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Tribological behaviour of surface-treated and post-oxidized tool steels at room temperature and 400 °C

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Abstract. The primary focus of this investigation is on tribological properties of plasma nitrided and post-oxidized tool steels during sliding against ultra-high-strength boron steel. The experimental work has been carried out at room temperature and at 400 °C by using a high-temperature pin on disc tribometer. The experimental materials were tool steels of three different compositions, which were plasma nitrided and post-oxidized at 500 °C. One of the tool steels was also post-oxidized at 480 and 520 °C. The results have shown that the friction and wear characteristics are influenced by the test temperature and the post-oxidizing temperature. The tool steel, post-oxidized at 500 °C, resulted in better friction and wear performance at room temperature and also improved wear resistance at elevated temperature. The observed wear mechanisms are mainly adhesive at room temperature and a combination of adhesive and abrasive at elevated temperature.

Key words: tool steel, high strength boron steel, wear, friction, post-oxidation, plasma nitriding.

1. INTRODUCTION

Recent years have witnessed an increasing demand for ultra-high-strength boron steels (UHSS) in energy absorbing systems, especially in automobile applications due to their high strength-to-weight ratio [1]. There are many challenges during forming of these steels owing to their poor formability, increased spring-back and their tendency to workharden. In order to overcome these problems, the UHSS components are usually produced through hot forming processes [2]. During hot forming, lubrication is not possible by means of conventional lubricating fluids as these rapidly decompose at elevated temperatures, typically above 300 °C. The high temperatures, encountered during hot forming operations, lead to increased tool wear and unpredictable friction behaviour. In view of this, the utilization of newly developed surface treatments is a possible way to control friction and minimize tool wear.

Nitriding is known to enhance surface hardness, fatigue strength, tribological properties and to an extent corrosion resistance of steel components. Plasma nitriding is commonly used owing to a number of advantages such as shorter treatment time, lower process temperature and minimal distortion compared to conventional techniques [3]. Post-oxidation of nitrided and nitrocarburized steels is an economical way of improving the tribological and corrosion properties. The process parameters of the post-oxidation treatment such as time, temperature and medium influence the type of oxides formed (i.e. Fe_3O_4, Fe_2O_3 and FeO) [4]. Typically, a thin layer of Fe_3O_4 is formed as the outermost layer on top of the compound layer [5]. This oxide layer enhances corrosion resistance and it also imparts a black surface which is aesthetically appealing. However, the tribological properties of these post-oxidized nitrided surfaces have not yet been investigated in detail. Sun [5] has investigated the room temperature sliding frictional behaviour of post-oxidized plasma nitrided steel during sliding against steel and alumina counterbodies. He found that, in the case of the steel counterbody, the frictional behaviour was mainly governed by a transfer film, consisting of the material from the steel counterbody, and differences could not be detected between different post-oxidation temperatures. However, in the case of plasma nitrided steel, post-oxidized at 400 °C, sliding against an alumina ball low friction was achieved. The low friction level was maintained until failure of the oxide layer occurred. Sun concluded that at higher post-oxidizing temperatures, a more porous and less adherent oxide film is formed, which leads to poor tribological behaviour. Zlatanović et al. [6] investigated oxide films, created on salt bath nitrocarburized and plasma nitrocarburized steel samples, respectively. They found that a well adherent Fe_3O_4 layer was formed on the nitrided samples whereas a Fe_2O_3 layer was formed on an untreated reference sample, indicating that the substrate plays an important role in determining the final oxide composition. Alsaran et al. [7] studied the influence of post-oxidation time on the tribological behaviour. They found that increased post-oxidation time resulted in an oxide layer, which gets easily spalled off. Prolonged post-oxidation time also adversely affected the friction and wear behaviour. Alloying elements also play an important role in the formation of oxide layers. The high temperature tribological behaviour is, to a large extent, governed by the oxide layers formed at the sliding interface. Aluminium and silicon yield a reduction of oxide film thickness thus leading to an increase in wear as temperature is increased [8]. During the tribological process, oxidized wear debris are produced either by a transient oxidation process (oxidation, removal of the oxide at the next transversal and reoxidation) or by formation, fracture, comminution and oxidation of metal debris particles. The layers, formed at temperatures below 300 °C, were found to be unstable and were removed rapidly. The layers, formed at higher temperatures, were more resilient and provided lower friction and wear rates [9]. Load also has a significant effect on the high-temperature friction and wear behaviour of materials. At lower loads, the protective oxide layers are able to sustain longer durations than at higher loads [10].

From this brief literature review, it is evident that the role of post-oxidation of the tool steel during its tribological interaction with ultra-high-strength boron steel has not been studied. The present work is thus aimed at investigating the friction and wear behaviour of different plasma nitrided tool steels, post-oxidized at different temperatures during sliding against the ultra-high-strength boron steel at room temperature and 400 °C. The structural, mechanical and tribological properties of these samples have been investigated by using a high-temperature pin-on-disc machine, 3D optical surface profiler, microhardness tester, X-ray diffraction (XRD), Scanning Electron Microscopy (SEM) and Energy Dispersive Spectroscopy (EDS).

2. EXPERIMENTS

2.1. Test materials and specimens

The experimental work performed in this research involved three different compositions of tool steels and one composition of UHSS. The chemical composition of the tool steels and UHSS are presented in Table 1. The tool steel specimens were plasma nitrided and subsequently post-oxidized. Tool steel 1 (TS 1) and tool steel 2 (TS 2) were post-oxidized at 500 °C whereas tool steel 3 (TS 3) was post-oxidized at three different temperatures, i.e. post-oxidation 1 (PO1) at 500 °C, post-oxidation 2 (PO2) at 520 °C and post-oxidation 3 (PO3) at 480 °C. The tool steel test specimens were cylindrical pins (Ø 10 mm, 10 mm long) with one end spherical (with a radius of 5 mm). The specimens were nitrided through a patented process known as CORR-I-DUR®, which is a combination of various thermochemical process steps involving plasma nitriding, gas nitrocarburizing and oxidation. The depth of the nitrided layers was between 0.25 and 0.3 mm and the duration of the process was 240 min. The mating UHSS test specimens were flat discs (Ø 24 mm, 7.9 mm thick). The UHSS discs were hardened by austenitization and subsequential quenching in water. The discs were thereafter ground and polished to a low surface roughness. A schematic of the test specimen configuration is given in Fig. 1. Wear of the tool steel and

Table 1. Compositions (wt%) of UHSS and different tool steels, Fe makes up the balance

Material	C	Mn	Cr	Si	B	P	S	Ni	Mo	V
UHSS	0.25–0.25	1.0–1.3	0.14–0.26	0.2–0.35	0.005	>0.03	>0.01	–	–	–
Tool steel 1	0.37	1.4	2.0	0.3	–	–	–	1.0	0.2	–
Tool steel 2	0.31	0.9	1.35	0.6	–	Max 100 ppm	Max 40 ppm	0.7	0.8	0.145
Tool steel 3	0.39	0.4	5.2	1.0	–	–	–	–	1.4	0.9

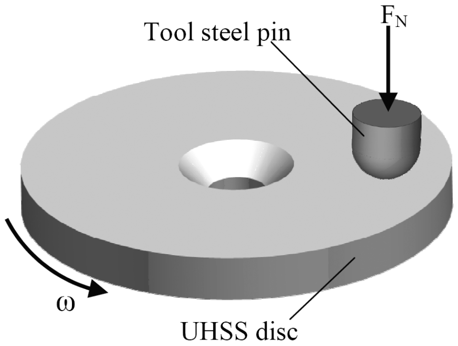

Fig. 1. Schematic of the test specimen configuration.

Table 2. Vickers hardness and initial surface roughness of different test specimens

Specimen	Hardness, HV	Ra, nm
UHSS	647	374
Tool steel 1, post-oxidation 1	590	294
Tool steel 2, post-oxidation 1	689	866
Tool steel 3, post-oxidation 1	648	604
Tool steel 3, post-oxidation 2	710	597
Tool steel 3, post-oxidation 3	605	992

UHSS specimens have been quantified by 3D optical surface profiler measurements of the wear scars and the calculation of wear volume is based on these measurements. The initial surface roughness and hardness of the test specimens are given in Table 2.

2.2. Test equipment

The experimental studies were carried out by using a high-temperature pin-on-disc machine. The 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. The disc spindle is driven through a belt and pulley drive system and the sliding speed can be varied by adjusting the radius of the test track and spindle speed. A strain gauge transducer, mounted at the rear end of the pin carrier head, measures the friction force. For high temperature tests (400 °C), an external hot air blower is used for heating the test specimens and the temperature is measured by an optical pyrometer.

The surfaces of unworn and worn specimens were characterized by SEM/EDS, XRD, 3D optical surface profiler and microhardness tester.

2.3. Test procedure

All test specimens and specimen holders were cleaned in petroleum spirit using an ultrasonic cleaner and were then rinsed with ethanol. The tests were performed at room temperature and at high temperature (400 °C) at a load of 4.905 N and a sliding velocity of 0.175 m/s. For high temperature tests, a heating sequence was run to heat the lower test specimen (UHSS disc) to 400 °C. As the disc specimen reached the desired temperature, the pin specimen was brought into contact and loaded against the disc and the test was started. All tests were repeated at least once and good reproducibility was observed. As an example, the scatter in friction measurements for TS3 PO1 was 0.72 ± 0.023. The uncertainty in wear measurements is given in Tables 3 and 4 as the average wear volume and standard deviation.

3. RESULTS AND DISCUSSION

The XRD analysis (Fig. 2) of tool steel specimens shows the presence of Fe_2N, Fe_3O_4, Fe_4N and a small amount of martensite. This correlates well to the expected phases since the aim is to obtain a Fe_3O_4 layer in view of its good corrosion resistance and wear properties. Ramesh et al. [11] have also reported that the oxide layer, produced by post-oxidation process, consists of magnetite (Fe_3O_4) and hematite (Fe_2O_3) and is formed over the compound layer containing ε-$Fe_{2\text{-}3}N$.

Table 3. Weight % of elements present in worn tool steel specimens and wear volume of tool steel (pin) and UHSS (disc) specimens from room temperature tests

Material	Elements					Wear volume, mm^3	
	O	Si	C	Cr	Fe	Pin	Disc
TS 1, PO1	–	0.22	0.65	1.87	96.06	0.146 ± 0.062	0.058 ± 0.028
TS 2, PO1	–	0.57	1.49	1.39	95.68	0.093 ± 0.004	0.051 ± 0.027
TS 3, PO1	6.85	–	4.28	1.19	87.68	0.01 ± 0.0003	0.033 ± 0.0004
TS 3, PO2	5.72	0.43	1.64	5.10	86.33	0.017 ± 0.004	0.041 ± 0.013
TS 3, PO3	–	0.38	0.50	5.31	93.06	0.023 ± 0.004	0.028 ± 0.016

Table 4. Weight % of elements present in worn tool steel 3 specimens and volume wear loss of tool steel (pin) and UHSS (disc) specimens from tests at 400 °C

Material	Elements					Wear volume, mm^3	
	O	Si	V	Cr	Fe	Pin	Disc
TS 3, PO1	0.51	0.20	0.65	5.41	93.23	0.48 ± 0.14	0.42 ± 0.05
TS 3, PO2	–	0.15	1.09	5.99	92.53	0.59 ± 0.14	0.59 ± 0.13
TS 3, PO3	–	0.16	1.25	5.52	92.92	0.56 ± 0.04	0.67 ± 0.14

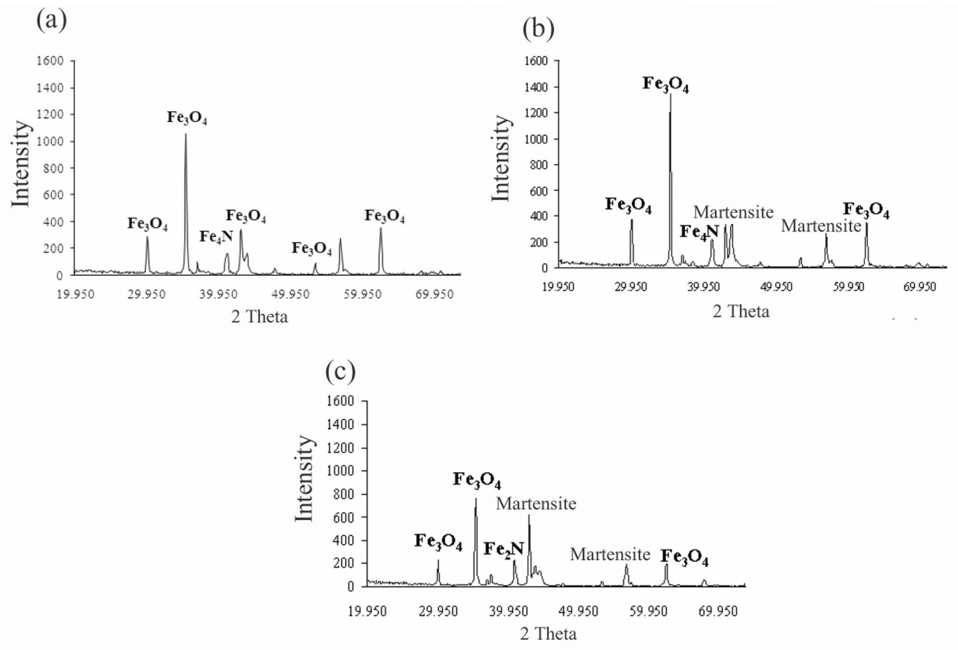

Fig. 2. XRD spectra of unworn tool steel 3 specimens: (a) post-oxidation 1, (b) post-oxidation 2, (c) post-oxidation 3.

3.1. Room temperature

Figure 3a shows the frictional behaviour of TS 1 and TS 2 and Fig. 3b the frictional behaviour of TS 3 at room temperature. The frictional behaviour is different for all the three tool steel materials, TS 1 and TS 2 show unsteady and higher friction values in the range of 0.9 to 1 whereas TS 3 shows steadier and lower friction values ranging from 0.7 to 0.8. The higher and more unsteady friction in case of TS 1 and TS 2 is caused by the presence of a different surface layer composition as seen from XRD analysis and it is known that the composition of the oxide layer is affected by the substrate [6]. TS 3, post-oxidized at 500 °C (PO1), shows a lower friction coefficient in the beginning and at the end of the test compared to TS 1 and TS 2 with PO1. XRD results reveal that the surface has a higher amount of iron oxides (Fe_3O_4), which can reduce friction. Alsaran et al. [9] also have reported that post-oxidation treatment after plasma nitriding results in the formation of Fe_3O_4, which is beneficial in reducing the friction and wear rate. TS 3 was also post-oxidized at 480 °C and 520 °C (PO2 and PO3 respectively) and the frictional results are shown in Fig. 3b. The initial friction is higher for PO2 and PO3 compared to PO1, which is attributed to the difference in surface layer compositions as indicated by the XRD results in Fig. 2. It is known that the post-oxidation temperature and time affects the composition and adhesion of the oxides layers [5,7] and it can be seen that the post-oxidations, carried out at 480 and 520 °C, results in surface layers that are

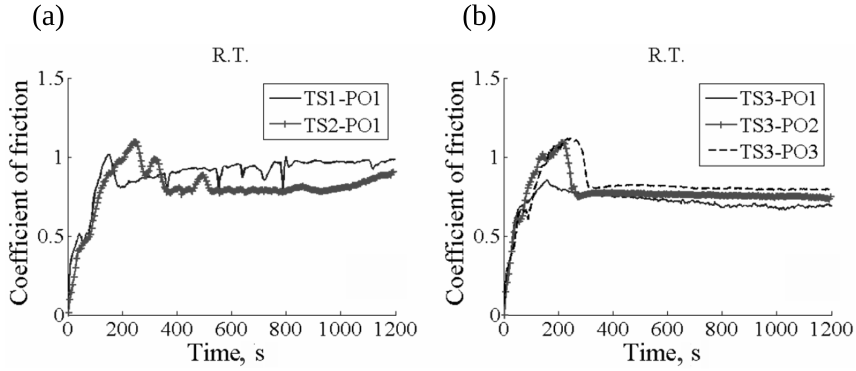

Fig. 3. Friction coefficients of tool steel 1 and tool steel 2 with post-oxidation 1 (a) and tool steel 3 (post-oxidations 1, 2 and 3) sliding against UHSS disc at room temperature (b).

worn away rather quickly as indicated by the high initial friction. The similar friction levels, observable towards the end of the tests in Fig. 3b are attributed to the formation of very similar surface layers, which determine the friction level.

Wear scars of pin and disc specimens have been analysed by SEM/EDS and showed some interesting results. Figure 4 shows SEM micrographs of wear scars and Table 3 shows the EDS analysis of TS 1 and TS 2 from tests at room temperature. The disc wear scars shows typical severe adhesive wear features and the mating pin surfaces have a very smooth wear scar with some wear debris accumulating around the edges. Adhesive wear is caused by formation of adhesive junctions between the contacting surfaces. If the strength of the junction is higher than the yield strength of one of the materials in contact then it will lead to transfer and/or removal of material as sliding progresses resulting in rough and irregularly torn surfaces. The occurrence of severe adhesion correlates well to the higher and unstable friction behaviour, observed in tests for these material pairs. In case of TS 3 with PO1, low wear on both the pin and disc specimens can be seen (Table 3). This is attributed to higher amount of iron oxides (Fe_3O_4) on the tool steel pin surface from the post-oxidation process, which is also reflected in the relatively lower friction obtained in these tests. The SEM micrographs (Fig. 5) support these observations. For TS 3 with PO2 and PO3, the wear is higher (compared to that with PO1) on both specimens. This indicates that the reduced amounts of iron oxides on the tool steels (as revealed by XRD analysis in Fig. 2) results in more adhesion, as can be seen in Fig. 5, and subsequent material removal. Another parameter, influencing wear in these experiments, is the changing contact pressure due to wear on the semi-spherical pin. The initial Hertzian contact pressure is around 800 MPa, which results in a highly concentrated contact. In case of TS 1 and TS 2, the final contact pressure is around 1.5 MPa, which is substantially lower compared to the initial contact pressure. Although the contact pressure is reduced, the severity of adhesion increases for TS 1 and TS 2 as compared to that of TS 3-PO1, in which the spherical shape is

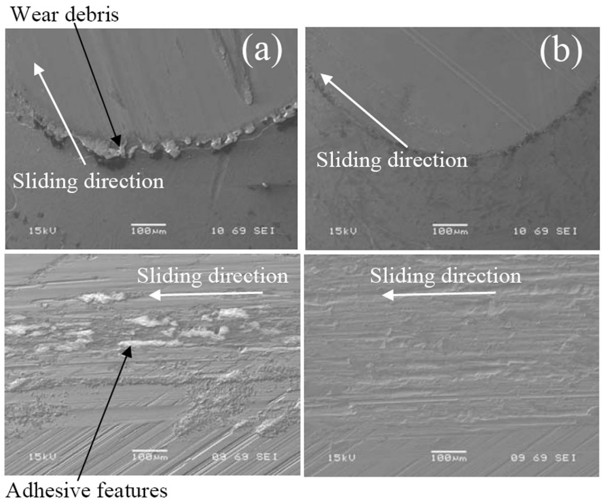

Fig. 4. SEM micrographs of wear scars (top-pin, bottom-disc) from room temperature tests: a – tool steel 1, post-oxidation 1; b – tool steel 2, post-oxidation 1.

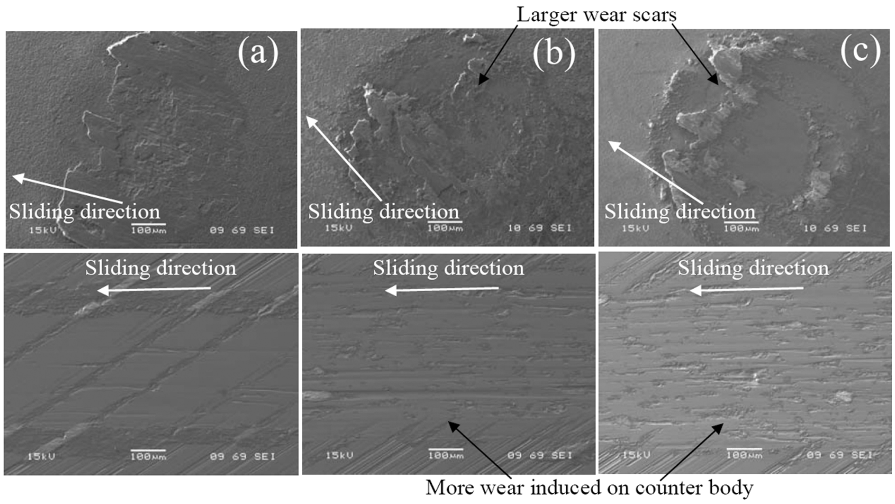

Fig. 5. SEM micrographs of wear scars (top-pin, bottom-disc) from room temperature tests: a – tool steel 3, post-oxidation 1; b – tool steel 3, post-oxidation 2; c – tool steel 3, post-oxidation 3.

maintained even after the test and thus only very small change in contact pressure takes place. This can also be seen by comparing Fig. 4a and b with Fig. 5a. This is explained by more wear on TS 1 and TS 2, which leads to removal of the outermost protective surface layers and consequently resulting in increased adhesion.

3.2. Temperature 400 °C

Tests at an elevated temperature of 400 °C were also conducted using TS 3 with the aim to study the friction and wear behaviour of different post-oxidation treatments. The friction coefficients as a function of time from the high-temperature tests are shown in Fig. 6. It can be observed that the overall trend in frictional behaviour is similar for all the three different post oxidations even if the friction levels differ. The friction initially increases and then levels out mid-way through the test duration. The difference in the friction level is partly attributed to the differences in hardness of the tool steel samples. The plasma nitrided tool steel pin specimen, with its spherical end in contact with the disc specimen, is likely to better retain its hardness at elevated temperatures and will give rise to higher ploughing component of friction as it indents into the relatively softer mating disc material. In Table 2 it can be seen that TS 3-PO2 is hardest, followed by TS 3-PO1 and TS 3-PO3, respectively. The friction levels are also in the same order (ranging from high to low) as can be seen in Fig. 6. In tribological tests at elevated temperatures, the oxidation of the test specimens plays an important role in determining their friction and wear characteristics. Figure 7 shows the SEM micrographs of worn TS 3 and the occurrence of severe adhesion is quite evident, which is also reflected in the higher friction levels observed at elevated temperatures.

The SEM micrographs of worn specimens and wear volume together with EDS results from tests at elevated temperature are given in Fig. 7 and Table 4, respectively. It can be seen that the main wear mechanisms are adhesive and abrasive in nature. The adhesive wear results in material transfer from the tool steel to the UHSS which is in marked contrast to the behaviour, observed at room temperature, where the material adhered to the pin. The abrasive grooves are caused due to the interaction with hard oxidized wear debris. It is also interesting to note that TS 3 shows a similar trend in terms of its wear rate at room temperature and at 400 °C. TS 3 with PO1 shows the lowest wear rate in both cases and TS 3 with PO2 shows the highest. It can be concluded that even though

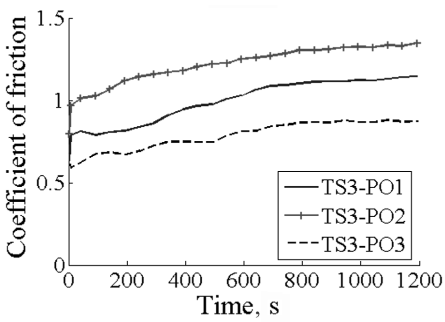

Fig. 6. Friction coefficient of tool steel 3, post-oxidations 1, 2 and 3 sliding against the UHSS disc at 400 °C.

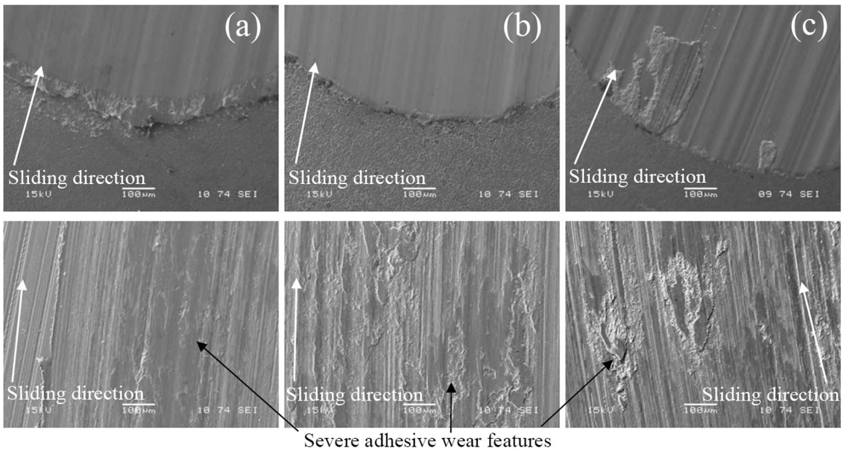

Fig. 7. SEM micrographs of wear scars (top-pin, bottom-disc) from tests at 400 °C: a – tool steel 3, post-oxidation 1; b – tool steel 3, post-oxidation 2; c – tool steel 3, post-oxidation 3.

TS 3 PO1 does not have the highest hardness, it is more wear resistant compared to the other post-oxidized tool steel specimens. This implies that formation of surface layers with an optimum balance between oxides and nitrides is essential for the improved friction and wear behaviour. From XRD analysis it is clearly seen that TS 3 PO1 has higher amount of oxides and lower amount of harder nitrided phases as compared to those in TS 3 PO2 and PO3, respectively.

The increase in friction towards the end of the test and increase in wear of tool steel is attributed to the increased contact area and softening of the materials. The increased contact area will result in more sites for formation of adhesive junctions and an increase in wear. Increase in 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 and insufficient support for the oxide layers.

4. CONCLUSIONS

Experimental studies pertaining to the friction and wear behaviour of different tool steels (plasma nitrided and post-oxidized at different temperatures) sliding against ultra high strength boron steel have been carried out at room temperature and at elevated temperature (400 °C). These results indicate that the operating temperature, composition and post-oxidation temperatures have significant influence on the tribological behaviour of the tool steel during sliding against ultra high strength boron steel. The salient conclusions from this study are as follows.

1. At room temperature, the friction characteristics of the tool steels 1 and 2 are different from tool steel 3. Tool steels 1 and 2 show an unstable friction

coefficients of about 0.8 and 0.9, respectively. In contrast, tool steel 3 has a relatively stable and lower friction coefficient of 0.7. Tool steel 3 with post-oxidation 1 showed 10–15 times better wear resistance compared to tool steels 2 and 1, respectively.

2. At 400 °C, the tribological properties are strongly influenced by the temperature. Friction at 400 °C is about 0.8 for tool steel 3 with post-oxidation 1 compared to 0.7 at room temperature. The hardness values of tool steel 3 specimens have significant effect on the initial friction behaviour and also influence the final friction. Wear at 400 °C was found to be about 20% lower in case of the tool steel 3 with post-oxidation 1 compared to the tool steel 3 with post-oxidation 3 and tool steel 3 with post-oxidation 2.
3. The wear mechanisms observed are predominantly adhesive at room temperature and a combination of abrasive and adhesive at higher temperature. There is hardly any wear of the tool steel 3 with post-oxidation 1 at room temperature.
4. Tool steel 3 with post-oxidation 1 resulted in lower friction and wear compared to the other tool steel variants at room temperature. At high temperature, tool steel 3 with post-oxidation 1 outperforms the other post-oxidized nitrided tool steels in terms of wear resistance.

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Pindtöödeldud ja järeloksüdeeritud tööriistateraste triboloogiline käitumine toatemperatuuril ning 400 °C juures

Abdul Khadar Syed, Jens Hardell ja Braham Prakash

Töö eesmärgiks on plasmanitriiditud ja järeloksüdeeritud tööriistateraste triboloogiliste omaduste uurimine liugkulumisel vastu ülitugevat boorterast. Eksperimendid viidi läbi toatemperatuuril ja 400 °C juures kõrgetemperatuursel *pin-on-disc* tribomeetril. Uuritavateks materjalideks olid kolm erineva koostisega tööriistaterast, mis olid plasmanitriiditud ja järeloksüdeeritud 480–520 °C juures. Selgitati välja, et hõõrde- ja kulumisnäitajad on sõltuvad katsetuse ning järeloksüdeerimise temperatuurist. Parim vastupanu hõõrdumisele ja kulumisele nii toatemperatuuril kui ka kõrgendatud temperatuuril oli 500 °C juures järeloksüdeeritud tööriistaterasel. Põhiliseks kulumise mehhanismiks toatemperatuuril oli adhesioon, kõrgendatud temperatuuril kombinatsioon adhesioonist ja abrasioonist.