Microstructural, Mechanical and Tribological Characterisation of CVD and PVD Coatings for Metal Cutting Applications

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Abstract

The present thesis focuses on characterisation of microstructure and the resulting mechanical and tribological properties of CVD and PVD coatings used in metal cutting applications. These thin and hard coatings are designed to improve the tribological performance of cutting tools which in metal cutting operations may result in improved cutting performance, lower energy consumption, lower production costs and lower impact on the environment. In order to increase the understanding of the tribological behaviour of the coating systems a number of friction and wear tests have been performed and evaluated by post-test microscopy and surface analysis. Much of the work has focused on coating cohesive and adhesive strength, surface fatigue resistance, abrasive wear resistance and friction and wear behaviour under sliding contact and metal cutting conditions.

The results show that the CVD deposition of accurate crystallographic phases, e.g. α-Al₂O₃ rather than κ-Al₂O₃, textures and multilayer structures can increase the wear resistance of Al₂O₃. However, the characteristics of the interfaces, e.g. topography as well as interfacial porosity, have a strong impact on coating adhesion and consequently on the resulting properties. Through the deposition of well designed bonding and template layer structures the above problems may be eliminated.

Also, the presence of macro-particles in PVD coatings may have a significant impact on the interfacial adhesive strength, increasing the tendency to coating spalling and lowering the surface fatigue resistance, as well as increasing the friction in sliding contacts.

Finally, the CVD-Al₂O₃ coating topography influences the contact conditions in sliding as well as in metal cutting.

In summary, the work illuminates the importance of understanding the relationships between deposition process parameters, composition and microstructure, resulting properties and tribological performance of CVD and PVD coatings and how this knowledge can be used to develop the coating materials of tomorrow.

Keywords: Tribology, Friction, Wear, Coatings, CVD, PVD, Alumina, Metal cutting

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1. Introduction

Metal cutting is one of the oldest and most common processes for shaping components in the manufacturing industry and involves methods such as turning, drilling and milling. The equality of these processes is that material removal is obtained by formation of a chip. Even though metal cutting stands for about 15 % of the value of all mechanical components manufactured worldwide metal cutting is one of the least understood manufacturing operations. The main reason for this is the complexity of the process resulting in extreme tribological conditions involving high contact pressures and temperatures.

The word tribology refers to the science of friction, wear and lubrication and is of great importance when surfaces are interacting in a relative motion, i.e. in everyday physical movement. Friction appears due to resistance of motion when a surface moves over another surface and results in wear of one or both surfaces. The friction is not a material parameter but a system parameter depending on the properties and surface topography of the two materials in contact, temperature, pressure, sliding velocity, atmosphere, etc. In the same way the wear, i.e. material removal, of the materials in contact will be a system parameter.

Everywhere, there are surfaces sliding against each other in enormous variations in type of everyday contact. It can be an ice skate against ice, shoe against asphalt, car breaks, hinges to a door, bearings in a bicycle wheel etc etc. All contacts give different tribological conditions but what they have in common is the extreme interaction in the very contact points. On a micro- and nanoscale the material will be extremely deformed and parts of the material will even melt, also in the smoothest contacts. The more severe the contact is the more severe the surface interaction will be. These phenomena are therefore very pronounced in an extreme contact such as the one present in turning. In this kind of contact a small cutting tool is used to shape a hard lump of steel into a well defined product and all deformation and hence the energy transformation will take place in just a few mm² at the time. Unarguable, the tribological conditions will be aggressive and result in high temperatures and pressures which put high requirements on the cutting tool in use. The tool will continuously be in contact with new and reactive work-piece material and the contact is featured by pronounced chemical dissolution, shear deformation and scratching.
In metal cutting cemented carbide with a large amount of hard carbides and a metallic binder is often used as cutting tool substrate material. By subsequent depositing of coatings such as Al₂O₃, Ti(C,N) or TiSiN by chemical vapour deposition (CVD) or physical vapour deposition (PVD) on the substrate the tribological characteristics can be significantly improved.

Accordingly, by using a thin coating of sub-microns in thickness of a wear resistant material the best properties concerning friction and wear will be located where they are most needed, while the substrate acts as a load carrier. The substrate should be able to resist mechanical fracture failure as well as deformation of the tool geometry while the coating should be designed to resist the surface deterioration. Further, when depositing a coating an interface is introduced and its strength, i.e. the adhesion, is of great importance to avoid spalling of the coating. In pure adhesion the interfacial atomic binding forces are of importance but also the potential of the coating system to decrease the shear stresses in the interface has a large influence. To gather the mechanical and tribological requirements and thus the tools operational functionality the coating system, i.e. substrate, interface and coating, have to obtain an appropriate combination of properties. Hence, the substrate may have a high strength and toughness while the coating has to be hard, chemically stable and wear resistant. Also thermal expansion, shear strength as well as elasticity of the components matter. In order to increase the interfacial strength all the above mentioned properties have to be optimised. These properties are dependent of many factors such as chemical and phase composition, coating architecture, thickness, texture, microstructure, porosity, defect density, residual stress state as well as surface topography of the coating. In turn these coating characteristics are controlled by different deposition process parameters, e.g. temperature, pressure and gas flow, and the understanding of the relationship between process parameters, coating characteristics, properties and finally tribological characteristics is needed in order to develop the coating materials of tomorrow.
Aim of the thesis

The aim of this thesis is to increase the understanding of the relationships between composition, microstructure, resulting properties and tribological performance of CVD and PVD coatings aimed for metal cutting applications. The tribological performance of several coating systems have been characterised by a number of friction and wear laboratory tests and evaluated by post-test microscopy and surface analysis. Much of the work has focused on properties such as coating cohesive and adhesive strength, surface fatigue resistance, abrasive wear resistance and friction and wear behaviour under sliding contact conditions and how these are influenced by coating microstructure. Also, the tribological mechanisms being active in metal cutting (turning) have been investigated and correlated to the corresponding mechanisms being active in sliding tribological contact (pin-on-disc testing). The knowledge obtained is intended to provide a knowledge base and new insights for the future work of designing new CVD and PVD coatings with improved tribological performance.


2. Tribology

Tribology is the science of solid surfaces in contact in relative motion and is an old knowledge of great importance regarding to everything in movement [1]. As a scientific discipline tribology is rather new and it is commonly known as the study of friction, wear and lubrication. To the nature it is a complex science with small possibilities to do theoretical calculations of the friction and wear. Hence, tribology is strongly associated with practical applications which makes elaborative work and empirically experience very valuable. The tribological properties are of utmost importance for the materials in contact and the system is very sensitive to the operating conditions and the environment. To understand the tribological behaviour knowledge in physics, chemistry, metallurgy as well as mechanics is necessary which makes the science interdisciplinary and notable open minded. However, by optimising the friction and wear in technological applications, such as in machine components or in metal working systems, both the environment and the economical costs could be saved.

2.2 Friction

Friction can be defined as the resistance to movement of a body against another and is of great importance for coated tools used in cutting operations. The friction is not a material property but a system response in the form of a reaction force. Generally the law of friction, known as Amontons-Coulomb Law see eq. (2.1), describes the friction coefficient as the relationship between the tangential force $F_T$ (frictional force) and the normal force $F_N$ (load).

$$\mu = \frac{F_T}{F_N} \quad (2.1)$$

This law is assumed to be accurate in tribological contacts with ordinary contacts pressures, as most of the contacts around are, and is often referred to as Coulomb friction. Further, during the contact generally the friction is composed of two different components, see eq. (2.2), i.e. the adhesive component ($\mu_a$) and the ploughing component ($\mu_p$).
\[ \mu = \mu_a + \mu_p \]  

(2.2)

The adhesive component is related to the materials in contact and controlled by an adhesive force acting at the areas of real contact, i.e. the areas in contact formed by asperities at the surfaces. The adhesive force originates from the force required to breaking the inter-surface bonds when the surfaces are sliding against each other. Hence, the adhesion of the two solids in contact is important and is dependent of the chemistry of the tribo surfaces in the sliding interface.

The ploughing component originates from the deformation force acting during the ploughing of the softest material in contact by the surface asperities of the harder material and is related to the surface topography of the surfaces in contact. Also, attached wear particles in the interface will act in a ploughing way.

One additional part to the ploughing component is the asperity deformation which is related to the deformation of the asperities in microscale, i.e. a scale lower than the micro-ploughing scale [1-2].

2.3 Wear mechanisms

In tribological contacts wear occurs due to the interaction between the two surfaces in contact and implies gradual removal of the surface materials, i.e. material loss. The wear mechanisms of importance regarding to coatings and metal cutting are abrasive, adhesive and tribo chemical wear. Typical more than one wear mechanism is acting at the same time.

An interrelationship exists between friction and wear, often a low friction results in low wear. Although, this is not a general rule and there is a lot of examples showing a high wear rate in spite of a low friction [1-3].

2.3.1 Adhesive wear

Adhesive wear has its origin in the shearing contact between the asperities of solid counter surfaces. In a microscopic point of view solid surfaces almost never are perfectly smooth; the surfaces are rather constituted of asperities of different sizes and shapes. During sliding elastic and/or plastic deformation of the asperities occur resulting in a contact area where the binding forces give a strong adherence and the surfaces will be welded together. The adhesive wear occurs when the tangential relative motion will cause a separation in the bulk of the asperities in the softer material instead of in the interface and hence material is removed, see Fig 1 [1-3].
The real contact area is constituted of all the areas of welded asperities at the surfaces and during sliding the material removal results in a wear that can be measured as a volume or weight decrease. However, it is more usual to present the wear in a wear rate or in a wear coefficient. In present thesis the wear rate is defined as the wear volume per sliding distance and load, and has the unit $\mu m^3/(Nm)$. The load, sliding distance and hardness of the softest material and also the probability to get a wear particle when a contact weld is broken are parameters that will control the adhesive wear of a material in a sliding contact [1-3].

2.3.2 Abrasive wear

Abrasive wear provides significant plastic deformation of the surface material and occurs when one of the surfaces in contact is significantly harder as compared to the other, or when harder particles are introduced in the tribo-system. Often a distinction is made between two- and three body abrasion where the latter refers to situations where hard particles are introduced in the interface, see Fig 2. However, the sharp and hard asperities or particles are pressed into the softer surface which results in a plastic flow of the softer material around the harder. Due to the tangential movement the harder surface will scratch the softer by the ploughing action which results in wear and remaining scratches or grooves. The abrasive wear can further be classified in different wear mechanisms: micro cutting, micro fatigue and micro chipping [1-3].
2.3.3 Tribo chemical wear

In tribo chemical wear the wear process is dominating by chemical reactions in the contact that are consuming the material. Here the environmental conditions in combination with mechanical contact mechanisms are of great importance. Hence, the chemical action, such as diffusion or solution, is always in combination and interaction with other wear mechanisms and is not a wear mechanism alone. It is more correct to talk about different mechanical wear mechanisms and consider the chemical effects as an additional influence parameter which will change the material properties of the surface in contact [2].

2.4 Tribo film formation

The high local temperatures and pressures obtained in the surface contact when two bodies are sliding against each other results in local shear deformation and fracture of the surfaces. The high local temperatures may accelerate chemical reactions or melt the surfaces locally and wear is hard to avoid. However, these conditions do not necessarily have to only be destructive for the surfaces but may also make it possible to form tribo films with new tribological properties. Usually tribofilms are divided into two groups: “Transformation type tribofilms” and “Deposition type tribofilm” and both are changing the surface topography, chemistry and mechanical properties. In the formation of “Transformation type tribofilms” transformation of the original surfaces is obtained by plastic deformation, phase transformation,
diffusion etc without any material transfer. On the contrary the formation of “Deposition type tribofilm” is obtained only by material transfer, i.e. by molecules fed from the counter surface, the environment or by wear debris [4]. Accordingly, the chemical reactivity, the chemical adherence and the surface topography may influence the formation of a tribofilm.
3. Tribology in metal cutting

Metal cutting has been and still is the major shaping process which is used in the production of engineering components. The tribological contact in cutting differs substantially from general sliding and involves severe tribological conditions with high temperatures and pressures which put high requirements on the cutting tool, also called cutting insert. The tool shall make it possible to get high productivity, excellent tolerances of the shaped geometries and surface finish as well as possibilities to shape tough workpiece materials. These goals may be reached by using wear resistant cutting tools with long lifetime and maintained micro geometry. Further, a low friction between the workpiece and the tool is beneficial since the cutting forces and hence the energy consumption as well as the load on the machine setup should be lower. Consequently, the tribological characteristics have a remarkable influence on the productivity and manufacturing possibilities in metal cutting.

3.1 Cutting process

The cutting process is characterised by that the shaping of a workpiece material is performed by formation of a chip. A common setup in turning can be seen in Fig 3. The cutting depth, \( a_p \), and the geometrical placement of the tool result in a width of the chip (\( b_1 \)). Further, the feed, \( f \), will give the theoretical thickness (\( h_1 \)) of the chip and together with the width the volume of the workpiece removal per every single reverse is obtained. The cutting speed, \( v_c \), gives the speed of the material removal. The cutting depth, feed and cutting speed are important parameters and result together with the tool geometry in certain tribological conditions [5].
3.2 Chip formation

The chip formation is in detail described in Fig 4, which shows the most important parameters and the three shearing zones (E_I - E_III). During the relative motion, $v_c$, between the workpiece material and the cutting tool the workpiece material is heavily deformed along the shear plane.

The workpiece materials can differ a lot in chemical composition and properties such as hardness, ductility and deformation hardening tendency and hence the machinability will vary. The shape of the chips varies from fragmented chips (e.g. in cast iron) to long continuous chips (in ductile metals) and thus the workpiece material, but also the tool material and geometry as well as the cutting data, control the chip formation.
The deformation of the workpiece material is divided into three different shear zones (E₁ - E₃) where all the energy consumption occurs. Along the shear plane the primary shear zone (E₁) is located and this is the area where the chip is formed. Hence, the deformation rate is very high. In the primary zone the stagnation point is located at the edge radius of the tool and will be a dead zone where the material is not moved. Here it is determined if the material will be deformed and moving to the chip or be left in the workpiece material. Hence, the location of the stagnation point is important and controls the chip flow and the tribological conditions. Also, at this point the probability to form a built-up edge is quite high, i.e. adhesion of workpiece material to the edge radius results in a different micro geometry of the tool. However, of the heat generated in the contact in metal cutting about 80 % originates from the mechanical deformation of the chip and the temperature will be high in this zone. Further, the chip stands for 75 % of the heat removal.

In the secondary shear zone (E₂) the highest temperatures and pressures are obtained, often over 1000°C and 2-3 GPa respectively. About 18 % of the generated heat has its origin in the contact between the heavily deformed chip and the rake face and 20 % of the total heat is conducted through the tool. The contact at the rake face is complex and results in shear and normal pressures which vary a lot over the surface.

The tertiary shear zone (E₃) is in the outermost surface layer of the workpiece material which is in contact to the edge radius and the flank face. Here the deformation rate is lower compared to the other zones and the temperatures are often about 500-600°C. The workpiece material absorbs about 5 % of the generated heat.

In summary, the material removal due to formation of a chip results in severe tribological characteristics that will give high temperatures and pressures which due to the complexity of the process will vary a lot over the tool [2, 5-6].

3.3 Friction

The pronounced variation and high levels of pressures and temperatures in metal cutting result in complex tribological conditions with a friction that will vary over the tool surface.

The contact between the workpiece material and the cutting tool can be divided into three different zones according to Höglund [7] among others, see Figure 5. In subzone A sticking between the two surfaces is obtained and no sliding between the cutting tool and workpiece material occurs. In subzone C only sliding contact occurs between the two surfaces and here the temperature and pressure are lower. Subzone B is a transition zone between subzone A and C where both sliding and adhesion between the workpiece
and cutting tool occur. Often the contact condition primarily is calculated for subzone C and it should also be assumed that the contact condition acting in subzone B is related to the acting in subzone C. For simplicity reasons it is common to only consider sliding contact [8], sticking contact [9] or a fixed combination of these phenomena [10].

![Figure 5. The three subzones A, B and C, divided by Höglund, in the contact between the workpiece material and the cutting tool [7].](image)

In tribological contacts the real area in contact is much smaller than the apparent area due to irregularities at the surface. Though, in metal cutting the high pressures result in heavily loaded sliding contacts which change the relationship between the areas. The real area will increase with increasing loading and at high enough loads it will reach the apparent area of contact. This results in that the Coulomb friction is usually not accurate [6, 11].

However, there are different methods to calculate the friction in metal cutting and not seldom it is assumed that Coulomb friction is accurate enough, i.e. the mean value of the contact conditions is calculated as a quota between a shear stress and a normal stress operating at the flank or rake face. In order to obtain tangential and normal forces that can be used in the calculations often so called plunging operations, where the tool feed is in the radial direction of the workpiece, can be performed. Here it is important to separate the load contributions at the rake and flank face respectively and the cutting forces of interest are the tangential force ($F_t$) and the radial force ($F_r$). When calculating the contact condition at the flank face the input parameters usually are the cutting forces ($F_c$ and $F_t$) when the chip thickness is going toward zero and these are obtained by extrapolating the forces at low feeds. In this scenario the frictional force and the normal force corresponds to $F_c$ and $F_t$ respectively at the flank face. On the contrary, at the rake face usually the cutting forces ($F_c$ and $F_t$), when the chip thickness is large, are used as input parameters. In this scenario the cutting forces obtained when the chip thickness is going toward zero is subtracted from the measured forces in order to
minimise the influence from the flank face. Here the frictional force and the normal force correspond to \( F_r \) and \( F_c \) respectively [2, 5-6, 13].

However, in order to get more accurate values of the contact conditions the localisation of the stagnation point and the contribution from the cutting edge have to be considered. One common method is to use the chip compression ratio and the rake angle to do calculations of the contact condition at the rake face and hence the stagnation point is taken into consideration [12].

### 3.4 Wear of cutting tools

The complexity of the tribological conditions in metal cutting also will influence the wear of the cutting tools. The surface of the tool will continuously come in contact with virgin and reactive workpiece material and the very contact is unaffected of the environment, i.e. the tribo-system is open and there is no oxidation of the very surfaces in contact. The tribological conditions can be seen as a combination of mechanical and thermal loading including chemical and physical processes.

The most common wear mechanisms obtained at the cutting tool during the machining are adhesive wear, abrasive wear and wear due to chemical instability (such as diffusion and solution). Depending on coating material and cutting data the dominating wear mechanisms will vary regarding to the area of the tool in contact.

#### 3.4.1 Microscopic wear mechanisms

**Adhesive wear**

Adhesive wear will be a consequence of formation of welded asperity junctions between the workpiece material and the coated cutting tool. The chemical reactivity of the materials in contact is of great importance. The shearing forces result in fracture of the junction and small fragments of the coating will be adhered at the chip or the workpiece. Shearing failure of the adhesive contact bridge of the asperities is often related to metallic materials but is obtained for all materials that are possible to deform plastically - also ceramics. Especially in metal cutting this will be seen and extremely shear deformation of the coating material can occur before material removal, i.e. superficial plastic deformation resulting in ridge formation. The wear mechanism is most common in zone B at the rake face where the temperatures are high [2].
**Abrasive wear**

Abrasive wear is obtained due to the abrasive action of hard particles in the work material and results in micro cutting, micro fatigue and micro chipping of the tool. Dependent on the work material, particles of different phases are obtained and often there are particles harder than the workpiece or coating material. Also hard particles can consist of highly strain hardened fragments from an unstable built-up-edge. Especially at the flank face, where the temperatures are somewhat lower, abrasive wear will dominate but can also occur in zone C at the rake face where sliding is common. The coating hardness is of great importance regarding to abrasive wear resistance.

Often the abrasive wear mechanisms, i.e. micro cutting, micro fatigue and micro chipping, are mixed together. In micro cutting the abrasive element is cutting a chip and in the refined case the entire scratch volume is removed. Also micro ploughing without material removal is common in abrasive contacts and results only in a plastic rearrangement of the surface material giving ridges at the sides of the scratch, and hence no wear. However, in micro ploughing the ridges and the scratch surface are heavily sheared and in a repeated contact these areas will be more brittle. Voids in the coating material or dislocation pile-ups may form the nuclei for the first crack to occur and the probability for crack initiation will increase in the repeated contact resulting in micro fatigue. Delamination wear is a kind of fatigue wear where cracks are nucleated below the surface and will propagate parallel to the surface resulting in delamination of long and thin wear sheets. Also spalling of entire coating fragments may occur. In some cases, especially when the bulk material is brittle (as ceramic coatings) the abrasive contact rapidly will result in significant crack initiation and propagation which will give micro chipping [2].

**Diffusion and solution**

In combination with different mechanical wear mechanisms the chemical effect on the surface in contact has a detrimental influence on the wear.

At high temperatures as obtained in metal cutting the wear can be accelerated due to chemical instabilities of the materials. The high temperatures and pressures result in partly molten material in the tribological contact and solution wear occurs. Also, diffusion wear plays an important role and is characterised as material loss by diffusion of tool material into the workpiece material.

When the coated tool is in contact with oxygen a thin layer of oxide is formed on the top of the surface. The formed oxide is often porous and has poor adhesion resulting in easy removal in contact with e.g. abrasive particles. The exposed surface will then continue to oxidise and a high wear rate is obtained [2].
3.4.2 Macroscopic failures

The dominating wear mechanisms varies over the tool and result in different macroscopic wear failures. The most important may be classified according to Figure 6 in:

- crater wear
- flank wear
- cutting edge notch wear

![Figure 6. Classification of a worn cutting tool showing crater wear, flank wear and notch wear. CFD stands for Chip Flow Direction.](image)

The crater wear is obtained at the rake face of the tool in form of a crater or a groove which will reduce the load bearing capacity. It is placed where the chip moves over the tool surface and typically 0.2-0.5 mm from the cutting edge in zone B and C. Especially when cutting materials with high melting point and at high cutting speeds the crater wear is dominating. Primarily the crater wear is caused by dissolution or diffusion of the tool material since it occurs in regions of maximum temperature rise. This is obtained in combination with adhesive and abrasive wear.

The flank wear occurs on the flank face of the tool, where the temperatures are lower, and is often controlling the tool lifetime. It is believed to be caused mainly by abrasive wear of the coated tool by hard particles, but also adhesive wear may be present.

Cutting edge notch wear is caused by oxidation in combination with abrasive and adhesive wear of the tool surface at the edges of the contact to the workpiece material.

The wear will deteriorate the performance and lifetime of the tool in use but there are also other limiting factors such as thermal and mechanical induced cracking, chipping of the cutting edge, fracture or plastic deformation. Plastic deformation is obtained due to thermal and mechanical loading and gives a change of the tool geometry and moves the edge line downwards.
The plastic deformation is often compensating the flank wear. Chipping of the cutting edge involves local fractures of smaller fragments at the edge line and may result in a poor surface finish of the shaped workpiece. Fracture of larger fragments may result in catastrophic failure and the tool will be unusable [2, 5, 15-16].
4. Design of coated cutting tool materials

4.1 Optimisation of substrate and coating properties

Most failures originate from the surface, either by wear, fatigue or oxidation. Hence, a lifetime cost and performance strongly can be improved when locating specific properties where they are most needed. In order to control friction and wear in different technical application one way is to utilize coatings, typically 0.1-10 µm in thickness. While the substrate material can be designed for strength and toughness to bear the load, the coating will represent the resistance to wear, oxidation and thermal loads which influence the friction characteristics. To gather the tribological requirements the coating/substrate system, i.e. substrate and coatings, have to obtain an appropriate combination of properties such as hardness, elasticity, shear strength, fracture toughness, thermal expansion and adhesion. Figure 7 illustrates four zones in the coating/substrate system with different properties that have to be considered. Both the substrate and the coating properties are determined by the chemical composition, microstructure as well as the porosity and homogeneity of the material. The connection between the two materials consists of the interface where the adhesion and shear strength is very important. In the surface where real contact is performed the chemical reactivity, and the roughness as well as the shear strength have to be considered. Always, there is a compromise in properties of the coating systems due that a good property in one part of the system will be a poor property in another part. For example, it is difficult to obtain both good adhesion in the interface and no surface interaction at the surface or both high hardness and high toughness of the coating. However, it is possible to optimise the properties in relation to each other [2-3].
As substrate material cemented carbide is commonly used due to its unique combination of strength, hardness and toughness. Usually it consists of hard carbide (or nitride) particles sintered together with a rather ductile metallic binder. By combining the hardness of the carbides and the ductility of the binder unique composite properties will be obtained and high resistance to wear, deformation, fracture, corrosion and oxidation will be achieved.

The dominating techniques to obtain hard coatings for cutting tools are chemical vapour deposition (CVD) and physical vapour deposition (PVD). Commonly used coating materials are e.g. CVD deposited Al₂O₃, Ti(C,N), TiC and TiN, and PVD deposited TiAlN, TiSiN, AlCrO and VN. The most important coating properties that the above mentioned coatings have in common are first of all a high hardness which will reduce the abrasive and adhesive wear. Also, the chemical inertness is high and hence diffusion and oxidation wear are limited. In particular the crater wear is affected of the chemical inertness and it is beneficial to use coatings such as Al₂O₃ in this area. The flank wear is reduced by using hard coatings such as Ti(C,N). Further, a low affinity to iron based materials results in low adherence of work-piece material. By optimising these properties of the coating low wear and friction may be obtained. This will result in e.g. lower cutting forces and hence lower energy consumption, a tool with longer lifetime and less need of harmful lubricants.

It should also be mentioned that an increase of coating thickness would not necessarily result in improvement of the tool life time, but in a decrease
of the toughness. The brittle coating acts as a crack initiator with a reduction of strength as a consequence.

However, cutting tools were the first successful commercial tribological application with thin surface coatings and still coatings, in combination with the substrate, are of great importance in order to get tools with beneficial properties for metal cutting [2].

4.2 Adhesion and interfacial fracture toughness

When using coated tools an interface is introduced and its strength, i.e. the adhesion, is of great importance. The adhesion is the ability for the coating to remain attached to the substrate and if the adhesion is inadequate the operational functionality of the surface will be lost. Also, the potential of the coating system to decrease the shear stresses in the interface have a large influence on the adhesion and hence the term adhesion is considerably complex. Cracking due to debonding in the interface is depending on local load levels and directions, resulting stress condition, strain and deformation [2]. Hence, in the coating system design it is important to consider the interaction between substrate and coating.

In pure adhesion the chemical reactivity, i.e. the atomic binding forces are of importance. Hence, the type of bonding results in different interfacial binding forces, see Fig 8 [17], which may consist of valence forces or interlocking forces or both [18]. When breaking the bonds interfacial cracks occur and thus the term interfacial fracture toughness is further describing the phenomenon.

![Figure 8. Binding energies of different bondings [17].](attachment:image.png)

In order to evaluate the adhesion different test methods have been developed during the years. Different pull-off tests, indentation tests and laser
techniques are commonly used but the most widespread technique is the scratch test. A tribological contact implicates locally a lot of different contact conditions and even though the scratch test do not simulate all of them it tells a lot about the coating system in contact. In scratch test as well as in other tribological contacts the mechanical properties of and the relationship between the coating and substrate have high importance. Such properties in particular are the hardness (H), Young’s modulus (E) and fracture toughness (Kc).

When the coating is deformed with the surface of the substrate the stresses in the interface or in the coating may be higher than the tensile or the shear strength of the material resulting in crack nucleation and propagation. This results in interfacial spalling, i.e. spalling of discrete coating fragments. In a repeated contact also the ideas of delamination can be referred to the micro spalling.

It has been shown that a low friction coefficient and a high Young’s modulus decrease the risk to get interfacial spalling [19-20]. In other words, a coating that can deflect with the substrate or resist deformation, i.e. accommodate substrate deformation without failure, under load will exhibit a lower tendency to spalling [19-21]. Also, high substrate hardness will increase the resistance to deflection.

 Often the relation H/E of the coating is used to describe the elastic limit of the strain, i.e. the amount by which the coating can be stretched before permanent deformation occurs. This will give an indication of the ability of the coating to deform with the substrate under load without yielding [22]. Also, the relation E/H or (E/H)^{1/2} is often used to evaluate the amount of plastic deformation that occurs in the coating and the substrate [23-24]. Further, residual stresses, i.e. stresses that remain in the surface after deposition, influence in spalling. Normally compressive stresses inhibit crack initiation and growth while tensile stresses promote cracking. But, high compressive stresses may also have a negative effect in spalling in connection to edges, corners or irregularities at the surface due to the resulting lifting force. However, the possibility of the coating to deflect with the substrate without failure is also dependent of the mismatch between the mechanical properties of the coating and substrate respectively where H, E and their internal relationship (H/E, E/H etc) do matter in variable extension dependent of the characteristics of the tribological contact. Both the cohesive and adhesive strength of the coating and substrate materials have a high influence on the interfacial fracture toughness [23-24].
4.3 Substrate material—cemented carbide

Cemented carbide usually consists of hard carbide (or nitride) particles sintered together with a ductile metallic binder which will fill all cavities. The content of carbides and binder control the properties such as toughness and generally the amount of the carbide phase is 70-97 wt% and the grain size averages varies between 0.5 and 10 µm.

The basic cemented carbide structure from which other types of cemented carbide has been developed is composed by tungsten carbide (WC), the hard phase, together with cobalt (Co) the binder phase. In addition to the tungsten carbide and cobalt composition cemented carbide may contain complex carbides, such as TiC, TaC or NbC, in varying proportions or binder phases of other metals such as Fe, Cr, Ni or Mo which can replace Co completely. However, the most common used cemented carbide structures contain about 90 – 94 wt% carbides and the remaining part is binder phase.

Sintered cemented carbide has usually a hardness of HV0.5 1300 – HV0.5 2200 and high strength in compression, often in the range 3000 to 10000 MPa [25].

4.4 Coating design and deposition

4.4.1 CVD coatings

Chemical vapour deposition (CVD) implies production of a solid deposit (coating) by chemical reactions between process gases. Today the CVD technique is widely used to get coating materials in a wide range of applications such as wear resistant coatings, thin film semiconductor devices or as biocompatible coatings. Different CVD techniques are in use which can initiate chemical reactions by heat, photons or electrons and plasma. In cutting tool applications thermally activated processes are most used and are often performed in hot wall CVD reactors which make it possible to coat thousands of cutting tools. In present thesis the CVD coatings investigated are deposited by a hot wall CVD reactor where the heat is generated by furnace elements in the reactor walls [2, 26-28].

Normally the deposition rate is in the range 0.1-10 µm/h and the temperatures and pressures are in the range 800-1200°C and from atmospheric (≈1 bar) to 10^{-2} mbar respectively. Other parameters optimised to get a high-quality coating are chemical concentrations, velocity of gas flows, geometry of the gas inlet and geometry of the substrate. The strength of the technique
is the ability to produce well-adhered, uniform and dense surface layers where grain orientation and size, coating composition and properties can be varied by selection of appropriate process parameters. As substrate cemented carbide often is used since it is not temperature sensitive which simplifies the condensation and chemical reactions on the surface. The CVD reactions that result in the growth of the coating are controlled by the thermodynamics that determine the driving forces and kinetics. Adsorption, diffusion and chemical reactions on the surface result in nucleation and growth of the coating.

CVD coatings are known to have intrinsic tensile residual stresses obtained when the tools cool down after deposition due to the differences in thermal conductivity between the substrate and the coating. That is also the reason for formation of thermal cracks, which are commonly obtained. These characteristics will be disadvantages in some applications such as intermittent cutting (milling) but in continuously cutting (turning) involving higher and more stable temperatures and loads CVD coatings are showing an excellent wear resistance. Also, the CVD process makes it easy to get a uniform coating over rather complex substrate geometries [2, 26-28].

Alumina

Alumina exists in a number of different crystallographic phases and in CVD coatings used in metal cutting the most common are the $\alpha$-$\text{Al}_2\text{O}_3$ and $\kappa$-$\text{Al}_2\text{O}_3$. The only stable phase is $\alpha$-$\text{Al}_2\text{O}_3$ while the others are metastable.

$\alpha$-$\text{Al}_2\text{O}_3$

$\alpha$-$\text{Al}_2\text{O}_3$ has properties well suited for cutting operations, e.g. chemical stability, high hardness, high melting point and good insulating properties. Especially at higher temperature the chemical stability of $\alpha$-$\text{Al}_2\text{O}_3$ is outstanding. The $\alpha$-$\text{Al}_2\text{O}_3$ is well known as corundum and the crystal structure is trigonal.

Generally, the $\alpha$-$\text{Al}_2\text{O}_3$ shows a better performance in cutting as compared to $\kappa$-$\text{Al}_2\text{O}_3$ but is more difficult to grow in the CVD process. CVD grown $\alpha$-$\text{Al}_2\text{O}_3$ shows columnar grained microstructure with grain sizes of several microns and the nucleation is believed to be beneficial on different titanium oxides [29-37]. However, the nucleation of $\alpha$-$\text{Al}_2\text{O}_3$ is poorly understood.

$\kappa$-$\text{Al}_2\text{O}_3$

As $\alpha$-$\text{Al}_2\text{O}_3$ also the $\kappa$-$\text{Al}_2\text{O}_3$ is used in metal cutting. The structure is orthorhombic and the microstructure is columnar with preferred growth along the $c$-axis. The grains are often smaller as compared to $\alpha$-$\text{Al}_2\text{O}_3$ and the width of the columns is less than 1 µm. The main drawback is that $\kappa$-$\text{Al}_2\text{O}_3$ is metastable and will transform to $\alpha$-$\text{Al}_2\text{O}_3$ at high temperatures (1000°C) as will be reach both in the CVD process and in the cutting process. This transformation results in a volume contraction of about 8% resulting in secon-
CVD of alumina

CVD alumina layers are commonly deposited from a gas mixture of AlCl₃, H₂, CO₂, CO, and HCl and are based on the hydrolysis reaction of AlCl₃ by water vapour:

\[ 2\text{AlCl}_3(g) + 3\text{H}_2\text{O}(g) \leftrightarrow \text{Al}_2\text{O}_3(s) + 6\text{HCl}(g) \quad (4.1) \]

The water is produced by high temperature reduction of CO₂ by H₂:

\[ 3\text{H}_2(g) + 3\text{CO}_2(g) \rightarrow 3\text{H}_2\text{O}(g) + 3\text{CO}(g) \quad (4.2) \]

Hence, the overall reaction can be described as:

\[ 2\text{AlCl}_3(g) + 3\text{CO}_2(g) + 3\text{H}_2(g) \leftrightarrow \text{Al}_2\text{O}_3(s) + 6\text{HCl}(g) + 3\text{CO}(g) \quad (4.3) \]

The rate-determining step in the deposition process is the formation of water vapour. In CVD it is important that the growth rate will be high at low temperatures resulting in dense and hard coatings with a uniform thickness and the natural way to do that is to increase the supersaturation of the reaction species and the pressure in the reactor. However, the probability to get irregular nucleation and growth of the Al₂O₃ crystals resulting in high porosity and poor adhesion is impending [31-32]. Further the growth of Al₂O₃ is sensitive for impurities which will lead to inhomogeneous growth and poor adhesion. Especially Co originating from the substrate results in such effects and usually the WC/Co substrate is pre-coated with Ti-C-N layers in order to ensure a Co-free nucleation surface. Also, e.g. TiC often shows epitaxial growth on WC which is believed to enhance the adhesion [31, 38-39].

Ti-C-N systems

The crystallographic phase of the alumina is strongly dependent of the characteristics of the nucleation surface. When depositing the α-Al₂O₃ the needed template have been found to be a titanium oxide deposited/oxidised on a Ti(C,N) coating structure [29-37]. The κ-Al₂O₃ does not need any titanium oxide to grow at but are preferentially grown directly on TiC or TiN. Accordingly, TiN, TiC and Ti(C,N) are important layers in the CVD alumina coating structure regarding to crystallographic matching and a surface free from impurities.
Often a first layer of TiN and Ti(C,N) are deposited directly on the cemented carbide in order to stop the diffusion of Co. Both TiC, TiN and Ti(C,N) have face centered NaCl (cubic) structure. The C and N content varies in a wide range and thus also the lattice parameter will vary. Also the hardness varies in the same way and is highest for TiC coatings while TiN layers often are decreasing the friction. All the layers show good wear resistance and additionally the TiN coating has an attractive colour (golden) which makes it easier to visualise the wear when using it as the outermost layer in a coating system [38].

Ti(C,N) are generally deposited by MT-CVD (moderate temperature, 700-900°C) which results in columnar grains. TiC and TiN are often deposited by conventional CVD at temperatures 900-1100°C.

4.4.2 PVD coatings

Physical vapour deposition (PVD) involves atomisation or vaporisation of solid source material which is then condensed on a substrate. PVD-techniques are often categorised by the principles behind the atomisation and are divided in evaporation or sputtering. Evaporation involves thermal vaporisation (thermal energy) of the deposition materials while sputtering is a kinetic controlled process. However, the PVD coatings in present thesis are deposited by arc evaporation why this technique is further explained.

In arc-evaporation low voltage and high current are used to create a plasma discharge between two electrodes. The arc discharge will hit a small area of the cathode surface (the source material) which yields a high current density. The high current results in extremely high temperatures (15,000°C) and evaporates atoms and emits electrons. The electrons will then collide with the atoms and ionise. Often the substrates are negatively charged and will attract positive ions that will condensate together and react with the applied gas, e.g. oxygen or nitrogen [40-41].

When the vaporised material has been condensed on the substrate a thin film is formed. Often the pressure is in the range 10^{-5} -10^{-2} mbar and the temperature 300-500°C which result in a deposition rate of about 0.001 to 75 μm/min. Hence, as compared to CVD the temperature is much lower which reduces undesired diffusion processes or reactions between substrate and coating. The bombardment of ions can often result in compressive stresses which will increase the hardness and fracture toughness [2].

Compared with other PVD-techniques arc-evaporation shows a very high degree of ionisation of the evaporated particles and consequently an applied bias of the substrate can be used to control the energy of the particles hitting the substrate and thus the microstructure, defect density and properties of the resulting coating. The arc evaporation technique also allows the growth of dense coatings at relatively low temperatures [43].
However, the arc process does not only involve the evaporation of ions and atoms from the cathode surface but also significantly larger particles. These are usually referred to as droplets or macro particles and will unfortunately result in a rough coating surface morphology with hard protruding asperities which will have a detrimental effect on coating properties. Hence, it is of interest to reduce the number of particles hitting the substrate and one way is to use shields or magnetic filters. However, these will reduce the deposition rate and is therefore not common in the cutting tool industry [43-45]. The PVD coatings evaluated in present thesis is deposited using arc evaporation without shields or filters.

**Titanium nitrides**

TiN, TiAlN and TiSiN are examples of hard coatings with high toughness and are commonly used in different cutting operations. The problem with TiN is that it is oxidising at 500-600°C and with the purpose to increase the oxidation resistance Al is incorporated. The resulting TiAlN has good oxidation resistance at high temperatures since that aluminium is forming a stable oxide (Al₂O₃) protecting the underlying and chemically unchanged TiAlN. High hardness in combination with oxidation resistance is of great importance in cutting where high temperatures and pressures are obtained.

Generally TiSiN has a higher hardness as compared to TiAlN, otherwise the coatings are showing similar mechanical properties. The crystallographic structure is known as [NaCl]-TiSiN (c-TiSiN) and [NaCl]-TiAlN (c-TiAlN) respectively [40].

**Vanadium nitrides**

Vanadium nitride (VₙN) is a nitride closely related to TiN and has a high hardness and fracture toughness. As for TiN the crystal structure is cubic and is usually described as [NaCl]-VN (c-VN). However, for VₙN coatings the hardness is not the property of highest interest. Instead the formation of a low frictional Magnéli phase (V₂O₅) is of more interest at the interface in sliding contact tribo-systems. This and the fact that it shows low adhesion to e.g. iron-base materials makes it interesting in e.g. metal cutting or sheet metal forming to decrease the tendencies to material transfer [46-47].

**Aluminium oxides**

Aluminium oxides generally shows a high chemical stability and low oxidation tendency. One of the disadvantages with CVD alumina is the residual tensile stresses due to the differences in thermal expansion between substrate and coating. Hence, many attempts to deposit alumina at lower temperatures using PVD have been performed. One way to obtain similar properties as for α-Al₂O₃ is to deposit AlCrO by using alumina-chromia solid solution [43]. The crystallographic structure is more or less a corundum structure, i.e. trigonal, and is dependent of the Cr content. Generally AlCrO shows a
lower hardness as compared to the TiAlN and TiSiN but much better oxidation resistance.
5. Coating characterisation

5.1 Chemical composition and microstructure

In the characterisation and understanding of the coating materials different surface analysis techniques can be used. In this thesis microscopy, spectroscopy as well as X-ray diffraction was used.

5.1.1 Light optical microscopy (LOM)

To get a first impression of the samples investigated a LOM have been used. It is often important to prepare a flat surface because the depth resolution is rather poor – 0.3 µm. The technique is fast and gives valuable information of e.g. chips of working steel material when combining LOM with etching of the surface investigated. This was done in Paper VII.

5.1.2 Optical interference profilometry (WYKO)

Measurements with optical interference profilometry are of great importance in the characterisation of surfaces and result in 2D and 3D pictures as well as a huge number of mathematical parameters describing the surface. Measurements of the virgin coating topography or the worn surface will increase the understanding of the tribological behaviour. This method, using visible light, is rather quick with a depth resolution of 5 nm but one drawback is the demand of reflecting samples which makes it necessary to use sputter coating deposition of a reflective gold layer when the coating samples are too transparent. In this thesis a Wyko NT-9100 was used (Paper III-VIII).

From each area of measurement values such as $R_a$, $R_z$, $S_{sk}$ and the bearing ratio of the closely-spaced irregularities and texture of the surface are calculated. Usually, one surface value is not enough to describe the surface roughness and several of the above mentioned parameters were used in combination. Also, the wear of the worn surfaces was obtained by measuring the volume of the wear track.

The $R_a$ value is an arithmetic average height of the deviation of the surface ($y$) from the mean line over one sampling length ($l$) and is calculated according to eq. 5.1.
\[ R_a = \frac{1}{L} \int_0^L |y(x)| \, dx \]  

(5.1)

The \( R_z \) value (the ten-point-height) is the difference between the mean value of the five maximum peak heights and the five minimum depths from an arbitrary chosen reference line, over the entire 3D surface, see eq. 5.2.

\[ R_z = \frac{R_1 + R_2 + R_3 + R_4 + R_5}{5} - \frac{R_6 + R_7 + R_8 + R_9 + R_{10}}{5} \]  

(5.2)

In order to further describe the surface the skewness (\( S_{sk} \)) was used and it describes the difference in symmetry of the surface profile around the mean line. For example an as-deposited PVD coating with a high amount of protruding droplets would result in \( S_{sk} > 0 \) while a polished one with shallow craters would result in \( S_{sk} < 0 \) [48].

Further, the term bearing ratio is often used to illustrate the roughness and shows how much of the surface profile that is displaced from the mean line. The curve could be found from a profile trace by drawing lines parallel to the datum and measuring the fraction of the line which lies within the profile.

5.1.3 Scanning electron microscopy (SEM)

The possibility to characterise as-deposited coatings as well as worn surfaces by obtaining micrographs with good resolution at high magnification is of great importance in the understanding of the tribological behaviour of a certain coating material in a certain tribological contact. This was possible in present thesis (all Papers) by using two kinds of SEM; a Leo Ultra and a Zeiss FEG-SEM. Compared to LOM the technique promotes much higher resolution and greater depth of focus, and thus higher magnification is possible to obtain [49].

By accelerating an electron beam into the sample different phenomena give information about the material investigated. The electrons are emitted from an electron source and the most commonly used sources are W, LaB\(_6\) and Field Emission Gun (FEG). The electrons are accelerated in the electron gun to energies in the range 1-20 keV and then the beam passes through a system of lenses and apertures. The used instruments are equipped with Secondary Electron (SE), Backscatter Electron (BE) and Energy Dispersive X-Ray (EDX) detectors. The FEG-SEM also has an in-lens detector.

In Figure 9 the activated volume and the escape depths of the detected electrons is shown. The principle behind the formation of SEs is the inelastic interaction between the primary electrons and the valence electrons of atoms.
in the sample, which results in the ejection of electrons from the atoms. Ejected electrons with energies less than 50 eV are termed “secondary electrons” (SE). Several SEs can be produced by one incident electron and due to their low energy only SEs generated near the surface (50 nm) can exit the sample and be detected. By changing the beam current, voltage or apertures the surface sensitivity, signal intensity and resolution may be optimised. However, the production of SEs is very topography dependent.

The BEs have higher energies as compared to the SEs and hence a larger escape depth. They are produced by interaction between the beam electrons and the atom nuclei in the sample. With BE images it is possible to show characteristics of geometry (topography) and atomic number (Z) contrast. Surfaces with a high average Z appear brighter as compared to surfaces with a low average. This is useful when e.g. characterising material transfer of steel to a coating.

When characterising very thin coatings or thin tribofilms the escape depth of the SEs and BEs can be a limitation and the voltage have to be as low as possible (about 1 kV) to minimize the escape depth. Also, when analysing ceramic coatings the low electric conductivity will give problems with charging of the surface.

![Diagram](image)

**Figure 9.** The activated volume and escape depths for the detected electrons and radiation [50].
5.1.4 Energy dispersive X-ray (EDX) analysis

When the electrons in the electron beam hit the sample surface an electron from the core shell of a sample atom can be removed creating a vacancy. The energy relaxation of the formed ion can occur through transfer of an electron from an outer shell to the created vacancy. The excess energy will then be emitted as a X-Ray photon which is characteristic for each element, see Fig 10a. The EDX detector will then detect and measure the energy of the photon [49].

By using EDX the elemental composition of the sample will be determined. The information depth is dependent of the activated volume and hence the primary electron energy, i.e. the voltage but also the chemical composition influences. The activated volume decreases with increasing atomic number.

In present thesis bulk composition and mainly thin interlayer, tribofilms and defects in the coatings were analysed with EDX. To perform this, low accelerating voltages (<5kV) were often used resulting in analysis depths of about 100 nm. But, at these low voltages only light elements can be detected.

![Diagram of energy release](image)

*Figure 10. Illustration of two paths for energy release in the form of a) a X-ray photon and b) an Auger electron [51].*

5.1.5 Auger electron spectroscopy (AES)

When the electrons are hitting the surface and creating a vacancy in the core shell the following relaxation of the formed ion and excess of energy do not necessarily have to emit an X-Ray photon. Instead an Auger electron can be emitted, see Fig 10b, and by measuring the energy of the detected electron the chemical composition can be determined.

The energy is released simultaneously by creating an Auger electron in a radiation free process. For low atomic number elements the most probable transitions occur when a K-level electron is ejected by the primary beam and
an L-level electron drop into the vacancy while another L-level electron is ejected (KLL-transition). Higher atomic number elements have LMM and MNN transition which are more probable as compared to KLL. Under practical analytical conditions the mean free path is approximately 25 nm and depends on the electron energy and the sample matrix. The energy distribution is initially narrow for the Auger electrons which then will lose energy when passing through the sample matrix. Electrons escaping from depths deeper than about 1-5 nm in the sample will only contribute to the spectrum background. Hence, each measured energy corresponds to a certain element and the AES makes it possible to detect small amount of elements. The technique is also very surface sensitive (30Å).

The peak height of the Auger electrons are related to the elemental concentrations but also to instrumental factors such as primary beam energy, sample orientation and the energy resolution and acceptance angle of the analyzer. Also, the chemical state, especially the oxidation state of the elements will influence on the peak intensity and shape.

In present thesis (Paper I-II and V) the advantages of the AES made it possible to analyse very thin tribofilms and interfaces in thin multilayered coating structures. The used AES equipment was a PHI 660 Scanning Auger Microprobe SAM and the analysis were performed with an accelerating voltage of 10 kV and a primary beam current of about 100 nA. Depth profiling of the CVD coatings was performed using 3.5 keV Ar⁺ ion sputtering in the SAM system [49, 51].

5.1.6 Secondary ion mass spectrometry (SIMS)

The principle of SIMS is that ions are produced from a solid sample by using a focused ion beam. The ion beam is directed toward the sample surface and removes material in the form of neutral and ionised atoms and molecules as well as electrons and photons. Usually the ionised secondary particles (about 10 %) are extracted by an electric field and accelerated into a mass spectrometer which separates the ions with respect to their mass-to-charge ratio (m/Z).

The in this thesis (Paper I) used SIMS instrument is equipped with a Time of Flight (ToF) mass spectrometer. The slowly sputtering energetic primary ion beam (Ga⁺) has an accelerating voltage of 0.5-25 keV and a current of 600 pA-20 nA. This analysing mode is termed static SIMS and makes it possible to analyse the top atomic layers while dynamic SIMS, which is used for depth profiling, implies a higher primary ion dose and hence will remove a larger amount of material during the sputtering. The intensities of the measured secondary ions are displayed in a mass spectrum as a function of m/Z. By sputtering a sample with a bunched primary ion beam while the mass spectrometer scans the mass range a mass spectrum is produced.
By using the dynamic mode sputtering and analysis will be combined and by monitoring the secondary ion count rate of selected elements as a function of time a depth profile is obtained. One of the advantages with the SIMS is the high detection rate which makes it possibly to analyse trace elements, with origin from the deposition process, in the coatings investigated. However, one drawback is the difficulty to get quantitative results due to the fact that the amount of secondary ions per incoming primary ion will vary regarding to many factors, e.g. between elements as well as between type of matrix [52].

5.1.7 X-Ray diffraction (XRD)

By using X-Ray diffraction the crystallographic phase composition can be determined. Usually a monochromatic X-Ray beam is sending CuK$_\alpha$ radiation (wavelength $\lambda=1.5418$ Å) into the sample at a given angle $\theta$. A crystalline sample will diffract the X-Ray beam with the lattice planes but only when the distance travelled by the reflected ray from a set of parallel lattice planes differs with a wavelength of $\lambda$. The plane spacing in the crystalline sample can be determined according to Bragg’s equation; $2d_{hk} \sin \theta = \lambda$. By using a moving detector the diffracted beam, which intensity varies with the angle, will be detected.

The intensity is plotted as a function of the angular position in a diffractogram giving the diffracted peaks of radiation. Each sample has a characteristic diffractogram and when a mixture of phases is present the diffractogram is a result of the individual patterns for each phase. By using reference databases the peak positions in the obtained diffractogram can be compared to known patterns and hence the present phases can be determined.

GI-XRD (Grazing Incidence XRD) is a method using a low fixed incident angle for the X-Ray source while the sample and detector are moving. By using very low incident angles the outermost surface layer of the coatings will be analysed. The higher the angle is the deeper into the coating the information will be obtained from. In the Th-2Th method also the X-Ray source is moving, with half of the angle of the detector. By this performance the depth of information is much larger and valuable information of the entire coating system is obtained. Further, if the purpose is to evaluate the texture of the coatings a goniometer is used. Here texture coefficients (TC) are calculated by using the measured intensities of the reflections and reference powder diffraction intensities [53].

In this thesis (Paper I-II and IV) the Th-2Th, GI and Texture investigations were carried out by using a Bruker D8 X-ray diffractometer system, a Philips MRD pro system and a Philips PW1050 powder goniometer respectively.
5.2 Mechanical and tribological properties

In metal cutting a large number of wear mechanisms are obtained and are overlapping each other why it often is difficult to separate the influence of single mechanisms on the friction and wear. In order to refine mechanisms and hence the understanding of different coating materials in tribological contacts, tribological testing is performed. The purpose is not to simulate a cutting process but rather to increase the understanding of the material behaviour in different well defined tribological contacts that can increase the tribological understanding in the cutting process. Further, mechanical testing gives valuable information, such as hardness and Young’s modulus, which also will increase the understanding of the tribological behaviour.

5.2.1 Coating adhesion

Adhesion is of great importance in metal cutting and to measure the adhesive strength of coating materials scratch testing is one of the most widespread techniques [2]. In the scratching contact the most widely used stylus is a conical indenter (Rockwell C) with a 200 µm radius spherical diamond tip and at specific loads, $F_c$, specific coating failures may occur [19-20, 54-57]. Commonly, the normal load corresponding to the first coating failure (e.g. local spalling) is defined as $F_{N,C1}$ and the load corresponding to continuous substrate exposure (continuously spalling) as $F_{N,C2}$. A schematic of a scratch is seen in Figure 11. In current thesis (Paper I-VI) the failure corresponding to the defined critical load is carefully described in each paper respectively. By increasing or decreasing the tip radius the adhesive contribution to the failure will increase and decrease respectively, and vice verse for the cohesive relationship. In order to further increase the adhesive contribution or with the purpose to simulate a more accurate tribological contact multi-pass scratching at sub-critical loads has been evaluated in several studies [58-60].

Further, scratch testing can be used in a modified set-up where a pin of a work material instead of a diamond stylus is sliding against the coating. This performance makes it possible to evaluate the friction and material pick-up characteristics of the coatings in sliding contact against e.g. different kind of steel grades.
The scratch test equipments used in this thesis were a Micro Combi Tester (MCT) and a Revetest (CSM Instruments Revetest®). The latter makes the modified set-up possible and has a maximum load of 200 N while the maximum load of the former one is 30 N. For both equipments the friction force and acoustic emission (A.E.) signals continuously were recorded during the tests. All experiments were performed in ambient air (21-22 °C, 30-32 % RH).

In present thesis the cohesive and adhesive characteristics of PVD coatings (Paper V and VI) were evaluated by using single and multi-pass scratch testing. For V₈N also modified multi-pass sliding tests against stainless steel were performed.

In Paper I and II the adhesive strength of CVD bonding and template layers were evaluated by using single scratch testing at low loads.

5.2.2 Coating hardness

Today there are a large number of different methods for hardness measurements. Micro Vickers is a commonly used method (Paper I-II) where a well defined Vickers stylus, four sided pyramid, is pushed down into the material at a specific load. After indenter removal the load carrying area is measured. The hardness is then calculated by using the area and the maximum load and is given as a pressure. The drawback with such methods is that the indent has to be visible to be possible to measure. For indents at low loads the area is very small and the area measurement error will be large. Errors are also obtained if ignoring the elastic recovery [2, 61]. Further, when measuring the hardness of thin coatings it is important to avoid contribution from the underlying substrate. Due to the plastic deformed zone beneath the indent a rule of thumb is that the depth of the indent should not be larger than 1/10 of...
the coating thickness to avoid the influence from the substrate. In order to avoid these problems depth sensing methods such as nanoindentation are used. This method senses the penetration depth and the displacement during loading and unloading is measured. The most common method of the hardness calculation is presented by Oliver and Pharr [61]. Usually a Berkowich diamond indenter, with a three-sided pyramid geometry, is used. With knowledge about the indenter geometry it is possible to obtain a hardness value from the depth of the plastic deformation by subtracting the elastic contribution from the total depth. Further, by analysing the unloading curve in the nanoindentation test the Young’s modulus (E) can be determined. The initial slope of the unloading curve is directly related to the elastic modulus. The hardness is related to the projected area corrected by the indenter constant for the used tip. Figure 12 shows a typical load-displacement curve with related indent in cross section [2, 61].

In present thesis a Micro Combi Tester (MCT) using a Vickers stylus at low loads was used (Paper IV-VII) and the hardness was calculated according to Oliver-Pharr. The Vickers indenter is not as sharp as the Berkowich indenter and hence the shape correction factor differs. It should also be mentioned that all the indents on the coatings were performed on flat areas free from defects at as-deposited coatings. As highest the maximum depth was twice the proposed value 1/10 (i.e. 2/10) of the coating thickness but still the residual plastic depth after unloading was lower than 1/10 of the coating thickness and the hardness values are still relevant. The used loads could be somewhat lower but then it is more difficult to do accurate indentation in areas of the coating totally free from defects due to the waviness.

Figure 12. A typical load-displacement curve obtained in nanoindentation and a corresponding cross section of an indent [61].
5.2.3 Abrasion resistance

With the purpose to refine the in cutting process common abrasive wear mechanism, micro abrasion testing was performed (Paper III-IV) at room temperature. This makes it possible to distinguish the abrasive deformation behaviour and wear resistance between different phases, structures and textures of the coating materials.

The micro abrasion tests of the coatings were evaluated using a Precision Dimple Grinder (Gatan Model 656). The equipment, normally used for preparation of samples for transmission electron microscopy (TEM), consists of a stainless steel grinding wheel (radius 10 mm) rotating against a horizontally mounted sample which in the present study was fixed, see Figure 13. In the micro-abrasion tests, a slurry with 1 or 6 µm diamond particles, a normal load of 20 g and a rotating speed of 220 rpm were used.

![Figure 13. The principles of the micro abrasion test (a) and the resulting crater (b).](image)

In order to keep the resulting craters, see Fig 13b, within the thickness of the coatings, the grinding time was set to 120 s for the finer 1 µm and 20 s for the larger 6 µm diamond particles.

5.2.4 Sliding wear resistance

Pin-on-disc test was used to simulate various sliding contacts to determine the sliding wear rate and the coefficient of friction. Compared to cutting the pressures are very low (less than 1 GPa) but still the method will increase the knowledge of coatings in sliding contacts and is known as a suitable method especially for contacts where one surface constantly is in contact [2].

The pin-on-disc test is a model test (CSM Instruments HT Tribometer®) where a stationary pin is sliding against a rotating disc while the friction force continuously is recorded. A cyclic movement according to Figure 14 is performed and the radius of the wear track is easy to change in the range 0-30 mm. Also the location of the track is easy chosen and many tests can be
performed at the tools. The normal load is selectable in the range 0-40 N and the sliding speed in the range 0.015-30 m/s.

Figure 14. The pin-on-disc setup.

Sliding wear testing was performed in paper VII using a quenched and tempered steel (AISI 4140) pin with a diameter of 10 mm and a polished ($R_a=25$ nm) hemispherical shaped end surface (radius 5 mm) was loaded against a CVD $\alpha$-Al$_2$O$_3$ coated cemented carbide insert (cutting tool). The normal load was set to 20 N and the relative sliding speed to 0.1 m/s. The test was run for 1000 s corresponding to a sliding distance of 100 m.

Also, the circular multi-pass scratch testing in paper VI was performed by using the pin-on-disc test equipment, but instead of a pin a Rockwell C diamond stylus (tip radius 50 µm) commonly used in scratch adhesion experiments (by using a scratch tester) was used. The multi-pass tests were performed at normal loads of discrete values between 0 and the critical load, $F_{N,C2}$ for each sample investigated. The speed was set to 90 mm/min (0.015 m/s).

All tests were performed in controlled room temperature (20-22 °C) and relative humidity (25-26 %) and repeated at least three times. The friction coefficient was continuously recorded.

However, the tribological contact in pin-on-disc is far away from that in cutting but anyway it will show mechanisms that also occur in the cutting process and hence will increase the knowledge. Also, the test makes it possible to increase the understanding of the general deformation behaviour, including crack formation, of different coating systems.
5.3 Performance in metal cutting

All the tribological testing methods were performed to increase the understanding of the tribological properties of coatings designed to be used in cutting operations. However, real cutting tests are important in order to increase the tribological understanding of the cutting process but also to increase the understanding of which mechanisms that are important to refine regarding to friction and wear. In present thesis two kind of cutting tests were performed.

The first type of cutting test was a coating lifetime test (Paper III) in the form of continuous turning of a plain carbon steel (AISI 4340). The feed, depth of cut and cutting speed were 0.4 mm/rev, 2.5 mm and 200, 250 and 275 m/min, respectively. The cutting times were chosen to 1, 3, 6 and 10 minutes in order to follow the flank and crater wear. After cutting, the tools were cleaned in HCl to remove the major part of adhered workpiece material. The most straight forward method to get quantitative values of the wear of the cutting tools is to measure the flank wear which have been done by using optical microscopy.

The second type of cutting test used in this thesis (paper VII and VIII) was so called plunging operations were the tool feed is in the radial direction of the workpiece. This method makes it possible to measure cutting forces that can be used in calculations of the contact conditions. The experiments were performed on AISI 4140 medium carbon steel at a cutting speed of \( v_c = 240 \) m/min. The feed during these experiments varied from \( f = 0.025 \) mm/rev to \( f = 0.3 \) mm/rev. During these experiments the cutting forces were measured by using a Kirster Z15814 piezoelectric measuring equipment. The obtained chips were measured after each machining operation by using a Mitutoyo 422-260 digital micrometer.
6. Coating engineering, characteristics and tribology

This thesis is about coating engineering, characteristics and tribology and the papers included focus on abrasive wear, sliding wear, interfacial fracture toughness and adhesion which all are important aspects for tools used in metal cutting operations. Also the nucleation of CVD alumina was evaluated in detail. This chapter present the most noteworthy results obtained within this thesis work.

6.1 Nucleation

In metal cutting operations the phase composition of e.g. Al₂O₃ coatings has a large impact on the tribological performance. It is well known that when depositing polymorphs with only slight differences in thermo chemical stabilities, the nucleation process controls the phase composition of the coating. Consequently, the phase composition (α/κ) in the CVD Al₂O₃ layer is influenced by the underlying surface and in order to control the nucleation process and growth of α-Al₂O₃ a template is used. The template is developed empirically and the reason why the nucleation of a certain polymorph is favoured by a certain template is poorly understood. An exactly composition of the template is not known, nor if it is chemically stable or not. In Paper I and Paper II two different sample series were investigated, a single template with sharp interfaces and a commercial gradient multilayer structure with a bonding layer under the template.

6.1.1 Single template

In Paper I a relatively thick template was deposited onto a Ti(C,N) pre-coated substrate and the results clearly show that α-Al₂O₃ may nucleate on Ti₄O₇. Further, the results show that while the as-deposited, uncoated template consists of Ti₄O₇ (triclinic structure), the template coated with a thin α-Al₂O₃ layer has been completely transformed to Ti₃O₅ (monoclinic structure) during the coating deposition process. One plausible reason to this transfor-
Information is that Al₂O₃ is thermodynamically more stable than Ti₄O₇. In this scenario oxygen diffuses to the Al₂O₃.

Also, the AES and SIMS sputter depth profiles display a constant concentration of Ti and O within the layers. The AES depth profile of the template-Al₂O₃ structure, see Fig 15, show that the template mainly consists of Ti and O, and has a thickness of approximately 175 nm. The thickness of the Al₂O₃ layer on the template is approximately 200 nm.

![Graph showing concentration vs depth for template and Al₂O₃](image)

*Figure 15. AES depth profile of the template and the deposited thin α-Al₂O₃.*

The SIMS depth profiles, Fig 16 show similar results as the AES analysis but the advantage with SIMS is the high detection power which makes it possible to detect trace elements. However, the information is primary of a qualitative nature. In present study traces of Cl, Fe and Cr were detected, and in small quantities Fe and Cr may influence the CVD process. The presence of these elements can be traced back to the CVD process where the supply tubes and the reactor are exposed to HCl forming volatile Fe- and Cr-chlorides. These elements were then involved in the growth process of the coating.
6.1.2 Gradient bonding template multilayer structure

In the second test series (Paper II) produced in order to increase the understanding of the nucleation of α-Al₂O₃ the template was constructed of a gradient multilayer structure, instead of sharp interfaces, using a bonding layer under the template. The template system is used commercial and is known to grow α-Al₂O₃ coating systems with excellent performance and wear resistance in cutting operations. However, commercial coating systems result in extremely thin templates which are difficult to analyse. In Paper II three different samples were investigated, a bonding layer (sample I), a template (sample II) and a thin Al₂O₃ layer (sample III).

It was found that the grown template consists a Ti₄O₇ phase and during subsequent deposition of the thin α-Al₂O₃ layer the entire Ti₄O₇ phase transforms to Ti(C,N,O). Hence, as in paper I it was shown that α-Al₂O₃ can nucleate on Ti₄O₇.

As can be seen in Figure 17 the chemical composition of the Ti(C,N,O)-bonding layer (sample I), is rather uniform with Ti, N and C contents of approximately, 48%, 23% and 23%, respectively. Furthermore, there is a small amount of O, 4-6%, in the layer, which slightly increases towards the surface that gives a needle-like morphology believed to increase the adhesion.

The Ti₄O₇-template (sample II) shows Ti and O contents of approximately 30% and 45%, respectively. Also, the layer contains a significant amount of N and a small amount of carbon (close to 0 at the surface).

When finally depositing the subsequent thin α-Al₂O₃ layer (sample III) the O content of the underlying template has decreased to less than 10%, and the plateau, present in sample II is absent. Accordingly, a large amount of O in the Ti₄O₇ layer will react with the AlCl₃ and form Al₂O₃. This reaction is much more pronounced for the gradient layers as compared to the layers in

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**Figure 16.** SIMS depth profiles for (a) positive ions, (b) negative ions for the thin α-Al₂O₃ layer deposited on the template. Ti⁺ corresponds to the Ti signal in positive mode when supplied oxygen is used.
Paper I. Also, it is a small amount of Al in all the coatings investigated, except for the coatings under the thin multilayer structure.

Figure 17. AES depth profiles for the bonding layer (sample I), template (sample II) and the subsequent deposited thin \( \alpha \)-Al\(_2\)O\(_3\) (sample III).

6.2 Coating adhesion

The adhesion in the interfaces in a coating structure is of great importance regarding to interfacial failure in cutting operations. In Paper I and Paper II it has been shown that factors such as chemical bondings, crystallographic mismatch, interfacial porosity, surface topography as well as the interfacial construction (i.e. gradient vs. sharp interfaces) will influence the adhesion.

6.2.1 Interfacial porosity and sharp interfaces

In Paper I scratch testing at low loads shows that interfacial micro spalling occurs at the rather sharp interface between the Ti(C,N) and the relatively
thick template. In contrast, the interface between the template and the $\alpha$-Al$_2$O$_3$ layer does not show any tendency to spalling and consequently the interface Ti(C,N)/template is the weakest interface within the multilayer structure. The critical normal loads for continuous micro spalling were found to be 3.7 N for the template (sample A) and as low as 0.5 N for the sample with a thin deposited $\alpha$-Al$_2$O$_3$ layer (sample B). Also, when micro spalling occurs the spalled off areas are significantly larger for sample B as compared with sample A. Further, it should be mentioned that the edges of the spalled off areas follows the network of the thermal cracks.

Figure 18 shows the backside of coating fragments that were spalled off from sample A (Fig. 18a) and B (Fig. 18b), respectively, during the scratch test. As can be seen the underside of the templates show very different features regarding to interfacial porosity. For the Ti$_3$O$_5$ template the presence of relatively large pores, up to 400-500 nm in size, in the coating is pronounced which would explain why the critical load for continuous micro spalling is lower for sample B as compared with sample A. The pores promote cracking and fracture along the interface and consequently adhesive failure of sample B. Also the contact area decreases.

![Figure 18. SEM images from the underside of fragments that were spalled off during the scratch test from a) sample A and b) sample B.](image)

The reason for the creation of the interfacial porosity during the phase transformation of the template is not fully understood. It is believed that the Ti$_4$O$_7$ $\rightarrow$ Ti$_3$O$_5$ transformation could not be responsible for the development of a porous interface since volume contraction encountered in this transformation is small. A more probable explanation is that the template to a small extent is transformed back to fcc TiO, Ti(C,O) or Ti(C,N,O) due to interdiffusion at the Ti(C,N)/template interface. This volume contraction in the order of 25-30% may explain the observed porosity.

One probable explanation for why micro spalling occurs in the interface Ti(C,N)/template and not in the interface between the template and the $\alpha$-Al$_2$O$_3$ layer may be a more pronounced crystallographic mismatch between Ti(C,N) (cubic fcc) and the templates; Ti$_4$O$_7$ (triclinic) and Ti$_3$O$_5$ (mono-
clinic) than between the templates and $\alpha$-Al$_2$O$_3$ (trigonal). Also the fact that
the interface comprises of a contact between a covalent (Ti(C,N)) and an
ionic (Ti$_6$O$_{2n-1}$) material may weaken the interface.

6.2.2 Gradient multilayer structure and surface topography
In paper II a thin gradient multilayer structure (Ti(C,N,O)/Ti$_4$O$_7$/\(\alpha$-Al$_2$O$_3$)
was deposited on a thicker inner TiN/Ti(C,N)/TiN multilayer structure resulting
in lack of sharp interfaces. The Ti(C,N,O)-bonding layer and the
Ti$_4$O$_7$-template structure is typically used in commercial Ti(C,N)/Al$_2$O$_3$
coating systems. Often in metal cutting operations spalling of the Al$_2$O$_3$ layer
occurs, why the understanding of the adhesion is of great importance.

Results of scratch adhesion tests show that the strength of the interfaces
within the bonding/template/\(\alpha$-Al$_2$O$_3$ multilayer structure is high and that the
lowest interfacial strength for all samples investigated was displayed by the
interface between the cemented carbide substrate and the inner TiN layer of
the TiN/Ti(C,N)/TiN multilayer structure, see Fig 19a. Besides spalling
along the cemented carbide/TiN interface, minor spalling occurs along the
upper Ti(C,N)/TiN interface in the inner multilayer structure, see Fig 19b.
The reason for this may be explained by the smooth surface of the Ti(C,N)
layer, i.e. a smooth surface results in less surface area as compared to a
rough surface and accordingly fewer chemical bonds between the two layers,
resulting in a lower adhesion. Thus, increasing the surface area, i.e. forming
a rougher Ti(C,N) surface, would probably increase the adhesive strength of
the inner TiN/Ti(C,N)/TiN multilayer structure. In the present study, the
morphology of the Ti(C,N,O) bonding layer was designed by controlling the
O-content in the CVD-chamber. It was found that an O content of around 5
% in the Ti(C,N,O) resulted in beneficial bonding layer morphology with a
beneficial nano-scale roughness and strong adhesion. Also, the fact that the
resulting bonding/template/\(\alpha$-Al$_2$O$_3$ multilayer structure does not show any
sharp interfaces but instead can be regarded as a gradient structure is be-
lieved to improve the adhesion strength of the system. These results are in
good agreement with the results in Paper I which showed that the absence of
a bonding layer and a sharp interface between the template and the underly-
ing layer resulted in poor adhesion.
6.2.3 Coating defects

All interfacial defects such as porosity etc, will decrease the adhesion of the coatings. In Paper V the influence on the adhesive strength of defects in form of droplets and craters were evaluated for V$_x$N-PVD coatings. In a repeated scratching contact of the coatings it was shown that a high defect density, i.e. a high amount of coating defects of a critical size, strongly influences the adhesion in a detrimental way. The results will further be discussed in the chapter 7.4.1.

6.3 Interfacial fracture toughness

In the interfaces in a coating structure chemical bondings, interfacial porosity, surface topography as well as the interfacial construction (i.e. gradient or sharp interfaces) have been shown to influence on the adhesion. However, the potential of the coating system to decrease the shear stresses in the interface also have a large impact on the adhesion. Hence, interfacial fracture toughness further describes the resistance to cracking due to debonding in the interface in the coating system. In Paper V and Paper VI this has been shown more in detail.

6.3.1 Influence of defects and mechanical properties

In Paper V the influence of defects (i.e. droplets and craters) on the cohesive and adhesive strength was evaluated. Multi-pass scratch testing by using a diamond stylus was performed on V$_2$N and VN coatings, both contending a high amount of surface defects. Figure 20 shows scratch induced coating failure maps illustrating the effect of normal load (F) and number of scratching cycles (N) on type of major coating failure of the two coatings investi-
In the maps, three different regions, I – III, can be distinguished, i.e. regions corresponding to: I) plastic deformation, II) local interfacial spalling or chipping, III) continuous interfacial spalling along the scratch.

As can be seen, the more defect rich and harder V₂N coating displays a smaller area of Region I as compared with the less hard VN coating for the number of scratching cycles investigated. Also, the slope, dF/dN, of Region II differs between the two different coatings investigated.

The A.E. signal obtained during the scratching event is shown in Figure 21 and corresponds to interfacial cracking/spalling. For the V₂N coating the resulting continuous interfacial spalling appearance is the result of several local failures at different positions during several cycles while the VN coating may display interfacial spalling appearance during one or two cycles. These facts makes it plausible that the presence of coating defects of critical size at the coating/substrate interface is believed to have the strongest impact on the interfacial strength and thus will result in lower critical loads.

However, probably a number of parameters makes the interface more sensitive to interfacial cracking and the occurrence of defects in combination with a higher coating hardness, a higher friction coefficient ($\mu_{V2N}=0.03$ as compared with $\mu_{VN}=0.01$) during the scratching event and a larger mismatch in coating/substrate mechanical properties (e.g. the H/E-parameter) influence. In this way the V₂N coating is believed to be more sensitive to interfacial cracking and spalling.

Consequently, the intrinsic mechanical properties of the coating and substrate in combination with the presence of critical interfacial defects may explain the differences in interfacial fracture toughness.
6.3.2 Coating fatigue resistance

A wide range of laboratory techniques are available to assess the mechanical and tribological characteristics of hard coatings for metal cutting tools but still the question is current about how to simulate a tribological contact in an accurate way. Single- and multi-pass scratch testing is believed to be a rather accurate contact, especially at lower loads, but it is difficult to perform tests in a large number of cycles as in e.g. impact testing. Therefore circular multi-pass scratch testing by using a pin-on-disc equipment was proposed in Paper VI as a test concept to evaluate the fatigue, wear, chipping and spalling behaviour of thin hard coatings under repeated/cyclic sliding contact conditions.

When comparing the results from the conventional single pass scratch test with the circular multi-pass scratch test it can be concluded that both tests display similar major coating failure mechanisms as long as the normal loads in the multi-pass test is relatively high, typically $F_{\text{norm}} \geq 0.60$, and is restricted to a low number of cycles, typically less than 10. Within this region coating failure is due to; plastic deformation and minor cracking (no coating material detached), chipping (cohesive failure) in combination with local interfacial spalling (adhesive failure) and continuous interfacial spalling (adhesive failure) outside the scratch. However, while the conventional single pass scratch test mainly gives information about the mechanical response of the coating-substrate composite, the circular multi-pass scratch test also gives information of the tendency to surface fatigue and wear when exposed to a large number of contact cycles. Besides, it also makes it possible to de-
termine a “fatigue limit” for the coating-substrate composite. In Figure 22 a coating failure map of the TiAlN coating is shown. The map illustrates the influence of normalized normal load and number of cycles on dominant failure modes, i.e. plastic deformation, cracking and wear, local spalling and continuous spalling in four different regions; A, B, C and D. In Table 1 the observed mechanisms are illustrated by simplified schematic drawings.

![Scratch induced coating failure map showing the influence of normal load and number of cycles on dominant failure modes, i.e. cracking/chipping, local spalling and continuous spalling.](image)

Figure 22. Scratch induced coating failure map showing the influence of normal load and number of cycles on dominant failure modes, i.e. cracking/chipping, local spalling and continuous spalling.

The ranking of the coatings investigated at low loads in the “interfacial fatigue area” is, keeping in mind that all coatings were deposited with identical interfacial properties, believed to be controlled by the differences in mechanical properties. Thus, the ranking with respect to spalling resistance obtained at low normal loads, i.e. TiSiN > TiAlN > AlCrO, can be explained by the fact that the TiSiN coating displays the highest hardness and AlCrO the lowest hardness. A high hardness is reducing the tendency to plastic deformation of the substrate material and thus the stresses at the coating-substrate interface.

The results show that the test method is quick, simple and reproducible and can preferably be used to obtain relevant data concerning the mechanical and tribological properties of different coating-substrate composites. Other advantages of the proposed test method are that the test method utilizes a commercially available tribometer and uses small, simple flat samples making it possible to easily evaluate e.g. the effect of different types of pre- and post coating deposition surface treatment processes, etc. Also, the small size
of the test region makes it possible to perform tests within small restricted areas. Consequently, the test method can be used as a virtually non-destructive test if the active region of the investigated sample is avoided. For example, it can be used to evaluate the cohesive and adhesive interfacial strength, fatigue and wear resistance of coated cemented carbide cutting tools prior to cutting tests in order to correlate the obtained data to the tool life.

In this test approach which comprises a large number of loading cycles, it is important to consider the extensive wear of the diamond stylus. In the present study, two high quality diamond styli with guaranteed spherical geometry were used and the wear/damage of these was restricted to mild polishing and initial "half-crown" shaped cracking after completing the test series. High scratching (sliding) speeds should preferably be avoided to decrease tribo-oxidation and graphitization of the tip. Further it is important to use high quality diamond tips with the [100] direction oriented along the z-axis and to do regular microscopic observations.
Table 1. *Observed coating failure mechanisms in the circular multi-pass scratch test.*

<table>
<thead>
<tr>
<th>Region A ($F_{\text{norm}} \leq 0.15$)</th>
<th>Mild plastic deformation, minor cracking and wear caused by chipping or delamination.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Coating properties of utmost import-</td>
<td>ance.</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Region B1 ($0.15 \leq F_{\text{norm}} \leq 0.20$)</th>
<th>Chipping and interfacial spalling controlled by <em>high cycle fatigue</em> ($N &gt; 1000$).</th>
</tr>
</thead>
<tbody>
<tr>
<td>Coating / interface properties of utmost import-</td>
<td>ance.</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Region B2 ($0.20 \leq F_{\text{norm}} \leq 0.25$)</th>
<th>Chipping and interfacial spalling controlled by <em>high cycle fatigue</em>, ($200 &lt; N &lt; 1000$).</th>
</tr>
</thead>
<tbody>
<tr>
<td>Coating / interface properties of utmost import-</td>
<td>ance.</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Region C ($0.25 \leq F_{\text{norm}} \leq 0.60$)</th>
<th>Chipping and interfacial spalling controlled by <em>low cycle fatigue</em>, ($10 &lt; N &lt; 200$).</th>
</tr>
</thead>
<tbody>
<tr>
<td>Interface properties of utmost importance.</td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Region D ($F_{\text{norm}} \geq 0.60$)</th>
<th>Chipping and interfacial spalling due to elastic recovery caused by pronounced plastic deformation of the cemented carbide substrate.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Interface/substrate material proper-</td>
<td>ties of utmost importance.</td>
</tr>
</tbody>
</table>
6.4 Abrasive wear resistance

The higher hardness of the coating the higher the wear resistance will be in abrasive contacts, that is a general rule. In the cutting process abrasive wear is common especially at the flank face but also at the rake face (zone C). Hard coatings such as Ti(C,N) and TiC are important coatings in order to increase the abrasive wear resistance in the cutting process. Although, also Al₂O₃, which above all is used due to the chemical stability and high hot hardness, has to have a high abrasive wear resistance. In present thesis the influence from other parameters than hardness were investigated, see Paper III and IV, by using tribological methods. Hence, as can be seen below phase and texture of the coatings as well as the use of a multilayered structure may influence the abrasive wear resistance.

6.4.1 Influence of phase and texture

Figure 23 shows the wear rates of four textured α-Al₂O₃ coatings and a κ-Al₂O₃ coating evaluated in the micro-abrasion tests using 6 and 1 µm abrasive diamond particles. As can be seen, for all samples the larger 6 µm diamond particles result in a wear rate more than one order of magnitude higher than for the finer 1 µm diamond particles. Generally the softer κ-Al₂O₃ coating shows a higher wear rate as compared with the textured α-Al₂O₃ coatings. Of the latter coatings, the (1010) textured α-Al₂O₃ coating shows a lower wear rate than the other textured α-Al₂O₃ coatings which show approximately the same wear rate.

![Figure 23. Abrasive wear rate of textured α-Al₂O₃ and κ-Al₂O₃ coatings tested against 1 µm (a) and 6 µm (b) diamond particles in the micro-abrasion wear test.](image)

In the tests, micro cutting (due to the cutting action of the diamond particles) and micro chipping (due to poor cohesive strength of the coating) were
found to be the dominant wear mechanisms and can be seen in Figure 24. The lower hardness of the κ-Al₂O₃ coating results in more pronounced abrasive scratches, i.e. the wear due to micro cutting becomes more aggressive as compared with the α-Al₂O₃ coatings. Also, the κ-Al₂O₃ coating displayed the highest tendency to micro chipping (see Fig 24c) especially for the larger 6 μm diamond particles. Thus, micro chipping is dominating in the κ-Al₂O₃ coating while micro cutting dominates for the α-Al₂O₃ coating. This indicates that the κ-Al₂O₃ phase may show lower fracture toughness as compared with the α-Al₂O₃ phase and that the difference between different α-Al₂O₃ textures is relatively small.

![Figure 24. Examples of dominating wear mechanisms, i.e. micro cutting and micro chipping, for two of the textured α-Al₂O₃ and the κ-Al₂O₃ coatings. For the α-Al₂O₃ especially the micro cutting is dominating (a) but for e.g. the 001-texture also micro chipping (c) is present to a large extent. The κ-Al₂O₃ (b) shows a worn surface where micro chipping to the largest extent is dominating.](image)

The reason to the differences in wear resistance between the textured coatings is not obvious. Probably the lower wear rate of the ⟨1014⟩ textured α-Al₂O₃ coating is due to a relatively low tendency to micro chipping as compared with the other α-Al₂O₃ coatings. Steijn [62] found that the wear rate of the prismatic and pyramidal planes of single crystals of sapphire (α-Al₂O₃) is significantly lower (50 times) as compared with the basal planes. The reason why the present study only reveal relatively small difference in wear rate between the different textured α-Al₂O₃ coatings (i.e. it is only the ⟨1014⟩ textured α-Al₂O₃ which display a significantly different (lower) wear rate) is probably due to the fact that polycrystalline α-Al₂O₃ structures most probably will behave differently as compared with single crystals. For ex-
ample, differences in grain size, morphology, degree of texturing, etc. will most probably influence the results obtained. Furthermore, the fact that the thermal cracks influence the wear show that local defects, increasing the local stress inducing cohesive failure, can be critical in controlling the wear rate.

6.4.2 Multilayer structures

As in the case for the textured $\alpha$-Al$_2$O$_3$ coatings and the $\kappa$-Al$_2$O$_3$ the abrasive wear resistance was evaluated in micro abrasion tests using 1 and 6 µm sized abrasive diamond particles for three multi layered coating structures. The wear rate is shown in Figure 25. For both types of abrasives $\kappa$-Al$_2$O$_3$ single layer coating shows a higher wear resistance as compared with the $\kappa$-Al$_2$O$_3$ multilayer coatings, which show an increasing wear resistance with increasing number of individual layers.

![Figure 25. Abrasive wear rate of $\kappa$-Al$_2$O$_3$ single layer and $\kappa$-Al$_2$O$_3$-Ti(C,N) multilayer coatings with 8, 15 and 32 individual layers tested against a) 1 µm and b) 6 µm diamond particles in the micro abrasion wear test.]

Detailed characterisation of the worn coatings by using SEM shows that two different wear mechanisms can be distinguished; *micro cutting* (due to the cutting action of the diamond particles) and *micro chipping/spalling* (due to poor cohesive/adhesive strength of the coating), see Fig 26. The 8-layered $\kappa$-Al$_2$O$_3$ multilayer coating shows a high tendency to micro chipping/spalling, while the 32-layered $\kappa$-Al$_2$O$_3$ multilayer coating shows a relatively low tendency. The $\kappa$-Al$_2$O$_3$ single layer coating did not display any
tendency to micro spalling at all. Hence, the wear rate of the coatings correlates with their tendency to micro chipping/spalling.

*Figure 26. Dominating wear mechanisms obtained for the κ-Al₂O₃-Ti(C,N) multi-layer coatings.*

The increase in wear resistance with increasing number of individual κ-Al₂O₃ layers is probably due to the fact that the volume fraction of the harder Ti(C,N) in the coating will increase with increasing number of individual κ-Al₂O₃ layers and that a finer multilayered structure will show a higher resistance to crack growth and thus micro chipping/spalling. Also, the detrimental effect of any residual tensile stresses in the coating will be lower in the case of a finer multilayered structure [63]. Nevertheless, since all multilayer coatings are prone to spalling the single layer κ-Al₂O₃ coating, not showing any micro spalling, display the lowest wear rate. Figure 27 shows porosity in the exposed interface obtained due to spalling and it is obvious that the occurrence of interfaces showing poor adhesion will have a detrimental impact on the wear resistance. However, the negative effect with interfaces may be compensated with the larger Ti(C,N) content when the number of layers is increased, as mentioned above.
6.5 Sliding wear resistance

In metal cutting the tribological contact is about steel sliding against the coating material and hence it is of interest to increase the understanding also in less severe contacts in order to get a basic knowledge. In present thesis (Paper V and Paper VII) especially the influence of topography is evaluated.

6.5.1 The influence of topography – vanadium based coatings

The fact that coating topography strongly influences the tribological performance of the tribosystem under sliding contact conditions is well known and is further illuminated by the results in Paper V and Paper VII.

In Paper V modified scratch tests were performed on V$_2$N and VN coatings, constituting a high amount of surface defects, with a counter material of stainless steel. The abrasive action of the protruding droplets does not only result in a high wear rate of the softer counter material but also in a high friction coefficient due to micro ploughing as well as material pick-up, resulting in an increasing friction since stainless steel slides against itself, see Fig 28.

For VN based coatings the formation of V$_5$O$_7$, a low friction Magnéli phase, at the sliding interface is known to improve the friction characteristics of the coating. However, a prerequisite for this is a smooth coating surface in order to reduce the ploughing effect of hard coating asperities in the softer counter materials. AES analysis of post-polished coating surfaces exposed to a tribological contact proofs the formation thin vanadium based oxide films which thus may explain the low initial friction coefficients obtained in the sliding tests. The AES line profile, see Figure 29, is obtained within a small selected region within the scratch track on the V$_2$N coated sample showing
transferred stainless steel and V$_2$N coating free from transferred steel. However, due to a relatively large number of defects, i.e. craters, still being present in the polished coating surface the friction coefficient will increase with increasing number of passes due to an increased adhesive wear since stainless steel is transferred to craters, see Fig 28b, in the polished coating surface. Thus, the negative effect of a rough surface will far outweigh the potential positive effects of a lubricious vanadium oxide, i.e. V$_x$O$_y$, at the sliding interface.

![Figure 28](image)

**Figure 28.** The friction coefficient (a) for the coatings investigated changing regarding to number of passes. For the polished coatings $\mu$ increases in the repeated contact due to the material transfer in connection to craters (b).

![Figure 29](image)

**Figure 29.** The AES line profile (a) is obtained within a small selected region (b) within the scratch track on the V$_2$N coated sample showing transferred stainless steel and V$_2$N coating free from transferred steel. The formation of a V$_x$O$_y$ is illustrated.

### 6.5.2 The influence of topography – alumina coatings

In Paper VII the friction characteristics of three Al$_2$O$_3$ coatings with different surface micro topography were evaluated in sliding contact with quenched and tempered steel. While the as-deposited coating (A) shows a high initial friction coefficient, see Figure 30, which increases to a steady-state value of
\( \mu = 0.7 \) the post-polished coatings show a relatively low initial friction coefficients and a less pronounced increase in \( \mu \) with increasing number of sliding cycles, see Fig. 30b. This is especially the case for the Al\(_2\)O\(_3\) coating with the smoothest surface (P3) which displays a low and stable friction coefficient during the first 300 cycles. This gives that \( \mu_{\text{adhesion}} \) is in the range of 0.10 for Al\(_2\)O\(_3\) while \( \mu_{\text{ploughing}} \), which is dependent on the coating surface topography, varies between 0 (P3) to 0.40 (A) for the different coating topographies. Also the wear rate of the counter steel material is lower for the polished coatings, especially initially.

The steady-state friction coefficients displayed by the different Al\(_2\)O\(_3\) coatings are all relatively high. For the as deposited coating the major reason for this is the steel material transfer and hence a contact with steel sliding against itself. For the smoothest coating (P3) the material transfer is relatively low and the tendency to form a tribofilm will increase the friction as well as the wear rate of the steel material. Thus, the consequence of the formation of a tribofilm has the opposite effect as compared to the PVD V\(_x\)N-coatings where the tribofilm is known to decrease the friction.

![Figure 30](image)

**Figure 30.** The friction coefficient, \( \mu \), versus the sliding distance for a) the A and P3 surfaces during the entire testing sequence and b) the A, P1 and P3 surfaces the first seven per cent of the running.

### 6.6 Correlation between laboratory tests and cutting tests

In order to increase the understanding of the tribological mechanisms active in metal cutting a number of laboratory tribological tests are frequently used. Even though the contact conditions differ a lot between such tests and metal cutting, tribological testing is still very common and useful since it will give information about the general tribological response of the mating surfaces and promotes post-test microscopy and surface analysis. In Paper III, VII and VIII metal cutting tests have been performed in order to evaluate the reality and the correlation between tribological testing and metal cutting.
6.6.1 Abrasive wear

Figure 31 shows the flank wear rate of the coated cutting tools in a lifetime metal cutting test. As can be seen, the flank wear increases with increasing number of layers of the multilayered \( \kappa \)-Al\(_2\)O\(_3\) coated tools for all cutting speeds investigated. However, the single layer \( \kappa \)-Al\(_2\)O\(_3\) coated tools show the highest wear, i.e. the opposite trend as observed in the micro abrasion test.

![Figure 31](image)

Figure 31. Flank wear rate of \( \kappa \)-Al\(_2\)O\(_3\) single layer and \( \kappa \)-Al\(_2\)O\(_3\)-Ti(C,N) multilayer (8, 15 and 32 individual layers) coated tools in the cutting tests.

The reason why the flank wear of the multilayered \( \kappa \)-Al\(_2\)O\(_3\) tools increases with increasing number of individual \( \kappa \)-Al\(_2\)O\(_3\) layers is not obvious but the following explanations can be considered:

- coating composition
- \( \kappa \)-Al\(_2\)O\(_3\) \( \rightarrow \) \( \alpha \)-Al\(_2\)O\(_3\) phase transformation
- interfacial adhesion

Since Ti(C,N) displays a more drastic drop in hot hardness as compared to Al\(_2\)O\(_3\) in the temperature range of interest it is believed that the strengthening effect (observed at RT) of Ti(C,N) layers within the multilayer coatings will decrease with increasing temperature. Also, the possible phase transformation of \( \kappa \)-Al\(_2\)O\(_3\) to \( \alpha \)-Al\(_2\)O\(_3\), associated with a volume decrease of about 8% and thus a reduced cohesive strength, may also play an important role in the cutting tests (due to flash temperatures). Hence, the advantages seen at RT with a finer multilayered structure are diminished in metal cutting. Instead interfaces with poor interfacial adhesion have a detrimental effect due to the interfacial porosity.
The higher wear rate of the single layer $\kappa$-Al$_2$O$_3$ coated tools is probably due to a coarser microstructure as compared with the $\kappa$-Al$_2$O$_3$-Ti(C,N) multi-layer coatings. Here the drawbacks of the interfaces are compensated by a finer structure when comparing one and several layers.

Although abrasion seems to be an important wear mechanism on the flank in the cutting test no correlation is obtained when comparing the results obtained in the micro-abrasion and cutting tests. The main reason is the huge difference in temperature. It is believed that, while fracture mechanical failure mechanisms mainly control the wear in the micro-abrasion test these mechanisms are of less importance at higher temperatures where the coatings behave more ductile.

Nevertheless, the micro abrasion test is believed to obtain information about the wear resistance of CVD coatings used in low temperature applications as well as CVD coatings on cutting tools where fracture mechanical failure mechanisms such as micro chipping and spalling are of more importance, e.g. in intermittent cutting operations.

6.6.2 Influence of topography

The influence of surface micro topography on the tribological behaviour in sliding contact and metal cutting conditions were evaluated in Paper VII and VIII. Despite the different contact conditions, several similarities exist especially when it comes to the mechanical interaction between the surfaces and the tendency to material transfer and pick-up. Thus, in common for the conditions prevailing at the sliding interface in pin-on-disc testing and at the rake and flank faces in metal cutting is an increasing material pick-up tendency with increasing surface micro topography. In contrast, the influence of surface micro topography on the contact conditions displayed significant differences.

The results of the pin-on-disc tests show that the surface condition of a hard coating in sliding contact with a softer counter material has a strong impact on both friction and wear of the tribo system. Thus, during initial sliding a smooth coating surface significantly reduces the friction coefficient, the wear of the counter surface and the tendency to material transfer. However, with increasing sliding distance these positive effects usually decreases due to the formation of initiation of material transfer and formation of tribo layers, e.g. layers of oxidized counter material as observed in the present study.

The tribological contact conditions at the rake face of a cutting tool are characterized by high temperatures and high contact stresses. Also, the tendency to sliding between the rake face and the chip varies significantly over the rake face, ranging from pure sticking in zone (a) close to the cutting edge to pure sliding in zone (c) close to the end of the rake face-chip contact.
The results obtained clearly show that the length of the individual contact zones (a)-(c) changes with decreasing topography, i.e. a smoother coating surface increases the length of zones (a) and (b) and decreases the length of zone (c). Consequently, the contributions from sticking and sliding will vary depending on the surface micro topography. This makes it very difficult to calculate the contact conditions from the cutting forces and to draw any conclusions regarding the influence of surface micro topography on the contact conditions at the rake face-chip interface.

However, it was found that the surface micro topography has a significant impact on the chip surface in direct contact with the rake face. Hence, a smoother coating surface results in a smoother chip surface, a thinner subsurface deformation layer and a lower chip temperature, results indicating relatively low contact conditions in zone (c). In combination with a shorter sliding distance the above results indicates a reduced frictional work, at least in zone (c), with decreasing surface micro topography.

Figure 32 shows the contact conditions at the rake face calculated with two different methods, i.e. by measuring the obtained chip compression ratio (a) and by using the linear fitted versions of the cutting forces (b). Note that the graphs correspond to a chamfered and a standard tool respectively in order to show the most pronounced similarities. As expected the different methods used in the calculations of the contact relationship show a significant variation in the results obtained at equal values of the process parameters. The real value is thought to be somewhere in between the values obtained by using the two different experimental methods investigated. The difference is probably mainly due to the existence of a stagnation point and the difficulties in measuring the cutting forces on the rake face due to this phenomenon. However, the general trend seems to be that a rough coating surface gives a higher contact condition as compared to a smooth surface.

![Figure 32](image)

*Figure 32. The contact conditions, $\mu_r$, at the rake face calculated with two different methods, i.e. by measuring the obtained chip compression ratio (a) and by using the linearized versions of the cutting forces (b).*
The tribological contact conditions at the flank face of a cutting tool are less aggressive thus promoting more uniform contact conditions. Again, the results obtained clearly show that the length of the individual contact zones (a) and (b) changes with decreasing topography and that a smoother coating surface increases the length of zone (a) and decreases the length of zone (b). Also, a smoother coating surface results in a smoother machined work material surface and a thinner sub-surface deformation layer. Consequently, besides known parameters affecting the workpiece roughness, e.g. vibrations in the cutting process or the tool geometry, present study shows that also the coating surface micro topography of the tool will have an impact. However, despite these positive effects the calculations show that the contact conditions decreases with increasing surface micro topography, see Figure 33.

The reason for this is not fully understood. However, Figure 34 showing the influence of micro surface topography on the mean normal and shear stresses at the flank face illustrates that the increase in contact conditions, $\mu_{cl}$, is due to a significant increase in normal stress with increasing micro surface topography. The reason for this not obvious but may be related to the deformation of the workpiece material in the contact zone. A rough surface results in inhomogeneous deformation of the workpiece material and a larger sub-surface deformation layer, and this means that more workpiece material will flow against the flank. A larger amount of workpiece material sliding against the flank face results in an increased hydrostatic pressure and hence an increased normal pressure, i.e. a rougher surface results in a higher normal pressure on the flank face. Further, it is possible that the increase in length of the sticking zone (zone (a)) for the smoothest coatings may have a negative impact on the contact conditions, i.e. the plastic flow in the chip may play an important role.

The sliding contact in the pin-on-disc test configuration can be characterized as a “lightly” loaded sliding system where the real area of contact, $A_r$, is restricted to plastically deformed surface asperities and consequently significantly smaller than the apparent area of contact, $A_p$. In contrast, metal cutting can be characterized as a “heavy” loaded sliding system where plastic deformation extends into the bulk material and the real, $A_r$, and apparent, $A_p$, areas are believed to be more or less identical. While Amontons law holds for the former lightly loaded conditions this is most probably not the case for the latter heavy loaded conditions. Consequently, the influence of surface micro topography on the contact conditions may differ under pin-on-disc sliding contact and metal cutting conditions, respectively.

Finally, it should be commented that, due to the short machining times, no influence of surface micro topography on coating wear could be observed. However, since a smoother coating surface reduces the mechanical interaction with the workpiece material it is believed that a reduced surface micro topography also is likely to increase the coating life time.
Figure 33. The contact conditions, $\mu_{cl}$, at the flank face as a function of coating surface micro topography.

Figure 34. The mean values of the normal and shear stresses at the flank face as a function of the coating surface micro topography.
7. Main conclusions of thesis work

Chemical and Physical Vapour Deposition, being two atomistic coating deposition processes, makes it possible to deposit and design wear resistant coatings on cutting tools for improved performance and life-time. The present work has contributed to an increased understanding regarding the relationships between composition and microstructure, resulting properties and tribological performance of a number of commercial CVD and PVD coatings aimed for metal cutting applications.

In the work focusing on CVD Al₂O₃ it has been shown that the nucleation and growth of α-Al₂O₃ can be obtained on a Ti₄O₇ template with a subsequent phase transformation of the titanium oxide. When the template is rather thick it transforms from Ti₄O₇ to a Ti₃O₅ phase but when it is composed of a thinner gradient layer it transforms to Ti(C,N,O). In common for these phase transformations is the formation of pores at the corresponding interfaces which will affect the adhesive interfacial strength. These results illuminate the importance of an increased knowledge regarding the nucleation of α-Al₂O₃ and the possibility to control the resulting microstructure.

The importance of the possibility to accurately control the crystallographic phases, e.g. α-Al₂O₃ rather than κ-Al₂O₃, textures and multilayer structures was illuminated in the work focusing on abrasive wear resistance of Al₂O₃. It was found that the abrasive wear resistance of α-Al₂O₃ was significantly higher as compared to that of κ-Al₂O₃. This is most probably due to a lower hardness of κ-Al₂O₃ and a higher tendency to micro chipping during the abrasive contact. When comparing textured α-Al₂O₃ coatings only small differences in wear resistance was observed, the ⟨1014⟩ textured α-Al₂O₃ coating showing the highest wear resistance due to a slightly higher resistance to micro chipping. For κ-Al₂O₃–Ti(C,N) multilayer coatings the wear resistance was found to increase with decreasing layer thickness. In summary it was found that the abrasive wear of Al₂O₃ is due to a combination of micro cutting, micro chipping and micro spalling. Also, the presence of interfacial porosity in the coatings was found to significantly reducing the wear resistance.

In the work focusing on cathodic arc evaporated PVD coatings the presence of defects, i.e. macro particles, at the substrate-coating interface was found to have a significant negative impact on the substrate-coating adhesive strength, interfacial fracture toughness and fatigue resistance. Also, the presence of μm-sized macro particles on the surface may result in high friction
coefficients and material transfer during sliding contact against a softer counter material.

The new circular multi-pass test approach evaluated is quick, simple and reproducible and can preferably be used to obtain relevant information concerning the adhesion, fatigue and wear characteristics of different coating-substrate composites. When evaluating three different commercial PVD coatings it was possible to distinguish these with respect to fatigue behaviour, i.e. TiAlN displayed the the best performance under low cycle fatigue conditions while the harder TiSiN displayed the best performance under high cycle fatigue conditions.

Despite the different contact conditions, the influence of surface microtopography on the tribological behaviour in sliding contact and metal cutting conditions have much in common. Several similarities exist especially when it comes to the mechanical interaction between the surfaces and the tendency to material transfer and pick-up. In contrast, the influence of surface microtopography on the friction characteristics differs substantially. At the flank face the contact conditions increases with decreasing coating topography while it on the rake face seems to be an optimum value at a certain surface roughness. Also the roughness of the workpiece material as well as the mechanical interaction is increasing with increasing coating surface topography.

Finally, although field tests, i.e. metal cutting tests, will give the final answer regarding the tribological performance of coated cutting tools the use of well defined laboratory tests focusing on some specific coating failure or wear mechanism is of utmost importance in the development of new coating systems. In the present work this was illuminated by the use of the micro-abrasion test and the multi-pass scratch test. In common for these tests is the simplicity of using a small sample size which makes the test cheap and easy to combine with post-test microscopy and surface analysis. Especially, the small size of the test region (φ < 1 mm if necessary) makes it possible to perform tests within small restricted areas, i.e. both tests can be regarded as virtually non-destructive tests. Consequently, these types of laboratory tests have a significant potential when it comes to perform screening tests of a large number of coatings.
8. Future work

In the never ending work to increase the understanding of the tribological behaviour for coated cutting tools there is plenty of phenomena to evaluate.

Regarding the controlled nucleation and growth of $\alpha$-Al$_2$O$_3$ different titanium oxides are known to promote the growth of the $\alpha$-Al$_2$O$_3$ phase. However, it is still not known which type of oxide or morphology that will give the best adhesion and nucleation of $\alpha$-Al$_2$O$_3$. Also, the influence of trace elements such as Cl, Fe and Cr on the nucleation process is not fully understood. Hence, chemical and phase analysis in combination with adhesion tests of other bonding and template layer structures should be done in the future. For example, bonding layers of different chemical composition and morphology should give different templates and adhesion characteristics.

Industrial cathodic arc evaporation processes are usually not filtered and consequently the deposited coatings will contain a significant number of defects which may be detrimental in some cutting applications depending on the contact conditions. This is also a research area of interest especially in the case of very hard coatings which may be more sensitive to intrinsic defects. In this work the newly proposed multi-pass scratch test may play an important role. The possibility to run these tests at elevated temperatures should be evaluated.

The influence of the coating surface topography on the performance and life-time of coated cutting tools is a research area of great potential. Thus, the impact of coating surface topography on the cutting forces, the chip formation process, the work material surface integrity and especially the coating wear rate and cutting tool lifetime should be investigated for a number of important cutting tool / work material combinations. For example, quick-stop tests and subsequent detailed characterisation with e.g. TEM should be very interesting to perform in order to further investigate the chip flow.
9. Sammanfattning på svenska (Swedish)

Mikrostrukturell, mekanisk och tribologisk karakterisering av CVD och PVD beläggningar för skärande bearbetning

Skärande bearbetning är en av de äldsta och vanligaste metoderna för formning av komponenter i den tillverkande industri. Under denna benämning finns metoder som svarvning, borrning och fräsning och det gemensamma är att material avverkas genom bildandet av en spåna. Trots att skärande bearbetning står för ungefär 15 % av det totala värdet av världens tillverkade mekaniska komponenter så är den en av de minst förstådda tillverkningsmetoderna. Den största orsaken till detta är processens komplexitet som resulterar i extrema tribologiska förhållanden med höga tryck och temperaturer.

Ordet "Tribologi" innebär "Läran om friktion, nötning och smörjning" och är en vetenskap som har mycket stor betydelse när ytor växelverkar i en relativrörelse – d.v.s. i alla fysiska rörelser i vår omgivning. Friktion finns alltså överallt runtomkring oss och uppstår på grund av motståndet mot en rörelse när en yta rör sig mot en annan, vilket ofta resulterar i någon typ av nötning av en eller båda ytorna. Friktion är inte en materialparameter utan en systemparameter och beror av vitt skilda faktorer såsom ytegenskaper, topografi, temperatur, tryck, glidhastighet, atmosfär etc. På samma sätt beror nötningen, d.v.s. avverkning av material, av hela systemets egenskaper och inte av en eller två materialparametrar.

Vart man än blickar finns det ytor som glider mot varandra i ett närmast oändligt antal olika vardagliga kontakter. Det kan vara skridsko mot is, sko mot asfalt, bromsar i bilen, hjullager till cykeln, gångjärn till dörren etc. etc. Alla ger vitt skilda tribologiska förhållanden, men det gemensamma är att otroligt mycket sker i själva kontaktpunktorna. I mikro- och nanoskala deformeras yta extremt mycket och delar av materialet kan smälta, vilket även sker i den mildaste av kontakter. Ju aggressivare förhållanden blir desto mer utpräglad blir växelverkan mellan ytorna och ännu mer sker i mikro-nanoskala. Dessa fenomen är därför mycket utpräglade i extrema kontakter som till exempel den aktuella i svarning. Här ska ett litet skär fullständigt omforma en hård stålklump till en fulländad detalj och all energi om- sätts på några få mm². Onekligen blir miljön mycket aggressiv med höga tryck och temperaturer. Skäret möter kontinuerligt nytt och reaktivt arbetsmaterial och kontakten kännetecknas av utpräglad kemisk upplösning, plas-
tisk ytdeformation samt kraftig repning, vilket ställer höga krav på det för-
mande skäret.

Härdmetall som innehåller en stor mängd hårdare karbider (WC) och en
metallisk bindefas (Co) används ofta som skärssubstrat. För att förbättra de
tribologiska egenskaperna deponeras mikrometerutnna skikt såsom Al₂O₃,
Ti(C,N) eller TiSiN genom kondenseringsoch/eller kemiska reaktioner i gas-
form. Sådana tekniker är vanligtvis “chemical vapour deposition” (CVD)
och “physical vapour deposition” (PVD).

Så, genom att använda en tunn beläggning av ett nötningsbeständigt mate-
rial fås de bästa egenskaperna med avseende på friktion och nötning där de
behövs som mest, medan substratet fungerar som lastbärare. Substratets roll
är alltså framförallt att stå emot större brottmekaniska skador och föränd-
ring av skärgeometrin, medan beläggningen kan designas med fokus på att
öka motståndet mot den tribologiska nedbrytningen. Ytterligare en aspekt
som icke är att förövrama då en beläggning deponeras på ett substrat är att
det skapas en gränsyta vars styrka, dvs. vidhäftning, är mycket viktig för att
beläggningen inte ska flaga av under ingrepp. Vad gäller den vidhäftning
spelar de atomära bindningarna i gränsytan en central roll, men även syste-
mets möjligheter att reducera gränsytans skjutspänningar är otroligt viktiga.
För att ett skär ska fungera tillfredsställande måste det alltså designas med
hänyn till både substrat, gränsyta och beläggning och dess egenskaper be-
höver optimeras mot varandra. Därmed bör substratet framförallt ha hög
hållfasthet och seghet medan beläggningen bör vara hård, kemiskt stabil och
nötningsbeständig. Termisk längdutvidgning, skjuthållfasthet och elasticitet
spelar också en stor roll och med avseende på gränsyta bör alla ovanstående
egenskaper kombineras på bästa sätt för att minska belastningen i denna del.
Vidare styrs nämnda egenskaper hos beläggningen av en rad faktorer som
t.ex. kemisk sammansättning, typ av fas, tjocklek, textur, mikrostruktur,
porositet, mängd defekter, restspänningar och yttopografi. I sin tur styrs dessa
processparamettern vid deponering, bl.a. temperatur, tryck och gasflö-
den, och förståelse för relationen mellan dessa processparamettern, beläg-
ningskaraktistik och slutligen mekaniska och tribologiska egenskaper är
mycket viktig för att kunna designa funktionsdugliga skär.

För att erhålla en beläggning med optimala egenskaper är det bl.a. viktigt
att beläggningsmaterialet består av den bäst lämpade kristallografiska fasen.
Vad gäller Al₂O₃ som är den vanligaste beläggningen vid svarvning så an-
vänds två faser som kallas alfa- respektive kappafasen (α- Al₂O₃ resp. κ-
Al₂O₃). I skärtekniska processer är alfafasen den absolut bästa av dessa två
men är mycket svårare att deponera i CVD-kammaren och kräver en speciell
yta att kärnbilda på. Vad denna yta består av och varför den gynnar alfafasen
är tämligen outrett. Hur som helst så visar denna avhandling att alfafasen kan
kärnbildas och växa på Ti₄O₇. Beroende på beläggningssystemets uppbygg-
nad fasonvandlas titanoxiden sedan till Ti₃O₅, då nuklearingssskiktet (tem-
plate) är relativt tjock, respektive Ti(C,N,O), då det är tunt, när Al₂O₃ depone-
ras ovanpå. Då det tjockare skiktet omvandlas till Ti₃O₅ bildas en stor mängd porer i underliggande gränsyta vilket kraftigt reducerar vidhäftningen. För det tunnare skiktet används istället en gradientstruktur med bl.a. ett underliggande bindeskikt och gränsytorna är mindre skarpa. Detta förbättrar vidhäftning avsevärt men i de områden då avflagning ändå förekommer har det visat sig att en jämn yta hos underliggande skikt har en negativ inverkan; d.v.s. en liten kontaktaera ger färre bindningsmöjligheter.

Begreppet vidhäftning är som sagt komplex och involverar förutom den kemiska vidhäftningen mellan de använda materialen även systemets möjligheter att minska belastningen i gränsytan. Därmed spelar t.ex. hårdhet, E-modul och seghet en central roll. För PVD-beläggningar har det visat sig att defekter såsom droplets i gränsytan i kombination med en hög hårdhet har en stark negativ inverkan på vidhäftningen. I en jämförelse mellan arc-förångad V₃N och VN uppvisade den hårdare och mer defektra (droplets och craters) V₃N ett betydligt sämre motstånd mot avflagning. Dessa defekter visade sig också ha en negativ inverkan på friktionen i en glidande kontakt mot stål. Även om beläggningen är finpolerad och en lågfriktionstribofilm (vana-dinoxid) bildas så påverkar de kvarvarande defekterna (craters) påklettnings-tendenser av stål och därmed friktionen negativt i större utsträckning än vad de positiva effekterna av en tribofilm gör.

För att utvärdera de tribologiska egenskaperna i en så realistisk kontakt som möjligt har en ny metod för multi-reptest utvärderats där information om utmattning, nötning, urflisning och avflagning hos beläggningsystem erhålls. Metoden är snabb, enkel och reproducerbar och dessutom är testområdet som krävs litet vilket gör det möjligt att utföra tester mycket nära ingreppsområdet utan att förstöra funktionen hos skäret. Av de testade PVD-beläggningarna uppvisar TiAlN bäst prestanda vid lågcykelutmattning medan TiSiN gör det vid högcykelutmattning. Beläggningarna har samma bindeskikt mot substratet och därmed samma vidhäftning. De uppmätta skillnaderna beror således framförallt på skillnader i beläggningsmaterialens egenskaper (H, E, etc.).

En annan egenskap som har en central roll i skärande bearbetning är motståndet mot abrasiv (repande) nötning och vanligtvis spelar beläggningens hårdhet en stor roll. Hur som helst så har det påvisats att faktorer som fasssammansättning, textur och beläggningsuppbyggnad också har en stor inverkan för t.ex. CVD Al₂O₃. Alfafasen uppvisar ett betydligt bättre nötningsmotstånd än kappafasen och om dessutom texturen hos alfafasen styrs så kan nötningmotståndet ytterligare optimeras; (104) är t.ex. bättre än (001). För dessa material inverkar tendensen till mikrourflisning till stor del på nötningmotståndet. För en multilagrad struktur (Ti(C,N)- κ-Al₂O₃) fås även avflagning av delskikt, p.g.a. porositet i gränsytorna, vilket ger ett sämre nötningmotstånd jämfört med enkelskikt. Jämfört med ett litet antal lager ger dock många lager bättre nötningmotstånd framförallt p.g.a. att fördelar-
na med en fin mikrostruktur och större mängd hård Ti(C,N) uppväger nackdelarna med många gränsytor.

Vid skärtest är det betydligt fler faktorer som spelar roll jämfört med laboratorietester och med avseende på det abrasiva nötningsmotståndet blir resultaten precis de motsatta. När däremot inverkan av belägningstopografri utvärderas förekommer det likheter; en grov yta ger betydligt större tendens till påkletning av stål jämfört med en finpolerad yta samt resulterar i en grövre yta hos det bearbetade arbets materialet och en mer utpräglad växelverkan mellan ytorna. Hur som helst så gör komplexiteten i skärprocessen att liknande mekanismer ändå kan påverka kontaktförhållandena olika. På släppningssidan ökar kontaktförhållandet ju finare beläggningens yttopografi är medan det på spånsidan tycks finnas ett optimalt värde för en medelfin yta.

Denna avhandling ökar på många sätt förståelsen för hur egenskaperna hos beläggningsmaterial, såväl som hos belägningssystem, påverkar dess tribologiska uppträdande och därmed svarvskärs funktion. Denna kunskap leder i bästa fall till framtidens belägningssystem som uppvisar lägre friktion och högre nötningsmotstånd vilket i skärande bearbetning resulterar i lägre produktionskostnader, bättre skärprestanda och en mindre påverkan på miljön.
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