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Wear characteristics of plasma-nitrided CrMo steel under mixed and boundary lubricated conditions

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An investigation of the sliding wear mechanisms of a plasma-nitrided CrMo steel is undertaken using a running-in procedure that eliminates severe wear under mixed and boundary lubricated conditions. In the running-in procedure, using a pin-on-disc wear machine, smooth contact surfaces are obtained rather than the rough contact surfaces generally found in laboratory experiments. This enables the wear mechanisms to be investigated more clearly, particularly mild wear processes. The work shows no measurable nor visible wear under full fluid film lubricated conditions, the existence of polishing under mixed lubricated conditions and micro-pits under boundary lubricated conditions. The results presented in this paper indicate that a mild abrasive wear mechanism predominates under mixed lubricated conditions and a micro-fatigue wear mechanism under boundary lubricated conditions. Examination of the microstructure reveals the formation of white layer regimes on the contact surfaces after tests under boundary lubricated conditions, which suggests severe work-hardening of the contact surfaces.

1. Introduction

Plasma-nitriding is used as a surface treatment to improve the wear resistance and fatigue strength of steels [1, 2]. It is a thermochemical process in which atomic nitrogen, formed in a nitrogen–hydrogen plasma, is diffused into the surface of the steel at a relatively low temperature. In plasma-nitriding, a thin white layer with a high nitrogen content forms at the surface [3]. As the thickness of this layer builds up, nitrogen diffuses from it into the underlying material to form a diffusion zone [4]. This microstructure produces favourable residual stresses, a low surface energy and a high value of hardness [5]. The hardness of nitrided steels contributes to their superior tribological performance, especially in providing excellent abrasive wear resistance [6]. In addition to hardness, nitriding also produces a compressive residual stress in the surface layers which gives rise to a marked improvement in fatigue performance [7].

The wear behaviour of plasma-nitrided steels has been investigated by several groups [6–8]. Kato *et al.* [6] reported a decrease of two orders of magnitude in the wear rates of plasma-nitrided materials. It was pointed out by Bell and Sun [7] that the improvement in the wear behaviour of these materials depends on the depth and strength of the nitrided case. Karamis emphasized the importance of case depth and microstructure for abrasive wear resistance of a plasma-nitrided material [8]. Unfortunately, there is little understanding of the tribological behaviour and characteristics of plasma-nitrided steels under lubricated conditions. Information about the wear characteristics during sliding under lubricated conditions is limited due to the lack of an appropriate running-in procedure [9]. Several groups [10–12] have identified the existence of adhesive, tribo-oxidation, abrasive wear, corrosion from additives, and delamination wear. A survey of reported data indicates that there is often a roughening process at the running-in stage of laboratory experiments, which results in severe abrasive and adhesive wear. Lee and Ludema [13] have reported that the prediction of the tendency for scuffing cannot be based on the initial surface topography before sliding because of the roughening of the contact surfaces during the running-in stage under lubricated conditions.

This paper concerns an investigation of the tribological performance of a plasma-nitrided CrMo steel which is used in gear applications. The work is directed at gaining an understanding of the wear mechanisms and related microstructural characteristics during sliding under mixed and boundary lubricated conditions with the secondary objective of achieving an appropriate running-in process without severe wear.

2. Experimental procedure

2.1. Materials

The material used in this work was a CrMo steel containing 0.15% carbon, 0.89% manganese, 0.24% silicon, 1.13% chromium and 0.23% molybdenum.

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This material was plasma-nitrided to a 0.3 mm case depth at 530 °C using a 45 V glow potential and a 300 Pa pressure of an atmosphere of 25% nitrogen and 75% hydrogen for 60 h. Fig. 1 shows the hardness profile as a function of thickness. It shows an increase in the hardness from 294 kgf mm^{-2} $H_{V(0.05)}$ at a depth of 3 mm to 1052 kgf mm^{-2} $H_{V(0.05)}$ at the outermost surface. Fig. 2 (a–c) shows the microstructure of the material which consists of a white layer at the surface (Fig. 2a) and a significantly coarser microstructure at the centre of the section (Fig. 2c) than that at or near the surface (Fig. 2a and b).

A commercially formulated lubricating gear oil was used in this work. It had a 105 viscosity index, 68.4 cSt (40 °C) viscosity, 214 flash point, 0.46 KOH g^{-1} neutralization number, 1.20 ppm sulphur and 130 ppm phosphorus.

2.2. Tribological tests

A conventional wear machine with a rotating pin-on-disc configuration was used to investigate the sliding tribological behaviour of CrMo steel under lubricated conditions. The disc specimen with a roughness R_a value of 0.35 μm was rotated at a speed of 348 rpm with a test track of 110 mm diameter, which gave a pin sliding speed of 2 m s^{-1}. A load was applied to the pin specimen, which was 6.35 mm in diameter and had an R_a value of 0.25 μm, through a cantilever. All tests were carried out in air at room temperature. In the lubricated tests, a tube was situated in front of the sliding contact area between the pin and disc specimens. The fresh lubricating gear oil was supplied continuously to the contact area during the tests.

The tribological tests were carried out for a period of 10 h under the lubricated conditions in order to investigate the lubrication mechanisms and sliding wear of the CrMo steel. The applied load varied from 10–1000 N. The frictional force was continuously recorded on chart paper using a calibrated linear variable differential transducer. The friction coefficient was calculated from the recorded frictional force. The extent of wear was assessed in terms of the wear volume of the pin specimens.

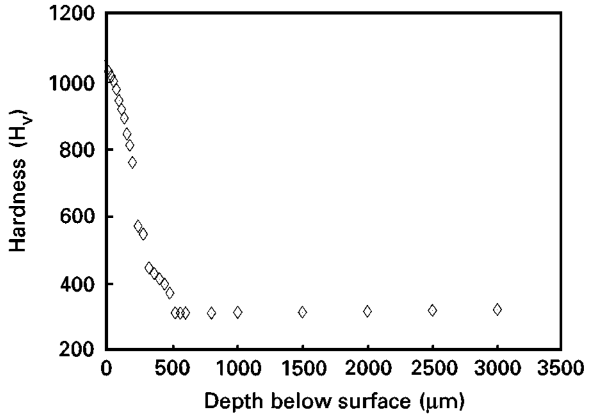

Figure 1 Microhardness profile as a function of depth into the plasma-nitrided CrMo steel.

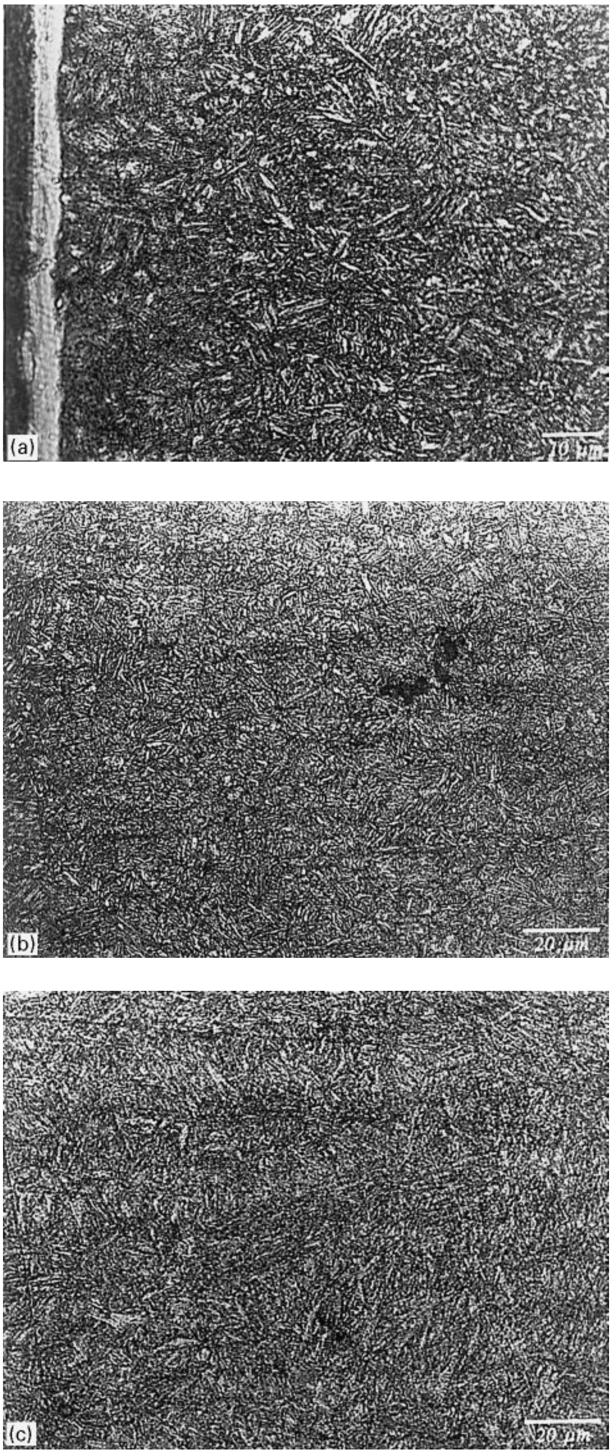

Figure 2 Light micrographs of plasma-nitrided CrMo steel cross-sections, show (a) a white layer on the outmost surface and metallurgical microstructure (b) near the top surface and (c) in the centre of the specimen.

2.3. Analysis of the morphology

The surface morphology of the wear tracks was examined using a light microscope and a scanning electron microscope (SEM) (Cambridge S250). The microstructure of the CrMo steel across the worn surfaces was investigated. An improved view of the morphology of a worn surface and the cross-sectional microstructure can be obtained with an 11° tilt [14]. In order to examine a taper specimen in both light and scanning electron microscopes, specimens were mounted in epoxy resin with an 11° angle between the worn surface and the viewing cross-section at room temperature. The mounted specimens were ground and polished and then the polished specimens were etched in 2% nital for the microstructural investigations.

3. Results

3.1. Tribological behaviour during sliding

The wear and friction of plasma-nitrided CrMo steel were examined during sliding under lubricated conditions. Fig. 3 shows the wear behaviour during sliding under a 450 N applied load. Examination of Fig. 3 revealed an increase in wear with sliding distance. Two types of variation in the wear during sliding are apparent: there was an initial period of rapid wear (a running-in stage) followed by a period of more gradual wear (an equilibrium wear stage).

Fig. 4 plots the friction coefficient as a function of sliding distance for the same lubricated conditions as those used in Fig. 3. From Fig. 4, it is clear that there is an initially high value of the friction coefficient which decreased rapidly and then remained relatively constant.

3.2. Variation of tribological behaviour with applied loads

The friction coefficient and wear rate of the CrMo steel were measured at different applied loads under lubricated conditions. Fig. 5 shows the relationship between the wear rates at an equilibrium wear stage and the applied load. Examination of Fig. 5 indicated that the wear rate increased with an increase in the applied load with an approximately linear correlation at the mild wear stage.

Variation of the friction coefficient during lubricated sliding in the equilibrium wear stage under different applied loads is presented in Fig. 6. It is clear from this figure that the value of the friction coefficient was high at a low applied load (about 0.055 at 10 N), and then it decreased with a modest increase in applied load (about 0.015 at 300 N). However, it then began to recover reaching a value of over 0.09 at 700 N.

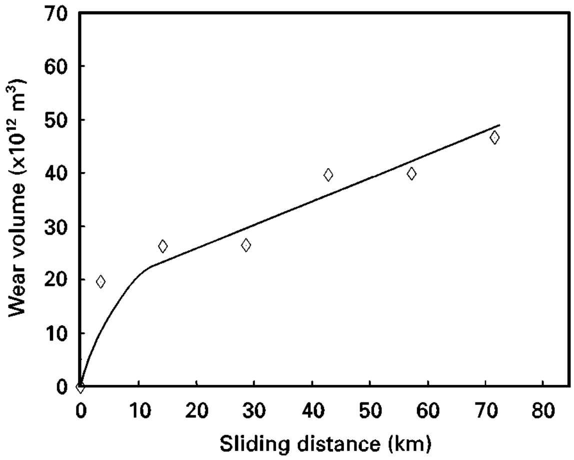

Figure 3 Sliding wear of CrMo steel during sliding at 2 m s^{-1} and under a 450 N applied load under lubricated conditions.

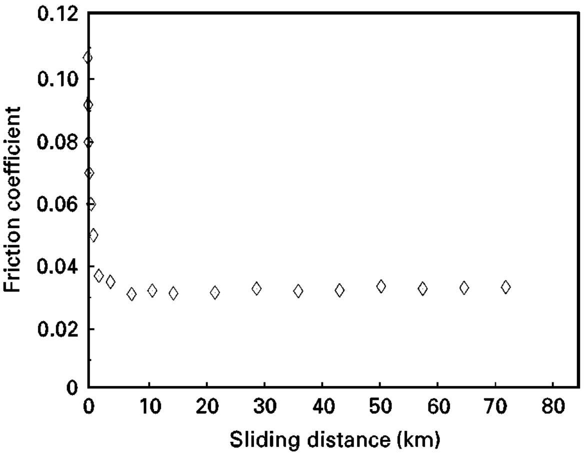

Figure 4 Friction coefficient of CrMo steel during sliding at 2 m s^{-1} and under a 450 N applied load under lubricated conditions.

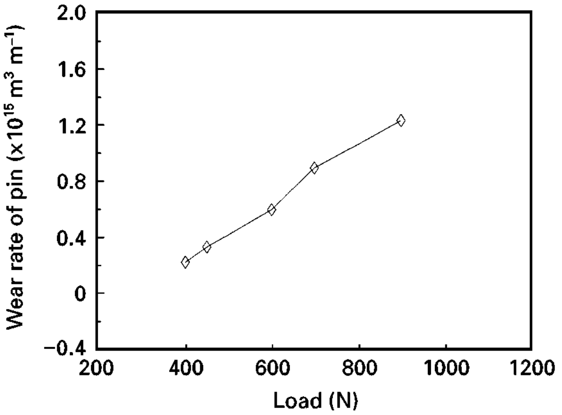

Figure 5 Sliding wear rates of CrMo steel at different applied loads at a 2 m s^{-1} speed under lubricated conditions.

4. Discussion

4.1. Running-in during sliding

When lubricated machine elements are run together for the first time, their ultimate load-carrying capacity is less than if they are preconditioned by running together for an initial period at a comparatively light load. This process is known as running-in. During this period the wear rate is often initially high, as is shown in Fig. 3. Further information can be obtained from the amount of wear debris particles in the lubricating oil, as determined by a particle quantifier (PQ) which measures the magnetic response of the particles in terms of a PQ value. The PQ value is directly related to the concentration of wear debris in the oil. Examination of PQ value of the gear oil used in the tests revealed an initial sharp decrease with increase in the sliding distance (Fig. 7), which is in agreement with a higher wear rate during the running-in period as compared to the equilibrium period.

The conditions and period for the running-in procedure are empirically determined since there is little measurable indication of how the sliding surfaces are

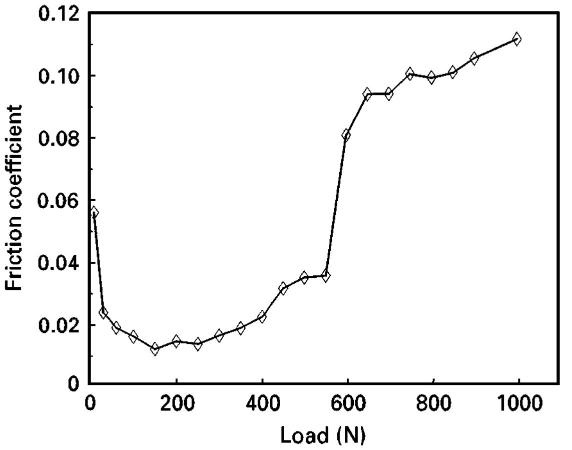

Figure 6 Friction coefficient of CrMo steel at different applied loads at a 2 m s^{-1} speed under lubricated conditions.

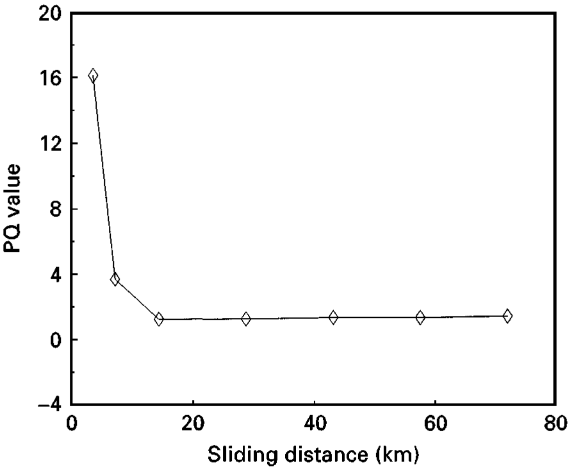

Figure 7 PQ index value of the gear oil collected during sliding of CrMo steel at 2 m s^{-1} under a 450 N applied load under lubricated conditions.

actually responding to the running-in procedure. In the laboratory, it is common to have a running-in procedure that consists of roughening the contact surfaces [11, 13]. However, it appears that the choice of running-in procedure can result in differences in the wear mechanisms at the mild wear stage under lubricated conditions. In order to investigate the wear mechanisms during the mild wear stage under lubricated conditions, it is necessary to use an appropriate running-in procedure. The transition from the relatively severe wear during running-in to the much milder wear in the equilibrium stage is due to the presence of high spots in the surface profile and initial misalignments between the mating surfaces. These effects lead to high local stresses and contact temperatures on the asperities which result, for instance, in the high friction coefficient shown in Fig. 4. As sliding progresses, the high spots and misalignments are removed and the contact stresses on the asperities reduce, which result in a lower friction coefficient. Examination of the worn surfaces indicated the operation of a polishing effect in this procedure, as is shown in Fig. 8.

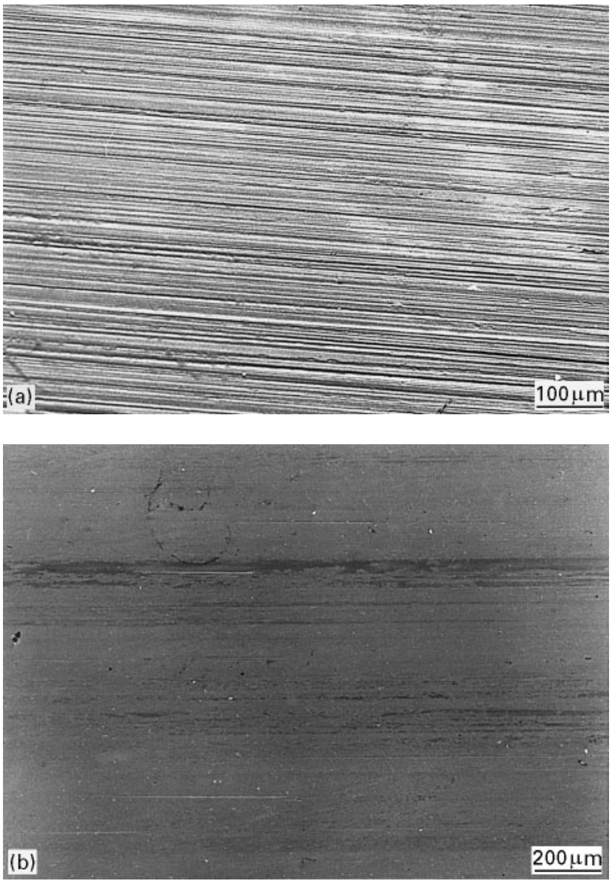

Figure 8 SEM micrographs of pin specimens (a) before and (b) after tests at a 300 N applied load at 2 m s^{-1} under lubricated conditions. Micrograph (b) shows the appearance of polishing.

4.2. Lubrication mechanisms at a mild wear stage

Under lubricated conditions, a Stribeck curve is usually used to indicate lubrication mechanisms [15, 16]. The Stribeck curve is a relationship between the friction coefficient and the dimensionless lubrication number (L_b), which can be formulated as;

$$L_b = \eta \frac{v}{Ra_t P} \tag{1}$$

where η is the dynamic viscosity coefficient in (N s m^{-2}), v is the sliding velocity with the units of (m s^{-1}), P is the normal pressure on sliding surfaces with units of (N m^{-2}) and Ra_t is the combined surface roughness ($[Ra_1^2 + Ra_2^2]^{1/2}$) with the unit of (m). A Stribeck curve is a function of several variables which include the geometry, materials, sliding conditions and thickness of a lubricating oil film between the sliding surfaces. Three main ranges of lubrication are distinguished: (i) boundary lubrication with a high value of friction coefficient at low values of L_b, (ii) mixed lubrication with the friction coefficient falling to a low value as L_b increases, and (iii) full fluid film lubrication with the friction coefficient rising as L_b increases further.

The lubrication number, L_b, was plotting against the friction coefficient for the sliding of plasma-nitrided CrMo steel under lubricated conditions to

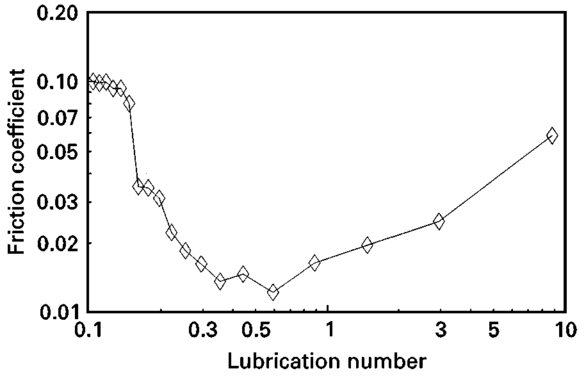

Figure 9 Relationship between the friction coefficient and lubrication number during sliding of CrMo steel under lubricated conditions.

produce a Stribeck curve as shown in Fig. 9, which provides an insight into the lubrication mechanisms. Examination of Fig. 9 suggests the occurrence of a full fluid oil film lubrication mechanism at high values of the lubrication number (10 N applied load); a mixed lubrication mechanism at intermediate lubrication numbers (300 N load) and boundary lubrication at low lubrication numbers (700 N load).

In order to confirm the lubrication mechanisms, voltages related to contact resistances between two contact surfaces were measured. Fig. 10(a–c) shows plots of the measured voltages during sliding at different applied loads under lubricated conditions. Examination of Fig. 10a indicated that the measured voltage (4 V) during sliding at a 10 N applied load was simply the supplied voltage which implies that the pin and disc specimens were completely separated by an oil film and that the lubrication mechanism was full fluid film lubrication.

With an increase in the applied load (to 300 N), the measured voltages between the sliding surfaces mostly remained at the level of the supplied voltage, although frequent fluctuations were observed (Fig. 10b) which suggests that the contact surfaces were mostly separated. From the fluctuation of the measured voltage, it can be inferred that some of the asperities on the sliding surfaces contacted each other during sliding. These contact points on the pin and disc surfaces were formed and destroyed rapidly during sliding, which caused the observed fluctuations in the electrical contact resistances. Furthermore, the measured voltages between the sliding pin and disc surfaces fluctuated rapidly. Consequently, it appears that the lubrication mechanism was of the mixed lubrication category.

At a higher applied load (700 N), there was a further decrease in the measured voltages, as can be seen in Fig. 10c. This decrease in the measured voltages suggest a further decrease and fluctuation of electrical contact resistances. It is expected that the real load-carrying area which is the summation of the asperity contact areas increases as the electrical contact resistances decrease. The existence of the electrical contact resistances indicated the presence of a non-metallic layer between the sliding surfaces. This may have been produced from the additives in the oil during sliding [17], which is consistent with a boundary lubrication mechanism [18].

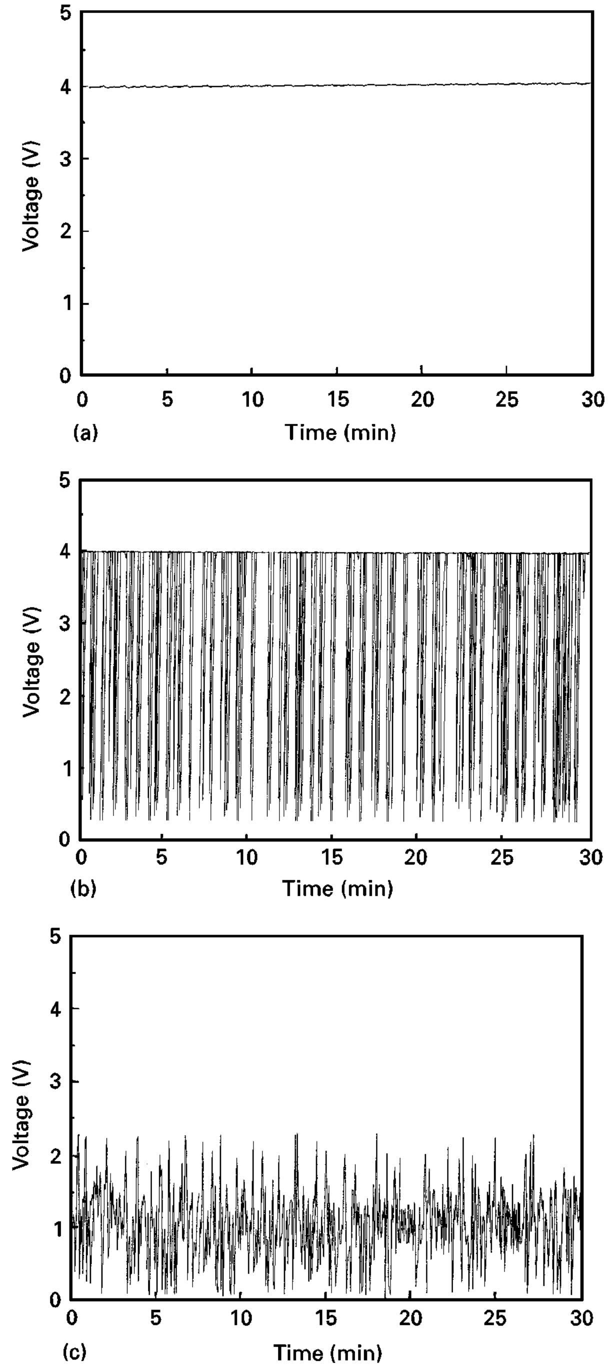

Figure 10 Measured voltages across the sliding interface during sliding of CrMo steel at 2 m s^{-1} in the gear oil at a mild wear stage: (a) 10 N, (b) 300 N, and (c) 700 N applied loads.

4.3. Wear mechanisms under mixed lubricated conditions

During mixed lubrication, both the friction coefficients and wear rates are low. There are few reports in the literature that consider sliding wear mechanisms under mixed lubricated conditions. In this work, an appropriate preparation process for matching the two sliding surfaces was applied before the sliding tests. With this process, it was found that there is no measurable or visible wear, as observed under a microscope, on the sliding surfaces after tests in the full fluid oil film lubrication regime and only extremely low wear in the mixed lubrication regime. This low wear regime was further investigated in order to elucidate the sliding wear mechanisms operating under these conditions.

As described above, the mixed lubricated experiments were performed using a 60 N applied load. However, it was found that no visible wear scar was observed on the worn surfaces after tests at this applied load. The worn surfaces were re-examined after tests at a 300 N applied load under lubricated conditions. As previously described, a polished surface was observed, as can be seen in Fig. 8. This polishing process could be the result of three wear mechanisms: (i) mild abrasive wear [19], (ii) mild adhesive wear [20], and/or (iii) tribochemical wear which can be caused by chemically active anti-scuffing (EP) additives in the lubricant oils [21]. An examination of the worn surfaces of the material at a high magnification after sliding tests in the mixed lubrication regime revealed evidence of a fine cutting process on most of the worn surfaces, as is shown in Fig. 11, which suggests that mild abrasive wear was the predominant wear mechanism.

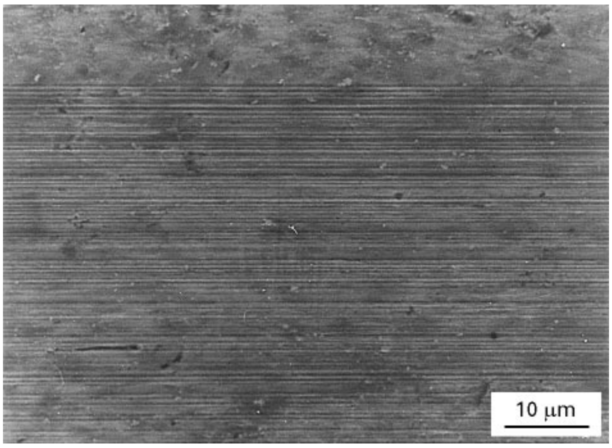

Figure 11 SEM micrographs of the worn surfaces of CrMo steel after sliding at 2 m s^{-1} under a 300 N applied load under mixed lubricated conditions. The micrograph shows mild abrasive wear.

4.4. Wear mechanisms under boundary lubricated conditions

The wear mechanisms under boundary lubricated conditions were investigated after lubricated sliding tests of plasma-nitrided CrMo steel at a 900 N applied load. A study of the worn surface using a light microscope indicated fine polishing after sliding. It was observed that a few black areas existed on the worn surface. Fig. 12(a and b) shows two SEM micrographs of typical areas on the worn surface after the tests. Fig. 12a shows the fine and polished surface with some small black regions. Close examination of the polished surface at a high magnification indicated that there was evidence for extremely mild abrasive wear, as is shown in Fig. 12b. Examination of the black regions at a high magnification suggested that they were fatigue wear tracks, which can be seen in Fig. 12b. Considering that the fatigue wear tracks are a few micrometres in size, this suggests micro-fatigue wear.

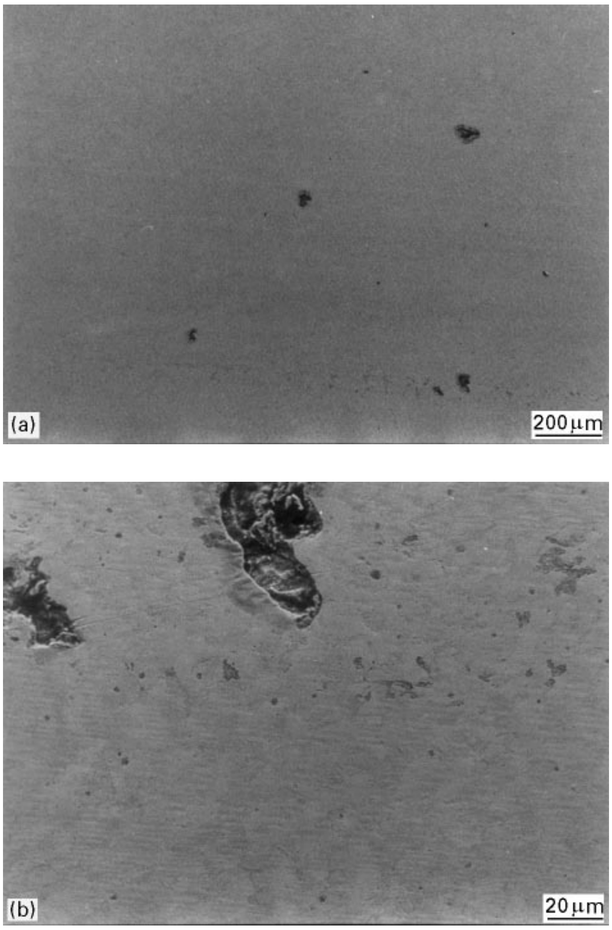

Figure 12 SEM micrograph of the worn surface of CrMo steel after sliding test at 2 m s^{-1} under a 900 N applied load under boundary lubricated conditions in the gear oil. The appearance of (a) the polished contact surfaces and (b) micro-pits in a range of boundary lubrication are clearly visible in these micrographs.

The cross-section through the worn surface was investigated since fatigue wear is affected by the formation of cracks beneath the worn surfaces. Fig. 13 shows an SEM micrograph of the cross-section through the worn surface after tests using a 900 N applied load. Fig. 13 shows no evidence for visible cracks below the worn surface after the sliding tests. It has been reported [22] that plasma-nitriding produces a thin layer of an alloy nitride which results in a high hardness and also a surface stress, including residual compressive stress, of up to 750 N mm^{-2}. The high hardness of up to 1050 kgf mm^{-2} $H_{V_{(0.05)}}$ on the top surface results in a high resistance to wear, scuffing and seizing, even under poor lubrication conditions [8, 23]. Results in the literature [22, 24] indicate that the high residual stress and surface compressive stresses result in a high fatigue strength. The high hardness and compressive residual stresses in plasma-nitrided CrMo steel, therefore, are mainly responsible for the low wear rates of micro-fatigue in the boundary lubrication regime.

A significant finding in this work is the formation of micrometre-sized white areas on the worn surface

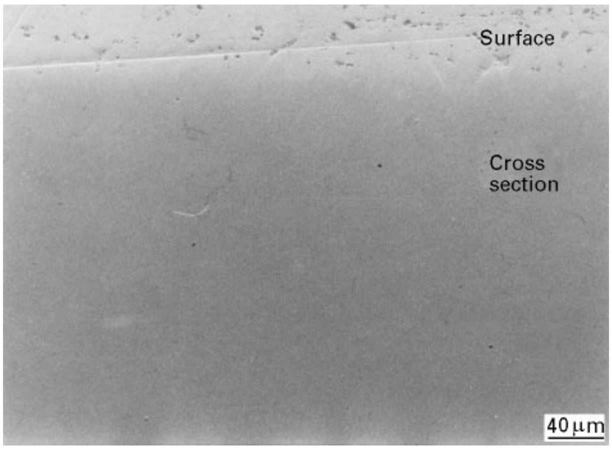

Figure 13 SEM micrographs of the cross-section through the worn surface of CrMo steel after sliding tests at 900 N applied load and a 2 m s^{-1} speed under boundary lubricated conditions in the gear oil. No cracks are apparent on the cross-section.

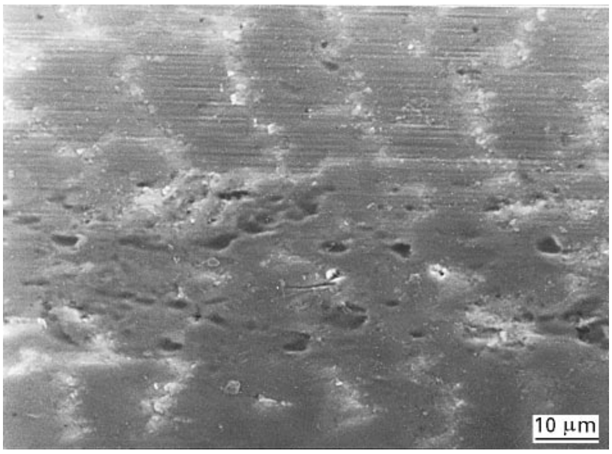

Figure 14 SEM micrograph of the worn surfaces after sliding tests of plasma-nitrided CrMo steel at a 900 N applied load in the gear oil. The micrograph shows the fishscale-like worn surface.

during the sliding of the plasma-nitrided steel at a high applied load under lubricated conditions. Fig. 14 shows an SEM micrograph taken at a high magnification on a sample after a lubricated test at a 900 N applied load. Examination of Fig. 14 indicated that there were many micrometre-scale white wave patterns transverse to the sliding direction and parallel to one another. This phenomenon, designated fish-scaling, has not been previously reported in the literature.

The polished worn surface was etched in 2% nital and re-examined in the SEM. Fig. 15 shows the SEM micrograph of the etched worn surface which is the same as that in Fig. 14. Comparing Figs 14 and 15 reveal that the white areas in Fig. 14 are the same as the white layer areas in Fig. 15. This phenomenon has also not been previously reported in the literature. In general, the hardness of a white layer is higher than that of the matrix material. The formation of the white layer areas on the sliding surface could increase the hardness on the sliding surfaces, which results in a low wear rate.

Another important finding is that the location of the micro-pits corresponds with that of the white layer areas on the worn surface. In Fig. 15, a micro-pit occurred at label A. An examination of Fig. 15 revealed that this pit was probably a location of a white layer area on the worn surface. The term “white layer” is taken to refer to not only an etch-resistant layer, but also to a hard layer formed on sliding surfaces during some machining or wear processes. Bulpett *et al.* [25] have reported that a process of grain refinement, and plastic deformation is a primary hardening mechanism for white layers formed by a thermo-mechanical process, which suggests that fatigue wear occurred in these white areas during our experiments. The existence of these white areas is worthy of further investigation.

It appears that the dominant wear mechanism for plasma-nitrided CrMo steel during lubricated sliding in a boundary lubrication regime is mild micro fatigue. During sliding, a fishscale-like sliding surface formed, which contained many white layer areas resulting in a high value of hardness at the sliding surface. The low wear rate could be attributed to the high hardness due to the formation of the white layer areas on the sliding surface and also to the high surface stresses created by the plasma-nitriding.

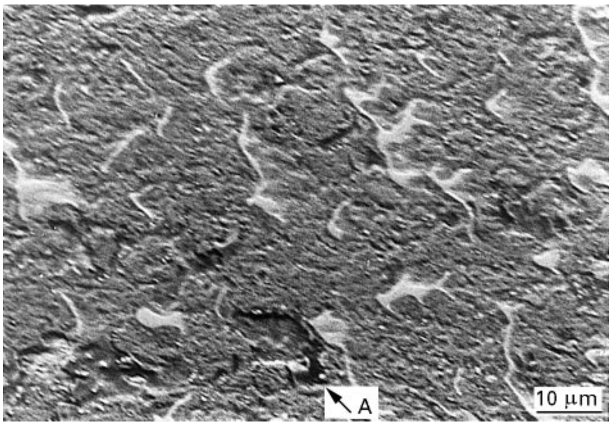

Figure 15 SEM micrograph of the etched worn surface of the pin specimen similar to that in Fig. 14, showing the correspondence of the fishscale-like worn surfaces with the formation of white layer regimes on the worn surfaces after lubricated sliding tests of plasma-nitrided CrMo steel at a 900 N applied load.

5. Conclusions

From the results presented above, the following conclusions can be drawn;

(1) Polishing occurs mainly during the sliding of plasma-nitrided CrMo steel under mixed lubricated conditions. The predominant wear mechanism under these conditions is a mild abrasive wear mechanism.

(2) Polishing is also observed during the sliding of plasma-nitrided CrMo steel under boundary lubricated conditions. It appears that the polishing can be attributed to extremely mild abrasive wear. However, a micro-fatigue wear mechanism operates for the wear process under these boundary lubricated conditions.

(3) It is found that white layer areas form on the worn surface during the sliding of plasma-nitrided CrMo steel under boundary lubricated conditions, which is attributed to micro-fatigue wear.

(4) No wear is observed during the sliding of plasma-nitrided CrMo steel under lubricated conditions in the full fluid film lubrication regime.

(5) A running-in procedure that does not incur severe wear will facilitate the observation of wear mechanisms during subsequent sliding under lubrication.

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