The Positive Effect of Nitrogen Alloying of Tool Steels Used in Sheet Metal Forming

IRMA HEIKKILÄ
Sheet metal forming processes are mechanical processes, designed to make products from metal sheet without material removal. These processes are applied extensively by the manufacturing industry to produce commodities such as heat exchangers or panels for automotive applications. They are suitable for production in large volumes.

A typical problem in forming operations is accumulation of local sheet material adherents onto the tool surface, which may deteriorate the subsequent products. This tool failure mechanism is named galling. The aim of this work is to explain the mechanisms behind galling and establish factors how it can be reduced.

The focus of this work is on the influence of tool material for minimum risk of galling. Experimental tool steels alloyed with nitrogen were designed and manufactured for systematic tribological evaluation. Reference tool materials were conventional cold forming tool steels and coated tool steels. The sheet material was austenitic stainless steel AISI 304, which is sensitive for galling. A variety of lubricants ranging from low to high viscous lubricants were used in the evaluation.

The properties of the tool materials were characterized analytically and their tribological evaluation included industrial field tests and several laboratory-scale tests. The testing verified that nitrogen alloying has a very positive effect for improving galling resistance of tool steels. Tool lives comparable to the coated tool steels were achieved even with low viscous lubricants without poisonous additives.

The hypothesis used for the explanation of the positive effect of nitrogen alloying is based on the critical local contact temperature at which the lubrication deteriorates. Therefore, the contact mechanism at the tool-sheet interface and the local energy formation were studied systematically. Theoretical considerations complemented with FEA analysis showed that a small size of hard particles with a high volume fraction gives low local contact loads, which leads to low frictional heating. Also, an even spacing between the hard particles and their frictional properties are of importance. Nitrogen alloyed tool steels have these properties in the form of small carbonitrides.

The finding of this work can be applied to a wide range of applications that involve sliding metal contacts under severe tribological loading.

Keywords: galling, nitrogen, tool steel, sliding contact, metal forming
To my mother
List of Papers

This thesis is based on the following papers, which are referred to in the text by their Roman numerals.


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The authors’ contribution

<table>
<thead>
<tr>
<th>Paper</th>
<th>Contributions</th>
</tr>
</thead>
<tbody>
<tr>
<td>Paper I</td>
<td>Planning, manufacture of alloys, evaluation, writing</td>
</tr>
<tr>
<td>Paper II</td>
<td>Planning, testing, evaluation, writing</td>
</tr>
<tr>
<td>Paper III-VI</td>
<td>Planning, major part of testing and evaluation, writing</td>
</tr>
</tbody>
</table>
1. Introduction ........................................................................................................... 9
  1.1 Practical experiences from sheet metal forming ........................................... 9
  1.1.1 Tribology in sheet metal forming .............................................................. 9
  1.1.2 Tool material aspects ............................................................................ 11
  1.1.3 Sheet materials difficult to form .......................................................... 14
  1.1.4 Lubrication .......................................................................................... 16
  1.2 Aim of the thesis ....................................................................................... 17
2. Contributions ...................................................................................................... 18
  2.1 Experimental .............................................................................................. 18
    2.1.1 Conventional tool materials ............................................................... 18
    2.1.2 Experimental tool steels .................................................................... 19
    2.1.3 Sheet materials .................................................................................. 20
    2.1.4 Lubricants ......................................................................................... 21
    2.1.5 Tribological testing ............................................................................ 22
    2.1.6 Characterization methods ................................................................... 26
  2.2 Summary of results ..................................................................................... 29
    2.2.1 Properties of tool steels ...................................................................... 29
    2.2.2 Properties of oxidized sheet materials .............................................. 33
    2.2.3 Adhesion at tool-sheet interface in dry contacts
      (Paper II, III, V) .................................................................................... 33
    2.2.4 Adhesion at tool-sheet interface in lubricated contacts .................. 34
    2.2.5 Adhesion of oxidized sheet materials in dry and lubricated tests
      (Paper VI) ............................................................................................. 38
    2.2.6 Temperature generation at tool-sheet interface
      (Not published) .......................................................................................... 39
  2.3 Discussion .................................................................................................... 40
    2.3.1 The critical contact temperature criterion ......................................... 41
    2.3.2 Adhesion and friction of tool steel phases ......................................... 42
    2.3.3 Influence of tool steel microstructure on galling ......................... 43
    2.3.4 Influence of lubrication for galling ................................................... 44
    2.3.5 Contact mechanisms ......................................................................... 45
    2.3.6 Effect of surface oxides on sheet materials for galling
      initiation .................................................................................................. 47
  2.4 Conclusions and future perspectives ............................................................ 48
    2.4.1 Conclusions ...................................................................................... 48
    2.4.2 Future perspectives ........................................................................... 49
1. Introduction

Metal forming was an important part of engineering – to produce commodities for everyday life – already in the pharaoh eras in ancient Egypt. For a few thousands of years the forming methods were founded on blacksmith experience alone. Systematic research on metal forming started in France by Coulomb and Tresca during the 17th century. Today we are living in the midst of millions of products manufactured through these decently well-calculated methods. Our lives are becoming formed by forming./1/

Metal forming processes include bulk forming and sheet metal forming processes. Typical bulk forming processes are rolling, extrusion and forging. This work will concentrate primarily on sheet metal forming, which is nearly always performed as a cold operation without any prior heating of the work material. A typical characteristic for the metal forming processes is that the need of forming forces is high. Robust machines such as large hydraulic presses are used to fulfill the force requirements. The goal of the forming operation is to transform a sheet blank into a useful part of complex geometry without affecting the thickness of the sheet. Sometimes elastic recovery of the sheet i.e. springback may be significant. The process of forming a sheet metal is very complex and often relies on a considerable amount of experience. The formability of sheet metals is affected by a large number of parameters such as material properties, tool design and setup and the complex interaction between the sheet, tool and lubrication. /1,2,/ 

1.1 Practical experiences from sheet metal forming

1.1.1 Tribology in sheet metal forming

Common sheet metal forming processes include bending, deep drawing, ironing, punching, stretch forming, spinning and stamping to name a few. Any forming process system is composed of the machine, the work piece and the interface between the tool and work piece, where the transfer of mechanical and thermal energies takes place. The physical phenomena describing a forming operation are difficult to express with quantitative relationships. The metal flow, the friction at the tool/material interface, the heat generation and transfer during plastic flow, and the relationships between microstructure, properties and process conditions are difficult to predict and analyze. For the understanding and optimization of metal forming operations
it is useful to consider metal forming as a system in order to be able to classify involved phenomena in a systematic way, c.p. Fig. 1a. /2,3,4/

![Figure 1. a) Typical tool arrangement for sheet metal forming b) close up of critical areas of the contact between die and sheet.](image)

In this thesis the critical areas of contact pressure, sheet deformation and sliding illustrated in Fig. 1 b, are studied in a systematic way. Important input variables are the properties of the forming tool and sheet material, the conditions at the tool/material interface, the environment and the deformation process. These variables contribute to the product quality and process efficiency as well. The direction of metal flow, magnitude of deformation and the involved temperatures greatly influence the success of the forming operation./2,3/

A typical instability encountered in forming operations is formation of local sheet material adherents onto the tool surface. This tool failure mechanism is named galling.

The galling process and its initiation mechanisms have been studied extensively. It has been stated that galling is a process composed of three stages: the initiation, gradual growth of adhesion pick-ups, and severe accumulation of work material onto the tool surface. During the two first stages, the amount of adhered work material is microscopic or hardly visible, and no disturbances can be observed in the process stability or product quality. Sometimes the formed adhesion pick-ups can disappear, if the interface between the tool and adhered pick-ups is weak enough for the adherent to be torn away during the following strokes. During the last stage of the galling process unstable friction conditions are formed and visible flaws such as indents or scratches can be found in the surface of the product. They are formed through a ploughing action of the large adhered accumulates./4,5,6,7/

Much of the galling research has concerned relatively rough tool surfaces and it has become clear that reducing surface roughness of the tool has a positive effect for avoiding galling. Studies on galling initiation mechanism on smooth polished surfaces are less common, but an increasing amount of research has been published on this area recently. Here, it is relevant to con-
sider interatomic forces between the contacting surfaces, tribochemistry and structure of the surfaces at a nano-scale./7,8,9/

1.1.2 Tool material aspects

The commonly used tool materials in sheet metal forming are cast iron, cast steel, aluminium bronzes, tool steels, and highly alloyed materials produced by powder metallurgy. For processes where relatively cheap tool materials are needed, for instance for large dies, or low loaded applications, cast tool materials or aluminium bronzes are chosen. Forming tools subjected to more demanding loading conditions need alloyed tool steels. Another group of material applied for demanding loading situations is carbides consolidated to a matrix material through powder metallurgical processes. Examples of such materials are cemented carbides and Ferro-Titanit (TiC + steel matrix). The tool life will generally increase if a surface treatment and/or a coating is applied to the material. Within the scope of this work uncoated and coated tool steels are examined. /5,10,11/

Tool steels

In sheet metal forming the most commonly used group of tool steels is cold work tool steels. High contents of alloying elements are used in cold work tool steels to produce alloy carbides in a martensitic matrix during heat treatment. Typical characteristics for cold work tool steels include high hardness (58-62 HRC) and high compressive strength, relatively good toughness, good machinability in soft annealed condition, dimensional stability during heat treatment and polishability. Typical alloying elements used for formation of alloy carbides include Cr, V, Mo, and W. The carbon content of the alloys is between 0.4-2.9 wt%. The content of formed alloy carbides can vary from 6-30 wt%. /11,12/

Tool steel materials delivered to customers have a microstructure of uniformly dispersed spheroidized carbide particles in a matrix of equiaxed ferrite grains, which will facilitate machining of the material into a desired geometry. The treatment used to achieve this microstructure is called soft annealing. The machined tool component is then ready for heat treatment which will involve pre-heating, austenisation, quenching and two tempering stages. Austenisation is critical to maximize the amount of martensite formed in quenching and to dissolve sufficient alloying elements for secondary hardening during the tempering. /13/

During austenisation the soft annealed structure reacts to form grains of austenite (face centered cubic crystal fcc) that grow until grains impinge upon another, thus establishing an initial austenite grain size. Continued heating results in growth of austenite grains and solution of excess carbide.
The austenite contains solute atoms such as C and N occupying the interstitial positions of the fcc structure and other elements substituting iron on the lattice points. An increase in austenisation temperature increases lattice vibrations and gives more open structure of austenite to accommodate more of the alloying elements. Longer times at the austenisation temperature are required for the steels containing strong carbide forming elements. Finely dispersed carbides retard austenite grain growth. Fine grain size is desirable in austenising to minimize the possibility of distortion and cracking in hardening and for an optimal combination of hardness and toughness./13,14/

Following austenisation a tool must be cooled at a rate great enough to yield a martensite structure with very little intermediate decomposition products. Strong carbide formers can tie up carbon so effectively that part of the austenite retains austenite after quenching. Thus, the microstructure produced in quenching consists of a mixture of martensite, retained austenite and retained carbides. In tempering, the toughness of martensite increases, retained austenite is transformed to martensite and precipitation of alloy carbides occur. The coarse retained carbides are stable throughout the tempering process./13,14,15/

Cold work tool steels can be manufactured through conventional ingot metallurgy or through powder metallurgy techniques. The manufacturing route has a large effect for the size and shape of the primary carbides formed during solidification. In ingot metallurgy the cooling rates are slow and the primary carbides become large, meanwhile in powder atomization the cooling rates are fast and the primary carbides become small. Powder metallurgy can also be utilized to produce high contents of alloy carbides. However, if the carbide content of the tool steel becomes higher than 30% problems with machinability are frequently encountered. The amount and size of carbides contribute to the wear resistance of the materials./11/

The stages commonly involved in the manufacture of PM tool steels are atomization in an inert gas, capsulation, hot isostatic pressing (HIP), forging, soft annealing and the subsequent machining and heat treatment of the tool. Moreover, also other techniques are available for the production and treatment of the powder which are used to introduce new alloying elements in the steel or for instance to reduce the oxygen contents./11/

**Nitrogen alloyed tool steels**

Nitrogen can be used as a substitute for carbon in tool steels. Nitrides or carbonitrides are formed in the microstructure instead of alloy carbides. The first studies on the effect of nitrogen on the properties of high-speed steels (HSS) were made by Kawai et al during the 1970 and 80s. This work resulted in an introduction of commercial high speed steel alloys with 0.4-0.6% N. /16,17/
The use of nitrogen as an alloying element in steel was not considered interesting until year 1912, when Andrew observed that N strongly stabilizes austenite and has a positive effect on mechanical properties of steel. Adcock made similar observations in year 1926 when alloying Cr and Cr-Fe alloys with N. During the World War II the interest for using N as an alloying element increased as there was a shortage of Ni. During the same period of time, the positive effects of N for corrosion resistance were discovered. /17,18/

During the 50s large efforts were put into developing the process technology for N alloying. The introduction of the Ar-O degassing (AOD) technique made it possible to alloy with N as a gas. In the 60s the first commercial N alloyed bearing steels appeared. From there on N alloying has been an important for the developments of many different steel grades including HSLA steels, high Mo corrosion resistant steels, duplex stainless steels etc. /17/

Nowadays, a lot of research concerns nitrogen alloyed PM tool steels and a crucial reason for this is the advances in PM technology which makes it a competitive method to produce alloys for cold and hot working applications. High nitrogen contents can be achieved by solid state nitriding powder particles prior compaction. For solid state nitriding fluidized beds or rotary furnaces are used with ammonia or ammonia-hydrogen gas mixtures at temperatures 500-600°C. Nitriding can also be made in pure nitrogen or nitrogen-hydrogen mixtures at 1050°C. Nitrogen can also be introduced as an alloying element during atomization, but then only lower N contents are achieved. An important tool for the development of N alloyed steels has been the use the thermodynamic computational calculations. /18,19/

Several authors have reported that the nitrogen additions of 0,4-0,6% to PM HSS gives generally an improved toughness and resistance to adhesive wear. High nitrogen additions in cold work tool steels have shown very positive effect for their galling resistance. The mechanisms how increased galling resistance is achieved are not comprehensively understood. /18,20,21,22/

**Coated tool steels**

Physical vapour deposition (PVD) and chemical vapour deposition (CVD) techniques are commonly used for coating cold work tool steels. Many of these coatings have high hardness and good wear properties and they exhibit good bonding to the substrate.

PVD is a process to produce a metal vapor that can condense on electrically conductive materials as a thin (2-5 µm) highly adhered pure metal or alloy coating. The process is carried out in a chamber at high vacuum using a cathodic arc source or other sources. Single or multi-layer coatings can be applied during the same process cycle. The metal vapor may react with vari-
ous gases to deposit oxides, nitrides, carbides or carbonitrides. One advantage with the PVD technique is that the process temperatures can be kept below the tempering temperature of the tool steel. The PVD coatings are usually polished after the deposition. /5,23/

CVD is an atmosphere controlled process conducted at elevated temperatures (800-1200°C) in a CVD reactor. The coatings (5-10 µm thick) are formed as a result of reactions between various gases and a heated substrate. The bond between the substrate and the coating is very good due to the formation of an intermediate diffusion layer. Complex geometries can be coated with the CVD process. However, hardening has to be performed in connection to or after the coating process, and requirements regarding the tolerances of the tool can limit the use of this coating type. /24/

Two of the most common PVD coatings on forming of steel are TiAlN and AlCrN. In demanding forming operations of stainless steel a low friction diamond like coating (DLC) composed of WC and amorphous carbon is commonly used. The most often used CVD coating in forming of carbon steel is TiCTiN and in forming of stainless steel TiC coating. /23,24/

The Toyota diffusion (TD) is a process, where a homogenous hard layer of carbides is formed on a steel substrate. Typically a layer of TiC or WC layer is used. The layer is deposited in a salt bath at high temperature (800-1250°C). There is a risk of dimensional changes due to the high temperatures involved. /25/

1.1.3 Sheet materials difficult to form

The most common materials suitable for sheet forming are carbon steels, stainless steels, aluminium and copper alloys and titanium. Sheets of magnesium alloys are used in limited extent because of their poor formability. Within each material type a wide range of commercial alloys is available. Each of them meets different demands on the final product in terms of mechanical properties, corrosion properties, thermal and electrical conductivity, weight, aesthetic appearance, cost aspects, etc. Several sheet material types are supplied with coatings, paints, films or a special topographic finish for aesthetic appearance. The range of the products manufactured from sheet metals covers a wide spectrum of commodities such as car bodies, household utensils, heat exchangers and constructional parts. /2/

Within the scope of this work sheet materials relatively difficult to form are considered. The focus is on stainless steel and to some less extent on titanium. A short description of characteristics of these materials relevant to galling is reviewed below.
Stainless steels

Stainless steels are classified by their crystal structure as austenitic, ferritic, martensitic, or duplex stainless steels.

Austenitic stainless steels have face centered cubic crystal (fcc) structure. They make up over 70% of the total stainless steel production. They are alloyed with minimum 16% Cr and at least 8% Ni, which gives them very good resistance against corrosion. These compositions together with additions of manganese will retain an austenitic structure at all temperatures, from the cryogenic region to the melting point of the alloy. The most widely used austenite steel is AISI 304 with a composition of 18% Cr and 8% Ni. The second most common austenite steel is the AISI 316 used in marine environments for its increased corrosion resistance. Austenitic stainless steels have a reputation of being sensitive to galling.

Ferritic stainless steels have body centered cubic (bcc) crystal structure. They are alloyed with Cr ranging from 12% and up to 27% for harsh environments. Most compositions include Mo, some Al and Ti, but no significant amount of Ni. Common ferritic grades AISI 409 and 430 are used in household utensils. Ferritic stainless steels have good formability and are much less sensitive to galling than the austenitic.

Martensitic stainless steels have bcc or tetragonal crystal structure. They are not as corrosion-resistant as the other two classes but have higher strength, good toughness, and a better machinability. These steels can be hardened by heat treatment. Martensitic stainless steel contains 12–14% Cr, 0.2–1% Mo, less than 2% Ni, and about 0.1-1% C. The common martensitic grades AISI 410 and 414 are used in stressed parts needing the combination of strength and corrosion resistance such as fasteners, springs and instruments.

Duplex stainless steels contain almost equal fractions of austenite and ferrite. They contain 19-23% Cr and up to 5% Mo, meanwhile the Ni content is lower than in austenitic stainless steels. These steels have high strength and good resistance to localized corrosion such as stress corrosion cracking. The low alloy content together with good properties makes their use cost-effective for many applications such as in transportation, containers and paper machines.

The good corrosion properties of stainless steels are achieved through a thin chromium-containing oxide layer that is formed by a reaction between Cr and O in the air. The layer is only a few atoms thick due in part to the ability of the Cr atoms and O atoms to pack together tightly and thus passivates the surface from further oxidation. This layer protects the steel underneath and is quickly reformed if the surface is damaged by scratching. /26/

Stainless steel sheets are delivered with different standardized surface appearances. These surfaces are created by rolling or by mechanical abrasion.
The final finishing process is preceded by several manufacturing stages. First, the steel is rolled to size and thickness with subsequent annealing. Thereafter the thick oxide layer formed in rolling is removed by pickling. The passivation layer forms after the pickling. If desired, a final finish is then applied to achieve the desired aesthetic appearance. The surface can contribute to the success of the lubrication in forming processes. /26,27/

During manufacturing, inclusions will form in the microstructure of stainless steels such as oxides, sulphides, complex inclusions and nitrides. They can typically cover about 0.08% of the steel volume and they can induce an abrasive action to the tool under sliding motion during the deformation process. /28/

**Titanium**

Titanium has a close-packed hexagonal (α phase) crystal structure. At about 890°C, titanium undergoes an allotropic transformation to a bcc (β phase) which remains stable to the melting temperature. Ti and alloyed Ti products are light in weight, have good corrosion resistance and are able to withstand extreme temperatures. However, the high cost of both raw materials and processing limit their use. Ti alloys are used in military and aerospace applications, medical devices, heat exchangers and in sport cars.

Ti alloys are less ductile than stainless steels and have a relatively low modulus of elasticity. Their properties limit stretch formability and results in a relatively large springback effect during forming. They are also highly sensitive to galling. /29,30/

1.1.4 Lubrication

Lubrication is employed to control the friction and to protect the tool and work piece from wear. Separate areas of the tool surface can be subject to hydrodynamically lubricated conditions, mixed or boundary lubricated conditions. The most exposed areas are those operating under boundary film conditions. In these areas, which are the ones focused on in this thesis, the sliding contact is supported both by an adsorbed lubricant film and by metallic contacts. Friction and wear control under boundary lubricated conditions require specific additives that form boundary layers at the contacting surfaces. The performance of these lubricants is connected to the thermal stability of the formed boundary layer.

The boundary layers can be formed through three mechanisms: physical adsorption, chemical adsorption and chemical reaction. Physically adsorbed layers adhere to metallic surfaces by polar heads of long hydrocarbons. In chemical adsorption, chemically reactive metallic surfaces and the lubricant form a metal soap at the contact interface. Physical adsorption further
strengthens the formed bond. Lubricant additives that form boundary layers through chemical adsorption are usually called friction modifiers. In the third group of boundary layers a chemical reaction between the lubricant additives and the metallic surfaces takes place which results in formation of metal salts. The additives used to form metallic salts are complex compounds based on P, S, B, and Cl and they give good stability even at high contact temperatures. These additives are called extreme pressure (EP) – additives. The drawbacks with their use are that they are either highly eco-toxic, poorly biodegradable or suspected to cause cancer. In addition, they are difficult to remove in the subsequent cleaning process. Recently, the use of lubricants with EP additives has been restricted through environmental legislation and other restrictions. A general trend is to use environmentally friendly, low viscous lubricants and hence to give more attention to the optimization of the whole tribological system involved in sheet metal forming. /3,31,32,33/

1.2 Aim of the thesis

The aim of this thesis is to get an increased understanding of the interaction mechanisms involved in galling phenomena in sheet metal forming. Large plastic strains of the work material caused by rough surface topography of the tool were eliminated in this work by rigorous polishing of all the tested tools and tool material surfaces. In this way the interaction mechanisms at the tribological interface depend sensitively on the material properties of the contacting bodies and the environment of the forming process. The focus of this work is on understanding the role of the tool steel microstructure for providing a suitable substrate for a lubricant film. A special interest is laid on nitrogen alloyed tool steels. The results of this work can be applied to a much broader area of tribology than that of forming tools, for instance at sliding contacts in machine elements.
2. Contributions

2.1 Experimental

2.1.1 Conventional tool materials

The test materials of this study were tool steels with varying alloy composition and coated tool steels, see Table 1 and 2.

Vanadis 23 and Vanadis 6 are PM tool steel grades from Uddeholms AB. Sverker 21 and Weartec are steel grades from Uddeholms AB manufactured by conventional ingot metallurgy and spray forming processes, respectively. Ferrotitanit is a tool material from Edelstahl Witten-Krefeldt GmbH and it has a high content of TiC particles embedded into alloyed steel binder phase. The material is manufactured by powder metallurgy techniques. The tool steels were heat treated to 62 HRC.

The coatings of this study were deposited by PVD, CVD. Also TD processes was applied as surface treatment. The substrate materials were PM tool steels.

Table 1. Conventional and coated tool steels of this study

<table>
<thead>
<tr>
<th>Tool steels</th>
<th>Manufacturing</th>
<th>Coated tool steels</th>
<th>Coating process</th>
</tr>
</thead>
<tbody>
<tr>
<td>Vanadis 23</td>
<td>PM</td>
<td>Vanadis 23 + TiCN / TiC</td>
<td>CVD</td>
</tr>
<tr>
<td>Vanadis 6</td>
<td>PM</td>
<td>Vanadis 23 + VC</td>
<td>TD</td>
</tr>
<tr>
<td>Sverker 21</td>
<td>Ingot</td>
<td>Vanadis 23/4 + TiN</td>
<td>PVD</td>
</tr>
<tr>
<td>Ferrotitanit</td>
<td>PM</td>
<td>Vanadis 23 + VN</td>
<td>PVD</td>
</tr>
<tr>
<td>Weartec</td>
<td>Spray forming</td>
<td>Vanadis 4 + TiAlN</td>
<td>PVD</td>
</tr>
</tbody>
</table>

Table 2. The nominal chemical composition of the conventional tool steels

<table>
<thead>
<tr>
<th>Tool steel</th>
<th>C</th>
<th>N</th>
<th>Cr</th>
<th>Mo</th>
<th>W</th>
<th>V</th>
<th>TiC</th>
</tr>
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<tbody>
<tr>
<td>Vanadis 23</td>
<td>1.28</td>
<td>0.05</td>
<td>4.10</td>
<td>5.00</td>
<td>6.40</td>
<td>3.10</td>
<td></td>
</tr>
<tr>
<td>Vanadis 6</td>
<td>2.07</td>
<td>0.05</td>
<td>6.80</td>
<td>1.50</td>
<td>0.10</td>
<td>5.35</td>
<td></td>
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<tr>
<td>Sverker 21</td>
<td>1.60</td>
<td></td>
<td>11.80</td>
<td>0.80</td>
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<td>0.80</td>
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<tr>
<td>Ferrotitanit</td>
<td>0.75</td>
<td></td>
<td>13.50</td>
<td>3.00</td>
<td></td>
<td></td>
<td>33</td>
</tr>
<tr>
<td>Weartec</td>
<td>2.90</td>
<td>7.10</td>
<td>2.60</td>
<td></td>
<td></td>
<td>8.80</td>
<td></td>
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</table>
2.1.2 Experimental tool steels

Influence of tool material composition and structure was primarily focused to the influence of nitrogen alloying. Some early studies indicated that nitrogen alloying of tool steel would reduce the risk of galling in sliding contacts /12/. To explore the validity of this finding for forming tool steels, a set of nitrogen alloyed steels were developed. A large test matrix covering several chemical compositions and carbon/nitrogen ratios were created, and the effects of their microstructure for galling resistance were evaluated in a systematic manner. In this thesis, four of these alloys have been selected for demonstration, see Table 3. The content of the alloying elements was determined by the aid of Thermodynamical equilibrium calculations. The alloy designated as Annealed is the Vancron 4 material heat treated at 1150 °C for 550 h in order to achieve a morphology for the hard phase particles close to that of conventional ingot steels.

Table 3. The nominal chemical composition of the experimental tool steels

<table>
<thead>
<tr>
<th>Tool steel</th>
<th>Alloying elements wt%</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>C</td>
</tr>
<tr>
<td>Vancron 1</td>
<td>1.28</td>
</tr>
<tr>
<td>Vancron 2</td>
<td>0.58</td>
</tr>
<tr>
<td>Vancron 3</td>
<td>1.96</td>
</tr>
<tr>
<td>Vancron 4</td>
<td>1.20</td>
</tr>
<tr>
<td>Annealed</td>
<td>1.20</td>
</tr>
</tbody>
</table>

The processing stages of these novel materials involved in the manufacture of the experimental tool steel alloys were gas-atomization to form the steel powder, nitriding of the powder particles, capsule filling, HIPing, forging and subsequent heat treatment. The size of each charge was 10 kg.

Nitrogen was introduced as an alloying element to the steels by nitriding powder particles in ammonia based gas mixtures at elevated temperatures (typically 550°C), in the furnace of Fig.2. The transfer process of nitrogen from gas phase into the solid material involves ammonia dissolution reactions on the particle surfaces and diffusion controlled transfer phenomena inside the metal particles. The alloying elements of high affinity to nitrogen such as vanadium, chromium and iron will react with nitrogen and nucleate nitrides in the material. During subsequent processing of the material the growth of nitrides continues. However, the thermodynamical stability of carbonitrides is very high, which results in slow growth rates of the carbonitrides during different steel processing stages. Consequently, the resulting particles are small, of the order of 1 µm or less.

All the experimental tool steels were hardened and tempered to a hardness close to 62 HRC.
2.1.3 Sheet materials

The majority of testing within this work was made with the austenitic stainless steel grade AISI 304. Also, the duplex stainless steel grade ASTM S32101, electro galvanized carbon steel DP 600+ZE and titanium ASTM grade 1 CP sheets were tested as received or with thick, protective surface oxide layers, see Table 4 and 5.

Table 4. Nominal chemical composition of the sheet materials

<table>
<thead>
<tr>
<th>Sheet</th>
<th>C (wt%)</th>
<th>Cr</th>
<th>Ni</th>
<th>Mn</th>
<th>Si</th>
<th>N (wt%)</th>
<th>Mo</th>
<th>Nb</th>
<th>Fe</th>
<th>Ti</th>
</tr>
</thead>
<tbody>
<tr>
<td>AISI 304</td>
<td>0.08</td>
<td>18.0</td>
<td>8.0</td>
<td>2.0</td>
<td>0.7</td>
<td>0.1</td>
<td></td>
<td></td>
<td>Bal.</td>
<td></td>
</tr>
<tr>
<td>ASTM S32101</td>
<td>0.03</td>
<td>21.5</td>
<td>1.5</td>
<td>5.0</td>
<td>0.7</td>
<td>0.1</td>
<td></td>
<td></td>
<td>Bal.</td>
<td></td>
</tr>
<tr>
<td>DP 600</td>
<td>0.10</td>
<td></td>
<td>0.8</td>
<td>0.2</td>
<td></td>
<td>0.015</td>
<td></td>
<td></td>
<td>Bal.</td>
<td></td>
</tr>
<tr>
<td>ASTM 1 CP</td>
<td>0.08</td>
<td></td>
<td></td>
<td></td>
<td>0.3</td>
<td>0.03</td>
<td></td>
<td></td>
<td>0.03</td>
<td>Bal.</td>
</tr>
</tbody>
</table>

AISI 304 material was tested as sheet material of thickness 0.5-1.0 mm, and in some tests as rods. The sheets were cold rolled, annealed, pickled and skinpassed. The latter treatment gives a surface finish commonly designated as 2B. The roughness of these sheets both parallel and across the rolling direction was $R_a = 0.10-0.28 \mu m$. The surface roughness of the bars was $R_a = 0.15 \mu m$ parallel and $R_a = 0.20 \mu m$ across the grinding direction.

The surface of duplex stainless grade ASTM S32101 was manufactured by cold rolling and pickling. The electro galvanized carbon steel DP 600 was tested without the lacquers or oils that are usually applied for this material. The technically pure titanium sheet grade was manufactured by cold rolling. The manufacturing influences the mechanical properties, meanwhile the
thermal conductivity is less sensitive for the manufacturing route of the sheets, see table 5.

Table 5. Some mechanical and thermal properties of the sheet materials

<table>
<thead>
<tr>
<th>Material</th>
<th>AISI 304</th>
<th>ASTM S32101</th>
<th>DP 600</th>
<th>ASTM 1 CP</th>
</tr>
</thead>
<tbody>
<tr>
<td>Yield strength MPa</td>
<td>210-230</td>
<td>600</td>
<td>350-480</td>
<td>170</td>
</tr>
<tr>
<td>Tensile strength MPa</td>
<td>520-540</td>
<td>840</td>
<td>600-700</td>
<td>240</td>
</tr>
<tr>
<td>Elongation %</td>
<td>45</td>
<td>40</td>
<td>16</td>
<td>25</td>
</tr>
<tr>
<td>Thermal conductivity W/m°C</td>
<td>15</td>
<td>15</td>
<td>43</td>
<td>16</td>
</tr>
</tbody>
</table>

In order to evaluate oxide layers as lubricants in sheet forming, thick protective oxides were grown on three of the sheet materials. The oxide layer on the duplex stainless steel was the natural oxide achieved in cold rolling. The oxide on Zn-coating was manufactured in humid atmosphere at room temperature. The oxide on the Ti-sheet was grown by heat treating in air at 550°C room that results in formation of an oxide with Magnéli structure /34/.

2.1.4 Lubricants

The lubricants chosen for this work covered a spectrum of metal forming oils with varying contents of additives, see Table 6. All the lubricants came from Quaker Chemicals Netherlands with the exception of Ilo PN 226 and Rhenus SF 150A, which were obtained from Castrol and Rhenus Lub GmbH & Co KG, respectively. Two of the lubricants, Ilo PN 226 and Draw 42K, contain a high amount of chlorine-based EP-additives, whereas all the other lubricants can be characterized as environmentally friendly lubricants with low contents of EP-additives.

Table 6. Lubricants and their properties

<table>
<thead>
<tr>
<th>Lubricant</th>
<th>Viscosity 40°C mm²/sec</th>
<th>Chlorine wt%</th>
<th>Base</th>
</tr>
</thead>
<tbody>
<tr>
<td>Coupex 046 EP</td>
<td>42</td>
<td>0</td>
<td>Mineral oil</td>
</tr>
<tr>
<td>Ilo PN 226</td>
<td>67</td>
<td>High</td>
<td>Paraffinic mineral oil</td>
</tr>
<tr>
<td>Draw 140 EL</td>
<td>162 (20°C)</td>
<td>Low &lt;0.05 wt%</td>
<td>Mineral oil</td>
</tr>
<tr>
<td>Draw 220 VP</td>
<td>220 (20°C)</td>
<td>0</td>
<td>Vegetable polymers</td>
</tr>
<tr>
<td>Draw 7000 RN</td>
<td>71 (20°C)</td>
<td>0</td>
<td>Mineral oil, water, soap</td>
</tr>
<tr>
<td>Draw 42K</td>
<td>220</td>
<td>High 33 wt%</td>
<td>Mineral oil</td>
</tr>
<tr>
<td>Draw 250I</td>
<td>250</td>
<td>0</td>
<td>Vegetable polymers</td>
</tr>
<tr>
<td>Rhenus SF 150A</td>
<td>150</td>
<td>0</td>
<td>Mineral oil</td>
</tr>
</tbody>
</table>
2.1.5 Tribological testing

*Industrial field test (Paper III)*

Experimental investigation of the progress of wear in a forming tool was made at an industrial process line at Grundfos A/S, Denmark. In this line a part for a water pump was manufactured through a deep drawing operation, similar to that illustrated in Fig. 1. The stroke rate of the process line was 250 000 - 300 000 strokes per month. The wear of the die was investigated at regular intervals. The area specified for the wear investigation was the inner die radius where the sheet material is subjected to large plastic deformations along the curvature and where the surface temperatures are the highest. The tool life is expressed as the number of strokes before severe galling is detected. The lubricant of the process was Ilo PN 226 and the sheet material was AISI 304 2B. The tested tool materials were Vanadis 23, Vanadis 6, Vancron 4, and Vanadis 23 with CVD TiC coating.

*Slider-on-sheet (Paper I,IV)*

The slider-on-sheet test has been used to simulate the tool-lubricant-sheet interaction generally prevailing in sheet forming operations. The method is developed by TNO Institute of Industrial Technology.\(^{5}\)

A sliding contact between the tool and sheet is realized by pressing and moving a slider against a sheet blank with a specific speed, see Fig 3. A constant normal force $F_n$ is applied to the slider. As the slider meets the end of the sheet blank, it is lifted from the sheet and moved back, so that the test can be continued along a new track under the same conditions. Thus, the slider is always in contact with a fresh sheet material. The friction force and normal force are recorded over each track and thus the coefficient of friction can be presented as a function of sliding length. The sliding test is generally interrupted if no galling is detected before a sliding length of 2000 m. When severe galling occurs, the coefficient of friction rises above 0.2 the testing is interrupted and the specific sliding length associated to occurrence of galling is registered.

*Figure 3. The slider-on-sheet test. The disk represents the tool. Note that the disk does not rotate.*
Flat-on-flat (Paper IV, VI)

The flat-on-flat test simulates typical conditions at a blank holder, e.g. Fig. 1. In this test, a sheet strip is pulled through a pair of flat tools that are pressed together with a clamping force \( F_n \), see Fig. 4. The test can be made in two different ways. The first one is to repeat pulling of the same sheet area through the tool. The lubricant is applied onto the strip only once and testing is repeated until galling occurs. The coefficient of friction is displayed as a function of number of strokes. The second way to make the testing is to introduce a fresh sheet strip to the contact area during each stroke. Here, the coefficient of friction is displayed as a function of sliding length. Different nominal contact pressures can be applied by changing the normal force \( F_n \).

![Figure 4. The flat-on-flat test.](image)

Bending Under Tension (BUT) (Paper V)

The BUT test is designed to simulate conditions in a deep drawing process, e.g. Fig. 1. Sliding contact between a tool and a sheet is provided by drawing a strip over a fixed cylindrical tool, see Fig. 5. The draw force and the back force tensions the strip plastically along the contact area with the tool. Both forces are monitored during the test. The strip is fed from a coil in such way that during each test stroke a fresh sheet material comes to the contact with the tool. The test result is presented as the variation of the friction coefficient with the number of strokes./35/

![Figure 5. The BUT-test.](image)
**Strip reduction (Paper VI)**

The strip reduction test resembles the ironing forming process, see Fig. 6a. In this test a sheet strip is drawn through a stationary tool bar and supporting plate, which gives a thickness reduction for the sheet strip, see Fig 6b. The reduction rate is successively increased until galling occurs at the tool. During each stroke a freshly polished tool surface is applied. The result of this test is the reduction rate at which galling commences.\(^{35}\)

![Figure 6. a) Ironing. b) The strip reduction test.](image)

**Compilation of the test parameters**

The most relevant parameters of the different sliding contact tests are summarized in Table 7.

<table>
<thead>
<tr>
<th>Test</th>
<th>Result</th>
<th>Pressure MPa</th>
<th>Velocity m/s</th>
</tr>
</thead>
<tbody>
<tr>
<td>Slider-on-sheet</td>
<td>µ vs. sliding length</td>
<td>500-600</td>
<td>0.5</td>
</tr>
<tr>
<td>Flat-on-flat</td>
<td>µ vs. stroke number</td>
<td>4-30</td>
<td>0.17-0.25</td>
</tr>
<tr>
<td>BUT</td>
<td>µ vs. stroke number</td>
<td>100</td>
<td>0.1</td>
</tr>
<tr>
<td>Strip reduction</td>
<td>Reduction vs. galling</td>
<td>Max. 2000</td>
<td>0.08</td>
</tr>
</tbody>
</table>

**Model test (Paper II)**

The load scanner test was used to evaluate adhesion in sliding contacts. In this test, two crossed cylindrical specimens slide against each other with gradually increasing normal force, cp Fig. 7. Thus, fresh surfaces are constantly introduced into contact. During sliding the upper test bar stays stationary, while the lower bar moves a predetermined distance thus creating a wear track on each test bar. Because of this test configuration, each position in the wear track corresponds to a well-defined load. During testing, the friction force and loading force are continuously measured. The friction is pri-
arily correlated to the deformation work applied to the softer test material. Adhesion on the tool specimen were studied by LOM and SEM. /37/

![Figure 7. Principle of load-scanner test. The lower test rod, representing the tool, moves horizontally as indicated by the arrows.](image)

**AFM friction test**

The adhesion forces between two different materials can be quantified through theoretical calculations by considering factors such as interatomic forces, chemical reactivity and the surface and interface energies. However, on real engineered surfaces there are always defects, adsorbed species, asperities and crystallographic orientations, which may contribute substantially to adhesion. It is a challenging task to understand and link macroscopic tribological behavior and interactions on an atomic scale. AFM is a technique to quantify attractive forces between contacting surfaces down to the atomic scale. /38,39/

The adhesion tendency at tool-sheet interfaces was simulated by making friction measurements on tool steel surfaces by using the tapping mode in AFM. In this mode, the microscope tip scans the tool surface without any physical contact, while the repulsive and attractive forces between the tip and sample can be determined. These forces are named as van der Waals forces. The microscope records the topography of the specimen simultaneously during the friction measurement. A tip made of silicon and coated with a 25 nm thick layer of W<sub>2</sub>C was used to evaluate the frictional properties of different tool steel phases. It had probably been more appropriate to use a stainless steel tip. However, such a tip is too weak for handling in the AFM. The applied load used in testing was 40 nN./39,40/
2.1.6 Characterization methods

**Thermo-Calc simulations**

Thermo-Calc calculations were used as an aid in alloy design of the experimental nitrogen alloyed tool steel compositions. Thermodynamical calculations were performed at the austenisation temperature of the steel in aim to estimate the contents of hard phases formed in heat treatment. Also, the chemical composition of the different tool steel phases could be achieved from the calculated data. Thermo-Calc software was also used to estimate the same features for the commercial tool steel compositions. The database TCFE6 used in the calculations was specially developed for steels containing of multiple alloying elements including nitrogen. /17/

**Optical and electron microscopy**

Light optical microscopy (LOM) is a widely used method for fast characterization of microstructure features such as phase content, inclusions, corrosion and wear. The spatial resolution of LOM is about 0.5 µm and its depth of focus is very small. Therefore LOM can be exploited for studying features with a topographic contour only to a limited extend. More detailed microstructure studies require use of methods that give higher resolution and greater depth of field. LOM was used for part of the wear analysis in this work./40/

In scanning electron microscopy (SEM) imaging and elemental composition is acquired by interaction between accelerated electrons and the material of the sample./39/ Electrons reflected from the sample can be collected to specific detectors. The most common modes of imaging use backscattered electron (BS) detectors for the elastically scattered electrons and secondary electron (SE) detectors for low energy electrons. For wear analysis SE-detection is used together with low accelerating voltage, which gives high surface sensitivity for the imaging. Also, the working distance (distance between specimen and the magnetic lens) can be increased to obtain a large depth of focus. Modern SEMs have a resolution up to 1-3 nm. Elemental compositions can be determined by analyzing the energy of the X-rays that are emitted from the sample during the electron irradiation. Energy dispersive or wavelength dispersive detectors are used for this purpose. However, the accelerating voltage must generally be kept above 10 kV in order to get statistically reliable information of the composition of the material. SEM was used for characterization of microstructures and wear in this work. /40/
Confocal microscopy

The confocal microscope creates sharp light optical images of specimen that have a rough topography. This is achieved by superimposing series of images and using only the reflected light from the specimen that is not from the microscope’s focal plane. Apart from allowing better observation of fine details it is possible to build three-dimensional reconstructions of a volume of the specimen by assembling a series of thin slices taken along the vertical axis. It is also possible to calculate surface roughness parameters from the achieved data. /41/

Wear progress of industrial forming tools was analyzed with Wyko NT3300 white light confocal microscope. Investigations were made at several sites of size 0.5 x 0.5 mm². Progress of wear was examined at regular intervals and the surface roughness parameters were registered for each investigation. Thus, the development of surface roughness as a function of stroke number could be achieved.

Atomic force microscopy

The topography of the forming tools is generally expressed by the technical roughness \( R_a \). The microroughness, i.e. the roughness level below the technical roughness, can vary significantly within the same technical roughness value. Atomic force microscopy (AFM) can be used to study the topography at a nano-scale. The three dimensional images that AFM provides can be quantified in x, y and z direction. Different carbides typical for tool steels were characterized regarding their topography and height with AFM using the topographic mapping mode. The technical \( R_a \) value was the same for all the studied samples./39/

GDOES

Glow-discharge optical emission spectroscopy (GDOES) is a technique to measure depth profiles of elements in solid samples by detecting the light emission from atoms in a glow-discharge plasma that gradually sputter the sample. This technique is quick, has high depth resolution and is convenient to use since ultra-high vacuum is not needed. GDOES was used to make depth profile analysis of the surfaces of tool steel samples. The aim was primarily to characterize the surface oxides and to measure the thickness of this oxide layer./42/
**XRD**

X-ray diffraction (XRD) is a technique that reveals the chemical composition and crystallographic structure of materials. The method is based on the recording of diffracted X-ray beams according to Bragg’s law during irradiation of the sample with monochromatic X-rays. Brukers X-ray diffractometer model DB was used to identify the crystal structure of surface oxides in sheet materials./43/

**Nanoindentation**

The nanoindentation technique makes use of a triangular-shaped Berkovic tip. The indentation depth is very small and thus the individual hardness of small phases can be identified. Nano hardness values can be converted to Vickers hardness values by multiplying the measured value with 94,59. Nanoindentation was used to measure hardness of carbonitrides in the tool steels. The indentation depth was 50 nm. In each steels the hardness measurements were made with 2 µm intervals along randomly positioned straight lines. High hardness values represent the hard phases, while the low numbers represents the matrix. /44/

**Image analysis**

Micro GOP image analysis was performed from BS SEM images to obtain the equivalent circular diameter ECD size distribution of carbonitrides in the Vancron steels./45/

**FEA**

Finite element analysis (FEA), Abaqus v6.12 Explicit, was applied to simulate a sliding contact between hard phases of a tool steel and an AISI 304 sheet. The hard phases were considered as hard linear elastic spheres and the sheet as an elastic plastic material. The volume fraction of hard phases was 20%. Simulation was made for a tool steel, where the ECD size of the hard phase was 1 and 5 µm, respectively, see Fig.8. The mesh consisted of hexahedral elements with reduced integration. A Coulomb friction of μ = 0,1 was assumed. The hardening of the sheet material was assumed to be isotropic and linear. The sliding velocity was set to 375 mm/s and the nominal normal stress was 125 MPa. A specific heat of 0.49 kJ/kgK was used for the sheet and 0.60 kJ/kgK for the hard phase. Fully coupled thermal-displacement analysis using explicit time integration was applied to simulate the temperature rise at the tribological interface /46/.
Figure 8. Model of hard phases in a tool steel of ECD size 1 µm (a) and 5 µm (b).

2.2 Summary of results

2.2.1 Properties of tool steels

Content, type and chemical composition of tool steel phases

The content and type of hard phases present in the microstructure after the final heat treatment were investigated. The content of phases was estimated through thermodynamical equilibrium calculation using Thermo-Calc database TCFE6, see Table 8. The calculations were made at the austenisation temperature.

Table 8. The estimated content and type of hard phases

<table>
<thead>
<tr>
<th>Tool steel</th>
<th>Hard phase and vol%</th>
<th>TiC</th>
<th>MC</th>
<th>M(C,N)</th>
<th>M₆C</th>
<th>M₇C₃</th>
<th>Tot%</th>
</tr>
</thead>
<tbody>
<tr>
<td>Vancron 1</td>
<td></td>
<td>18.3</td>
<td>4.0</td>
<td></td>
<td></td>
<td></td>
<td>22.3</td>
</tr>
<tr>
<td>Vancron 2</td>
<td></td>
<td>24.9</td>
<td>3.9</td>
<td></td>
<td></td>
<td></td>
<td>28.8</td>
</tr>
<tr>
<td>Vancron 3</td>
<td></td>
<td>33.1</td>
<td>3.9</td>
<td></td>
<td></td>
<td></td>
<td>37.0</td>
</tr>
<tr>
<td>Vancron 4</td>
<td></td>
<td>22.0</td>
<td>4.0</td>
<td></td>
<td></td>
<td></td>
<td>26.0</td>
</tr>
<tr>
<td>Vanadis 23</td>
<td></td>
<td>6.5</td>
<td>8.8</td>
<td></td>
<td></td>
<td></td>
<td>15.3</td>
</tr>
<tr>
<td>Vanadis 6</td>
<td></td>
<td>12.4</td>
<td></td>
<td></td>
<td>1.2</td>
<td>13.6</td>
<td></td>
</tr>
<tr>
<td>Sverker 21</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>15.0</td>
<td>15.0</td>
<td></td>
</tr>
<tr>
<td>Ferrotitanit</td>
<td></td>
<td>33</td>
<td></td>
<td></td>
<td></td>
<td>33.0</td>
<td></td>
</tr>
<tr>
<td>Weartec</td>
<td></td>
<td>16.0</td>
<td></td>
<td></td>
<td>8.1</td>
<td>24.0</td>
<td></td>
</tr>
<tr>
<td>Annealed</td>
<td></td>
<td>22.0</td>
<td>4.0</td>
<td></td>
<td></td>
<td>26.0</td>
<td></td>
</tr>
</tbody>
</table>

There are significant differences in the microstructure between these tool steels. Firstly, the total content of hard phases varies between 13.6-37.0 vol%. Secondly, their type and content are different. Thermo-Calc was utilized to evaluate the chemical composition typical for the different hard phases, see Table 9. MC has a composition close to VC, meanwhile M(C,N) is close to VN. M₆C is a W-Mo-rich carbide and M₇C₃ is a Cr-Fe-rich carbide.
Table 9. Calculated chemical composition of hard phases (wt%) using Thermo Calc.

<table>
<thead>
<tr>
<th>Phase</th>
<th>Structure</th>
<th>C</th>
<th>N</th>
<th>Cr</th>
<th>Mo</th>
<th>V</th>
<th>W</th>
<th>Fe</th>
</tr>
</thead>
<tbody>
<tr>
<td>MC</td>
<td>fcc</td>
<td>16</td>
<td></td>
<td>1-12</td>
<td>11-19</td>
<td>60-65</td>
<td>1-2</td>
<td></td>
</tr>
<tr>
<td>M(C,N)</td>
<td>fcc</td>
<td>5-9</td>
<td>13-15</td>
<td>4-15</td>
<td>1</td>
<td>63-73</td>
<td>&lt;1</td>
<td>1-3</td>
</tr>
<tr>
<td>M₆C</td>
<td>fcc</td>
<td>2</td>
<td></td>
<td>2</td>
<td>26</td>
<td>1</td>
<td>39</td>
<td></td>
</tr>
<tr>
<td>M₇C₃</td>
<td>hexagonal</td>
<td>9</td>
<td></td>
<td>46-52</td>
<td>1-2</td>
<td>5-14</td>
<td>31-37</td>
<td></td>
</tr>
</tbody>
</table>

The matrix of the studied tool steels is martensite with a carbon content of about 0.5-0.7, wt%, Fe 91-93 wt% and Cr 3-6 wt%. Also, small amounts of Si, Mn, Mo and V are dissolved in the matrix.

Examples of the microstructure of some tool steels in fully heat treated condition are given in Fig.9.

![Fig. 9. Microstructure of Vancron 4 (a) and Sverker 21 (b).](image)

Size and size distribution of hard phases

The size of the hard phases was expressed by their equivalent circular diameter (ECD) and by the height they protrude from the polished steel matrix. These size parameters were measured by AFM, see Table 10. Each value is an average from several measurements. The measurements were made on samples polished with 0.25 µm diamond paste as the final polishing stage.

Table 10. The size of hard phases in some tool steels measured by AFM

<table>
<thead>
<tr>
<th>Tool steel</th>
<th>Hard phase</th>
<th>ECD nm</th>
<th>Height nm</th>
</tr>
</thead>
<tbody>
<tr>
<td>Vancron 4</td>
<td>M(C,N)</td>
<td>1200</td>
<td>20</td>
</tr>
<tr>
<td>Vanadis 23</td>
<td>M₆C + MC</td>
<td>1900</td>
<td>6</td>
</tr>
<tr>
<td>Vanadis 6</td>
<td>MC</td>
<td>1800</td>
<td>6</td>
</tr>
<tr>
<td>Sverker 21</td>
<td>M₇C₃</td>
<td>3400</td>
<td>6</td>
</tr>
<tr>
<td>Ferrotitanit</td>
<td>TiC</td>
<td>5200</td>
<td>28</td>
</tr>
<tr>
<td>Weartec</td>
<td>MC</td>
<td>3400</td>
<td>12</td>
</tr>
<tr>
<td>Annealed</td>
<td>M(C,N)</td>
<td>1200</td>
<td>20</td>
</tr>
</tbody>
</table>
The carbonitride phase revealed in the experimental tool steel Vancron 4 has a smaller ECD size and protrude higher than the carbides in other steels, cp. Fig. 10a. A similar topography was found in the worn surface of the same steel in an industrial tool after being used for 5,7 million strokes, see Fig. 10b. The topography of industrial tools was not possible to measure with AFM for practical reasons.

![Figure 10](image)

Figure 10. Three dimensional presentation of the topography of the carbonitrides obtained by AFM in polished Vancron 4 (a), and by SEM on a used industrial tool (b).

It is seen that the majority of the carbonitrides in Vancron 1-3 are very small in size, see Fig. 11. The highest measured peak value was 1,25 µm for Vancron 3.

![Figure 11](image)

Figure 11. The size distribution of carbonitrides in Vancron 1-3.

When Vancron 4 was annealed at 1150 °C for 550 hours followed by conventional heat treatment to a hardness of 62 HRC, the ECD size of carbonitrides grew to 5-10 µm, see Fig. 12.
**Figure 12.** The carbonitrides in Vancron 4 after conventional heat treatment (a) and after annealing at 1150°C 550 hours (b).

**Hardness of hard phases**

The hardness and elasticity of the carbonitrides and carbides were measured with the nanoindentation technique and shown in Fig. 13. The hardness of the matrix was around 800 HV for all the tool steels.

**Figure 13.** Hardness and elasticity of carbonitrides and carbides.

**Thermal conductivity of hard phases**

The individual thermal conductivity of the hard phases could not be detected. They are literature values /47/. Instead, the combined conductivity from hard phases and matrix was evaluated by measuring the thermal conductivity from bulk samples of Vancron 4 and Weartec, see Table 11.
Table 11. *Thermal conductivity* \( W/(m \, ^{\circ}C) \) of \( V \), \( VN \), and \( VC \) and tool steels Vancron 4 and Weartec

<table>
<thead>
<tr>
<th></th>
<th>V</th>
<th>VN</th>
<th>VC</th>
<th>Vancron 4</th>
<th>Weartec</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>30.7</td>
<td>11.3</td>
<td>38.9</td>
<td>21</td>
<td>17</td>
</tr>
</tbody>
</table>

**Oxide layer**

The GDOES measurements made on Vancron 4, Sverker 21, Ferrotitanit, Vanadis 6 and Weartec showed that the surface of these materials is covered by an oxide layer of thickness 2-3 nm. The chemical analysis showed that the dominating elements of this layer are iron and oxygen. In Ferrotitanit also titanium is found in the surface oxide.

2.2.2 Properties of oxidized sheet materials

The surface of the deliberately oxidized sheets was imaged with SEI mode in SEM, see Fig. 14. The thickness of the oxide layers were < 0.5 \( \mu \m \). EDS in SEM was used for chemical analysis of the surface layer, and XRD to determine the crystallographic structure of the oxides, see Table 12.

![Figure 14. Surface topography of the oxidized sheets. ASTM S32101 (a), DP 600 (b), and ASTM 1 CP (c).](image)

Table 12. *Oxide types, and their chemical composition (wt%), and crystal structure*

<table>
<thead>
<tr>
<th>Sheet</th>
<th>Oxide</th>
<th>Composition wt%</th>
<th>Crystal structure</th>
</tr>
</thead>
<tbody>
<tr>
<td>ASTM S32101</td>
<td>Top layer ( \text{M}_3\text{O}_4 ) (Mn rich)</td>
<td>Fe 30/Mn 30/O 32</td>
<td>Cubic fcc</td>
</tr>
<tr>
<td>DP 600</td>
<td>( \text{Zn(CO}_3\text{)}_2(\text{OH})_6 + \text{ZnO} )</td>
<td>Zn 60-65/O 24-42/C 2-7</td>
<td>Monoclinic + hexagonal</td>
</tr>
<tr>
<td>ASTM 1 CP</td>
<td>( \text{Ti}_2\text{O} )</td>
<td>Ti 53-64/O 36-46</td>
<td>Primitive tetragonal</td>
</tr>
</tbody>
</table>

2.2.3 Adhesion at tool-sheet interface in dry contacts

* (Paper II, III, V)  

The findings from the experiments with the load scanner showed that the tool steel matrix displayed somewhat higher tendency for adhesion with the austenitic stainless steel than the MC, M(C,N) and M\( _6 \)C particles. The Cr-Fe
rich M7C3 carbide showed equal tendency for adhesion as the steel matrix. Also, the large TiC dispersoids in Ferrotitanit were very sensitive for adhesion. Large variations in the coefficient of friction were recorded for the tool steels.

The findings with the coatings from the load scanner tests demonstrated that VN shows very low tendency to adhere to austenitic stainless steel. On the TiN and VC coatings some transfer layers were formed, but far less than on tool steels. The coefficient of friction was generally lower for the coatings than for the tool steels. The vanadium carbonitrides displayed lower friction than carbides in tool steel in tapping mode tests with AFM, see Fig. 15. In the friction images of Fig. 14, the relative difference in contrast between the hard phase and the matrix shows qualitatively the difference in friction force. Dark color corresponds to low friction.

![Friction images](image_url)

*Figure 15. Friction image (left) and topography (right) of Vancron 4 (a), Sverker21 (b), Weartec (c), and Ferrotitanit (d) as obtained by AFM. The imaged areas are 30 x 30 µm².*

2.2.4 Adhesion at tool-sheet interface in lubricated contacts

*Industrial field tests (Paper III)*

The tool life in the deep drawing line was highly different for the different tool materials, see Fig. 16. Vancron 4 displayed excellent galling resistance compared to the other tool materials. The material was still free of galling after 15 million strokes, whereas Vanadis 6 and 23 dies were discarded from use because of galling only after a short time. The CVD coating was discarded from use due to early cracking and delamination.
The surface topography of Vancron 4 showed only small changes during its long life, see Fig. 17a. Typical for these changes is that tiny grooves were formed parallel to the sliding direction of the sheet. They were caused by abrasive action of inclusions present in AISI 304 and by wear debris. In Vanadis 6 microscopic galling was observed after 0.1 million strokes, see Fig. 17b. The test was terminated after 0.8 strokes because of heavy galling. Signs of mild abrasion were then seen in Vanadis 6.

Large differences were noticed for the galling resistance between the experimental tool steels alloyed with nitrogen and conventional tool steels in lubricated sliding tests against AISI 304. In the tests with a light mineral oil without EP additives, all the experimental tool steels displayed better galling resistance than the conventional tool steels, see Fig. 18. The scatter of the results for the experimental tool steels was large. Sometimes a galling resistance equally high as for the coated tools was achieved.
Figure 18. Galling resistance of the tool materials in slider-on-sheet test. Lubricant Coupex 046 EP (no EP additives).

Similar trends between the nitrogen alloyed tool steels and the conventional tool steels were observed for tests, where both tool material and lubricant were used as test variables, see Fig. 19. The lubricant with EP additives i.e. Draw 42K, gave excellent galling resistance for all the tool materials. The other lubricants with low environmental impact increased the tendency for galling. Here, the nitrogen alloyed tool steel and coated tool steel performed better than conventional tool steels.

Figure 19. Galling resistance of tool materials with different lubricants in slider-on-sheet tests.

The same tool materials (with the exception of TiN replacing TiC) and lubricants were tested in the flat-on-flat tester with similar result, see Fig. 20. Again, all tested materials behaved very well together with the chlorinated Draw 42K lubricant. It is also seen that both Vancron 4 and the TiN-coated steel displayed a very low friction throughout the whole test duration also for
the other three lubricants. In these oils the worst behavior is displayed by Sverker 21 for which the friction raises steeply after just a few strokes.

Figure 20. Friction versus number of test strokes of tool materials with different lubricants in the flat-on-flat test.

The results from the BUT experiments also indicated excellent galling resistance for the nitrogen alloyed tool steel compared to the conventional tool steel together with a vegetable based oil. This test also included the annealed experimental tool steel, which proved to be rather sensitive for galling, see Fig. 21.

Figure 21. Friction versus number of test strokes for three tool steels in the BUT test. Lubricant Draw 250 I.
2.2.5 Adhesion of oxidized sheet materials in dry and lubricated tests (Paper VI)

The influence of oxidation of the sheet materials was evaluated against the Vanadis 6 and TiAlN-coated steel. The oxidized duplex stainless steel (ASTM S32101) and titanium (ASTM 1 CP) displayed increased galling resistance compared to the same materials without the specific oxides in dry strip reduction and flat-on-flat tests. The oxidized titanium sheet displayed considerably better galling resistance in dry contacts than the pure titanium sheet in lubricated contacts (Rhenus SF 250A). The produced oxides on the electrogalvanized material displayed poor galling resistance. It was noticed that the specific surface oxides on duplex stainless steels increased their galling resistance in contacts lubricated with Rhenus SF 250A. The galling tests of the oxidized materials are summarized in Tables 13 and 14. However, the number of performed tests was limited.

Table 13. Results from strip reduction tests with TiAlN-coated tool. Yes=galling, no=no galling

<table>
<thead>
<tr>
<th>Dry contacts</th>
<th>Lubricated contacts</th>
</tr>
</thead>
<tbody>
<tr>
<td>Reduction %</td>
<td></td>
</tr>
<tr>
<td>S3201</td>
<td>S32101+M₃O₄</td>
</tr>
<tr>
<td>Zn</td>
<td>Zn(CO₃)₂(OH)₆ + ZnO</td>
</tr>
<tr>
<td>S32101</td>
<td>1 CP + Ti₂O</td>
</tr>
<tr>
<td>2</td>
<td>10/13/14</td>
</tr>
<tr>
<td>0/3/6</td>
<td>-</td>
</tr>
<tr>
<td>0/5/7/13</td>
<td>0/6/6,6</td>
</tr>
<tr>
<td>Galling</td>
<td>yes</td>
</tr>
<tr>
<td>yes</td>
<td>yes</td>
</tr>
<tr>
<td>no</td>
<td>no</td>
</tr>
</tbody>
</table>

Table 14. Results from the flat-on-flat test. Yes= galling, no= no galling

<table>
<thead>
<tr>
<th>Dry contacts</th>
<th>Lubricated contacts</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pressure MPa</td>
<td></td>
</tr>
<tr>
<td>TiAlN tool</td>
<td></td>
</tr>
<tr>
<td>S32101</td>
<td>S32101+M₃O₄</td>
</tr>
<tr>
<td>Zn</td>
<td>Zn(CO₃)₂(OH)₆ + ZnO</td>
</tr>
<tr>
<td>S32101</td>
<td>1 CP + Ti₂O</td>
</tr>
<tr>
<td>30</td>
<td>12</td>
</tr>
<tr>
<td>yes</td>
<td>yes</td>
</tr>
<tr>
<td>30</td>
<td>30</td>
</tr>
<tr>
<td>no</td>
<td>no</td>
</tr>
</tbody>
</table>

The surfaces of oxidized sheets were investigated in SEM after dry strip reduction test, see Fig. 22. It can be seen that the Mn-rich M₃O₄ displays some ability for shearing and Ti₂O displays good ability for shearing at the tribological interface. Zn(CO₃)₂(OH)₆ + ZnO is mostly flattened and did not display any shearing.
2.2.6. Temperature generation at tool-sheet interface (Not published)

Temperature generation at tool-sheet interface was simulated for a simplified contact situation where the contact was carried by all the hard phases protruding from the matrix, see Fig. 23-24 for the temperature rise in the sheet material. Small hard phases generate lower temperature rise in the sheet than the big hard phases. The simulated sliding lengths were very short, but still differences were observed between small and big hard phases.

Figure 22. SEM images of the surface of oxidized ASTM S32101 (a), Zn-coating (b), and ASTM 1 CP (c) after dry strip reduction tests.

Figure 23. Temperature rise in the sheet material in contact with hard phases of size 1 µm. The distance between the contact points is 2 µm, and the maximum temperature rise is 1°C.
2.3 Discussion

A general belief is that the material transfer involved in galling is a result of local lubricant failure. Lubricant failure in sliding contacts has been a subject of research for several decades. Correlation between the temperature and boundary layer removal, relation of lubricant desorption to local contact temperatures as well as formation of local contact temperatures have been considered at asperity level in this research. The surface roughness of the contacting materials, geometry of the asperities and their interaction has been similar as in engineering surfaces, and the surfaces applied for theoretical and empirical studies were relatively rough. The mean height of tool asperities was about 0.3 µm. /5/

Spikes and Cameron proved that failure in lubricated systems at low or moderate sliding speeds is controlled by the degree of coverage of lubricant molecules on the surface. Failure occurs below a critical coverage, when the surface is partly depleted of lubricating molecules. The lubricant film thickness and coverage is in turn controlled by the contact temperature, which is named as critical temperature $T_{cr}$. Thus, the formation of local (flash) temperatures above the critical temperature exposes the contacting materials to metal-to-metal contact. Similar findings have been reported by Blok. De Gee et al. showed that the existence of three wear regimes (no wear, mild wear, severe wear) in lubricated contacts could be related to the failure of the lubricant./48-51/

As soon as the local contact temperature is above $T_{cr}$, the soft work material starts to adhere as wedges on the tool asperities. A theoretically and empirically well tested strategy to avoid galling and exclude the occurrence of
wedge formation is to remove the wedge forming asperities by polishing the surface to a high finish. Another well tested strategy for avoiding galling is to use coatings that display low interfacial shear strength at the interface between the coating and sheet material. /5,7/

Theoretical considerations on lubricant failure of ideally smooth surfaces have shown that the most important parameters influencing the formation of high local temperatures are the contact length, the sliding speed and the geometry of the contacting bodies. The sliding length can be regarded as the time during which the contact is acting as a heat source. For instance, for low sliding speeds and short contact lengths, there is enough time to transfer the small generated amounts of heat by conduction. In that case, the thermal conductivity of the contacting materials will influence the maximum occurring temperatures. Diamond-like-carbon coatings possess a high thermal conductivity (around 450 compared to about 40 W/mK for steel) and it is successfully used to delay galling. They also generate a low interfacial shear resistance./5,52/

2.3.1 The critical contact temperature criterion

The hypothesis for explaining the good galling resistance for the nitrogen-alloyed steels is based on the critical local contact temperature at which the lubrication deteriorates.

The energy E generated in one local contact point in the interface between tool and sheet can be expressed as

$$E = \mu \cdot F \cdot s$$  \hspace{1cm} (1)

where $\mu$ is the local friction coefficient, $F$ is the contact load and $s$ the local sliding length. The local temperature rise $\Delta T$ in this point can qualitatively be expressed as

$$\Delta T = K \cdot E \cdot v \cdot t / a$$  \hspace{1cm} (2)

where $K$ is a constant, $v$ is the sliding velocity, $t$ is the duration of the contact and $a$ is the thermal conductivity. Consequently,

$$\Delta T = K \cdot \mu \cdot F \cdot v \cdot t / a$$  \hspace{1cm} (3)

In this work the criterion when galling starts to occur is the contact temperature that exceeds a critical temperature. It has been observed in this work that galling is initiated at a microscopic level. Therefore the temperature rise at the microstructure level is of interest. The input parameters that influence
the contact mechanism at the tool-sheet interface and the local temperatures were characterized systematically. Thus, the interest is on formation of local contact loads, and more specifically on the contact temperatures and the adhesion and the friction properties at these local contact points.

2.3.2 Adhesion and friction of tool steel phases

Dry sliding tests with the load scanner demonstrated that the adhesion tendency of the tool steel matrix to AISI 304 was generally somewhat higher than that of the hard phases of tool steels. The chemical composition of the tool steel matrix and austenite is very similar; primarily composed of Fe and Cr, c.p. Table 15. It is expected that during the sliding experiment the thin film oxide on AISI 304 was locally removed through plastic strain, and therefore a Fe-Cr (austenite) to FeO (tool steel oxide) contact was prevailing at the interface. Here, the chemical reactivity of fresh austenite to FeO and the O$_2$ molecules present in atmosphere as well as the surface energetic aspects were of importance for formation of adhesive bonding. Hanson et al. has studied the interface between austenitic stainless steel and TiN and showed that the interface is composed of Cr-rich oxides.\textsuperscript{53} It can be expected that the adhesive bond between the sheet and the tool steel matrix is composed of iron-chromium oxides. Further TEM EDS/EELS analysis is proposed to study the structure and chemical composition of oxides at the tool steel matrix/AISI 304 interface.

Table 15. The tool steel matrix – AISI 304 interface: elements and oxides at the interface

<table>
<thead>
<tr>
<th>Base material</th>
<th>Structure</th>
<th>Fe</th>
<th>Cr</th>
<th>C</th>
<th>Type</th>
<th>Thickness</th>
</tr>
</thead>
<tbody>
<tr>
<td>Matrix</td>
<td>Bcc, tetragonal</td>
<td>91-93</td>
<td>3-6</td>
<td>0.5-0.7</td>
<td>FeO</td>
<td>2-3 nm</td>
</tr>
<tr>
<td>AISI 304</td>
<td>fcc</td>
<td>80</td>
<td>18</td>
<td>0.08</td>
<td>CrO</td>
<td>&lt;1 nm</td>
</tr>
</tbody>
</table>

The same dry sliding tests demonstrated that the hard phases of tool steels displayed a lower adhesion tendency to AISI 304, but it was not possible to rank which hard phase showed the lowest tendency. However, it was noticed that the M$_7$C$_3$ carbides (about 50 wt% Cr, 35 wt% Fe) was prone to adhesion. Here, the chemical similarity of this phase to AISI 304 may have contributed to adhesion. The affinity of Cr to form an oxide is high in austenite and presumably also in carbides. It was also noticed that TiC dispersions were prone to adhesion, but here the most significant contribution probably came from the high vertical height of the TiC particles that caused a mechanical interlocking effect for the surfaces in contact.

Dry sliding tests with VN, VC and TiC coating against AISI 304 (Paper II) showed that the VN coating displayed no adhesion to stainless steel,
whereas the two other coatings were more sensitive for adhesion. The good sliding properties of VN at elevated temperatures have been explained by formation of a vanadium oxide that contains easily sheared crystallographic planes at the sliding contact. Vitos et al. have shown theoretically that VN gives a lower atomic friction against single crystals of iron than TiN.

The friction evaluation made with AFM demonstrated the action of van der Waals forces between tool steel phases and the tip material W2C. The weak van der Waals forces acting between materials can create bonds between neutral atoms and molecules. The AFM experiments showed that the vanadium rich carbonitrides in Vancron 4 had lower attraction to the tip than carbides in other investigated tool steels.

All the conducted experiments and evaluations indicate that the adhesion tendency and frictional sliding resistance of vanadium carbonitrides and vanadium nitride is lower than that of carbides in contact with stainless steel. The origins of the positive frictional properties of this phase and the VN coating are not completely understood.

2.3.3 Influence of tool steel microstructure on galling

The nitrogen alloyed steels displayed better galling resistance than conventional tool steels in industrial field tests and laboratory scale tests. For the experimental alloys Vancron 1, 2 and 3 a relatively large scatter was observed for their galling resistance. This was likely caused by variations in the microstructure and by a relatively high content of inclusions typical for laboratory scale manufactured alloys. Regardless of this, all the experimental tool steels outperformed the conventional ones throughout all testing. As the same high surface finish (Ra 0.05 μm) was applied for all tools throughout all testing, it is reasonable to assume that the microstructure of tool steels has a fundamental influence on the interaction mechanisms at the contact interface.

Different hard phases in tool steels have different thermal conductivity. The mononitrides of Group V transition metals (V,Nb) have lower thermal conductivity than carbides or pure V or Nb. Their thermal conductivity is related to the travel of phonons and electrons inside the material.

The microstructure of the tool steels is predominantly martensite, which varied between 67-87 vol % for the studied compositions. The carbon content, hardness, and the surface oxides were similar for all tool steels. Thus, we may conclude that the tribological behavior of the matrix is practically the same for all the tested tool steels.

A general characteristic of the tool steels that displayed inferior galling resistance is that they had a relatively low total content of hard phases, between 13.6-15.3 vol%. The hard phases in these steels were MC, M6C, and M7C3 carbides. For Sverker 21 manufactured through ingot metallurgy the
M$_2$C$_3$ carbides were large (ECD $\approx 3.4 \mu$m) and their distribution varied considerably, see Fig. 9 b. Their vertical height was about 6 nm. There was a tendency of these carbides to adhere with to AISI 304. In the PM tool steels Vanadis 23 and Vanadis 6 the carbides are smaller (ECD $\approx 1.8-1.9 \mu$m). The hardness of the hard phases in these steels is around 2000 HV. The accuracy of the hardness measurements can have been influenced by the surrounding matrix for the PM steels as the size of their hard phases was small.

The nitrogen alloyed tool steels and Weartec had a high content, 22.3-37.0 vol%, of hard phases. A better galling resistance was observed for the Vancron steels. The most striking difference between Weartec and Vancron steels was in the chemical composition of the hard phases, their horizontal and vertical size and in their distribution. The carbonitrides have a ECD size of 1-1.2 µm and their vertical height is around 20 nm. The carbides in Weartec have a ECD size of 3.4 µm and height is around 12 nm. Vanadiumcarbonitrides have lower hardness than vanadiumcarbides. The thermal conductivity of pure vanadiumnitride is lower than that for vanadiumcarbide. The carbonitrides displayed low friction.

A comparison between Vancron 4 and the material designated as Annealed indicates that a material with a large particle size is more sensitive for galling than a material with small hard particles.

It can be concluded that a favourable microstructure with respect to galling resistance comprises of a high volume fraction of hard phases in a form of small particles. There is also an indication that a relatively large vertical height of the hard phase contributes to good galling resistance in lubricated contacts. In addition, carbonitrides showed lower friction than carbides.

The coated materials displayed very good galling resistance. The coatings were composed of one phase that displayed low adhesion and low friction to stainless steels.

### 2.3.4 Influence of lubrication for galling

The galling sensitivity of any forming operation or test is closely connected to the properties of the used lubricant. It was noticed that the lubricants with high chlorine content gave very good galling resistance in the sliding contact tests for all the tested tool materials. These lubricants form a persistent chemical reaction layer onto the tool under activation of frictional heating. However, above a critical contact temperature the layer loses its lubricating ability.

In the industrial field tests, a highly chlorinated lubricant was used and the results showed differences in tool life for different materials. Obviously, the laboratory scale sliding tests were too short to induce severe galling with the chlorinated lubricants. In the industrial trial with Vanadis 6 microscopic galling was observed after 118 000 pressings, but severe galling was
achieved only after 800,000 pressings. Thus, the time from galling initiation to severe galling can be relatively long. In this case, the tool was in active use for nearly three months before galling became a problem. It is obvious that laboratory scale tests cannot be performed over such long periods.

In the sliding contact tests with the lubricants with low environmental impact, the best galling resistance was observed for the nitrogen alloyed tool steels and with coated tool steels, while the tool steels with low contents of alloy carbides were sensitive to galling.

It is concluded that the ranking between the tool materials became similar for the experiments conducted in industry and laboratory and that the sliding lengths (time) needed for galling initiation were closely connected to the properties of the used lubricant.

2.3.5 Contact mechanisms

In boundary lubrication, the sliding contact is supported by both adsorbed lubricant molecules and by metallic contacts. As the hard phases protrude from the steel matrix, it is reasonable to assume that the local contact pressure is higher on the hard phases, while the steel matrix is covered with the lubricant.

The number of contact points influences the load carried by each particle. A theoretical consideration of the load per contact point was made for different ECD sizes of hard phases assuming that the entire contact load is carried by the hard phase particles, see Fig. 25, where the nominal contact pressure is set to 500 MPa. The magnitude of the contact force per particle increases with increasing size of the contact point. For instance, the decrease in contact force per particle when the particle size is reduced from 2 to 1 µm is 4 times.
The density of contact points and magnitude of local contact force per contact point vs. ECD for a nominal contact pressure of 500 MPa, assuming that all load is carried by the hard phase particles.

The smaller the hard phase particles are in size the lower is the contact load per particle. Due to the lower contact loads and shorter sliding lengths, the heat generation at these points will be lower. With small evenly distributed particles, the generated frictional heat will be more evenly distributed across the tool surface, which facilitates the heat dissipation. Lowering the surface temperature decreases the risk of reaching the critical oil temperature. Thus, a small size of the hard phase is an important property for good galling resistance. This is part of the explanation why the Vancron steels showed a good galling resistance.

The performed FEA gave convergent results for the heat generation on contact points of different sizes. Contact points of larger size became warmer than the small ones due to more intense frictional heating.

The thermal conductivity of the carbonitrides is not known, but one can expect this property to be close to that of vanadiumnitrides, which display low thermal conductivity. However, apparently not low enough to have a negative effect on the local heat formation. Instead, the limited amount of generated heat at these points, their size and distribution are more decisive than the low thermal conductivity.

However, the material designated as Annealed had a carbonitride size of 5 µm and volume fraction of 22 %, and was sensitive for galling. Here, the size of the contact points with carbonitrides was relatively large, which resulted in formation of large contact points, each of which carried a relatively large normal force, and the local temperature thus became high.

The tool steel Weartec had a high volume fraction of carbides of relatively large size. Due to the large size of carbides, the number of contact points became relatively low and thus the local normal force and temperature rise
became unfavorable compared to that generated by the small carbonitrides in the Vancron steels. Also, the frictional properties of the hard phase are in favor for the carbonitrides.

The ECD size of the carbides in Vanadis 23 and Vanadis 6, around 1,8-1,9 µm, and vertical height around 6 nm and the content of hard phases was low. Obviously a larger part of the contact load was carried by the matrix. This resulted in formation of large contact areas of high reactivity. The matrix effect of these materials became more dominant and they displayed a higher sensitivity to galling.

If a lubricant containing EP additives is used for lubrication, a protective layer will be formed on the matrix and on any work material pick-ups. A rapid growth of the galling pick-up can be avoided with use of lubricants with EP additives. The abrasive particles inevitably present in forming can partly remove the smallest pick-ups. With other types of additives the shear resistance at the area of galling pick-ups is increased due topographic effects and as the additives are less effective than the EP additives, the growth of galling pick-ups becomes more rapid.

2.3.6 Effect of surface oxides on sheet materials for galling initiation

In both dry and lubricated tests the galling resistance of Mn-rich M₃O₄ on duplex stainless steel and Ti₂O on titanium was better than that of the same sheets without thick oxides. The reactivity of these oxidized sheets to the tool materials was obviously low, as adhesion to the tool was not a problem. The oxide layer on duplex stainless steel was Cr-rich at the interface to the bulk steel, whereas the surface of the oxide was Mn-rich due to the lower affinity of Mn to form oxides. It is not known if the Mn-rich oxide displays lower tendency for adhesion to the tool than the Cr-rich oxide. It has already been demonstrated that Cr in austenite reacts rapidly with oxygen and bonds strongly to the tool steel matrix. The reactivity of Ti₂O was considerably lower than that of Ti, and dry forming of oxidized Ti sheet was possible without galling. As both Cr and Ti react strongly with oxygen, it can be speculated the chemical reactions at the contact interface controls the formation of adhesion bond and initiation for galling when lubricant failure occurs.

The high galling resistance of oxidized sheets is also connected to the ability for the oxides to shear at the contact interface. The Ti₂O on titanium displayed better ability for shearing than the Mn-rich M₃O₄ oxide. The M₃O₄ oxide has an fcc structure and the sliding planes available there have obviously contributed to a relatively low shear resistance. The Ti₂O has rutile crystal structure, also named Magnéli phase, and obviously the sliding planes in this primitive tetragonal structure have very low shear resistance as
reported by several investigators. The surface structure of rutile Ti$_2$O and Mn-rich M$_3$O$_4$ is expected to be very complex and their interaction with molecules of O$_2$ and H$_2$O in ambient environment even more complicated, but it is not known if these effects are important for the shearing behavior at the tribological interfaces. A TEM study on a cross-section of the contact area should reveal which sliding planes were activated for shearing. It can be concluded that the surface oxides give a lubricious effect in sliding contacts under conditions when they display a low adhesion tendency to the tool material and display an ability to be sheared at the contact interface.

2.4 Conclusions and future perspectives

2.4.1 Conclusions

The following conclusions can be drawn from the sheet forming tests. All tool specimens were polished to a very high initial surface finish.

**General conclusions**

- By powder technology it proved possible to make nitrogen alloyed tool steels of desired chemical composition and structure.
- The laboratory tests proved to simulate the galling characteristics of all tested steels.
- The galling resistance of tool steels in lubricated contacts is related to the microstructure of the steel.
- The hard phase particles protrude from the matrix surface even for highly polished tool steels.
- Nitrogen alloying of tool steels has a very positive effect for the galling resistance in lubricated sliding contacts.
- Galling initiation can be avoided in dry forming of austenitic stainless steels and titanium alloys by applying thick surface oxides on the work material. Oxides are less prone to adhere compared to the base material. The oxides that have easily sheared sliding planes available at the contact interface act as lubricants.

**Detailed conclusions**

- The relatively large test matrix of tool steel alloys and lubricants show that nitrogen alloyed tool steels give an improved galling resistance compared to conventional tool steels even for low viscosity oils.
- Small size combined with an even distribution of the hard phase particles is the most important feature giving a good galling resistance for
tool steels. These features influence the formation of local contact temperatures through two mechanisms:

1. Heat generation: It is assumed that the hard phase particles represent contact points. For a given volume fraction, the smaller they are, the lower will the local contact forces be, and the heat generated by friction at each particle will be low.


- The frictional properties of hard phases are important for the frictional heating and adhesion, but only if the criteria of small size and even distribution of the hard phases are fulfilled.
- The origins of low friction properties of the vanadiumcarbonitrides are not known. Formation of easily sheared sliding planes with and without tribo-oxidation could be studied by TEM.
- A TEM study could be performed on Ti$_2$O surfaces to examine which sliding planes are activated for shearing.

**Industrial relevance**

- Nitrogen alloyed tool steels can be used as a cost effective alternative to coated tool steels.
- The galling resistance of nitrogen alloyed tool steels can further be improved by decreasing the size of the carbonitrides below 1 µm (ECD).
- The investigations have contributed to the commercial introduction of a nitrogen alloyed tool steel named Vancron 40. The tool steel is manufactured by powder metallurgy by Uddeholms AB.

**2.4.2 Future perspectives**

- The galling resistance of tool steels can be further improved by increasing the N and V contents above the tested values. It would result in formation of increased number of carbonitrides of even smaller size.
- For less demanding forming applications, the N and V contents could be decreased from the tested values, and conventional tool materials could be replaced with the proposed alloys for improved tool life.
- During the transition period towards more environmentally friendly lubrication, the use of N alloyed tool steels provides an alternative to the coated tools.
- The use of lubricious oxide layers on the sheet materials could be an alternative way to improve galling resistance in specific applications.
such as processes at elevated temperatures or at very demanding cold forming operations.

- The concept of using nitrogen alloying to produce small hard phases could be applied to other materials used in lubricated sliding contacts.
- VN or lubricious oxides could be explored as new coatings for tool materials.
Metallformning innefattar ett stort antal viktiga operationer inom verkstadsindustrin och används för att tillverka mer eller mindre komplicerade produkter. Inom formning skiljer man mellan skärande bearbetning (svarvning, borrning, gängning, sågning, mm.), gjutning (kokillgjutning, pressgjutning, stränggjutning etc.) och plastisk formning (valsning, bockning, träddragning, plåtpression, osv.). Plastisk formning sker antingen vid förhöjd temperatur (varmformning) eller vid rumstemperatur (kallformning). Gemensamt för de plastiska formningsoperationerna är att man går från ämne till färdiga produkt enbart genom formändring. Ingen materialavverkning sker. Vid plåtformning pressas ämnet, som vanligtvis är en plan plåtskiva, mellan två verktygshalvor av stål. Verktygen har graverats för att ge ämnet sin slutliga form. Det finns många exempel på produkter som skapats genom plåtformning, t ex. inom vitvarubranchez, diskbänkar, karosserier och stänkskärmar till bilar, detaljer i värmeväxlare för havsvatten etc. Om slutprodukten är en tredimensionell struktur, som i exemplet ovan krävs att plåten samtidigt både sträcks och bockas för att undvika att få veck i slutprodukten.

Under formningen glider plåten mot de båda verktygshalvornas ytor under högt tryck, och det finns risk att plåtmaterialet kletar fast till verktyget. Lokala påkletningar på verktygen gör att man fortsättningsvis får skador i form av intryck eller repor i slutprodukterna. Detta fenomen, som benämns galling, är ett alltför välkänt fenomen inom verktygsbranchen.


Fördelningen av och storleken på de hårdas partiklarna påverkar de lokala temperatur–stegringar som uppstår vid kontakterna mellan verktyg och plåt. Detta i sin tur påverkar smörjmedlets förmåga att skydda ytorna vid glidande kontakter. Vid ökande temperaturer tappar smörjmedlet viskositet och blir tunnare. Över en viss kritisk temperatur blir smörjmedelsfilmen så tunn att det lokalt kan förkomma osmord kontakt mellan verktyg och plåt. I sådana områden är risken stor för galling.

Friktionsegenskaperna hos de hårdas partiklarna varierar mellan olika verktygstål, och de kvävehaltiga karbonitriderna har befunnits ha bättre egenskapen att ge låg friktion än de traditionella karbiderna. Orsaken till detta ligger framför allt i att de är så små, vilket i sin tur kan härledas till deras höga värmebeständighet. Denna egenskap gör att de tillväxer långsammare än karbider under tillverkningen av verktygsstålet.

Det är också möjligt att motverka galling genom att förse plåtmaterialet med ett skikt av ytoxider som kan fungera som ett fast smörjmedel.

En slutsats är att kvävelegerade verktygstål kan användas som alternativ till belagda material vid det pågående teknologiskiftet mot miljövänliga smörjmedel. Gallingmotståndet hos verktygsstålet kan skräddarsyas genom val av kvävehalt och tillsatser av nitridbildande legeringsämnen.

De resultat som presenteras i avhandlingen är direkt tillämpliga på plåtformningsverktyg. Bland annat genom utnyttjande resultat från denna studie har ett stort svenskt stålforetag redan utvecklat ett kvävelegerat verktygsstål som säljs under namnet Vancron 40. Resultaten kan också tillämpas för att ta fram nya legeringar för nya tillämpningar inom många andra områden där två metallytor glider mot varandra under höga tryck.
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References

33. S.M. Hsu, Boundary lubrication of materials, MRS Bulletin (USA), Vol 16, no 10, pp. 54-58, 1991
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