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# 1. ABSTRACT

Sputter coatings are used industrially as hard coatings for protection of tools and tool devices against wear and corrosion'.

The most significant objectives of this project were the development of coatings based on new target materials and the optimization of the lifetime of cutting tools by means of tool design according to the properties of the new coatings.

Sintered targets with variable chemical composition based on tungsten alloyed with titanium, nickel, chromium and other metals were used to deposit thin films as nitrides, carbides and carbonitrides.

The study of the deposition conditions was carried out initially in a laboratory equipment. This work lead to the construction of a prototype with similar characteristics to a high rate sputtering system.

Thin films were deposited over high speed steel and cemented carbide cutting tools with standard and. later with optimized geometries. The deposition conditions in the" prototype were optimized for grinded (cutting tools) and polished (samples)" surfaces, establishing the standard process parameters.

Samples coated with GOLD-TiN, SILVER-TiN and TiCN were supplied by means of industrial production of standard coatings deposited from a titanium target. The study of the coating Properties. was carried out in view of the optimization of the target chemical composition. Each target alloy was used to 'produce coatings for cutting tools to acchieve higher lifetime and an optimal wear behaviour during service.

Comparative studies were carried out on a Fin-on-disc equipment, establishing the relationship. between the wear rate and the friction coefficient of the sputtered coatings. The study of the wear behaviour was carried out at room temperature, as well as at temperatures up to S00 degrees Celsius for high speed steel coated samples and up to 600 degrees Celsius for cemented carbide coated samples.
A fretting test equipment was used to complete the study of the wear behaviour of the coatings at room temperature.

The wear behaviour of the new developed coatings was studied under real cutting conditions using coated high speed steel taps and thread formers and cemented carbide mills and drills. Accordinq to the properties of the optimized coatings, new tool geometries were developed. Finally, the cutting conditions were optimized for the tools with new geometries.

## 2. INTRODUCTION

The aim of the project was to develop new coated cutting tools with better cutting performances than those obtained by conventional coatings and tool geometries. Two important objectives should be reached, concerning the deposition of W-C-/N-Me (Me=Metal) coatings and the adjustment of the tool geometries to the properties of the new developed coatings.

The definition of the target chemical composition, which leads to the optimal film properties, as high hardness and tenacity, high temperature and abrasive wear resistance, low friction coefficient and high deposition rate, was certainly the most important subject 'to be analysed.

A first study on the deposition conditions was carried" out in a laboratory 'equipment, where different target materials were used, establishing simultaneously the basic work for the definition of a PVD coating equipment with characteristics similar to "an industrial system; The conception of the sputtering system' included a planar magnetron cathode, an ion beam source positioned in front of that cathode and a rotational substrate holder. This system allows the' coating deposition using simultaneously the ion beam source to increase the plasma ionisation.

The vacuum equipment with an ultimate pressure under 10-6 mbar works at pressure values under 3 x 10-3 mbar and at deposition rates up to 0,35 μm/min. in order to guarantee the purity, Of the coating chemical composition and the rentability of the deposition process.

The application of coatings to" cutting tools leads generally to severe alterations of the cutting conditions. For adjusting these conditions to achieve the best results, it was necessary to acquire complete' basic data for standard tools coated under standard conditions and establish the relationships between the coating properties and the cutting conditions and the tool geometries depending on the tool and worked materials:

Standard tool geometries were used to optimize the pre-heating, etching and deposition conditions in the prototype equipment and to study the influence of the coating properties and film thickness on tool geometry and on their cutting performances.

The correlation of the film properties with the wear phenomena was investigated, in order to develop new cutting tool geometries designed' for optimum surface treatment and manufacturing productivity. These tools should work at high cutting speed and feed rates, also without lubricant or coolant.

## 3. TECHNICAL DESCRIPTION

### 3.1. SELECTION OF THE BASE MATERIAL FOR THE COATING

The deposition conditions were initially optimized, when reactive sputtering from a pure tungsten target. The study of the influence of the elements nitrogen and carbon on the" properties of the films showed that the increase of nitrogen flow rate makes the film more. oriented in (110) direction and decreases the coating hardness,' specially when the $W_2N$ phase is present. Scratch-test results have shown that W-N films fail always cohesively, firstly by tensile cracking and after by chipping. The structure of W-C/N films is different, where the phase WC carbide was detected. This phase is isomorphous of WN.

In addition to the element tungsten, a preliminary selection of the metallic elements for the target material was carried out, using chromium, nickel and titanium to analyze the influence of the 'second metallic element. The study was carried out in non-reactive mode to compare the results with those of amorphous and crystalline W-C-Co.

The deposition of W-C-Ni coatings have shown that the addition of Ni leads to the amorphization of the films when its content in the film overcomes a thresold value of 5 % atomic. In this system was detected the presence of. ''free" Ni. The crystallisation mechanisms were observed depending on the chemical composition: for Ni < 9 at% two phases WC + $W_2C$ are formed and for higher Ni contents an initial precipitation of WC + Ni occurs.

In comparison to previous results obtained with the system W-C-Co, films deposited from W-C-Cr, W-C-Ni and W-C-Ti targets present adhesive failures and lower cohesive critical loads. The hardness of these films is lower than that of crystalline W-C-Co films and slightly higher than amorphous W-C-Co films. The adhesive critical load increases with increasing titanium content and with increasing negative substrate bias. Any influence of the chromium content "or of the substrate bias on the film properties cohesive and adhesive critical loads, as well as on the microhardness, could be found for W-C-Cr films.

The morphology of' the films sputtered from a pure tungsten target in a reactive atmosphere is more compact than that of the W-Ti-N films. No signs of nitrides could be detected in films of the W-N system, where nitrogen was found only in a solid solution. The reactivity of tungsten in the plasma environment is very low compared with that of titanium. W-Ti-N films present nitrides in their . structure at the same nitrogen/argon ratio levels as for the W-N system.

## 3.2. ADJUSTMENT OF THE DEPOSITION CONDITIONS OBTAINED IN THE LABORATORY EQUIPMENT TO THE PROTOTYPE

The concept for the prototype considered some special features:

- each sample to be treated can be rotated with constant speed
- the samples are preheated by means of electron bombardement
- the sputter cleaning process is acchieved very easily by means of inversion of the substrate tension
- during the deposition of films it is possible to variate the distance between cathode and substrate
- the ion beam source can also contribute to increase the ionization level, when working simultaneously with the magnetron cathode. '.

The deposition conditions were optimized concerning the "influence of the ultimate pressure on the coating properties and the influence of the pre-heating and ion etching parameters on the surface roughness and on the mechanical properties of the coatings.

T"he influence of the ultimate pressure on the coating properties was studied comparing the results of the hardness and scratch tests, obtained on films deposited after the deposition chamber have. been evacuated during different times. The results show that both hardness and scratch" test critical loads increase with decreasing ultimate pressure. In order to achieve reasonable results and simultaneously to satisfy the industry requirement of low evacuation time, the deposition was carried out at an ultimate pressure of $7.5*10^{-6}$ mbar.

It was observed that the surface roughness of the coatings varies as a function of the pre-heating and etching time. The substrate temperature shows an almost linear proportionality to the pre-heating time up to a temperature of about 500° C. The electron bombardment of the substrate during the pre-heating procedure has ". any influence on the substrate roughness. However, when the substrate was pre-heated, the final values of the substrate roughness were higher than those obtained for the same etching conditions but without previous substrate pre-heating. This leads to the conclusion, that the substrate temperature has an influence on the mobility of the surface atoms increasing the sputtering (etching) rate with increasing temperature.

The optimal value of the substrate roughness to acchieve the best mechanical properties is a mean value of 0,09 µm Rmz.

## 3.3. OPTIMIZATION OF THE TARGET CHEMICAL COMPOSITION

The influence of the element nitrogen and of the substrate bias were studied when depositing films from a W-Ti target with 10, 20 and 30 weight % titanium content.

'l'he study of thin films deposited from the target with 10 weight' % titanium showed that the substrate bias voltage during film deposition influences the properties of the W-Ti-N films. The increase of the substrate bias gives rise to dense films and decreases the nitrogen atomic content in the film. Concerning the mechanical properties, small variations were detected when changing the substrate bias.

The element nitrogen is very important. The morphology of the films is typically columnar not depending on the nitrogen flow rate. The film structures ranged from the tungsten phase up to, tungsten/titanium nitride phases. Increasing the nitrogen content of the film, increases as well its compactness. The conditions corresponding to the transition between structures without and with formation of nitrides present the best compromises between hardness and adhesion values.

Similar studies were carried out using reactive gas $CH_4$ and both $CH_4$ and $N_2$. Coatings containing carbon had higher hardness (4000 Vickers) but lower scratch-test critical loads.

The study of the films deposited from the target with 20 weight % titanium showed that the nitrogen content of these films is higher than bevor, for the same $N_2$/Ar flex rate. This is due to the higher reactivity of titanium towards nitrogen in comparision with tungsten. The results of the scratch-test show lower values for the cohesive and adhesive critical loads, as well as the hardness of the films (3200 Vickers).

Similarity as before, the study of the films deposited from the target with 30 weight % titanium showed that the increase of the titanium content in the target lead to a decrease of the values for the film hardness (2200 Vickers) and for the scratch-test critical loads.

The morphology of the films is similar with those deposited from targets with lower titanium content and the nitride phase could be as well detected for values over 25 % nitrogen content.

Concerning the deposition conditions it was confirmed that the deposition with the ion gun switch-cd on, this means with increased plasma ionisation, leads to better film properties.

Under consideration of the results obtained with the different W-Ti targets and once the user demands for TiAlN coatings were growing, the influence of the element aluminium in the target chemical composition was studied. For this purpose a sintered target with W-Ti-Al (75-20-5) weight % was used to deposit films on high speed steel "and cemented carbide tools.

For this study only $N_2$ was used as reactive gas. The hardness achieved values up to 2400 Vickers, the scratch-test critical loads obtained were excellent and no signs of adhesive failure were detected. The deposition conditions selected for coating the cutting tools gave rise to compact films presenting the nitride phase.

Once the target" materials used -in this project are very expensive in their production methods, tests were also carried out with a Ti-Cr (50/50%) target produced by means of vacuum plasma spraying. The study was limited to the analysis of the influence of the 'target production process on the deposition conditions and chemical composition of the deposited films.

During the deposition tests any plasma instabilities were observed on the target surface. Different $N_2$/Ar flow rates were used. The results concerning the chemical composition show that the plasma spraying process is an alternative to the sintering production method.

## 3.4. COATING CHARACTERIZATION

The chemical composition of the coating depend not only on the percentage of the respective elements 'involved in the deposition process, but also on other technological parameters, as the bias voltage; deposition temperature, total pressure in the vacuum chamber and deposition rate.

Comparing the results of the cutting tests with the values obtained for film hardness and scratch-test critical loads any relationship could be established.

The study of the wear behaviour of those coatings carried out with the pin-on-disc equipment can be resumed as follows:

- the high temperature wear resistance is "directly linked to the cutting tool endurance. life. The best results should correspond to the lower average depth of the wear scar
- The high temperature friction coefficient should be as low as possible, average value and stability
- The homogeneity of the wear scar at the end of the friction test

Some coatings could be optimized to fullfill the three previous criteria.

"The cutting tests under real conditions lead to results, which should be compared with those of the theoretical studies., Unfortunately any relationship was found, only one deposition test with a good high temperature friction coefficient achieved gut results.

The results of the fretting experiments seem to have a better relationship to the cutting tests under real conditions. The coatings with lower friction coefficient had the best results deposited on cemented carbide mills 'and working without lubricant (air cooling). The coated high speed steel taps did not show this result with evidence.

## 3.5. CHARACTERIZATION OF COATINGS DEPOSITED BY INDUSTRIAL PROCESS

The influence of the sample hardness on the measured value for the coating depends extremely on coating thickness and on the substrate hardness. This influence was studied for stainless steel, high speed steel and cemented carbide.

The study of the surface roughness was carried out for different initial values. The coating induces a lower surface roughness for the samples with higher roughness values. On the opposite, very smooth surfaces get higher roughness, when coated. This depends on the type of coating, as well as on the film deposition rate. A decrease of the deposition rate favorises the film growth mechanisms and increases the coating quality.

The morphology of W-Ti-N coatings deposited" when increasing substrate bias and comparative an industrial SILVER-TiN coating with high nitrogen atomic content in the film show that the film growth structure for SILVER-TiN is not evident, but for the W-Ti-N film a typical columnar growth can be observed.

Variations of the, deposition parameters in the industrial equipment were carried out for comparative purposes. The deposition parameters. were changed according to the working conditions, once the influence of the deposition rate and of the Argon partial pressure should be studied. The nitrogen flow rate was also changed when depositing SILVER-TiN coatings.

The results of these comparative studies showed that:

1) When changing the deposition rate from 2,0 up to 3,5 nm/sec or the Argon partial pressure from 1,3 Up to 2,2 x 10-3 mbar, any influence of the deposition rate could be detected, as well as of the Argon partial pressure, on the hardness and on the surface roughness of the GOLD-TiN coating

2) When changing the deposition rate from 1,2 Up to 11,0 nm/sec the hardness of the SILVER-TiN coating increases. At higher deposition rates there is a formation of hot spots, on the target surface, due specially to unpurities of the vaccum athmosphere.

3) The deposition of TiCN was *done* using CH4 and C2H2. When increasing the Argon partial pressure, the formation of arcs on the target surface can be avoided. For a pressure above 2,0 x 10-3 mbar the plasma can be kept stable during a long time (film thickness up to 5 microns). When decreasing the deposition rate, the plasma gets more stable and the hardness of the, coating increases.

## 3.6. DEVELOPMENT OF A MULTILAYER COATING SYSTEM

The development of a new coating based on a typical multilayer system similar to TiCN (titanium carbonitride) was carried out, once the results obtained with W-C/N-M coatings were not satisfying.

The influence of the most important technological parameters on the coating properties was studied, as well as the chemical composition and the structure of the different layers.

The chemical composition of the functional coating was controled by means of the reactive gas flow, which influence on the microhardness of the films was studied.

The influence' of the target load (deposition rate) on the microhardness is 'very significant for values under 20 W/cmZ'. Above this limit, the Vickers hardness keeps its value constant (2800 NV).

The substrate bias has any influence on the film growth, on the. surface' roughness and on the adherence of the coating for values above 80 and up to 150 Volt.

## 3.7. DEVELOPMENT OF NEW CHARACTERIZATION METHODS

Two tests were developed to try to get more information about film stresses and film strength, as well as to characterize the adhesion of the coating to the substrate:

### CST - CRACK STRENGTH TEST

This test can be described as a Bend-Break-Test of, brittle materials. For this purpose, a soft substrate material with low hardness is coated with a film of specified thickness. The surface is loaded by the diamond indenter of a Vickers microhardness measurement equipment. With increasing load, a limit value is reached which induces the film to break through into the substrate.

The development of the CST test and the definition of the test conditions were realized using aluminium alloy samples, as, substrate material, and multiple layer coatings were deposited with variable , thickness up to 50 µm for Silver-TiN.

### SST - SOFT SCRATCH TEST

Scratch testers use a diamond indenter and are used to measure a theoretical adhesion (the critical 'load Lc) quality of thin films. Any kind of relation can be established between the measured critical load values and the results of the lifetime tests with cutting tools,. as well as any kind of relation can be established between critical load and the measured hardness values.

The coating structure and hardness are important factors; which have a great influence on the critical load values obtained with the diamond scratch tester.

The method proposed to test the adhesion quality of thin films is very similar, but instead of a diamond indenter here a cemented carbide indenter is used.

The study of the working conditions when using the SST equipment was done, using high speed steel and cemented carbide substrates. The coating thickness was chosed at three levels, depositing approx. 3, 5 and 7 µm for single and multiple layer coatings.

The SST load range was defined at stages 0, 1, 2 and 3 (equivalent to 250, 500", 750 and 1000g) . The relativ movement of the cemented carbide indenter is carried out in two ways: in one way to obtain a surface pressure under an angle of 75° and in the opposite way to obtain shear forces under an angle of 15°.

## 3.8. WEAR CHARACTERIZATION AND GEOMETRY DEVELOPMENT

The wear behaviour of the cutting tools shows, as expected, that the critical regions are the cutting edges for both cemented carbide end mills and HSS taps. The friction surfaces of the cutting tools, which are in contact with the worked material during service, are also critical regions, as the 'free angle side of the cutting edge.

The most significant aspects to be considered for the geometry definition are certainly the creation of stable cutting edges, the optimization of the surface roughness and the increase of the substrate material strenght. The coating gives a higher protection to the cutting edge, increases the material strenght of the substrate surface and decreases the friction coefficient.

To calculate the exact profile of cemented carbide cutting tools to be developed or optimized, a software programme was used, which allows to calculate the optimal profile for the tool, to simulate a cutting tool 'and to design the profile" 'for the grinding wheel, with which the designed tool will be produced. Different types of drills were tested coated with standard coatings. This development showed the influence of the coating properties on the tool geometry.

The results-of the cutting tests with different PVD coatings and special surface treatments of high speed steel taps show the dependency of the tool wear behaviour from those parameters.

Other tests run to determine the torque by thread forming using taps from type FORMEX with different poligon forms and with an oxinitriding surface treatment. The results have shown that "with a flat poligon form the torque is higher than that of a pointed form. The lifetime for both k'inds of poligons was similar.

## 3.90 ANALYSIS OF THE EFFECT OF SUPPLEMENTARY COATING THICKNESS

Certainly there is a difference between HSS taps or formers on one side and cemented carbide drills and end mills on the other side.

With increasing coating thickness, the lifetime of the cutting tools increases proportionally. For any kind of tool there a thresold for the coating thickness, which should be estimated.

HSS taps are sensible to variations of the coating thickness for" films with higher roughness, but not as much as-by formers.

The development of heat on the contact surface during service leads to the disruption of the lubricant film. Under this working conditions, the properties of the coating can change, as well as the structure of the substrate material. The adherence of the coating to the substrate can be lost.

Cemented carbide tools need thicker coatings for better protection of the cutting edges. The limits for the coating thickness are different for drills and for end mills.

Drills can be coated with a film thickness higher then 3 µm and the 'lifetime still increases, depending on the worked material.

The common material used for cutting tests was heat treatable tool steel, for which the coating thickness should acchieve values up to 4,0 microns. For working general structural steel a coating thickness up to 5 microns can be used with very good lifetime. results. With increasing strentgh of the worked material the coating thickness must be decreased.

## 3.10. CUTTING TOOLS. WITH NEW GEOMETRIES' AND NEW COATINGS

Cemented carbide for tool production was obtained from different producers. The specifications were done corresponding to the type of tool to be tested considering the worked material.

A new cemented carbide quality for tool production was optimized and the study of the wear behaviour of coated tools was carried out.

The new cemented carbide type is CERMETS, which has other chemical and mechanical properties as usual standard materials, as better wear resistance, higher chemical stability, higher resistance against temperature differences and lower bend strenght.

With the purpose to use CERMETS for tool production it was necessary to adjust the tool geometry to the mechanical properties of that material. Tests of mill cutters with two, three, four and six flutes were carried out, leading to better lifetime of the tool compared with conventional cemented carbide.

New geometries for standard tools were used for further development. Thes'e tools were coated and tested, in such a way that the geometrical limits could be defined for those tools.

Coated tools with new geometries were produced and tested uncoated and coated with standard coatings, as well as with the new developed multilayer coating. Tests were carried out with coated cemented carbide drills and HSS taps and formers.

The optimization of the coating properties lead to deposition tests with standard end mills and new developed drills, as well as for new designed taps and formers. Mainly, an interlayer at the' beginning of the deposition process was introduced to increase the adherence of the coating to the substrate. Also switching on the ion gun during the deposition increases the quality of the coating.

The, best results were achieved by the multilayer coated tools for emulsion coolant and by W-C/N-Me coated tools for air coolant.

Working with coated HSS taps and formers, low torque values and high lifetime of the W-C/N-Me coated taps could be acchieved.

The coating thickness distribution depends on the deposition conditions, which can be optimized for that purpose, but the most important aspect is certainly the tool geometry.

Considering the tool geometry and the functional surfaces, where the wear is more significant, it is not possible to influence the coating thickness distribution by means of changing the tool geometry.

## 4. RESULTS AND CONCLUSIONS

The results acchieved with the new develcped W-C/N-Me coatings showed a continous positiv development considering the life-time of the cutting tools. The optimization' of the target chemical composition and of the respective deposition conditions lead to very significant results, specially those related with the cemented carbide end mills. These were tested under real cutting conditions and showed different wear behaviour when. using lubricant or only press air as coolant..

Coatings containing tungsten seem to have better high temperature wear resistance, also compared with some standard coatings. Under dry cutting conditions, the life-time could be increased up to 200 %, also when using the 'optimized SILVER-TiN and MULTILAYER coatings.

The best results were acchieved with coatings deposited from a W(70)-Ti(30) target. Nitride coatings acchieved the best results under dry cutting conditions and carbonitride coatings using emulsion as coolant and' lubricant.

Once more could be confirmed that with increasing titanium content the quality of the coating, increased similarily. On the other hand, the introduction of aluminium in the chemical composition of the target leads to a decrease of the coating quality.

The results acchieved with coated HSS taps and formers were not very promising. The higher life-time of the tools was acchieved with coatings deposited also from the W(70)-Ti(30) target. With nitride and carbonitride coatings some good results were acchieved. The life-" time was lower than that of the standard coatings and acchieved not more than 75 % of the reference values.

For high speed steel taps the cutting forces can be reduced. Resuming the results of the tests, means the surface treatments the clamp friction and tool wear can be reduced and the tap time life increased.

The quality of the development departments, concerning the characterization of hard coatings during service could be increased. Many lifetime tests were carried out with cutting tools, to create the standard basic data for industrial applications and for further developments of coatings.

The development of new characterization methodes for thin films was very useful and the results show very interesting relations to practical applications. The quantification of these parameters gave new impulses to the project. The pin-on-disc and fretting tests, as well as the crack strength and the soft scratch tests, can be used more often in the future.

The development of new tool geometries lead to better results of the lifetime with standard coatings. For these purposes a new multilayer coating was developed and deposited on cemented carbide end mills and drills, which can be commercialized.

The development of W-C/N-Me coatings lead to the optimization of the chemical composition of the target. The films deposited from the target W,Ti have very good adhesion and cohesion properties, high hardness and low friction coefficient values.

With the new multilayer coating following advantages could be acchieved:

- higher strength than the other coatings, which allows to use it "for cutting tools working under interrupted cutting conditions

- higher temperature resistance than the standard Gold-TiN coating, i.e. higher cutting speed and feed rate are possible

- higher abrasive wear resistance than the standard TiAlN coating .

- lower affinity to titanium, nickel or brass,