Tribological Behaviour of Surface Treated and Postoxidised Tool Steels at Elevated Temperature

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PREFACE

This thesis is a summary of my work as a Master’s Student of Advanced Material Science and Engineering (AMASE). This work has been carried out in the field of high temperature tribology at the Division of Machine Elements under the supervision of Professor Braham Prakash.

I am grateful to everyone who has supported me during this work, especially to Jens Hardell for his guidance, discussion and great support during my thesis work. Finally I thank my supervisor Professor Braham Prakash, without whose invaluable advice and great ideas this thesis would not have been possible. I am also thankful to Johnny Grahn for his great support during my thesis.

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I am thankful to my friends (Adroits) without whose great support, suggestions and encouragement during my thesis work.

Finally I would like to dedicate this thesis work to my Parents Syed Jahangeer and Bismilla begum.

– Syed Abdul Khadar
ABSTRACT

This work is a part of an ongoing research project at the Division of Machine Elements at Luleå University of Technology. The research mainly deals with the tribological behaviour of surface treated and post oxidised tool steels at elevated temperature. There are many tribological interfaces that are exposed to elevated temperatures. The material which subjected to elevated temperatures results in highly complex interfaces due to chemical reactions, changing in microstructure and morphology. The mechanical properties will also have a great influence on the tribological properties at elevated temperatures.

Another important consideration is lubrication since it is not possible to use conventional lubricants at high temperatures. Instead it may be beneficial to utilise surface treatments and pre-oxidised surfaces for friction and wear control. The aim of this work is to study the tribological properties of plasma nitrided tool steels which are also post-oxidised to form an oxide layer on top of the nitrided layer.

The experimental work has been carried out by using pin on disc machine at room temperature and at 400 °C. The experimental materials were tool steels of three compositions. All tool steel samples were plasma nitrided and post oxidised at 500 °C. One of the tool steels was also post oxidised at 480 °C and 520 °C. The mating disc specimen was made from hardened high strength boron steel. The specimens have been analysed by using SEM/EDS, XRD and 3D optical surface profiler.

The results have shown that both friction and wear of tool steel and high strength boron steel pairs are temperature dependent. Increase in temperature results in increasing of friction and wear. The tool steel wear is higher at elevated temperature when compared to wear at room temperature. The tool steel samples post oxidised at 500 °C has better friction and wear performance not only at room temperature but also at higher temperature.
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I. Introduction

1. Tribology

The word Tribology was first reported in a landmark report by Jost (1966) which was published by the UK department of education and science. The word is derived from the Greek word Tribos meaning rubbing, so the literal translation would be “the science of rubbing”. The committee led by Peter Jost, was given the task of highlighting the need for more education and research within in the field of lubrication. The jost report concluded that approximately 1% of the gross national product of the UK could be saved by decreasing friction and wear. Historically it can be found that even the ancient Egyptians possessed some knowledge about Tribology even though they did not use that term. Leonardo da Vinci performed his friction experiments in the 1490’s and showed that friction was dependant in the normal load on the material but not on the contact area [1].

Dictionaries define tribology as the science and technology of interacting surfaces in relative motion and related subjects and practices [1]. In a larger sense, the study of how things change with time and of the forces that cause them to do so is the objective of dynamic investigation [2]. The function of tribological research is to bring about a reduction in the friction and wear of mechanical components and protect them against failure and provide long, reliable life by selection of materials, operating parameters and lubricants. Mechanical systems such as bearings, seals and gears are the examples of components that are investigated. Whenever two or more solid surfaces are in contact with relative motion between the surfaces, tribology is involved [1]. The bodies in relative motion may be gas and solid (aerodynamic friction), or liquid and solid (lubrication friction) or a solid and solid (dry or solid friction) or the friction may be due to the internal heat dissipation process (internal friction) [3].

2. Friction

The first part in tribology is friction which can be defined as the force resisting the relative motion of two or more surfaces in contact or a surface in contact with fluid. Two of the most significant inventions of early man are friction related. Use of frictional heating to cooking, and discovered that rolling friction is much lower than that of sliding friction. The second discovery led to the invention of the wheel. Friction plays an important role not only in the daily life activities but also in the field of industrial applications. It aids in starting the motion of a body and changing its direction and subsequently stopping it, if there is no friction we
could not move and grip objects. Without the presence of friction rolling mills could not operate, most threaded joints would not hold, friction welding would not exist neither the sound of the violin nor the squeal sound of brake [3].

In moving machinery friction is responsible for dissipation and loss of much energy. For example, it has been estimated that 10% of oil consumption in the United States used simply to overcome friction. The energy loss caused by friction requires an energy input in order to maintain the sliding motion. The energy dissipated from the system is primarily in the form of heat which may have to be removed by cooling to avoid the damage of machinery. Not all of the energy will dissipate as heat but some of the energy dissipates in the form of various deformation processes, which results in the wear of the material where the contact has been occurred and resulting in their eventual degradation of point where replacement of whole component may be essential [3].

The fundamental experimental laws that govern friction of solid bodies are quite simple and are usually named after Coulomb, who formulated them in 1875 (much of this work was built on earlier work by Leonardo da Vinci and Amontons). The laws stated by Coulomb are in very general terms [3]:

- Static friction may be greater than kinetic or dynamic friction
- Friction force is proportional to applied load
- Friction is independent of sliding velocity
- Friction force is independent of contact area

Even though these are very simple and applicable in many instances; there are also numerous conditions under which they may break. Friction is commonly represented by friction coefficient ($\mu$). The friction coefficient is a dimensionless scalar value which is the ratio between the friction force, $F$, and the load, $N$. The frictional force is always normal to the applied load or force [3].

$$\text{Coefficient of friction } \mu = \frac{F}{N}$$

The friction coefficient typically ranges from 0.03 for a very well lubricated bearing, to 0.5 to 0.7 for dry sliding and even $\geq 5$ for clean metal surface in vacuum. The coefficient of friction depends on whether the objects are initially moving or stationary and on the types of materials involved. For flat surfaces the normal force is equal to the weight of the object, for inclined surfaces only a fraction of the object’s weight pushes directly into the surface, so the normal force is less than the object’s weight. Different kinds of relative motion between objects give rise to different types of friction between them which are [3]:

2
- Static friction
- Sliding or kinetic friction
- Rolling friction and
- Fluid friction

Static friction occurs between stationary objects. The coefficient of friction for two materials may differ depending on the type of friction involved. Static friction prevents an object from moving relative another surface. It is the force that keeps an object from sliding off on a surface, even when the surface is slightly tilted. The static friction is time dependent, the length of the rest time will affect the static friction. It can either increase or decrease with rest time. In order to move something, you must first overcome the force of static friction between the object and surface on which it is resting. This force depends on the coefficient of static friction between the object, surface and the normal force of the object (\(\mu_s\)) [4].

An object sliding on a surface or brakes slowing down the wheel are both examples of sliding or kinetic friction. Sliding friction acts in the direction opposite to the direction of motion. When sliding friction is acting another force must be present to keep an object in motion. In the case of object sliding over a surface, this force is gravity. The force of sliding or kinetic friction depends upon the coefficient of kinetic friction between the object and the surface on which it is moving (\(\mu_k\)) [4].

Rolling friction hinders the motion of an object rolling along a surface. Rolling friction slows down a ball rolling on a surface, and it slows down the motion of tire rolling along the ground. Rolling friction is caused by energy dissipation inside the material, also called hysteresis. The coefficient of rolling friction is 1/100 that of sliding friction [4].

Objects moving through a fluid experiences fluid friction. It acts between the object and fluid and hinders the motion of the object. The force of fluid friction depends upon the object’s shape, material, speed and as well as the fluid’s viscosity [4].

3. Wear

The second part of tribology, wear, can be defined as the loss of material from a tribological surface. However, this does not mean that material is lost from the tribo–system since material can be transferred from one surface to another. In most cases wear is detrimental for a system and will reduce its efficiency or even lead to complete failure. Wear, just like friction, is not a material property, it is a system response. Wear can be reduced by proper selection of materials, lubricants and operating conditions. In some systems, there can be components that are designed to wear (brake pads). There are several different wear
mechanisms that can occur either alone, more commonly, and simultaneously. Examples of wear mechanisms are [5]:

- Adhesive wear
- Abrasive wear
- Fretting wear
- Surface fatigue and
- Tribochemical or corrosive wear

These are not distinct mechanisms, but rather combinations of the adhesive, chemical and abrasive forms of wear. Most of the industrial wear situations occur because of adhesive and abrasive wear mechanisms. Wear by all mechanisms, except by fatigue mechanism, cause gradual removal of material.

3.1 Adhesive wear

Adhesive wear occurs when two nominally flat solid bodies are in sliding contact, whether lubricated or not. Adhesion (or bonding) occurs at the asperity contacts at the interface and these contacts are sheared by sliding, which may results in detachment of a fragment from one surface and attachment to the other surface. [1]. Adhesive wear mechanism is schematically shown in Fig 1.

![Fig 1. Model of deformation in adhesive asperity contact](image)

As the sliding continues, the transferred fragments may come off the surface to which they are transferred and be transferred back to the original surface, or be ejected as loose wear particles. The formation of loose particle often results from chemical changes in the fragment. The fragments have large surface area and tend to oxidised readily, which reduces adhesive strength, and readily break loose. In some cases the formation of loose particles involves the residual elastic energy of adherent fragments [1].
3.2 Abrasive wear

Abrasive wear occurs when asperities of a rough, hard surface or hard particles slide on a softer surface and damage the interface by plastic deformation or fracture. In the case of ductile materials with high fracture toughness, hard particles or hard asperities results in plastic flow of softer material. In the case of brittle materials, wear occurs by brittle fracture. In these cases the worn zone consists of significant cracking [1]. The rate at which the surface abrades depends on the characteristics of material surface, presence of abrasive particles, the speed and other environmental conditions. Abrasion is typically categorized according to types of contact, as well as contact environment. Types of contacts include two-body and three-body wear. The former occurs when an abrasive slides along a surface, and the later, when an abrasive is caught between one surface and another [3]. Fig 2 shows the schematic representation of two body and three body wear mechanisms.

Fig 2. Two and three body abrasive wear mechanism [8]

Several mechanisms have been proposed to explain how material is removed from a surface during abrasion. These mechanisms include fracture, fatigue and melting. Because of the complexity of abrasion, no mechanism completely accounts for all the loss. Fig 3 depicts some of the processes that are possible when a single abrasive tip transverses a surface. They include plowing, wedge formation, cutting, microfatigue and micro cracking [3].
Plowing is the process of displacing material from a groove to the sides. This occurs under light loads and does not result in any real material loss. When the ratio of shear strength of the contact interface relative to the shear strength of the bulk rises to a high level, it has been observed that a wedge can develop on the front of abrasive tip. The most severe form of wear for ductile material is cutting. During cutting process the abrasive tip removes a chip, results in the removal of material [3].

![Fig 3. Five processes of abrasive wear](image)

(a) Plowing  (b) Wedge  (c) Cutting  (d) Microfatigue  (e) Microcracking

### 3.3 Fretting wear

Fretting is the small amplitude oscillatory movement that may occur between contacting surfaces, which are usually at rest. One of the immediate consequences of the process in normal atmospheric conditions is the production of oxide debris hence the term “fretting wear” or “fretting corrosion” is applied. The movement is usually because of the external vibration, but in many cases it is a consequence of one of the members being in contact subjected to cyclic stress which gives rise to early fatigue cracks. This is termed as “fretting fatigue” or “contact fatigue”. There are some cases where the movement is not simply tangential, but is complicated by the normal force also oscillating to the extent that the surface loses contact in each cycle. This leads to hammering effect and termed as impact fatigue [3].

Fretting wear is affected by different physical variables such as amplitude of slip, normal load, frequency of vibration and the circumstances of the fretting situation, such as type of contact, mode of vibration and the condition of surfaces. It is generally agreed that
when fretting wear is takes place between two flat surfaces or cylindrical surfaces, the progress with time can be observed in three stages

- In the initial stage when metal-to-metal contact will occur which results in local welding, roughening of the surface, high friction and low contact resistance. If the movement is cyclic, fatigue cracks are initiated in this stage.
- In the second stage, reduction of the coefficient of friction will occur because of the formation of beds of compacted oxide.
- Onset of steady state in which the friction is more or less constant and the contacting resistance is generally high with occasional momentary falls to a low value.

Fretting wear can be controlled or prevented by improved design, surface finish, coatings, inserts and lubrication [3].

### 3.4 Surface fatigue

Surface fatigue seems to be a simpler wear mechanism since it appears to be based only on the action of stress on the surface without needing a physical solid contact of the surfaces under consideration. The effect of surface fatigue wear process is normally associated with repeated stress cycling in rolling or sliding contact. In fatigue, failure in the material arises because of stress-reverse effects [2]. Adhesive and abrasive wear mechanism are operative during direct contact between two surfaces moving relative to each other, if the surfaces are separated by fluid film these wear mechanisms do not operate. However, in an interface the contacting stresses are very high and surface fatigue wear mechanism can be operative. The maximum compressive stresses occur at the surface, but the maximum shear stresses occur at some distance below the surface. Time to surface fatigue failure depends upon the amplitude of the reversed shear stresses, the interface lubrication condition and the fatigue properties of the material [1].

### 3.5 Tribochemical or corrosive wear

Tribochemical or corrosive wear occurs when sliding takes place in a corrosive environment. In air, the most dominant corrosive medium is oxygen. Friction modifies the kinetics of chemical reactions of sliding bodies with each other, and with the gaseous or liquid environment, to the extent that reactions which occur at high temperatures also occur at moderate and ambient temperatures during sliding. Chemistry dealing with this modification of chemical reaction by friction or mechanical energy is referred as tribochemistry, and the wear controlled by this reaction is referred as tribochemical wear. The combined effect of
friction and chemical reaction can result in total material losses that are much greater than the additive process taken alone [1].

In the absence of sliding, the chemical products forms a protective layer of less than a micrometer thick on the surface, which would tend to slow down or even arrest the corrosion, but the sliding action wears the chemical film away, so that the chemical attack continues [1].

4. Lubrication

Finally, the third part of tribology is lubrication, a method to reduce friction and wear between two moving surfaces. This is achieved by introducing an easily sheared material (liquid, solid or gas) between two surfaces. These sheared materials are called lubricants, reduces friction and wear at the interface and carry away the heat and debris generated during the sliding process. Depending on the lubricant film thickness, the interfacial height and the degree of geometric conformity, different lubrication regimes can be distinguished [1]. The lubrication regime is usually determined by using Strubeck curve which is shown schematically in Fig 4.

There are three lubrication regimes; boundary lubrication (BL) where the load is carried by the asperities and friction is governed by the surface films that are formed. In mixed lubrication (ML) a hydrodynamic pressure starts to build up and the load is partially carried out by the oil film. In full film lubrication (FL) the two surfaces are completely separated and the entire load is carried by the lubricant film [5].

As previously mentioned, lubricants may be in the form of liquid, solid and gaseous. The choice of an appropriate lubricant depends on system needs, cost, safety and environmental conditions. Minimizing the number of lubricants used generally reduces the
maintenance cost. Liquid lubricants contain different additives which are added to the base oil to improve certain properties such as load carrying capacity, viscosity or oxidation behaviour. The lubricant can also be solid material with a lamellar structure that allows easy shearing (graphite, molybdenum disulphide or boric acid). Today’s advanced engineering technologies allow for preparation of multilayer surface coatings with built in layer with friction reducing properties [5].

5. High temperature Tribology

High temperature tribological studies (typically above 300 ºC) play an important role in the technological applications where the components operate at elevated temperatures. Examples of such applications can be found in aerospace, power generation and metal working industries. In such applications, lubrication by conventional means (liquid lubricants) is not possible because of the rapid decomposition and deterioration of the lubricants. To overcome these problems, solid lubricants are used which reduce the friction and wear. The behaviour of all tribochemical layers will not have same effect; it will depend on their properties. As in the case with the most other metals, steels are thermodynamically unstable in air, and form oxide layers which may protect or adversely affect the surfaces depending upon the nature of oxides formed.

Alloying elements play important role in the formation of oxide layers. Fontalvo et al. [6] studied the effect of aluminium and silicon on the friction and wear behaviour of hot work steel. They concluded that the aluminium and silicon yield a reduction of oxide film thickness thus lead to an increase in wear as temperature is increased.

Tribological behaviour at elevated temperatures in reciprocating sliding condition was investigated by Glascott et al. [7]. They concluded that at 300 ºC the oxide layers are brittle, resulting in higher friction and wear. Oxide layers at 600 ºC are smooth and protecting the surface, resulting in lower friction and wear.

Stott et al. [8] studied the effect of load and hardness on friction and wear behaviour of different dissimilar combinations of pin and disc specimens in reciprocating sliding condition at 500 – 600 ºC and revealed that the temperature and load will have significant effect on the tribological behaviour of material. At lower loads, the protective layers are able to sustain longer durations than at higher loads. In high speed steel and carbon steel combinations at 500 ºC, the wear mechanism is abrasion of the pin as hard oxide debris were observed. Loose debris cannot be retained in between the contacting surfaces and formed shallow grooves and cause high wear. But in the case of soft material (high- chrome
steel) wear grooves are deeper and facilitated the entrapment of wear debris leading to development of wear protective glaze.

6. Effect of surface quality on tribological properties

Surface topography of the material has a significant effect on the tribological properties of a material. Roughness is a measure of the texture of a surface. It is quantified by the vertical deviations of the real surface. If the deviations are large the surface is said to be rough and if the deviations are small, surface is smooth. Rough surfaces wear more quickly and have high friction.

Table 1. Commonly used height parameters [7]

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Formula</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>Roughness average (CLA or Ra)</td>
<td>$R_a = \frac{1}{L} \int_0^L</td>
<td>z</td>
</tr>
<tr>
<td>Root mean square roughness (RMS or Rq)</td>
<td>$R_q = \sqrt{\frac{1}{L} \int_0^L z^2 , dx}$</td>
<td>Average separation of the five highest peaks and the five lowest valleys within the sampling length: $R_q = \frac{p_1 + \ldots + p_5 + v_1 + \ldots + v_5}{5}$</td>
</tr>
</tbody>
</table>

Table 1 shows the commonly used height parameters. The typical amplitudes between the peaks and valleys for engineering surfaces are about one micrometer. Almost every known surface, apart from the cleaved faces of mica, is rough. Height characteristics are commonly described by parameters such as centre-line average or roughness average (CLA or Ra), root mean square roughness (RMS or Rq), mean value of the maximum peak-to-valley height...
(Rtm), ten-point height (Rz) and many others. In engineering practice, however, the most commonly used parameter is the roughness average [9].

7. Surface treatments

Surface treatments of materials become a vital factor for improving the properties of a component, and/or extending its lifetime. Surface treated materials can have one or several functions. For example, the application of paint can have both an anticorrosion and decorative effect. The surface treatments are widely used for the ferrous based materials. The process of surface treatments tailors the surface of engineering materials to

- Control friction and wear
- Improve corrosion resistance
- Change physical properties (e.g. conductivity, resistivity and reflection)
- Vary appearance
- Reduce cost.

Surface treatment processes may be classified into two main categories, microstructural treatments and diffusion treatments.

7.1 Microstructural treatments

In most microstructural treatments, a thin surface shell of ferrous metal (steels and cast irons) is case hardened by localized surface heating and quick quenching to produce hard, wear resistance martensitic structure with a tough, ductile core. The fatigue strength is increased because the treated surface has compressive residual stresses. Two heat treatment techniques are being widely used, induction heating and flame hardening. In addition to these two, laser and electron beam hardening also are becoming popular. In addition to these techniques a few old techniques are also in use such as chill casting and work hardening [10].

7.2 Diffusion treatments

At elevated temperatures chemical species can be diffused into most iron and steel alloys at significant rates. This fact is taken advantage of by exposing heated ferrous parts to an appropriate medium, which may be solid, liquid, gas or plasma. The diffusion treatments involve atom by atom transportation from medium to the part, thus modifying the surface chemistry of the part by creating a new alloy composition. A variety of diffusion treatments will increase the hardness of metals, particularly steel and iron alloys. Surface treatments such as carburizing, carbonitriding, nitriding, nitro-carburizing, boriding, ion implantation and ion
beam milling are the most prominent for tribological applications. Other techniques which are also used include chromizing, aluminizing, siliconizing and shearardizing [10].

7.3 Nitriding
Among all of the other surface treatments methods which are applied to improving the tribological properties, nitriding is one of the most commonly used methods applied for steel and its alloys, especially for tool steel to improve the hardness and surface wear resistance at elevated temperatures. Nitriding treatments are ferritic surface hardening heat treatment that introduces nitrogen atoms into the surface of metal at a temperature range of 500 to 590 ºC where the metal is in ferritic phase. The nitrogen is introduced in the surface of the steel by reaction with a solid phase, gas or plasma containing nitrogen. The processing time is rather long (100 hours at 500 ºC to obtain a depth of 0.5 mm) when compared to carburizing. This process does not need quenching which minimise the distortion and a good surface finish is possible. Nitrogen is most effective when the steel contains Al, Cr, Mo, V, and M, these elements from stable nitrides. All hardenable steels must be hardened and tempered before being nitrided. Process methods for nitriding are
- Gas (box furnace)
- Liquid
- Nitro carburizing and
- Plasma nitriding.

Gas nitriding is a case hardening process in which nitrogen is introduced into the surface by exposing it to the nitrogenous gas, mostly Ammonia at particular temperature (quenching is not required).

Liquid nitriding (salt bath) is a generic term for number of different salt processes. It is a subcritical case hardening process which contains either cyanides or cyanates. The metal is immersed in salt bath at a subcritical temperature. The surface properties may vary with the chemical control and the process used.

Plasma (ion) nitriding is an extension of conventional nitriding process in combination with plasma. Fig 5 shows the schematic representation of ion - electron discharged mechanism. It is a low temperature and low distortion surface treatment process. This process is performed in vacuum, where a high electrical energy is used to form plasma. The furnace will act as an Anode and the specimen will act as Cathode. The high energy current mainly consists of two factors, the negatively charged ions move towards the anode.
and positively charged ions will move towards the cathode. The nitrogen atoms are positively charged and accelerated towards the negative electrode and hit the surface with a high kinetic energy due to which the specimens are heated to high temperature and the nitrogen ions diffuse easily into the surface and form the compound as Fe$_2$N or Fe$_4$N.

![Fig 5. Ion - electron discharged mechanism [14].](image)

Plasma nitriding is particularly suitable for surface modification of low alloy steels as hot working and gearing steels. The superior property imparted by plasma nitrided stainless steel, hot and cold rolled steels is good wear resistance [11].

*Nitro carburizing* is modified form of nitriding which involves the introduction of nitrogen and carbon (small amount of sulphur). This process will be performed only in the ferritic region of steels. The operating temperature is in the range of 540 to 600 °C. The amount of nitrogen diffused is very high compared to the carbon. Nitrocarburized steel surfaces offer good resistance to scuffing, improved fatigue properties and corrosion resistance.
II. Objectives

The main objectives of this thesis are

➢ To experimentally investigate the friction and wear behaviour of different tool steels during sliding against high strength boron steels at room temperature and at high temperature.

➢ To understand the various mechanisms involved in the friction and wear process of tool steels and high strength boron steel.

➢ To study the plasma nitrided and post oxidised samples at different temperatures.

The improved knowledge of high temperature tribological behaviour of these materials will enable in proper selection of materials and surface treatments.
III. Experimental approach

The experimental work performed in this thesis has been carried out by using standard tribological test equipments. It is a systematic approach to investigate the different steel specimen performance against high strength boron steel at room and high temperatures.

1. Experimental materials

The studies presented in this thesis involved three different compositions of tool steels.

1. Tool steel 1 (TS 1)
2. Tool steel 2 (TS 2)
3. Tool steel 3 (TS 3)

The tool steels specimens have been studied in this thesis work are in nitrided and post oxidised condition. Tool steel 1 and tool steel 2 were post oxidised at 500 °C whereas, tool steel 3 was post oxidised three different temperatures i.e. post oxidation 1 at 500 °C, post oxidation 2 at 520 °C and post oxidation 3 at 480 °C respectively.

The high strength boron steel is a commercially available material and it was hardened through quenching and the surface was then ground and polished to a low surface roughness. The chemical composition of both the tool steel specimens and boron steel disc are presented in Table 2.

Table 2. Compositions (wt %) of the materials employed in these studies

<table>
<thead>
<tr>
<th>Material</th>
<th>C</th>
<th>Mn</th>
<th>Cr</th>
<th>Si</th>
<th>B</th>
<th>P</th>
<th>S</th>
<th>Ni</th>
<th>Mo</th>
<th>V</th>
</tr>
</thead>
<tbody>
<tr>
<td>Boron steel</td>
<td>0.25</td>
<td>1.0</td>
<td>1.3</td>
<td>0.14</td>
<td>0.26</td>
<td>0.2</td>
<td>0.005</td>
<td>&gt;0.03</td>
<td>&gt;0.01</td>
<td>-</td>
</tr>
<tr>
<td>Tool steel 1</td>
<td>0.37</td>
<td>1.4</td>
<td>2.0</td>
<td>0.3</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>1.0</td>
<td>0.2</td>
<td>-</td>
</tr>
<tr>
<td>Tool steel 2</td>
<td>0.31</td>
<td>0.9</td>
<td>1.35</td>
<td>0.6</td>
<td>-</td>
<td>- max 100 ppm</td>
<td>- max 40 ppm</td>
<td>0.7</td>
<td>0.8</td>
<td>0.145</td>
</tr>
<tr>
<td>Tool steel 3</td>
<td>0.39</td>
<td>0.4</td>
<td>5.2</td>
<td>1.0</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>1.4</td>
<td>0.9</td>
<td>-</td>
</tr>
</tbody>
</table>

2. Surface treatments and specimen geometry

The specimens were nitrided through patented processes known as Corr- I- Dur® which is a combination of various chemical process steps involving plasma nitriding, gas nitrocarburizing and oxidation. The depth of nitrided layers was between 0.25 - 0.3 mm and the duration of the process was 240 minutes (4 hours). The specimens were post-oxidised at
different temperatures with a view to obtain different friction and properties. The post-oxidation was a part of the plasma nitriding process.

The upper specimens are the tool steel specimens of pin shape (diameter 10 mm x 10 mm long) with one end spherical and the lower specimen is flat disc made from high strength boron steels (diameter 24 mm x 7.9 mm thickness). See Fig 6.

**Fig 6. Schematic of the test specimens used in these studies**

3. **Experimental equipment**

3.1 **Pin on disc machine**

The TE-67 Pin on disc machine consist of a pin carrier assembly and disc carrier assembly is held fixed by the base frame in order to maintain the alignment between the specimens to enable usage of flat-on-flat geometry. Fig 7 shows the Pin on disc machine set up for room temperature and high temperature. The test disc is secured by a central bolt on to the disc carries spindle. The spindle is driven through a pulley belt by the D.C. motor beneath the machine. The motor has tachogenerator feed back to ensure stable running speeds. The speed range can be changed by adjusting the wear track radius the test. The scale rule is used to set the radius to the middle of the pin.

The pin specimen has 8 mm mounting diameter so that it can be clamped in the collet chuck. The pin assembly is pivoted to the frame so that it will move up and down. The strain gauge transducer mounted at the rear end of the pin carrier head measures the friction force exerted on the pin during the rotation. Two types of force transducers are available, for low load 5 kg and for high loads 100 kg.
Fig 7. Pin on disc machine setup. (a) Room temperature. (b) High temperature

The standard configuration allows test at room temperature. Temperature is measured using a simple trailing thermocouple running on the disc surface. For high temperature (max 400 ºC) applications an external air blower is used. At high temperatures the trailing thermocouple and optical pyrometer are used to record temperature. The pneumatic load actuator can handle steady and fluctuating loads with sine or triangular shaped waves. A linear potentiometer measures the movement of the pin piston and can provide in-situ wear measurements. Both dry and lubricated situations are possible. For the lubricated situation there is an enclosure to submerge the disc, which features two electrical resistance heating elements to control temperature. Table 3 refers to the technical specifications of pin on disc machine.

Table 3. Technical specification of Pin on disc machine

<table>
<thead>
<tr>
<th>Speed</th>
<th>30 - 1000 rpm, 60 - 2000 rpm</th>
</tr>
</thead>
<tbody>
<tr>
<td>Load</td>
<td>Pneumatic 25 - 1000N, Dead weight 5 - 100 N</td>
</tr>
<tr>
<td>Friction force</td>
<td>0 – 50 N, 0 - 1000 N</td>
</tr>
<tr>
<td>Wear measurements</td>
<td>Linear potentiometer 0 – 2.5 mm &lt; 2µm</td>
</tr>
<tr>
<td>Contact potential</td>
<td>40 mV dc signal</td>
</tr>
</tbody>
</table>

3.2 Wyko 1100 NT 3D optical profiler

Technical specifications of Wyko 1100 NT optical profiler are shown in table 4. The Wyko 1100 NT optical profiler uses interferometry to measure a surface. It has two main features, Vertical Scanning Interferometry (VSI) and Phase Shifting Interferometry (PSI). In VSI mode, it utilizes the white light interferometry which is a bright and dark. The bright pattern originates from the smooth reference surface and the dark pattern from the test specimen. The
pattern will be photographed by a CCD camera and transferred to the computer. The PSI mode is similar to the VSI but, it uses filtered light and the reference surface is translated instead of the objective. It also uses the difference in intensity instead of fringes for height data acquisition. The acquired data can be generated as a 2D and 3D mode for analysis.

Table 4. Technical specifications of Wyko 1100 NT optical profiler

<table>
<thead>
<tr>
<th>Magnification</th>
<th>1.25 X – 100 X</th>
</tr>
</thead>
<tbody>
<tr>
<td>Vertical resolution (VSI)</td>
<td>&lt; 1nm</td>
</tr>
<tr>
<td>Vertical resolution (PSI)</td>
<td>1 Å</td>
</tr>
</tbody>
</table>

3.3 Scanning Electron Microscope (SEM)

The SEM uses an electron beam instead of light to form an image. A beam of electrons produced at the top of the microscope by the electron gun passes through a vertical vacuum chamber. The electron beam passes through electromagnetic coils and lenses which focus the beam on to the specimen. When beam hits the sample it produces x-rays and electrons (primary back scattered, secondary and auger). Detector collects these X-rays, secondary and back scattered electrons and converted them into use full form of signals and sent to the display screen. Fig 8 shows the schematic representation of Scanning electron Microscope (SEM).

![Fig 8. Schematic representation of Scanning Electron Microscope (SEM)](image)

The SEM has many advantages over traditional microscopes. It has a large depth of field, which allows larger area of interaction with electron beam, higher resolution, so closely spaced specimens can be magnified at much higher levels. Much more control on degree of magnification, different imaging modes (secondary electron imaging and back scattered...
electron imaging) and SEM images are much easier to interpret than Transmission Electron Microscope (TEM) images.

Energy Dispersive Spectroscopy (EDS) is a technique used to qualitative elemental analysis or chemical composition of a sample. The generation of X-rays due to bombardment of electrons on to the sample are used for EDS. The X-rays are detected by the EDS detector and sent to analysis. However some limitations are considered such as detection of light elements, the detector should be kept at 77K all the time to reduce the noise in the system.

The *JSM 6460* is a low vacuum (1-270 Pa range) SEM with a conventional tungsten filament electron source that is equipped for both imaging and micro analysis in low vacuum and high vacuum. In high vacuum mode, SEM capable of giving secondary electron images of resolution up to 3nm at the maximum acceleration voltage (30 Kv). The tungsten filament will produce a strong extremely stable electron source with high beam current for back scattered imaging. The magnification ranges from 5 to 300000 X and the accelerating voltage ranges from 0-30Kv.

The microscope is equipped with Energy Dispersive Spectrometer (EDS) capable of chemical analysis with a detectable range of Carbon (C) – Uranium (U). An Electron Back-Scattered Diffraction (EBSD) camera is also available with mapping software that can provide crystallographic information such as pole figures, grain boundary orientation and crystal orientation.

### 3.4 X-ray diffraction

X-ray power diffraction is an analytical tool used for identification of phases in crystalline materials and provides information about unit cell dimensions. X-ray diffraction is based on constructive interference of monochromatic X-rays and crystalline sample. X-rays are produced by cathode ray tube, filtered for monochromatic rays, collimated to concentrate and directed towards the sample. The interaction of incident rays with the sample produces constructive interference when it satisfies the Bragg’s law \( n\lambda = 2d \sin \theta \) as shown in the Fig 9. This relates the wavelength of the electromagnetic radiation and diffraction angle and lattice spacing. When the incident electrons will have sufficient energy to dislodge inner shell electrons of the targeted material, X-ray spectra are produced.
The Philips X’pert MRD X-ray diffractometer is a multipurpose diffractometer consisting of Cu long five focus electrode. The generated X-rays of energy 45Kv tension and 40 ma current. The 2\(\theta\) values are ranging from -9.3475º to 157.6525º; Theta values are from -4.6234º to 176.3766º and can only operate at room temperature.

### 3.5 Micro hardness tester

The term micro hardness usually refers to static indentations made with a load of few grams to 1kgf. The indenter is usually either Vickers diamond indenter or Knoop elongated diamond indenter. The procedure for measuring is similar to the macro Vickers test, except that the test carried out in the microscale under microscope with high precision. The test sample requires good surface metallographic finish (lower the load higher the surface finish).

The Mxt-ox- microhardness tester consists of both Vickers and knoops diamond pyramids with load ranging from 10 grams to 1000 grams. It consists of a CCD camera to view the indentation and is connected to a computer to analyze the indentation.

A semi-microelectronic weighing balance is used for weighing the pin specimens. The weighing balancer can be used to weigh a maximum of 220 grams with a precision of 0.00001 grams.

### 4. Test parameters

The test parameters for all the experiments are for constant sliding distance. The wear track radius and rotational speed is different for the samples. Tests at room temperature and high temperature (400 ºC) are carried out with constant load of 4.905 N. The test parameters for tests at room temperature and high temperature are shown in table 5 and table 6 respectively.
Note: High temperature tests are performed at 400 °C with 4.905 N loads. Two tests are performed on each disc with different wear track radius, speed and time with two different specimens of same composition.

Table 5. Test parameters for tests at room temperature

<table>
<thead>
<tr>
<th>Test specimen (pin)</th>
<th>Wear track radius (mm)</th>
<th>Rotational speed (rpm)</th>
<th>Time (sec)</th>
</tr>
</thead>
<tbody>
<tr>
<td>TS3 (Post oxidation at 500 °C)</td>
<td>16</td>
<td>104</td>
<td>1211</td>
</tr>
<tr>
<td>TS3 (Post oxidation at 480 °C)</td>
<td>19</td>
<td>88</td>
<td>1205</td>
</tr>
<tr>
<td>TS3 (Post oxidation at 520 °C)</td>
<td>22</td>
<td>76</td>
<td>1205</td>
</tr>
<tr>
<td>TS1 (Post oxidation at 500 °C)</td>
<td>25</td>
<td>67</td>
<td>1203</td>
</tr>
<tr>
<td>TS2 (Post oxidation at 500 °C)</td>
<td>28</td>
<td>60</td>
<td>1200</td>
</tr>
</tbody>
</table>

Table 6. Test parameters for high temperature tests

<table>
<thead>
<tr>
<th>Test specimens (Pin)</th>
<th>Wear track radius (mm)</th>
<th>Rotational speed (rpm)</th>
<th>Time (sec)</th>
</tr>
</thead>
<tbody>
<tr>
<td>TS3 (Post oxidation at 500 °C)</td>
<td>16 and 22</td>
<td>104 and 76</td>
<td>1211 and 1205</td>
</tr>
<tr>
<td>TS3 (Post oxidation at 480 °C)</td>
<td>16 and 22</td>
<td>104 and 76</td>
<td>1211 and 1205</td>
</tr>
<tr>
<td>TS3 (Post oxidation at 520 °C)</td>
<td>16 and 22</td>
<td>104 and 76</td>
<td>1211 and 1205</td>
</tr>
</tbody>
</table>

5. Test procedure

We studied the tribological properties of five different tool steel specimens sliding against a high strength boron steel disc. The tests are performed by standard test equipments at room temperature and at high temperature (400 °C) with dry friction (in the absence of lubrication). All the test specimens’ surfaces are characterized by Wyko NT 1100 Topometer and the topographical properties are measured. The phases present in the tool steel specimens are measured with X-ray diffraction. Test procedure employed in this thesis work mainly consists of four steps, cleaning, weighing, testing and surface analysis.
The test specimens and holders were cleaned using ultrasonic cleaner in petroleum spirit and the specimens were then rinsed with ethanol in order to remove all contaminants. All test specimens are then weighed by using a semi-microelectronic weighing balance. All samples are weighed three times to reduce the errors and the average value is considered as final value. For tests at room temperature the pin on disc machine is calibrated and suitable parameters (speed and time) are calculated for each test, all tests are performed with constant sliding distance and with a load of 4.905 N.

After completion of each test, the test specimens are cleaned again and weighed to determine the weight gained or loss to the specimens and to calculate the volume loss of specimens. The surface morphology is studied by SEM and Topometer and the chemical composition of pin specimens and disc wear scars (after the test) are studied with the help of EDS.

For high temperature tests, the test parameters have been listed in Table 6. In high temperature tests (see Fig 10), first the heating sequence will run where the lower test specimen (Boron steel disc) will be heated to 400 °C with the help of an external blower and the temperature is measured with the help of thermo couple and optical pyrometer. As the disc specimen reached to the desired temperature, the pin specimen will be brought in to contact immediately, loaded against the disc and the test was started. Upon completion of test the specimens are allowed to cool to room temperature and the same cleaning, weighing and surface analysis are performed as mentioned above.

![Fig 10. Schematic representation of the high temperature test](image)

For the second test at high temperature the same discs which have been used for the first high temperature test are used with the same specimen geometry but with different wear track radius (22 mm). After completion of the test the same cleaning, weighing and surface analysis are performed.
IV. Results and discussion

1. Surface characteristics of the unworn specimens

All the test samples surface characterization was done by topometer. The untreated disc specimen (high strength boron steel) surface roughness was measured and ranges from 0.35 to 0.46 µm and for the tool steel pin specimens ranges from 0.65 to 1.35 µm. All the tests are carried out with similar roughness values for all pin specimens. Fig 11 shows the surface topography of disc and pin samples. Tool steel specimens curvature and tilt effects are eliminated by using algorithm with the help of software in order to get good surface characterization results.
The tool steel specimens are tested with the help of X-ray diffraction in order to know the different phases present in the specimens. The results shown that different quantity of oxide formation elements are present in all tool steel specimens which are post oxidised at different temperatures. Fig 12 shows X-rd spectra of tool steel 3 which is post oxidised at different temperatures.
Fig 12. X-ray spectra for tool steel specimens (a) Tool steel 1 post oxidation 1 (b) Tool steel 2 post oxidation 1 (c) Tool steel 3 post oxidation 1 (d) Tool steel 3 post oxidation 2 (e) Tool steel 3 post oxidation 3.
2. Hardness measurements

The surface hardness of boron steel disc and tool steel pin specimens also play an important role in the friction results, wear mechanisms and amount of wear. Table 7 refers to the hardness measurements of test samples. The measured hardness values are different for all the specimens.

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Vickers hardness (HV)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Boron steel disc</td>
<td>647</td>
</tr>
<tr>
<td>Tool steel 1- Post oxidation at 500 °C</td>
<td>590</td>
</tr>
<tr>
<td>Tool steel 2- Post oxidation at 500 °C</td>
<td>689</td>
</tr>
<tr>
<td>Tool steel 3- Post oxidation 1 at 500 °C</td>
<td>648</td>
</tr>
<tr>
<td>Tool steel 3- Post oxidation 2 at 520 °C</td>
<td>710</td>
</tr>
<tr>
<td>Tool steel 3- Post oxidation 3 at 480 °C</td>
<td>605</td>
</tr>
</tbody>
</table>

*Note: Hardness values are the average values for all the specimens at 25 grams load.*
3. Friction results

3.1 Frictional behaviour at room temperature

In our studies at room temperature the friction coefficient of pairs has been measured for constant rotational distance (211.1 m). Constant sliding distance was maintained by varying the speed, wear track radius and the rotating time of disc.

![Friction coefficient of tool steel 3 - post oxidation 1, 2 and 3 sliding against high strength boron steel disc at room temperature](image)

The results have shown that the coefficient of friction for tool steel 3 post oxidation at 500 ºC is lower, post oxidation at 520 ºC is moderate and post oxidation at 480 ºC is higher. The initial frictional behaviour for post oxidation 1 is different when compared to post oxidation 2 and 3 but the friction behaviour is similar and steady (refer Fig 13). The higher friction coefficient in the beginning of the test is because of the hardness differences of three samples.

From X-ray diffraction it has been shown that different amount of iron oxide (Fe$_3$O$_4$) is present in the three samples. In post oxidation 1, it is higher; in post oxidation 2 moderate and in post oxidation 3 the least. These results correlate to the friction coefficient, as higher amount of iron oxides on the tool steel surface will reduce the adhesion and provide a lower coefficient of friction. The flash temperature in between the pin and disc specimens is enough to produce the compact oxide layers which reduce friction.
Fig 14 shows the friction coefficient for tool steel 1 and tool steel 2 post oxidised at 500 °C. From the results it can be observed that the initial friction is higher and similar, this may be due to the hardness difference in the specimens when compared to the tool steel 3 post oxidation 1 specimen. The frictional behaviour is similar for tool steel 1 and 2 and the test results show higher and unsteady friction coefficient. X-ray diffraction shows lower amount of iron oxides forming elements when compared to tool steel 3 samples, which cause higher friction due to the less oxides on tool steel specimens. The unsteady and higher frictional behaviour may be attributed to the fact that more force was needed for plastic deformation of the steel disc.

The friction coefficient and wear mechanisms are different for both tool steel 3 post oxidation 1, 2, 3 and tool steel 1 & tool steel 2 post oxidation 1. Tool steel 3 has lower friction and less wear compared to the tool steel 1 and tool steel 2. Tool steel 3 shows initially higher and end up with steady state friction where as the tool steel 1 and tool steel 2 shows higher friction initially but has a more unsteady behaviour. The main reason for this behaviour is the presence of less oxide compact layers during the test in both tool steel 1 and tool steel 2. The presence of more wear debris in tool steel 1 and tool steel 2 will also leads to higher wear of pin and disc.
3.2 Frictional behaviour at 400 ºC

In our studies, we conducted high temperature test with tool steel 3 at different wear track radius on different disc specimens for each set. The test parameters are presented in table 8. In first set, the test was conducted at 16 mm wear track radius and the second test was conducted at 22 mm wear track radius on the same disc as in the first test. Table 8 shows the test combinations of specimens and table 6 shows the test sequence at 400 ºC.

Table 8. High temperature test combinations

<table>
<thead>
<tr>
<th>Disc specimen</th>
<th>Test 1, Wear track radius</th>
<th>Test 2, Wear track radius</th>
</tr>
</thead>
<tbody>
<tr>
<td>Disc 1</td>
<td>Post oxidation -1, 16 mm</td>
<td>Post oxidation -1, 22 mm</td>
</tr>
<tr>
<td>Disc 2</td>
<td>Post oxidation - 2, 16 mm</td>
<td>Post oxidation - 2, 22 mm</td>
</tr>
<tr>
<td>Disc 3</td>
<td>Post oxidation - 3, 16 mm</td>
<td>Post oxidation - 3, 22 mm</td>
</tr>
</tbody>
</table>

3.3 Friction results using 16 mm wear track radius

In this test, the friction coefficient as a function of time is more complicated and the friction coefficient increased to 1.15 ± 0.3. The behaviour of friction coefficient with time is shown in Fig 15.

As seen, post oxidation 1 has moderate friction coefficient where as post oxidation 2 and 3 has higher and lower friction coefficient respectively. Due to the high temperature the wear debris will get oxidised which will cause increase or decrease in frictional behaviour. The frictional behaviour is similar in all the cases which is gradually increasing and reaches to
steady state in the end. The difference in the friction coefficient is because of the hardness difference of the pin samples, post oxidation 1 has moderate and post oxidation 2 and 3 has higher and lower hardness.

Table 9. Weight % of elements present on tool steel 3 after test at 400 °C and 16 mm wear track radius

<table>
<thead>
<tr>
<th>Element</th>
<th>Post oxidation 1</th>
<th>Post oxidation 2</th>
<th>Post oxidation 3</th>
</tr>
</thead>
<tbody>
<tr>
<td>O</td>
<td>0.51</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Si</td>
<td>0.20</td>
<td>0.15</td>
<td>0.16</td>
</tr>
<tr>
<td>V</td>
<td>0.65</td>
<td>1.09</td>
<td>1.25</td>
</tr>
<tr>
<td>Cr</td>
<td>5.41</td>
<td>5.99</td>
<td>5.52</td>
</tr>
<tr>
<td>Fe</td>
<td>93.23</td>
<td>92.53</td>
<td>92.92</td>
</tr>
</tbody>
</table>

From EDS spectra, the oxygen concentration is different for all the three samples wear scars which lead to different friction coefficient values by the formation of oxide layers. Table 9 shows the weight % of elements present on tool steel 3 after test. The oxygen concentration is negligible in both tool steel 3 post oxidation 2 and 3 when compared to post oxidation 1. Higher the oxides lower the friction and vice versa. The high friction coefficient may be the cause of specimen geometry since the harder, spherical shaped pin will indent the softer disc it may cause a lot of ploughing. If we reverse the geometry (pin specimen from high strength boron steel and disc specimen from tool steel) we may get similar friction coefficient as at room temperature.
3.4 Friction results using 22 mm wear track radius

The second test was conducted on the same disc with different wear track radius. Before the test, hardness measurements are done for all the disc specimens and observed drastic decrease in the hardness to 485 HV. This is due to the heating and cooling effect during first test which may changes the microstructure of the disc.

![Graph showing friction coefficients](image)

*Fig16. Friction coefficient of tool steel 3 - post oxidation 1, 2 and 3 at 400 °C sliding against high strength boron steel disc at 22 mm radius*

From the second test we can observe that there is a reduction in the friction coefficient and the frictional behaviour is similar for all the three samples. Fig 16 shows the friction coefficient of tool steel 3 post oxidation 1, 2 and 3. There is no frictional gradient between them and the initial friction is also same. This is because of decrease in the hardness of the disc specimen. There is much time to form thicker oxide layers which will have significant effect on the frictional behaviour. The higher hardness pin specimen can penetrate into the softer disc specimen and causes higher material removal from the disc rather than from the pin. The deformation depends on mechanical properties such as young’s modulus, hardness and the real contact conditions. From EDS analysis the amount of oxygen present on the disc wear scar is same for all the three samples which cause similar frictional behaviour and frictional coefficient.
4. Wear results

4.1 Wear loss and surface damage of specimens at room temperature

Our studies have shown some interesting wear results of both tool steel and boron steel specimens. Fig 17 shows the wear loss of disc and pin specimens of different tool steels. From Fig 17 (a), tool steel 3 post oxidation 1 (500 °C) wear loss is negligible compared to other two and the respective disc wear loss is also lower. This is mainly due to the formation of oxide layers.

Fig 17. Pin and disc volume wear loss (a) Tool steel 3 at room temperature (b) Tool steel 1 and 2
Fig 17 (b) shows wear loss of tool steel 1 and 2, higher volume loss of pin specimens and lower volume loss of disc. The main reason behind it is the formation of fewer oxides during the test which causes higher friction and wear and leads to severe adhesion.

Wear scars of pin specimens and disc specimens have been studied by SEM/EDS and topometer for both room temperature and high temperature tests to get more information about the amount of wear and the wear mechanism generated by sliding.

![Fig18. SEM images of wear scars of tool steel 3 pin (top) and boron steel disc (bottom) specimens at room temperature (a) Post oxidation 1 (b) Post oxidation 2 (c) Post oxidation 3](image)

Fig 18 in combination with Fig 13, the friction behaviour seems to be governed by the hardness and oxide forming elements. From Fig 18 (a), it is clear that there is a negligible wear on the pin specimen and very low wear on the disc. The disc material is adhering to pin specimen. The image shows the presence of polishing grooves in the wear scar of disc specimens which prove that there is very low material loss from the disc. The lower friction is possibly the result of more iron oxides on the tool steel pin surface which reduces the adhesion and thus friction and wear. From EDS the oxygen concentration is high on both tool steel and boron steel specimens and helps to form oxides in large quantities.

From Fig 18 (b), there is more wear on the tool steel and boron steel disc, the boron steel material is adhered on tool steel. The higher friction rates are because of the wear debrrises caused by higher adhesion and causes to higher material removal from tool steel and boron steel. From the disc wear scar, the wear mechanism involved in this process is purely
adhesive; the deep grooves are because of the presence of wear debris in the process which results increase in the friction.

From Fig 18 (c), the post oxidation 3 sample will have higher wear rate when compared to the other two tool steel 3 samples. The frictional and wear behaviour of this material shows similar behaviour as the other two. The higher friction results because of the less compact oxide layers and the deeper grooves on disc wear scar reveals the higher volume loss due to interaction with wear debris during the sliding process. The steady state friction is caused by the debris particles getting embedded in the grooves and there is no damage to the samples.

From EDS and SEM, the wear scars show low oxygen concentration. The material adhering to tool steel reveals the change in chemical composition and the presence of boron steel elements on to the pin sample is the evidence of material transfer form disc to pin specimens. Table 10 shows the Weight % of elements present in tool steel 3 after test, from which it is clear that oxygen concentration is different for all the three specimens.

<table>
<thead>
<tr>
<th>Element</th>
<th>Post oxidation 1</th>
<th>Post oxidation 2</th>
<th>Post oxidation 3</th>
</tr>
</thead>
<tbody>
<tr>
<td>O</td>
<td>6.85</td>
<td>5.72</td>
<td>-</td>
</tr>
<tr>
<td>Si</td>
<td>-</td>
<td>0.43</td>
<td>0.38</td>
</tr>
<tr>
<td>C</td>
<td>4.28</td>
<td>1.64</td>
<td>0.50</td>
</tr>
<tr>
<td>Cr</td>
<td>1.19</td>
<td>5.10</td>
<td>5.31</td>
</tr>
<tr>
<td>Fe</td>
<td>87.68</td>
<td>86.33</td>
<td>93.06</td>
</tr>
</tbody>
</table>

From SEM images it is revealed that the wear mechanisms are different for three samples. There is no specific wear on disc with tool steel 3 post oxidation 1 and the material is adhering on to the tool steel pin specimen. The other two samples behaviour is also similar but the disc volume loss is higher when compared to post oxidation 1.

Fig 19 shows the wear scars of tool steel 1, tool steel 2 and the respective boron steel wear scars. The frictional behaviour and wear characteristics of these two materials are different from tool steel 3. From the SEM images, clear evidence of the absence of material transportation from boron steel disc to tool steel specimen. The wear mechanism is similar to the tool steel 3 and the wider wear scars reveals the higher volume loss from the both tool steel and boron steel specimens. The high friction obtained during room temperature test may be attributed to the occurrence of severe adhesion which can be ascertained from the SEM micrographs.
Fig 19. SEM images of wear scars of pin (top) and disc (bottom) specimens
(a) Tool steel 1 (b) Tool steel 2

The image shows the longer and deeper grooves explaining the plastic deformation of material and governing wear mechanism which appears to be mainly adhesive accompanied by some abrading action of oxidised wear debris causing higher material removal. From XRD and EDS, it shows the negligible amount of oxide forming elements on tool steel and the more amounts of harder nitrided elements. Table 11 shows the weight % of elements present in tool steel 1 and 2. The harder nitrided wear debris causes the higher friction coefficient and unsteadiness in the behaviour which results in more material removal from disc rather than from pin. When the nitrided layer was removed from the tool steel the softer pin material will be in contact with the disc and causes the higher material removal from both pin and disc.

Table 11. Weight % of elements present in tool steel 1 and 2 at room temperature test (after test)

<table>
<thead>
<tr>
<th>Element</th>
<th>Tool steel 1</th>
<th>Tool steel 2</th>
</tr>
</thead>
<tbody>
<tr>
<td>O</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Si</td>
<td>0.22</td>
<td>0.57</td>
</tr>
<tr>
<td>C</td>
<td>0.65</td>
<td>1.49</td>
</tr>
<tr>
<td>Cr</td>
<td>1.87</td>
<td>1.39</td>
</tr>
<tr>
<td>Fe</td>
<td>96.06</td>
<td>95.68</td>
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From the SEM images we can observe clear and wider wear scars, higher volume loss of both pin and disc specimens and more wear debris in these two tests which may cause the higher and unsteadiness in the friction coefficient. The pin and disc SEM images clearly shows the adhesive wear scars and deeper grooves of material removal on the disc.

4.2 Wear loss and Surface damage of specimens at 400 °C

Wear loss of tool steel and boron steel specimens at high temperature are different from wear loss at room temperature. Fig 20 shows the wear loss of tool steel 3 and boron steel specimens at 400 °C. The test reveals that, for all of the tool steel 3 samples, wear increased manifolds when the temperature was increased. It is to be noted that the tool steel 3 post oxidation 1 sample has less wear compared to others at room temperature and even at high temperature.

![Graph showing wear loss comparison at different oxidation stages](image)

*Fig 20. Pin and Disc volume wear loss of tool steel 3 at 400 °C*

In order to compare the tests at room temperature and the tests at high temperature, two high temperature tests are carried out with similar tool steels specimen of tool steel 3 on the same disc with different wear track radius. First test was at 16 mm radius and second test was at 22 mm radius.

In tribological tests at elevated temperatures, the oxidation of test specimen plays an important role to determining their friction and wear characteristics. The governing wear mechanism is a combination of abrasion and adhesion and we can observe the change in wear of the pin specimens, which is negligible at room temperature and remarkable at high
temperature. There are several ways that these oxides or oxide wear debris can influence the wear behaviour. The higher wear is because the wear debris may be removed or retained in the contact and form protective layers. The oxides can also act as third bodies and get embedded and causes increasing wear with increasing in temperature.

The increase in friction towards the end of test and increase in wear of tool steel is may be attributed to increasing the contact area and softening of the material. Increasing wear with temperature has also been reported by Hardell et al. [12] and Wang et al. [13] as a result of increased abrasive wear due to softening of the specimen thus providing in sufficient support for the oxide layers to be formed. From SEM images, see Fig 21, the tool steel material is adhered to boron disc and the wear behaviour is different for all the three.

Fig 21. SEM images of wear scars for tool steel 3 Pin (top) and boron steel disc at 400 °C at 16 mm wear track radius
V. Conclusions

Experimental studies pertaining to the friction and wear behaviour of different tool steels sliding against high strength boron steel have been carried out at room temperature and at elevated temperatures (400 °C). The experiments were carried out in ambient air without any lubricants and the salient conclusions are as follows:

- At room temperature, the friction characteristics of tool steel 1 and 2 are different from tool steel 3. Tool steel 1 and 2 have shown higher and more unsteady friction where as tool steel 3 shows lower friction with a steadier behaviour. Different post oxidation temperatures produces different amount of oxides, post oxidation 1(500 °C) was highest, post oxidation 2 (520 °C) was moderate and post oxidation 3 (480 °C) was lowest. These oxides result in decreasing the friction and wear.

- At 400 °C, tribological properties are strongly influenced by the temperature and the friction was higher and gradually increased. The hardness of tool steel 3 materials have significant effect on the initial friction behaviour which influences the final friction. The three post oxidation samples have similar frictional behaviour but different wear rates. Wear increases from post oxidation 1 to 3 and the boron steel wear loss is less when compared to tool steel due to the softening of the tool steel specimens.

- The wear behaviour observed is mainly adhesive at room temperature and abrasive and adhesive at higher temperature. There is hardly any wear of tool steel 3 post oxidation 1 at room temperature.

- Tool steel 3 with post oxidation 1 has shown to have lower friction and wear compared to the other investigated tool steel variants both at room temperatures and high temperatures.
VI. Future work

Future work on tool steel 1, 2 and 3 should focus on the following aspects:

- For tool steel 3, varying the post oxidation temperature and time to find an optimum combination of iron oxides and iron nitrides.
- For tool steel 1 and 2, experiments should be carried out with post oxidation 2 and 3 and investigate the tribological properties.
- Experiments should be performed at elevated temperatures by reversing the specimen geometry to investigate the tribological properties and compared them with the present work.
- Experiments should be carried out by changing the heat treatment process time and post oxidation process condition.
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