Fundamental friction phenomena and applied studies on tribological surfaces

Viktoria Westlund

Licentiate Thesis

Department of Engineering Sciences
Uppsala University, September 2017

Abstract

This thesis is based on two different projects, one more focused on applied research and one on more basic research. The first project examines the potential of nitriding as an alternative to case hardening in tribologically loaded components while the second project involves micro scale studies of the relations between roughness, transfer and friction between metals.

The first project consists of an evaluation of the tribological properties of nitrided steels. The aim is to increase the understanding of the wear and friction behavior of different nitrided steels in relation to the choice of steel grade, microstructure, thickness and composition of the compound layer, among other parameters. This study is a part of a bigger project called SurfNit, which primary objective is to optimize the nitriding process in order to increase the use of nitrided steels for applications like high stress components. Today case hardening is the standard heat treatment for these applications but nitriding is both more environmentally friendly and less time consuming. In the present study, the steel grade with the highest content of nitride-forming elements and highest hardness showed the best wear resistance, regardless of the composition of the compound layer. Further, steels of a given grade but with different phase compositions of the compound layer showed differences in their wear behaviour. It was also shown that nitrided steels with ε-phase in the compound layer acted more brittle than those containing ϒ'.

The goal of the more basic project is to increase the understanding of the mechanism behind sliding friction. The main focus has been the relation between friction and material transfer. A better understanding can be of help when developing new tribological materials, for example wear resistant components that can operate without lubrication. It could also enable specific recommendations for surface finishes to avoid material transfer and be of help when trying to make more realistic tribological models. Scratch tests have been performed on samples with different surface roughness and different surface composition. It was shown that nano scale topography had a bigger impact on both material transfer and friction compared to micro scale topography. Experiments both in air and in situ in a SEM have been performed to determine the effect of presence of air on the friction and material transfer. The experiments in air resulted in more material transfer and higher friction than those performed in vacuum. Both lubricated and unlubricated contacts have been studied in order to see how surfaces otherwise separated by boundary lubrication will be affected if the lubrication fails.

Till mamma och pappa,
bättre stöd får man leta efter

List of Papers

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

I Westlund, V., Troell, E., Haglund, S., Jacobson, S. Nitrocarburizing and plasma nitriding - effects of steel grade, compound layer and post treatments on tribological properties

II Westlund, V., Heinrichs, J., Olsson, M., Jacobson, S. (2016) Investigation of material transfer in sliding friction- topography or surface chemistry? *Tribology International*, vol. 100: p. 213-223

III Westlund, V., Heinrichs, J., Jacobson, S. On the role of material transfer in friction between metals – initial phenomena and effects of roughness and boundary lubrication in sliding between aluminium and tool steels

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Abbreviations

NC	Nitrocarburizing
PN	Plasma nitriding
ASPN	Active-screen plasma nitriding
C	Case hardening
DLC	Diamond like carbon
PVD	Physical vapor deposition
FEG-SEM	Field emission gun scanning electron microscopy
EDS	Energy dispersive spectroscopy
AFM	Atomic force microscopy
FIB	Focused ion beam
EBSD	Electron backscatter detection

1 General Introduction

The work presented in this thesis is based on two different projects. The first project examines the potential of nitriding as an alternative to case hardening in tribologically loaded components. Today case hardening is the standard heat treatment for this application but nitriding is both more environmentally friendly and less time consuming. This project is part of a bigger project called SurfNit which primary objective is to optimize the nitriding process in order to increase the use of nitrided steels in for applications like high stress components. SurfNit is a collaboration between the companies in Värmebehandlingscentrum (Bodycote, Scania, Volvo Group Trucks, Volvo CE, Parker Hannifin, Atlas Copco, Sarlin Furnaces, Stresstech, Ovako and Uddeholms) and the research institutes Swerea IVF and Swerea KIMAB as well as Uppsala University and Oerlikon Baltzers Sandvik Coating.

The second project involves micro scale studies of the relations between roughness, transfer and friction between metals. Although there are numerous economic benefits in being able to control the friction, the mechanisms of sliding friction are still not fully understood. The goal of the project is therefore to increase the general understanding of the mechanisms behind sliding friction. New knowledge can be of use when choosing and developing tribological materials, surface treatments and coatings. It could also enable very specific recommendations on what type of surface finish is required for specific applications to avoid wear or material transfer. A more extensive knowledge would also help make tribological models more realistic.

Although the first project is more applied research while the second one is more basic research, the materials used in both projects include different cold working tool steels and nitrided steels as well as post treatments like DLC coating; all chosen for their potential in real tribological applications such as cold metal forming or high stress automotive components.

2 Introduction to Part I: The potential of nitriding as an alternative to case hardening in tribologically loaded components

Today case hardening is the most common surface hardening method when producing high stress components such as gears and other drive train details. Case hardened steel consists of a hard, carbon enriched, martensitic surface layer on top of a softer but also tougher bulk material. This combination of hardness and toughness gives high fatigue strength and wear resistance.

Although case hardening works well in many high stress applications, nitriding processes may offer potential benefits. Nitriding processes include surface hardening methods like nitro carburizing and plasma nitriding. Nitrided steel consists of a hard and thin ceramic surface layer called the compound layer and a thick diffusion zone underneath that, with an increasing hardness towards the surface. The core of the steel is still soft, ensuring the toughness of the material. Advantages of using nitriding instead of case hardening is lower process temperatures, shorter process times, less shape distortion and overall nitriding is more environmentally friendly. But to be able to replace case hardening, the wear properties, as well as the fatigue strength have to be on the same level or better for the nitrided steel. [1]

Several parameters can affect the tribological properties of nitrided steels; the choice of steel grade, composition and thickness of the compound layer etc. The composition and thickness of the compound layer can be controlled by changing process parameters such as the time, temperature and atmosphere in the furnace, but it is complex. [1]

The research on the topic has shown scattered results [2–7]. To be able to optimize the nitriding process it is necessary to understand the wear behavior of the compound and diffusion layer.

2.1 Aim

The aim of the project was to make it possible to use nitrided steel to a higher extant for high stress components by optimizing the nitriding processes. The focus of this work was to evaluate the tribological properties and increase the understanding of the wear behavior in relation to the microstructure and composition of different nitrided steels.

3 Surface hardening methods

3.1 Case hardening

Case hardening is a thermochemical surface hardening method where low carbon steel is hardened by enriching the surface with carbon at a high temperature (900-950°C), making a martensitic phase transformation possible when quenching the steel (by rapid cooling in oil). The furnace atmosphere consists mainly of nitrogen, carbon monoxide and hydrogen. The nitrogen acts like a carrier gas while the carbon monoxide and hydrogen reacts on the surface and form free carbon atoms and water. The carbon can diffuse into the surface while the water returns to the atmosphere.

To avoid a brittle surface, the steel is heated again after quenching to relief surface stresses; this step is called annealing. The process results in a hard surface layer and a tough core, giving the steel a high strength and good wear resistance. The case-hardened depth of the steel varies depending on the carbon content and the curability of the steel as well as the dimension of the goods and the cooling time, but it is in the millimeter range. [8][9][10]

3.2 Nitriding processes

3.2.1 Nitrocarburizing

Nitrogen and carbon is added to the steel surface via nitrogen, ammonia and carbon dioxide in the furnace atmosphere. The ammonia decomposes at the steel surface into free nitrogen atoms and hydrogen gas (***Eq. 1***). The carbon dioxide reacts with hydrogen and forms water and carbon monoxide (***Eq. 2***) that in turn forms free carbon atoms and water at the surface, when reacting with hydrogen (***Eq. 3***). Nitrogen acts like a carrier gas. The temperature is around 570°C, which is below the austenite phase transformation temperature. Therefore less shape and size distortion is obtained than during case hardening. Less distortion reduces the need for post operations like grinding and polishing.

$$NH_3 \leftrightarrow N + \frac{3}{2}H_2 \quad \text{Eq. 1}$$
$$CO_2 + H_2 \rightarrow CO + H_2O \quad \text{Eq. 2}$$
$$CO + H_2 \rightarrow C + H_2O \quad \text{Eq. 3}$$

The hardness and hardening depth varies with the alloy content of the steel. The ability of nitrogen to diffuse into the steel controls the hardened depth, which is why low alloyed steels get higher hardened depths than high alloyed steels hardened at the same temperature and time.

When a steel is nitro carburized a thin surface layer (0-30 µm) called the compound layer, see ***Fig. 1***, is created and underneath that a thick (0.05-0.8 mm) diffusion zone. The compound layer consists of iron nitrides and iron carbides, $Fe_2(N, C)_{1-x}$ and Fe_4N_{1-z} called ε-phase and ϒ'-phase, respectively. The nitride formation starts with nucleation of ϒ'-phase at the steel surface. After a while ε-phase is also formed on the surface, often resulting in a compound zone with ε-phase closest to the surface and ϒ'-phase further in. By adjusting the nitrogen content in the furnace atmosphere it is possible to affect the ϒ'/ ε-phase ratio. High carbon content favors the formation of ε-phase. The carbon content also affects the composition of the compound layer, making the process control complex. In the diffusion zone some of the nitrogen and carbon is solved in the ferrite while some forms nitrides and carbides with various alloying elements. [8][9][10]

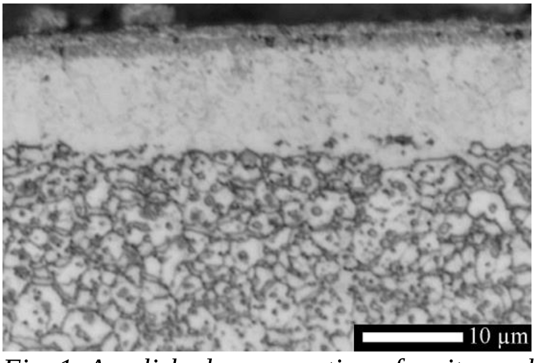

Fig. 1. A polished cross section of a nitrocarburized Orvar steel sample depicted with a light-optic microscope. The light grey area is the compound layer. It appears that the area closest to the surface is porous.

3.2.2 Plasma nitriding

Plasma nitriding processes occur in an ionized gas. The atmosphere consists mainly of nitrogen, hydrogen and a carbon containing medium like methane and the pressure is very low. The plasma is ignited by introducing an electric potential between the goods, cathode, and the walls of the oven, anode. The ionized nitrogen is then accelerated towards the goods. When the ionized nitrogen hits the surface, atoms are knocked out. Iron atoms that are knocked out form iron nitrides in the plasma and then recondense at the surface of the goods, supplying nitrogen to the surface.

The process can be performed in a wide temperature range 400-600°C. Usually a lower temperature than in nitrocarburizing is used, ensuring even less shape distortion. Plasma nitriding consumes 10 times less gas than gaseous nitriding processes do. Just as for the nitrocarburizing process a thin compound zone is created (0-20 µm) and a diffusion zone below that consisting of the same type of nitrides and carbides.

Often a cleaning step is introduced before the nitriding, called sputtering. During this step argon or hydrogen is introduced in the atmosphere of the furnace, which is then ionized and accelerated towards the goods surface to get rid of the outmost atom layers.
[8][9][10]

3.2.3 Active screen plasma nitriding

Active screen is a type of plasma nitriding where a cage is placed around the goods. This cage acts like a cathode instead of the goods itself. The cage works like a shield protecting the goods from getting bombarded by ionized nitrogen with high kinetic energy. It also radiates heat resulting in a more even temperature distribution, making it possible to nitride goods with different shapes and sizes at the same time. These changes give a more even nitriding. [8]

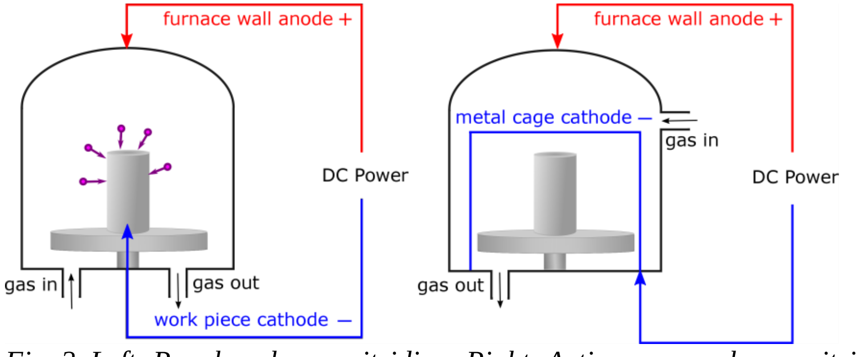

Fig. 2. Left: Regular plasma nitriding; Right: Active-screen plasma nitriding

3.2.4 Post treatments

By post oxidizing steel after nitriding, the corrosion resistance is increased. The process is quick and performed at around 450-550°C. An iron oxide layer (Fe_3O_4) with a thickness of around 1-3 μm is obtained on top of the compound layer.

DLC coating with PVD results in a high surface hardness and a high wear resistance as well as low friction during sliding contact. To get a good adhesion, the surface has to be very clean before the coating process. It is of high importance that the substrate is hard enough to support the coating to prevent it from flaking off; therefore the steels used in this work were first plasma nitrided and then given a chromium nitride layer before they were DLC coated. [8]

3.3 Process control

During the nitrocarburizing process much water is produced as well as the byproduct ammonium carbonate (a salt obtained during the cooling process when residual ammonia, water and carbon dioxide react). Because of the high humidity and because the ammonium carbonate can clog the analysis equipment it is hard to measure and control the gas mixture. Today the composition of the atmosphere is measured with a hydrogen sensor, measuring only the hydrogen content and then estimating the amount of residual ammonia [11]. If carbon monoxide is used instead of carbon dioxide less water and less ammonium carbonate will be produced, making it possible to use an IR gas sensor[12] resulting in a better control of the atmosphere during the nitriding process.

By controlling the gas mixture, process time, temperature and cooling rate it is possible to optimize the properties of the nitrided steel to fit the application. The composition of the atmosphere has a strong effect on the compound zone. A high nitrogen activity results in a thick and porous compound zone, while a low nitrogen activity gives a thin and compact one. Having a high carbon activity is favorable for the formation of ε-phase. [8][13][14]

The cooling rate can be controlled by changing the coolant. By using oil, a high cooling rate is obtained, while using a gas results in a low cooling rate. The hardened depth of nitrided steel depends on the diffusion rate of nitrogen. The higher the temperature and the longer the process time, the more nitrogen is able to diffuse into the steel.

4 Materials

4.1 Steel grades

The steel grades used in this work were chosen to cover a range of different alloy compositions, see ***Table 1***. Some of the alloy elements are strong nitride formers, this includes silicon, chromium, molybdenum and vanadium. Adding manganese and nickel helps the process by increasing the curability of the steal. Nitriding of low alloyed steels results in relatively low hardness compared to high alloyed steels since less nitrides are able to form. High alloyed steels on the other hand lower the nitrogen diffusion rate, which decreases the hardness depth. The hardness of the diffusion zone is highly dependent on the amount of alloying elements. Precipitation hardening is the main hardening mechanism for high alloyed steel while for steels with low amount of alloying elements, solution hardening is dominating. [8]

Table 1. Chemical composition, wt-%, of the alloying elements in the different steel grades used in this work.

Steel \ Alloying elements	C	Si	Mn	S	Cr	Mo	Ni	V
42CrMo4	0.38-0.45	0-0.40	0.60-0.90	0-0.035	0.90-1.20	0.15-0.30		
Orvar	0.39	1.0	0.4		5.2	1.4		0.9
Ovako277	0.14-0.17	0-0.030	1.20-1.40	0.023	2.10-2.30	0.45-0.55	0.45-0.55	
34CrNiMo6	0.32-0.39	0.10-0.40	0.50-0.80	0-0.035	1.30-1.37	0.15-0.30	1.30-1.70	
20NiCrMo2-2	0.18-0.23	0.20-0.35	0.65-0.90	0.008-0.040	0.40-0.70	0.15-0.25	0.40-0.70	

4.2 Test matrix

The different combinations of heat treated and post treated steel samples used in this study can be seen in ***Table 2***.

Table 2. Material matrix, heat treatment and post treatment for each steel grade tested.

Steel grade	Heat treatment	Post treatment
42CrMo4	Nitrocarburizing (NC)	
	Nitrocarburizing	Post oxidization
	Plasma nitriding (PN)	DLC coating
	Active-screen plasma nitriding	
Orvar	Nitrocarburizing	
	Plasma nitriding	
	Plasma nitriding	DLC coating
Ovako277	Nitrocarburizing	
	Plasma nitriding	
34CrNiMo6	Nitrocarburizing	Different surface finishes
20NiCrMo2-2	Case hardening(C)	

5 Methods

5.1 Seizure test

Seizure is an extreme form of failure, defined as the stopping of a relative motion between two surfaces in contact when the friction force becomes higher than the driving force. The high friction force is an effect of severe damage to the sliding surfaces, which could be caused by a breakdown of the lubrication film. This rarely happens in practice and the word seizure is loosely used when talking about a high sudden increase in friction, a roughening of the original surfaces, clearly visible adhesive wear and sometimes localized surface welding. [17]

In this work a seizure test was chosen as a first screening test, facilitating an overview of the different sample performances. For this particular test, seizure is defined as when the coefficient of friction reaches a value of ≥ 0.3. The tests were performed in a turning machine with the sample of interest pressed against a rotating cylinder with a thin pre-deposited oil film. The normal load was increased until seizure was reached. The test has the advantage of being really fast, making it possible to test several samples during a short period of time. The disadvantage is that since the seizure limit is rather arbitrarily selected and the wear features can vary, it is difficult to do extensive interpretation of the wear results.

5.2 Reciprocal test

As a complement to the seizure test, a sliding contact test that was interrupted after a pre-defined number of cycles to evaluate the wear loss and wear mechanisms was used. The test setup consists of two crossed cylinders, the top cylinder is stationary and the bottom one reciprocating. The top cylinder received a smaller and deeper wear scar than the bottom cylinder and was therefore easier to study when comparing the amount of wear. Since the effect of different compositions of the compound layer on wear and friction was of interest, load and number of cycles was chosen to keep the wear within the compound layer. For every steel grade chosen, two samples were tested, one with more ϒ’-phase in the compound layer and one with more ε-phase.

5.3 Scratch test

The conventional use of scratch test is to test coating adhesion, as was first developed by Heavens [18]. The normal load of the indenter is linearly increased from a minimum to a maximum. The critical load is defined as the load when the coating starts to flake off. The critical load can be detected by an acoustic sensor. [10]

In this work a scratch test was used to evaluate the material’s resistance to brittle fracture. In the scratch test the materials are subjected to severe plastic deformation and those not having visible cracks were interpreted as having a high resistance to brittle fracture.

5.4 Sample characterization

5.4.1 Thickness and composition of the compound layer

The thickness and composition of the compound layer was investigated by electron backscatter diffraction (EBSD) as detected in a FEG-SEM. EBSD is a microstructural crystallographic technique that detects backscattered electrons forming diffraction patterns. These diffraction patterns contain information about the crystal structure of the sample. The sample has to be very smooth and defect free to produce clear diffraction patterns. [15]

Below (***Fig. 3)*** is a crystal structure map created with EBSD. Similar crystal structures are given similar colors. The black areas consist of undefined data points.

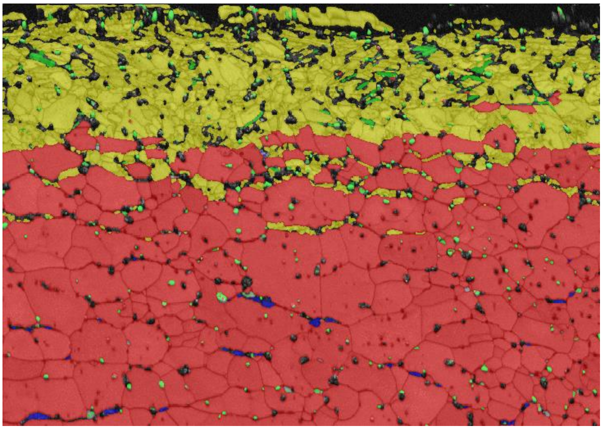

Fig. 3. A crystal structure map of a polished cross section of a nitrocarburized Orvar steel sample. The ε-phase in the compound layer is shown in yellow and the ϒ'-phase is shown in green. The blue areas represent cementite and the red area the steel substrate.

5.4.2 Surface roughness and mechanical properties

An interference microscope was used to measure the surface roughness of the samples. Interference microscopes uses the wave properties of light to measure height variations by comparing the path of two light beams, one reflected on a reference surface and one reflected on the sample. The reflections interfere, resulting in either constructive interference (the waves are in phase) or destructive interference (the waves are out of phase). If the surface has a sharp slope, the light will not be reflected from this area and no height information will be obtained. [16]

The hardness was measured by Vickers indentation on the surface with a load of 1 kg. It was also measured 50 µm below the surface on a cross section, with a load of 0.1 kg. Finally, a hardness profile was created using nano indentation on a cross section from the surface down to a depth of 400 µm, using loads in the mN range.

5.5 Analysis of the worn surfaces

The amount of worn material was calculated as the volume of the wear scars. The geometries of the wear scars were measured with an interference microscope and also studied in a high resolution FEG-SEM and chemically analyzed using EDS mapping.

6 Introduction to Part II: Micro scale studies of the relations between roughness, transfer and friction between metals

The starting point of research on the mechanisms of sliding friction was with Bowden and Tabor in the 1950's [19]. They suggested an adhesion model explaining the mechanism behind sliding friction. In the model, adhering asperities are formed when a normal load is applied and the friction force is the result of energy loss when the asperity junctions break and plastically deform as the surfaces are set into motion, see ***Fig. 4***. Although much research has been performed in the field since then, their model is still highly topical.

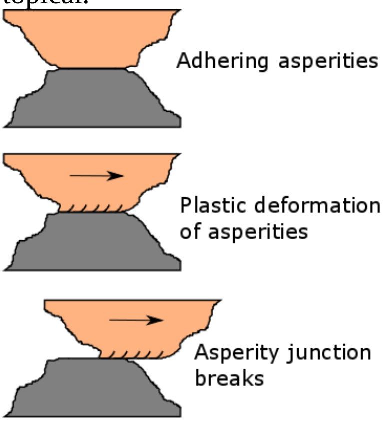

Fig. 4. A schematic of the adhesion model.

It has been shown in previous research [20][21][22] that the topography has a strong impact on the material transfer and that material transfer in its turn has a strong impact on the friction, but the micro- and nano-scale interaction is still left to uncover. A deeper understanding of the interaction between friction and material transfer is of great interest for industries involved in cutting and forming operations, who must avoid galling, i.e. material transfer to the tools.

One line of experimental work today in the field of nano-scale friction is the use of Atomic force Microscopy (AFM). AFM makes it possible to measure frictional force at the atomic level. The technique uses a very small tip that is put very close to the surface examined and then the interactions between the tip and the surface are registered by a high sensitive force sensor as the tip scans the surface. A problem with these types of experiments is the weak correlation to real tribological contacts. No plastic deformation is obtained, and any material transfer or wear occurs on an extremely small scale. [23]

The friction during unlubricated sliding contact varies fast and seemingly randomly. Parameters that can strongly alter locally over the surface and thereby may be part of the explanation to the friction fluctuations, include the roughness and chemical composition. By using an in situ technique in the SEM it is possible to correlate the friction variation registered with the events observed. The technique uses a small tip, representing the work material, that is spring loaded against a tool steel flat. The load is high enough to plastically deform the tip. The tool steel flat is then set into motion making short tracks over the surface. The test set up is shown in ***Fig. 5.***

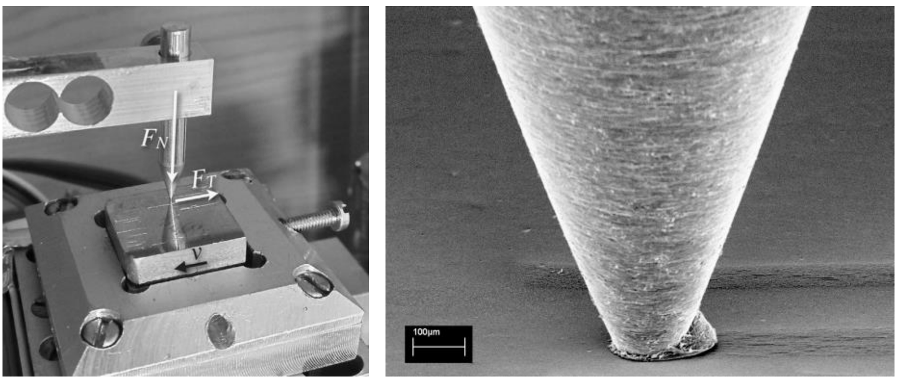

Fig. 5. To the left; the in situ test equipment. To the right; aluminium tip in sliding contact with a tool steel flat.

6.1 Aim

The aim of this project was to increase the understanding of the mechanisms behind sliding friction. The focus was on the interaction between friction and material transfer, including the influence from both the initial surface roughness and the roughness due to transfer, as well as surface composition. Both lubricated and unlubricated contacts were studied to get a more comprehensive picture of when a boundary lubricant film works, as well as of the worst case scenario, when the lubrication fails and a metal to metal contact is obtained.

7 Materials

7.1 Work material

To represent the work material in real tribological contacts, different tool steels were chosen. Dievar, consisting of only a martensitic matrix and Vancron 40 consisting of a martensitic matrix with two different hard phases, carbides M_6C (5 vol %) and carbonitrides M(C, N) (19 vol %) [24] were used in the experiments to study the effect of adding hard phases on the friction and material transfer. To be able to separate the role of surface chemistry from that of surface topography on friction and material transfer, sliding on both uncoated and DLC coated Vancron 40 was studied. The DLC coating was chosen because of its excellent properties such as high wear resistance, often low friction in sliding contact. It is further quite chemically inert and has been proven able to prevent or strongly limit transfer of otherwise adhesive metals [25][26][27]. The coating follows the topography of the substrate, preserving it, which facilitates comparisons with an uncoated substrate. To be able to study the effect of the topography on the friction and material transfer, tool steel samples were polished in various steps to obtain different final surface topographies.

7.2 Tool material

Aluminium was chosen to represent the work material in a real tribological application such as cold metal forming. Aluminium is known to cause much material transfer and galling, which makes it a hard metal to form. The aluminium alloy 6082 with silicon and magnesium as the main alloys was formed from sheets into the tips used in the experiments. The tips were manufactured using a lathe and were then ground and polished to form a sharp tip with a rounding diameter of ~100 μm.

8 Methods

8.1 Sliding tests in air and in situ in SEM

The very initial mechanisms of sliding contact, including deformation, material transfer and friction, were studied in a sliding test in situ in a Scanning Electron Microscopy (SEM). The test procedure includes a needle-like aluminium tip put into contact with a tool steel surface with a normal load of 3 N. This load causes the tip to plastically deform, resulting in a flat end with a radius of around 100-200 μm. The tool steel is then put into motion (2mm/min) and the friction force is measured. Multiple passages over the same track can be performed with the tip, making it possible to study the gradual buildup of material transfer and the friction changes that comes with that. This test set up enables events to be observed while they are happening, which makes it possible to relate specific events to friction variations.

The method correlates to typical tribological contacts in for example the forming industry, showing the same type of plastic deformation and material transfer.

Similar sliding tests were performed both in air and in situ in a SEM. This allows studying the effect of the presence of air, particularly the oxygen, on the friction and material transfer.

8.2 Dry and lubricated contact

By performing sliding tests also outside the SEM, it was possible to compare lubricated and unlubricated contacts. The effect of different surface topography on friction and material transfer when having boundary lubrication was investigated. It was also tested if a surface with a considerable amount of material transfer and a high sliding friction could be “saved” in the terms of lowering the friction and stopping more material from transferring as well as smoothening the roughness of the material transfer, by adding a boundary lubrication film. This is of great interest since even components intended to have a separating lubricating film can accidentally come in contact, resulting in material transfer.

In this project a thin, well-defined lubricant film is applied by adding a mixture of 6 wt % base oil (PA08) in hexane and then waiting for the hexane to evaporate.

8.3 Sample preparation

8.3.1 Mechanical grinding and polishing

The tool materials were mechanically ground and polished in several different steps to get a range of different surface topographies. To get surfaces as smooth as possible, a dispersion of small diamond particles on a soft polishing cloth was used. When polishing the tool material Vancron 40, consisting of hard phases embedded in a softer matrix, the final polishing step was done using a dispersion of diamond particles. Since the hard phases have a higher wear resistance than the matrix the polishing resulted in surfaces with more or less protruding hard phases.

8.3.2 Ultra clean surfaces

When the surfaces are very smooth, having only nanometer -scale roughness, the sliding friction is very sensitive to contamination layers, absorbed gas, oxide layers, etc. This is because the surface asperities are so small they only affect the outermost layer, which is the contamination layer (unless the surfaces are ultra clean) [23]. A contamination layer is often easily sheared and acts like a lubricant, decreasing the friction and material transfer during sliding contact. Due to this it is of utmost important that the storage of the samples is well-controlled. If a box is used to store the samples, it has to be cleaned very thoroughly; even the slightest amount of oil or lubricant residuals can diffuse to the sample surface, contaminating it. Storing the samples in a plastic bag leads to contamination in the form of softener from the plastic adhering to the surfaces. Residuals of acetone and ethanol used to clean the specimens also affect the tribological behavior of the surfaces.

8.4 Sample characterization

Before the experiments were performed, the surface topographies of the tool steels were measured and depicted using an interference microscope as well as an atomic force microscope, AFM. The AFM technique has a higher lateral resolution, valuable for the really smooth surfaces. The arithmetic average roughness value, Ra, was used to compare the topography of the different surfaces. This value describes the mean value of the measured vertical

deviation from the average level of the surface profile [28]. How to calculate the Ra value is illustrated in ***Fig. 6***.
Unfortunately the Ra value does not give a complete picture of the surface roughness; two surfaces can have the same Ra value but have different characters. Therefore it is of importance to also have the depicted surface in mind and not only consider Ra values.

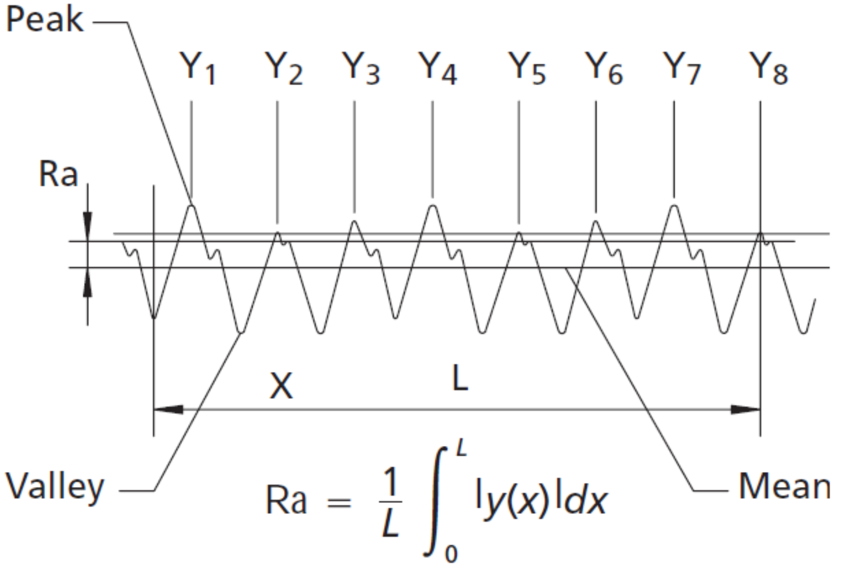

Fig. 6. The arithmetic average roughness value, Ra.

8.5 Surface analysis

To be able to see really thin material transfer films, a low acceleration voltage (3 kV) was applied in a FEG-SEM. When needed to enhance the topographic view of the surfaces, they were tilted to a high degree angle (70°). Two of the aluminium tips were also cross sectioned using a focused ion beam (FIB) instrument to get a better picture on how the tip was interacting with the tool steel surface.

9 Summary of results

9.1 Paper I: Nitrocarburizing and plasma nitriding – effects of steel grade, compound layer and post treatments on tribological properties

The aim of this work was to investigate the tribological properties of nitrided steels with different steel grades, post treatments, surface finishes, composition and thickness of the compound layer. Focus was put on determining the effect of each parameter on the friction and wear by using three different test set ups. A seizure test was used as a first screening; a reciprocating sliding wear test was used to evaluate the wear resistant and a scratch test was used to determine the resistance to brittle fracture.

The plasma nitrided steel samples had a thinner, more homogenous and less porous compound layer than the nitrocarburized steels. The compound layer of the plasma nitrided steels consisted mostly of ϒ’-phase, while the nitro carburized steels had compound layers with mostly ε-phase.

It was found that the properties of the compound layer had an effect. Nitrided samples with compound layers containing mostly ϒ’-phase showed a less brittle behavior than the samples with compound layers consisting of mostly ε-phase. The steel grade with the highest content of nitride-forming elements and therefore the highest hardness showed the least wear in the reciprocating sliding wear test, regardless of the composition of the compound layer.

It was also shown that adding a post treatment in the form of a DLC coating or post oxidization resulted in higher seizure loads; higher wear resistance and lower friction.

The effect of the surface finish on the tribological properties was also investigated. It was shown that the surface finish of the surface with the most shallow wear depth, whether due to higher wear resistance or to that the wear is spread over a larger area, had a major effect on the friction.

9.2 Paper II: Investigation of material transfer in sliding friction – topography or surface chemistry?

The aim of this study was to increase the understanding of the mechanism of sliding friction on the micro- and nano-scales. The main focus was to investigate the role of the surface roughness and surface chemistry on the friction and material transfer, respectively.

To compare two different surface chemistries, one DLC coated and one uncoated tool steel were prepared. Different surface topographies were obtained by varying the last polishing steep. Since it is impossible to make a perfectly smooth surface and thereby get rid of the contribution from mechanical interlocking to the material transfer, attempts were made to make surfaces with the same topography but with different surface chemistries to be able to link any change in material transfer to the difference in chemical bonding. Although such surfaces with roughly the same Ra values but with different surface chemistries were made, the real surface profiles looked quite different, making it impossible to make a fair comparison.

It turned out that the uncoated tool steel was never "smooth enough" to result in a low friction and transfer free surface. For the DLC coated tool steel, the smoothest surface, with an Ra value of 2 nm, did provide a low friction and transfer free surface. Whether this was the result of a weaker chemical bond or because the DLC coated surface was smoother than the uncoated tool steel could not be determined.

The as-deposited DLC coatings had a nano topography that caused material transfer and a gradual increase in friction. It turned out that this nano topography affected the friction and material transfer more than did the micro topography, even though the amplitude was only 10 % of the micro topography.

It was also shown that even if the friction levels were similar, the material transfer could vary.

9.3 Paper III: On the role of material transfer in friction between metals – initial phenomena and effects of roughness and boundary lubrication in sliding between aluminium and tool steels

In this study a series of micro-scale sliding tests were performed with the aim to investigate the first sliding contact between tool steels and a softer work material and the corresponding initial material transfer. Tests were performed both in air and in situ in a SEM to try and determine if the presence of air, particularly the oxygen, affects the friction and initial material transfer. Performing sliding tests in air also made it possible to study lubricated contacts, which is not feasible in the SEM.

The experimental set-up consisted of a tool steel flat in sliding contact with a work material tip. Two different tool steel grades were studied, a martensitic matrix steel and a powder metallurgy (PM) steel with carbide and carbonitride hard phases. Aluminium tips were used to represent a work material.

When performing the same tests both in air and in the SEM, different results were obtained. For the smooth matrix tool steel the tests resulted in high friction (~0.65) in air and quite low friction (~0.13), although increasing after the first four passages, in the SEM. After the tests in air, the aluminium tip was covered by a thick oxide layer with embedded steel fragments. The tool steel was scratched and a lot of aluminium was transferred to it. When performing the same test in the SEM, the aluminium tip had no noticeable oxide layer and no transferred steel fragments and the tool steel showed very little transferred aluminum.

Unlike the results for the matrix tool steel, when performing the same sliding test on the PM steel in air no steel was transferred to the aluminium tip and the tool steel was not scratched by the tip.

When adding a boundary lubrication film to the tool steel, low friction was obtained for the smooth steel sample but for the rough sample almost twice as high friction was obtained. Even when adding the lubricant to the steel flats after they were tested in dry conditions, where the original smooth surfaces had become severely roughened by transfer, the friction level was drastically reduced.

As the results from the study show, the relation between friction and transfer in sliding contact depends on the initial roughness of the tool steel, the composition, presence of oxygen and lubricants.

Outlook

An interesting way to continue the research on the mechanisms of sliding friction would be to prepare and test specially designed surfaces with less technical applicability to be able to answer fundamental research questions like:

- What is the contribution from surface chemistry to the friction?
- How does the crystallographic orientation of a material affect the friction?

To be able to optimize the nitriding processes, more information about the tribological mechanisms and properties of nitrided steels is needed. A way to achieve that would be to perform studies with fewer parameters varied. It would be of interest to study nitrided steels with for example the same thickness of the compound layer but with different compositions, to isolate the effect of the composition on the wear and friction of the material.

Acknowledgements

Jag vill börja med att tacka min huvudhandledare Staffan Jacobson för att ha gett mig möjligheten att jobba med detta projekt. Tack för alla idéer, all expertishjälp och inte minst all hjälp vid artikelskrivningen. Jag vill också tacka Jannica Heinrichs som fungerat som en inofficiell handledare kring det ena projektet och som varit till stor hjälp både vid artikelskrivning och vid det mer praktiska laborationsarbetet. Jag vill också nämna Mikael Olsson som bistått med prover och som också är medförfattare i en av artiklarna. Eva Troell som varit projektledare för SurfNit och alla projektmedlemmarna. En person som förtjänar ett extra stort tack är Jan-Åke Gustafsson som backat upp med teknisk support varje gång min försöksutrustning vägrat fungera, vilket tro mig har varit ofta!

På det mer personliga planet vill jag tacka fikagänget Frida, Lisa, Elin och Charlotte som funnits där både de dagar då allt har gått bra men också de dagar när allt skitit sig. Tack för allt mentalt stöd och trevliga stunder!
Jag vill tacka mamma och pappa, mina alldeles egna cheerleaders. Oavsett vad jag gör är ni där och stöttar, ända sedan den allra första streckteckningen jag ritade, som jag misstänkter inte var så mycket att hurra för, fram till nu med denna avhandling.
Jag vill tacka min blivande man Adrian som har funnits där och hejat på under doktorandtiden och som förgyllt min vardag.

Slutligen vill jag tacka alla på tribologiavdelningen, det är så mycket roligare att gå till jobbet när man har så trevliga kollegor!

References

[1] Public report NitroVBC; *Minskade formförändringar och bränsleförbrukning genom utveckling och förbättring av nitrerprocesser för värmebehandling*, (2013)

[2] C. Binder, T. Bendo, G. Hammes, A. N. Klein and J. D. B de Mello, *Effect of nature of nitride phases on sliding wear of plasma nitrided sintered iron,* Wear 332–333, (2015), 995–1005

[3] C. Binder, R. Binder, A. N. Klein and J. D. B. de Mello, *Effect of nature of nitride phases on microabrasion of plasma nitrided sintered iron*, Tribology - Materials, Surfaces and Interfaces 4, (2010), 191–196

[4] S. P. Brühl, A. Cabo, T. Walter and G. Prieto , *Tribological behaviour of nitrided and nitrocarburized carbon steel used to produce engine parts,* Industrial Lubrication and Technology 68, (2016), 125–133

[5] F. T. Hoffmann and P. Mayr, *Nitriding and nitrocarburizing,* ASM handbook 18 , (1992), 877–883

[6] S. Karaoğlu, *Structural characterization and wear behavior of plasma-nitrided AISI 5140 low-alloy steel*, Materials Characerization 49, (2002) 349–357

[7] A. Molinari, G. Straffelini, M. Pellizzari and M. Pirovano, *Wear Behaviour of Diffusion and Compound Layers in Nitrided Steels*, Surface Engineering 14, (1998)

[8] T. Holm, P. Olsson and E. Troell, *Stål och värmebehandling- en handbok,* (2010)

[9] Bodycote värmebehandlingsguide, Downloaded from www.bodycote.com, 2017-09-19

[10] P. Møller and L. Nielsen, *Advanced Surface Technology Vol. 2,* (2013), chapter 22

[11] H-J. Spies, H. Le Thien and H. B. Biermann, *Controlled nitriding,* Metal science and heat treatment 46, (2004), 7–11

[12] T. Dinh, I. Choi, Y. Son and J. Kim, *A review on non-dispersive infrared gas sensors: Improvement of sensor detection limit and interference correction*, Sensors and Actuators, B: Chemical 231, (2016) 529–538

[13] D. Panfil, M. Kulka, P. Wach, J. Michalski and D. Przestacki, *Nanomechanical properties of iron nitrides produced on 42CrMo4 steel by controlled gas nitriding and laser heat treatment,* Journal of

Alloys and Compounds 706, (2017), 63–75
[14] J. Michalski, J. Tacikowski, P. Wach, E. Lunarska and H. Baum, *Formation of single-phase layer of gamma'-nitride in controlled gas nitriding,* Metal Science and Heat Treatment 47, (2005) 35–38
[15] Oxford instruments, http://ebsd.com/, (downloaded 2017-09-19)
[16] P. Hariharan, *Basics of interferometry 2nd edition,* (2007)
[17] A. Houghton, R. Lewis, U. Olofsson and J. Sundh, *Characterising and reducing seizure wear of inconel and incoloy superalloys in a sliding contact*, Wear 271, (2011) 1671–1680
[18] O. S. Heavens, *Some factors influencing the adhesion of films produced by vacuum evaporation,* Le Journal de Physique et le Radium 11, (1950) 355–360
[19] F. P. Bowden and D. Tabor, *The friction and Lubrication of Solids,*American Journal of Physics 19, (1951), 428-429
[20] B. Podgornik, S. Hogmark and O. Sandberg, *Influence of surface roughness and coating type on the galling properties of coated forming tool steel*, Surface and Coatings Technology 184, (2004), 338–348
[21] B. Podgornik and S. Hogmark, *Surface modification to improve friction and galling properties of forming tools,* Journal of Materials Processing Technology 174, (2006), 334–341
[22] P. Menezes, Kishore, S. Kailas and M. Lovell, *The role of surface texture on friction and transfer layer formation during repeated sliding of Al Mg against steel,* Wear 271, (2017), 1785–1793
[23] H. J. Kim and D. E. Kim, *Nano-scale friction: A review,* International Journal of Precision Engineering and Manufacturing 10, (2009), 141–151
[24] J. Heinrichs, M. Olsson and S. Jacobson, *New understanding of the initiation of material transfer and transfer layer build-up in metal forming—In situ studies in the SEM*, Wear 292–293, (2012), 61–73
[25] J. Heinrichs, M. Olsson and S. Jacobson, *Mechanisms of material transfer studied in situ in the SEM:* Wear 292–293, (2012), 49–60
[26] J. Heinrichs, M. Olsson, I. Z. Jenei and S. Jacobson, *Transfer of titanium in sliding contacts—New discoveries and insights revealed by in situ studies in the SEM,* Wear 315, (2014), 87–94
[27] M. Olsson and U. Bexell, *Friction characteristics and material transfer tendency in metal powder compaction,* Wear 271, (2011) 1903–1908
[28] S. Jacobson and S. Hogmark, *Tribologi,* (2011)