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Surface Engineering in Sheet Metal Forming

PER CARLSSON

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Abstract

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In recent years, surface engineering techniques have been developed in order to improve the tribological performance in many industrial applications. In sheet metal forming processes, the usage of liquid lubricants can be decreased by using self lubricated tribo surfaces which will result in more environmentally friendly workshops. In the present work two different concepts, i.e. the deposition of thin organic coatings on the steel sheet and PVD coatings on the tool, have been evaluated. The sheet materials investigated include Zn and 55%Al-Zn metal coated steel sheet, which in general are difficult materials to form under dry conditions since they are sticky and thus have a high tendency to adhere to the tool surface. The PVD coatings include CrN, TiN and various DLC coatings. The work comprises tribo testing and post test characterisation using surface analytical techniques in order to evaluate the tribological properties of the tribo surfaces. The tribological tests of different tribo couples were conducted by using modified scratch testing and ball-on-disc testing. From these test results different friction and wear mechanisms have been identified.

The deposition of thin organic coatings on the steel sheet metal has been found to be promising in order to control the friction and to avoid metal-metal contact resulting in galling. However, it has been found that the tribological characteristics of organic coated steel sheet are strongly influenced by coating chemical composition, the substrate surface topography and the coating thickness distribution.

The performance of the PVD coatings depends mainly on the chemical composition and topography of the coated surface. By choosing PVD coatings such as diamond like carbon (DLC) low and stable friction coefficients can be obtained in sliding contact against Zn. Surface irregularities such as droplet-like asperities may cause an initial high friction coefficient. However, after a running in process or by polishing the PVD coating low friction coefficients can be obtained resulting in a stable sliding contact.

The combination of imaging (optical profilometry, LOM, SEM) and chemical analytical techniques (EDS, AES, ToF-SIMS) gave valuable information concerning the friction and wear properties of the tribo surfaces investigated.

Keywords: Friction, wear, metal coated steel sheet, dry lubricants, galling, PVD coatings

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ENCLOSED PAPERS

This thesis comprises the following papers, which in the summary will be referred to by their Roman numerals.

I P. Carlsson, U. Bexell and M. Olsson
Tribological behaviour of thin organic permanent coatings deposited on hot-dip coated steel sheet - a laboratory study
Surface and Coatings Technology 132 (2000) 169-180

II P. Carlsson, U. Bexell and M. Olsson
Friction and wear mechanisms of thin organic permanent coatings during sliding conditions
Wear 247 (2001) 88-99

III P. Carlsson, U. Bexell and M. Olsson
Tribological performance of thin organic coatings deposited on galvanized steel – influence of coating composition and thickness on friction and wear
Wear 251 (2001) 1075-1084

IV U. Bexell, P. Carlsson and M. Olsson
Tribological characterisation of an organic coating by the use of ToF-SIMS
Applied Surface Science, 203-204 (2003) 596

V P. Carlsson and M. Olsson
Tribological behaviour of thin organic coatings on 55%Al-Zn steel sheet - influence of transfer and tribo film formation
Submitted to Wear

VI P. Carlsson and M.Olsson
Improved anti-galling properties in sheet metal forming by the use of surface engineering
Proceedings of Nordtrib 2002, 10th Nordic Symposium on Tribology, Stockholm, Sweden, June 9-12, 2002

VII P. Carlsson and M. Olsson
PVD Coatings for sheet metal forming processes –
a tribological evaluation
Surface and Coatings Technology, in press

The author's contribution to the papers is as follows:

I, II	Part of planning, experimental work, major part of evaluation
III	Major part of planning, experimental work, evaluation
IV	Part of planning, evaluation
V, VI, VII	Major part of planning, all experimental work and evaluation (except ToF-SIMS)

The following papers have also some relevance to this work although they are not included in the thesis:

A P. Carlsson, U. Bexell and M. Olsson
Automatic scratch testing - a new tool for evaluating the stability of tribological conditions in sheet metal forming
Proceedings of GALVATECH 2001, 5th International Conference on Zinc and Zinc Alloy Coated Steel Sheet, Brussels, Belgium
June 26-28, 2001

B P. Carlsson, U. Bexell and S.E. Hörnström
Corrosion behaviour of Aluzink® with different passivation treatments
Proceedings of GALVATECH 2001, 5th International Conference on Zinc and Zinc Alloy Coated Steel Sheet, Brussels, Belgium
June 26-28, 2001

C P. Carlsson, U. Bexell, M. Olsson and H. Klang
A study of the initial stages of atmospheric corrosion of formed hot dip zinc coated steel
Proceedings of EUROCORR -97, Trondheim, Norway
September 22-25, 1997

D P. Carlsson, U. Bexell, M. Olsson and H. Klang
Initial atmospheric corrosion behavior of formed hot dip zinc coated steel - an energy dispersive x-ray spectroscopy study
Proceedings of SCANDEM -97, Göteborg, Sweden
June 10-13, 1997

E U. Bexell, P. Carlsson and M. Olsson
Characterisation of thin films of a non-organofunctional silane on Al-43.4Zn-1.6Si alloy coated steel by ToF-SIMS
Proceedings of the 12th International Conference on Secondary Ion Mass Spectrometry (SIMS XII), Brussels, Belgium
September 5-10, 1999

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PREFACE

This thesis reflects my scientific work carried out at Dalarna University. The financial support was provided by the Swedish Foundation for Knowledge and Competence Development (KK-stiftelsen), SSAB Tunnplåt AB, Becker Industrial Coatings AB and Chemetall Skandinavien Ytteknik AB. Dr Greger Håkansson, Bodycote Värmebehandling AB, and Dr Mats Larsson, Balzers Sandvik Coating AB, are acknowledged for providing the PVD coatings and valuable information. Dr Heribert Domes, Chemetall GMBH, is recognised for providing the thin organic coatings.

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Last, but not least I would like to thank my family for always being there.

Borlänge, January 2005

Per Carlsson

1

INTRODUCTION

1.1 Background

Steel is the most widely used material in the world owing to its versatility and low cost. Steel is therefore one of the basic ingredients in the development of industry and the whole society. Carbon steel sheet is used in the automotive, appliance and building industries. New applications of high strength steels in automotive and other sheet metal forming industries have placed increased demands on the forming capability of these steels. For different applications for duty in aggressive atmospheres, the steel sheet has to be coated by a protective coating. The most important anti-corrosion coatings are various types of zinc coatings. Zinc coatings can be divided into hot-dip galvanized and electrogalvanized coatings the most common chemical compositions are pure zinc coatings and zinc-alluminium alloyed coatings. Continuously hot-dip metallization is the most cost effective technique to protect steel sheet and have been used since the 60´s. The coating must have a good formability and a good adhesion to the steel substrate to survive severe forming operations in the press shop. Pure zinc coatings are soft and ductile which give them excellent formability properties. However, soft coatings have a high tendency to adhere to the forming tool which after several successive forming cycles can lead to surface problems such as scratches or, even worse, cracks in the formed product. This surface related problem is often called galling (Figure 1) and is generally eliminated by lubricating the steel sheet with a lubricating oil. Before painting the oil must be removed by using more or less unhealthy cleaning agents.

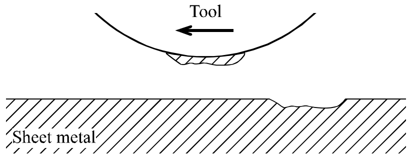


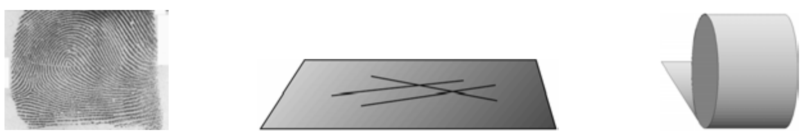
Figure 1. Schematic showing galling in sheet metal forming.

1.2 Motivation

In recent years the demands on the environment have been of increasing importance. Consequently, concepts able to substitute the lubricating oils are of outmost importance. Finding a winning solution for the intricate problem would be a break through and will result in more environmental friendly workshops.

The problem is a typical tribological problem, where the interaction mechanisms between two surfaces in relative motion have to be identified. To solve the tribological problem a basic understanding of the tribological conditions prevailing during sheet metal forming is important, not only for specific tool/sheet metal forming operations, but also for the development of new surface engineering concepts able to reduce problems such as high friction forces and high tendencies to galling.

In sheet metal manufacturing high requirements are placed on the surface finish. Not only for aesthetic reasons but also in order to obtain a surface topography that is optimal in a forming point of view. Besides a corrosion attack the surface may also be affected by mechanical degradation, *e.g.* by localized damage such as scratches, during fabrication or transportation due to the low hardness of the coatings (Figure 2). Moreover, hot-dip Zn coatings are sensitive to fingerprints. All these surface related problems can be eliminated or significantly reduced by protecting the surface in different ways. The corrosion protection can be enhanced by applying chromates or oils on the surface. The galling problem is often eliminated by using an excess of lubricating oils. These solutions are relatively toxic and consequently have a negative impact on the environment and therefore development of new environmentally friendly concepts to solve these problems is necessary.



Finger print — Scratches — Crevice corrosion in a steel strip coil

Figure 2. Surface related problems for hot-dip Zn coated steel sheet.

Except for liquid lubrication mainly two concepts exists able to reduce friction and wear at the sheet metal/forming tool interface, see Figures 3a and b. The first focusing on the sheet metal, *i.e.* the deposition of a thin dry lubricant on the sheet metal, the second focusing on the tool, *i.e.* the deposition of a thin low friction and wear resistant PVD coating [1, 2].

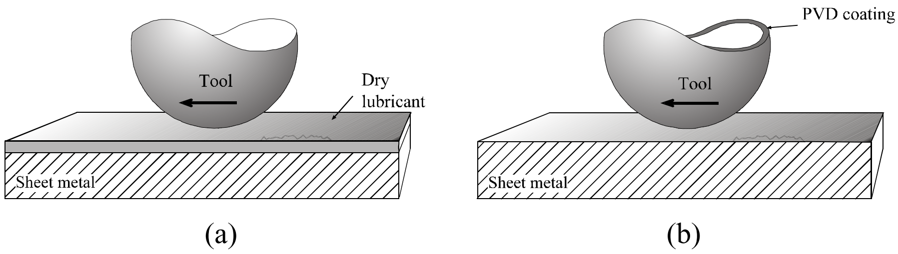


Figure 3. Surface engineering concepts evaluated in this thesis. (a) Deposition of a dry lubricant on the metal sheet and (b) deposition of a low friction (anti-sticking) and wear resistant PVD coating on the forming tool.

1.3 Limitations and objectives

The fundamental mechanisms controlling the forming properties of sheet metals depend on a complex combination of factors such as steel sheet and tool materials, tribological conditions and press parameters, etc., see Figure 4. Of these, the tribological mechanisms controlling the performance of the two types of surface engineering concepts discussed above will be focused on in the present thesis.

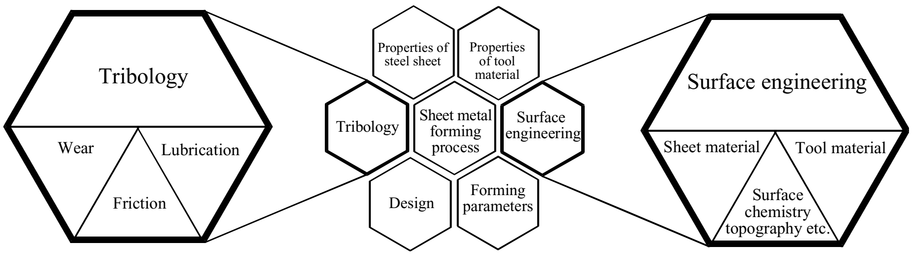


Figure 4. Factors influencing the forming properties of sheet metals.

The aim of the thesis is to find well performing surface engineering concepts for either the sheet material or the forming tool with the intention to work under dry/unlubricated sliding conditions. The purpose of the present work is to characterise the tribological properties of a number of newly developed polymer based thin organic coatings deposited on metal coated steel sheet. Furthermore, the tribological properties of different PVD coatings in sliding contact with Zn and 55%Al-Zn coated steel sheet have been evaluated. The work includes laboratory testing and post test characterisation of the samples using surface analytical techniques such as SEM/EDS, AES and ToF-SIMS in order to evaluate the tribological properties of the coatings.

2

HOT-DIP GALVANIZED STEEL SHEET

For over 150 years, hot-dip galvanizing has had a proven history of commercial success as a method of corrosion protection of steels for a wide range of applications worldwide. Today, there are mainly three types of Zn deposition techniques used on the market: hot-dip galvanizing, electrodeposition and thermal spraying. This thesis will be limited to hot-dip galvanized steel sheet applied by a continuous process. Today, this is the most common way to protect carbon steel sheet from corrosion. Hot-dip Zn coatings will significantly improve the corrosion resistance of the steel sheet due to a slower corrosion rate of the coating material. Besides, the coating will give a cathodic protection of the steel in damaged areas of the coating and at cut edges of the sheet.

In the continuous metallising process, cold-rolled steel sheet is preheated to 540-700ºC in order to burn off surface contaminants and to improve the mechanical properties. The sheet is then cooled in a N_2-H_2 atmosphere in order to reduce iron oxides on the surface. After the heat treatment, the steel enters the metal bath for hot-dip galvanizing. The thickness of the metal coating is controlled by removing the excess of melted metal with gas jet knives. The coating thickness is typical 10 to 25 µm. After the dipping process the coated steel is cooled to room temperature which results in a dendritic microstructure.

The surface finish is of outmost importance when it comes to produce a high quality steel sheet product. A lot of research work has therefore been done in the steel sheet industry concerning surface finishing issues. The surface finish will not only affect the aesthetic properties but also properties such as lubrication during forming and paintability. The surface finish in general and more particularly the topography is controlled by the last rolling step which is referred to as temper rolling. During this rolling process the desired topography is obtained by using textured rolls. The resulting surface topography of the steel sheet has an essential role when controlling the lubrication in sheet metal forming [3].

In the present work two different metal coated steel sheet materials, *i.e.* hot-dip Zn and 55%Al-Zn coated steel sheet, manufactured by SSAB Tunnplåt AB, Borlänge, have been investigated. In the following chapters the structure, mechanical properties and the corrosion resistance of Zn and 55%Al-Zn coated steel are illuminated. The chapters concerning corrosion are in the vicinity to the central line of this thesis, but are essential for a basic understanding of the usage of Zn and 55%Al-Zn coated steel.

2.1 Hot-dip Zn coating

Hot-dip galvanized steel sheet has been produced commercially since 1962 by SSAB Tunnplåt AB under the trade name Dogal. In the Dogal production line the metal bath consists of 99.7% Zn and 0.3% Al and is kept at a temperature of approximately 460ºC.

2.1.1 The structure of hot-dip Zn coating

In order to suppress the reaction between iron and zinc approximately 0.3 % Al is added to the metal bath. This will result in a more ductile coating by suppressing the formation of brittle Fe-Zn phases. In general, hot-dip Zn coatings show very good adhesion to the steel substrate, due to the formation of a metallurgical bond, consisting of Zn, Fe, Al phases, between the coating and the steel substrate. At a microscopic level the galvanized Zn coating has a structure consisting of dendritic arms. At a macroscopic level, as seen by the naked eye, the coating consists of large grains called "spangles". Each spangle contains dendrite arms consisting of pure Zn eta (η) phase with a specific crystal orientation. Owing to the high affinity of aluminium to oxygen, a 3-4 nm thick layer of aluminium oxide, γ-Al_2O_3, is formed on the surface immediately after the steel strip has been immersed in the metal bath [4]. This oxide improves the lustre or reflectivity of the coating.

2.1.2 Mechanical properties of hot-dip Zn coating

The metal coated steel sheet material may be described as a composite consisting of a metallic coating connected to the steel substrate by a brittle intermetallic phase. During forming operations micro cracks are generated, mainly within the intermetallic phase but also within the Zn-coating. In general, pure Zn coatings crack within grains by transgranular cleavage cracks, which essentially are parallel to each other. More in detail observations have shown highly anisotropic deformation behaviour of zinc crystals. [5-7]

2.1.3 Corrosion resistance of hot-dip Zn coating

The purpose of using zinc as a coating material is mainly due to two reasons. First, the zinc coating serves as a barrier to the environment, with much higher corrosion resistance than the steel substrate. Second, the zinc coating provides galvanic protection due to its more active corrosion than the more noble steel substrate (cathode). Therefore, the zinc coating serves as a sacrificial anode and gives a cathodic protection of the steel in damaged areas of the coating and at cut edges of the sheet. Many studies have been performed to investigate the atmospheric corrosion process of zinc exposed to different environments [8-11] as well as the cathodic protection mechanism [12-15] that occur in connection to defects or at cut edges of coated steel sheet.

2.2 55%-Al-Zn coating

55%Al–Zn coated steel sheet was first produced commercially in 1972 by Bethlehem Steel in the US under the trade name Galvalume, in 1976 by BHP in Australia as Zincalume, and in 1981 by SSAB Tunnplåt AB in Sweden as Aluzink. The material is most commonly used in the building industry and is also used for motor vehicle components and for electric household appliances. The product was originally developed with the intention to obtain a coating with an improved atmospheric corrosion protection than normal continuously hot-dip galvanized steel sheet [16].

2.2.1 The structure of 55% Al-Zn coating

In the 55% Al-Zn production line the metal bath consists of approximately 55% Al, 1.6% Si and remainder Zn. The Si is added to the bath in order to suppress the exothermic reaction between iron and aluminium [17].

When the steel strip enters the metal bath the intermetallic layer forms at the interface between the steel substrate and the coating. The bath temperature is kept at approximately 600ºC. According to the phase diagram (Figure 5) is Al-α phase primary precipitated when cooling beneath 600ºC. The Al-α phase is precipitated as dendrites (Figure 6). The dendrite arm spacing is strongly correlated to the cooling rate [18]. The coating structure consists of fcc crystals which preferentially have been precipitated with the close-packed (111) plane parallel to the sheet surface, which is shown by a 6-fold rotational symmetry. Finally, zinc-rich phases and silicon particles are precipitated in the interdendritic regions. A cross-sectional view of the 55%Al-Zn coating is seen in Figure 7. The silicon precipitates, in fact plates, 5-20 µm in size, can be seen in the interdendritic regions. At the substrate-coating interface the intermetallic layer, 0.5-2 µm, is observed. This layer consists of Fe-Zn-Al and Fe-Zn-Al-Si compounds [17, 19] and acts to bond the coating metallurgically to the steel substrate.

Also on 55%Al-Zn, a few nanometer thick layer of aluminium oxide, γ–Al_2O_3, is formed on the surface immediately after the steel strip has been immersed in the metal bath [20, 21]. The aluminium oxide film is generated by diffusion of Al from the bulk to the surface due to its high affinity to oxygen.

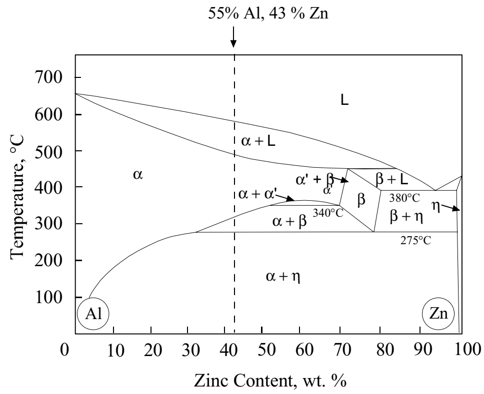


Figure 5. Phase diagram for zinc-aluminium alloys.

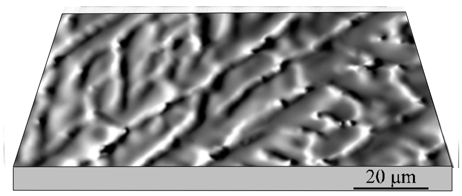
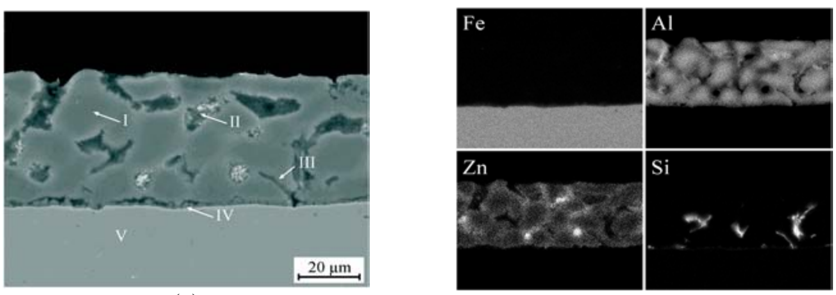


Figure 6. Backscatter electron image (topo) from 55%Al-Zn coated steel showing the dendritic structure of the coating.



(a) (b)

Figure 7. Cross-section view (a) and EDX elemental maps (b) of an as received 55%Al-Zn coating. I - Al-rich dendrite arm, II - Zn-rich interdendritic region, III - Si-particle, IV - intermetallic layer and V - steel substrate.

2.2.2 Mechanical properties of 55%Al-Zn coating

The ductility of the 55%Al-Zn coating is significantly lower than the steel substrate. This is mainly due to the complex structure of the coating consisting of a wide range of phases with individually different mechanical properties which results in an uneven distributed strain when an external stress is applied to the coating. The plastic strain is mainly located to the more ductile Zn-rich interdendritic regions. At such small strains as 5% cracks are initiated in the interdendritic regions along silicon flakes or at local defects in the coating. The ductility of the coating is often quantified by measuring the area fraction of cracks as a function of strain. A heat treatment (over ageing) is one way to make the coating more ductile and consequently reduce the crack formation. This is common for products formed to a tight radius which implies high strains, see Figure 8.

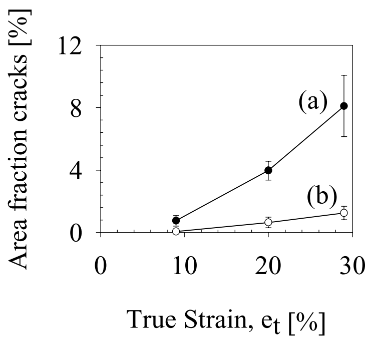


Figure 8. Area fraction cracks as a function of true strain. (a) 55%Al-Zn coated steel and (b) post heat-treated. From refs [22, 23].

2.2.3 Corrosion resistance of 55%Al-Zn coating

Townsend et. al have performed thirty-year atmospheric corrosion tests of hot-dip coated steel sheet [24]. The results show that the 55% Al-Zn alloy coating provides the best combination of durability and galvanic characteristics for the long-term corrosion protection of steel sheet.

Due to the complex structure of the 55% Al-Zn alloy and also variations in the prevailing atmospheric condition is the corrosion process very difficult to monitor. However, in general the corrosion of 55% Al-Zn alloy is initiated in the zinc rich interdendritic regions.

Although the 55% Al–Zn alloy coating has a high corrosion resistance, it is well known that the sheet is sensitive to wet storage staining, *i.e.* blackening. This kind of appearance is similar to black staining of aluminium. Odnevall et al. [25] investigated blackening and performed corrosion product characterization of sheet panels exposed under different temperature, wet storage and pH conditions. The results showed that Bayerite ($Al(OH)_3$) mainly was precipitated on the aluminium rich dendrite arms and a basic zinc aluminium carbonate ($Zn_6Al_2(OH)_{16}CO_3 \cdot 4H_2O$) was precipitated in the zinc rich interdendritic regions. Blackening of 55%Al-Zn surfaces is connected to differences in optical properties of embedded metallic zinc and/or aluminium particles of different shape and size in the corrosion layer.

To improve the resistance against wet storage staining the 55% Al–Zn alloy coating is usually protected by deposition of a corrosion-inhibiting oil, a chromium passivation treatment or a thin organic coating. In paper B the corrosion behaviour of 55%Al-Zn with different passivation treatments were investigated.

3

TRIBOLOGY IN SHEET METAL FORMING

Sheet metal forming consists of deformation processes in which a steel sheet is shaped by tools or dies. The performance of sheet metal forming processes depends on the characteristics of the type of forming process, the sheet metal, the tool material, the tribological conditions at the tool/steel sheet interface, the amount of plastic deformation used and the finished-products requirements. The high formability of metal steel sheet gives great opportunities to form products to a wide range of shapes. In this chapter the three most common types of sheet metal forming processes will briefly be described, while the focus will be concentrated upon the tribological mechanisms which may appear in the interface between the tool and the steel sheet. From now and hereafter sheet metal forming will be abbreviated as SMF.

3.1 Sheet metal forming (SMF) processes

In SMF a flat steel sheet material is deformed plastically and formed to a final shape. In the case of complicated product shapes sometimes several processes are necessary to obtain the final shape. The most widely used SMF processes are bending, stretching and deep drawing. Bending can be found in most assembling industries due to its high flexibility while stretching and deep drawing are used for production of cups and cans for the food industry and for car body panels in the automotive industry. The tribological as well as plastic deformation properties of the steel sheet are of outmost importance when optimizing SMF processes. Unexpected and unknown tribological behaviour may lead to time(cost)-consuming production stops and lower productivity. For all SMF processes different kind of contact types can be identified. With a tribological contact means that the tool and the sheet surface are brought together under relative motion and the surfaces in the contact areas interact with each other. The most extreme tribological conditions can be identified for deep drawing processes. This is mainly due to the high contact pressure between the steel sheet and the tool in combination with long sliding distances. The following chapters comprise a brief description of some SMF processes.

3.1.1 Bending

Bending is a widely used SMF operation since different kinds of shapes are possible to produce with the same equipment. In V–shape bending the punch gradually presses the sheet into a V-shaped die, see Figure 9. By stopping the process after a certain punch length different shapes can be produced.

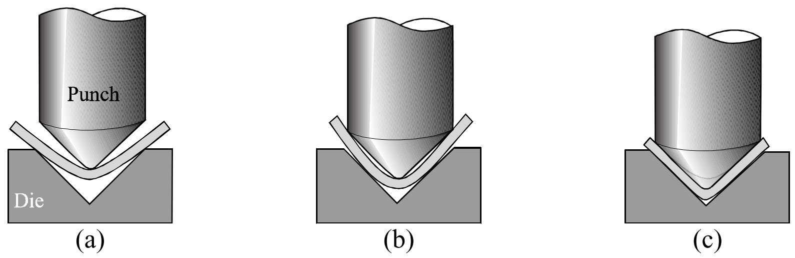


Figure 9. Schematic of the bending operation. (a) free bending (b) initiating full punch and (c) full punch.

Other types of bending operations are U-bending, roll bending and roll forming.

3.1.2 Stretch forming

In stretch forming the sheet (blank) is firmly clamped at its circumference after which a punch deforms the sheet, see Figure 10. The basic difference between deep drawing and stretching is that in stretching the steel sheet is not allowed to deform between the blankholder while in deep drawing the sheet deforms and slides between the blankholder.

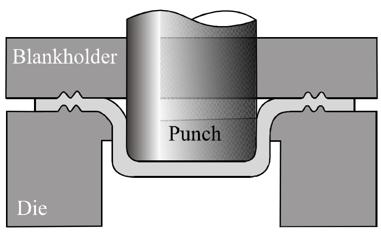


Figure 10. Schematic of the stretching process.

3.1.3 Deep drawing

In deep drawing the sheet is clamped by a blankholder and deformed by a punch. The sheet is allowed to slide between the blank holder and the die in order to avoid wrinkling of the sheet, see region I in Figure 11. In deep drawing local tribosystems and contact types can be identified. In region II the highest wear rate of the tool and the sheet is expected due to a bending and unbending deformation of the sheet and high contact pressures ($P \approx 100$ MPa) [26].

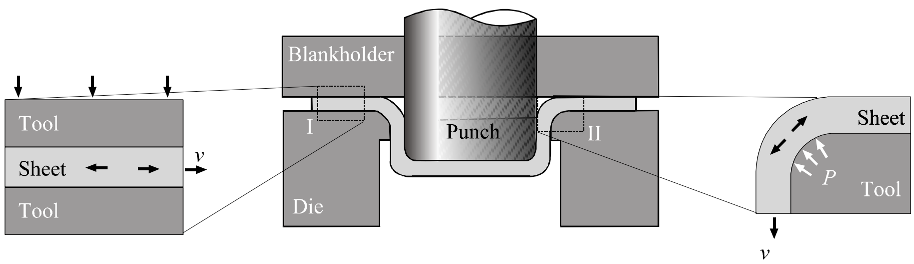


Figure 11. Schematic of the deep drawing and identified contact types.

The interacting tribological mechanisms in SMF processes will be further discussed in the following chapters.

3.2 Tribology aspects

In order to develop new steel sheet materials and/or forming tool materials, it is necessary to understand and control basic tribological processes at the sliding interface during a forming operation. The term *tribology* is defined as "the interdisciplinary science and technology of interacting surfaces in relative motion" [27]. Or in other words tribology is the scientific discipline that comprises friction, wear and lubrication.

In dry SMF the tribo system consists of tribo surfaces surrounded by humid air. Generally, a liquid lubricant is present in the tribo system and will strongly influence the tribological performance of the tribo contact. However, in this thesis liquid lubricants are not investigated and are therefore excluded from the tribo system. The tribo surfaces are the forming tool surface and the metal coated steel sheet surface, which are in contact at surface asperities. The tribo system in SMF is defined as an open tribo system since wear particles and/or lubricating oil can leave the tribo system.

In SMF the control of the friction level has a significant role since it influences the stress and strain distribution in the sheet. It should be neither too high nor too low to obtain a sheet with desirable quality. A too low friction coefficient lead to slippery sheet panels which result in handling problems in the press shop. In deep drawing the friction force between the blankholder and the die has to be sufficient high in order to obtain required plastic flow and to avoid wrinkling. However, a too high friction force will most often result in surface related problems (scratches, etc.) as well as cracks on the formed product. Consequently, there is of outmost importance to control the friction level.

The wear of the forming tool and the metal coated sheet are generally avoided by using a surplus of lubricating oils. As a result of even higher demands on more environmental friendly processes the usages of lubricating oils are desired to be minimized or if possible eliminated.

The stability of the tribological conditions in SMF operations will, to a large extent, influence the productivity and the quality of the formed product [28]. In general, the conditions prevailing at the tool/sheet metal interface are strongly influenced by the adhesion of sheet metal material to the tool surface, *i.e.* the galling tendency, see Figure 12, and by the deformation and/or wear of the sheet metal and the tool surfaces [29]. Unstable tribological conditions usually cause expensive interruptions in the forming process and poor quality (surface finish) of the products.

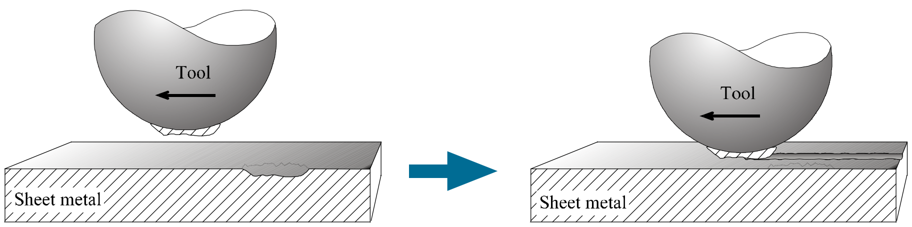


Figure 12. Material transfer from the sheet metal to the forming tool due to adhesive wear, resulting in scratch formation (abrasive wear).

3.3 Friction in SMF [30]

When the forming tool surface is sliding against the metal coated steel surface under a constant load there will be a friction force between them directed opposite to the sliding direction. The friction coefficient is the ratio between the friction force and the normal load:

$$\mu = \frac{F_T}{F_N} \tag{1}$$

This law of friction is known as the "Amontons-Coulomb Law" referring to work done by the two scientists in 1699 and 1785, respectively. In this approximation the friction coefficient does not depend on the normal load, the size of the apparent contact area and the sliding speed. However, this law is not always a correct way to explain the prevailing friction conditions.

During forming of metal coated steel sheet the friction coefficient is controlled by two different friction components:

- An adhesive force acting at the areas of real contact,
- A deformation force acting during the ploughing of the harder tool surface asperities in the softer sheet metal surface.

Consequently, the friction coefficient can be written as follows:

$$\mu = \mu_a + \mu_p \tag{2}$$

where the first component, μ_a, is the adhesive component which is material related and the second one, μ_p, is the deformation or ploughing component which is related to the surface topography of the tribo surfaces in contact.

The importance of the adhesion between two solids in sliding contact has been emphasized by Bowden and Tabor [31] in explaining the tribological phenomena. The adhesive friction component is dependent on the chemistry of the tribo surfaces at the sliding interface.

Since the tool surface is harder than the sheet metal any surface irregularities on the former surface may result in ploughing in the latter surface, thus increasing the friction force. In this thesis almost all tribological experiments have been performed by using a ball-on disc sliding contact geometry and consequently the macro ploughing component can be estimated from the contact geometry. Besides, surface irregularities and attached wear particles on the tool surface have a significant contribution to deformation which is referred to as micro ploughing in paper II (chapter 6.2).

3.4 Wear mechanisms in SMF[30]

Wear is the gradual removal of material from contacting surfaces in relative motion. By definition a heavily deformed material due to ploughing therefore does not necessarily have to be subjected to a wear mechanism. Similar to the mechanisms of friction, there are three basic wear mechanisms that are distinguished in the classification of wear in SMF:

- adhesive wear
- abrasive wear
- tribo chemical wear

Generally, more than one single mechanism occurs at the same time. However, there is generally a primary mechanism that determines the material removal rate. In *adhesive* wear, the junctions that give rise to the resistance to sliding can also cause removal of discrete particles at the sliding interface. These wear particles are often attached to the tool surface which in a subsequent forming operation may result in *abrasive* wear of the metal sheet and to contribute to the ploughing contribution of friction. The transition to abrasive wear will generally result in a more pronounced damage of the sheet metal surface due to the ploughing and cutting mechanisms of the abrasive elements. *Tribo chemical* wear mechanisms comprise a combination of chemical, mechanical and thermal processes occurring at the interface and the environment.

3.4.1 Adhesive wear

In SMF adhesive wear is the most critical wear mechanism. In general adhesive wear is identified as scoring, galling, seizing, and scuffing. These terms are used depending on type of application and type of appearance. Although adhesive wear has a wide range of appearances it is defined as wear by transference of material from one surface to another during relative motion due to a process of solid-phase welding. During adhesive wear particles that are removed from one surface are either permanently or temporarily attached to the other surface.

A more in detail explanation of adhesive wear is as follows. Solid surfaces are almost never perfectly smooth, but rather consist of microscopic asperities of various shapes, see

Figure 13. When two such surfaces are brought into contact the asperities come into contact and elastically or plastically deform until the real area of contact is sufficient to carry the load. A bond may then occur between the two surfaces that is stronger than the intrinsic strength of the weaker of the

two materials in contact. When relative motion between the two surfaces occurs, the weaker of the two materials fails, and material is transferred to the contacting surface. Due to deformation hardening the transferred particles may result in a transition to abrasive wear. In subsequent interactions, this transferred material may be retransferred to the original surface (probably at a different location) or may become partly or totally separated as a wear debris particle of an irregular morphology.

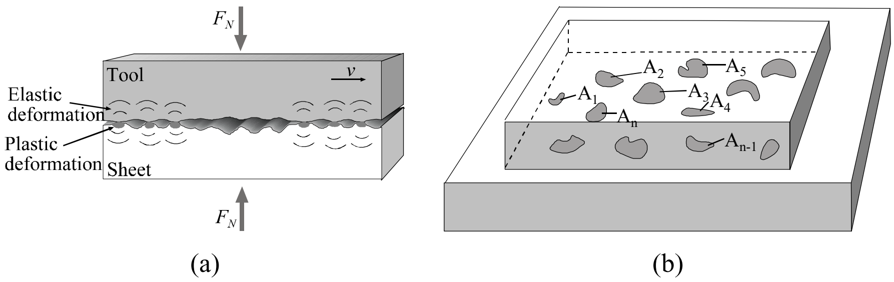


Figure 13. a) Plastic and elastic deformation of asperities in contact and b) real contact area of two surfaces in intermediate contact.

For adhesive wear to occur it is necessary for the surfaces to be in intimate contact with each other and the surfaces have a high tendency to adhere to each other. So, to reduce the adhesive wear two types of solutions are proposed:

- separating the surfaces by adding a lubricating film between the sliding surfaces.
- changing the chemistry of either one or both of the surfaces to lower the adhesion between the sheet and the tool surfaces.

3.4.2 Abrasive wear

Abrasive wear occurs when one hard surface (usually harder than the second) cuts material away from the second soft surface. In SMF, pure abrasive wear is not a very common wear mechanism. However, in those cases when the tool has a significant roughness the asperities can induce scratches on the steel sheet surface. Furthermore, in a sliding contact it can appear as a secondary effect under certain conditions since adhesive wear can generate wear debris which then causes further wear by two or three body abrasion.

3.4.3 Tribo chemical wear

Tribo chemical wear mechanisms comprise a combination of mechanical and thermal processes occurring at the interface and the surrounding environment. For example, tribo induced oxidation, resulting in the formation of oxide based tribo films, is frequently observed in the sliding contact against metals. In general, tribofilm formation is promoted by the frictional heat and the generation of fresh metal surfaces and wear particles. for metals are oxide films generated by thermal and mechanical processes prevailing at the sliding interface. Since the frictional heat generated is proportional to the friction coefficient a high friction coefficient will promote the generation of oxide based tribo films.

3.5 Tribo and transfer film formation

A repeated sliding tribo contact will in most cases result in the formation of either a transfer or a tribo film on either one or both of the tribo surfaces. A transfer film is formed by transfer of the softer sheet metal to the harder tool surface. A tribo film is generated by a tribo induced chemical reaction at a tribo surface, which means that the tribo surface is subjected to a change in surface chemical composition of the following ways:

- oxidising chemical reaction,
- alloying by solid state inter diffusion

Tribo films are sometimes desirable and sometimes detrimental. An example of a desirable tribofilms is lubricious oxide films, which retain the tribo contact in the mild wear regime. In chapter 6.4 the influence of tribo and transfer film formation on the tribological performance will be discussed in more detail.

3.6 Lubrication

The friction coefficient for an unlubricated forming operation is rarely lower than 0.5, and in many cases even higher. In SMF such high values would often lead to intolerably high friction forces leading to fracture and frictional energy losses. In SMF industry, therefore, lubricants are used to reduce and control the frictional force between surfaces.

In SMF the lubricant is often placed on the steel sheet with intention to separate the sliding surfaces and work as a layer of material with lower shear strength than the surfaces themselves. The benefit of using a lubricant in SMF is that the surfaces are separated so the probability for adhesive wear is minimised. However, the lubricant may not completely prevent asperity contact, although it will reduce it and may also reduce the strengths of the junctions formed [3]. In a historical point of view it is a well-known fact that a lot of research work has been put on the lubrication in SMF [32]. However, as mentioned before the use of lubricants have a negative impact on the environment, so the only way to lower the adhesive wear is to change the chemistry of the tribo surfaces. In the next chapter different concepts to reduce the interaction between the contacting surfaces and thus the adhesive wear by changing the chemistry of the steel sheet and the forming tool will be presented.

4

SURFACE ENGINEERING IN SMF

Surface engineering, *i.e.* techniques and processes capable of creating and/or modifying surfaces to provide enhanced performance such as corrosion and wear resistance are today frequently used in the industry. Today, there is an increasing demand for environmental friendly SMF production processes so therefore great attention is being paid to develop new surface engineered concepts which have potential to function under dry conditions, *i.e.* without using liquid lubricants. In SMF mainly two such concepts of surface engineering have been proposed (Figure 3), *i.e.* the use of dry lubricants deposited on the metal sheet and the deposition of a low friction anti-sticking coating on the forming tool surface. In common for these concepts are that they are "self-lubricating" which means that they lower the shear strength within the tribological contact thus lower the friction coefficient. The main differences between these concepts are hardness and coating ductiliy which can be related to the chemical composition and structure of the individual coating. The polymer based thin organic coating has a low shear strength and a high material transfer tendency, while the PVD-coating has typical ceramic properties like high hardness and high wear resistance.

In this thesis the types of dry lubricants are limited to thin organic coatings deposited on steel sheet. Positive effects with the introduction of thin organic coatings, as compared with the use of conventional liquid lubricants, are thought to be: reduced environmental problems in the press shop, excluded handling of lubricants in the press shop, temporary corrosion protection and improved anti fingerprint properties [33-35]. Furthermore, the deposition of thin organic coatings is aiming for an improved quality of the formed products (reduced scrap) and reduced tool wear. The idea is that the thin organic coatings should be applied continuously onto the steel sheet by the steel manufacturer thus making the process cost effective.

The other way to improve environment for SMF processes is to apply a wear resistant and low frictional anti-sticking PVD coating on the tool surface. These coatings have excellent intrinsic properties, *i.e.* high hardness, toughness and high chemical stability, which extend the life of the forming tool. Today, the state-of-the-art research work is focused on multilayers, diamond and diamond like carbon coatings (DLC). These coatings show excellent performance within a wide range of applications, *i.e.* hard disc, microelectronics, machine elements, etc.

The benefits of deposition of PVD-coatings on tools in SMF applications include: improved working environment in the workshop (no degreasing liquid lubricants), reduced/controlled friction forces, increased galling resistance and as a result reduced wear of the tool reduced usage of cleaning chemicals prior to the painting process (cost effective and environmental friendly).

4.1 Dry lubricants (thin organic coatings)

In the literature dry lubricants are generally classified into inorganic and organic compounds. The first class includes laminar solids (*e.g.* graphite and MoS_2), non laminar solids (*e.g.* PbO and CaF_2) and soft metals (*e.g.* Pb and Sn) while the second class includes various types of fats, soaps, waxes and polymers.

In general, two different types of dry lubricants exist on the market, temporary and permanent dry lubricants. While the temporary coatings should be cleanable and removed after the forming process the permanent coatings should not be removed after the forming process. In the latter case, the costs spent for cleaning agents and for destruction of used agents are significantly reduced and consequently the interest for permanent dry lubricants has increased during the last years.

Typical polymer based thin organic coating formulations consist of a resin (coating-forming material) and different types of additives, *e.g.* forming additives and corrosion inhibitors [36]. The main function of the resin in a thin organic coating is to hold the functional additives on the surface, *i.e.* the binder itself does not need intrinsic functional properties. However, the resin material should have a sufficient load carrying capacity, chemical resistance and wear resistance. Resins may be organic or inorganic, or combinations of these. Forming additives, *e.g.* waxes, are included in order to reduce the coefficient of friction as well as the adhesion between the tool and the steel sheet during the forming process. Finally, corrosion inhibitors, *e.g.* chromates, are added in order to provide the required transit corrosion protection of the steel sheet. Unfortunately, the chemical compositions of the thin organic coatings are under industrial secret and consequently only limited information about the compositions of the coatings have been received within the present work. Therefore an exact correlation between the composition and structure and the resulting properties of the organic coatings is very difficult to obtain.

4.1.1 Deposition processes of thin organic coatings

One of the most important properties of a thin organic coating is that it should be easy to apply in a continuous process line. The coatings investigated in this thesis have all been applied by a spray and squeeze rolling deposition technique, see Figure 14. Both production line deposited coatings as well as coatings deposited in the laboratory have been investigated.

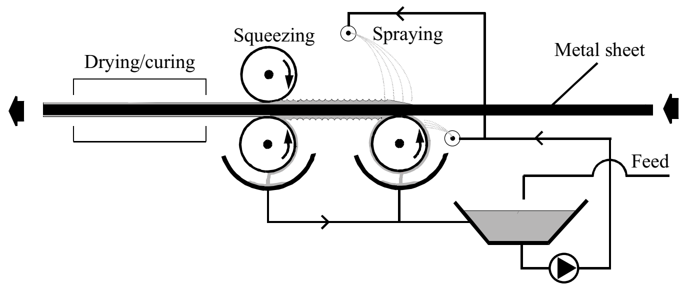


Figure 14. Schematic of the spray and squeeze deposition process used at SSAB Tunnplåt AB.

Recently, a reverse direction roll coating deposition technique has been developed and introduced to the manufacturing industry, see Figure 15. The main advantage of using this type of deposition technique is that the deposited organic coating displays a much more even thickness distribution. This is mainly due to the fact that thicker wet films can be deposited using this technique. The disadvantage of this technique is that it is complicated to tune in and gives a high wear rate of the rolls (consisting of rubber).

4.1.2 General aspects concerning polymers and polymer tribology

Polymers are an extensive class of natural or synthetic substances composed of very large molecules, and having a wide range of mechanical, physical and chemical properties. They have excellent thermal and electrical insulation properties, low density and high resistance to chemicals but are mechanically weaker and exhibit a lower elastic moduli than metals. The advantage of polymers is that they can be easily manufactured into complicated shapes. The basic manufacturing processes are extrusion, moulding, casting and forming of sheets.

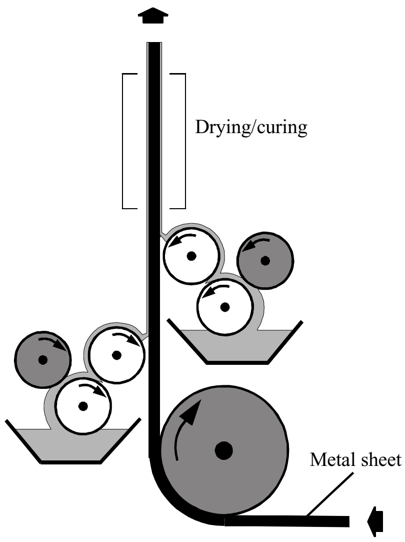


Figure 15. Schematic showing the reverse direction roll coating deposition technique.

Many tribological investigations have been carried out to understand the wear and friction mechanisms of polymers in sliding contact with steel. In particular, great attention has been focused on transfer film mechanisms in polymer friction. Since most polymers are self-lubricating materials, the transfer film of polymers can act as a lubricant [37-41]. In order to improve the tribological properties of polymers a number of potential additives can be added to the polymer. For instance many polymers contain additive which improves their friction characteristics (*e.g.* waxes) and mechanical strength (ceramic fillers etc.).

4.2 PVD coatings

During the last years there has been an increasing interest for development of low friction/high wear resistant PVD coatings which have the potential to form metal sheets under dry conditions. Due to the low deposition temperature of the PVD deposition technique (200-500ºC) it is suitable for different types of tool steel grades. One of the aims of this thesis is to find out what types of PVD coatings those have potential to work as tool material in a forming application of zinc and 55%Al-Zn.

4.2.1 Tribological properties of PVD coatings

As mentioned before, the use of liquid based lubricants in SMF has mutually a negative impact on the environment and on the whole economy. Therefore, it is a huge need of finding solutions to make the forming processes dry.

In the literature, basically two different types of PVD coatings, CrN and diamond-like carbon (DLC) based coatings, have shown promising results when used in those metal cutting and forming applications where pick-up of work material on the tool surface should be avoided.

CrN coatings offer high thermal stability and oxidation resistance [42], high corrosion resistance [43], high wear resistance [44] and a low adhesion to some engineering work materials such as Cu. Furthermore, the relatively low intrinsic stress state [45] and the relatively low deposition temperature make it possible to deposit relatively thick CrN coatings (possible to polish resulting in a very smooth surface) on most steels without any risk for thermal softening [46].

DLC coatings for tribological applications have extensively been investigated for the last 10 years. DLC coatings consist basically of a mixture of diamond (sp^3 bonds) and graphite (sp^2 bonds) and are generally divided into hydrogen-free tetrahedal amorphous ta-C DLC coatings and amorphous hydrogenated a-C:H coatings. In the tribological applications of interest in the present study, mainly a-C:H DLC coatings doped with metals or metal carbides have shown promising results due to a combination of high wear resistance and low friction (low adhesion to some engineering work materials) [47-49].

Podgornik et al. [50] have investigated the galling properties of four different PVD coatings; TiN, TiB_2, TaC and a tungsten-carbide doped DLC coating (WC/C) in sliding contact with austenitic stainless steel. The results showed that the deposition of a DLC coating of the WC/C type may result in excellent protection against pick-up of work material. However, in order to reduce the pick-up tendency of work material the coated surface should be as smooth as possible.

Vercammen et al. [51] have investigated different state-of-the-art DLC coatings in a comparative study. The results from that study showed that the tribological properties of the DLC coatings vary strongly depending on coating composition/configuration and processing conditions (techniques). In summary it was found that the observed differences in performance was related to differences in intrinsic properties of the coating such as mechanical properties, roughness, coating thickness and internal stress state.

Murakawa et al. [52, 53] and Taube [54] have been investigated the potential of using DLC coated dies in deep-drawing of aluminium. The results show that the DLC coated dies exhibit excellent anti-sticking properties for aluminium under oil-lubricated conditions. In another work by Murakawa et al. [55], the tribological properties of amorphous hard carbon films sliding against zinc plated steel sheets were investigated. It was found that the DLC coatings showed excellent deep drawing performance against zinc under dry forming conditions.

In paper VI and VII the friction characteristics and material pick-up tendency of TiN, CrN and DLC coatings in sliding contact with Zn and 55%Al-Zn were investigated using modified scratch and ball-on-disc testing (Chapter 5.1). These papers focus on the influence of chemical composition and surface topography on coating performance. In order to increase the understanding of the friction and wear mechanisms controlling material transfer at the sliding interface the worn surfaces were subjected to a careful characterization using surface analytical techniques (Chapter 5.3). The most important finding from these papers includes that DLC coatings exhibit a low friction coefficient and no material pick-up in dry sliding contact with Zn. However, the low friction/no material pick up characteristics were only obtained after a running in procedure. A more in detail description of the results are summarised in Chapter 7.

5

TRIBOLOGICAL TESTING AND SURFACE CHARACTERISATION

Due to the difficulty of evaluating the tribological characteristics of real forming applications the aim of this thesis was to simulate and study the tribological mechanisms prevailing at the intimate contact (interface) between a forming tool surface and a mating steel sheet surface. The simulations were performed by laboratory testing which makes it possible to follow the friction and the visual appearance of the surfaces (*e.g.* discoloration) continuously. The tests can be stopped for surface analysis during an interesting steady-state friction regime or when a sudden event occurs, *i.e.* when changing wear mechanism resulting in a sudden increase or decrease in friction. The small samples (sheet as well as ball counter surfaces) allow surface analyses along the wear track and of the wear spots which gives valuable information about the correlation between the surfaces and the friction characteristics thus obtaining detailed information of the prevailing tribo mechanism controlling friction and wear. The sequence of the experimental work is illustrated in Figure 16.

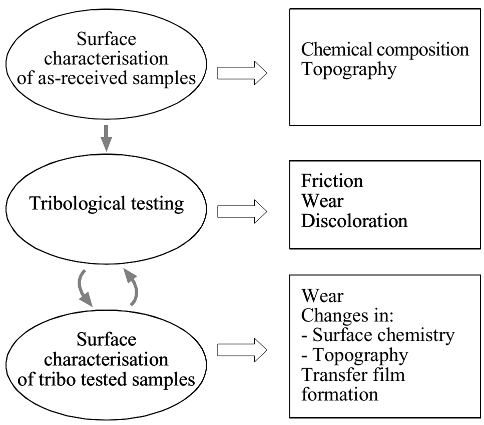


Figure 16. Schematic of the experimental procedure.

5.1 Tribological testing

In this thesis three different test methods have been used in order to simulate the contact conditions in SMF

- bending under tension testing
- ball-on-disc testing
- modified scratch testing

The tribological tests, sheet materials and counter surfaces used in the present thesis are summarized in Table 1.

As mentioned earlier (chapter 3.3), the contribution to friction is divided into an adhesive component, μ_a, and a ploughing component, μ_p. In a real SMF operation the contribution to friction is almost completely restricted to the adhesive contribution. In the tribological model tests (modified scratch and ball-on-disc) a ball bearing steel ball is sliding on the sheet aiming to simulate a sliding tool. Since the spherical geometry yields a significant contribution to friction due to the ploughing effect a too small ball radius should be avoided since it will result in a too high μ_p. In contrast, a very large ball radius results in a minimized μ_p thus isolating μ_a. However, too big ball bearing steel balls is not realistic and practical to use/analyse so in our experiments ball diameters of 6, 7.5 and 8 mm were selected, see Figure 17.

Table 1. Tribological tests used in the present thesis.

Tribo test	Sheet material	Counter surface	Papers
Bending under tension test	Polymer coated 55%Al-Zn and Zn	Calmax, hardened tool steel	I
Ball-on-disc	Polymer coated 55%Al-Zn and Zn	Ball bearing steel	I, III, V
	55%Al-Zn and Zn	TiN, DLC, CrN	VI, VII
Modified scratch test	Polymer coated 55%Al-Zn and Zn	Ball bearing steel	I, II, III, IV, V
	55%Al-Zn and Zn	TiN, DLC	VI

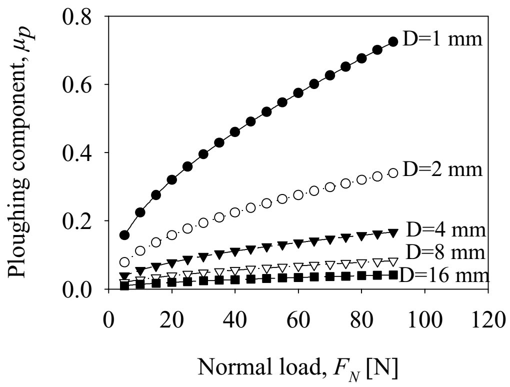


Figure 17. Ploughing component, μ_p, vs normal load calculated for a number of ball diameters.

5.1.1 Bending under tension testing

Bending under tension (BUT) testing was used to evaluate the materials in a well-established, well-controlled laboratory forming test set-up [56-58], see Figure 18. In this test, a sheet strip (size: 650×50×1 mm) is stretched 90° over a cylinder (radius R=5.0 mm), with a prescribed back tension force, F_2, adjusted to prevent sliding until a certain amount of plastic deformation of the strip occurs, while measuring the pulling force, F_1.

$$\mu = \frac{(2R + t_0)}{pR} \cdot \ln\left(\frac{F_1 - F_{BUB}}{F_2}\right) \tag{3}$$

where F_{BUB} is the bending - unbending force[i] and t_0 is the sheet thickness. A more comprehensive description of the BUT test equipment is given in reference [59].

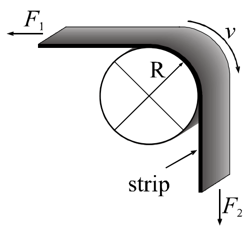


Figure 18. Principle of bending under tension testing.

[i] F_{BUB} is measured by stretching a strip over a freely rotating cylinder

Ten strips were tested for all materials investigated without changing the tool cylinder. The total sliding distance and sliding speed were in all experiments 100 mm and 0.1 m/s, respectively, and the back tension stress was set to 198 MPa. The tool material was a quenched and hardened tool steel (Calmax) ground to a surface finish of $R_a \approx 0.1$ µm.

All tests were performed under dry sliding conditions, *i.e.* without any lubricant present, in ambient air. Before testing the uncoated samples were ultrasonically cleaned in acetone and alcohol while the coated samples were rinsed in alcohol.

5.1.2 Ball-on-disc testing

Ball-on-disc testing (Figure 19) was used to evaluate the materials in a well-controlled multiple-passage sliding contact [60]. In this test a steel ball was used and drawn over the surface several revolutions in the same circular track at a normal load of 20 N and a sliding speed of 10 mm/s. During the experiments, the tangential (friction) and normal forces were continuously measured with strain gauges and recorded by a personal computer.

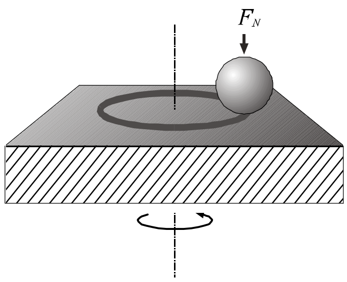


Figure 19. Principle of ball-on-disc testing.

5.1.3 Modified scratch testing[i]

Modified scratch testing (Figure 20) is based on conventional scratch testing using a commercial scratch tester, CSM Revetest®. However, instead of the Rockwell C diamond stylus (radius 200 µm), frequently used in abrasion/scratch testing, a steel ball (diameter 8.0 mm), made of ball bearing steel, was drawn over the coated steel sheet surface in order to obtain a well-controlled sliding contact [61]. A linearly increased (0-100 N) normal load or a constant normal load of 20 N, a sliding speed of 20 mm/min and a

[i] A more correct name of the test should be “sliding testing” or “adhesive wear testing”.

sliding distance of 20 mm were used in all the experiments. In both the scratch and ball-on-disc tests the ball was rotated and cleaned in acetone and alcohol between the measurements in order to get a fresh and clean sliding surface. All experiments were performed in controlled room temperature and relative humidity.

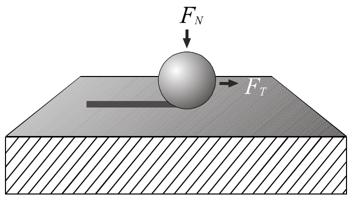


Figure 20. Principle of modified scratch testing.

5.2 Relevance of the tribological test methods

The main question concerning laboratory testing is whether the selected test can simulate the contact conditions and wear mechanisms prevailing in the real application. To manage to do this the contact types and conditions (temperature, relative humidity, contact pressure and sliding speed) in the forming process have to be identified. It is not hard to understand that determining all these parameters is not realistic. For example the contact areas and local contact pressures are altering over time from one part of the sheet to another depending on geometrical and topographical parameters in combination with the movement of the tool relative to the sheet. It is however known since long time ago that the wear and friction mechanisms are governed by the sliding interaction between the asperities at a microscopic level.

The relevance of the three test methods used were evaluated and compared in paper I. The results obtained show that; all tests display a high degree of repeatability and reproducibility; the tests rank the materials identically; the worn surfaces of the tested samples obtained in the different tests display the same wear characteristics and the friction coefficients obtained in the different tests are almost the same. The relatively small differences in friction coefficients observed, were probably due to different geometrical configurations and sliding speeds used in the tests. Besides, the differences in surface topography of the tool used in each test may influence on the friction coefficient.

From the study in paper I it was found that the main advantages of using modified scratch and ball-on-disc testing include; well-controlled contact geometries in combination with a fast and straight forward test procedure. Furthermore, the use of small and simplified samples makes post-test surface characterisation of worn tribo surfaces easier to perform and consumes less material. The strength of modified scratch testing is that the friction characteristics are recorded under well-controlled test conditions. In addition, thoroughly performed surface analyses along the wear track gives valuable information about the correlation between the surface and the friction characteristics. In contrast, ball-on-disc testing gives valuable information about the wear resistance of surface coatings. The wear resistance is determined by counting the number of revolutions before the coating fails (substrate exposure). The coating failure event is easily identified since the friction coefficient dramatically increases due to strong adhesive wear.

5.3 Surface characterisation techniques

The use of advanced surface analytical techniques has an important role in this thesis. Table 2 comprises the techniques used in order to characterise the as-deposited as well as the tribo tested coated surfaces. The main reasons for using these techniques (despite availability) are their potential of providing topographical as well as chemical information on a microscopic level. Especially, information on the composition of the topmost atomic layers of tribo surfaces is essential in order to obtain the complete information and interpretation of tribo induced surface changes. The most important and most frequently used techniques are therefore described in the following sections.

Table 2. Surface analytical techniques used in this thesis.

Technique	Information	Paper
Light optical microscopy (LOM)	Structural changes, discoloration characteristics	I-VII
Scanning electron microscopy (SEM) [62]	Surface topography, morphology	I-VII, A, B, C
Energy dispersive spectroscopy (EDS) [62]	Elemental/chemical composition	I-III, V-VII, A, C
Auger electron microscopy (AES) [63-65]	Elemental/chemical composition	I-III, V-VII, A, B, D
Time of flight Secondary ion mass spectrometry (ToF-SIMS) [66-68]	Elemental and molecular composition	III, IV, A, D, E
Non contact interference profilometry	Surface topography	II, III, VII

5.3.1 Scanning electron microscopy – SEM

Scanning electron microscopy, SEM, has two major advantages over conventional optical microscopy due to its higher resolution and greater depth of focus. A higher resolution makes it possible to study a surface at a higher magnification while the greater depth of focus makes it possible to study topographical objects at low magnifications. When the electrons strike the sample a variety of signals are generated see Figure 21. It is from the detection of specific signals that an image can be produced or a sample's elemental composition can be determined. The most important signals, which provide valuable information, are the secondary electrons, backscattered electrons, characteristic X-ray photons (sect. 5.3.2) and Auger electrons (sect. 5.3.3). The primary electrons interact with the atoms in the material and can either be scattered elastically or inelastically. Due to the scattering, the primary electrons only travel down to a specific depth, thus resulting in an activated pear-shaped volume, see Figure 21.

When the atoms in the specimen are bombarded with the primary electron beam, many of the atomic inner-shell electrons are ejected, thus ionising atoms of the specimen. The vacances are filled by other outer-shell electrons.

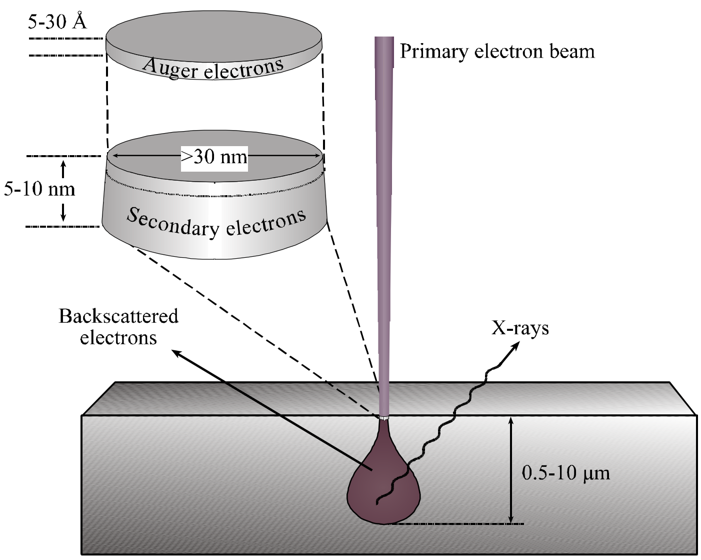


Figure 21. Schematic cross-sectional view of the incident electron beam, the activated volume and emitted signals.

The surplus energy thus created can be released in either of two ways: by emitting an X-ray quantum or an Auger[i] electron, see Figure 22. Both these signals can be detected and analysed in order to obtain information about the chemical composition of the near surface region.

In this thesis SEM analysis has been the primary analytical technique in order to illustrate surface morphologies of as-received as well as worn samples.

5.3.2 Energy Dispersive X-ray Spectroscopy- EDS

During inelastic scattering of the beam electrons, characteristic X-rays can be formed by an inner shell ionisation process (Figure 22a). As can be seen an incident electron can interact with a tightly bound inner-shell electron, ejecting the electron and leaving a vacancy in that shell. The atom is left as an ion in an excited energetic state and relaxes immediately to its ground state through transitions of outer-shell electrons to fill the inner-shell vacancy. The energy difference of the transition is emitted as a photon of electromagnetic radiation, *i.e.* X-rays. This photon is characteristic for each atom due to sharply defined bonding energies of the electrons for the specific atom.

For detection of emitted characteristic X-rays the SEM is commonly equipped with an energy dispersive X-ray spectrometer, EDS. The EDS gives valuable information about the elemental composition of the (sub)surface material down to a depth of 0.5 – 5 µm, depending on accelerating voltage, average atomic mass and specimen orientation.

In all papers EDS-analysis has been a very useful technique in order to characterise the surface chemistry of as-received as well as worn samples.

5.3.3 Auger Electron Spectroscopy – AES

Auger Electron Spectroscopy is an analytical technique to probe the elemental composition in a thin surface layer of a material, see Figure 21. The main advantages of which makes AES a powerful tool for surface analysis are the high surface sensitivity and the high lateral resolution, which is equal to the spot size of the incident electron beam, see Figure 21. The surface sensitivity (depth of resolution: 5-30 Å) is a result of the relatively short inelastic mean free path for Auger electrons. As the electron beam can be focused to a very small probe size and is easy to position or deflect, AES is an excellent technique for performing small spot analysis, line scan and elemental mapping.

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[i] The Auger process was first described by Pierre Auger (1899-1993), French physician and professor at Sorbonne.

In competition with the formation of X-rays an Auger electron may be generated when an electron hits an atom in a material and a core shell electron is expelled (Figure 22b) and consequently leaving a vacancy. The ionised atom relaxes by filling the vacancy by an electron from an outer shell. The surplus of energy may be transferred to a third electron, a so called Auger electron. The Auger electron is emitted with a kinetic energy corresponding to the energy generated by the electron transition which is characteristic for the specific atom and its electron configuration. Auger instruments are often equipped with an ion gun, thus enabling ion etching of the surface to produce elemental depth profiles. In this thesis work AES depth profiling was a very useful technique in order to characterise thin layers (tribo films) formed on worn samples.

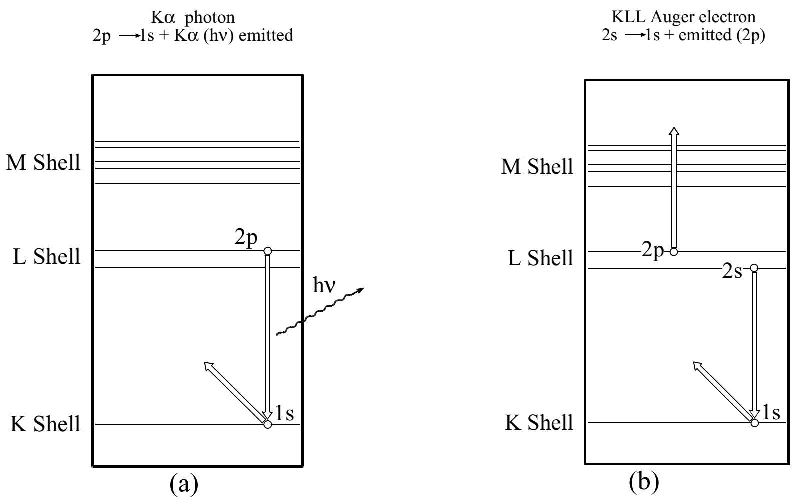


Figure 22. Schematic view of the X-ray emission process (a) and Auger electron emission process (b).

5.3.4 Time of Flight Secondary Ion Mass Spectrometry – ToF SIMS

One of the most surface sensitive chemical analytical techniques is secondary ion mass spectrometry, SIMS. In SIMS a high energetic primary ion beam is bombarding a solid sample resulting in the ejection of neutral and ionized atoms and molecular fragments from the surface region (Figure 23). The ionized species are mass analysed and a mass spectrum is produced. When combined with other surface sensitive techniques such as X-ray photoelectron spectroscopy, XPS, or AES a detailed understanding of surface structure and composition can be obtained.

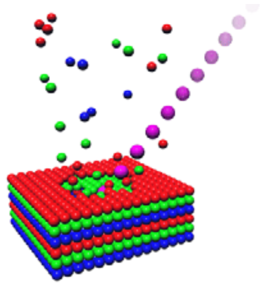


Figure 23. Schematic sketch of bombardment of a surface by the incident primary ion beam resulting in emission of secondary ions. From http://www.simsworkshop.org/graphics.htm created by Greg Gillen.

Modern state-of-the-art SIMS systems are equipped with a Time-of-Flight analyser, *s.c.* ToF analyser. Time-of-Flight Secondary Ion Mass Spectrometry (ToF-SIMS) uses a pulsed primary ion beam to desorb species from the sample surface. The resulting secondary ions are accelerated with a specific energy into the field-free mass spectrometer from the sample to the detector. The mass of the ions are determined by measuring their time-of-flight from the sample surface to the detector. By rastering a well-defined ion beam across the surface an image is obtained. The entire mass spectrum is obtained simultaneously from every pixel in the image.

ToF-SIMS is a very useful technique capable of analysing both organic and inorganic samples. In Paper III, IV and VI tribo induced chemical compositional changes of thin organic coatings was studied by ToF-SIMS. For a more in detail presentation of the ToF-SIMS technique the thesis by U. Bexell is recommended [69].

5.3.5 Optical interference profilometry

The surface profilometry measurements were performed with a WYKO NT-2000 3D interference microscope. From each area, R_a, R_q and R_z were calculated over the entire array of values (736×480=353280 pixels) in order to obtain a measure of the closely-spaced irregularities and texture of the surface. R_a is defined as the average roughness (eq. 4); R_q is the root-mean-squared roughness (eq. 5) and R_z is the difference between maximum peak height and maximum valley depth over the entire 3D surface (eq. 6):

$$R_a = \frac{\sum \left| z_i - \bar{z} \right|}{n} \tag{4}$$

$$R_q = \sqrt{\frac{\sum \left| z_i - \bar{z} \right|^2}{n-1}} \tag{5}$$

$$R_z = z_{\max} - z_{\min} \tag{6}$$

The advantages of using optical interference profilometry over conventional stylus contact instruments includes quick to perform measurements, depth resolution of 5 nm, the results yields easy interpreted 3D images. One drawback of the technique is that transparent samples can not be analysed. However, by sputter coating deposition with a reflective gold layer of a non-transparent metal layer this problem can be solved.

6

TRIBOLOGICAL PERFORMANCE OF THIN ORGANIC COATINGS IN SMF

This chapter comprises the essential results and findings from papers I-VI dealing with the tribological performance of thin organic coatings in SMF. All together more than 40 formulations of thin organic coatings have been evaluated with respect to their tribological properties using modified scratch testing and ball-on-disc testing as screening tests. Of these formulations, 19 have been evaluated more in detail and post-test characterised using different types of microscopy and surface analyses techniques. In the following sections the friction and wear mechanisms controlling the performance of the different types of thin organic coatings evaluated will be presented. For more detail the reader is referred to papers I-VI.

6.1 General observations

In general a low and stable friction coefficient indicates a stable sliding contact and a high resistance to galling. In contrast a sudden increase in friction coefficient was in all cases related to material transfer from the soft metal coating (Zn or 55%Al-Zn) to the counter surface resulting in galling. The difference between well and poor performing organic coatings can easily be observed by the naked eye. This is displayed by the photography in Figure 24. All three organic coated samples were tested in the bending under tension test and it was found that a good performing sample (a) shows no traces of discoloration or scratches, while the other samples displayed a high tendency to galling. It should be noted that one of the samples (c) performed so bad that it was fractured during the forming event due to high frictional forces. Since the aesthetic appearance is crucial when producing steel sheet the discoloration tendency has to be minimised. Therefore, for product development purposes, the discoloration appearance of the organic coated steel sheet samples was notified in real time during ball-on-disc testing.

These results have not always been published but have been vital for the continuing product development at SSAB Tunnplåt AB. By analysing the tool surface in the cases where galling occur, it was confirmed that material originating from the metal coating had been transferred to the tool surface, see Figure 25.

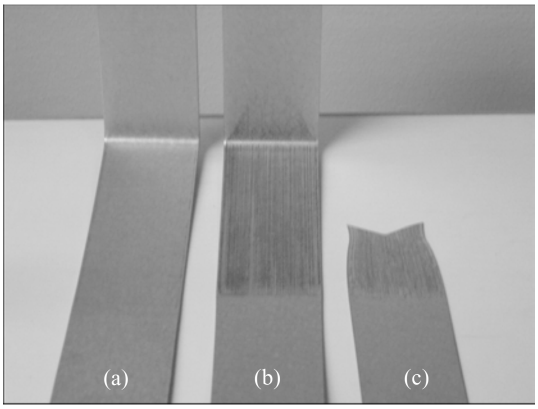


Figure 24. The difference between good and bad performing organic coatings as observed BUT experiments. (a) A good performing thin organic coating, (b) a sample surface which shows a high degree of scratching and discoloration due to galling and (c) a sample which have fractured due to very high frictional forces caused by galling.

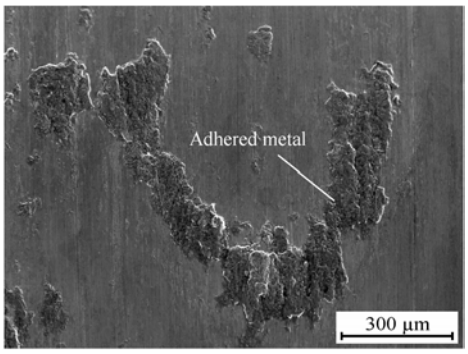


Figure 25. SEM micrograph of the corresponding tool surface in the BUT experiments illustrating a high galling tendency.

6.2 Friction characteristics

The factors influencing the friction coefficient is very complex to monitor. However, it is believed that the coefficient of friction of the organic coating can be controlled by its chemical composition, *e.g.* by adding forming additives with specific properties and the coating thickness. Besides, the topography of the substrate material also influences the friction characteristics

In the modified scratch test with increasing load (0-100 N) it is evident that the friction characteristics of thin organic coated steel sheet (in the mild wear regime) are mainly controlled by the two different components in (eq. 2), μ_a, and μ_p the adhesive component and ploughing component, respectively. The former component is material related and the latter one is the deformation or ploughing component which is related to the mechanical properties of the materials in contact and test geometry conditions.

$$\mu = \mu_a + \mu_p \qquad (2)$$

The sphere-flat geometry (Figure 26) of the sliding interface makes it possible to estimate the latter component, see *e.g.* ref. [30]. Calculating μ_p from the width of the wear track at 20 N normal load gives a value of 0.04 for both types of hot-dip coated steel materials. In a multiple passage sliding test performed at a constant load (paper I) this value easily can be identified as the decrease in friction between the first and the second passage.

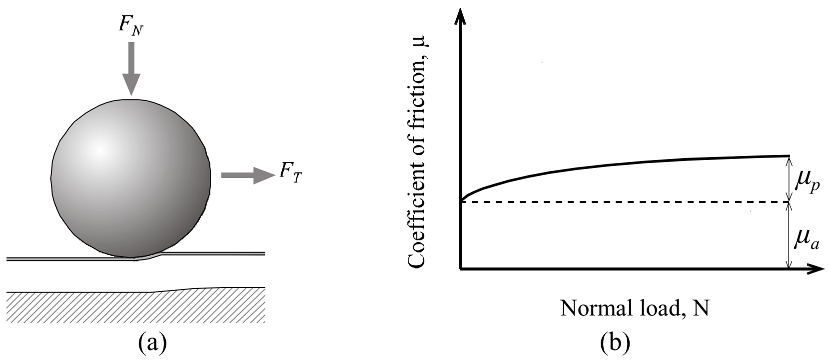


Figure 26. Schematic of the sphere-flat geometry and ploughing deformation of the surface resulting in the typical friction characteristics of metal coated steel coated with a thin organic coating as observed with increasing normal load.

In the cases when the organic coating fails high friction coefficients (μ~0.50) are obtained. The high coefficient of friction is mainly due to high adhesive forces being active at the metal/metal sliding interface. However, the micro ploughing action caused by small hard wear particles attached on the steel ball will also contribute to an increased friction coefficient. These particles most likely originate from oxides formed during scratching and/or from hard precipitates. Consequently, a third friction component, μ_{mp}, corresponding to a micro ploughing action can be added to (eq. 2), *i.e.*:

$$\mu = \mu_a + \mu_p + \mu_{mp} \tag{7}$$

Papers I, III and V clearly show the potential of using ball-on-disc testing when evaluating the wear performance of thin organic coatings. In those papers the friction characteristics of the organic coated samples were classified into different groups of performance. Typical friction characteristics for well performing organic coatings (Figure 27a) include a low initial steady-state friction level followed by a sudden increase in friction at a critical number of revolutions. The initial friction corresponds to an intact organic coating thus separating the steel sheet from the steel ball surface. The rapid increase and subsequent scatter in friction corresponds to a breakthrough of the organic coating followed by micro welding and subsequent material transfer of soft metal coating to the steel ball. Poor performance (Figure 27b) appears by an instant increase in friction from start of the test. Some organic coatings exhibited a transition in the friction curve (Figure 27c), which gave rise to a massive experimental work to explain the somewhat peculiar behaviour. The results from that work emerged into paper V which in more detail is discussed in chapter 6.4.

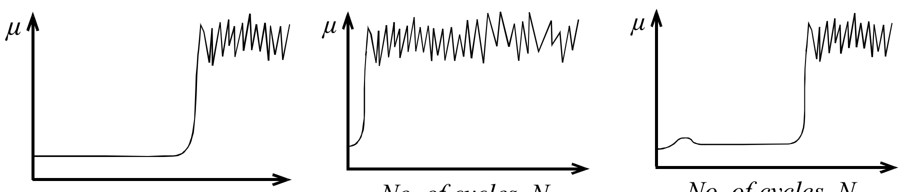


Figure 27. Schematic illustration showing the three different types of friction characteristics obtained by ball-on-disc testing. (a) Well performing organic coating, (b) poor performing organic coating and (c) well performing organic coating but showing a transition in the friction curve.

6.3 Wear mechanisms

Mild wear of the topmost surface— In papers III and IV tribo chemical changes and mild wear mechanisms of tribo tested surfaces were characterised using ToF-SIMS. This analytical technique was chosen since the information of depth is in the atomic level together with the capability of characterising organic species. In paper III it was found that tribo induced changes may take place during initial stages of a sliding event. In this paper the chemical changes was restricted to near surface wear of a forming additive. In contrast, the worn surfaces observed in paper IV did not show any changes in surface chemical composition

Coating failure mechanisms — Besides a continuous wear (resulting in a continuous reduction of the coating thickness) of the organic coating two different coating failure mechanisms have been observed in the cases when galling occurs (Figure 28), *i.e.*:

i) Adhesive failure

ii) Plastic flow and ridge formation

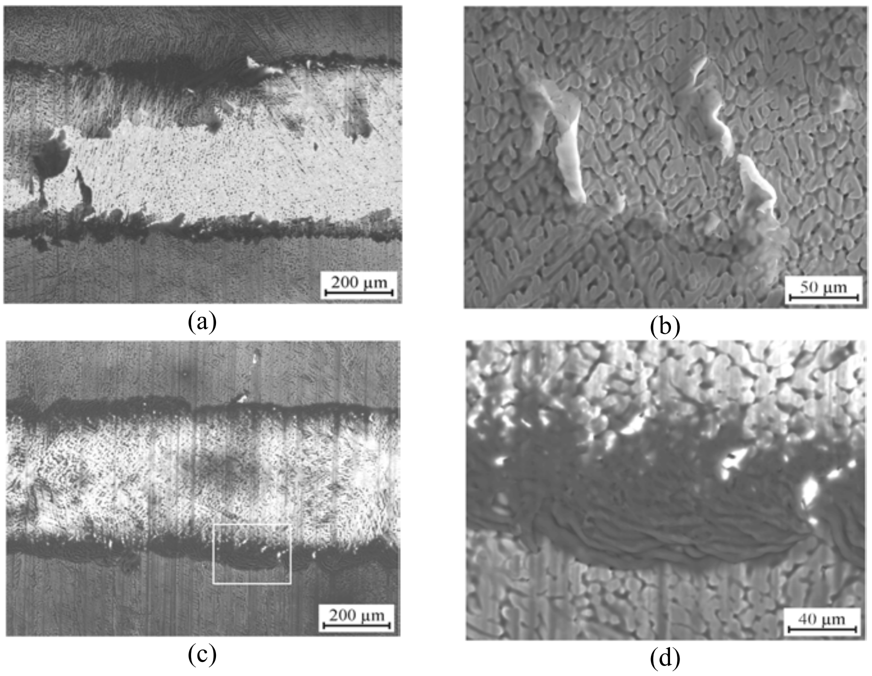


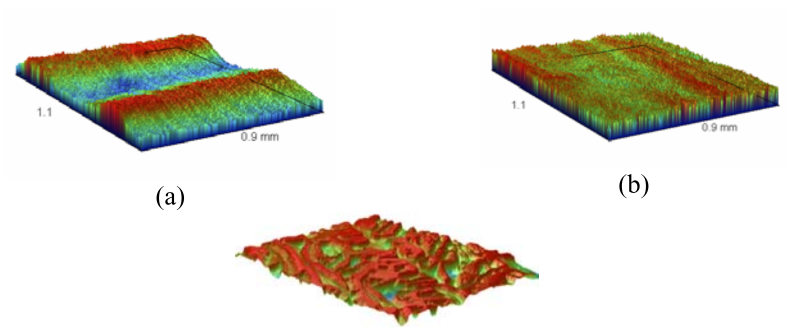
Figure 28. Coating failure mechanisms observed in the modified scratch test. Adhesive failure resulting in complete substrate exposure (a and b) and plastic flow and ridge formation due to low load carrying capacity (c and d)y. Scratch direction from left to right.

The former mechanism is promoted by a poor adhesion between the coating and the substrate, while the latter mechanism is promoted by a low hardness (low load carrying capacity) of the coating. In general the tendency to adhesive failure and plastic flow/ridge formation will increase with coating thicknesses above 1.5-2.0 μm. The critical normal load for the appearance of both types of mechanisms can easily be obtained from modified scratch testing.

However, the wear characteristics of the thin organic coatings are also strongly influenced by the substrate surface topography as will be discussed in the following section.

6.3.1 Influence of topography and coating thickness on localised coating failure

The topography of 55%Al-Zn coated steel can be divided into a long and a short range waviness. This type of topographic feature has also been observed on Zn coated steel samples (not shown here). The long range waviness is identified as a wavy surface with a wavelength of approx. 1 mm resulting from the stretch levelling of the steel strip after the hot-dip coating process. Figure 29 shows two different types of long range surface topographies of the 55%Al-Zn metal steel surfaces investigated. As can be seen at a low magnification sample (a) shows a pronounced waviness, while sample (b) shows a smooth surface topography. At a higher magnification a short range waviness can be found on both types of surfaces (a and b). This waviness is due to the microscopic variation in surface topography of the dendritic Al-Zn morphology (Figure 29c).



(a) (b)

(c)

Figure 29. 55%Al-Zn coated samples showing different substrate topography as measured by non-contact optical profilometry. (a) long range waviness (1×1 mm^2),(b) smooth surface (1×1 mm^2) and (c) short range waviness as observed at higher magnification (50×50 μm^2).

Studies in the SEM using the backscatter compositional imaging mode shows that the surface topography strongly influences the thickness distribution of the organic coating, see Figure 30.

Figure 31 shows the influence of organic coating deposition on the resulting surface topography of the organic coated samples. As can be seen, the organic coated samples display a less pronounced wavy surface profile with increasing coating thickness since the low level regions tend to be filled with the organic substance during the coating deposition process. In Paper II AES analyses of as deposited thin organic coatings confirm that the coating thickness is strongly influenced by the surface topography and that the coating thickness on the asperities is only about 5-10% of the nominal thickness value, *i.e.* 50-100 nm.

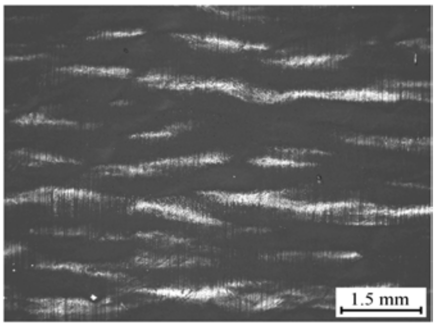


Figure 30. SEM back scatter electron image (BEI COMPO-mode) showing the variation in coating thickness over the surface. The low BEI-signal in the black regions corresponds to a high coating thickness, i.e. a low mean Z-value (atomic weight).

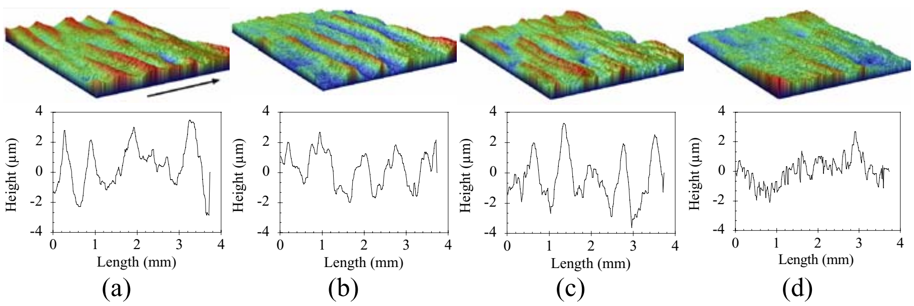


Figure 31. Influence of organic coating deposition on the resulting surface topography of the organic coated samples. Uncoated substrate (a) and low (b), medium (c) and high organic coating thickness (d). The arrow in fig. (a) indicates the profile recording direction.

As a consequence of the non-uniform coating distribution combined with a rough surface topography the friction characteristics as well as the wear properties will vary over the surface. In a sliding contact between a rough surface topography and a smooth tool surface the real contact area is located to a few contact points corresponding to the highest asperities resulting in high contact pressures at the asperities, which in combination with thin thicknesses of the organic coating at the top of the asperities may result in localised wear of the organic coating, see Figure 32. It is believed that this mechanism to a large extent controls the tribological performance of the organic coating.

Figure 32. Schematic view of an as deposited organic coating (a) and an example of localised break through of the coating at the asperities (b).

The correlation between local variations in the coating thickness and the variation in friction coefficient is illustrated in Figure 33. In this test an increasing load (0-100 N) was applied on the steel ball along the sliding track (20 mm). The bright areas in the image correspond to areas with a low organic coating thickness. As can be seen the friction coefficient increases in the areas where the coating is thin, which indicates that the coating locally has failed and a metal-metal sliding contact has occurred. It is believed that the main reason for local failure of the organic coating is due to a combination of a high contact pressure and low coating thicknesses of the organic coating at the high level regions. The curvature of the wave formed surface has been calculated to be approximately 15 mm so the steel ball is in contact with the sheet metal during the test.

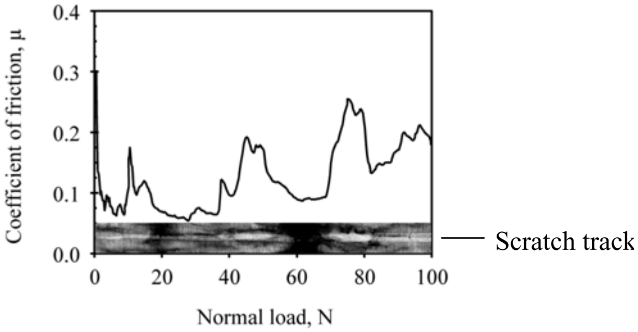


Figure 33. The correlation between local variations in coating thickness and resulting variations in friction coefficient as observed in the modified scratch test.

6.3.2 Severe adhesive wear and wear particle formation

Local failure of the organic coating will result in a metal-metal contact and a pronounced change in friction and wear characteristics, *i.e.* comparable to those observed in dry sliding of Zn and 55%Al-Zn. If the successive sliding contact continues it will result in sever adhesive wear resulting in material transfer and formation of wear particles, see Figure 34 from paper I. In the case of Zn, these particles are build-up by a flake like multilayered structure and in the case of 55%Al-Zn the particles are formed by mechanically milling and subsequent merging of smaller wear debris. The difference in wear particle formation mechanisms can be explained by the clear distinction in structure and mechanical properties between the Zn and 55%Al-Zn alloy. The sequences of wear particle formations are illustrated in Figure 35 and are discussed in the following. In case of Zn the relatively soft pure metal allows plastic shearing of successive layers. By propagation of shear cracks the wear particle finally detaches from the surface. Successive sliding events will result in further growth of the wear particle. This observation is confirmed by Kayaba et. al. [70]. On the other hand the ductility of 55%Al-Zn is strictly limited due to the complex chemical composition and structure. The 55%Al-Zn coating consists of aluminium dendrites (in fact consisting of Al-rich precipitates), Zn-rich interdendritic regions and a fine dispersion of plate-like Si precipitates, with individually different mechanical properties [71]. It is believed that the soft sticking zinc phase in combination with hard Al-rich precipitates will favour the mechanism consisting of mechanically milling and merging of wear debris. This mechanism has also been confirmed by Sasada [72].

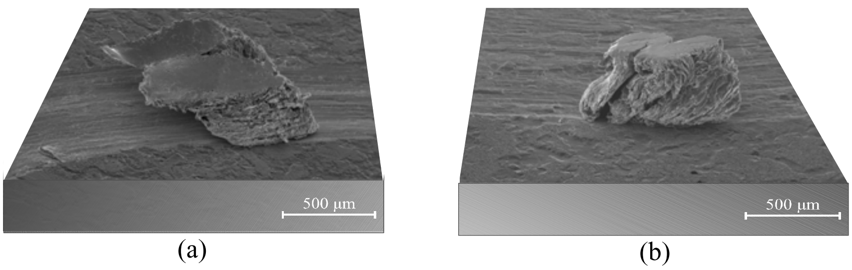


Figure 34. SEM micrographs showing wear particles stuck to test discs. (a) Multilayered wear particle observed in the case of Zn and (b) mechanically milled and merged debris resulting in an irregular shaped wear particle observed on 55%Al-Zn.

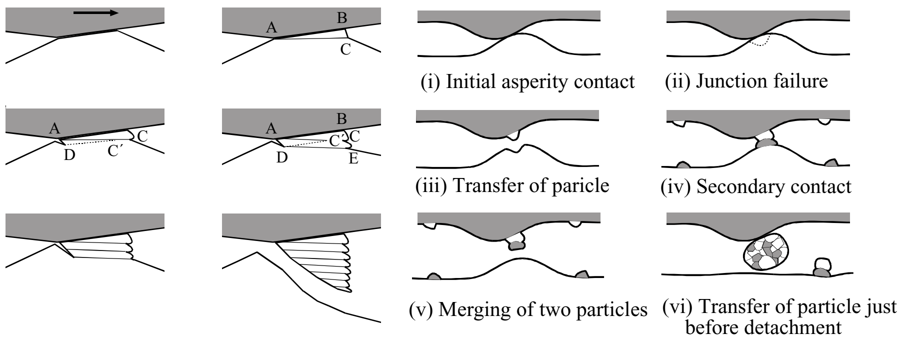


Figure 35. Formation of wear particles by a) plastic shearing of successive layers resulting in a multilayered structure (as observed in the case of Zn) from ref [70] b) mechanically milling and merging of small wear debris (as observed as in the case of 55%Al-Zn) from ref [72].

6.4 Influence of transfer and tribo film formation on the tribological behaviour of thin organic coatings

As mentioned in chapter 6.2 (Figure 27c) some well performing organic coatings showed a transition in the friction curve. To correlate the tribo mechanisms prevailing at the sliding interface with the observed friction characteristics ball-on-disc tests were run and interrupted after certain number of cycles in order to follow the gradual changes of the tribo surfaces during test duration.

SEM observations of the wear track on coated steel sheet surfaces and the corresponding contact spot on the steel balls show distinct differences in polymer transfer properties between samples showing bad, “ordinary” and “transition” friction behaviour. The bad performing organic coating has a high tendency to form relatively large loosely attached organic coating fragments within the wear spot (Figure 36a). These fragments are easily detached from the sliding interface in a multiple sliding event which explains the poor lubricating properties of the organic coating. In the case of organic coatings showing “ordinary” friction behaviour the transfer of polymer from the sheet to the steel ball is less discrete and forms a transfer film containing lumps (Figure 36b). For organic coatings showing the “transition” friction behaviour, the transfer of polymer from the sheet to the ball is more continuous, resulting in a well covering transfer film (Figure 36c). Since the latter transfer mechanism generates a more stable contact condition with no, or significantly less, areas of exposed Al-Zn alloy the tendency to a metal-metal contact resulting in a high friction coefficient will be low. Consequently, the differences in friction and wear behaviour of the different organic coatings investigated are probably due to the differences in chemical composition and mechanical properties, which affects the ability to form a stable transfer film at the sliding interface.

AES and EDS analysis of organic coatings showing the transition friction behaviour show that both the wear track on the steel sheet as well as the contact spot on the ball in the steady-state region to a large extent are covered by an Al rich oxide film. The formation of this type of tribo film is mainly due to the stable contact conditions governed by the initially formed transfer film which will prevent an early metal-metal contact and stimulate oxidation of aluminium at the sliding interface. Thus, the tribo-contact in the steady-state region is similar to the sliding contact between (if we assume that the oxide consists of a pure alumina) self-mated alumina. In the present study the friction values measured in the steady-state region was found to be approximately 0.20 which is a typical friction value during the initial mild wear regime of self-mated alumina [73].

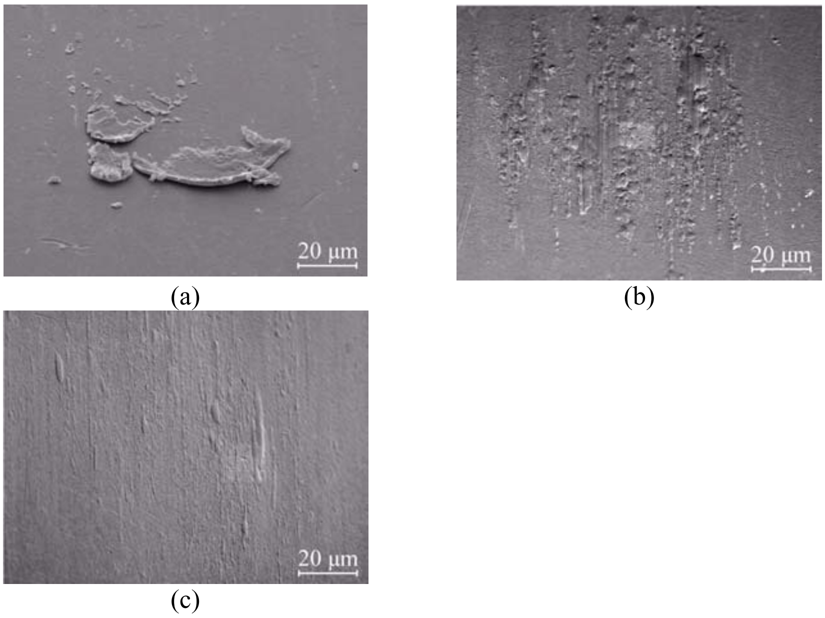


Figure 36. SEM images of the contact spot on the steel ball illustrating the differences in initial material transfer observed for the different organic coatings after 1 cycle in the modified scratch test. Image (a) showing characteristic loosely attached organic coating fragments on the ball surface typical for organic coatings with poor lubricating properties. Image (b) showing transfer film containing lumps typical for organic coatings exhibiting "ordinary" friction behaviour. Image (c) showing thin covering transfer film formation typical for organic coatings exhibiting the" transition" friction behaviour.

7

TRIBOLOGICAL PERFORMANCE OF PVD COATINGS IN SMF

This chapter comprises the most interesting results from the papers VI and VII in which the tribological performance of PVD coatings in SMF was evaluated. The PVD coatings investigated in these studies include; TiN, CrN, and various DLC coatings, see Table 3. They are all commercially available on the "PVD-market" and have been deposited on ball-bearing steel balls and further tested tribologically by modified scratch testing (paper VI) and ball-on-disc testing (paper VII). In the following chapters it will be revealed that self-lubricated PVD coatings on the forming tool have potential to create "unlubricated" forming processes. However, to create a successful and well performing PVD coated surface some essential requirements have to be taken into consideration. The aim of this chapter is therefore to give a better understanding of the underlying tribo mechanisms governing the performance of PVD coatings in SMF applications.

7.1 General observations

From a practical point of view one of the most interesting results from this thesis reveals that low friction DLC coatings may be used in the dry forming of hot-dip Zn coated steel sheet. It has, in particular, been found that the friction and wear characteristics of the PVD coatings investigated are mainly influenced by the surface condition of the tribo couples. The most important factor is the surface chemical composition of the tribo couples. By changing the surface chemical composition of one of the tribo surfaces the performance of the tribo couple is significantly changed. A clear evidence of the influence of the surface chemical composition is the distinct difference in performance between well performing DLC coatings and poor performing TiN and CrN coatings in sliding contact against Zn. On the other hand by changing the sheet metal it was found that well performing DLC coatings in sliding contact against Zn did not work at all in sliding contact against

55%Al-Zn. Furthermore, beside the surface chemical composition the surface topography of the PVD coating plays an important role in order to keep the friction coefficient at a low and stable value. For example all PVD coatings show surface irregularities such as micro droplets and dimples in the as-deposited condition. During the initial sliding these irregularities are perfect sites for soft metal to adhere and consequently these irregularities will increase the pick-up tendency of counter material and thus the material transfer tendency, see Figure 37. However, in some cases the initially adhered Zn as-well as the micro droplets disappear from the surface resulting in a smooth surface and a low and stable friction coefficient. In order to reduce the tendency to material pick-up and subsequent problems with galling it is therefore of outmost importance that the forming tool show a smooth surface before coating deposition and that the coating process does not significantly affect the surface topography. If so, it may also be necessary to polish the coated tool surface in order to eliminate the surface irregularities which may interact with the work material and initiate material pick-up. However, the results obtained illustrate that it may be difficult to form 55%Al-Zn coated steel sheet without any help from a dry or liquid lubricant although the surface is free from irregularities. The reason for this is probably due to the complex chemical composition and structure of the 55%Al-Zn coating showing aluminium dendrites (consisting of Al-rich precipitates), Zn-rich interdendritic regions and a fine dispersion of plate-like Si precipitates, with individually different mechanical properties [71]. It is believed that the soft sticking zinc phase in combination with hard Al-rich precipitates will result in an unstable tribological contact.

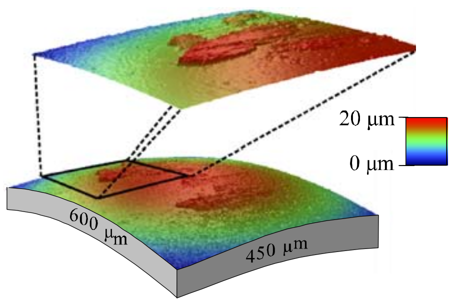


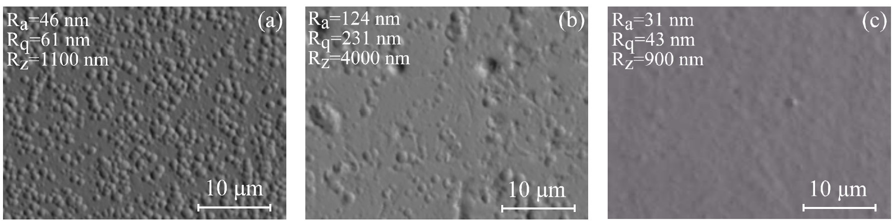
Figure 37. 3D optical profilometry images showing protruding lumps of adhered Zn within the contact spot of DLC IV as observed after 4 cycles in sliding contact with Zn.

Table 3. PVD coatings investigated in this thesis.

Coating	Investigated in	Coating thickness* [µm]	Vickers Hardness*
DLC (WC/C)	Paper VI	2.0-2.5 **	1000
TiN	Paper VI	3.5	2300
CrN	Paper VII	5.0-6.0	2000
DLC I (WC/C)	Paper VII	2.0-2.5	1500
DLC II (WC/C)	Paper VII	2.0-2.5	1200
DLC III (WC/C)	Paper VII	2.0-2.5 **	1000
DLC IV (CrC/C)	Paper VII	2.0-2.5	2500

* As given by the coating supplier
** Deposited on a 4-5 µm (Ti,Al)N coating

Figure 38 shows the surface morphologies and R-values of three different DLC coatings with individually different friction characteristics. As can be seen, the surface of coating DLC I is covered by small (µm in size) droplet-like asperities, while coating DLC IV show a smooth surface with the smallest R-values. Coating DLC III shows larger droplet-like asperities and shallow dimples on the surface which explains the large R-values. Measurements of polished surfaces (not shown here) show significantly lower R-values, the only exception being coating DLC III which still in the as-polished condition show high R-values. SEM studies of the polished surface show that this is due to detachment of small coating fragments, locally resulting in the exposure of the underlying (Ti,Al)N coating. The discrepancy in surface morphology is believed to mainly be due to differences in PVD coating deposition process parameters.



(a) (b) (c)

Figure 38. SEM images showing the surface morphologies of DLC I (a) DLC III (b) and DLC IV (c).

7.2 Friction characteristics

The friction characteristics of the PVD coatings in Table 3 have been evaluated in both as-deposited and polished conditions. In general the friction characteristics of as-deposited PVD coatings can be illustrated by two types of characteristic friction curves. A typical poor performing PVD coating (Figure 39a) is identified by an instant increase in friction coefficient from the start of the test followed by high and unstable (scattering) friction values. A typical well performing PVD coating (Figure 39b) is identified by a relatively high initial friction coefficient, $\mu_{initial}$, followed by a peak, μ_{peak}, and further by a running-in process resulting in low and stable friction values. A mild polishing treatment (using 1 micron diamond) eliminates the initial peak, μ_{peak}, resulting in low and stable friction coefficients from the start of the test (Figure 39c). The results from all tribo tests are summarized in Table 4.

Table 4. Friction coefficients for tribo couples investigated. Well performing tribo couples with potential to work in a real SMF process is marked with grey.

Modified scratch test (Paper VI)		**Coefficient of friction, μ (scratch 1-3)**		
Tribo couples		μ_1	μ_2	μ_3
Steel-Zn		0.38	0.46	0.48
TiN (as-deposited)-Zn		0.37	0.46	0.48
WC/C-Zn		0.38	0.42	0.22
Steel-AlZn		0.90	0.89	0.95
TiN-AlZn		0.66	0.89	0.94
WC/C-AlZn		0.64	0.86	0.91
Ball-on-disc test (Paper VII)	**As-deposited**		**Polished**	
Tribo couples	$\mu_{initial}$	$\mu_{steady\text{-}state}$	$\mu_{initial}$	$\mu_{steady\text{-}state}$
CrN-Zn	0.24	0.55	0.22	0.55
DLC I-Zn	0.25	0.17	0.17	0.15
DLC II-Zn	0.34	0.16	0.18	0.17
DLC III-Zn	0.22	0.40	0.21	0.40
DLC IV-Zn	0.35	0.16	0.22	0.14
CrN-55%Al-Zn	0.47	0.41	0.40	0.39
DLC I-55%Al-Zn	0.46	0.40	0.43	0.39
DLC II-55%Al-Zn	0.45	0.40	0.43	0.39
DLC III-55%Al-Zn	0.46	0.40	0.43	0.39
DLC IV-55%Al-Zn	0.45	0.41	0.23	0.41

As can be seen from Table 4 a polishing treatment significantly improves the tribological performance of the PVD coatings. The effect of polishing is obvious by the difference in initial friction characteristics between as-deposited and polished samples.High initial friction values are typical for as-deposited samples, while low initial values are characteristic for polished samples. Well performing PVD coatings with potential to work in a real SMF process is marked with grey in the Table. These coatings are all DLC coatings of various types and show steady–state friction coefficients in the interval 0.14-0.17. The only well performing PVD coating in sliding contact against 55%Al-Zn was DLC IV in polished condition, see Figure 39d. However, the good performance (μ=0.23) was limited to the first 35 cycles.

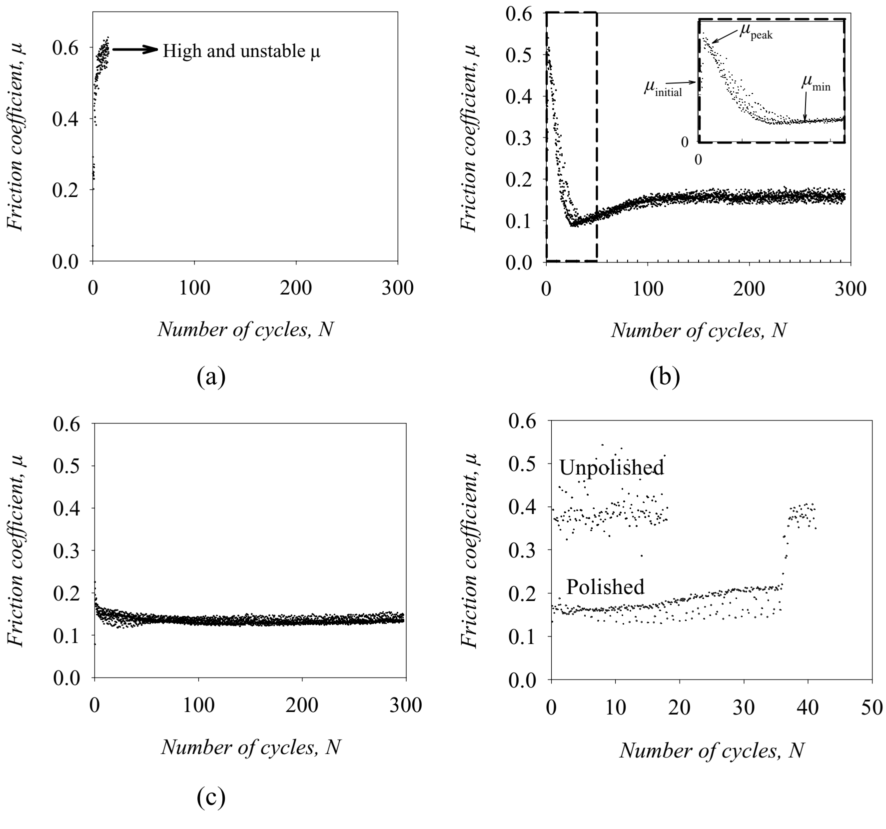
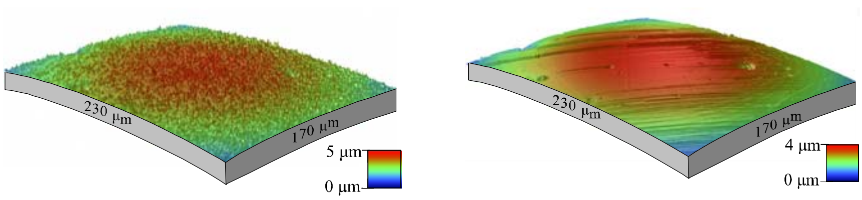


Figure 39. Friction characteristics of CrN (a), DLC IV (b) and polished DLC IV (c) in sliding contact against Zn. (d) Friction characteristics of unpolished and polished DLC IV in sliding contact against 55%Al-Zn.

7.3 Wear mechanisms

7.3.1 Mild polishing wear

The wear of the PVD coatings was monitored by following the changes in surface characteristics of the contact spot on the PVD coated steel balls, see Figure 40. It was found that a gradual wear of the PVD coatings can be observed for well performing DLC coatings. The droplet-like asperities are worn resulting in a smooth surface containing small scratches. The depth and inter distance of the scratches were studied in detail and it was found that the average depth was approximately 50 nm and the average inter distance was calculated to be 3 µm. However, some large scratches were about 400 nm in depth. The origin of these scratches is believed to be a two body abrasion due to hard wear particles (like worn droplets) in the sliding interface. However, no such particles have been found within the wear track, so there is no evidence for this hypothesis.



(a) (b)

Figure 40. Optical profilometry image of (a) as-deposited DLC I coating and (b) worn in DLC I coating after 300 cycles in sliding contact against a fresh Zn surface.

7.3.2 Surface chemical changes of the wear spot

In paper VII SEM/EDX analyses show that the high initial friction coefficient/high Zn pick-up tendency observed during the sliding between a fresh as-deposited DLC-surface and Zn was probably due to the macro particles found on the as-deposited DLC surface acting as abrasive elements. Thus, the ploughing action of these particles during the sliding event was believed to result in a relatively high friction coefficient and a pronounced pick-up tendency of Zn (Figure 41). After repeated sliding, the major part of these particles will be detached from the surface due to the relatively high tangential forces being active at the sliding interface, and consequently the friction coefficient and pick-up tendency will decrease. From now on the steady-state friction coefficient is mainly controlled by the intrinsic low friction characteristics of the PVD coating chemistry of the tribo film at the sliding interface.

In paper VI Auger depth profiles of the contact area showed that the surface mainly consists of carbon, whereas a thin tribo film of zinc oxide and graphite was observed at the periphery of the contact area. Thus, the low friction, anti-sticking properties of the surface is believed to be due to the lubricating properties of both graphite and zinc oxide.

The reason why the DLC-coating does not reduce the friction in contact with 55%Al-Zn is not clear but it is believed that it is due to the abrasive action of fine aluminium oxide particles making it impossible to generate a stable low friction interface, *i.e.* the low friction steady state regime can not be achieved.

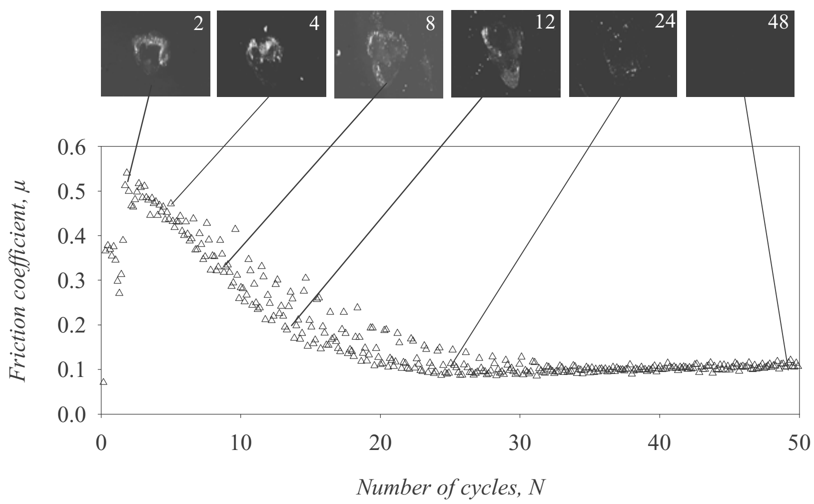


Figure 41. EDX Zn-maps showing the amount and distribution of adhered Zn within the contact spot of the DLC IV coated ball in sliding contact with Zn after 2, 4, 8, 12, 24 and 48 cycles and the corresponding friction curve.

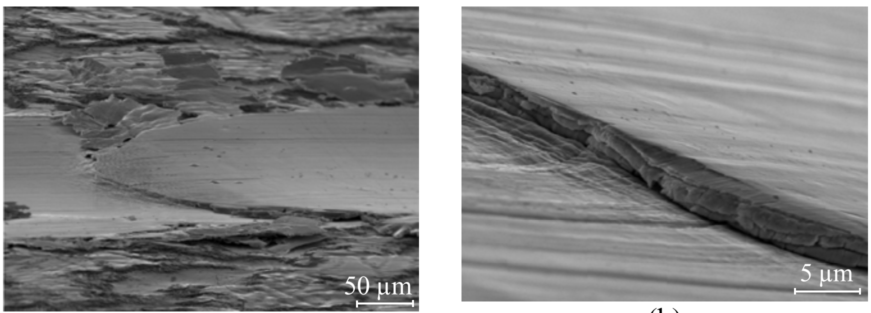
7.4 Surface characteristics of the metal sheet

7.4.1 Change in surface chemical composition

The change in surface chemistry of the topmost atomic layers within the wear track of Zn in sliding contact against DLC has been examined by AES in paper VII. The results show that initially the as-present aluminium rich oxide is locally worn off. Repeated sliding contact in the ball-on-disc test will result in the formation of an oxide rich tribo film in the wear track on the Zn surface. AES depth profiling of the tribo film indicates that it is a layered structure with a ZnO-rich layer on top of an Al_2O_3-rich layer. The separation of the tribo film into two oxide layers, ZnO and Al_2O_3, respectively, was proposed to be due to the differences in lubricating properties of the oxides. The more lubricious ZnO is believed to be enriched at the sliding interface due to its lower shear strength. However, another explanation can be that the high access of Zn added to that aluminium oxide fragments are due to mechanically mixing embedded in the soft metal Zn coating.

7.4.2 Mechanical deformation and lip formation

After 100-200 cycles the formation of relatively large (1 mm in size) sheared lips are observed within the wear tracks on the Zn coated steel sheet samples exposed to a sliding contact against the DLC coatings I, II and IV, *i.e.* the coatings showing a low friction coefficient steady-state regime, see Figure 42a. The friction behaviour in the steady-state regime is believed to be controlled by the shearing between adjacent layers within these lips or at the DLC coating/lip interface. In higher magnification (Figure 42b) it can be seen that the lips consist of a multilayered structure which indicates that the lips are generated by near surface shearing of the relatively soft Zn surface.



(a) (b)

Figure 42. Observed lip formation within the wear track on Zn coated steel after 500 cycles sliding contact against DLC IV.

8

FINAL REMARKS

8.1 Practical implications

In the present thesis it has been shown that the two concepts (the deposition of a thin organic coatings on the steel sheet and the use of PVD-coated tool) have great potential to make SMF processes dry which reduces environmental problems in the press shop due to less handling of lubricants and also reduced usage of cleaning chemicals prior to the painting process. Common for these concepts is that they should result in an improved quality of the formed products (reduced scrap) and also lead to a less frequent tool surface refinishing and reduced tool wear. The focus has been placed on the tribological performance of the surface engineered concepts. However, for a practical use one has to consider the advantages and limitations of each concept.

The main advantage of the use of thin organic coatings on steel sheet as compared with the deposition of PVD coatings on the forming tool is the multifunctional effect of the organic coating, which means that the organic coating results in an improved performance of the steel sheet product in terms of an improved temporary corrosion protection and improved anti fingerprint properties. Furthermore, the steel sheet product needs no cleaning and pretreatment before painting since the coating work as a pretreatment itself. The idea is that the thin organic coatings should be applied continuously onto the steel sheet by the steel manufacturer thus making the process cost effective. However, some of the organic coatings investigated in this thesis were difficult or almost impossible to apply in the coating line at SSAB Tunnplåt AB. For example, it has been found that some coatings were difficult to apply due to a high tendency of the organic dispersion to form lumps on the steel sheet surface and on the coating rolls. Other coatings were sticky which resulted in a strong adhesion between the sheet layers in the coil. Thus, the primary prerequisite for choosing a certain type of organic coating is that it should be easy to apply and have stable properties after coating deposition.

The main advantage of the use of a PVD coated forming tool is cleaner and environmentally friendlier SMF workshops since no addition of lubricants is needed. Furthermore, the use of PVD coated forming tools results in controlled friction during the forming process and extended life time of the tool due to reduced wear. Before introducing a PVD-coating into practical applications it is of outmost importance to take into consideration the critical effects of the chemical composition and surface topography. In this thesis it has been shown that the DLC coatings initially have a high tendency to pick-up Zn. This effect can be observed in the friction curve with initially high friction coefficient values due to a combination of a high micro-ploughing component from droplet like asperities and adhered Zn which lead to a high adhesive friction component. However, a running-in procedure consisting of several successive sliding events will result in a smooth DLC surface without any adhered Zn. During further sliding the friction coefficient is maintained at low and stable level. In a real forming process when producing expensive parts, e.g. car body panels a running in procedure of the DLC coating is not realistic. A polishing treatment of the DLC can be a possible solution but is very difficult to perform for complex geometries. Thus, a PVD coating for sheet metal forming applications should be designed in order to promote a running-in, which results in smooth coating topography and a stable friction coefficient. For example, by finishing the DLC coating deposition process with a high graphite content top layer this effect can be achieved.

In chapter 6 the potential of using dry lubricants on the steel sheet to avoid galling was demonstrated. Especially, during severe forming operations high local pressures may cause local coating failure due to either a low load carrying capacity or low adhesion of the polymer. Consequently, localised cold welding followed by material transfer from the sheet to the tool will be expected. In Figure 43 the excellent combination of using dry lubricants and low friction PVD coatings (DLC) is demonstrated by ball-on-disc testing. By combining the two surface engineered concepts a sudden failure of the organic coating is not critical since the self-lubricating DLC-coating will avoid cold welding and material transfer at the sliding interface. Thus, this is a clear evidence of how combinations of surface engineering concepts have potential to improve the stability and quality of SMF processes.

8.2 Future trends

In the future the need for maintenance and a reduction of environmental degradation will be a frequently discussed issue and an important task to solve in SMF processes. Therefore, new environmentally friendly surface treatments will have an important role in the industry. Of these, multifunctional pre-treatments such as the deposition of thin organic coatings on metal coated steel will probably become more and more important. The focus will be aimed on cost effective environmental friendly products with optimised multifunctional properties. Consequently, further work on optimising the forming properties, corrosion properties and paintability properties of thin organic coatings is needed. However, in order to further improve the performance of organic coated steel sheet, optimisation of the production lines is needed to obtain a well-defined and smooth surface topography as well as a well-controlled organic coating thickness.

As this thesis has shown, low friction and high wear resistant PVD coatings will most probably play an important role in the future in the development of SMF applications. Today, many PVD coatings need a running-in process or need to be polished in order to obtain controlled friction coefficients. Since the polishing is difficult to perform and the running-in process is time/material consuming future PVD coatings have to be further designed to promote a running-in process, resulting in a smooth coating topography and a stable friction coefficient. This can be obtained by ending the PVD process with the deposition of a relatively low wear resistant coating material such as graphite. Alternatively, the PVD-process may be finished by a sputter etching step thus removing the surface irregularities in a more effective way.

The development of new materials for SMF processes will be of a never ending interest in the future and the eagerness of solving the intricate problem sets the limit whether the aim of dry forming press shops will be achieved or not.

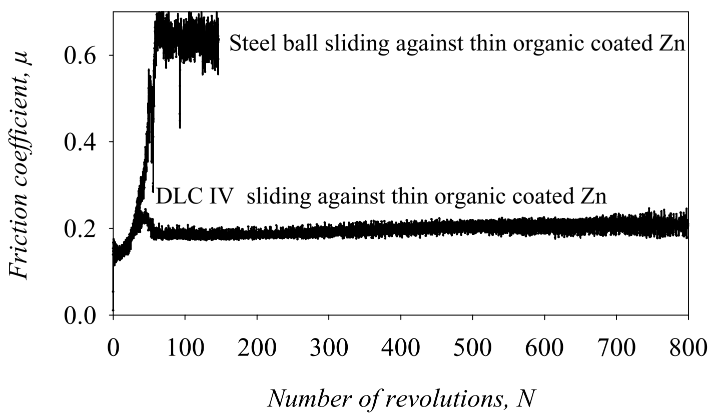


Figure 43. Ball-on-disc friction data showing the potential of combining the surface engineered concepts resulting in a maintained low friction coefficient after the thin organic coating has failed.

9

Conclusions

This thesis deals with self lubricated tribo surfaces, *i.e.* thin organic coatings on steel sheet and PVD coatings on tool, aimed for making sheet metal forming processes more environmentally friendly. The sheet materials investigated include Zn and 55%Al-Zn metal coated steel sheet, which in general are difficult materials to form under dry conditions since they are sticky and thus have a high tendency to adhere to the tool surface. In order to avoid this problem two types of surface engineered concepts, *i.e.* the deposition of thin organic coatings on the steel sheet and PVD coatings on the tool, have been evaluated. The PVD coatings include CrN, TiN and various DLC coatings. The work comprises tribo testing and post test characterisation using surface analytical techniques such as SEM/EDS, AES and ToF-SIMS in order to evaluate the tribological properties of the tribo surfaces. These tests also give valuable information of the wear and friction characteristics of coatings.

From the obtained results the following conclusions can be drawn:

- Tribological model tests such as modified scratch testing and ball-on-disc testing are easy to perform techniques, which can be used as screening tests to rank thin organic coatings and PVD coatings with respect to their performance in forming applications.
- The deposition of thin organic coatings on the steel sheet metal has been found to be promising in order to control the friction and to avoid metal-metal contact resulting in galling. However, it has been found that the tribological characteristics of organic coated steel sheet are strongly influenced by coating chemical composition, the substrate surface topography and coating thickness distribution.
- During a tribological contact the tendency to material transfer of the organic coatings to the tool surface strongly prevent the formation of metal-metal contacts and the performance of the SMF process.

- Of the PVD coatings investigated the DLC coatings have potential to work in a real SMF process under dry conditions, especially for Zn coated steel.
- The performance of the PVD coatings depends mainly on the chemical composition and topography of the coated surface. By choosing PVD coatings such as diamond like carbon (DLC) low and stable friction coefficients are obtained in sliding contact against Zn.
- Surface irregularities such as droplet-like asperities may cause an initial high friction coefficient. However, after a running in process or by polishing the PVD coating controlled friction coefficients can be obtained.
- The combination of imaging (LOM, SEM) and chemical analytical techniques (EDS, AES, ToF-SIMS), gave valuable information concerning the friction and wear properties of the tribo surfaces investigated.

10

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