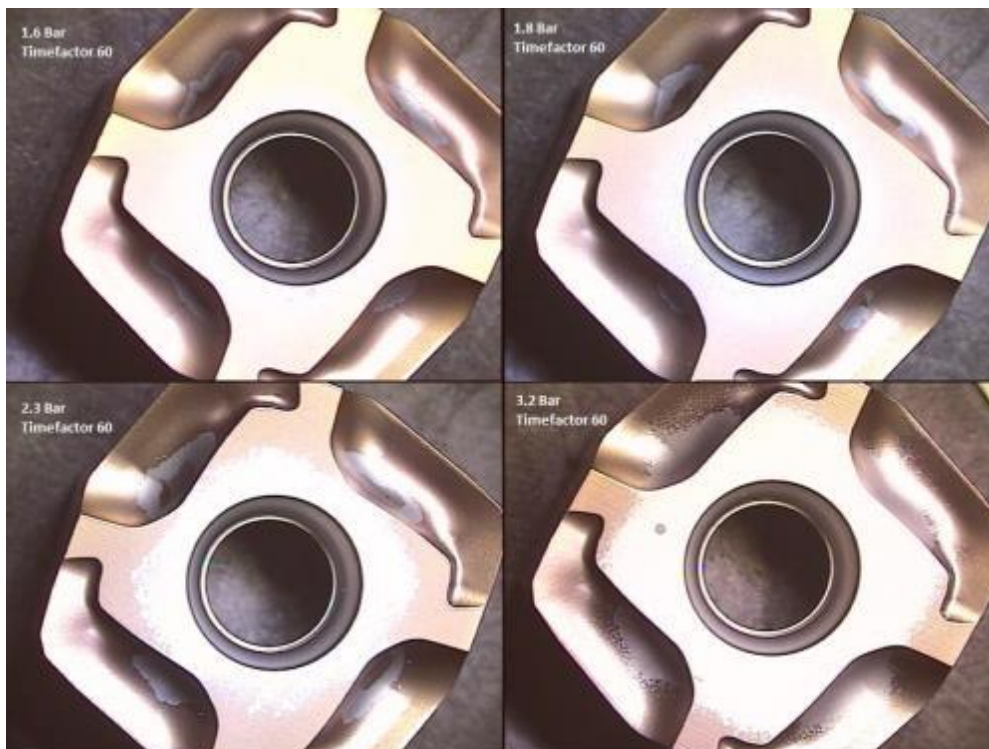
**Table 4: Result matrix of inserts blasted with M2 on both sides of the inserts. Red boxes mean that the coating was either worn down to the substrate or that flaking in the chip breaker occurred.**

<u>M2</u>	Time 60		Time 120		Time 200	
Side of insert:	Better	Bad	Better	Bad	Better	Bad
1.6 Bar						
1.8 Bar						
2.3 Bar						
3.2 Bar						
3.7 Bar						





**Figure 14: Better side of inserts evaluated using M2. The coating is neither worn down to the substrate nor flaked until a pressure of 3.2 bar is used with time factor 60.**



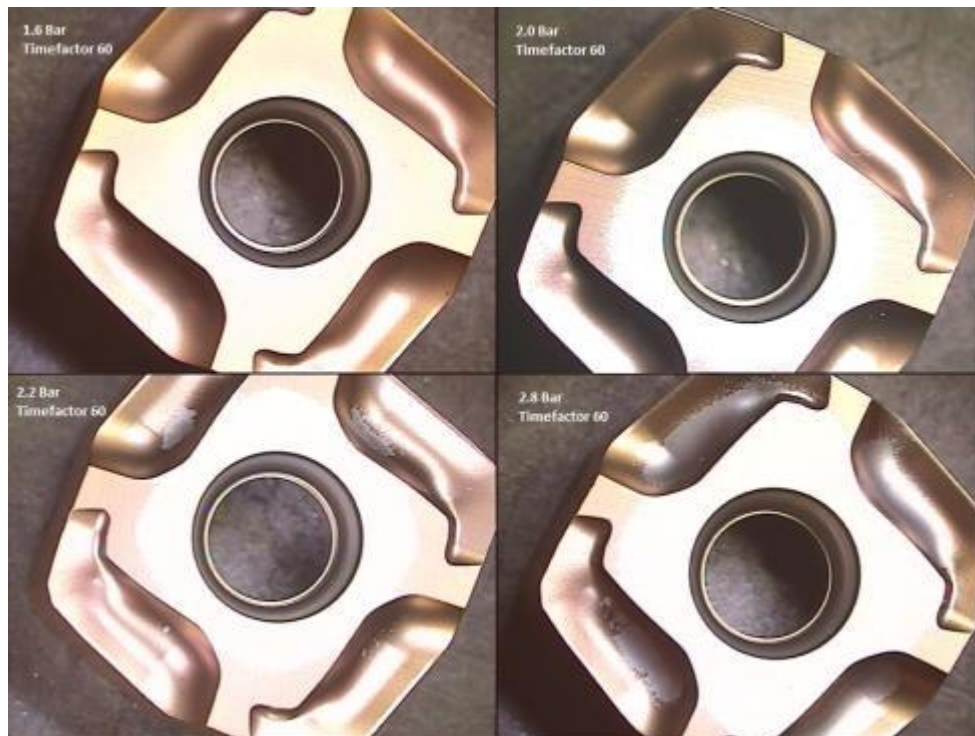
**Figure 15: Bad side of inserts evaluated using M2. It is clearly seen that it flakes in the chip breakers, even at the lowest tested pressure and time factor. The difference from testing with M1 is that the coloring layer of the coating is not worn down before flaking.**

### 3.2.3 Testing with M3

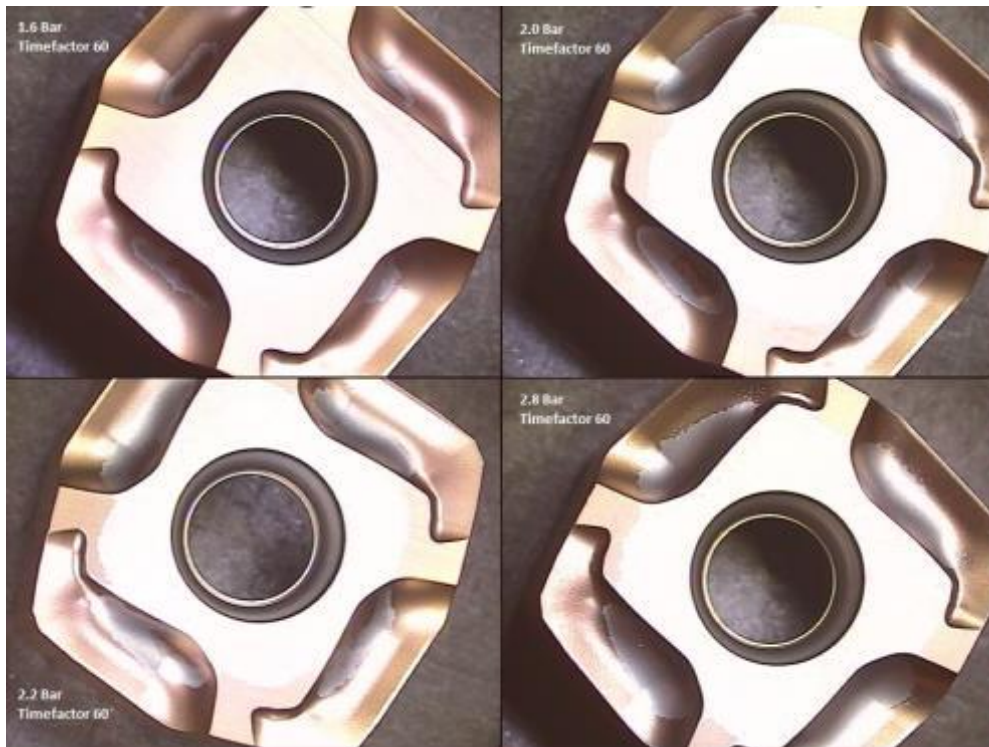
The last blasting method that showed promising results was M3. In a similar manner as the evaluation done using M1 and M2, the coating could withstand the lowest pressures at the better side before flaking, which is shown in Table 5 and Figure 16, whereas the coating flaked on the bad side of the inserts for all pressures and times as observed in Figure 17. The wear pattern looked similar to the other methods where the coating wore off at the top of the inserts and started to flake in the chip breakers at a pressure of 2.2 Bar on the lowest time factor.

**Table 5: Result matrix of inserts blasted with M3 on both sides of the inserts. Red boxes mean that the coating was either worn down to the substrate or that flaking in the chip breaker occurred.**

<b>M3</b>	<b>Time 60</b>		<b>Time 120</b>		<b>Time 200</b>	
<b>Side of insert:</b>	<b>Better</b>	<b>Bad</b>	<b>Better</b>	<b>Bad</b>	<b>Better</b>	<b>Bad</b>
<b>1.6 Bar</b>						
<b>2.0 Bar</b>						
<b>2.2 Bar</b>						
<b>2.4 Bar</b>						
<b>2.8 Bar</b>						



**Figure 16: Better side of inserts tested with M3. The coating flaked when a pressure of 2.2 bar was used as can be seen in the bottom left corner. Otherwise, the coating was not worn down to the substrate.**



**Figure 17: Bad side of inserts tested with M3. Just as for M1 and M2 tested inserts flaking occurred in the chip breakers of the inserts at the lowest used pressure.**

### **3.3 Conclusions test screening**

The results from the test screening showed that three testing methods will be investigated further, M1, M2 and M3.



## 4 Experimental procedure

### 4.1 Different amount of pre-treatment blasting

A batch of inserts was produced with untreated inserts with the same geometry as in the test screening. These inserts were received directly from grinding after the sintering process, meaning that the following processing could be controlled. This was done to be able to process inserts with varying adhesion quality between the coating and surface by applying different degrees of pre-treatment blasting and to find out what type of process instability in pre-treatment that could be found by the test methods. The inserts were analyzed in untreated state using LOM and later blasted with varying pressure and time as pre-treatment. Images of both sides of an untreated insert are shown in Figure 18 below. The different sides of the insert will from now on be called top and bottom side instead of better and bad side.



**Figure 18: LOM image of top and bottom side of an untreated insert.**

Four different amounts of pre-treatment blasting, henceforth denoted P1-P4 was used:

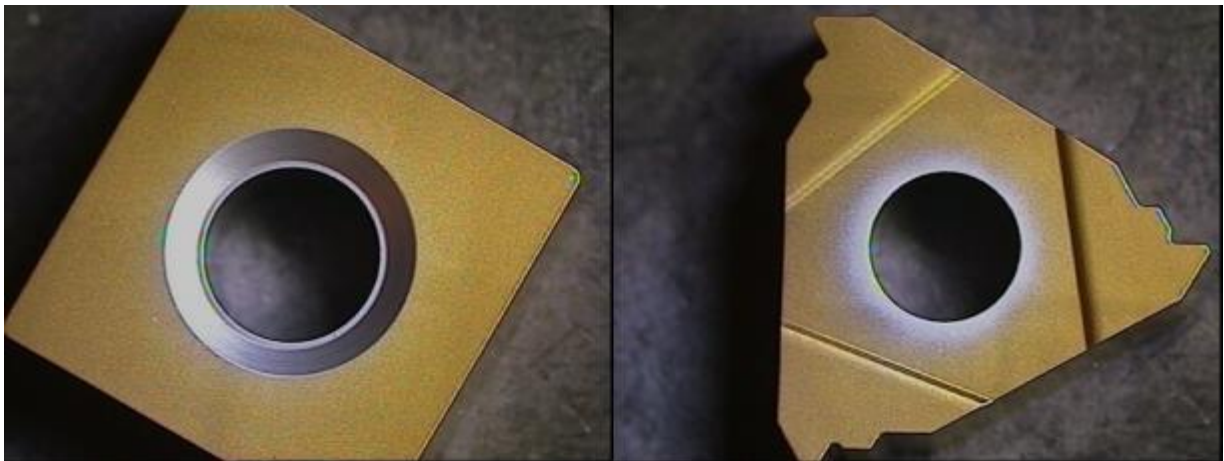
- **P1:** No blasting.
- **P2:** Low blasting, 1.8 bar pressure with time factor 70 in 150 mesh wet blasting process.
- **P3:** Standard blasting, towards an edge radius of around 35  $\mu\text{m}$  which implies process parameters of approximately 3.2 bar pressure with time factor 70 in 150 mesh wet blasting process.
- **P4:** Standard blasting + 500 mesh, same standard recipe as standard blasting, but with the addition of 500 mesh blasting at 3.2 bar pressure with time factor 70 in wet blasting process to increase the cleaning further.

30 inserts was used in each blasting setup, giving a total amount of 120 inserts. Inserts 1-5, 11-15 and 21-25 for each amount of pre-treatment blasting were marked on both sides to be able to analyze the same part before and after blasting as well as before and after PVD coating. The rest of the inserts were only marked on one side. After coating, inserts 1-10 were evaluated



using method M1, inserts 11-20 using M2, and 21-30 using M3, as seen in Table 6 and Table 7. The amount of pre-treatment blasting was decided from what is used in production for this type of inserts. After pre-treatment blasting, the inserts' edge radii were measured in PER, optical laser measurement, in order to see any differences between the amounts of blasting. Nine inserts from each blasting setup were measured. Also, pictures using LOM were taken on both sides after blasting to evaluate any differences on the surface. This was followed by another cleaning before PVD coating.

A TiAlN coating process in the latest generation machine was chosen since it is a common PVD process. It is a different process with similar properties as the coating used for the inserts in the test screening part. This process uses a TiAl alloy that reacts with nitrogen gas during the process to create a TiAlN coating that is approximately 4  $\mu\text{m}$  thick. All inserts were placed with the top side up on 3mm pins with spacers between them. Ten inserts were placed on each pin with insert number 1, 11, 21 at the bottom of each pin. They were all placed in the same PVD process together with other inserts used in production and 6 extra furnace control inserts as well as three extra threading inserts. The flat geometry of the furnace control insert and threading insert is shown in Figure 19.



**Figure 19: Furnace control insert to the left and a threading insert to the right.**

A typical formation of inserts in the PVD furnace consists of three parts; table, tree, and pin rotate during the process to achieve as smooth coating as possible. No failure was reported during the process. The inserts were analyzed individually after coating to evaluate any potential differences in surface appearance.

M1, M2, and M3 were used to evaluate the coating quality in all pre-treatment setups. The recipe for the different testing methods was decided by testing the harshest treatment where the better side on the test screening inserts did not flake. Pressures of 2.3 bar for M1 and M2 and 2.0 bar for M3 were used on two inserts from each pre-treatment blasting amount with time factor 60. Insert 6 and 10 in M1, 16 and 20 in M2 and 26 and 30 in M3 were used for all pre-treatment blasting setups. Inserts 6, 16, and 26 were blasted on the top side and inserts 10, 20, and 30 were blasted on the bottom side. All inserts flaked on both sides resulting in that a lower pressure was used for the tests. The pressure that was decided to be used for testing was

1.6 bar using M1, and 1.8 bar using M2 and M3, where all inserts were blasted with time factor 60.

Six inserts from each amount of pre-treatment blasting were used for the main tests. The test cycle for all tested inserts is shown in Table 6 and Table 7. All inserts for each testing method were blasted on the same plate. After testing, all blasted inserts were evaluated in LOM and some inserts using SEM.

**Table 6: Test cycle of insert with pre-treatment No-blasting and Low blasting.**

<b>Amount of pre-treatment:</b>	<b>P1 30 inserts</b>			<b>P2 30 inserts</b>		
<b>Test method: (marking)</b>	M1 10 inserts (1-10)	M2 10 inserts (11-20)	M3 10 inserts (21-30)	M1 10 inserts (1-10)	M2 10 inserts (11-20)	M3 10 inserts (21-30)
<b>Insert used for blast testing:</b>	<b>Top side:</b> 1, 2, 7 <b>Bottom side:</b> 3, 4, 8	<b>Top side:</b> 11, 12, 17 <b>Bottom side:</b> 13, 14, 18	<b>Top side:</b> 21, 22, 27 <b>Bottom side:</b> 23, 24, 28	<b>Top side:</b> 1, 2 and 7 <b>Bottom side:</b> 3, 4, 8	<b>Top side:</b> 11, 12, 17 <b>Bottom side:</b> 13, 14, 18	<b>Top side:</b> 21, 22, 27 <b>Bottom side:</b> 23, 24, 28

**Table 7: Test cycle of insert with pre-treatment standard blasting and standard blasting + 500 mesh.**

<b>Amount of pre-treatment</b>	<b>P3 30 inserts</b>			<b>P4 30 inserts</b>		
<b>Test method: (Insert number)</b>	M1 10 inserts (1-10)	M2 10 inserts (11-20)	M3 10 inserts (21-30)	M1 10 inserts (1-10)	M2 10 inserts (11-20)	M3 10 inserts (21-30)
<b>Inserts used for testing:</b>	<b>Top side:</b> 1, 2, 7 <b>Bottom side:</b> 3, 4, 8	<b>Top side:</b> 11, 12, 17 <b>Bottom side:</b> 13, 14, 18	<b>Top side:</b> 21, 22, 27 <b>Bottom side:</b> 23, 24, 28	<b>Top side:</b> 1, 2 and 7 <b>Bottom side:</b> 3, 4, 8	<b>Top side:</b> 11, 12, 17 <b>Bottom side:</b> 13, 14, 18	<b>Top side:</b> 21, 22, 27 <b>Bottom side:</b> 23, 24, 28

#### 4.2 Effect of etching in the PVD process.

In order to evaluate the effect of etching in the PVD process has on the coating, two PVD coating runs were made with lower and no etching bias respectively in a PVD process.

50 inserts were pre-treated using P3 blasting setup, resulting in blasting with 3.2 bar pressure with time factor 70 in 150 mesh wet blasting process to achieve an edge radius of approximately 35µm. The inserts were divided into two groups, where thirty inserts were coated with the same TiAlN process as the inserts in the previous part, but with a lower etching bias than usual. Ten of these inserts were placed with the bottom side up in the PVD process and the rest with the top side up. The inserts were charged on pins, with the pins not used for the experiment filled with scrap inserts, or so called dummies. Additionally, six furnace control inserts and six threading inserts were also included in the process. The same coating was used for the remaining twenty inserts, but this time with no etching at all in the PVD process. The inserts were charged in the same way, and three furnace control inserts and three threading inserts were also used in this process. No failures were reported during the PVD process. The surface quality of the inserts was evaluated using LOM.

All inserts were evaluated using with the same pressure and time, as the inserts with different amounts of pre-treatment blasting. However, only M1 and M2 were used for these tests. The test plan for blasting testing in this part can be seen in Table 8 below. The inserts were also analyzed using LOM to map the amount of wear.

**Table 8: Test plan for blasting testing on inserts with less etching than normal in the PVD process.**

<b>PVD process:</b>	<b>Lower etching bias (20 inserts)</b>		<b>No etching bias (20 inserts)</b>		<b>Bottom side up in PVD (10 inserts)</b>
<b>Test method:</b>	M1 10 inserts (1-10)	M2 10 inserts (11-20)	M1 10 inserts (1-10)	M2 10 inserts (11-20)	M2 10 inserts (21-30)
<b>Insert used for blast testing:</b>	<b>Top side:</b> 1, 2 and 7 <b>Bottom side:</b> 3, 4 and 8	<b>Top side:</b> 11, 12 and 17 <b>Bottom side:</b> 13, 14 and 18	<b>Top side:</b> 1, 2 and 7 <b>Bottom side:</b> 3, 4 and 8	<b>Top side:</b> 11, 12 and 17 <b>Bottom side:</b> 13, 14 and 18	<b>Top side:</b> 21, 22 and 27 <b>Bottom side:</b> 23, 24 and 28

### 4.3 Directional dependence in PVD furnace

In order to investigate if the adhesion is affected by how the inserts is turned in the furnace, was a test with the inserts pointing in different directions in the PVD process made. 50 inserts were used, all blasted using pre-treatment P3. In the PVD process, 20 inserts were placed normally with the top side up, 20 inserts were placed with the top side down and ten inserts were placed on a magnetic tube, five with the top side pointing at the targets and five with the bottom side. A picture of the magnetic tube can be seen in Figure 20. Inserts placed with the top side up will from now on be denoted TSup, and the inserts with the bottom side up BSup. The same PVD process as earlier was used, and the inserts were placed in the furnace with a normal production order. The inserts were evaluated using both M1 and M2. Analysis was made using LOM.



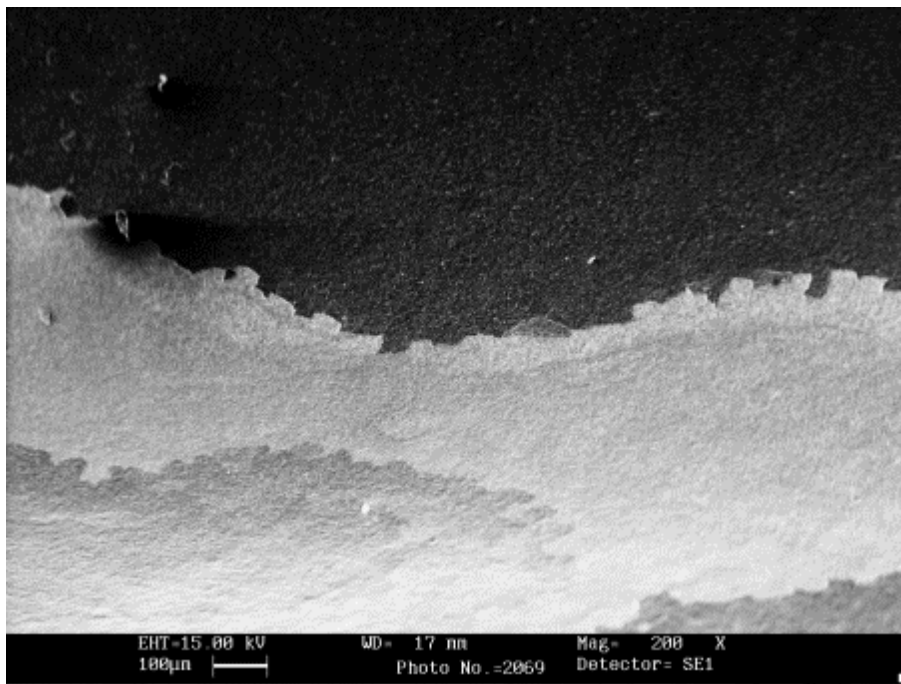
**Figure 20: Picture of charging of inserts on magnetic tube.**

## 5 Results

### 5.1 Wear analysis in SEM

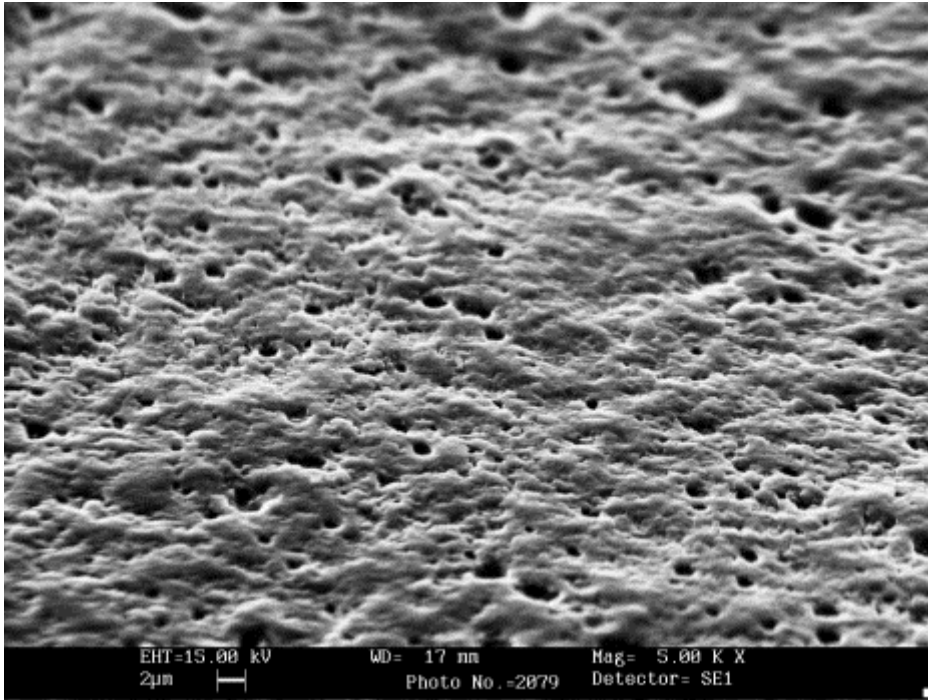
The results from wear analysis in SEM of the test screening part are shown in Figure 21- Figure 24.

In Figure 21 below, it is clear that flaking occurs when blasting with M2 at the lowest pressure and time. This can be seen since there are sharp edges around the light grey areas which have flaked. The same thing was observed on inserts tested with M1 and M2 on the bad side of the inserts. These figures can be seen in Appendix 1.

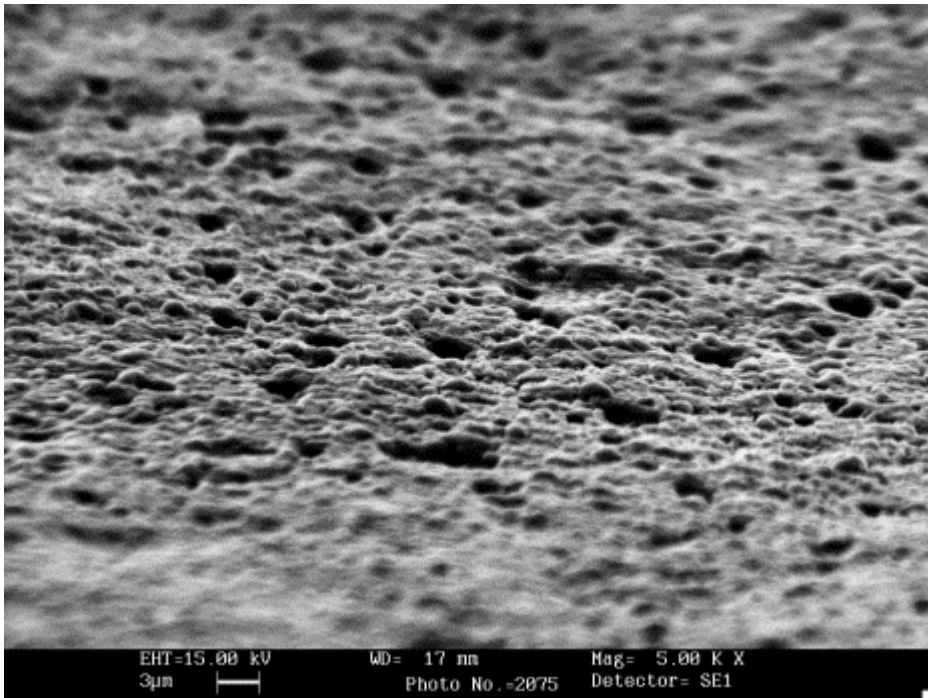


**Figure 21: Flaking on the bad side of an insert tested in M2, shown in 200 times magnification. The light grey areas have flaked down to the substrate whereas the dark grey area is the coating.**

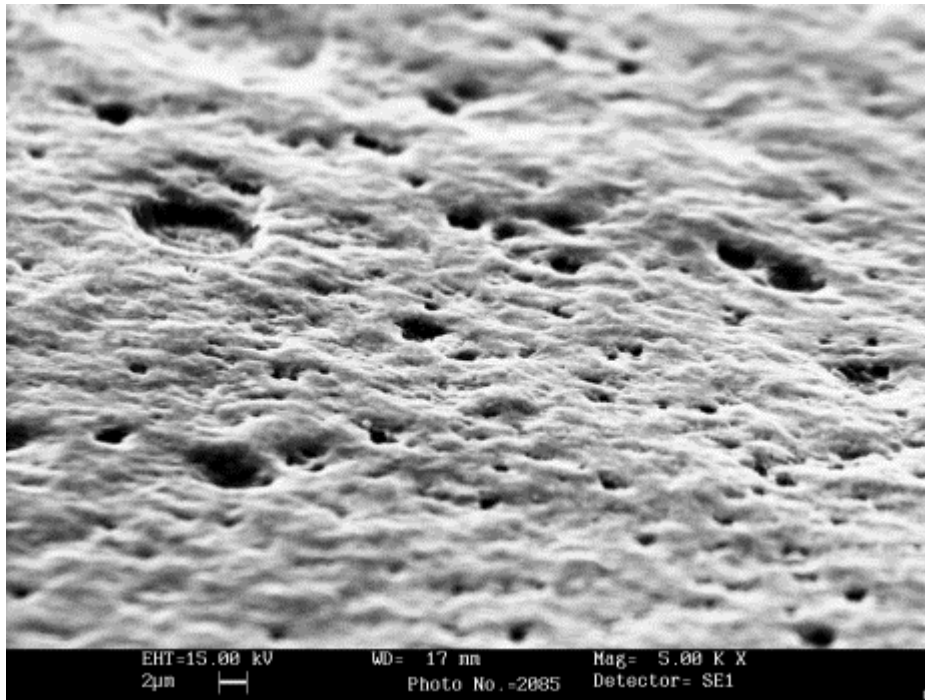
Another observation made in the SEM analysis was the difference in wear with the different blasting particles. A significant difference on the surface of the inserts could be observed at higher magnifications, where the inserts tested using M4 had a smoother surface than the inserts tested with M1 and M2. The inserts tested using M2 received the roughest surface of the three. Examples of surface conditions of the different inserts can be seen in Figure 22- Figure 24 below.



**Figure 22: SEM image showing remaining coating on the surface of an insert tested with M1 at the pressure and time when it started to flake, 1.6 bar and time factor 60.**



**Figure 23: SEM image showing the remaining coating on the bad side of an insert tested using M2 at the pressure and time when it started to flake, 1.6 bar and time factor 60.**



**Figure 24:** SEM image on the bad side of an inserts tested with M4 at the lowest pressure and time with 5000 times magnification. A relatively smooth surface on the remaining coating compared to the inserts tested using M1 and M2.

## 5.2 Main tests

The results from the blasting tests on inserts with different pre-treatments are presented in six different categories below.

- Influence of different amounts of pre-treatment on the surface structure before testing.
- Influence of etching bias in the PVD process on coating adhesion.
- Evaluation of different amount of pre-treatments effect on adhesion.
- Adhesion evaluation between the different testing methods.
- Varying etching bias in the PVD process, and.
- Directional dependence in PVD furnace.

### 5.2.1 Influence of different amounts of pre-treatment on the surface structure before testing

After coating, pictures were taken on the same inserts as after pre-treatment blasting. The picture of the inserts after coating is seen in Figure 25. A small variation between the inserts is where the P1 insert received a dirty surface with many small black spots on the top side and a shiny and color shifting surface on the bottom side as can be seen in first row of the figure. The P2, P3 and P4 inserts exhibited similar surface qualities, especially on the bottom side. On the top side of these inserts, a small difference could be seen, where a slightly higher number of small black spots were observed on the P4 inserts than the P2 and P3 inserts.



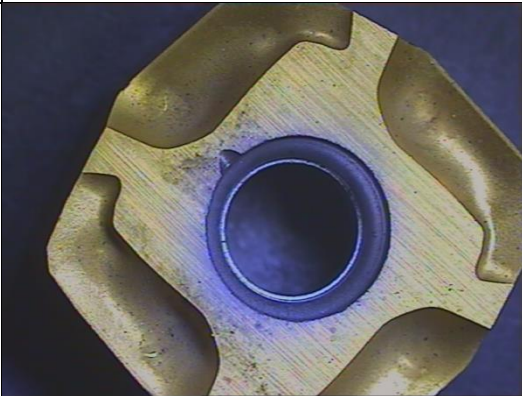
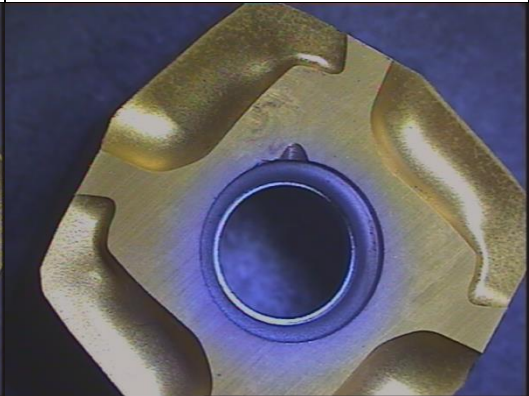
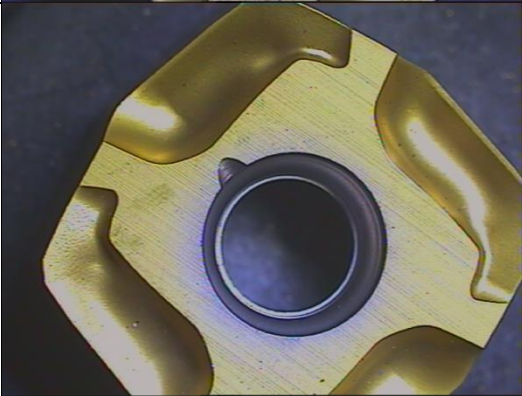

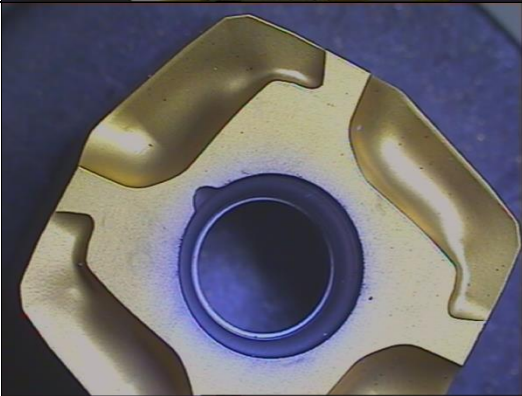
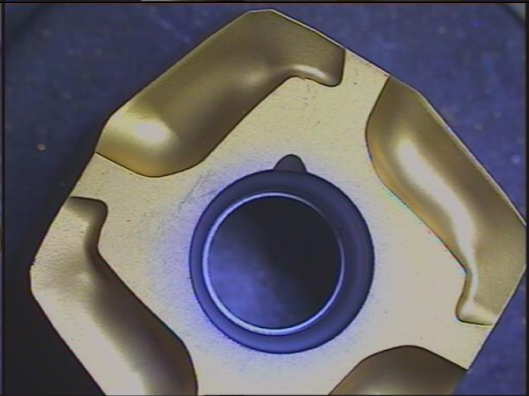
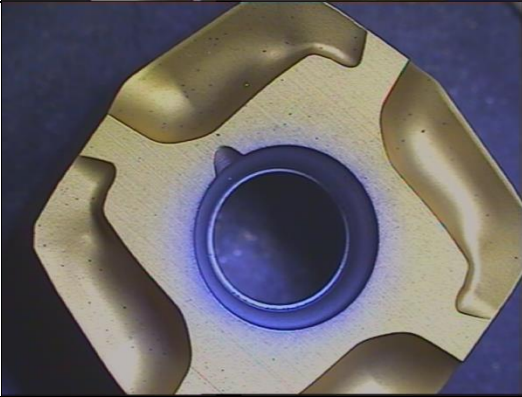
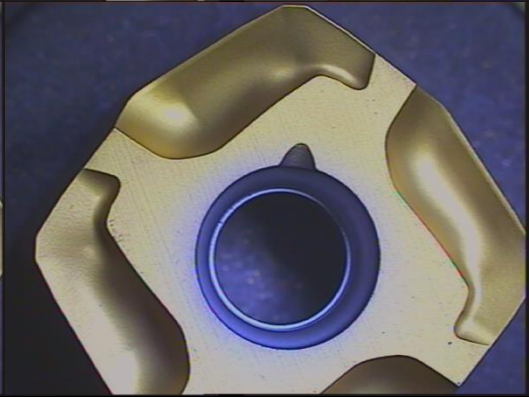
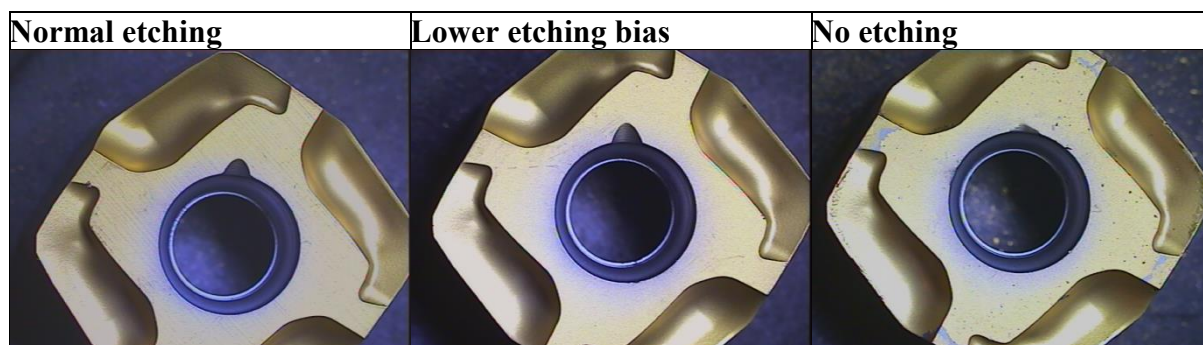
<u>Pre-treatment</u>	Top	Bottom
P1		
P2		
P3		
P4		

Figure 25: Top and bottom side of insert 1 after coating for all four pre-treatment blasting setups.



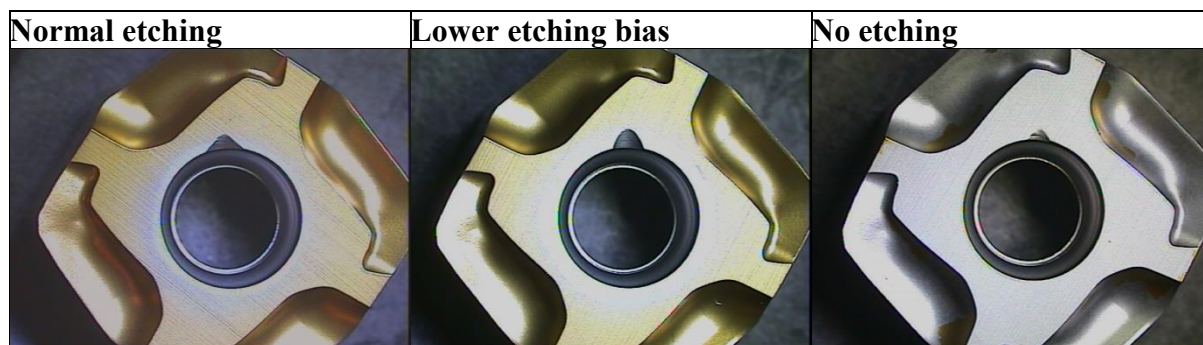
### 5.2.2 Influence of etching bias in the PVD process on coating adhesion

A comparison was made between the lower etching bias inserts and the inserts processed as a normal production order in order to evaluate if the method can separate different levels of plasma etching before coating. This also shows the importance of etching in the PVD process which can be seen when comparing the inserts from the normal PVD process and the process with lower etching bias. Figure 26 shows the difference in surface structure after coating. The only deviating insert is the one coated without any prior etching. The difference consisted in observed flakes on parts of the insert.



**Figure 26: Showing the bottom side of insert 14 after coating for three different amounts of etching in the PVD processes.**

The results when testing using M1 is seen in Figure 27. It is shown that the normal etching and the lower etching inserts look the same, whereas the inserts processed without etching did not have any coating left after testing.



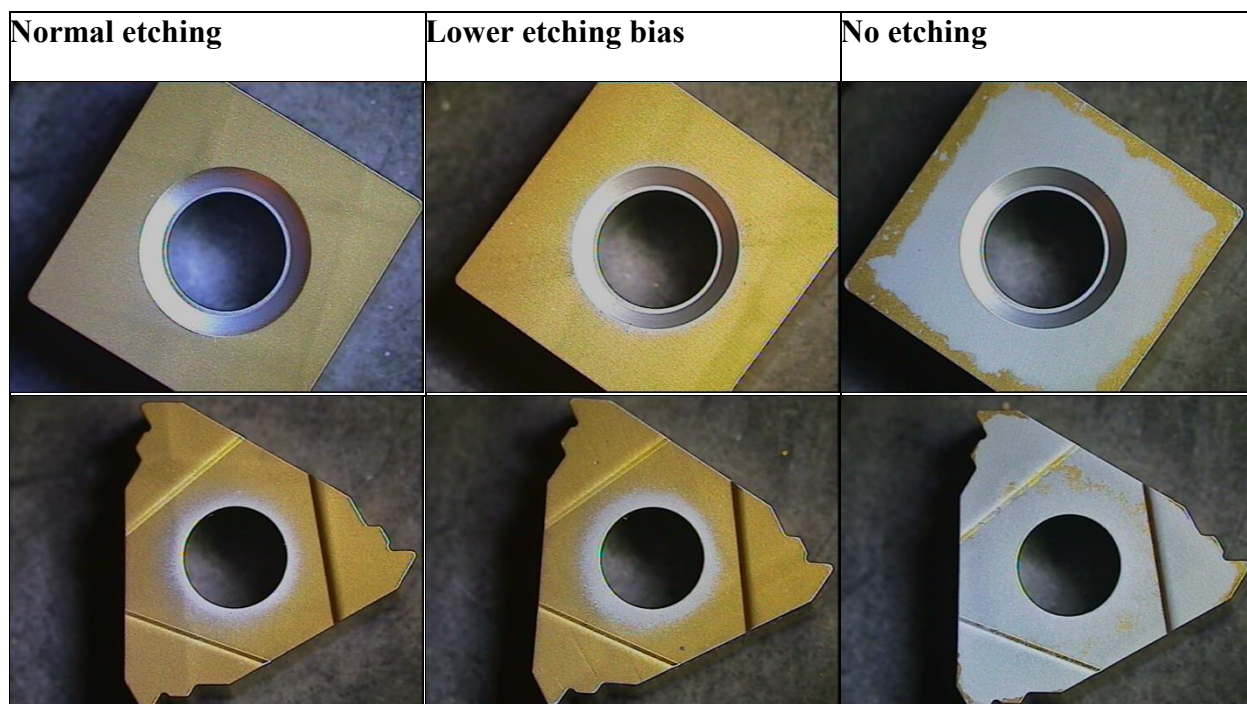
**Figure 27: The bottom side of insert 4 after testing using M1. The insert has been processed with different amounts of etching in the PVD process.**

A significant difference was showing after testing using M2, shown in Figure 28 below. The insert with normal etching process only exhibited slight flaking in one chip breaker which is seen in the top left corner. The insert with lower etching bias in the PVD process exhibited extensive flaking and the insert without etching did not have any coating left.



**Figure 28:** Showing the bottom side of insert 14 after testing with M2. The insert has been processed with different amounts of etching in the PVD process.

The varying quality can also be confirmed by observing the oven control and threading inserts that were included in all the PVD processes. Figure 29 shows that the inserts without etching loses almost all coating after testing whereas the inserts processed with lower etching bias and normal etching both withstand the wear from blasting. All inserts were evaluated using M2 but the same tendency as the evaluated inserts using M1 shown in Figure 27 was observed.



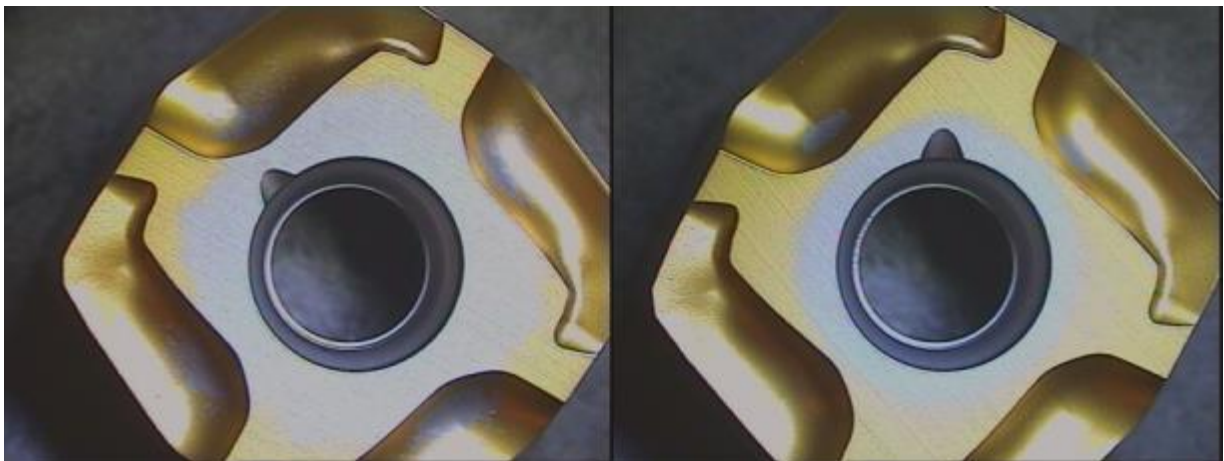
**Figure 29:** Top side of furnace controls (top row) and threading inserts (bottom row) of the normal etching, lower etching bias and no etching processes. All inserts were evaluated using the M2 method.

### 5.2.3 Evaluation of different amount of pre-treatments effect on adhesion

The result from the tests where inserts had been exposed to different pre-treatment blasting setups is visualized in Table 9. Red marking in the table means that the insert flaked in all chip breakers, when blasted after coating, whereas yellow color means that flaking occurred in one or two chip breakers, and white boxes denote that no sign of flaking was observed. The numbers in the tables denote the number of the insert included in the test. Figure 30 show pictures of typical appearances of the classification. Pictures of the insert are shown in Appendix 2 but some typical pictures from the different pre-treatment and after testing with M1, M2 or M3 can be seen in Figure 31.

**Table 9: Results from tests with different amounts of pre-treatment and later tested in M1, M2 and M3. Red boxes indicates that all chip breakers flaked, yellow area indicates that one or two flaked and white area means that the insert did not show flaking.**

<u>Amount of pre-treatment:</u>	<u>P1</u>			<u>P2</u>			<u>P3</u>			<u>P4</u>		
<u>Test method:</u>	<u>M1</u>	<u>M2</u>	<u>M3</u>	<u>M1</u>	<u>M2</u>	<u>M3</u>	<u>M1</u>	<u>M2</u>	<u>M3</u>	<u>M1</u>	<u>M2</u>	<u>M3</u>
Top side	1	11	21	1	11	21	1	11	21	1	11	21
Top side	2	12	22	2	12	22	2	12	22	2	12	22
Top side	7	17	27	7	17	27	7	17	27	7	17	27
Bottom side	3	13	23	3	13	23	3	13	23	3	13	23
Bottom side	4	14	24	4	14	24	4	14	24	4	14	24
Bottom side	8	18	28	8	18	28	8	18	28	8	18	28



**Figure 30: The insert to the left is a typical red mark in the tables below whereas the insert to the right is a yellow mark since it only exhibits flaking in two of the four chip breakers. The figure is showing insert 21 and 24 with P4 pre-treatment.**

When M1 was used as testing method, flaking in more than two chip breakers only occurred for inserts with pre-treatment, P1. Two of the inserts with P2 pre-treatment flaked in less than two chip breakers on the top side whereas the remaining did not show any flaking. There was no observed difference between the inserts with P3 and P4 pre-treatments after testing. Neither

of them flaked and looked similar after testing. With the exception for the P2 inserts, no difference in wear between the sides of the inserts was observed.

When M2 was used for testing the result, a difference in wear was observed between the top and bottom sides for two pre-treatment setups; P2 and P4, images is shown in Appendix 3. The inserts for both these pre-treatments flaked on the top side in all chip breakers whereas less flaking or no flaking was observed on the bottom side. The most pronounced difference between the sides was observed for the inserts with P4 pre-treatment, where all evaluated inserts exhibited flaking on the top side but none on the bottom side. All P1 inserts flaked on both sides and the P3 inserts flaked in less than two chip breakers on both sides, without any observable distinction between the sides.

For the inserts tested with M3 no clear difference between the sides could be observed except for the P4 pre-treated inserts, where all chip breakers exhibited flaking upon testing on the top side, but only in two or less chip breakers upon testing on the bottom side. Images of these inserts can be seen in Appendix 3. The results indicate that the M3 method wore the coating more than the other two testing methods. This can also be seen in the figures in Appendix 2, where the worn area is larger on the top side compared to the bottom side, both in the chip breakers and on the flat part of the inserts.

#### 5.2.4 Adhesion evaluation between the different testing methods

The difference in wear between the different testing methods is presented in Figure 31. The top side of inserts that have been placed in the same position in both PVD process and testing but with different pre-treatment is shown in all pictures. In the first row, the inserts tested in M3 and pre-treated in P1 is presented. They differ from the other inserts, since the wear is more severe than for the others. However, the inserts flaked using all three different testing methods when pre-treated with P1.

The insert with P2 pre-treatment also exhibited a difference between the testing methods. Heavier wear was seen on the insert tested using M3 than on the other two. An onset of flaking could be observed for the insert tested using M2, where small parts of the coating in the chip breaker were removed. The inserts tested using M1 did not show any flaking at all when using P2 as pre-treatment, which is observed in the second row of Figure 31. Also, another observation that can be made in the figure is that the area where the coating is worn down to the substrate on the flat side of the inserts is bigger for the inserts evaluated using M3 than for the other two methods. This is also the case when investigating the P3 and P4 pre-treated inserts as can be seen in the bottom half of the figure.

The standard pre-treated inserts, P3, is shown in the third row, and for these inserts only the M3 inserts were flaking in the chip breaker. The wear on the flat side of the insert is also larger for M3 than for the two inserts blasted by M1 and M2. Inserts pre-treated using P4 exhibited flaking when tested using M3 but also when using M2. However, when testing with M1, no flaking was observed. The flaking when using M2 and M3 look similar to each other in the chip breaker whereas the M3 method wore the slightly more on the flat part of the insert.

As a summary of this part of the results, all inserts flaked using the M3 testing method. This means that M3 as a method cannot be used to separate the different pre-treatments. Whereas



only some of the inserts flaked using M2 testing method and only the insert with P1 pre-treatment flaked using M1 as testing method. This makes the M2 method the most promising method because it gave a difference between all levels of pre-treatments, P1-P4, whereas the others did not.

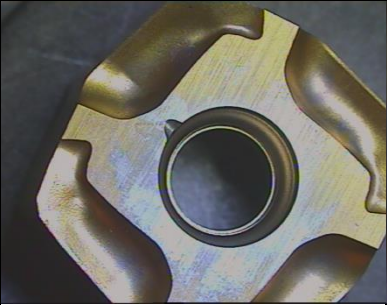

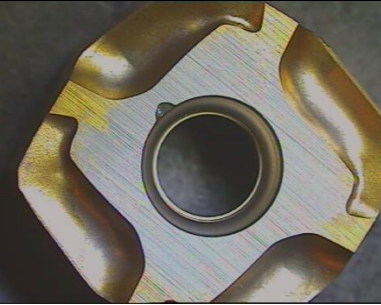
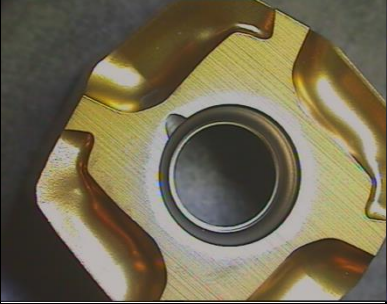
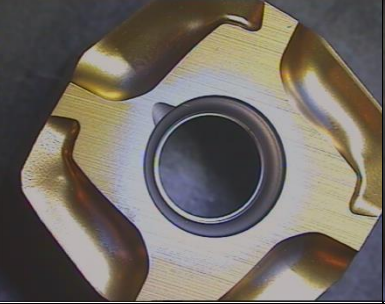
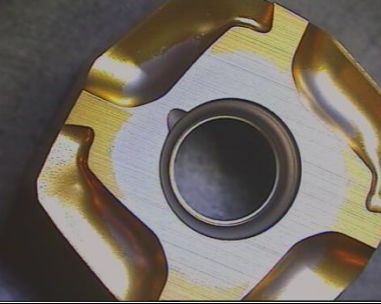
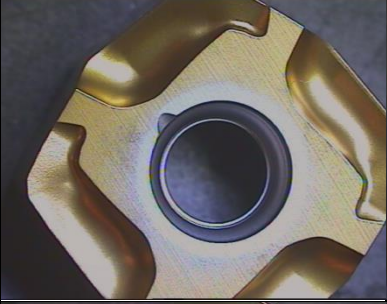

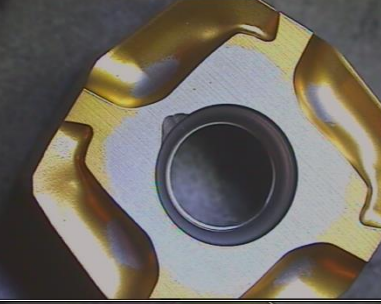
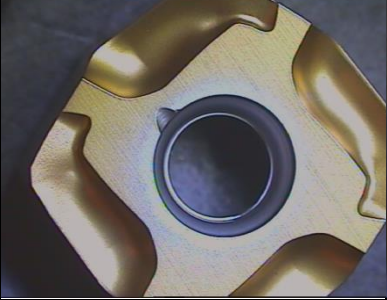
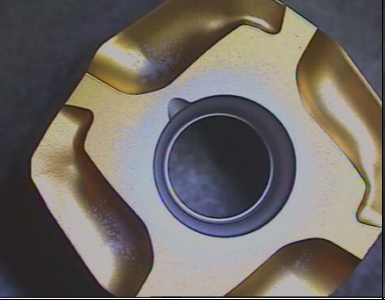

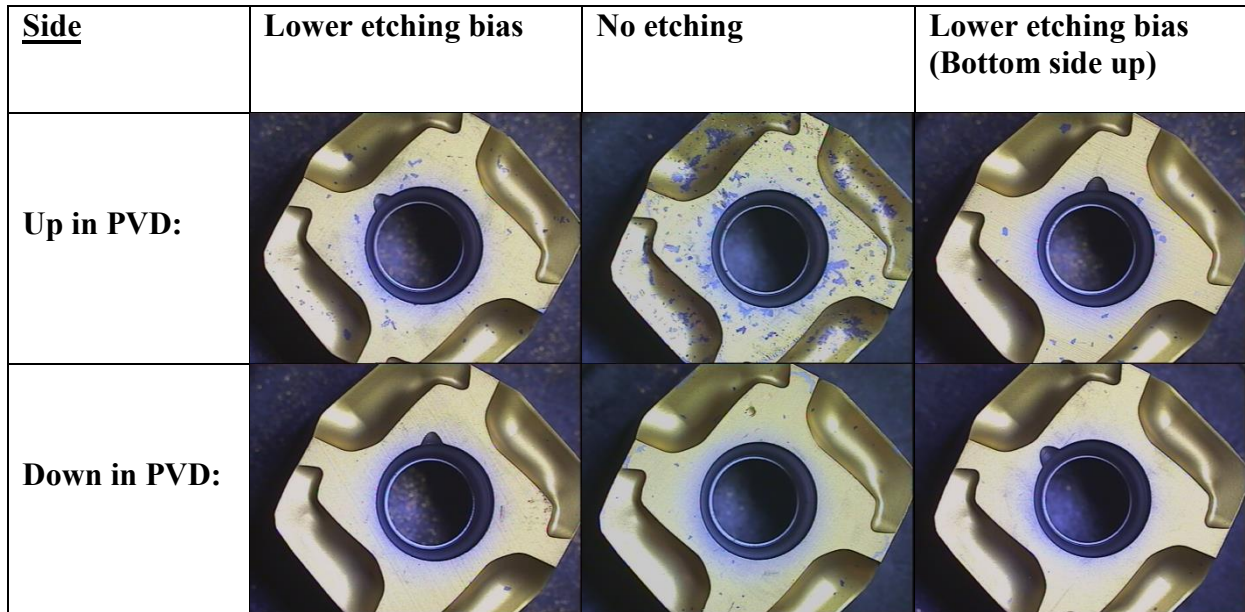
	<u>M1</u>	<u>M2</u>	<u>M3</u>
<u>P1</u>			
<u>P2</u>			
<u>P3</u>			
<u>P4</u>			

Figure 31: The top side of inserts 2, 12, and 22 for all pre-treatment setups and all three test methods.

### 5.2.5 Varying etching bias in the PVD process

When reducing the etching bias, the coating adhesion suffered severely, with large areas of flaking, Figure 32. The phenomenon was especially observed on the insert side facing upwards in the PVD furnace during etching. As seen in Figure 32, the inserts that was coated without any etching at all suffered even worse from flaking. The downwards facing side in the PVD process looks normal for the inserts that was processed with lower etching bias.



**Figure 32: Comparison of the surfaces between the inserts sides and amounts of etching in the PVD process.**

The results from the blasting tests with inserts processed with less or no etching in PVD can be seen in Table 10. As seen, all inserts processed with lower etching bias flaked in all blasting tests except for the ones tested in M1 testing method. These inserts only showed flaking on the top side which was pointing upwards in the PVD process.

**Table 10: Results from blasting test on inserts with different amounts of etching in the PVD process. Red area indicates that all chip breakers flaked, yellow area indicates that two or less flaked and white area mean that the insert did not show flaking.**

<u>PVD process:</u>	<u>Less Etching</u>			<u>No etching</u>	
<u>Tested in:</u>	<u>M1</u>	<u>M2</u>	<u>M2</u> <u>Bottom side up in</u> <u>PVD</u>	<u>M1</u>	<u>M2</u>
Top side	1	11	21	1	11
Top side	2	12	22	2	12
Top side	7	17	27	7	17
Bottom side	3	13	23	3	13
Bottom side	4	14	24	4	14
Bottom side	8	18	28	8	18

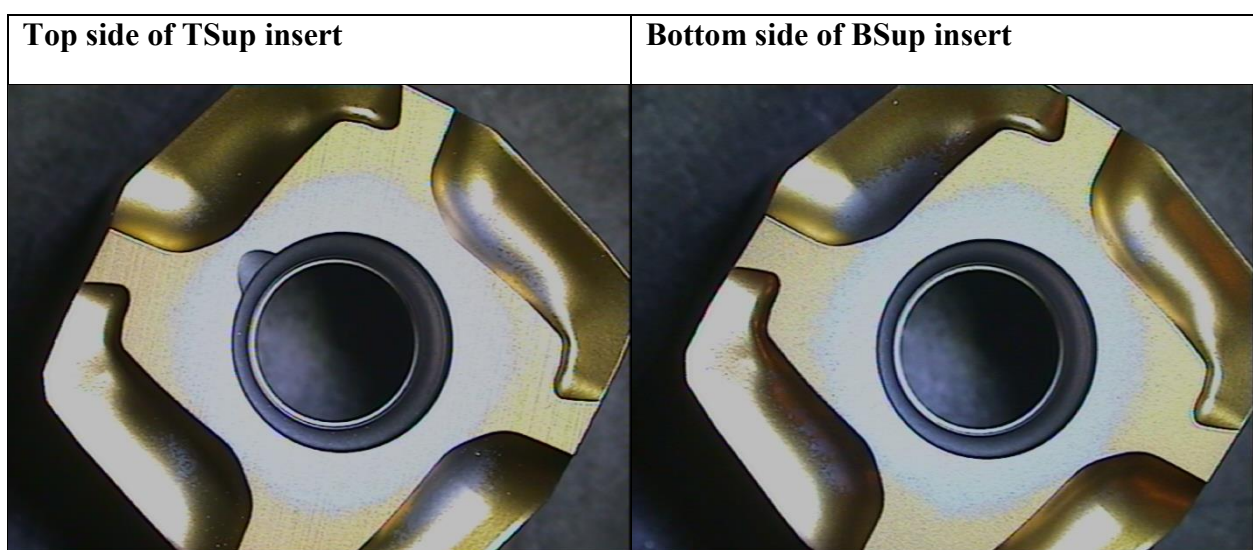
### 5.2.6 Directional dependence in PVD furnace.

The result from the investigation of which side is pointing upwards in the PVD process is presented in Table 11. It is observed that there is a difference between the sides when testing with the M2 testing method, where the side of the insert pointing upwards in the PVD process flaked whereas only one insert tested on the other side showed signs of flaking for each. A typical view of flaking can be seen in Figure 33.

The results can be hard to follow for the inserts tested in M1, since the numbered BSup inserts was tested on the other side compared to the TSup inserts. However, the results shows that only two inserts showed signs of flaking for TSup and BSup tested in M1, all on the top side regardless if it was pointing upwards or not in the PVD process.

**Table 11: Results from blasting tests on inserts with varying sides pointing upwards in the PVD process. Red area indicates that all chip breakers flaked, yellow area indicates that two or less flaked and white area mean that the insert did not show flaking.**

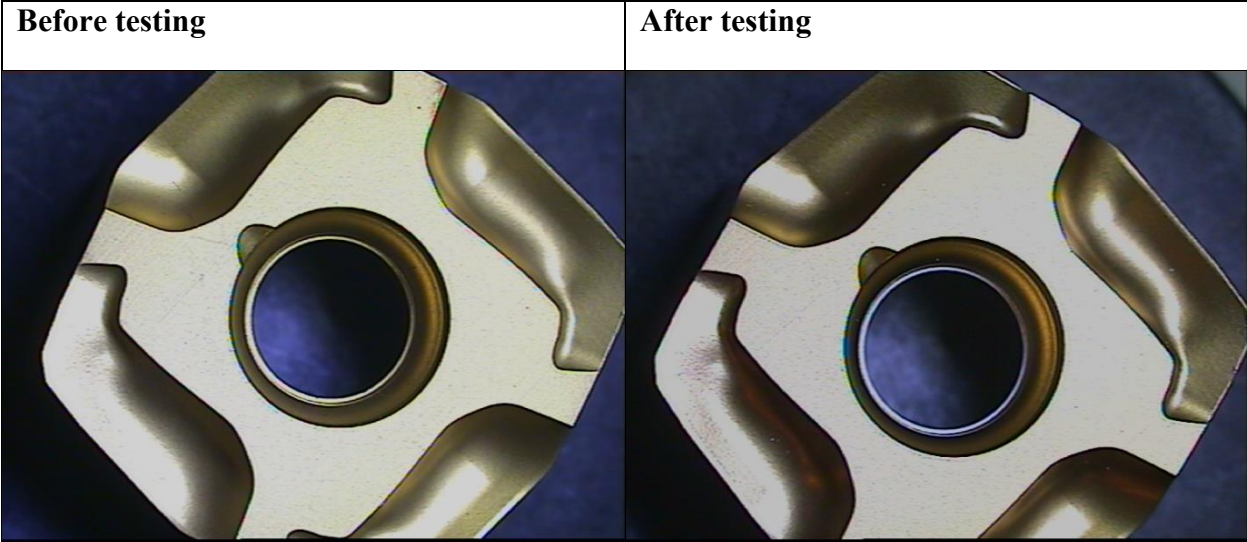
<u>Inserts:</u>	<u>TSup</u>	<u>BSup</u>	<u>TSup</u>	<u>BSup</u>
<u>Tested in</u>	<u>M1</u>	<u>M1</u>	<u>M2</u>	<u>M2</u>
Top side	1	3	11	11
Top side	2	4	12	12
Top side	7	8	17	17
Bottom side	3	1	13	13
Bottom side	4	2	14	14
Bottom side	8	7	18	18



**Figure 33: Showing flaking on the side pointing upwards in PVD and tested in M2.**



In the test made where the inserts were placed in the PVD furnace attached to a magnetic tube, no sign of flaking was seen on any of the inserts. A comparison of an insert before and after testing from the magnetic tube coating can be seen in Figure 34.



**Figure 34: Comparison of insert 1 coated attached to a magnetic tube before and after testing in M2.**



## 6 Discussion

The SEM images of the inserts used in the test screening showed that different amounts of wear occurred when using different blasting media. Figure 22 - Figure 24 that show the surfaces in 5000 times magnification indicate that larger blasting particles cause bigger parts of the surface to be separated, making the surface appear smoother. Wear of the coating could also be seen when particles harder than the coating were used, even when these were small. When using small particles that were softer than the coating, the surface that did not flake looked almost unaffected.

The actual volume removed by one single particle is very difficult to decide from the images since numerous particles have been impacting the surface during treatment. The images suggest that the dominating wear mechanism in wet blasting using softer particles than the coating is erosive wear. Support for this can be found in the lack of grinding like wear marks or striped structure, which should have been present if two-body or three-body abrasive wear had been dominant [4].

The results from the main tests, as well as from the test screening, indicate that blasting is a promising method of analyzing the adhesion of PVD coatings on cutting tools. Especially the results from the test screening suggest this, since a clear difference between a bad and better side of the inserts was observed. The main tests gave a more refined picture of the methods tested. It was found that when blasting with small alumina particles, M2, not only the difference between bad and good pre-treatment and etching could be found, but also the difference between different amounts of etching and pre-treatment blasting.

The importance of pre-treatment blasting as surface cleaning method can be observed in Figure 25, where inserts with different amounts of pre-treatment is presented before testing. Inserts pre-treated with P1 received a very uneven and dirty coating surface as a result of no cleaning and pre-treatment before the PVD process. These inserts later flaked in all tests, making it obvious that some amount of surface cleaning is needed before coating. A micro-blasting have also showed to improve the hardness, increasing toughness and operating life as well as producing a better surface finish [15]. This can somewhat be confirmed by the blasting tests where the inserts pre-treated with no micro-blasting flaked instantly. Why the methods chosen could not find differences between the other different amounts of pre-treatments is not clear, but this could be because the incoming quality differs, even if this could not be categorized in visual inspection before testing. Individual differences in surface oxides and or different amount of surface cobalt etc. is hard to see in visual inspection, but could perhaps explain why the differences could not be found by the chosen method. The influence of the incoming quality can also be seen when comparing the results for the etching test where the 340 inserts behave a bit differently from the process control inserts, both ordinary furnace controls and the gear milling inserts.

When comparing different amounts of etching in the PVD process, a difference was observed between the normal etching, lower etching bias, and no etching. The coating of the insert produced with no etching flaked even before testing, and after testing in M1 or M2 not much was left of the coating. Different results was seen, depending on which test method, M1 or M2, that was used. The inserts produced with lower etching bias did not flake on the bottom

side when testing with M1 whereas it flaked when using M2. This could be explained by the lower pressure used for M1 compared to the pressure used for M2. With this result, it would be safe to say that the testing method M2 seems to have slightly more merit than M1 as an adhesion testing method, since a stepwise flaking was showing when lowering the etching bias when testing with M2. The risk that this stepwise effect of different amount of etching only works for this particular geometry is confirmed by observing the furnace controls and threading inserts from the different processes. The flat geometry of these inserts could have resulted in a more evenly distributed coating and therefore fewer spots where flaking can propagate. With this considered, the importance of etching in the PVD process before coating can still be seen by observing these insert after testing, where both the furnace controls and threading inserts flaked substantially when coated without etching.

By comparing the different blasting testing methods, M1, M2, and M3, it is clear that the wear at the evaluated pressure was too harsh when using M3. All the inserts using the M3 method flaked on both sides, and in doing so essentially disqualifying M3 as a method for adhesion evaluation. However, both M1 and M2 still look promising since the P3 pre-treated inserts passed evaluation without flaking. The reason for why the inserts flaked using the M3 method, whereas it didn't for the M2 method could be explained by that it is two different machines used with differences in angle of impact, slurry concentration as well as different blasting medias, where particle size probably has the highest influence on the results [10]. Another thing that can be observed is that the flat part of the inserts received more substrate revelation for M3 method than for the other two. This could again be explained by the fact that different machines were used, but also that the angle of impact in M3 method, 90 degrees, could be close to the most beneficial for erosive wear by ceramic particles on a harder surface [4]. The most promising method for usage in the future is the M2 method, since it resulted in a clear difference between the amounts of pre-treatment which is expected because of the different surface qualities. Evaluation using M1 method is also possible, since there was a difference in flaking between the P1 and P3 pre-treated inserts.

The results from the lower etching bias testing in PVD process showed that most of the inserts flaked on both sides. The only inserts that could withstand some amount of blasting were the inserts processed with lower etching bias that were evaluated using M1. The same pressure and time in the blasting tests was used to be able to compare the results with the different pre-treatment testing. The results indicate that the adhesion between the coating and substrate suffered more from lowering the etching bias in the PVD process than from changing the pre-treatment processing. The coating quality from low etching bias tests were also worse before testing compared to the coatings in pre-treatment testing, suffering from flaking, especially on the side facing up in the PVD process. The reason for this could be that the etching functions as a last cleaning step in the process before coating. Furthermore, when using less or no etching it is possible that some impurities were still on the inserts, resulting in bad adhesion. Oxides will also be removed by the etching since it operates in vacuum, which makes it possible that some oxides were not removed when using lower or no etching bias. This is confirmed by earlier studies where Ar and H<sub>2</sub> plasma etching has improved the adhesion of PVD films [16].

When the importance of which side facing upwards in the PVD process was investigated, the results did not show any consistency between the testing methods, M1 and M2. Results from

testing using the M1 method indicated that the top side of the inserts flaked, regardless if it was up in the PVD process or not. This was not the case when testing using the M2 method. Instead, the results showed that there is an importance of which side is up, because that side flaked during testing. Another interesting thing from this investigation is that the inserts that were coated attached to a magnetic tube did not flake at all, regardless of which side that was coated. This makes the results even more interesting since it shows that the coating quality depends on whether the side points towards the targets or not. Further investigations would have to be made using this geometry to investigate what the problem is with the PVD process, because there is a difference in quality depending on how the inserts are coated. One reason for the varying quality of the side pointing towards the targets in PVD could be that dirt from the targets gets stuck in the chip breaker of this geometry which the etching could not get rid of.

## **7 Conclusions**

- Blasting shows promising results as a quality evaluation method of PVD coatings.
- The importance of etching in the PVD process is proved by blasting testing with testing methods, M1 and M2.
- Testing methods M1 and M2 shows most promising results for future investigations.

## **8 Future work**

There are some things that can and should be done in order to implement blasting as a testing method for PVD coatings. First of all, the batches where a failure is reported should be tested using both the blasting method and the method used today in order to compare the validity of the blasting test. The result using the blasting method should be at least as good as the method used in Västberga in order to replace it. This would, as mentioned in the purpose of the study, save a lot of money for the company since the threading insert can be removed from the PVD process.

There are also some tests that should be made in order to conclude where the limit of a good/bad adhesion of the coating is. In order to conclude this, several geometries should be tested in the same way with different adhesion quality by, e.g., using different amounts of pre-treatment. It would be interesting to investigate an insert with flat geometry, where the coating would be evenly distributed over the surface instead of the complicated geometry with chip breakers that were used in this thesis. Furthermore, a leakage test in the PVD process should be performed. Leakages in the process can happen from time to time, and a valve can be integrated in order to create a simulated controlled leakage of the amount that can occur in a real process. This would likely produce a poor coating that could help the investigation of where the limit between good and bad cohesion of coatings is in the blasting tests.

Tests should also be made in order to investigate why only one side appeared as bad in the test screening which also was showing in tendencies of the main tests. One thing is that some inserts can be turned before pre-treatment blasting and later sent to the same PVD process with later investigation by blasting testing.

## **9 Acknowledgments**

First I would like to thank my supervisor at Sandvik Coromant, Dr. Benny André for all the great support during this thesis. Also special thanks to Dr. Sam Ogden and Dr. Michael Bromark for all the help with analysis and all the great input with the report.

I would also like to thank all the operators and technicians in the pre-treatment and PVD departments for all the cooperation and help with the testing.

Thanks to my supervisor at KTH Royal Institute of Technology, Ass. Prof. Peter Hedström and also my examiner Anders Eliasson.

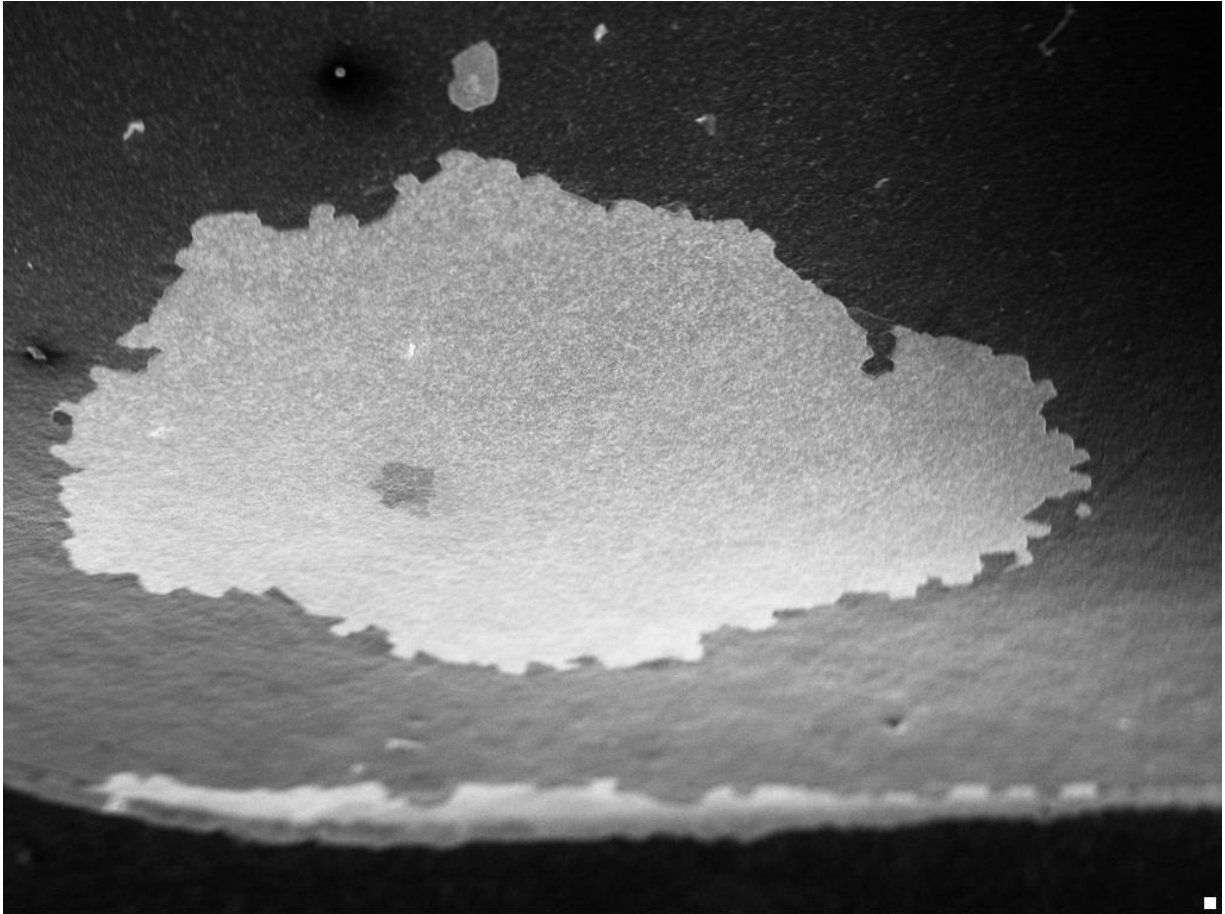
And last but not least thanks to all the personnel at GHTF in Gimo; Paul, Camilla, Arnaud, Johan, Erik, Doron and Simon for their hospitality, valuable inputs and pleasant talks.

## 10 References

- [1] "About Us: Sandvik Coromant," 9 March 2016. [Online]. Available: [www.sandvik.coromant.se](http://www.sandvik.coromant.se).
- [2] K. Choy, "Chemical vapour deposition of coatings," *Progress in Materials Science*, no. 48, pp. 57-170, 2003.
- [3] S. Jacobson, Tekniska Ytor, Uppsala, 2011.
- [4] S. Jacobson and S. Hogmark, Tribologi, Uppsala, 2011.
- [5] J. Fulemova and Z. Janda, "Influence of the cutting edge radius and the cutting edge preparation on tool life and cutting forces at inserts with wiper geometry," *Procedia Engineering*, no. 69, pp. 565-573, 2014.
- [6] H. Ollendorf and D. Schneider, "A comparative study of adhesion test methods for hard coatings," *Surface and Coatings Technology*, no. 113, pp. 86-102, 1999.
- [7] K.-D. Bouzakis, E. Bouzakis, G. Skordaris, S. Makrimalakis, A. Tsouknidas, G. Katirtzoglou and S. Gerardis, "Effect of PVD films wet micro-blasting by various Al<sub>2</sub>O<sub>3</sub> grain sizes on the wear behaviour of coated tools," *Surface & Coatings Technology*, no. 205, pp. 128-132, 2011.
- [8] K.-D. Bouzakis, F. Klocke, G. Skordaris, E. Bouzakis, S. Gerardis, G. Katirtzoglou and S. Makrimalakis, "Influence of dry micro-blasting grain quality on wear behaviour of TiAlN coated tools," *Wear*, no. 271, pp. 783-791, 2011.
- [9] K.-D. Bouzakis, G. Skordaris, E. Bouzakis, A. Tsouknidas, S. Makrimalakis, S. Gerardis and G. Katirtzoglou, "Optimization of wet micro-blasting on PVD films with various grain materials for improving the coated tools' cutting performance," *CIRP Annals - Manufacturing Technology*, no. 60, pp. 587-590, 2011.
- [10] J. Fröberg, Interviewee, *Production engineer*. [Interview]. 18 05 2016.
- [11] Y. Leng, "Light Microscopy," in *Materials Characterization*, Asia, John Wiley & Sons, 2008, pp. 1-44.
- [12] S. Hogmark, S. Jacobson and Å. Kassman-Rudolphi, Svepelektronmikroskopi, Uppsala: Ångströmslaboratoriet, 2010.
- [13] D. D. Kopeliovich, "Materials Engineering - Metallographic examination - Scanning Electron Microscope," [Online]. Available: [www.substech.com](http://www.substech.com). [Accessed 08 08 2016].
- [14] P. Eriksson, "TM GHT60826: Optimering av mätparametrar i PER," Internal report, 2011.
- [15] D. Kennedy, J. Vahey and D. Hanney, "Micro shot blasting of machine tools for improving surface finish and reducing cutting forces in manufacturing," *Materials and Design*, no. 26, pp. 203-208, 2005.
- [16] H. C. Barshilia, A. Ananth, J. Khan and G. Srinivas, "Ar + H<sub>2</sub> plasma etching for improved adhesion of PVD coating on steel substrates," *Vacuum*, vol. 86, pp. 1165-1173, 2002.

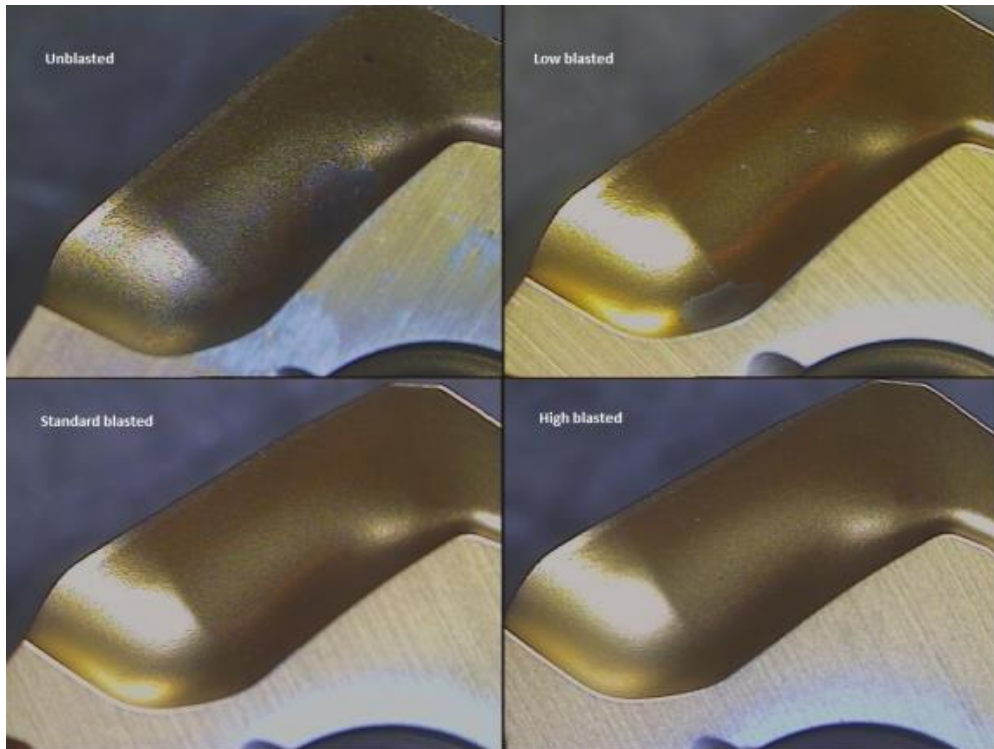
## Appendix

### Appendix 1

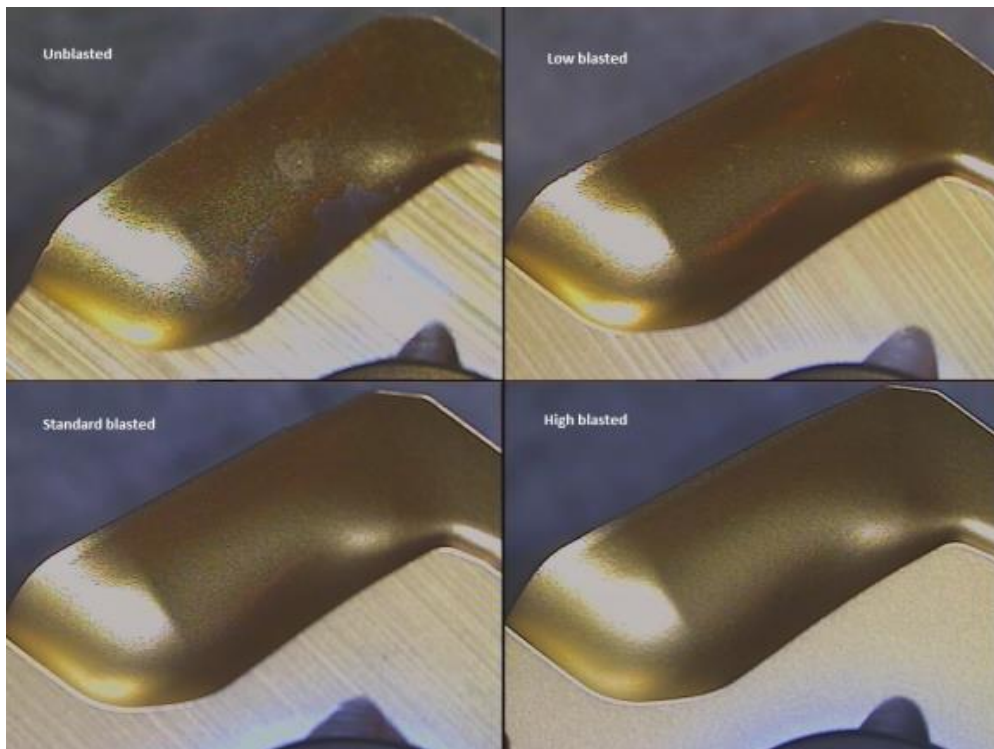


**Figure 35: SEM image of a flake on an insert tested using M1 method.**

## Appendix 2

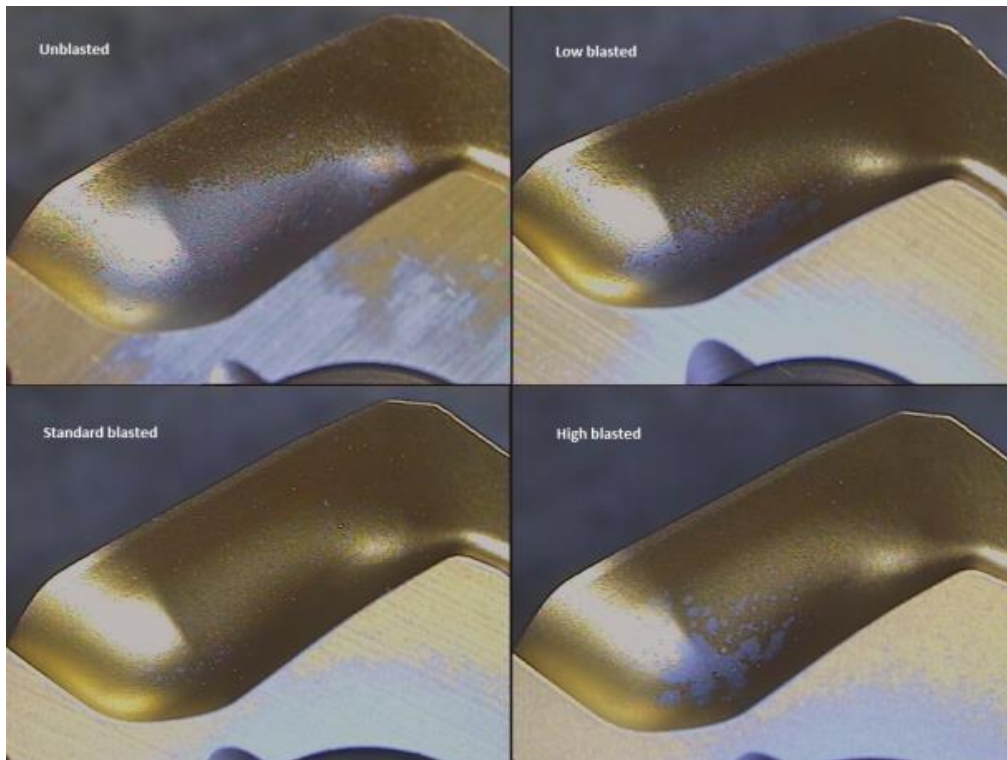


**Figure 36: Comparison of the top side of the inserts tested using M1. The picture is showing insert number 2 with different pre-treatment.**

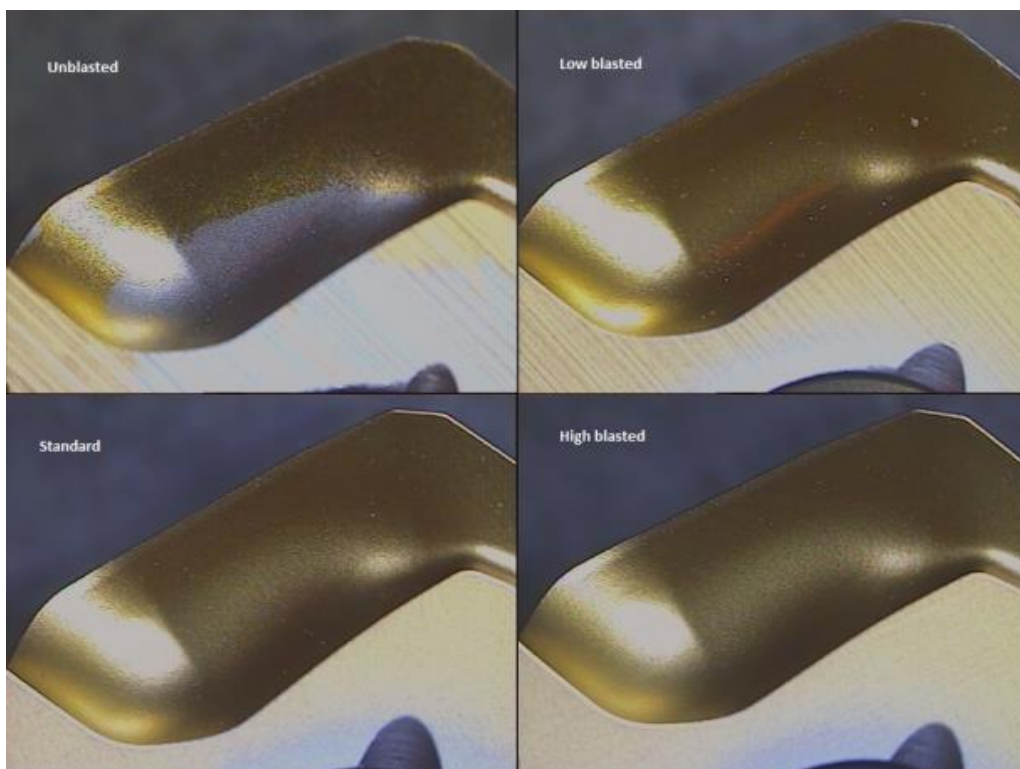


**Figure 37: Comparison of the bottom side of the inserts tested using M1. The picture is showing insert number 4 with different pre-treatment.**

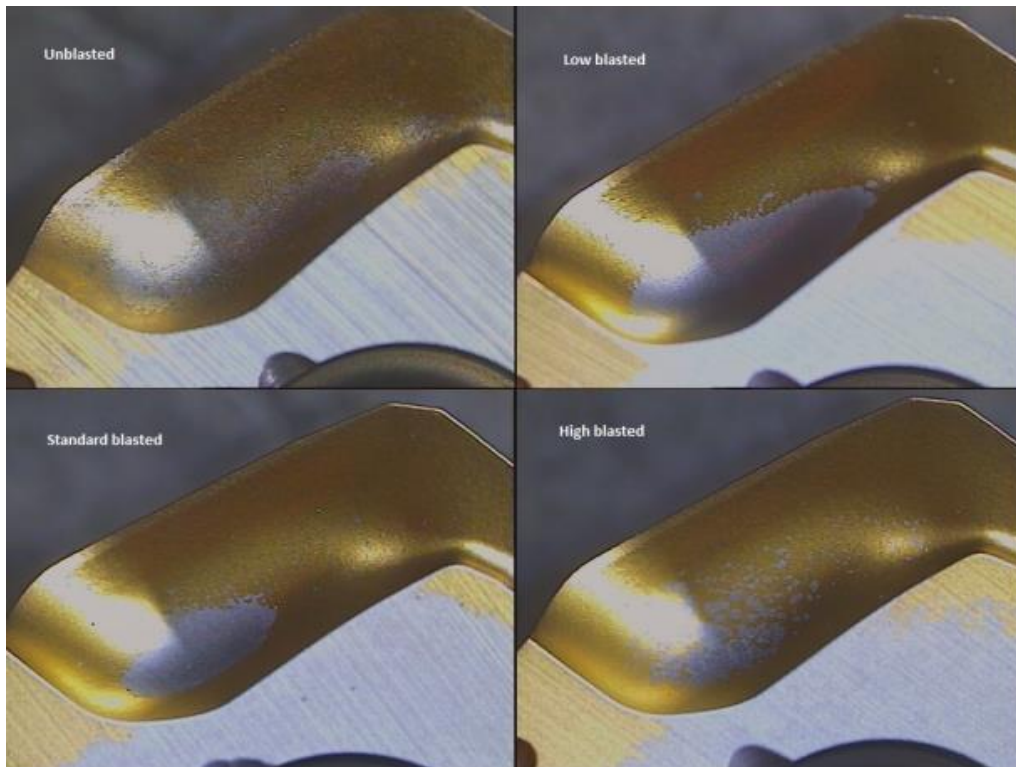




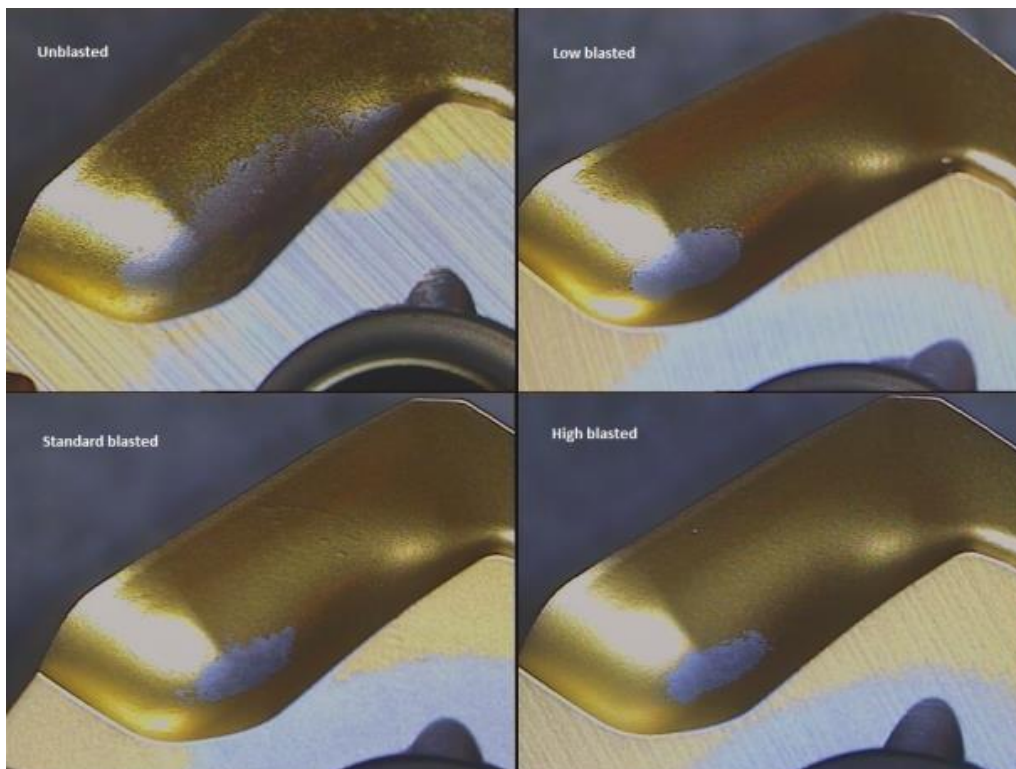
**Figure 38: Comparison of the top side of the inserts tested using M2 method. The picture is showing insert number 12 with different pre-treatment.**



**Figure 39: Comparison of the bottom side of the inserts tested using M2. The picture is showing insert number 14 with different pre-treatment.**



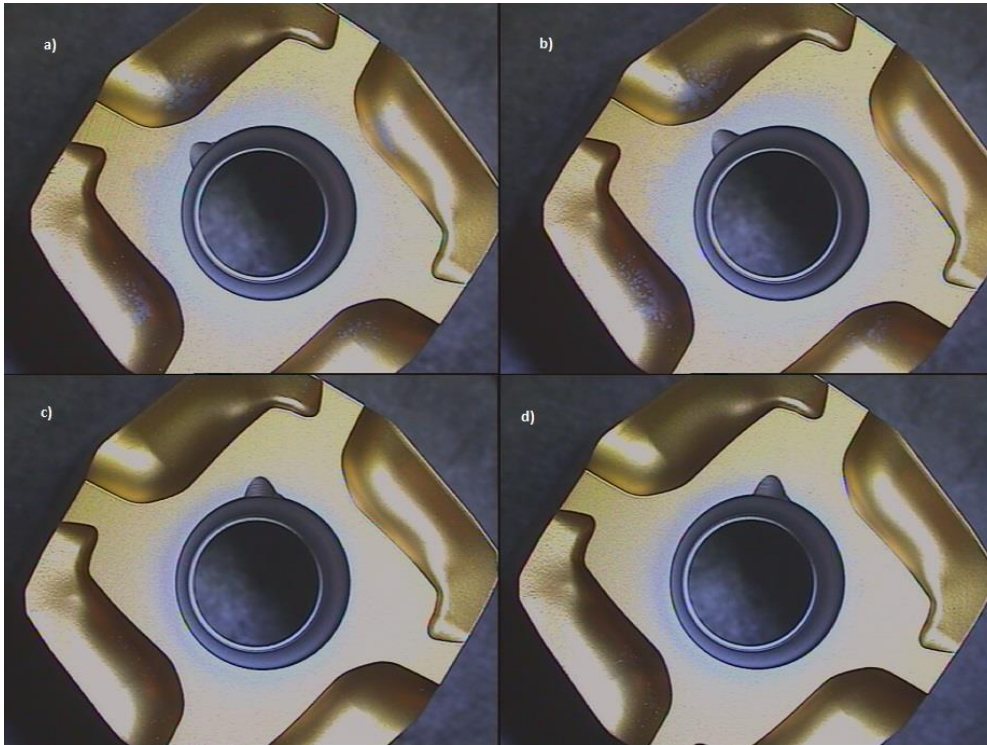
**Figure 40: Comparison of the top side of the inserts tested using M3 method. The picture is showing insert number 22 with different pre-treatment.**



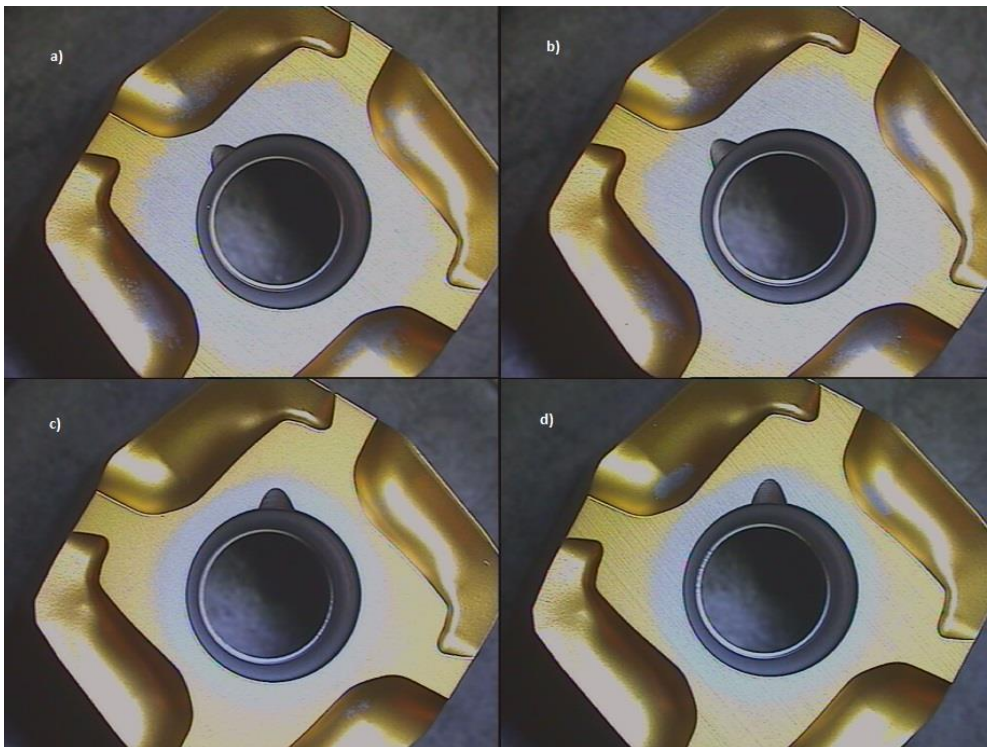
**Figure 41: Comparison of the bottom side of the inserts tested using M3 method. The picture is showing insert number 24 with different pre-treatment.**



### Appendix 3



**Figure 42: Insert tested using M2 and pre-treated with P4. Difference in flaking between the top and bottom side, where a) and b) shows the top side whereas c) and d) show the bottom side.**



**Figure 43: Inserts tested using M3 and pre-treated with P4. Difference in flaking between the top and bottom side, where a) and b) shows the top side whereas c) and d) show the bottom side.**

