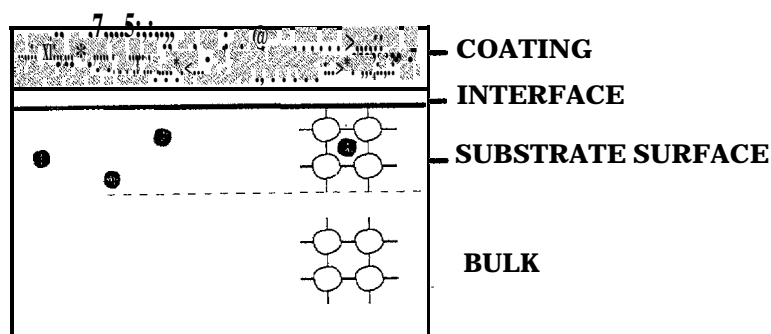


SYNTHESIS REPORT

FOR PUBLICATION

Surface generation, surface pretreatment and surface
characterization of tool materials in relation to PVD coating :
A new and systematic approach

(Project BE-4003, Contract BREU-0523, starting date: 01.02.92)



Project coordinator: WTCM-SURFACE TREATMENT B

Partners:

RWTH AACHEN (WZL)	D
THYSSEN EDELSTAHL WERKE AG (TEW)	D
PHILIPS PMF-CMTI (PMF)	NL
LIMBURGS UNIVERSITAIR CENTRUM (LUC)	B
CRIF/WTCM-MATERIALS (CRIF)	B
BELGIAN TOOLING COMPANY (BTC)	B
HELSINKI UNIVERSITY OF TECHNOLOGY (HUT)	SF

Reference Period: 0.1.02.1992 to 31.07.1995

Duration: 36+ 6 months



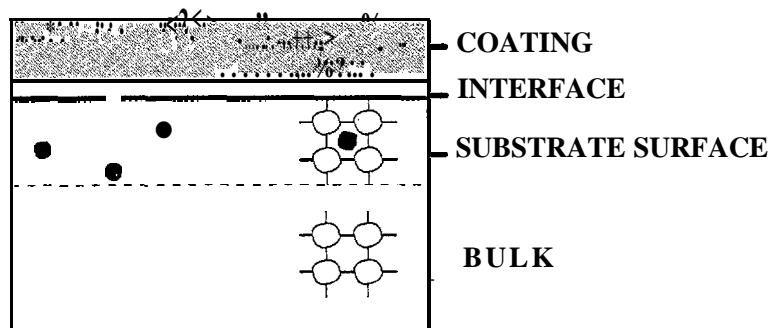
PROJECT FUNDED BY THE EUROPEAN
COMMISSION UNDER THE
BRITE-EURAM PROGRAMME

DATE: 15.01.1996

PRE-TREATMENT IN RELATION TO PVD: SOME GUIDELINES

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A new and systematic approach

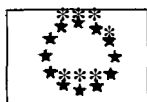
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Critical issues in generating and pretreating tool surfaces in relation to PVD coating : some guidelines

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1 Objectives

Based on a Brite-Euram research project that dealt with the study of tool surface pretreatment prior to Physical Vapour Deposition (PVD) coating, some guidelines could be deduced to make it possible for tool makers and tool users to deliver tools with an optimal surface state in respect to PVD and to reach a more efficient use of PVD coated tools.

A future objective is the completion of these guidelines, based on the experience of European tool makers, job-coaters and end users, which must finally lead to an European standard.

2 Introduction

Physical vapour deposition (PVD) is a technique often used for about ten years to deposit TiN coatings on tools. The purpose of this coating is to enhance the efficiency of the process in which the tool is used and to improve the quality of the final product. An advantage of the PVD process, in comparison with CVD (Chemical vapour deposition) is the low process temperature, so that various types of hardened steel can be coated without changes in properties.

A disadvantage of PVD techniques is that the initial surface characteristics of the substrate (a very clean surface, burr-free cutting edges, . . .) have a large influence on the nucleation of TiN particles and on the initial stage of layer growth. This affects to a large extent the properties of the coating and its performance as a functional coating - e.g. the resistance against mechanical, thermal and chemical loads applied on coated tools or components - and its reliability.

Surface characteristics prior to coating, such as chemical contamination, residual stresses, texture, roughness and topography are partly the result of surface manufacturing treatments, e.g. grinding, polishing, spark erosion and shot-peening. Each treatment can be characterized by several parameters such as manufacturing agents (grinding and polishing agents, electrode materials, . . .) and energy input.

These surface characteristics prior to coating influence the adhesion, microstructure, residual stresses and texture of the coating. Failure of a coating is usually due to a non-optimal surface finish of the tool. To obtain the desired tailored tool surface properties, standard surface pretreatments like decreasing or sputter cleaning are not sufficient. They must be partly generated already during the manufacturing of the tool surfaces.

In the mean time PVD technology has evolved towards a further upscaling and implementation of the existing coatings TiN, (Ti,Al)N, Ti(C,N), CrN, . . . It seems that each coating can benefit from some particular properties to be used in certain applications. From the substrate side some efforts have been made by other research groups to study the influence of machining operations on the coating performance. Cleaning procedures will be replaced more and more by plasma cleaning alternatives as an environmental friendly solution.

Various surface generation techniques, tool materials and coating techniques were evaluated in this project and compared with today's state of the art. A lot of characterization techniques were helpful to understand the tool surface characteristics and their influence on the performance of the tools.

3 Technical description

Flat sample procedure:

In the first part of the project flat samples were made with 34 different surface variants as a combination of 5 manufacturing processes:

- . grinding
- sink-electro-discharge machining (S-EDM)
- . wire-electro-discharge machining (W-EDM)
- . mechanical polishing
- . electrochemical machining (ECM)

and 6 kind of materials:

- . high speed steel (1 .3343)
- . cold work tool steel (1.2379)
- . cold work tool steel (1.2083)
- . hot work tool steel (1 .2343)
- . carbides
- . cermets

The chemical composition of the investigated tool steels, carbides and cermets are given in [table 3.1](#). The tool steels are in a hardened state with HRC hardness values :65 (1.3343),62 (1.2379), 53 (1 .2083), 41 (1.2343). The annealing temperature of mould steel C is 500°C so coating temperature must be lower to avoid softening of the tool. The cold work tool steel has been tempered two times.

The 34 processing variants one can find in [table 3.2](#). More details about the different surface generation processes are given in [table 3.3](#).

Material Standard NR. *ISO*DIN	Chemical composition (weight %)
A = high speed steel 1.3343 * HS 6-5-2 * S 6-5-2	0.9 C -0.31 Si -0.38 Mn - 0.019P - 0.0002S -3.87 Cr -4.8 Mo - 1.75 V-6.15 W
B = cold work steel 1.2379 “160 CrMoV 12 * X 155 CrMoV 121	1.6 C -0.38 Si - 0.45 Mn - 0.02P- 0.0006 S -11.2 Cr -0.67 Mo - 0.99 V -0.12 W
C = mould steel 1.2083 *40 Cr13 *40 Cr13	0.44 C -0,28 Si - 0.81 Mn - 0.017P -<0.003 S - 12.87 Cr -0.05 Mo -0.05 V -0.05 W
D = hot work tool steel 1,2343 * 35 CrMoV 5 * X 38 CrMoV 51	0.39 C- 1 Si - 0.31Mn - 0.021 P-0.002S-4.94Cr- 1.25 Mo -0.4 V -0.02 W
E = carbides TTR	78.5 WC - IO (Ti,Ta,Nb)C -11.5 Co
F = cermets TTI 25	26 TiC-27 TiN-20 WC- 8.6 TaC - 11 NbC - 8.2 Ni -8.6 co

Table 3.1: Designation (Standard Nr. * I SO * DIN) and chemical composition of materials used.

Substrate and processing variants						
Process						
substrate material	A 1.3343 S 6-5-2	31.2379 { 155 CrMoV 21	C 1.2083 X 42 Cr 13	D 1.2343 X 38 CrMoV 5 1	⌈ Carbide ⌋ TR	⌈ Cermet ⌋ Ti 25
I Grinding	1 corundum ($v_c = 35$ m/sec) 2 CBN ($v_c = 100$ m/sec) 3 CBN ($v_c = 60$ m/sec)	. corundum $v_c = 35$ m/sec) ! CBN $v_c = 100$ m/sec) ‡ CBN $v_c = 40$ m/sec)			diamond $v_c = 25$ m/sec) ‡ diamond $v_c = 35$ m/sec)	. diamond $v_c = 15$ m/sec) ! diamond $v_c = 25$ m/sec) ‡ diamond $v_c = 35$ m/sec)
II S-EDM			1 finishing 1 tool: Cu(+) hydrocarbon 2 finishing 2 tool: graphite(+) deionized water 3 polish. 1 tool: Cu(+) hydrocarbon 4 polish. 2 tool: graphite(+) deionized water	1 finishing 1 tool: Cu(+) hydrocarbon 2 finishing 2 tool: graphite(+) deionized water 3 polish. 1 tool: Cu(+) hydrocarbon 4 polish. 2 tool: graphite(+) deionized water		
III W-EDM	1 polishing 1 1 maincut, 1 trimcut 2 polishing 2 1 maincut, 5 trimcuts (SF)	! polishing 1 ! maincut, 1 rimcut ! polishing 2 ! maincut, 3 rimcuts ‡ polishing 3 ! maincut, 5 rimcuts (SF)			polish. 1 maincut, 3 rim cuts ! polish, 2 maincut, 6 rimcuts	! polish. 1 ! maincut, 3 rimcuts ! polish. 2 ! maincut, 6 rimcuts
IV Polishing			1 best S-EDM finish. 2 best S-EDM polish.			
V ECM			1 finishing 1 2 finishing 2	1 finishing 1 2 finishing 2		! finishing 1 ‡ finishing 2

Table 3.2: 34 surface variants generated on flat samples and coated during the project.

Grinding of A and B was carried out with corundum and with CBN (cubic boron nitride) grinding wheels. It was aimed to obtain $R_z < 2\mu\text{m}$, no burrs, and no or a negligible white layer. Carbides (E) and cermets (F) were ground with diamond. The grinding wheels were dressed before with diamond (steels) and with Al_2O_3 and diamond for the carbides and cermets.

Sink-electro-discharge-machining (S-EDM) of tool steels C and D was carried out using a Cu tool (+ hydrocarbon dielectric) or a graphite tool (+ water dielectric). Both the polishing and finishing mode were used.

Wire EDM (W-EDM) uses a CuZn37 wire (+ deionized water). Both EDM techniques are followed by shot-peening.

The mechanical polishing is performed as finishing step on S-EDM machined tool steels C and D.

Electro Chemical Machining (ECM) with CuZn37 tool (+ NaNO₃ electrolyte) is done as a finishing step after W-EDM.

Technique	Process parameters
grinding (fig. 3.1a)	<ul style="list-style-type: none"> •cutting speed ($v_J \approx 35$ m/s (Al₂O₃) or higher (CBN, up to 100 m/s) (steels) = 15 to 35 m/s (diamond) (carbides, cermets) -surface-peripheral- longitudinal-grinding (fig. 2a) “cutting fluid = emulsion 4 % “grinding wheels = <ul style="list-style-type: none"> •EKK 46/60 -611- V20 (Al₂O₃) •B64 VSS 281/GH 1 SC V360 (CBN) •D64 V-plus2813 6H 1 SC C 100 (ceramic bound diamond) •cutting depth (a_e) = <ul style="list-style-type: none"> •5 μm (steels) •2 μm (carbides, cermets)
S-EDM (fig. 3.1b)	<ul style="list-style-type: none"> •finishing : <ul style="list-style-type: none"> “discharge power 11 A •pulse duration 150 ms “quiescent period 10 ms “polishing <ul style="list-style-type: none"> -discharge power 5 A •pulse duration 25 ms “quiescent period 7 ms
W-EDM (fig. 3.1c)	<ul style="list-style-type: none"> •reducing discharge current and pulse duration from main cut to trim cuts = <ul style="list-style-type: none"> •44 A, 2,6 μs (main cut) •0.7 A, 0.4 μs (5th trim cut) •wire speed = 90 mm/s gap width = 13 mm (main cut), 17 mm (trim cut)
ECM (fig. 3.1d)	voltage 24 V, current 0.5 A, pulse duration 1 ms, gap width 70 μm, quiescent period 9 ms
shot-peening	7 bar (stat), blasting angle 45° at a distance 8 cm during 30 s (glass balls of 100 to 200 pm)

Table 3.3: Process details of various surface generation techniques

After surface generation the samples were protected by an anti-corrosion lacquer (Molykote), which could easily be removed afterwards. Two cleaning methods were applied before PVD deposition: for WTCM solvent and vapour decreasing and alkaline cleaning (70°C) (both ultrasonically), rinsing in deionized water and drying in hot air, and for TEW acetone cleaning and alkaline cleaning (70°C) (both ultrasonically), rinsing in deionized water and applying a tensid treatment.

Two PVD coating techniques were applied

- ♦ electron beam ion plating (EBIP, WTCM, W) (± 500°C),
- ♦ random arc ion plating (RAIP, TEW, T) (± 450°C).

The thickness of the coating is about 3 pm.

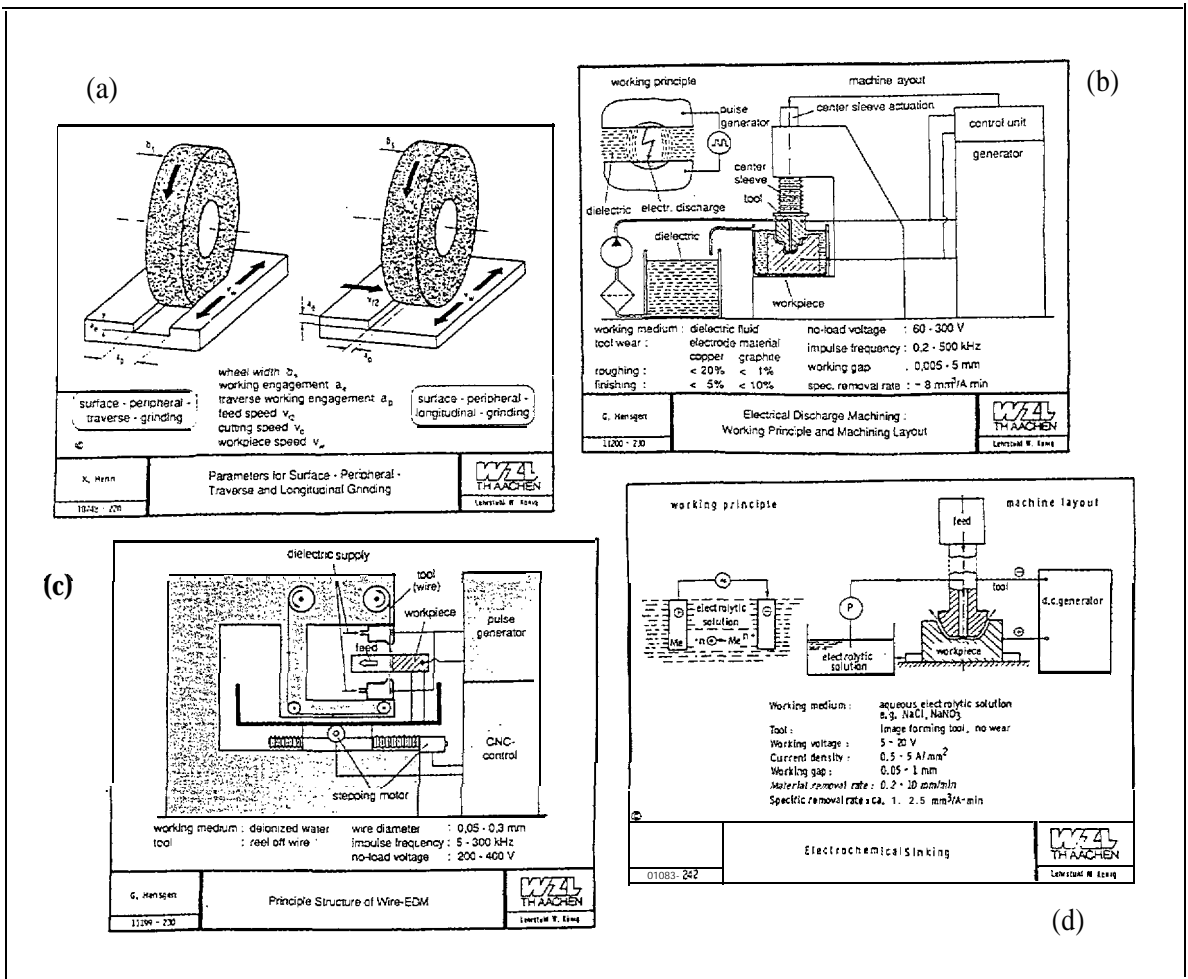


Figure 3.1: Different manufacturing techniques used in the project; (a) grinding, (b) S-EDM, (c) W-EDM, (d) ECM.

The electron beam ion plating starts with heating and Ar ion etching and the deposition of a Ti intermediate layer of 0.2 μm (bias voltage - 150 V). The random arc ion plating starts with infrared heating, H₂ and Ti ion etching and the deposition of a Ti interlayer (0.05 μm) (bias voltage -200 V).

All samples were characterized by roughness measurement, Knoop hardness measurement, metallographic cross-section examination, scanning electron microscopy, X-ray diffraction, and some particular samples by SIMS, scanning tunneling microscopy and TEM. The coated samples were also characterized for their adhesion by scratch test and by Rockwell C indentation test and cross-section.

A link was made between the surface generation and the coating quality. Based on this the following quality criteria was established:

Based on the scratch test those samples with a Lc6 (spalling failure) value were classified as samples with insufficient adhesion properties and therefore further investigated in task 6. Those without a Lc6 value are allowed to be used for tools (task 5), which doesn't mean that the coating adhesion is guaranteed during performance.

The skipped surface variants were tried to be optimised so that they were still suitable for PVD coating. Especially the cleaning of carbides and cermets and the S-EDM, W-EDM and ECM processes had to be optimised (a) by adapting the cleaning method and (b) by intermediate steps before PVD coating.

Coating deposition on real tools:

In the second part of the project those surface variants which met the quality criteria were used for real tools (turning, milling and deep drawing). The influence of the tool surface generation on the quality of coated tools was investigated.

Testing of coated tools:

As cutting operations, milling and turning were applied.

High speed steel (A) and cermet (F) inserts were ground with Al_2O_3 , CBN and with diamond. The rake face (fig. 3.2.a) is ground according to the flat samples procedure. The flank face (fig. 3.2.b) is ground now with the so called longitudinal side grinding process (fig. 3.3, position 1) with grinding occurring from flank to the rear of the insert, so avoiding burr formation at the cutting edge. While the flank grinding marks are perpendicular to the cutting edge, they become parallel to the cutting edge for the cermets (fig. 3.3, position 2). This is done to avoid chipping of the cutting edge. Roughness measurements and metallographic investigations showed no significant differences compared to what could be seen on the flat sample surfaces. The only upscaling feature (from flat samples to real tools) was a thinner TiN coating at the flank face in the electron beam ion plating process. This could be solved by an extra sample rotation step in the deposition chamber.

Turning and milling were carried out with these inserts on workpiece material 42 CrMo4V (1.7225). This material showed a harder surface zone (300 HB) compared to the core (260 HB). In order to avoid tool wear due to changing hardness of the workplaces the shafts were turned only in the surface zone. Cutting with high speed steel was done with a lubricant (3 % emulsion) and dry with the cermets.

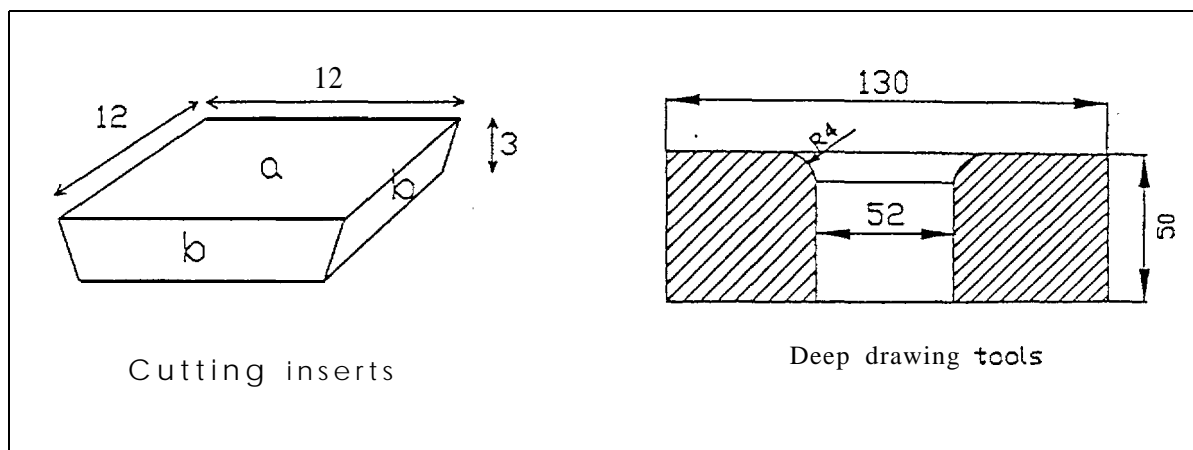


Figure 3.2: Schematic view of cutting inserts and deep drawing tool.

4 Guidelines for generation and preparation of tool surfaces for PVD coating

4.1 Grinding

- When grinding is applied in a proper way, i.e. working with a sharp grinding wheel (to avoid burrs, plastical deformation and oxidised surfaces),
- keeping the surface roughness lower than half the coating thickness ($R_z < 2\mu\text{m}$), no bad adhesion will occur.
- There are thin white layers formed during grinding, but they do not cause bad adhesion.

Grinding is known as a surface generation process which forms so called white layers at the tool surface. It is caused by the thermal and mechanical influence of the grinding particles working the tool surface. Very often Al_2O_3 or CBN (cubic boron nitride) grains are used in grinding wheels. CBN has a higher thermal conductivity and therefore creates a so called cool cut process for the tool surfaces to be ground. This gives rise to thinner white layers without retained austenite (lower grinding temperature).

Typical grinding surface characteristics (see [table 4.1](#)) found in this project are:

- white layer on ground steel exists of untempered martensite and retained austenite
- lower steel hardness results in lower grinding temperature, thus thinner white layer
- even when no white layer was visible with optical microscopy, a white layer was detected (XRD)
- grinding of carbides and cermets results in small plastical deformation of binder and pull out of carbides instead of white layer formation

GRINDING		
Al_2O_3 (steels A, B)	CBN (steels A, B)	diamond (carbides, cermets)
$R_z = 1 \mu\text{m}$	· higher due to higher grain size	$R_z = 1.5 \mu\text{m}$
· A) small white layer (-2pm) of retained austenite and untempered martensite · B) lower grinding temperature → no white layer visible with optical microscopy	· small white layer without retained austenite	· no white layer
· grinding marks	· no grinding marks	· plastical deformation of binder and pullout of carbides

Table 4.1: Surface characteristics of ground surfaces.

The resulting influence on the TiN coating adhesion was (figure 4. 1):

- good adhesion on all ground samples (which is in contrast with literature findings)
- CBN ground surfaces give rise to another TiN growth mode which must be related to the different outermost surface of CBN ground surfaces compared to Al_2O_3 ground surfaces

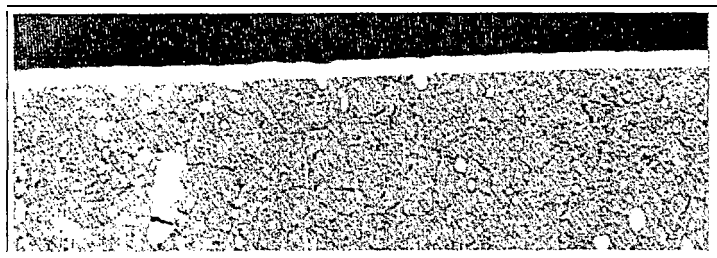


Figure 4.1: Cross-section of corundum ground 1.3343 with W coating

Surface roughness was about 1 and 1.5µm for grinding of steel with Al_2O_3 and carbides and cermets with diamond. The higher grain size of CBN wheels causes a higher surface roughness.

The so called white layers can clearly be seen on metallographic cross-sections after chemical etching. This means in fact that the substrate material becomes chemically activated and that a thin surface layer remains unetched, which resulted in the designation white layer. When grinding parameters are too severe also oxidation can occur which can result in a contamination of other tools in the PVD deposition chamber. This can happen e.g. with unsharp grinding wheels. These wheels cause also more a ploughing effect rather than a cutting effect. So contaminations can be included in the tool surface which can lead then to electrical discharges on the tool surface with a resulting serious damage if the necessary precautions are not taken by the job-coater. For ground surfaces the thin white layer consist of a crystalline structure. The latter can be defined as a transformation layer which results from austenitization at the higher grinding temperature and subsequent cooling. In the case of the high speed steel (A) the microstructure of the top layer (100nm) of this white layer (1,5 µm) showed a austenitic structure with small grains (1 0-25nm) coming from a martenisitic structure before grinding. The cold work tool steel is also a ledeburitic steel with a martensitic structure. After grinding with corundum a martensitic and austenitic structure is present.

Adhesion of PVD coatings on such ground surfaces is often found in literature to be insufficient. This is in contradiction with the findings in this project.

Tools made of cemented carbides and cermets are ground with diamond wheels. Due to the high grinding temperature cracks in the tool surface and dissolution of the binder material of the tool can result.

Burrs on fictional edges have to be avoided because during the first contact with the workpiece or counter tool the burr will break off and the coating will be removed on the edge. This damage can have an influence on the life time of the tool as will be described in paragraph 5.

4.2 Sink-Electro-Discharge-Machining (S-EDM)

- Due to the amorphous regions in the white layer of a S-EDM machined surface in general a bad coating adhesion is obtained.
- The roughness after S-EDM is also too high for the use of coatings.
- Polishing (or another finishing technique) afterwards is necessary.

Tool surfaces generated by EDM get a higher energy input which results in a thicker white layer, partially remolten. Especially sink EDM (S-EDM) is known as a technique resulting in high roughness values. State of the art is then polishing the tool surface. In general adhesion of PVD coatings on EDMed surfaces is very bad.

Typical S-EDM surface characteristics (see [table 4.2](#)):

- in all cases a high roughness is obtained ($R_z = 20-27\mu\text{m}$)
- forming of thick remolten layer (30-100 μm) with amorphous regions and retained austenite or Fe_3C
- cracked surfaces

Influence on TiN coating adhesion (see [figure 4.2](#)):

- bad adhesion of TiN coating on amorphous regions and bad overall characteristics of the final surface because of cracks and roughness



[Figure 4.2](#): Cross-section of S-EDM machined 1.2083 with TiN coating

The white layer formed during S-EDM is very thick (30-100 μm). In case a Cu tool with hydrocarbon is used, amorphous regions and Fe_3C (probably because of cracking of hydrocarbon) is formed. When a graphite tool and water is used, amorphous regions and retained austenite is formed.

A clear relationship is deduced between the amorphous zones and the bad adhesion of a TiN coating as will be described in paragraph 4.3.

The roughness of these surfaces is too high for a meaningful deposition of a coating for wear applications. The coating will be removed directly and preferentially at the roughness peaks.

S-EDM (+ shot-peening)	
Cu + hydrocarbon (steels C, D)	graph ite + water (steels C, D)
• $R_z = 20 \mu\text{m}$	• $R_z = 27 \mu\text{m}$
• thick white remolten layer (-30 μm) with formation of Fe_3C and amorphous regions	• thick white remolten layer (-100 pm) due to focused energy input (formation of retained austenite and amorphous regions)
• cracked surface layer	• no cracked surface layer
• shot-peening contamination	• shot-peening contamination
• Cu contamination	
Mechanical polishing	
mechanical polishing removed afterwards the white layer completely $R_z \sim 0.1 \mu\text{m}$	

Table 4.2: Surface characteristics of S-EDM machined surfaces

Because of the high roughness and bad coating adhesion one is obliged to finish the surface with another technique as grinding or polishing.

4.3 Wire-Electro-Discharge-machining (W-EDM)

- Better surface condition and lower roughness R_z is obtained in comparison to S-EDM. Again white layers with amorphous structure are formed.
- Therefor never deposit a coating on a W-EDM machined surface without verifying if there are no amorphous regions present at the surface.
- The amorphous regions have to be recrystallized first by means of annealing or have to be removed by means of mechanical polishing or high energy ion bombardment.
- The recently Superfinishing W-EDM is a suitable process giving a low R_z value and a good adhesion. A white layer is still present but much thinner and probably not in an amorphous state (not investigated yet).

Wire - EDM uses the same principle of material removal as for sink - EDM. In contrast to sink - EDM, the tool, which consists of an unwinding wire electrode, can not be mapped on the workpiece. Shapes are produced by guiding the moving wire along the workpiece. The energy input is lower than for S-EDM which results in a thinner white layer.

Typical surface characteristics are (see [table 4.3](#)):

- lower roughness than for S-EDM, about 3pm
- white remolten layer of retained austenite and amorphous regions
- wire-EDM (W-EDM) results in a thinner white layer than for S-EDM
- super finishing W-EDM is a suitable process giving a low R_z value and a good adhesion.

W-EDM (+ shot-peening)	
steels (A, B)	carbides, cermets
• $R_z = 3 \mu\text{m}$	• $R_z = 3 \mu\text{m}$
• white remolten layer of retained austenite and amorphous regions (energy input between grinding and S-EDM)	• only remolten layer
• shot-peening contamination	• shot-peening contamination
• 1 main cut, 1 or 3 trim cuts	• 1 main cut, 3 or 6 trim cuts (no influence)
Super Finishing W-EDM	
• $R_z = 1.5 \mu\text{m}$	
• no shot-peening	
• 1 main cut, 5 trim cuts	

Table 4.3: Surface characteristics of W-EDM and SF-W-EDM machined surfaces.

Influence on TiN coating adhesion:

- bad coating adhesion due to presence of amorphous regions (figure 4.3.1)
- amorphous regions have to be crystallised or removed

The adhesion was better as one can see in figure 4.3.1 for the W coated sample (b) because of the partial annealing of the white layer due to a higher coating process temperature.

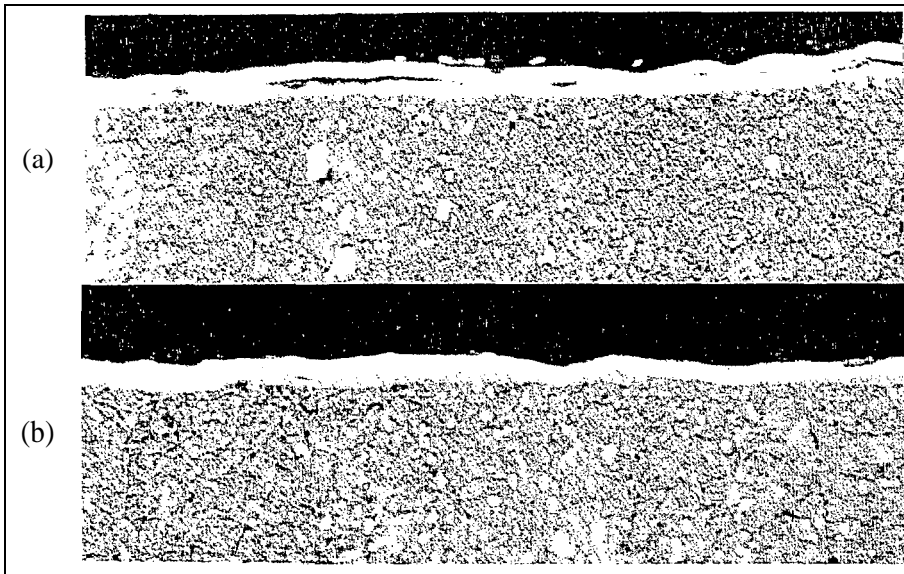


Figure 4.3.1: Cross-section of W-EDM machined 1.3343 with T (a) and W (b) coating.

Usually one main cut and six trim cuts are applied.

For W-EDM machined surfaces (of steel) the white layer is partially amorphous (amount depending on the kind of steel) alternated with regions containing small grains (10-50nm). A clear relationship is deduced between the amorphous zones and the bad adhesion of a TiN coating. The interface at the amorphous region is shown in figure 4.3.2 at a high magnification (22000x). The white zone is an amorphous zone (a). The TiN coating grown on this zone is not textured like a normal TiN coating and showed a bad adhesion. At the crystalline parts (b) of the specimen the coating adheres much better.



Figure 4.3.2: TEM bright field image of W-EDM machined A material (AlI): (a) amorphous area, (b) region containing small grains.

Adhesion tests like indentation test and scratch test showed that the adhesion was dependant on the region were performed. Indentation tests showed delamination of small particles over a large area, which is totally different from adhesion on ground surfaces. Due to the higher deposition temperature of the WTCM process the adhesion of WTCM coated samples was a little better than those coated by TST. The reason for this is the partial recrystallisation of the white layer.

Possible solutions to avoid this amorphous white layer are:

- annealing of the white layer
- removing the white layer
- high energy ion bombardment
- optimization of EDM

The amorphous regions in EDMed surfaces can be crystallised by an annealing step. This can be done during the coating process itself if the coating process temperature is high enough (e.g. electron beam ion plating) or by high energy Ti or etching (e.g. random arc ion plating). Also an annealing outside the PVD chamber is possible. An additional heat treatment is sometimes applied by tool makers to relax internal stresses. Disadvantage of this method are dimensional changes and possible hardness loss.

An additional manufacturing step to remove the white layer is in case of complex geometries sometimes impossible or very expensive.

From all possible solutions the most promising one is the Super-Finishing-W-EDM. Here only a very thin white layer with very low roughness is formed. A better adhesion is reached in this case, probably because of the fact the white layer is without an amorphous structure .

4.4 Mechanical polishing

- Polished surfaces can give adhesion problems if the surface is not degassed well (during heating phase of the deposition process) before coating deposition.
- The polishing technique determines the characteristics of the Beilby layer. Coating adhesion is dependant on the surface characteristics.

Mechanical polishing gives rise to the so called Beilby layer on top of the tool surface and includes e.g. the small polishing particles and residues of other polishing agents. During the micro cutting of the surfaces polishing residues are included in the surface. Although usually a very clean surface seems to be generated in literature a lot of PVD adhesion problems are reported on. Reason for this is that the residues are not removed during physiochemical cleaning and the degassing of these inclusions during the heating of the coating process. Especially polishing paste seems to cause this problem.

Typical surface characteristics:

- formation of Beilby Layer
- very smooth surface

Influence on TiN coating adhesion (see figure 4.4):

- sometimes adhesion problems

Mechanical polishing was used here as finishing technique after S-EDM machining (see table 4.2).

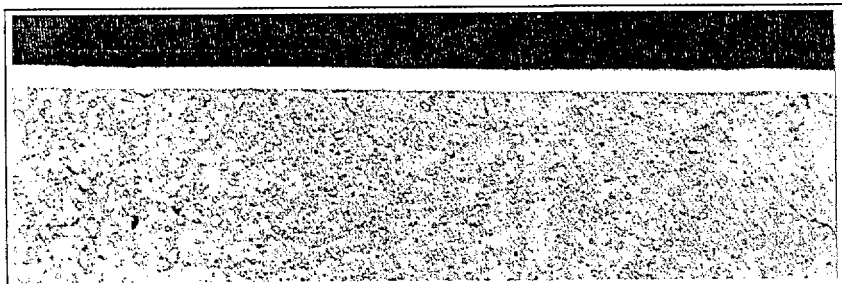


Figure 4.4: Cross-section of polished 1.2083 with W coating.

The adhesion problem of polished samples can be solved using the electron beam ion plating technique by heating them during a prolonged time (up to 1 h) and by ion etching them during at least the same time as the heating time. This is related to the degassing during heating and the following redeposition needs therefore a larger ion etching step.

For deposition techniques using high energy etching no bad adhesion occurs.

4.5 Electrochemical machining (ECM)

- Not suitable for cermets because of dissolving of Co, which also results in a high roughness.
- When applied as finishing step on steel after EDM, be sure the amorphous white layer is removed totally.

ECM is a rather new technique which initiates no white layer and no stresses in the tool surface. It is therefore investigated as a promising finishing step after EDM and is based on an electrochemical dissolution of the tool surface material.

Typical surface characteristics (table 4.5):

- no white layer formation for steels (electrochemical), but sometimes white layer of preceding W-EDM was present
- it seems not to be a suitable finishing step for cermets (dissolving of Co and high R_z values).

ECM (cermets) after W-EDM (+ shot-peening)
$R_z = 5 \text{ à } 11 \text{ }\mu\text{m}$ Co dissolution due to ECM shot-peening contamination

Table 4.5: Surface characteristics of surfaces when ECM machined after W-EDM machining

Influence on TiN coating adhesion (figure 4.5):

- the behaviour of ECMed steel surfaces depends on the remaining white layer as a result of the preceding W-EDM process.
- the dissolving of Co degrades the support action of the surface for the deposited coating

On steel only convincing results were obtained when ECM machining was applied after grinding. When applied after EDM the white layer of the preceding EDM was not removed and caused bad adhesion.

When cermets were ECM machined, roughness values of 5 to 11 μm were obtained.

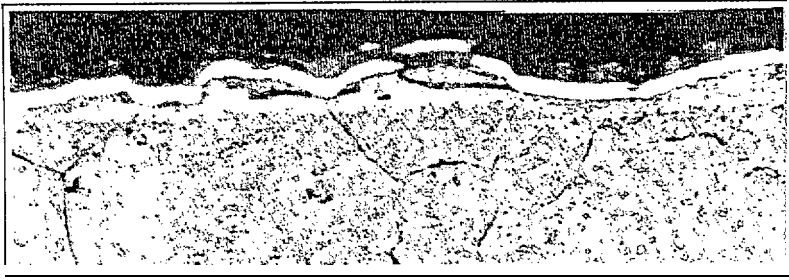


Figure 4.5: Cross-section of ECM machined 1.2083 with T-coating.

4.6 Shot-peening

- Shot-peening gives no rise to bad adhesion and can be applied in those cases where necessary.
- Take care for inclusions of other contaminations which can disturb the coating process.
- Shot-peening is not to be used to remove the amorphous layer of EDM surfaces.

Shot-peening is often used after EDM to work harden the surface or to create compressive stresses. It normally does not remove the white layer. The shot-peening particles are built in the tool surface and can disturb the coating process.

Typical surface characteristics:

- dependant on the steel hardness, the surface has more or less a plactical deformed structure
- some contamination (Si, Ca,..) could be measured

Influence on the TiN coating adhesion:

- shot-peening does not influence the adhesion

4.7 Cleaning and handling

- Job-coaters have to take care for dissolving of the Co binder of cermets and carbides in their alkaline baths (due to presence of complex former).
- Logical and careful cleaning and handling (storage) is of the highest importance for good coating quality.

Because of the sensitivity of the PVD coating adhesion for the initial surface characteristics, the surface must have a high degree of cleanness (free of dust and disturbing elements). Therefor the cleaning applied after the generation and before the coating process is very important.

Typical cleaning problems:

- complex geometries like holes etc.
- dissolving of Co (cermets and carbides) in alkaline cleaning baths

Influence on the TiN coating adhesion:

- presence of contaminations or cleaning products causes flaking of the coating
- the coating adhesion was in general bad on all carbide and cermet samples due to a binder dissolving during cleaning before coating (see figure 4.7)

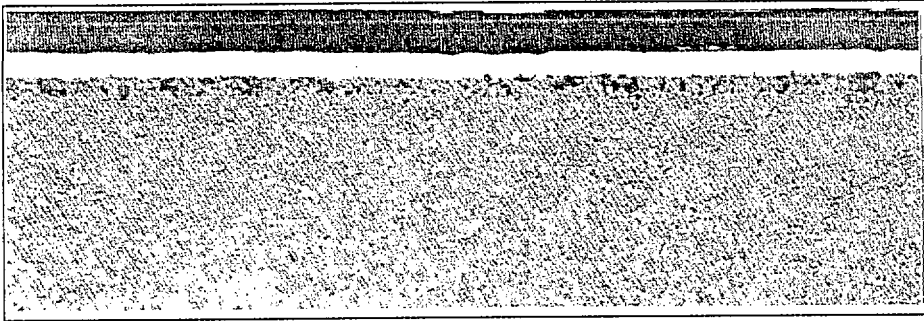


Figure 4.7: cross-section of ground carbide with W coating with typical Co-dissolving.

After surface generation the samples were protected by an anti-corrosion lacquer (Molykote), which could easily be removed afterwards.

First of all tool surfaces must be cleaned in a proper way, this means that the cleaning medium must remove the contaminations on the surface and that afterwards the contaminated cleaning agent is able to leave the tool surface without drying spots. This can be done based on a logical handling of the tools in the cleaning baths. All handling of the tools afterwards, e.g. storing, mounting in the PVD equipment or masking some parts of the surface must be carried out with great care to avoid recontamination.

Cleaning of complex tools can give rise to problems related to adhesion. For example holes in tool surfaces are difficult to clean and coat in an efficient manner.

The dissolving of the Co binder in carbides and cermets during alkaline cleaning can be solved by

- avoid the use of alkaline cleaning product with complex former.
- or reducing the bath temperature (e.g. 30°C) and the cleaning time (e.g. 3 rein),

A complex former is an additive (for example Na-EDTA) which complexes or ties up metals. Due to this action Co is dissolved and tied up in the cleaning product.

5 Influence of surface generation on use of coated tools for different applications

5.1 Turning and milling

5.1.1. Turning and milling of tempering steel using TiN coated HSS

- Tool life is increased significantly compared to uncoated tools.
- The different pregrinding conditions have no influence on the performance of coated HSS.
- Largest possible thickness at flank and rake face is desirable, specially for turning.
- For milling, burrs generated during manufacturing are not allowed.

In milling operations and, moreover, in turning operations a PVD TiN coating significantly increases tool life in comparison to uncoated inserts.

In turning and milling, the coating influence predominates as compared to the pregrinding influence.

In both applications, cutting edges coated using the electron beam ion plating and no substrate rotation (EBIP) process have poorer tool life behaviour as compared to cutting edges coated by the random arc ion plating process (RAIP). The cause is the very thin flank coating due to the first technique, which leads to very high flank wear values. In the tests, almost all the W inserts fail earlier for this reason, through faster attainment of the VB criterion,

Specific variation of the coating thickness distribution shows that in the range of thickness under investigation (up to 4 µm), a homogeneous distribution with the greatest possible thickness on the face and flank is desirable. Tool lives in turning operations can be increased by this means.

In milling operations, burred cutting edges will have lower tool life than unburred ones. In turning operations, the microgeometry of the cutting edge has no effect on tool life. Thus, in milling operations corner rounding reduces running-in wear and opens the way to increased tool lives.

The very thin flank coating of the EBIP coated inserts leads to very high flank wear values. In the tests, almost all these inserts fail for this reason, through attainment of the VB criterion. By contrast, the RAIP coated inserts fail due to flank wear only at low cutting speed and low feed. If a TiN coated crater lip is formed, these cutting edges achieve the longest tool lives irrespective of the cutting parameters.

Obviously the cermet inserts coated by random arc ion plating still have adhesion problems in contrast to the flat samples, which must be due to upscaling phenomena of the cleaning procedure. The coating performance has proven to be very dependent on the working

parameters used and was influenced by upscaling factors, caused by upscaling from the flat samples to complex tools.

5.1.2. Turning and milling of tempering steel using TiN coated cermets

- No influence of different pregrinding conditions on turning and milling performance.
- Coating thickness and micro geometry is not important for turning with cermets.
- Coating thickness and micro geometry is important for milling.
- For milling breakage of coated cermets was failure mechanism.
- Maximum tool life is reached at v_c 200m/min.

Because the results for turning and milling are very different, they will be dealt with separately here:

Turning results:

The coating significantly reduces both crater and flank wear of cermet inserts.

There is no significant influence of different grinding parameters on the wear behaviour of the coated cermets in the turning process. Of course grinding has to be performed as described in 4.1.

Slight differences between the coating process variants are observable. Especially at low cutting speeds, crater wear on EBIP coated cermets is lower than that on RAIP coated inserts. On the latter ones, crater wear is encouraged by poor coating adhesion.

The reduced flank coating thickness of the EBIP coated inserts (because of no substrate rotation) does not appear to cause a significant increase in flank wear.

Related to microgeometry no negative observations were available.

The end of tool life for all insert variants is signalled by attainment of the width of wear land criterion.

Milling results:

There is no significant influence of the different tested grinding conditions detectable in the case of milling with cermets.

The thicker flank coating of the RAIP coated inserts is more resistant to the erosive wear attack than the more thinly EBIP coated inserts. On the other hand, crater wear is slightly higher on RAIP inserts. As in the turning tests, this is attributable to poorer coating adhesion.

In contrast to turning, the dimensions of the teeth of the cutting edge due to the grinding marks have a slightly negative effect on wear behaviour in the milling process.

With one exception at reduced feed, the end of tool life of the cermet inserts occurs due to chipping of the main cutting edge. The position of the cuboid chipped segments is oriented on the position of the ridge cracks. The breakage behaviour of the coated cutting edges is worse than that of the uncoated edges. This difference is particularly apparent in the comparison between the EBIP coated cutting edges and the uncoated cermets. The number of ridge cracks increases with rising cutting speed and feed.

Apart from the absolute extreme, the tool life curve for the range of cutting speeds in the tests ($100 \text{ m/min} \ll v_c \ll 250 \text{ m/min}$) exhibits a relative minimum at $v_c = 100 \text{ m/min}$ and a relative maximum at $v_c = 200 \text{ m/min}$. There is a continuous steep fall in tool life for all variants with increasing feed. The cutting speed variation confirms the tool life behaviour of TiN coated cermets already noted in analogous tests (strip turning test) with interrupted-cut machining of tempering steel (“camel’s hump curve”).

The coated cutting edges exhibit superior wear behaviour on the face and flank. Leaving aside breakage behaviour, flank wear is more decisive for tool life.

5.2 Deep drawing

- No influence of different tested pregrinding conditions found (if ground as described in paragraph 4.1).
- If no polishing of the radius is carried out delamination of the coating occurs (high line loads).
- Coating of massive tools needs adapted coating process parameters, as for example longer heating and etching time.

Although the flat samples showed a sufficient adhesion, the coated tools can have a bad adhesion behaviour. Heating during a too long period and subsequently ion etching during a too short period was the cause of this problem.

After deep drawing 500 cups we could conclude the following :

- an important difference in the tool behaviour when deep drawing austenitic stainless steel was the better quality of the cups produced for coated dies compared to uncoated one
- for the uncoated cold work tool steel, already after making 100 cups intolerable cold welding takes place when deep drawing Zinc/Nickel coated steel. The coated tools have a tool life of at least 500 cups without any problem.
- No real life time tests were performed, only preliminary tests with promising results (related to cold welding, cup quality and tool wear)

Comparing the coated dies with Ferro-Titanit and Hardbronze is interesting on an economical base. Coated 1.2379 is indeed much cheaper than these materials. There is not yet information available about this comparison.

6 Conclusions

Various combinations of tool materials and surface generation processes were evaluated with respect to PVD coating adhesion.

On simple geometries the only adhesion problems were related to

- dissolving of the Co binder in carbide and cermet during cleaning
- the bad adhesion of the coating on the amorphous regions in the EDMed surfaces
- a rather short ion etching step after heating.

These problems could be solved.

Typical upscaling problems are related to :

- inhomogeneous coating thickness
- chipping of sharp edges
- cleaning of complex geometries
- heating and ion etching of complex geometries
- softening of the mould steel due to exceeding the annealing temperature

For the cutting and deep drawing application promising results were presented which must enhance the industrial implementation of PVD coating technology. The findings of this project related to surface generation and pretreatment will be fit in a standard methodology so that PVD coated tools will be of a better quality.

It is clear that all the different steps are important and have to be carried out with a high quality level. The success of a coated tool in its industrial application can only be guaranteed if surface generation, cleaning and coating deposition are executed in an optimal way. This can only be realised if collaboration between tool maker, job-coater and end-users becomes daily practice.

7 Acknowledgements

The authors acknowledge in the name of all the partners the European Commission for financial support (contract BREU 523) and especially dr. Hartley for his opinions and advise during the project. This project was carried out in collaboration with the Technical University of Aachen (WZL-RWTH, D), the Institute for Materials Research (IMO-LUC, B), Helsinki University of Technology (SF), CRIF/WTCM Materials Technology (B), BTC (B), Thyssen Stahl AG (D) and Philips PMF-CMTI(NL).