CRAFT

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Diamond Coating for CUtting and Metrology **Took** (**DICCUM**)

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1 Objectives

The aim is to raise the durability and functional quality of cutting edges, measurement probing units and construction components by a factor of several 10th compared with uncoated tools. The development of diamond coating with good adherence opened a wide field of applications which is described in the following. The workprogramme was focussed on the coating of cutting edges (see point 4):

1. Measuring and contour probing bodies

The reliability of automated measuring and contour probing as an essential component of computer-aided production and of fast orientation systems is decisively determined by the quality of the contour probing a measuring bodies and their resistance to abrasion and chemical erosion. The state of the art for contour probing is, for instance, the use of rubyset heads, the manufacture of which is very expencive and which can be used only for contours whose contour slides are not less than ea. 1 mm.

2. Machine elements subject to high wear

In centerless grinding the so-tailed supporting plate is an essential construction element for the functioning of the machine and for the maintenance of the principle of centerless grinding. The hardness and geometry of this workpiece gliding surface decisively determines the result of the grinding process and its economic efficiency. The workpiece gliding surface at present consists of hardened steel or carbide. The programme has as its aim to coat the workpiece sliding plate with diamond and to obtain the required results through its hardness and optimal coefficient of the sliding friction. Art advantage of DLC coating there is a combination of super hard state of the amorphous film with included graphite like parts, which are necessary for the optimal sliding properties without lubrication,

3. Cutting edges for ultra-precision cutting technology

A partial field of UP production technology concerns the production of laser (reflectors). The (reflectors} geometry and surface quality can can be attained only with cutting edges of monocrystalline diamond. These cutting edges can be produced only by accepting exorbitant expense. The programme aims at creating the necessary precision of the cutting edges geometry on base material which can be processes at relatively reasonable cost, for which the subsequent diamond coating offers the same cutting quality as the monocristalline diamond edges.

4. Cutting edges for machining systems in macrocutting

Cutting tool edges are manufactured according to the state of the art from PCD (polycrystalline diamond) compact materials, ceramic, carbide or high-alloy types of steel. The manufacturing costs of these tools are relatively high and the edge life differs according to material and is also poor compared to diamond. The programme aims at using suitable base materials for the production of standardized cutting systems and partially coating them with diamond. In the field of cutting production techniques a further area of use is thus opened up for improved tools which can be used, among other things, for turning, milling, drilling, grinding and dressing (diamond dressing tools). From the point of view of material techniques, this concerns the processing of aluminium and copper alloys and ceramic elements in machine tools building, in the automobile and aircraft industry as well as of alloys on a cobalt-chrome basis, titanium, tantalum and ceramic facing materials in dental techniques. The large field of processing of steel and iron-based materials needs alternative super hard tools, as cubic boron nitride (CBN), even as coating. The proposed research plan aims at carrying out exemplary practical cutting studies using diamond-coated tools for the processes of turning, milling and grinding. Here principally the influences, dependent on the coating process, on the adhesion to the substrate and on the wear of the diamond layer, are to be investigated and tool-specific recommendations derived from this for improvement of the coating process. In a further phase of work it is intended to carry out comparative tests on the wear performance of diamond coated tools and those of carbide, PCD and natural diamond. This investigation has as its aim to evaluate the performance potential of diamond coating from the economical and technological points of view and to derive recommendations for practical use.

At the end of the programme it is expected that the coating of suitable base materials and the resultant improvement in wear characteristics can be demonstrated, a well founded statement on process lay-out and control can be made and a further field of use will be known.

2 Workprogramme

The workprogramme can be derived from the objectives described before:

1. Design and construction of a coating chamber
2. Testing of the deposition chamber
    - investigation of the basic deposition conditions
3. Coating experiments
    - determination of coating conditions
    - preparation of substrate surface
    - blowing with diamond media
4. Layer investigations
    - determination of crystallinity with RAMAN controlling and AFM (Atomic Force Microscope)

- adhesion strength
- layer thickness
- layer homogenity
- resistance to thermoshock

5. Construction of a testing stand for abrasion tests, development of sample geometry
6. Practical cutting tests
    - Carrying out of preliminary tests to establish test conditions suitable for the tools / materials, which will remain constant

      in the subsequent tests
    - Carrying out of practical experiments under the test

      conditions on various manufactured diamond cutting edges.

      Establishing of coordinated cutting forces, tool quality and

      wear of the cutting edge
    - Comparative durability / wear tests of coated tools and tests

      to obtain information on the repeatability of the quality of

      cutting edge coating
    - Feedback of the test results into the coating process and

      eventual modification of the CVD coating with regard to process-specific demands (improved adhesion, layer

      thickness, roughness of the cutting edge).
    - Trials in industrial use of tools with diamond-coated tools

In the practical test phase findings are especially to be sought to establish whether diamond coatings on turning tools are an alternative to tools whith conventional cutting materials under technological (removal performance, worksurface quality, wear of cutting edge) and economic aspects.

3 Practical investigation and results

3.1 Design and construction of a DLC/diamond deposition chamber and process development

3,1.1 Construction and building of the deposition chamber

The construction of the deposition chamber has been realized into two steps:

Step 1:

the deposition chamber **DIC-1** was designed to realize and to investigate the basic deposition conditions.

The chamber DIC-1 was characterized by a small reactor space. From the vacuum chamber diameter of 300 mm an area of about 100mm diameter for the plasma process (cleaning steps and the different coating steps) was available.

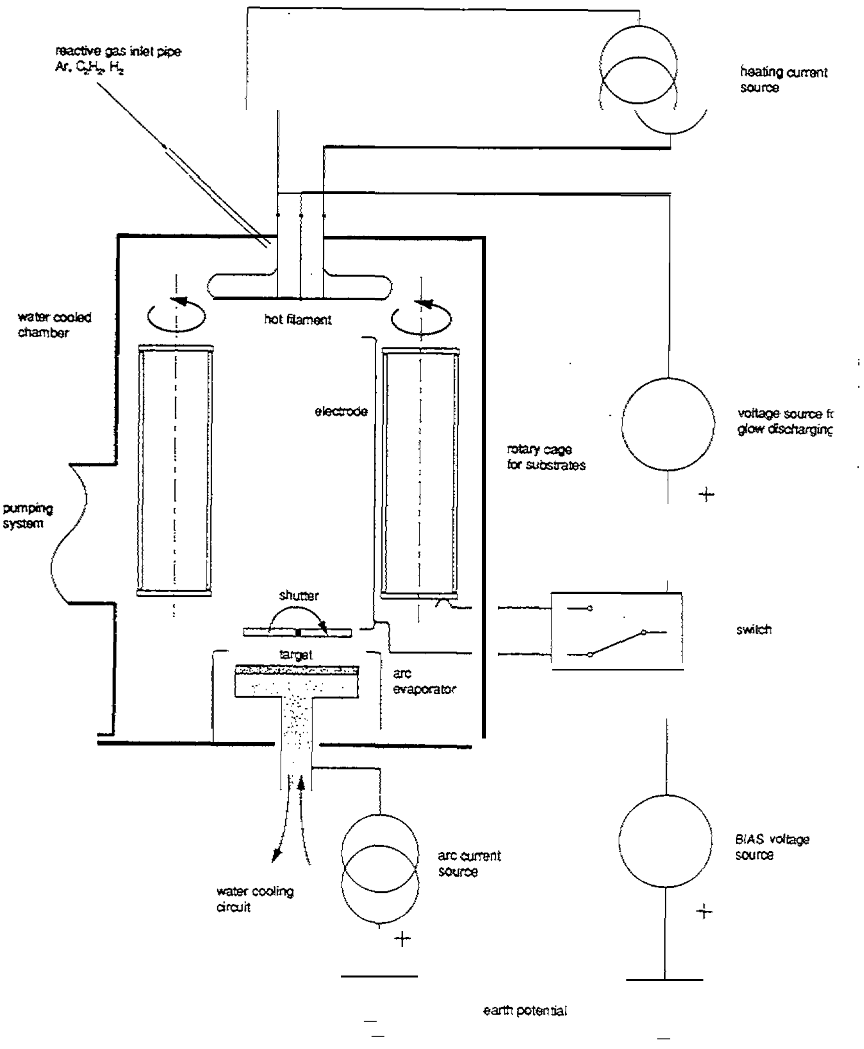

Figure 1: Scheme of PVD/CVD coating system

Table 1: Summary o the design o deposition chamber DIC-2

	Process steps	Realization	Surface treatment method
	Radiation heating with coiled filament	The coiled filament is heated with current till it glows. The rotating basket turns with the substrates.	Heating, destroy water film, remove dissolved gases.
	Electron heating with hot cathode glow discharge	As 1, but the temperature of the coiled filament will be raised till thermal electrons emit, an additional voltage will be applied: minus to the coiled filament, plus to the substrate a noble gas Is let in, subsequently burns a strong glow cathode smolder discharge, what results in a fast heating of the substrate surfaces by the electrons from the plasma. Process parameters: chamber pressure some pa, voltage 60...200 V, current some A	some pa, voltage 60...200 V, current some A, plasma cleaning, but without material removal.
	Plasma cleaning in the glow cathode plasma	As before, but the glow cathode discharge burns between hot filament and chamber wall, the chamber waii is connec ted with the plus - pole of the voltage source, a further voltage source produces a negative potential on the substrates in respect to the glow cathode discharge, subsequently the negative ions will be extracted from the plasma and accelerated toward the substrate surface, plasma cleaning and slight sputtering, Attention: the sputter rates depend onmaterial, at alloys and composites a predominate sputtering of one material can occur.	Plasma cleaning with clear material sputtering, predominantly from foreign substances, at edges and in connection with heating
	modified frame” CVD	As 2 or 3, but a reactive gas or -mixture will be iet in, for example for DIAMOND / DLC:methane or acetylene.	Layer deposition from the gas phase
	ARC - coating	ignition of a vacuum arc between the chamber wail and the target of the ARC - evaporator the ARC burns usual at 18...28 V and 80 . . . 120 A in a metal vapour plasma, which is set free at the erosion of the target, additional reaction gases (N_2, C_XH_X) will be iet in, during coating the substrates are charged negative (BIAS); The following target materials are usual for industrial use: Ti, Zr, Ai, TiAl; possible films are TiN, TiCN, TiC, TiAlN, ZrN et. al., conditions for optimal fiim adhesion and texture: > 200 “C, absolute pure plasma cleaned surfaces.	Layer deposition from plasma from the vapour phase
	Subsequent heating, possible with thermal diffusion	As 1 or 2, possible with reactive gas inlet. Surface temperature increase possibly to > 500 “C.	increase of the interface region, possible additional diffusion with one gas component,

The components of the chamber has been a small hot filament, a thermic insulating shield and the ARC-evaporator for the interface-layer. The different process steps were investigated provisionally and selected parameters have **been measured and** chosen for the next step.

The DIG-I was not suitable for coating of a huge number of substrates. Furthermore the system was also not suitable to carry out the whole coating steps in one closed cycle. Another disadvantage of DIC-1 have been the limited control functions, aiming at the flexible deposition of a multilayers coating.

Step 2:

Already during the first experiments using DIC-1 the new deposition chamber, **DIC-2**, has been designed (see fig. 1). The DIG-2 is characterized by an enlarged reaction space. The vacuum chamber is a cubus of 700x700x700 mm. The thermic insulating shield limits the reaction space and the basket up to 400 mm. The reaction gas flux is directed through the reaction space. A long hot filament (400 mm) has been fiexed at the top of the vacuum chamber, surrounded by the basket with the substrates.

For the control of the whole equipment a computerized system has been installed to ensure the reproduction of the deposition process parameters. The whole design of the deposition chamber, describing the various process, is summarized in table 1.

The main process step of the DIC-2 technology is represented by the deposition of DLC's or diamond. This process step is characterized by a background plasma caused by the HTF. The plasma burns between the HTF and the shield. The basket including the substrates is connected with the negative bias voltage. A gas mixture consisting of ionized argon, hydrogen, and acetylene is guided over the substrate surface. From this follows the plasma is working as the,, flame". Therefore we call this method modified acetylene flame CVD.

3.1.2 Testing of the deposition chamber and optimization

The coating arrangement has been tested thoroughly, where the process parameters have been adapted step by step to the planned values (HTF-current: 150 A, background plasma: 150 V/10 A, bias voltage: 400 V, plasma for heating: 100 V/l 0 A).

Meanwhile the system has been improved to such a degree, that all necessary parameters for the diamond - or DLC - layer deposition can be repeated with sufficient accuracy, A good reliability of the involved assembly and of the mechanical and electrical components has been reached, That is necessary, because of the iong cycle time more than 4 hours for the DLC-coating.

It turned out, that the realization of the **high process temperature** at the substrate surface (800 - 1.000°C - as shown also in the literature) is a difficult problem.

Figure 2 shows the measured temperature curve at the chamber wall (up to 120°C) and the temperature at the substrate surface. For the determination of the temperature at the chamber wall the thermocouple was used. Using metal and alloy wires of known melting points mounted at the substrates and the basket temperature reference points have been determined. The temperature curves for the substrate surface and the rotating basket shown in figure 2 was extrapolated on the basis of the measured values. Especially the components in motion like substrate holders, rotating basket and screen undergo here a high wear. Contrary to all expectations, the control of the **plasma discharges** gives no problems, even above 500°C in the reaction space.

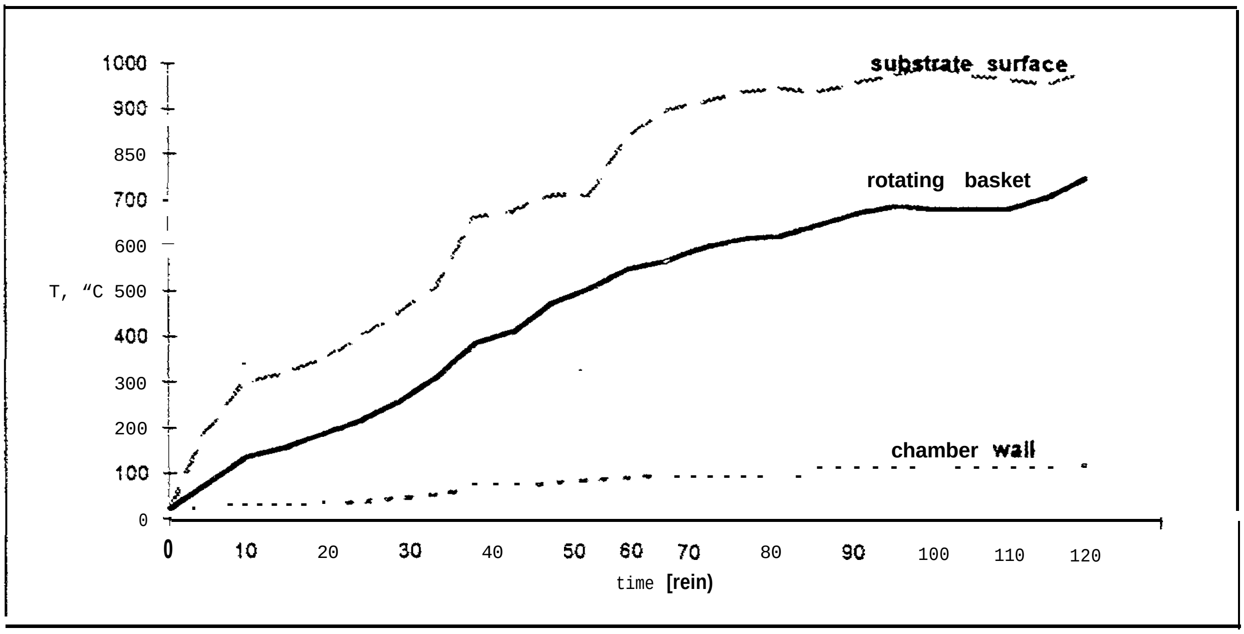

Figure 2: Temperature course on chamber wall, rotating basket and substrate surface

For the selection of the process parameters at different plasma discharges, which are necessary for the DLC - diamond coating process, measurements over a long period have been carried out.

3.1.3 Coating experiments

In the following the different coating steps are shown and described to determine the coating conditions:

- **electron heating with thermionic glow discharge.**

  This process is supported by using an additional cover shield, to exclude contamination and to reduce the power for the whole process.

- **cleaning by glow discharge plasma with argon.**

- **deposition of an intermediate layer using Arc**

This thin layer of TiN (or TiC, TiCN, Ti) is in our process essential to deposite a diamond - or DLC - layer. We have reached good results on the basis of a Ti-TiN-Ti - interlayer of 0.5 µm thickness. The layer determination have been carried out provisionally by means of microscopical observations, simple hardness tests (scratching) and comparison with reference materials.

- **DLC / diamond deposition using CVD - process (modified acetylene flame CVD) in C_xH , . gas.**

The experiments to determine the coating- and deposition conditions for DLC- and diamond layers are carried out in two different directions:

1. Coating experiments **without** plasma support

From the results can be derived that deposition of carbon - DLC -layers is possible in the chosen arrangement, also without plasma support. The only requirements are:

- HTF-temperature higher than 2000°K

. constant acetylene flow through the reactor, where the gas flow coats over the substrate.

The deposited layers have been changed from graphite-like depositions up to deep black middle-hard DLC- layers. Deep black hard layers are coated at substrate temperatures of about 400°C and show a high hydrogen content, which will have to be avoided to prevent the deposit from brittleness. The layers have a tendency to flake off as well as at pure HSS and hard metal samples and at substrates coated with Ti and TiN caused by the internal stress of the coated layers. Therefore these experiments have not been continued.

2. Coating **with** plasma support

Using plasma support DLC - layers of a transparent, light gray, dark gray or black colour have been deposited. At deposition temperatures above 500°C at the substrate the deposits proved to be extremely stable, adhesive and hard. Detailed measurements of the physical parameters have been done. The first coating experiments using plasma support have been carried out in a shortened process regime (only a short plasma cleaning time; the coating time for DLC was limited to a layer thickness of about 1 µm), so that the adhesive strength is not guaranteed over a longer period. This has been proved by scratch testing in the spot.

Meanwhile the process course, existing of

- heating - electron heating - plasma precleaning - Ti- and TiN - intermediate layer deposition as adhesion intermediary and corrosion barrier - DLC- or diamond- coating phase I - DLC- or diamond- coating phase II has been defined (see fig. 3).

Including preparation of the sample, the putting in, vacuum pumping and cooling down, about 4 hours are necessary for one process course. The typical process parameters are:

0 Charging the substrate holder

1 Evacuation of the whole arrangement up to 10-3 Pa

10...20 min

2 Heating of the substrate by radiation heating from the hot filaments

20...30 min

3 Ion-etching of the substrates at 300 V bias-voltage and a background plasma between hot filament and screen, typical parameters: plasma voltage/plasma current; 135 V/7A, HTF-current/voltage 120 A/28 V, working pressure in reaction space 0,1 ... 0,4 Pa

10 min

4 ARC-coating with TiN [0,7 . ..1.5 µm) and Ti (0,1 . . 0,3 pm) typical parameters: coating rate 0,2 µm/min, evaporator current 100 A, reaction gas nitrogen working pressure in reaction space 0,1 . . . 0,2 Pa

4 . . . 5min

5 Ion etching of the substrates at 300 V bias-voltage and a background plasma between hot filament and screen, typical parameters: plasma voltage/plasma current 135 V/7 A, HTF current/voltage 120 A/28 V,-working pressure in reaction space 0,1 ... 0,4 Pa

10 min

6 Heating by electron-shock heating up to a process temperature higher 800 *C, between hot filament and substrates will be ignited a plasma, the substrates are connected with positive potential, typical parameters: working pressure 0,08 ,..0,1 Pa, reaction gas argon, hydrogen and acetylene, bias-voltage 300 V, process temperature 750...900 °C at substrate surface, coating rate 0,01...0,03 µm/min

10 min

7 DLC-coating, typical parameters: working pressure in reaction space 0,1..,0,2 Pa, reaction gases argon, hydrogen and acetylene, bias-voltage 300 V, process temperature 750 . . . 900 *C at substrate surface, coating rate 0,01 . ..0.03 µm/min

30...60 min

8 Postheating of the substrates by electron shock-heating, typical parameters as in <u>pos. 7</u>

10 min

9 Cooling down in vacuum, chamber pressure better 10^{-3} Pa

60 min

10 Ventilation and take out the samples

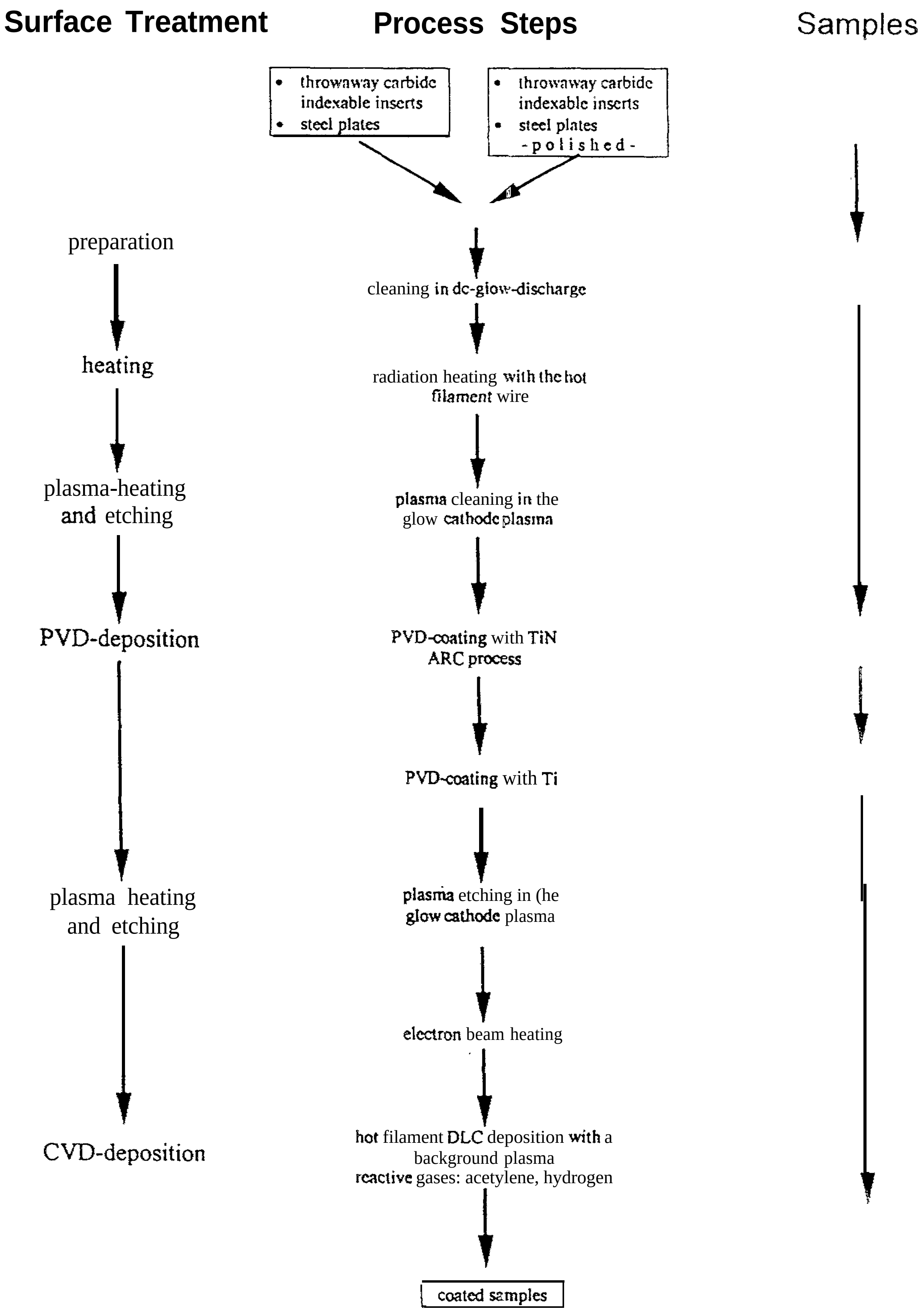

Figure 3: DLC-coating of tungsten carbide inserts

Preparation of surface (cleaning, etc.)

After testing different methods for surface preparation before coating the following procedure have been chosen as the most effective method.

The hard metal substrate material plates, which are intended for the quality tests of the coatings are pretreated in the following manner:

1. fine grinding (if necessary) using diamond paste of grain size D 54
2. prepolishing using diamond powder or paste **of** grain size D 20
3. finishing using diamond paste D 3 (3/2 µm)
4. Ultrasound cleaning, 15 min.

The preparation of HSS tool **surfaces is** made in a similar way. The polishing time depends on the handling degree and the experience of the engineer. With the **above** described procedure a roughness can be reached of 2-3 pm, which is sufficient for a reasonable deposition quality. The hard metal cutting edges which are intended for the **wear tests** have been prepared only by **ultrasonic cleaning.**

3.1.4 Coating experiments to improve the deposition of diamond like structures

The experiments have been done on WC and alsoTantal- and Niob - Substrates. These metals (Ta, Nb) are able to form carbide phases, which support the adhesion properties of the DLC - structures on the substrate. They could be therefore an excellent intermediate layer for the successful deposition of DLC on hard metal or steel substrates.

After the standard preparation of the substrates the influence of the following parameters has been investigated to get DLC - layers with a high part of sp^3- structure (diamond) using the initial parameters:

• gas mixture hydrogen / acetylen 4:1	10 min.
• gas mixture hydrogen / acetylen 1:4	45 min.
• working pressure	0,1 Pa
• 1. HTF (hot filament)	IOOA
• 2. HTF	115 A / 39 V
• Plasma	120 V / 5 A

1. Additional bias voltage

Using a voltage potential of 200 V / 1 A the amount of diamond structure in the layer is higher than without additional bias voltage. The figures 15 and 16 shows the Ramanspectrum of such a sample: the peak at 1332 cm^{-1} is characterized by the diamond structure; the frequency at 1580 cm^{-1} represents the sp^2- structure of graphite.

2. Substrate temperature

The substrate temperature belongs to the most important parameters to carry out a successful process. To investigate the influence of the substrate temperature the geometry of the HTF was changed. The experiments have been done at temperatures of 680 ± 30°C, 770 ± 40 °C, and 840 ± 50 °C. From the Raman-spectra can be derived that the quality of the layer's is decreasing with increasing substrate temperature. This may be interpreted by the fact that with increasing temperature the velocity of the graphitization process is increasing. On the other hand the strngth inside the diamond like layer is decreasing, which maybe derived from the position of the diamond peak.

3. Concentration of the carbon containing gas

The concentration of the carbon containing gases is very important especially at

higher substrate temperatures.

At the maximum of the substrate temperature (840 - 890°C) the mixture of hydrogen and acetylen have been added to the system. The current parameters

at a working pressure of 0,1 Pa were:

● 2. HTF	130 A / 30 VF
● Plasma	130 V/4,5A
● Bias	200 V / 1 A

The experiments have been done changing the acetylen content: It can be derived from the achieved results that with decreasing acetylen content the quality of the layer is increasing.

3.2 Practical investigation on physical properties and wear resistance of diamond coated inserts

3.2.1 Test Programme

3.2.1.1 Measuring methods

The development of new kinds of coatings and the production results of combined coating processes (TiN and diamond for example) require quality control of the layer.

There is a large variety of parameters, which have an influence on the coating respectively on the cutting behaviour. To measure these parameters a wide range of methods is available. With regard to investigation time and costs a complete measurement of all parameters is not practicable. Therefore a selection of measuring methods has to be made. The coating quality on cutting tools can be described by the methods listed in table 2.

The main idea of this work is to create a standard-procedure for testing diamond-coated cutting inserts [1].

At the University of Bremen visual investigations of the coated inserts were performed using macro- and microscopes. Additional cutting tests of the diamond-coated inserts were carried out using the turning **process.** The **turning** process and the measured wear of the cutting edge (VB) are shown in figure 4 in principle.

Diamond-coated cutting inserts

⇩

- Visual investigations (macro- and microscope)

⇩

- Cutting tests
  - cutting forces
  - wear land on the clearance face VB
    chip analysis
  - process control

⇩

- chemical composition and structure investigations by ESCA (electrospectroscopy for chemical analysis) and Raman-analysis on sample inserts

⇩

- layer thickness by ball grinding process
- adhesive strength by scratch test
- hardness by micro hardness test

⇩

- AFM (atomic force microscope) -analysis on sample inserts

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Table 2: Test-programme for diamond-coated cutting inserts

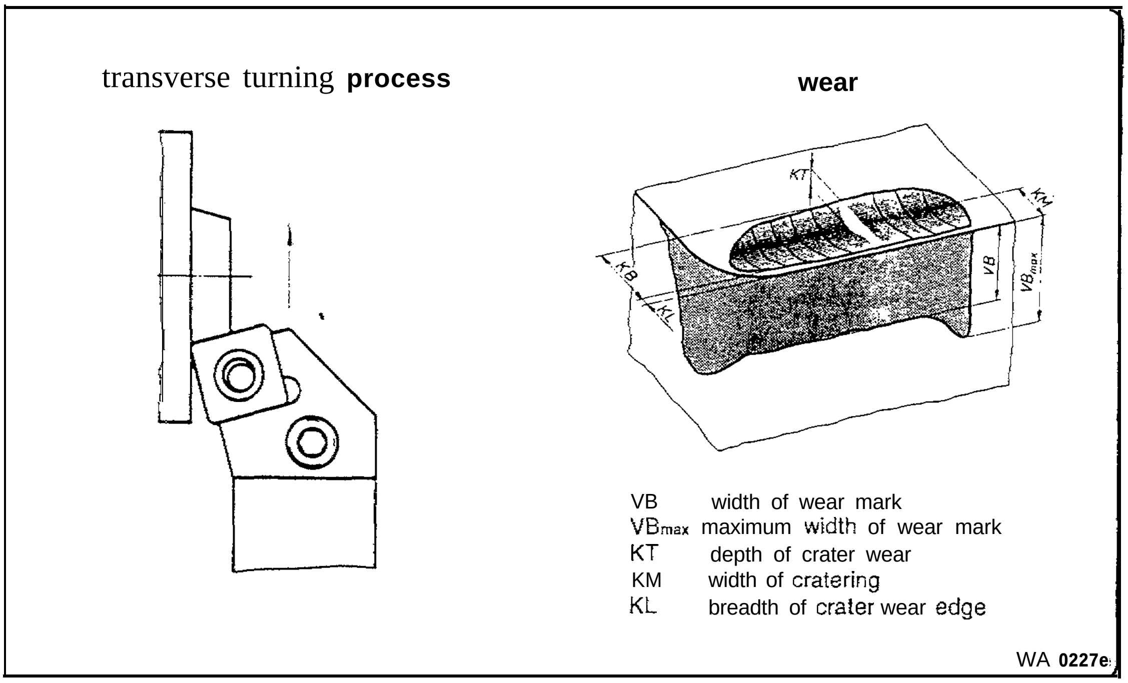

Figure 4: Principle of a turning process on the tool wear (VB)

The basic goal of these tests was to investigate the cutting forces, the tool wear and the process control. In addition to these results the ESCA (Electrospectroscopy for Chemical Analysis) and Raman-Analysis delivered information on the layers structure. The ball grinding process gives information on the layers thickness. Using a scratch test the adhesive strength of the layer can be judged. The hardness of the layer was investigated by micro hardness tests. The roughness, porosity, morphology and topography of the layer can be rated by an AFM (Atomic Force Microscope) -analysis.

Beside these investigations micrographs and SME-photographs of the diamond coated inserts were take. In order to gain information ont the layers structure in more detail.

3.2.1.2 Material, machine and inserts

The general aim of these investigations is to analyse the quality of diamond-coated inserts by turning the aluminium alloy AlSi 18 Cu Mg Ni. This work material is a hypereutectic alloy, which is widely used in the automotive and the aircraft industry [2].

Especially the silicium in the aluminium alloy makes an abrasive effect on the tool flank during the turning process. Therefore this work material is suitable to test the quality of coated inserts.

All cutting tests have been performed with coated tungsten carbide inserts on a Hembrug Super-Mikroturn CNC-machine. This machine has a main spindle with 3000 min^{-1} revolution, a power of 5,3 kW and feedrates up to 1500 mm/min.

. The tools used in these cutting tests are Sandvik CCMW 09 T 304 HI O, Type K 10. The inserts were diamond/DLC coated by Vollstädt-Diamant GmbH (VD).

Furthermore the quality of other **inserts were tested to become reference indications.** These inserts were coated by other companies.

3.2,1.3 Cutting conditions

Before starting the cutting tests some preliminary tests were practiced. By this way the optimum cutting parameters were found:

- process: transverse turning
- cutting speed: v_C = 500 m/min
- feed rate: f = 0,5 mm
- depth of cut: a_p = 0,5 mm
- cooling: without coolant

These cutting parameters were hold constant during all cutting tests. The cutting test ends by a cutting volume of V_W = 130 cm3.

3,2.2 Investigation results

3.2,2.1 Cutting forces

By using the standard cutting parameters extensive cutting tests have been performed. [n all tests the work material was preworked to a circular bar with a length of 30 mm and a diameter to 190 mm. The transverse turning process was used.

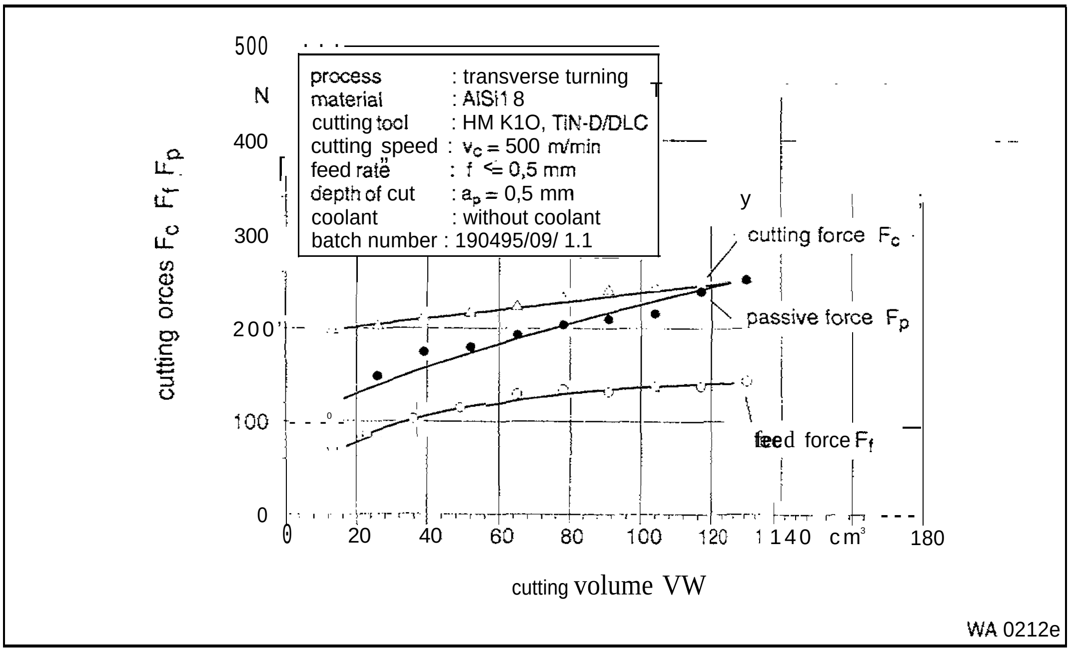

Figure 5: Forces by employing a diamond-coated insert (charge-number 19495/09/1.1)

A cutting volume of V_W = 13 cm^3 was taken off in one cut. For testing the quality of diamond/DLC-coated inserts ten cuts have been practiced. After each cut the flank wear was measured. During the cutting process forces have been measured in three directions by a force-sensing device.

A look at the measured cutting forces shows that the level of the forces by turning with a VD (Vollstädt-Diamant GmbH)-coated insert is similar to the level by using a Sandvik TiN-coated insert. In figure 5 and 6 the curves of the cutting force F_c, passive force F_p and feed force F_f are shown.

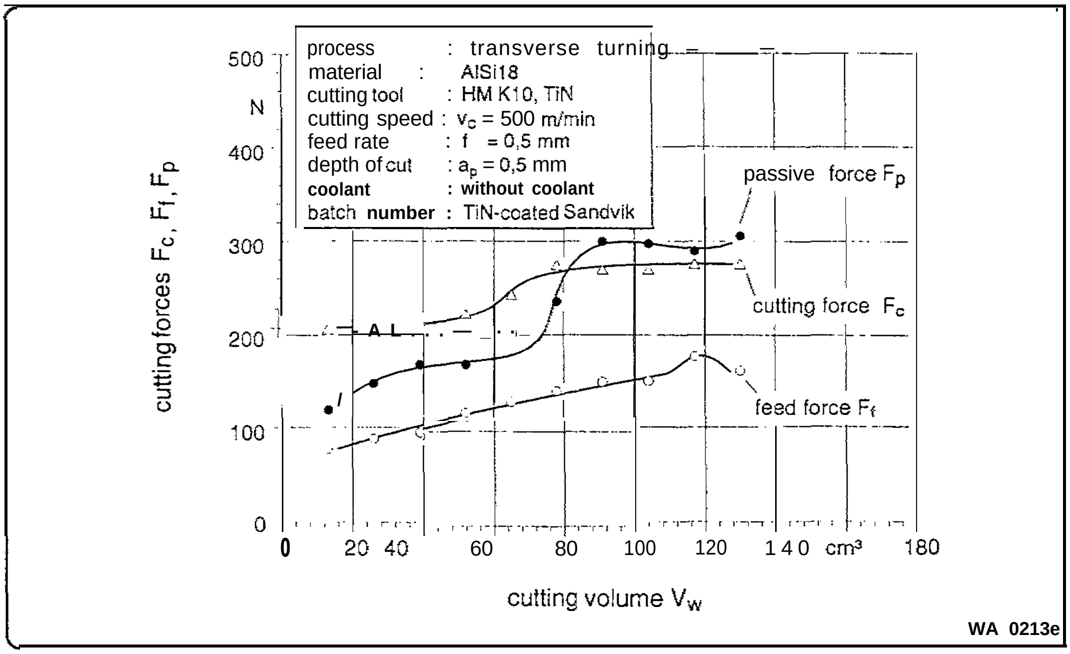

Figure 6: Forces by employing a TiN-coated insert (Fa. Sandvik)

From figure 5 can be seen that the cutting forces perform a constant course, when using the VD-diamond-coated insert, Figure 6 shows a similar course of the cutting forces for the Sandvik TiN-coated insert, but after a cutting volume at 80 cm^3 the forces increase due to a higher flank wear.

3.2.2.2 Wear of cutting edges

The next step in this investigation was to analyse the flank wear. To give a statement ont the quality of diamond coated inserts it is necessary to investigate an uncoated insert HM-K10. In figure 7 the course of the flank wear by turning with an uncoated insert is shown. After a cutting volume of VW= 130 cm^3 the flank wear amounts to VB = 1,93 mm.

From the cutting tests it has been found that the flank wear of most of the VD-coated inserts (VD 240395/1 6- VD 030595/1 5) can not be measured because the inserts were not coated entirely. Just the tool face was coated. Therefore, only the wear of the substrate material can be analysed. The tool flank and the cutting edge were not or only partly coated. These inserts could therefore not be employed in the cutting tests.

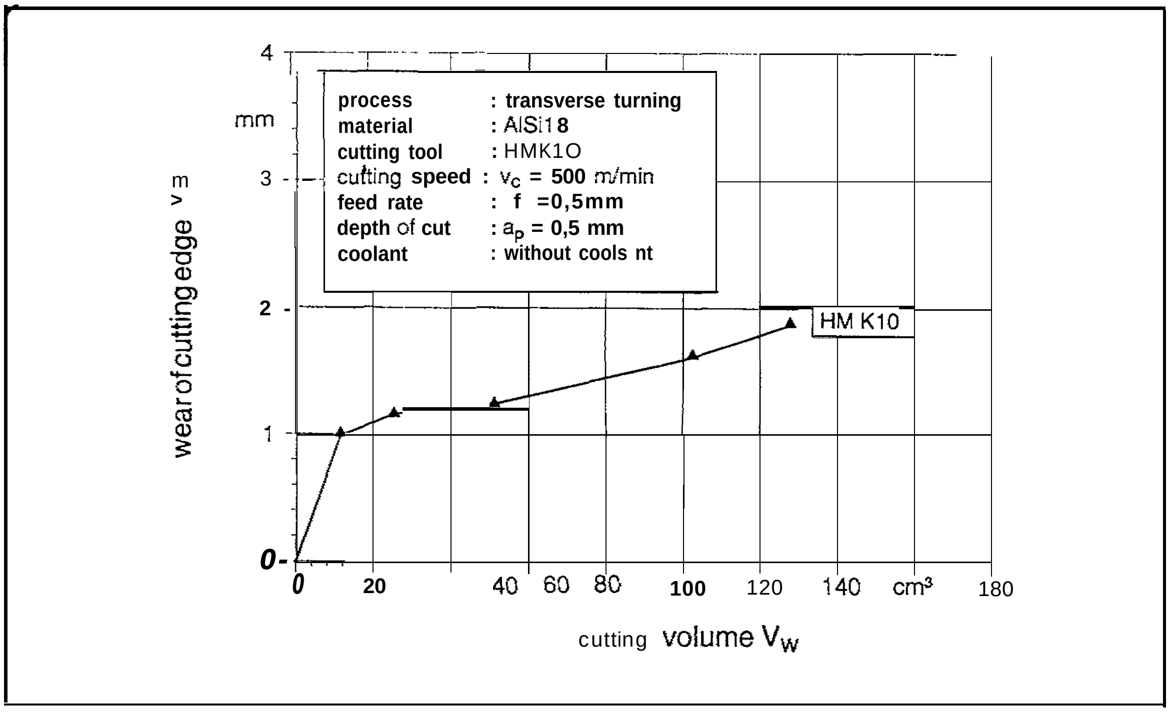

Figure 7: Flank wear of an uncoated insert

Due to the results of the measurement of the flank wear it can be stated that there are quite some problems in the adhesive strength of the layers. After the first cut (cutting volume V_W = 13 cm^3) the layer is completely removed. This is due to the fact that the VD-coated inserts were not entirely coated on the land face.

Only the three charges VD-2905951OI, VD-290595/02 and VD-030795/03 shows a real flank wear of the layers. These inserts ***were*** coated with different coating parameters. The results of the cutting tests with these inserts seem to be sufficient. [n figure 7 the comparison of the flank wears of the VD-coated inserts and an uncoated insert is given. Especially, the charges VD-290595/01 and VD-290595/02 reached a flank wear under VB = 1,2 mm. The reference inserts (Ref. 1 and Ref.2) showed a clear flank wear of the coating. The assessment of the results make clear that the Ref. 1 -insert proved to be successful in cutting tests. The adhesive strength of these layers seems to be sufficent.

Figure 8 shows the course of the flank wear of the Ref1 .-insert. After a cutting volume of V_w ‾ 130 $cm^3$the flank wear amounts to VB = 0,58 mm.

In figure 9 the flank wear of different inserts after a cutting volume of V_w= 130 $cm^3$is shown. The inserts were etched after cutting in order to remove the dummy chip. This made the measurement of the real flank wear possible.

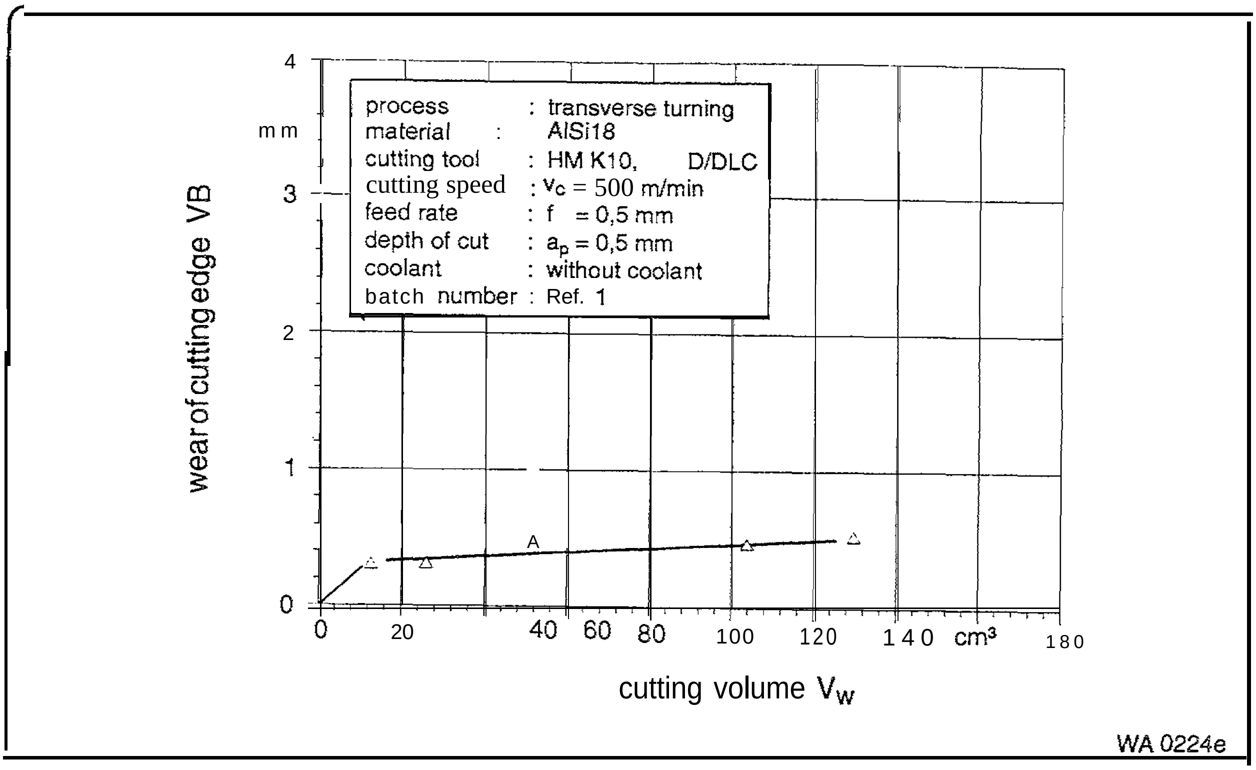

Figure 8: Flank wear of the Ref1 .-insert

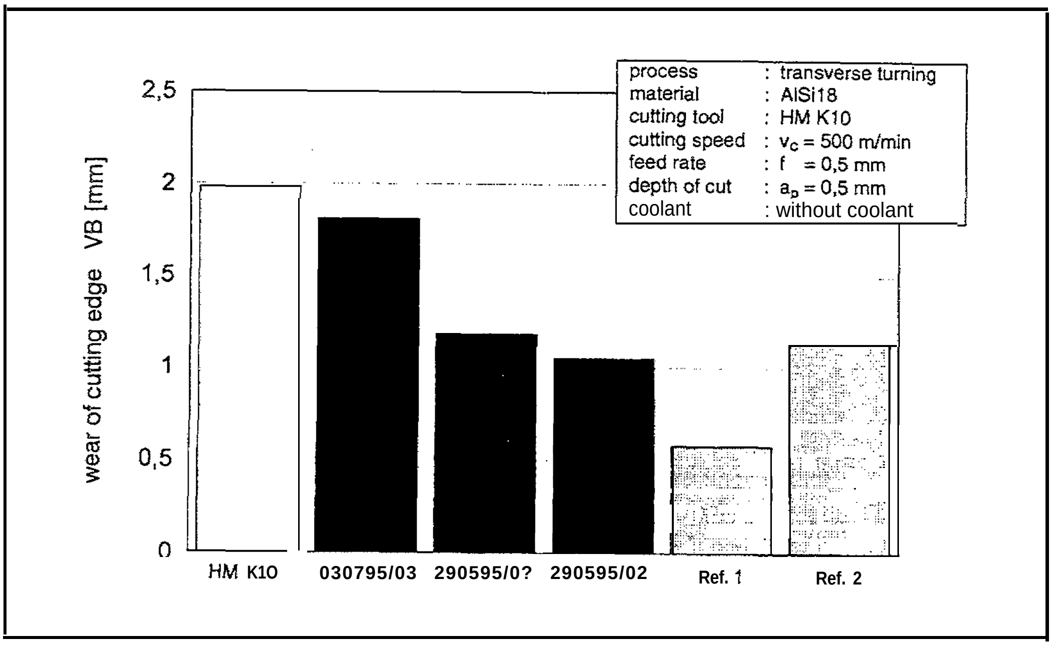

Figure 9: Wear of cutting edge for different batch numbers

The results of these measurements vary between VB = 1,98 mm (max.) for the uncoated HM-K1 O insert and VB = 0,58 mm (min.) for the Ref. 1 -inserts. This makes clear that the charges VD 290595/01 and VD 290595/02 achieve relatively good results. The aim of a following investigation must be to achieve a better reproducibility of these coating results.

3,2.2.3 Layer thickness

The layer thickness was investigated by the ball grinding process. Using this method the layer thickness can be measured. In figure 10 the layer thickness of all investigated inserts are shown.

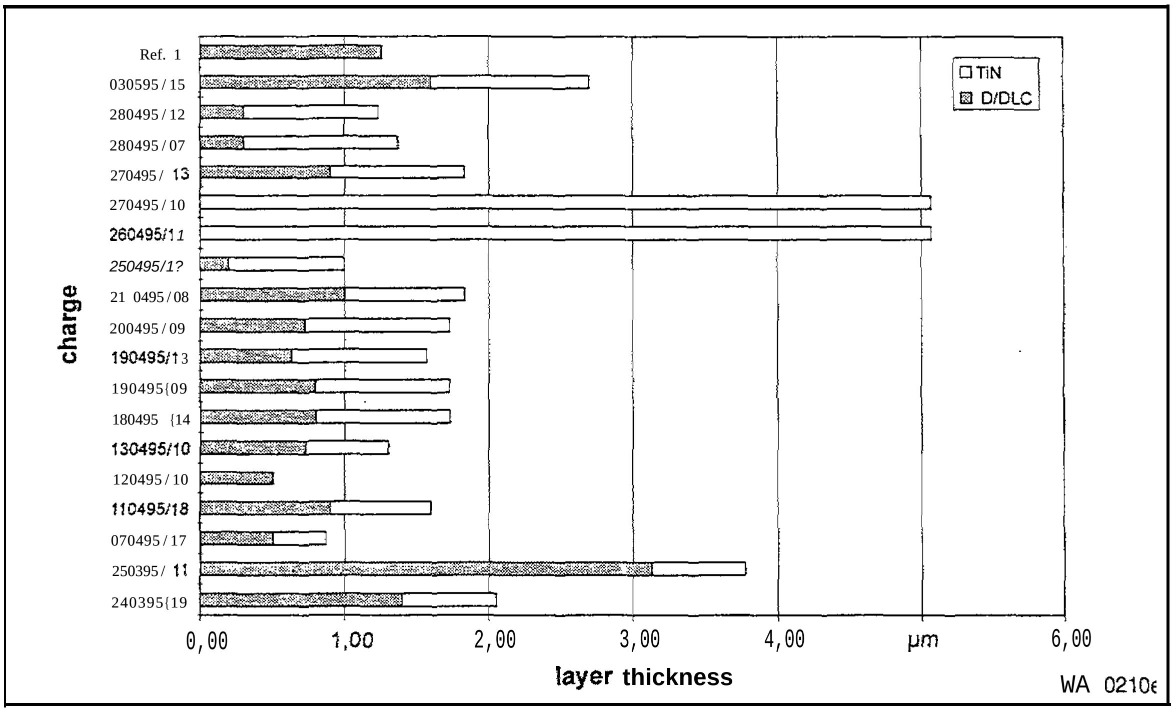

Figure 10: Layer thickness

The data shows that the layers have a thickness smaller than 4 pm. in figure 10 one can compare the thicknesses of the TiN-layer and of the diamond/DLC-layer. From this figure it can be seen, that the VD-coated inserts show a wide range of thickness (0,2 -3,1 µm). Most of these inserts are coated with a D/DLC-layer less than 1,0 pm.

3.2.2.4 Scratch tests of adhesion strength

The scratch tests give more information about the adhesive strength of the layers. In figure 11 the results of the scratch tests are shown.

The necessary forces for scratching through the layers are very small. In general the forces for scratching the DLC-layer are smaller than 10 N. This shows that the layers are not resistant to wear. Only two VD-inserts with the charge numbers 190495}13 and 130495}10 could prove to be wear resistant,

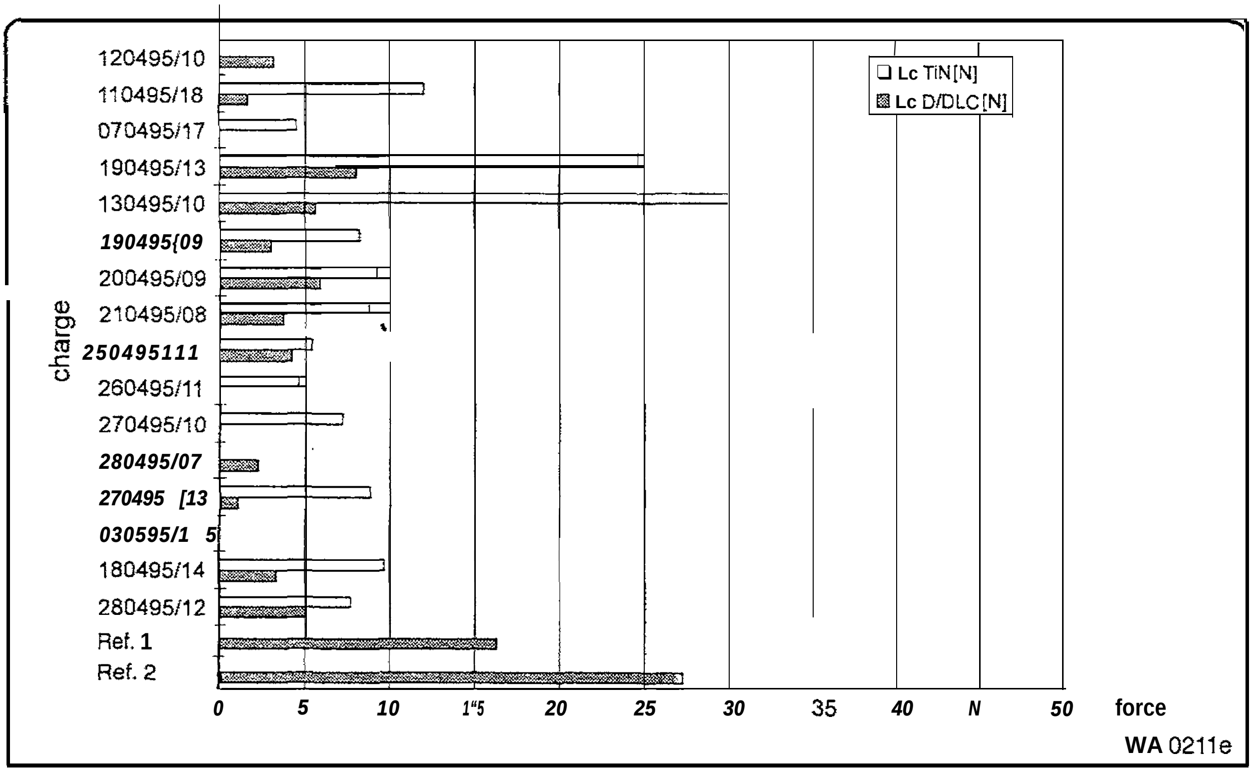

Figure 11: Visual evaluation of the scratch tests

Because of the thin layers it is not possible to perform micro-hardness tests. But the results of the scratch tests make clear that the hardness of the layer is very low. The layers are soft and weak. Especially, the effect of the transformation of the coating material in to simple carbon causes problems. The irregular and unclear layer surface is not effective against wear.

3.2.2.5 Atomic Force Mocroscopy (AFM)-analysis

An AFM-analysis gives information on the morphology, topography, porosity and roughness of the layers.

In figure 12 the tool face of a VD-coated insert is shown. When analysing this AFM-photograph it can be noticed that the layer is very irregular. The color difference is very high. An index for the layers roughness is the RMS-factor. It is comparable to the arithmetical mean deviation of the profile. The result of the measurement is a factor of RMS = 530 nm. This can be regarded as an indication for a high roughness.

This figure shows a fractional coated layer with areas without coating. This finding might be an explanation of the poor results of the scratch tests. By looking at the AFM-photograph of the tool face of the VD-290595/02 insert a regularly layer structure can be seen (figure 13). The RMS-factor is RMS = 90 nm.

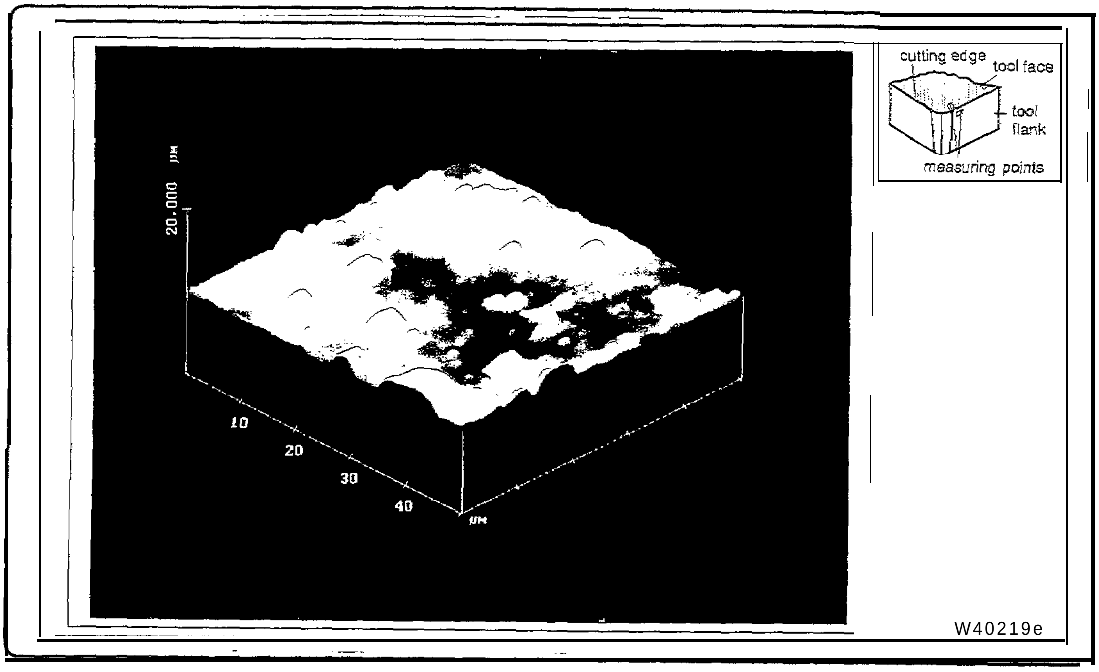

Figure 12: AFM-photograph of the tool face of a VD-coated insert

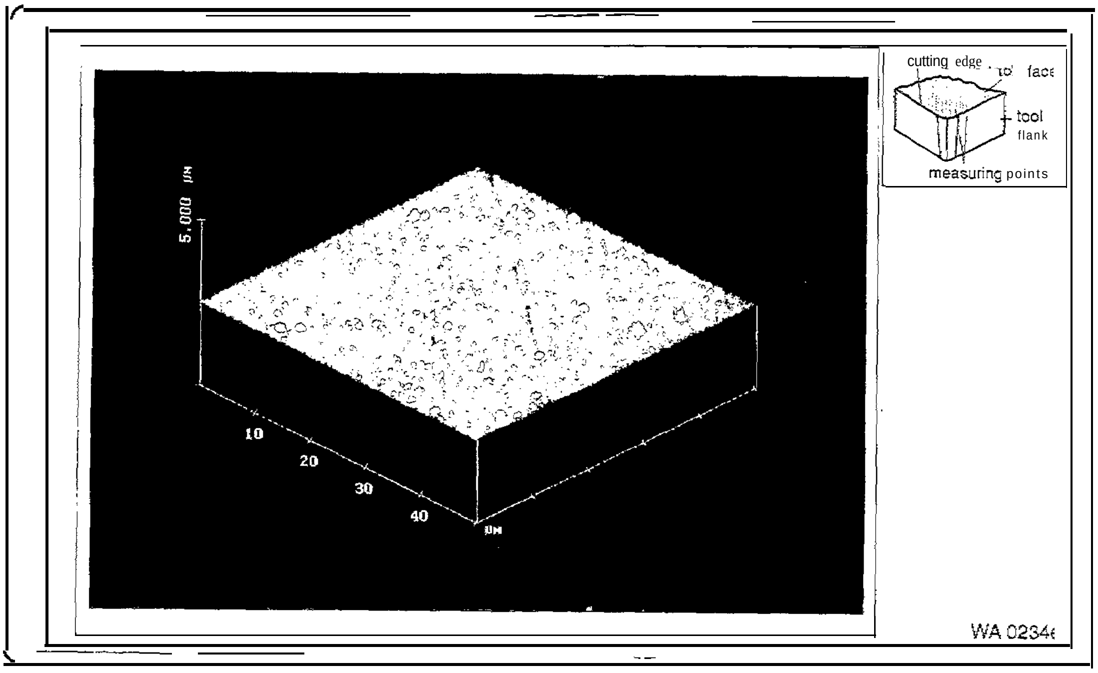

Figure 13: AFM-photograph of the VD-290595/02 insert

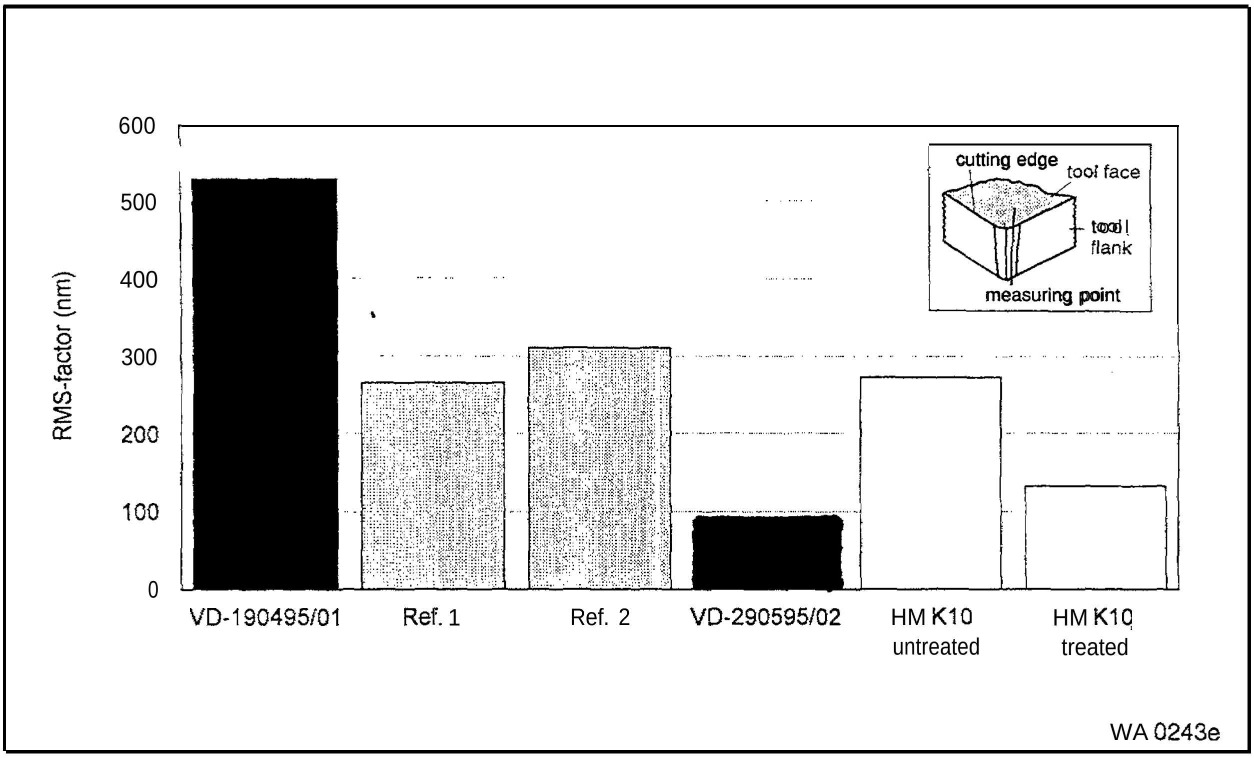

Figure 14: RMS-factors of the tool faces

In figure 14 the RMS-factors of differnt inserts are shown. The comparison of these results makes clear that the VD-290595/02 insert has a very plane surface, The RMS-factor amounts to RMS =90 rim. It can be supposed that after the coating process the surface of the insert was ground.

3.2.2.6 ESCA and Raman-Analysis

With sample inserts investigations by ESCA were practiced. The measurements were fruitless. Because of the strong contamination of the layers with carbon an assessment by the method of ESCA was not possible.

The investigation by Raman-spectroscopy gives information about the chemical structure of the layers [3]. The measurements were practiced with 1800-reflection in a N IR Broker Fourier-Transform Raman Spectroscope. An excitation of 1064 nm was used. The dissolvement is 2 cm-l. The measured point has a diameter of 1 mm.

The "Mineralogisch-Petrographisches Institut" of the University of Hamburg has performed these measurements. Four selected inserts were investigated. To summarise the results one can state that no diamond-structures or diamond-like-structures could be located on most of the VD-coated inserts. The maxima at

1287 cm^{-1} and 1600 cm^{-1} are carbon-/graphite-structures with sp^2- and sp^3- phases (figure 15).

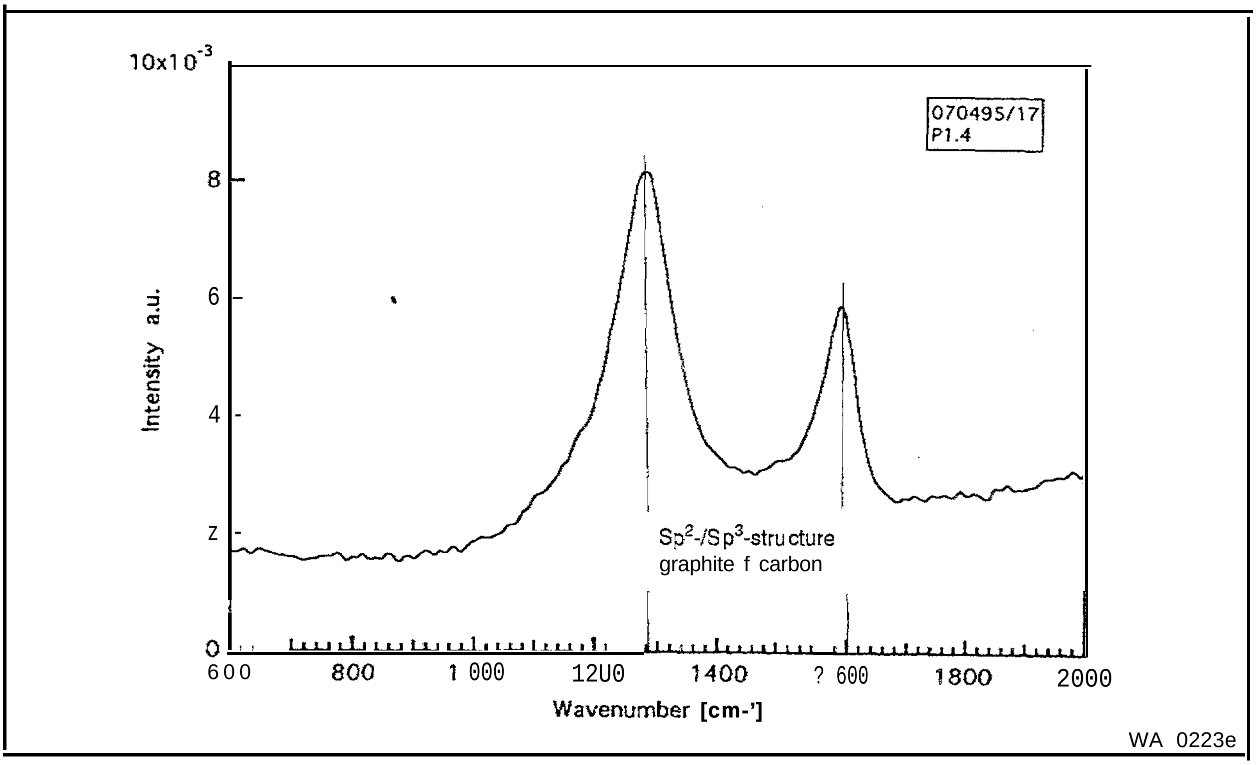

Figure 15: Raman-analysis of a VD-coated insert

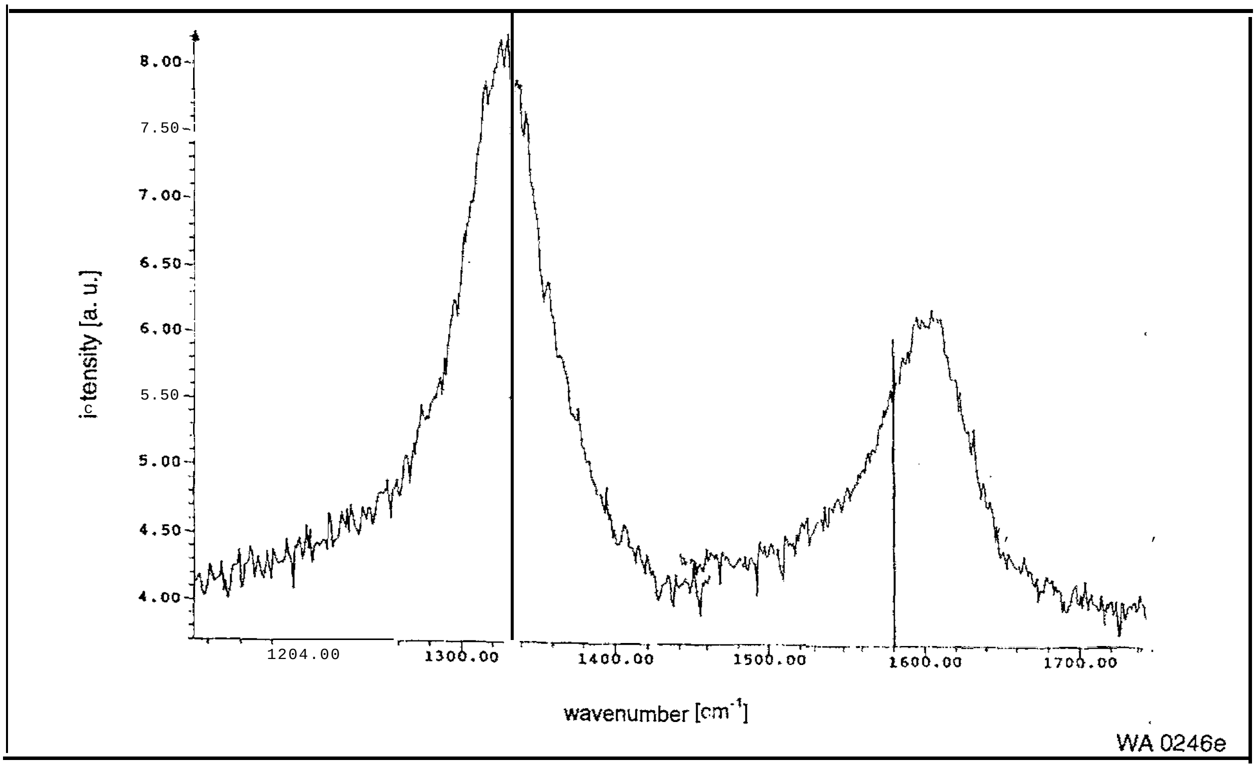

Figure 16: Raman-Analysis of the VD-290595/02 insert

Only the VD-290595/02-insert shows a maximum near to 1339 cm-' (figure 16). This suspected a diamond- or DLC-layer. The low intensity indicates some volume percent of diamond-structures. This result seems to be succesful.

3.2.2.7 Reproducibility of the coating results

The utilization of chemically vapor deposited diamond films as anti-wear-coatings on cutting inserts is often limited by a lack of its adhesion. Therefore, the reproducibility of the coating results is very important for an industrial application of this coating process. The aim of the project was to reproduce the good coating results of the charges VD-290595/01 and 290595/02. At the end of the project time this aim should be reached.

The results of the cutting tests of the last four charges coated by Vollstädt Diamant GmbH are shown in figure 17.

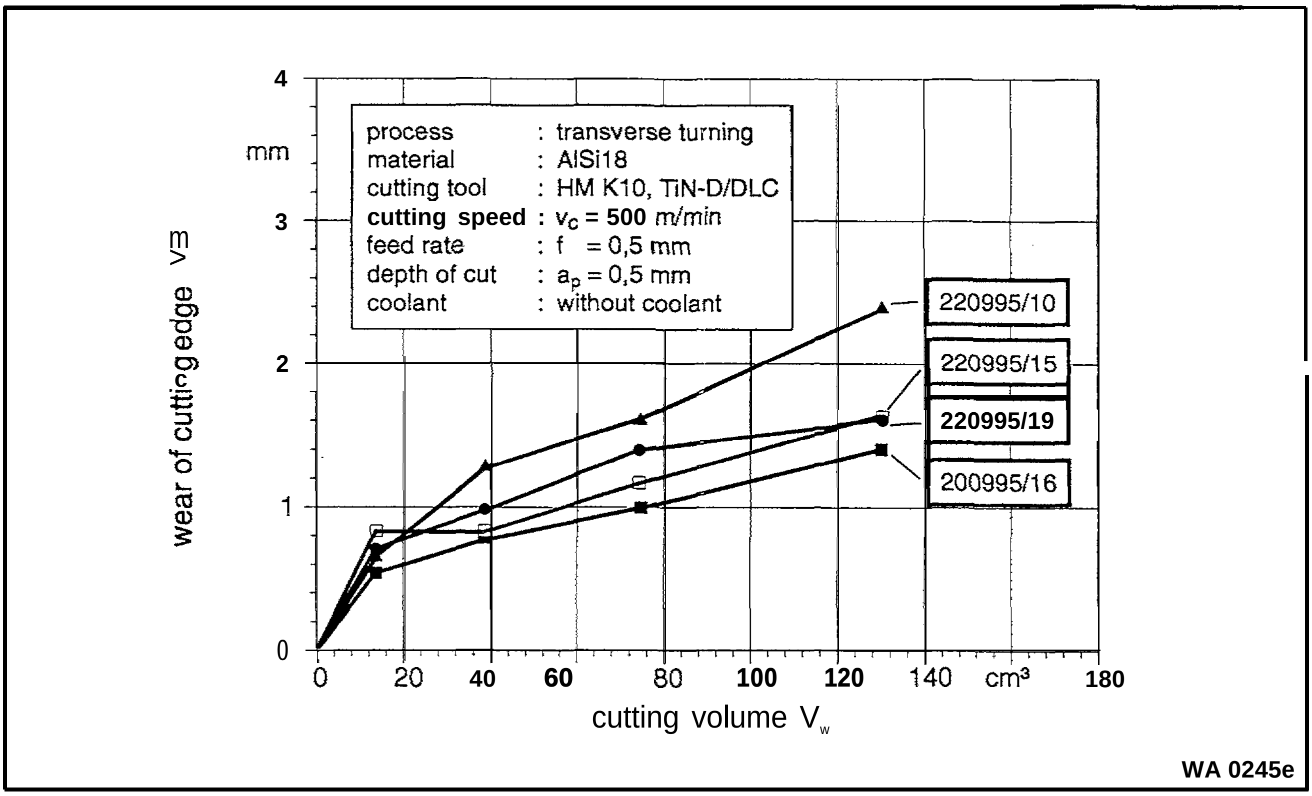

Figure 17: Wear of cutting edge dependent on cutting volume

From these cutting tests it has been found that the flank wear of most of the VD-coated inserts (VD 200995)16, VD 220995/1 O, VD 220995/15 and VD 220995/19) can not be measured because of the problems with the adhesive strength of the layers. Therefore, only the wear of the substrate material can be analysed. After the first cut (cutting volume V_w = 13 cm^3) the layer is completely removed. Unfortunately, it was not possible to repeat the good results. Apparently the coating process can not be controled for a reproducible process course. The flank wear of the substrate material of these inserts can be seen in figure 17.

4 Conclusions

According to the content of the project „ Diamond coating for cutting and metrology tools" (DICCUM) the target of the project has been the surface hardening of cutting and other tools by means of a modified acetylene - flame CVD - coating using superhard materials, mainly diamond, and diamond like carbon (DLC).

The project has been realized in these main steps

- design and construction of the deposition chamber dummy
- development of the coating technology and coating experiments
- wear test of the coated cutting tools.

The deposition chamber DIC-1 has been designed and built to realize the basic deposition conditions.

The deposition chamber DIC-2 represents a deposition system which is suitable to carry out a whole cycle of 5LC - or diamond coating, consisting of the following coating procedure: plasma heating - plasma etching - interlayer deposition - DLC / diamond deposition (modified acetylene flame CVD). The DIC-2 has been constructed and built using alternative combinations of coating technique, e.g. plasma supported PVD and acetylene flame CVD, to improve the coating conditions. The deposion system DIC-2 includes the following improvements, which allow the coating of tools close to the pilot state:

- large coating space with thermal shielding and rotating basket
- control system for stable gas flow and plasma working power.

From the experiments the following results and problems can be derived, which should be taken into account for the coating experiments:

- some improvements of the equipment are necessary, i.e. realization of higher gas flows by installations of another diffusion pump, the completion of control equipment, implementation of components and control algorithms for the gas flow and the plasma stabilization - these ones are already prepared,
- reconstruction of the thermal shielding of the rotating basket to improve the homogeneity of the gas flow depending on the coating results, which are planed for the following period,
- temperature measurements on the substrate in motion
- further increase of the plasma working power on the substrate surface

The coating experiments, using mainly cutting edges as substrate materials have been carried out using different deposition conditions, which include gas flow rate, gas composition, target power, substrate temperature, deposition time, plasma heating, intermediate layers.

After optimization the following deposition conditions and procedures have been stated to obtain the best qualify of coated edges with a high part of diamond structure:

- mechanical cleaning of the substrate using diamond micron technique
- deposition of intermediate layer (Nb, Ta, Ti - TiN)

• gas mixture hydrogen/acetylen	4:1	(10 min.)
• gas mixture hydrogen/acetylen	1:4	(45 min.)
• working pressure	0,1 Pa	
• Hot filament 1	100 A	
• Hot filament 2	115 A/ 39 A	
• Plasma	120 V/5A	

The workprogramme aimed at carrying out exemplary practical cutting tests using diamond coated tools for the turning process. Here principally the influences, dependent on the coating process, on the adhesion to the substrate and on the wear of the diamond layer, were investigated and tool-specific recommendations derived from this for improvement of the coating process, The main aim was to raise the durability and functional quality of the cutting edges.

The basic goals of the cutting tests were to investigate the cutting forces, the tool wear and the process control. In addition to these results the ESCA (Electrospectroscopy for Chemical Analysis) and Raman-Analysis delivered information on the layers structure. The ball grinding process has given information on the layers thickness. Using a scratch test the adhesive strength of the layer could be judged. The roughness, porosity, morphology and topography of the layer were rated by an AFM (Atomic Force Microscope) -analysis. [n addition to these investigations micrographs and SME-photographs of the diamond coated inserts were made.

The experimental tests performed show that diamond / DLC-coated inserts used for cutting tests could not deliver satisfactory results. Only the charges VD-290595/01 and VD-290595/02 seem to be succesful. The Raman-Analysis shows that there are diamond-like structures on these inserts. But it was impossible to reproduce these good coating results.

The aim of the project, which was the development of a controllable coating process for diamond coating was not reached. Due to the problems with the adhesive strength of the layers the cutting tests with most of the investigated inserts give only information about the flank wear of the substrate material. The effect of a strong contamination with carbon on the layer leads to irregular structures of the layers. The adhesive strength is not guaranted. The proposed chamber and technology on the basis of the DICCUM-project, which has been applied for patent has to be improved for being used at an industrial level, concerning the technological and economic basis.

5 References

Layer investigation

[1]	Jehn, H.	Charakterisierung dünner Schichten, DIN-Fachbericht 39 Beuth Verlag, Berlin, 1993
[2]	Erinski, D.	Untersuchungen über den Einfluß des Werkstoffgefüges auf das Zerspanverhalten von Al-Si-Gußlegierungen Dissertation, Universität Bremen 1990
[3]	Knight, D. S.	Characterization of diamond films by Raman spectroscopy, Materials Research Society, 4 (1989) 2, p. 385-393
[4]	König, W.	Fertigungsverfahren Band 1, VDI-Verlag, Dusseldorf 1981

Coating precess

[1] E. 1. Tochitsky, A. V. Stanishevskii, V. V. Akulich et. al.. The formation of diamond-like carbon coatings for raising the wear resistance of precision friction pairs. Surface and Coatings Technology, 47(1 991) 792-296

[2] E. 1. Tochitsky, O. V. Selifanov, V. V. Akulich et. al.. Electrical erosion pulsed plasma accelerators for preparing diamond-like carbon coatings. Surface and Coatings Technology, 47 (1 991) 522-527'

[3] H. E. Hintermann, A. K. Chattopadhyay. New generation superabrasive tool with monolayer configuration. Diamond and Related Materials, 1 (1992) 1131-1143

[4] E?. Lux, R. Haubner, P. Renard: Diamond for tooling and abrasives. Diamond and Related Materials, 1 (1992) 1035-1047

[5] S. J. Bull, A. Matthews. Diamond for wear and corrosion applications, Diamond and Related Materials, 1 (1992) 1049-1064

[6] Y. Saito, T. Isozaki, A. Masuda et al.. Adhesion strength of diamond film on cemented carbide insert. Diamond and Related Materials, 2 (1993) 1391-1395

[7] G. K. Wolf. Erzeugung verschleißmindernder Randschichten durch Ionenstrahlverfahren. Mat, -wiss. u. Werkstofftech., 24(1 993) 109-119

[8] H.-J- Scheibe, B. Schultrich. DLC-Film Deposition by Laser-Arc and Properties Study. Materials of the 9th International Conference on Thin Films ICTF9, Sept. 6-10, 1993, Wien

[9] M. Nesladek, J. Spinnewyn, C. Asinari et al.. Improved adhesion of CVD diamond films to steel and WC-CO substrates. Diamond and Related Materials, 3 (1993) 98-104

[10] J.-P. Peyre, D. Duchateau. Evolutions des Dépôts PVD-CVD. CETIM-Informations, No. 138, Avril 1994

[11] F. Miller. Superharte Schichten aus Diamant und kubischem Bornitrid. Metalloberfläche 48, 6(1994), 404-406

6 Annexes

List of publications

1. Workshop, 09. + 10.06.1994 EG-Verbindungsbüro fur Forschung und Technologies, Steinbeiss-Europa-Zentrum, Stuttgart
2. 5th Conference on Industrial Technologies, 06.-08,12.1994 at Brussels
3. Patent 12/P-01 /DE : Verfahren und Einrichtung zur Abscheidung von Funktionsschichten auf einem Substrat mittels plasmagestützter Schichtabscheidung