Nanotribological characterization of advanced tool steels

Nanotribologisk karakterisering av avancerade verktygsstål

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Abstract

Tribological problems of tool–work piece interaction is a key aspect influencing product quality, process performance and tool lifetime. For example, in sheet metal forming operations, sliding contact may cause adhesive wear of sheet materials with build up of worn material on the tool surface. This tribological problem often resulted in loss of tolerance and product quality and is called galling.

It was demonstrated that tendency to adhesive wear depends on the steel grade, which means high importance of chemical- and phase-constitution of the tool steel. It was suggested that adhesion to the matrix is critical due to metal to metal contact while carbide phase prevent adhesion of counterbody materials. Nevertheless, in macroscale tests it is difficult or even impossible to separate contribution of each phase into the wear mechanism.

In the present work, selected steels are to be investigated at nanoscale by means of AFM facilities. Main attention will be paid on adhesion and frictional properties of steel matrix and primary phase. As expected and reported in several articles, carbides, carbonitrides and nitrides may behave differently, contributing into the final performance differently. Additional attention will be paid on phase size and distribution, meaning they are also important parameters influencing tribological behaviour.
## Table of contents

Aims of research: ....................................................................................................................... 2  
Research activities: .................................................................................................................. 2  
Expectations: ............................................................................................................................ 2  

**Literature survey** ................................................................................................................. 2  
**Tool steels** .......................................................................................................................... 2  
Water Hardened Tool Steels (W-Steels) ............................................................................ 3  
Low-Alloy Special-Purpose Tool Steels (L-Steels) ........................................................... 3  
Shock-Resisting Tool Steels (S-Steels) ............................................................................. 3  
Oil-Hardened Cold-Worked Tool Steels (O-Steels) ......................................................... 4  
Air-Hardened Cold-Worked Tool Steels (A-Steels) .......................................................... 4  
High-Carbon High-Chromium Cold-Worked Tool Steels (D-Steels) ............................ 4  
Hot-Worked Tool Steels (H-Steels) ................................................................................... 5  
High Speed Tool Steels (T- and M-Steels) ...................................................................... 5  
Mold Steels (P-Steels) ........................................................................................................ 5  

**Selected tool steels** ............................................................................................................. 6  
Vancron 40 ........................................................................................................................... 7  
Vanadis 6 ............................................................................................................................... 9  
Vanadis 4 Extra .................................................................................................................. 11  
Sverker 21 ........................................................................................................................... 13  
Caldie ..................................................................................................................................... 13  

Comparison of selected tool steels ....................................................................................... 15  

**Atomic Force Microscopy** ................................................................................................ 15  

**Materials and methods** ....................................................................................................... 22  
Project procedure, practical tasks ......................................................................................... 22  
Stage 1: Preparation of specimens ....................................................................................... 22  
Stage 2: SOFS tribotestings ................................................................................................. 23  
Stage 3: Nanoscale characterization of damage and wear mechanisms ............................ 23  

**Project Procedure, Analysing** .......................................................................................... 24  
AFM – Atomic Force Microscope ....................................................................................... 24  
OP - Optical Profilometer .................................................................................................. 25  
SEM – Scanning Electron Microscope ............................................................................... 26
Aims of research:
The aims are (i) to investigate microstructural features of the selected tool steels, (ii) to measure adhesion tendency and friction at nanoscale, (iii) predict tribological performance, (friction coefficient, volume wear), of each tool steel in relation to the surface roughness, surface morphology and phase constitution and (iv) verify prediction with macrotests.

Research activities:
This research includes several stages;

- Thorough literature research.
- Calibration of AFM-tip and running pilot tests.
- Characterization of polished surfaces of selected tool steels by means of AFM, OP, SEM, characterize microstructure and amount of phases per volume unit.
- Running AFM friction and adhesion tests for selected phase constituents.
- Running wear tests with current control of friction coefficient in milli-Newton load reciprocating sliding.

Expectations:
To investigate influence of steel microstructure and tribological behavior of phases (matrix and carbide phases) on macro behavior of selected tool steels. To demonstrate influence of chemical and phase constitutions on tribological behavior. To conclude influence of microstructure on tribological performance.

Literature survey

Tool steels
Tool steels are usually high-carbon steels that obtain high hardness by quench and tempering heat treatment. Because of the combination of high strength, hardness, and toughness, tool steel applications are cutting tools in machining operations, dies for die casting, forming dies and other applications. All these different types of steel grades have been organized into groups. The main groups are Water-Hardened Tool Steels (W), Low-Alloy Special-Purpose Tool Steels (L), Shock-Resisting Tool Steels (S), Oil-Hardened Cold-Worked Tool Steels (O), Air-Hardened Cold-Worked Tool Steels (A), High-Carbon High-
Chromium Cold-Worked Tool Steels (D), Hot-Worked Tool Steels (H), Tungsten High Speed Tool Steels (T), Molybdenum High Speed Tool Steels (M) and Mold Steels (P). [1]

**Water Hardened Tool Steels (W-Steels)**

These types of steel have the lowest amount of alloying elements of all tool steels and are basically carbon steels. Therefore, the hardenability is low, and water quenching is required to form martensite. Even though water quenching is used, only the surface of a tool steel may harden. In these steels, martensite of high hardness will be formed because of the high carbon content. Due to the low-alloy content only iron carbides are produced by heat treatment and, therefore, lower wear resistance compared to more highly alloyed steels. Some applications of water hardened tool steels are cold header dies, shear blades, blanking dies, reamers, threading dies, taps, twist drills, lathe tools, coining dies, woodworking tools and cutlery.

**Low-Alloy Special-Purpose Tool Steels (L-Steels)**

These types of steel are very similar to water hardened tool steels besides that they contain larger amount of alloy content. This quantity of higher alloy content increases wear resistance as well as hardenability compared to water hardened tool steels. Low-Alloyed special-purpose tool steels can in regard to the higher hardenability be oil quenched which minimize dimensional changes during hardening. But parts with more simple shapes or heavy sections (like rolling-mill rolls) can be water or brine quenched. Some applications are tools like precision gages, cold heading dies, swaging dies, rock drills, shears, woodworking tools, punches and dies, drills, broaches and cutlery. Other could be non-tool uses as bearings, bearing races and small- to medium-size rolls.

**Shock-Resisting Tool Steels (S-Steels)**

This type of steel has been developed to produce good combinations of high strength, high hardness, and high toughness or impact fracture resistance. The major difference with this steel type, other than commonly used alloying elements such as manganese, chromium and molybdenum, is the major alloying element silicon. Silicon offers, in some tempered conditions, a microstructure with reduced sensitivity to fracture and also provides tempering resistance. In some grades, Tungsten is added in significant amounts. The carbon content (around 0.5%) in S-steels makes it possible to produce high-strength tempered martensitic microstructure without incorporation of coarse carbides that would lower toughness. S-steels are developed mainly for spring applications where good fatigue resistance are required. The
combination of high hardness and toughness also makes it applicable to chisel, punch and die applications.

**Oil-Hardened Cold-Worked Tool Steels (O-Steels)**

These types of steel gain its high hardness and wear resistance from high carbon and modest alloy content. The carbon makes it possible to form martensite, whereas the alloying elements provide sufficient hardenability to make hardening by use of oil quenching. The O-Steels are restricted to cold-work applications due to that the alloying elements are insufficient to provide carbides necessary for cutting at high speeds or hot-working applications. Due to the high carbon content austenitizing for hardening can be made at relatively low intercritical temperature where austenite and carbides coexist. Fine grain size is obtained due to the undissolved carbides but the austenite is still sufficient to provide martensite of high hardness. Oil quenching provides a relative freedom from cracking of intricate sections.

**Air-Hardened Cold-Worked Tool Steels (A-Steels)**

These steels are high carbon content steels with moderately high alloy content. High alloy content is needed to achieve air-hardening but it also provides distribution of large alloy carbide particles superimposed on the microstructures developed by heat treatment processing. The alloying elements have much higher hardness relative to martensite and cementite and therefore contribute to higher wear resistance. Regardless it's not sufficient for high speed machining or hot-work applications, there are other steel grades with even more alloy content which are far more suitable, which results that it is used mainly in cold-work applications.

**High-Carbon High-Chromium Cold-Worked Tool Steels (D-Steels)**

These are the most highly alloyed cold-work steels. Chromium is the major alloying element with a concentration up to 13 %, but manganese, molybdenum, vanadium, nickel, tungsten and cobalt may be added as well. These types of steels are deep hardened, except those which contain molybdenum that is oil quenched. They are hardenable by air-cooling from austenitizing temperatures and therefore have very low susceptibility to distortion and cracking during hardening. This steel type was at first developed for applications like hot-work trying to replace high-speed steels used for cutting tools but was found to have insufficient hot hardness and had a tendency to be too brittle for machining, however it
produced large amount of high-hardness carbides which are excellent for cold-work applications.

**Hot-Worked Tool Steels (H-Steels)**

These types of steel are made for hot-work applications. They all have in common the capacity to resist softening during long or repeated exposures to high temperatures such as hot-work or die cast applications. These types can be divided into three different kinds of classes; chromium hot-work steels (Cr, Si, Mo, Va), tungsten hot-work steels and molybdenum hot-work steels. Multiple alloying elements are also added to the tungsten and molybdenum classes and performances are generally somewhat better than the chromium steels. All hot-work tool- and die- steels should have these general characteristics: resistance to deformation at working temperatures, resistance to shock, resistance to high-temperature wear, resistance to heat treatment distortion, resistance to heat checking and good machinability. Selection of a hot-work tool steel depends on matching the manufacturing and performance requirements, selection is largely affected by the temperatures developed in dies, load aspects or the manner of cooling the die.

**High Speed Tool Steels (T- and M-Steels)**

These steels have in common to maintain high hardness at elevated temperatures. The name “high speed” inherits from cutting tools that generate considerably heat during high speed machining of steels and other materials. These steels can even resist softening at so called red hot machining, sometimes referred to as red hardness. They posses a number of unique alloying and processing features to reach high performance. Some of these features are sufficient alloy and carbon content to provide excess alloy carbides in heat-treated tools, hardening at temperatures close to or at their melting points, hardening with fine austenitic grain sizes, deep hardening by cooling in still air and prominent secondary hardening during tempering. They can be either tungsten or molybdenum based but in performance aspects they behave almost the same. Molybdenum based high speed steels are more widely used because of their lower cost. This difference in cost inherit from that the weight of molybdenum is one half of tungsten, therefore less molybdenum is required to provide equivalent performance.

**Mold Steels (P-Steels)**

These types of steels are used in plastic molding processes and some die cast applications. These steels differ a bit from the other types of tool steels, a list of essential characteristics
required for a mold steels are hubbability, machinability, polishability, wear resistance, high surface hardness, high core strength, toughness, minimum dimensional change on hardening, resistance to corrosion and resistance to hardness loss on tempering.

**Selected tool steels**

The different types of tool steels examined in this project are listed below, all steels are provided by Uddeholm Tooling AB.

- **Vancron 40 I** - 1150 C / 20h, 60 HRC
- **Vancron 40 II** - 1120 C / 30 min + 560 C 2x2h, 60-62 HRC
- **Vancron 40 III** - 1150 C / 10 min + deep cool + 520 C 3x1h, 66 HRC
- **Caldie** - 1120 C / 30 min + 525 C/2x2h, 60 HRC
- **Vanadis 6** - 1120 C / 30 min + 525 C/2x2h, 60 HRC
- **Vanadis 4 Extra** - 1120 C / 30 min + 525 C/2x2h, 60 HRC
- **Sverker 21** - 1120 C / 30 min + 500 C/2x2h, 60 HRC

Counterbody material used in wear test was a PVD-coated Vancron 40.
Vancron 40

This is a cold work tool steel with excellent galling/adhesive wear resistance. It is a nitrided powder tool steel in other words a surface coating is integrated into the finished tooling material. By adding a surface coating into the material results in a tool surface with very low friction that reduces galling or stick of soft work materials. Main area of use for Vancron 40 is in cold work applications, blanking and forming, cold extrusion, deep drawing or powder pressing where adhesive wear or galling is the major failure mechanism. Vancron 40 is characterized by:

- Very high adhesive wear resistance
- Very high galling resistance
- Good chipping and cracking resistance
- High compressive strength
- Good through hardening properties
- Good dimensional stability in hardening
- Very good resistance to tempering back
- Good WEDM properties

The hardenability for Vancron 40 ensures good through hardening properties at quenching in salt bath or gas quenching in vacuum furnace. It can be heat treated to give a wide range of hardness.

- Pre-heating in two stages: 600–650°C (1110–1200°F) and 850–900°C (1560–1650°F).
- Holding time: 30 minutes (10 minutes at 1100°C (2010°F)).

To achieve a hardness between 58-65 HRC the austenitizing temperature is varied in the range 950–1100°C (1740–2010°F). The recommended austenitizing temperature is 1020°C (1865°F) with 30 minutes holding time followed by quenching and tempering at 560°C (1040°F)/ 3 x 1 h resulting in a hardness of 60–62 HRC. In order to avoid a too low working hardness it is recommended to austenitize at a higher hardening temperature than normal and if the hardness will be too high temper down the hardness to the right hardness level.

<table>
<thead>
<tr>
<th></th>
<th>C</th>
<th>N</th>
<th>Si</th>
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<th>Cr</th>
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<td>4.5</td>
<td>3.2</td>
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Fig 1: Hardening of Vancron 40 tool steel.

Fig 2: CCT-Graph (Continuous Cooling) of Vancron 40 tool steel. Ausenitizing temperature 1050°C (1920°F). Holding time 30 minutes.
Fig 3: TTT-Graph (Isothermal Transformation) of Vancron 40 tool steel. Ausenitizing temperature 1050°C (1920°F). Holding time 30 minutes.

Vanadis 6

This is a powder metallurgical cold work tool steel. High wear resistance is often associated with low toughness but Vanadis 6 has very high wear resistance as well as good toughness. It is most suitable for long run tooling of work materials where the main failure mechanisms are mixed (abrasive–adhesive) or abrasive wear and/or chipping/cracking and/or plastic deformation. Some of the applications could be: blanking and fine blanking of harder work materials, forming operations where a high compressive strength is essential, powder compacting substrate steel for surface coating, plastics moulds and tooling subjected to abrasive wear conditions or knives. Vanadis 6 is characterized by:

- Very high abrasive-adhesive wear resistance
- High compressive strength
- Good toughness
- Very good dimensional stability at heat treatment and in service
- Very good through-hardening properties
- Good resistance to tempering back
- High cleanliness.

<table>
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<th>Mn</th>
<th>Cr</th>
<th>Mo</th>
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<td>0.4</td>
<td>6.8</td>
<td>1.5</td>
<td>5.4</td>
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</table>
Fig 4: CCT-Graph (Continuous Cooling) of Vanadis 6 tool steel. Aussenitizing temperature 1050°C (1920°F). Holding time 30 minutes.

Fig 5: TTT-Graph (Isothermal Transformation) of Vanadis 6 tool steel. Aussenitizing temperature 1050°C (1920°F). Holding time 30 minutes.
Vanadis 4 Extra

This is a powder metallurgical cold work tool steel with good combination of wear resistance and ductility for high performance tools which offers very good machinability and grindability compared to other high alloyed PM-tool steels. One advantage with Vanadis 4 Extra is that the dimensional stability after hardening and tempering is very good. This steel would therefore be suitable for CVD coating. Applications for this type of tool steel is where adhesive wear and/or chipping are the dominating failure mechanism such as blanking and forming of austenitic stainless steels and advanced high strength steels. Other examples for application areas could be steels with thicker work material, high strength work materials or even high strength steel sheet materials. Examples; Blanking and forming, fine blanking, cold extrusion tooling, powder pressing, deep drawing, knives or substrate steel for surface coating. Vanadis 4 Extra is characterized by:

- Very good ductility
- High abrasive-adhesive wear resistance
- High compressive strength
- Good dimensional stability during heat treatment and in service
- Very good through-hardening properties
- Good temper back resistance
- Good machinability and grindability

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<tr>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>Cr</th>
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<td>1.4</td>
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<td>0.4</td>
<td>4.7</td>
<td>3.5</td>
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Fig 6: CCT-Graph (Continuous Cooling) of Vanadis 4 Extra tool steel. Ausenitizing temperature 1020°C (1870°F). Holding time 30 minutes.

Fig 7: TTT-Graph (Isothermal Transformation) of Vanadis 4 Extra tool steel. Ausenitizing temperature 1020°C (1870°F). Holding time 30 minutes.
Sverker 21

This is a high-carbon, high-chromium tool steel alloyed with molybdenum and vanadium. This is a very common grade for cold work applications with very good abrasive wear resistance but with rather limited cracking resistance. It is suitable for manufacture of medium run tooling for applications where abrasive wear is dominant and the risk of chipping or cracking is not so high. Some applications could be blanking and forming of thinner, harder work materials. Sverker 21 is characterized by:

- High wear resistance
- High compressive strength
- Good through-hardening properties
- High stability in hardening
- Good resistance to tempering-back

Caldie

This is an ESR-grade (Electro Slag Refined) steel. It has good weldability, castability, through hardening properties, machinability and grindability. It is suitable for short to medium range run tooling where the predominant failure mechanism are chipping and/or cracking and where compressive strength (hardness over 60 HRC) is necessary e.g. cold work ultra high strength steels where high crack resistance is of importance. Coldwork applications could be, thread rolling dies or machine knives for fragmentation of plastics and metals. It is also suitable as a substrate for applications where surface coatings are necessary. Caldie is characterized by:

- Very good chipping and cracking resistance
- Good wear resistance
- High hardness (>60 HRC) after high temperature tempering
- Good dimensional stability in heat treatment and in service
- Excellent through-hardening properties
- Good machinability and grindability
- Excellent polishability
• Good surface treatment properties
• Good resistance to tempering back
• Very good WEDM properties

Fig 8: CCT-Graph (Continuous Cooling) of Caldie tool steel. Ausenitzing temperature 1025°C (1880°F). Holding time 30 minutes.

Fig 9: TTT-Graph (Isothermal Transformation) of Caldie tool steel. Ausenitizing temperature 1025°C (1880°F). Holding time 30 minutes.
**Comparison of selected tool steels**

One of the main research activities in this project was to conclude what type of tribological behaviour the selected tool steels suffer compared to each other. Since Vancron 40, Vanadis 4 Extra and Vanadis 6 are all PM-steels (Powder Metallurgical) and Caldie and Sverker 21 are traditional cold work tool steels the main failure mechanisms for these steels are adhesive wear, abrasive wear and chipping. During dry sliding conditions, like the case in this project, the most interesting mechanism to compare are adhesive wear or galling as it is called as well. Tribological studies show that the relation between friction and wear could be referred to as adhesive wear. Therefore the relation between galling resistance of the selected tool steels is of interest. Galling is a process of surface roughening that result from high contact pressure and high traction at slow speeds generally without lubricant. Comparing the selected tool steels in terms of resistance to galling the Vancron 40 –steels have the highest resistance followed by Vanadis 6, Vanadis 4 Extra and and Caldie. Sverker 21 is the worst steels in terms of galling compared to these other steels. The resistance of galling at less severe conditions is something yet to discover. In this study these less severe conditions will be analysed to see if the same pattern in behaviour could be mentioned.

![Fig 10: The left image showing adhesive wear of Vancron 40, right image showing galling of Vancron 40.](image)

**Atomic Force Microscopy**

The differences of macro- and micro/nano-tribology are that in macrotribology test components of large mass and with heavy loads are used which leads to wear all the way down to bulk material. Micro/nanotribology on the other hand is focused on small masses with almost no loads and surface wear of only a few atomic layers. Micro- and nanotribological techniques are ideal to study the friction and wear processes of micro- and nano structures. Such studies are valuable of fundamental understanding of interfacial phenomena in macrostructures to provide a bridge between science and engineering.
Atomic force microscopy (AFM) is used as a routine microscopy technique to explore topography of surfaces on micro- and nano scale. In AFM, one studies atomic forces between sample and probe tip at nm level. The AFM can work in several different modes, tapping-, non-contact- and contact mode are some of them. Generally, the method use atomic forces between sample and a sharp probe tip to provide a microscopic image of the surface. The tip is mounted on a cantilever, which is sensitive enough to deflect forces as low as $10^{-8}$ to $10^{-9}$ N. In the so-called contact mode, the AFM uses constant force between sample and tip, which create an image that results in contours of constant force between the tip and sample. In general, the tip is pushed or dragged over the sample surface recording topography. In this mode, the tip is in contact with the sample and can therefore damage the tip or even the surface. This means also that AFM provides experimental access to mechanical properties of surfaces on the atomic scale, for example friction.

A non-contact mode, AC-mode, was developed for the reason to avoid damage. This mode record information of the surface using an electrical potential between sample and tip. The non-contact mode can, therefore, be useful for more sensitive samples like biological material or polymers. Another important discovery made by the non-contact mode is that it can result in true atomic resolution, that is, the possibility of imaging individual atomic features. The presence of a regular atomic pattern obtained in contact-mode does not in fact mean that true atomic resolution has been achieved. The non-contact mode sets the cantilever into vibration using the resonance frequency as a tool to control the tip-sample distance. During tip approach the sample-tip interactions leads to changes of the resonance frequency. Another advantage of the non-contact potential attractive mode is the possibility to detect long range interactions (on the scale up to a few 10nm) like van der Waals, magnetic or electrostatic forces.
In applications today, AFM is used in the static deflection mode of the cantilever. This, generally, means that the deflection of the cantilever is measured from its zero position preset value, which controls the force the tip apply the sample. This mode can only be used after contact with tip and sample has been established. The position of the tip is defined by the balance of repulsive versus attractive interactions between sample and tip and is stable if the tip is pushed towards the sample by the cantilever. As mentioned before, this mode cannot record true atomic resolution since the applied forces are so large that deformation of about a hundred atoms occurs.

A well known term in AFM context is “jump to contact”. This is one phenomenon explaining why true atomic resolution cannot be obtained. When the tip approaches the sample at about 100 nm the attraction forces pull the tip towards the sample creating a so called jump to contact. After contact, the force balance is given by the repulsive interaction of the front part of the tip apex with the sample, the attractive interaction of the entire tip with the sample and the deflection of the cantilever. In this mode, the tip is constantly adjusted to maintain a constant deflection, and, therefore, a constant height above the surface. Another crucial thing about contact mode is that the tip’s stiffness needs to be lower then the spring constants holding the atoms together, which is about 1-10 nN/nm. Most tips of today have a spring constant of $< 1$ nN/m.
When the tips get worn, accuracy and resolution decreases, as seen above in fig 12. One way to measure the tip sharpness is to use some kind of reference sample as in Fig 13 below, a porous aluminium sample, PA01, was used. It shows how a sharp new tip influenced resolution of the method after one session and then after more sessions of sliding contact mode. Note that not only decrease in sharpness of tip may occur, chipping of the tip is very common as well. This of course makes the tip useless in terms of sharpness and accuracy.

When performing so called friction measurements, the AFM measures the lateral force applied on the cantilever or as a practical approach the “twist” of the cantilever. With this tool one can quantitatively determine areas of higher or lower friction, see fig. 14a. When the tip
slides over an area with higher friction the tip tilts more and the lateral deflection is measured. These regions show up as darker or lighter areas in AFM imaging, see fig 15. When sliding over an obstacle, the tip may also tilt a little bit, registering a friction, but this friction pattern is not indeed actual friction, it only show that the tip “climbs” on top of the obstacle as seen in fig 14a.

**Fig 14a:** Showing how Lateral Force imaging is made.

**Fig 14b:** Comparison between height map (left) and friction map (right), darker areas in the friction map showing lower friction.
Fig 15: Comparison between height map (left) and friction map (right), illustration of how “climbing” is formed in lateral imaging, scan direction from left to right.

Adhesion properties at nanoscale can be measured in AFM using the so called force-distance curve measurements. A typical force-distance curve is showed in fig 16. The point denoted as 1 is the starting point, when the sample started to approach the probe. In the segment 1–2 there is no interaction between the sample and the tip. At point 2, the cantilever jumps to contact as an effect of an attraction contact by surface forces. From this, an interaction between sample and probe is observed. The point denoted as 3 indicates the last point in the range of the optical detection system. Line 3-4 and point 4 - adhesion.

Fig 16: Showing a typical force-distance curve. Point 1-2 cantilever in rest, 2: jump to contact, 2-3: contact between sample and cantilever, 4: loss of contact between tip and sample.
The non-contact mode utilizes the vibrations of the tip and the attraction between tip and sample to create the images, and it is in contradiction to the contact mode imperative for the tip not to touch the sample when approaching. The detection scheme for imaging is based on measuring changes to the resonant frequency or amplitude of the cantilever.

Tapping mode utilizes the oscillation of the cantilever in the same way as in non-contact mode but instead of not touching the surface it intermittently touches or “taps” on it. This mode uses very stiff cantilevers as the tips could get stuck in, for instance, contamination layers. Tapping mode is very often used on soft samples, lateral resolution is better and lateral forces such as drag, common in contact mode, are practically eliminated.

Recent research made by AFM facilities shows that AFM is a powerful tool for micro/nano frictional measurements. A study made by Heikkilä [14] show that frictional differences in both matrix and hard phases could be obtained for Vancron 40, Sverker 21, Weartec and Ferrotitanit steels. She also states that Atomic Force Microscope can be used to image adhesion characteristics of tool steel phases. Szlufarska et. al. [16] concludes that in particular experiments with the scanning force microscope provided well-defined interfaces for tribological studies. The materials and conditions studied continue to broaden to this day. The last two decades of developments in scanning force microscopy enabled measurements of forces in the sub-nanoneutron regime, and led to characterization of tribological properties of nanometre-scale contacts in various environments for a wide range of materials. Macroscopic tribology often focuses on determining the friction coefficient and wear rate for the materials of interest. Neither, the friction coefficient nor the wear rate is an intrinsic physical property, as both can be strongly dependent on the specific structure, chemistry and elastic/plastic properties of the surfaces, on the chemical environment in which the measurements are performed, and on the sliding history of the interface. These properties can also depend on the mechanics of the instrument itself that is used to make the measurement. Because of the complex nature of nanotribology, fundamental understanding requires experiments at well-defined interfaces. Therefore, single-asperity contact measurements have been a very useful tool in such studies. AFM measurements can be performed in a variety of environments: ambient air, controlled atmosphere, liquids or ultrahigh vacuum (UHV) giving a wide range of application areas. Another important thing to mention is since the tip is attached to the cantilever, the technique is referred to as being load controlled, which means that the load can be set to whatever the user wants to. Tribological studies using these techniques are still
scarce but because of their great promise for unravelling nanotribological phenomena, they are worthy of further pursuit.

**Materials and methods**

In this study Atomic Force Microscope (AFM), Optical Profilometer (OP), Sliding on Flat Surface (SOFS) and Scanning Electron Microscope (SEM) was used. To be able to characterize the surfaces of each tool steel a standard polishing routine was made. In AFM it is critical to have a clean polished surface and for these studies an Ra of about 0.05 µm was accepted. This was to ensure that the range of roughness lies within the size of carbides and other inclusions. To conclude the roughness of the polished steels samples OP measurements were carried out. AFM was used to characterize the surface in terms of frictional and adhesion properties as well as morphology and roughness. The purpose of these studies was to find out if there are any correlations between micro/nano scaled and macro scaled frictional properties. To measure the macro scale frictional behaviour of the steels SOFS-testing was performed. SOFS is a tribometer, measuring friction coefficient during dry sliding conditions.

To be able to measure features of wear track the SOFS made on the surface, OP and SEM studies were made. In OP, the wear tracks were analysed in terms of depth and width. In SEM, the wear tracks were analysed in terms of chemical composition and morphology.

**Project procedure, practical tasks**

**Stage 1: Preparation of specimens**

1. Following measured hardness of the selected grades, one standard polishing procedure is to be selected based on reference handbook.
2. Microstructural characterization of the polished specimens
3. Surface characterization, morphology and roughness, by means of; AFM, OP and SEM
4. AFM tribology – selection of specific carbides and matrix regions, frictional measurements of the friction on selected phases
5. AFM tribology – selection of region and measurements of the friction over the whole region

Expected results: Characterized surfaces which can be comparable with each other. Friction properties of different phases
Stage 2: SOFS tribotestings

1. SOFS - Calculation of average contact pressure in dependence of surface roughness measured previously
2. SOFS - One step tests with selected number of cycles/strokes, load if the same, sliding distance is 10mm
3. SOFS - measurements of friction coefficients during sliding

Expected results: Macro-scaled friction properties.

Stage 3: Nanoscale characterization of damage and wear mechanisms

1. AFM - surface roughness and morphology of the track, friction along the track.
2. OP - surface roughness and morphology of the track, track profile and depth.
3. SEM - damage mechanisms, fracture of particular phases, smearing the matrix?
4. Auger - chemistry of the track bottom, if counterbody wear was observed.

Expected results: Nano-scaled friction properties and surface behaviour after sliding contact.

Table 1: Test matrix stages 1, 2 and 3

<table>
<thead>
<tr>
<th>Grades/Stages</th>
<th>Stage 1</th>
<th>Stage 2</th>
<th>Stage 3</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Standard Polishing</td>
<td>OP, Ra, and Rt</td>
<td>AFM, Friction</td>
</tr>
<tr>
<td>Va 40 1</td>
<td>X</td>
<td>X</td>
<td>X</td>
</tr>
<tr>
<td>Va 40 2</td>
<td>X</td>
<td>X</td>
<td>X</td>
</tr>
<tr>
<td>Va 40 3</td>
<td>X</td>
<td>X</td>
<td>X</td>
</tr>
<tr>
<td>Vanadis 6</td>
<td>X</td>
<td>X</td>
<td>X</td>
</tr>
<tr>
<td>Vanadis 4x</td>
<td>X</td>
<td>X</td>
<td>X</td>
</tr>
<tr>
<td>Caldie</td>
<td>X</td>
<td>X</td>
<td>X</td>
</tr>
<tr>
<td>Sverker 21</td>
<td>X</td>
<td>X</td>
<td>X</td>
</tr>
<tr>
<td>Duration days</td>
<td>2</td>
<td>2 weeks</td>
<td>1 weeks</td>
</tr>
<tr>
<td>Totally</td>
<td>8 weeks maximum</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Project Procedure, Analysing

AFM – Atomic Force Microscope

Scanning procedure

i) Tip Shape (20min)
   - Place sample PA01 in AFM
   - Mount NSC18 / A1BS – tip (Make sure to get a minimum of
     3.5V in signal)
   - Scan surface to get tip shape
   - Save data

ii) Place sample you want to investigate in AFM (20min)
   - Measure resonance frequency \( \omega \)
   - Calculate
     \[
     k_c = m \omega^2
     \]
     \[
     m = L_t * b * \rho
     \]
     \[
     \omega = \sqrt{\frac{k_c}{m}}
     \]
   - Known parameters: \( L, t, b, \rho \)
   - Set \( k_c \)-value in SPMLab

iii) Select these modes: (0min)
   - Lateral Force Mode (Forward, Backwards)
   - Deflection Mode (Forward, Backwards)
   - Heigth Mode (Forward, Backwards)

iv) Scan surface (100min)
   - Make sure that “Deflection” doesn’t reach above 5 % of “Height”
   - 90x90 \( \mu \text{m} \), Save data
   - 40x40 \( \mu \text{m} \), Save data
   - 10x10 \( \mu \text{m} \), Save data

v) Perform Point Spectroscopy of three different phases (20min)
   - Point 1, matrix
   - Point 2, large carbide
   - Point 3, small carbide
   - Save data

vi) Tip Shape (20min)
- Place sample PA01 in AFM
- Scan surface to get tip shape
- Save data

Total time estimation; stage i) – vi) 180min = 3h

**Calibration of tip-shape:**

i) Place sample PA01 in AFM
ii) Mount new NSC18 / A1BS – tip (Make sure to get a minimum of 3.5V in signal)
iii) Scan surface to get tip shape
iv) Place sample you want to investigate in AFM
v) Make an adhesion test (point spectroscopy)
vi) Place sample PA01 in AFM
vii) Scan surface to get tip shape
viii) If tip isn’t deformed repeat step iv) – vii) consequently until tip is worn.

**Calibration of tip stiffness:**

i) Mount NSC18 / A1BS – tip (Make sure to get a minimum of 3.5V in signal)
ii) Measure resonance frequency
iii) Calculate

\[ k_c = m\omega^2 \]

\[ m = L*t*b*\rho \]

\[ \omega = \sqrt{\frac{k_c}{m}} \]

Known parameters: \( L, t, b, \rho \)
Set \( k_c \)-value in SPMLab

**OP - Optical Profilometer**

Time estimation 40min

**Scan areas:**

- 90 x 90 μm
- 40 x 40 μm

**Magnification:**

- 50 x magnification with scan area 90 x 90 μm
100 x magnification with scan area 40 x 40 µm

**Procedure:**

**Before wear test**

i) Start up the computer and place the sample under lowest magnification lens.

ii) Make sure that the software has the correct data in: magnification, scan area etc.

iii) Find a spot for investigation on the sample by adjusting the x-y-joystick.

iv) Focus until interference pattern moves slightly out of the picture.

v) Scan the area of investigation and save the raw data. Remember to name the file correct: chemical composition, stage of investigation (damaged surface or not).

vi) Scan and save raw data for earlier stated magnification and scan areas.

**After wear test**

i) Start up the computer and place the sample under lowest magnification lens.

ii) Make sure that the software has the correct data in: magnification, scan area etc.

iii) Find a spot for investigation on the sample by adjusting the x-y-joystick.

iv) Focus until interference pattern moves slightly out of the picture.

v) Scan the area of investigation and save the raw data. Remember to name the file correct: chemical composition, stage of investigation (damaged surface or not).

vi) Scan the wear track at min 3 positions: start, middle and end.

vii) Scan and save raw data for earlier stated magnification and scan areas.

**SEM – Scanning Electron Microscope**

**Scan areas:**

90 x 90 µm

40 x 40 µm

10 x 10 µm

**Procedure:**

**Before wear test**

i) Start up the computer and place the sample, with holder, in vacuum chamber.

ii) Scan areas of interest using BSE, SE and EDS. Remember that EDS has a limitation in investigation area: 9-10 µm.

iii) Scan the area of investigation and save pictures with the right scale bar. Remember to name the file correct: chemical composition, stage of investigation (damaged surface or not).

iv) Scan and save pictures, with scale bar, for earlier stated scan areas.
v) Tilt sample and repeat step i – iv.

**After wear test**

i) Start up the computer and place the sample, with holder, in vacuum chamber.

ii) Scan wear track with earlier stated scan area, using BSE, SE and EDS. Remember that EDS has a limitation in investigation area: 9-10 µm.

iii) Scan the area of investigation and save pictures with the right scale bar. Remember to name the file correct: chemical composition, stage of investigation (damaged surface or not).

iv) Scan and save pictures, with scale bar, for earlier stated scan areas.

v) Tilt sample and repeat step i – iv.

**SOFS – Sliding On Flat Surface**

*Track length:*

- 10 mm

*Load:*

- 10 N

*Procedure:*

i) Clean sample and wheel in ethanol.

ii) Place the sample in special holder and fasten the whole package to a magnetic table.

iii) Make sure that the computer software is set to slide in reciprocating mode, with sliding length of 10 mm. Decide which distance to use for recording raw data.

iv) Apply linear increasing load and investigate the load of interest for investigation of samples.

v) Save data with correct filename.

vi) Apply min load, from iv, and do 1 sliding test for each sample. (10x3 min)

vii) Save data with correct filename.

viii) Repeat vi, with 50 sliding test for each sample. (20x3 min)

ix) Save data with correct filename.

x) Repeat vi, with 500 sliding test for each sample. (250x3 min)

xi) Save data with correct filename.
Results:
To be able to measure surface-roughness, after standard polishing routines were made, the specimens were put into an Optical Profilometer using the setup shown above. The specific Ra-values for the different steels are shown in Table 2:

Table 2: Ra-values of different steels after polishing, measured in Optical Profilometer.

<table>
<thead>
<tr>
<th></th>
<th>Va 40 I</th>
<th>Va 40 II</th>
<th>Va 40 III</th>
<th>Vanadis 6</th>
<th>Vanadis 4 Extra</th>
<th>Caldie</th>
<th>Sverker 21</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ra (µm)</td>
<td>0.04</td>
<td>0.05</td>
<td>0.05</td>
<td>0.02</td>
<td>0.05</td>
<td>0.1</td>
<td>0.05</td>
</tr>
</tbody>
</table>

Optical Profilometer was also used to characterize what kind of track the SOFS made on the surface. All steel grades illustrate the same behaviour; some kind of layer is built on top of the tracks.

Fig 17: Showing Optical Profilometer measurements, after sliding contact in SOFS. The 1st top left corner image is for Vancron 40 I, 2nd image Vancron 40 II, 3rd image Vancron 40 III, 4th image Vanadis 6, 5th image Vanadis 4 Extra, 6th image Sverker 21 and 7th image Caldie.
SOFS-measurements were mainly carried out to be able to distinguish the frictional behaviour of each tool steel in terms of macro-friction. These tests were also the main findings in understanding of what happened with the material at micro- and nano-scale. The steels behaved similar. Started out with a quite low value of friction coefficient and then elevated to a certain point where they then stabilised after several slidings, see picture 18. The steel, which differs from the others, is Caldie, see picture 18. Table 3 shows how the frictional coefficient changed from zero slidings to 100 slidings. The sliding distance was 15 mm with a reciprocating sliding of a total of 100 slides, giving a total sliding distance of 1.5 meters, with a load of 10 N. See Appendices for detailed information.

Table 3: Showing $\mu$-values of different steels after zero slidings and at which $\mu$-value the specific steel stabilized on.

<table>
<thead>
<tr>
<th></th>
<th>Va 40 I</th>
<th>Va 40 II</th>
<th>Va 40 III</th>
<th>Vanadis 6</th>
<th>Vanadis 4 Extra</th>
<th>Sverker 21</th>
<th>Caldie</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\mu$, starting point</td>
<td>0.3</td>
<td>0.3</td>
<td>0.3</td>
<td>0.4</td>
<td>0.6</td>
<td>0.2</td>
<td>0.5</td>
</tr>
<tr>
<td>$\mu$, stabilized</td>
<td>0.5</td>
<td>0.9</td>
<td>0.9</td>
<td>0.9</td>
<td>0.9</td>
<td>0.8</td>
<td>0.5</td>
</tr>
</tbody>
</table>

Fig. 18: Showing behaviour of friction propagation after sliding contact in SOFS. The 1st top left corner image is for Vancron 40 I, 2nd image Vancron 40 II, 3rd image Vancron 40 III, 4th image Vanadis 6, 5th image Vanadis 4 E, 6th image Sverker 21, 7th image Caldie.
AFM-measurements were carried out to see if different phases contribute to different frictions and if the steels could be characterized based on friction and height maps. Almost every steel that was investigated showed differences in frictional behaviour in two or more phases. And it is therefore possible to characterize different steels based on friction and height maps.

Fig. 19: Demonstration of how the AFM measurements of surface topology was made, this is a Vancron 40 III specimen, 1\textsuperscript{st} image top left showing a height scan over 40x40\(\mu\)m, 2\textsuperscript{nd} image 20x20\(\mu\)m, 3\textsuperscript{rd} image 10x10\(\mu\)m, 4\textsuperscript{th} image 5x5\(\mu\)m, 5\textsuperscript{th} image 2,5x2,5\(\mu\)m and the last and 6\textsuperscript{th} image 1,2x1,2\(\mu\)m.

With the AFM used in this study it is not possible to pinpoint the exact friction coefficient value. To do that, one has to use so called FFM (Friction Force Microscopy), but it is enough to be able to distinguish higher friction regions from lower. Low friction areas are plotted as darker regions while higher friction areas are plotted as lighter regions as shown in back-scanned images, fig 20.
Fig. 20: Demonstration of how the AFM measurements of friction maps was made, this is a Vancron 40 I specimen, 1st image top left showing a lateral scan over 20x20µm, 2nd image 10x10µm, 3rd image 5x5µm, 4th image 2.5x2.5µm, 5th image 1.2x1.2µm and the last and 6th image 0.6x0.6µm.

The Vancron 40 tool steel (I, II and III) showed signs of three different frictional phases, Vanadis 6 and Vanadis 4E showed signs of two different frictional phases, while Caldie and Sverker 21 only showed signs of one frictional phase, matrix phase.

Table 4: Shows a summary of how much amount of high friction phases the steels have and also adhesion measurements in different frictional areas.

<table>
<thead>
<tr>
<th></th>
<th>Va 40 I</th>
<th>Va 40 II</th>
<th>Va 40 III</th>
<th>Vanadis 6</th>
<th>Vanadis 4 Extra</th>
<th>Caldie</th>
<th>Sverker 21</th>
</tr>
</thead>
<tbody>
<tr>
<td>Adhesion carbide phases (µN)</td>
<td>Small 0.030 Big 0.056</td>
<td>Small 0.112 Big 0.100</td>
<td>Small 0.050 Big 0.060</td>
<td>0.050</td>
<td>0.0875</td>
<td>X</td>
<td>0.110</td>
</tr>
<tr>
<td>Adhesion matrix (µN)</td>
<td>0.050</td>
<td>0.096</td>
<td>0.060</td>
<td>0.030</td>
<td>0.062</td>
<td>0.025</td>
<td>0.080</td>
</tr>
</tbody>
</table>

To be able to characterize the tracks, in terms of material constitution, SEM investigations was made. Another big conclusion to be made in SEM was to find that the surface looked the same in Scanning Electron Microscopes compared to in Atomic Force Microscope, fig 21.
The purpose of SEM investigations was to find out that something happens during sliding contact and conclude where and what has happened. The main results from SEM were that the particles found in OP built on top of the track mainly consist of a thin oxide layer which forms during sliding, see fig 22.

Fig 21: Comparison between Vanadis 4 Extra SEM imaging 40x40µm (to the left) and AFM imaging 40x40µm (to the right).

Fig 22: Showing oxide layer which formed on top of Vancron 40 I specimens wear track.
Fig. 23: SEM image showing an oxide particle which was formed in the wear-track of a Sverker 21 specimen.

Fig. 24: Showing a line scan over an oxide particle which was formed in the wear-track of a Sverker 21 specimen. Plot showing the amount of oxygen (dark blue) related to other alloying elements.
To ensure that the hardening process was made in a proper way a so called retained austenite measurement was performed. Table 5 below shows that retained austenite was not any problem in these materials.

**Table 5: Results of Retained Austenite measurement.**

<table>
<thead>
<tr>
<th>Retained Austenite (%)</th>
<th>Va 40 I</th>
<th>Va 40 II</th>
<th>Va 40 III</th>
<th>Vanadis 6</th>
<th>Vanadis 4 Extra</th>
<th>Caldee 21</th>
<th>Sverker 21</th>
</tr>
</thead>
<tbody>
<tr>
<td>&lt; 2</td>
<td>&lt; 2</td>
<td>&lt; 2</td>
<td>&lt; 2</td>
<td>&lt; 2</td>
<td>2 ± 1</td>
<td>2 ± 2</td>
<td></td>
</tr>
</tbody>
</table>

Below follows detailed information of each steel tested in this project in terms of Optical Profilometer, Atomic Force Microscope and Scanning Electron Microscope.
**Vancron 40 I**

SOFS-test show that the friction coefficient increases from 0.3 up to 0.5 at relatively constant rate. From OP measurements it shows that the track width was about 200µm and that some kind of layer, about 0.08µm high, was built upon the track, SEM measurements prove that the layer consist of oxide. AFM measurements show three different kinds of frictional behaviour, matrix, carbide and small particles. Notable is that the small particles, about 10x10nm in size, register a much lower friction then carbides and matrix.

![Friction Coefficient trendline of Vancron 40 I obtained from SOFS with applied load of 10N and 100 reciprocating slidings.](image)

*Fig 25: Friction Coefficient trendline of Vancron 40 I obtained from SOFS with applied load of 10N and 100 reciprocating slidings.*
Fig 26: Optical Profilometer map of Vancron 40 I wear-track 5X magnification, $Ra = 0.04\mu m$, a) 2D-line measurement, b) 3D-map.

Fig 27: AFM measurements of Vancron 40 I, height map (left), friction map (right).

Fig 28: AFM measurements of Vancron 40 I, height map (left), friction map (right).
Fig 29: SEM measurement of Vancron 40 I in different resolutions.

Fig 30: SEM measurement of Vancron 40 I. To the left is a SE2 scan and to the right is a BSE scan. This picture shows that the layer on top of the steel is in fact different in composition.
**Vancron 40 II**

SOFS-test show that the friction coefficient increases from 0,3 up to 0,5 at a relatively quick rate and then continue increasing at a constant rate up to 0,9. From OP measurements it shows that the track width was about 300µm and that some kind of layer, about 0,08µm high, was built upon the track, SEM measurements prove that the layer consist of oxide. AFM measurements show three different kinds of frictional behaviour, matrix, carbide and small particles. Notable is that the small particles, about 10x10nm in size, register a much lower friction then carbides and matrix.

![Friction Coefficient trendline of Vancron 40 II obtained from SOFS with applied load of 10N and 100 reciprocating slidings.](image)

Fig 31: Friction Coefficient trendline of Vancron 40 II obtained from SOFS with applied load of 10N and 100 reciprocating slidings.
Fig 32: Optical Profilometer map of Vancron 40 II wear-track 5X magnification, $Ra = 0.05\,\mu m$, a) 2D-line measurement, b) 3D-map.

Fig 33: AFM measurements of Vancron 40 II, height map (left), friction map (right).

Fig 34: SEM SE2 scan of Vancron 40 II showing to the left the wear track and to the right the center of the wear track with a higher resolution.

Fig 35: SEM measurement showing difference in SE2 scan (left image) and BSE scan (right image).
**Vancron 40 III**

SOFS-test show that the friction coefficient increases from 0,3 up to 0,9 at a relatively quick rate and stabilizes. From OP measurements it shows that the track width was about 200µm and that some kind of layer, about 0,10µm high, was built upon the track, SEM measurements prove that the layer consist of oxide. AFM measurements show three different kinds of frictional behaviour, matrix, small carbides and big carbides.

![Friction Coefficient trendline of Vancron 40 III obtained from SOFS with applied load of 10N and 100 reciprocating slidings.](image1)

**Fig 36:** Friction Coefficient trendline of Vancron 40 III obtained from SOFS with applied load of 10N and 100 reciprocating slidings.

![Optical Profilometer map of Vancron 40 III wear-track 5X magnification, Ra = 0,05µm, a) 2D-line measurement, b) 3D-map.](image2)

**Fig 37:** Optical Profilometer map of Vancron 40 III wear-track 5X magnification, Ra = 0,05µm, a) 2D-line measurement, b) 3D-map.
Fig 38: AFM measurements of Vancron 40 III, height map (left), friction map (right).

Fig 39: AFM measurements of Vancron 40 III, height map (left), friction map (right). Green areas showing one carbide phase, purple areas showing another carbide phase.
**Vanadis 6**

SOFS-test show that the friction coefficient increases from 0.4 up to 0.75 at a relatively quick rate and then stabilizes around 0.9. From OP measurements it shows that the track width was about 200µm and that some kind of layer, about 0.10µm high, was built upon the track, SEM measurements prove that the layer consist of oxide. AFM measurements show two different kinds of frictional behaviour, matrix and carbides.

*Fig 40: Friction Coefficient trendline of Vanadis 6 obtained from SOFS with applied load of 10N and 100 reciprocating slidings.*
Fig 41: Optical Profilometer map of Vanadis 6 wear-track 5X magnification, $Ra = 0.02 \mu m$, a) 2D-line measurement, b) 3D-map.

Fig 42: AFM measurements of Vanadis 6, height map (left), friction map (right).

Fig 43: SEM measurement of Vanadis 6 showing an overview of the wear track to the left, and the centre of the wear track with a higher resolution to the right.

Fig 44: SEM measurement of Vanadis 6 showing even higher resolution of the wear track to the left, and the centre of the wear track with a higher resolution to the right.
Vanadis 4 Extra

SOFS-test show that the friction coefficient increases from 0.5 up to 0.9 at a relatively quick rate and then stabilizes around 0.9. From OP measurements it shows that the track width was about 200µm and that some kind of layer, about 0.18µm high, was built upon the track, SEM measurements prove that the layer consist of oxide. AFM measurements show no signs of frictional differences.

Fig 45: Friction Coefficient trendline of Vanadis 4 Extra obtained from SOFS with applied load of 10N and 100 reciprocating slidings.
Fig 46: Optical Profilometer map of Vanadis 4 Extra wear-track 5X magnification, $Ra = 0.04\mu m$, a) 2D-line measurement, b) 3D-map.

Fig 47: AFM measurements of Vanadis 4 Extra, height map (left), friction map (right).

Fig 48: SEM measurement of Vanadis 4 Extra showing an overview of the wear track to the left, and the centre of the wear track with a higher resolution to the right.
**Sverker 21**

SOFS-test show that the friction coefficient increases from 0.2 up to 0.8 at a relatively quick rate and then stabilizes around 0.8. From OP measurements it shows that the track width was about 300µm and that some kind of layer, about 0.15µm high, was built upon the track, SEM measurements prove that the layer consist of oxide.

---

**Fig 49: Friction Coefficient trendline of Sverker 21 obtained from SOFS with applied load of 10N and 100 reciprocating slidings.**

**Fig 50: Optical Profilometer map of Sverker 21 wear-track 5X magnification, Ra = 0.05µm, a) 2D-line measurement, b) 3D-map.**
Fig. 51: SEM image showing an oxide particle which was formed in the wear-track of a Sverker 21 specimen.

Fig. 52: Showing a line scan over an oxide particle which was formed in the wear-track of a Sverker 21 specimen. Plot showing the amount of oxygen (dark blue) related to other alloying elements.
Caldie

SOFS-test show that the friction coefficient is stable around 0.5. OP measurements show very poor signs of track width only one scratch was observed. SEM measurements prove that the track was around 30µm in width and that a layer of oxide was formed in the track.

Fig 53: Friction Coefficient trendline of Caldie obtained from SOFS with applied load of 10N and 100 reciprocating slidings.

Fig 53a: Optical Profilometer map of Caldie wear-track 10X magnification, Ra = 0.01µm, a) 2D-map, b) 3D-map, c) 2D-line measurement.
Fig 54: Optical Profilometer map of Caldie wear-track at 5X magnification, $Ra = 0.01\mu m$

Fig 55: SEM measurement of Caldie showing an overview of the wear track to the left, and the centre of the wear track with a higher resolution to the right.

Fig 56: SEM measurement of Caldie showing an even higher resolution image of the wear track to the left, and the centre of the wear track with a higher resolution to the right.
Discussion

The OP shows that the surface roughness after polishing is very low, this was desirable to be able to distinguish carbides from matrix and vice versa after doing SOFS testing. It is also imperative with as smooth surface as possible to be able to use AFM method for analysing surfaces with different phases in regard to friction and adhesion properties. The polishing routine for these steels were standard polishing methods. Measuring surface roughness for these tool steels were a bit more difficult then expected. The Optical Profilometer microscope uses a type of interference pattern scanning over the surface and recording roughness.

As figure 57 shows, you see only that specific interference pattern. This in the other hand, means that the specimen is very well polished and it shows that the polishing routine were successful.

The measurements over the tracks show that the steels get worn as well as it is something sticking or growing upon the wear-tracks. This is consequent throughout all specimens. To be able to analyse what happened on the track one have to use another kind of analysing equipment such as SEM.

In SOFS the main issue was to recognize a pattern of how the steels behave in terms of friction. To be able to distinguish whether it was the matrix or other phases which corresponds to this frictional behaviour is very hard to say. When the tracks were investigated in SEM it was found that carbides interact first and works as frictional enhancers. But one must realize that the SOFS-machine suffers many child diseases and is very unreliable in terms of consequent testing. Even though it was programmed in the same order, with constant load and same amount of number of slides every time, it did not behave in the same way all the time. One other thing to take into account is the wear of the counterbody which in this study was not investigated at all.
The AFM method used was sliding contact mode with a silicon tip NSC 18 / A1BS. To be able to perform as accurate tests as possible great amount of work was put into calibrating the tip and making sure that same conditions for each steel was held. As the tips get worn accuracy and resolution decreases. To maintain the sharpness of the tips measurements of tip shape was measured before and after each session. Fig 58 shows how a sharp tip can look like after only one session of sliding contact mode and then after more sessions be useless in terms of sharpness and accuracy.

![Fig 58: Shows how the tip shape gets worn from new (left), after one session (middle) and after several sessions (right).](image)

All Steels had a friction coefficient between 0,2 - 0,5 at the beginning of the friction test in SOFS, one steel; Caldie, showed no changes in friction coefficient during sliding while the other steels like Vancron 40 had a certain increase of friction coefficient. Vancron 40, Vanadis 6 and 4 Extra as well as Sverker 21 showed this behaviour. The highest measured friction coefficient was 0,9. Some notes about these values should be that Vancron 40 I differs from the other Vancron 40 specimens and that the Vanadis specimens have a higher friction coefficient at the beginning but at the end the same as for some of the Vancron’s.

*Table 6: Showing $\mu$-values of different steels after zero slidings and at which $\mu$-value the specific steel stabilized on.*

<table>
<thead>
<tr>
<th>Steel</th>
<th>$\mu$, starting point</th>
<th>$\mu$, stabilized</th>
</tr>
</thead>
<tbody>
<tr>
<td>Va 40 I</td>
<td>0,3</td>
<td>0,5</td>
</tr>
<tr>
<td>Va 40 II</td>
<td>0,3</td>
<td>0,9</td>
</tr>
<tr>
<td>Va 40 III</td>
<td>0,3</td>
<td>0,9</td>
</tr>
<tr>
<td>Vanadis 6</td>
<td>0,4</td>
<td>0,9</td>
</tr>
<tr>
<td>Vanadis 4 Extra</td>
<td>0,6</td>
<td>0,9</td>
</tr>
<tr>
<td>Sverker 21</td>
<td>0,2</td>
<td>0,8</td>
</tr>
<tr>
<td>Caldie</td>
<td>0,5</td>
<td>0,5</td>
</tr>
</tbody>
</table>

The wear tracks for the steels differ all the way from 20µm - 300µm in width. It was on Caldie which the track was 20µm, the other specimens had tracks with a width of 200µm - 300µm. On each of the wear tracks a so called tribolayer emerged. Even at very first contact this layer was built on. The layer consists of some kind of oxide. The thickness of the oxide layer were in general the same for all the specimens, very thin, about 0,10 - 0,20µm thick.
As stated earlier the main attention of this study was to find out whether AFM facilities was a powerful tool for analysing steels in terms of frictional behaviour and adhesion properties on micro/nano scale. Both in educational purposes and in the plain research field the results were evident with expected results. As Heikkilä stated in 2005, hard phases for instance in Vancron 40 had lower friction than matrix phase, was something expected also in this study and clearly the measurements of Vancron 40 et al. conclude this. One thing to mention is the small particles with even lower friction then carbides in the Vancron 40 specimens. It can be concluded that the particles are embedded in the matrix due to that they don’t register any height in AFM height measurements. These small particles wasn’t found in Irma’s research and could therefore be interesting in future work point if view. The other steels like Vanadis 6 and Vanadis 4 Extra also showed signs of those carbides contribute with lower friction. The reason to believe the images from AFM is simply shown in Fig 59 below, note sliding direction from right to left. The circles is showing a carbide on the surface, now take a look at the right side of it. It is a little bit lighter around the edge of the carbide, showing signs of the tip “hitting” something. The so called “hit” makes the tip deflect and register a false impression of that it would be higher friction. If you look at the other side of the carbide the edge is darker, showing signs of that the tip more easy slips down from the carbide. This concludes that lighter areas are high friction - and darker areas are low friction areas.

![Fig 59: Comparison between height map (right) and friction map (left), illustration of how “climbing” is formed in lateral imaging](image)

Comparing these micro/nano scaled friction properties of the steels with the macro friction test made show signs of that the small particles may have a great amount of effect on the macro friction. Vancron 40 steels showed signs of lower friction coefficient properties at the beginning of the wear tests while Vanadis 6 and Vanadis 4 Extra’s friction coefficient was a
bit higher. This could be an effect of the small particles acting as friction reducers at beginning of sliding contact.

Conclusions

General conclusions are;

- Even at very first sliding contact an oxide layer forms on each steel grade analysed in this project.
- It is possible to characterize different steels in terms of friction and height mapping from AFM facilities.
- Friction measurements on micro/nano-scale can show how the steels behave in macro frictional environments.

Main conclusion to be made in this research is that AFM facilities can be used as a tool for measuring frictional properties of different phases in tool steels on micro/nano scale. Correlations between friction measurements made in AFM facilities and macro scaled friction measurements are possible.
References

[11] - Recent Advances in Manufacture & Use of Tools & Dies and Stamping of Steel Sheets, Nader Asnafi, October 5-6, Olofström Sweden, 2004
[16] - Recent advances in single-asperity nanotribology, Izabela Szłufarska1, Michael Chandross and Robert W Carpick, Department of Materials Science and Engineering, University of Wisconsin, Madison, WI 53706, USA, Sandia National Laboratories, Albuquerque, NM 87123-1411, USA, Department of Mechanical Engineering and Applied