Tribological behaviour of plasma nitrided cast iron D6510 and cast steel S0050A under the inclined-impact sliding condition with extremely high contact pressure

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Abstract. Plasma nitriding as a surface modification was applied on two substrate materials: cast iron D6510 and cast steel S0050A. After measurement of the friction coefficients of the treated samples using a pin-on-disc tribotester, an inclined impact-sliding wear tester was utilized to investigate their tribological behaviour under tilting contact with extremely high contact pressure. While numerous surface fatigue cracks, severe chipping, and peeling of the compound layer were observed for the treated cast steel sample, the treated cast iron sample had far fewer surface fatigue cracks without chipping or peeling of the compound at the same test condition. The governing mechanisms of the treated cast iron sample’s superior resistance to surface fatigue failure were revealed by studying the cross-sectional hardness and nitrogen concentration profile. Energy-dispersive X-ray spectroscopy (EDS) analysis indicated that the treated cast iron sample had a smaller nitrogen concentration gradient, which led to a smaller hardness gradient as measured. The results suggest that a smaller hardness gradient between the compound layer and the diffusion zone and a thicker hardened case was able to improve the wear resistance and surface fatigue cracking resistance against high contact loads. Moreover, the smaller friction coefficient of the treated cast iron sample could also be beneficial for improving the wear resistance.

1. Introduction
The application of nitriding to substrate materials results in a hardened layer with limited distortion and dimensional variations [1–5], making it an efficient and cost-effective method for the surface modification of precise mechanical parts, like stamping dies and gear teeth.

The tribological behaviours of nitrided iron and steel have been extensively studied [2, 3, 5–7]. However, this knowledge may not be always appropriate to be used for the selection of die materials and surface modification methods in terms of industrial stamping application. For instance, a cast steel has usually been considered as a better material than a cast iron. But, this may not be true at some situations. A laboratory testing method should imitate the loading conditions of real applications.

For a stamping die used for metal forming of advanced high strength steel (AHSS), the loads in the working corner areas where the most wear may occur consist of both normal and tangential forces. Moreover, the maximum stress [8] at the radius of the die could be as high as 4000 MPa and the sliding energy density [9] could be as high as 0.4 J/mm². Such harsh conditions cannot be simulated by the
traditional pin-on-disk apparatus, and a new kind of wear tester is needed for the tribological evaluation of the nitrided iron and nitrided steel.

In this study, plasma nitriding was used to form case hardened layers on ductile cast iron D6510 and cast steel S0050A. After measuring the friction coefficients of the treated samples, a newly developed inclined impact-sliding wear tester [10–12] was utilized to study the tribological behaviours of the treated samples. The difference in the wear resistance of the samples was explained with respect to the characteristics of their case hardened layers.

2. Sample preparation and experiments

The materials chosen for this study were two commonly used stamping die materials: ductile cast iron NAAMS-D6510 and cast steel NAAMS-S0050A. The chemical composition of the ductile iron is 3.41 wt.% C, 2.22 wt.% Si, 0.41 wt.% Mn, 0.42 wt.% Mo, 0.56 wt.% Cu, 1.12 wt.% Ni, 0.1 wt.% Cr, 0.02 wt.% S, 0.04 wt.% P, 0.05 wt.% Mg, and the balance Fe. The chemical composition of the cast steel is 0.45 wt.% C, 0.36 wt.% Si, 1.05 wt.% Mn, 0.41 wt.% Mo, 0.90 wt.% Cr, 0.03 wt.% S, 0.03 wt.% P, 0.12 wt.% V, and the balance Fe [13].

For the specimen preparation, two sets of samples were cut into 25.4 mm × 25.4 mm × 5 mm plates and subsequently machined to ensure that they had the same surface finish. The plates were cleaned, degreased, and then plasma-nitrided in a system developed by Advanced Heat Treat Corp. with a cathodic heating shield and a pulse plasma generator of 6 kHz frequency. The plasma nitriding was conducted at 570°C for 4 hours. Heating in the plasma process from room to the final temperature was performed in a mixture of 30% nitrogen and 70% hydrogen with the total pressure of 2.9 mbar. For the sake of simplicity, the treated samples will be referred to as TI for the plasma nitrided cast iron sample and TS for the plasma nitrided cast steel sample.

Metallographic sections prepared from the nitrided samples were used for measuring hardness and nitrogen concentration profiles. The surface hardness and hardness profiles of the case hardened layers of the treated samples were determined using a Vickers microhardness tester with indentation loads of 0.0981 and 0.245 N, respectively. The nitrogen concentration profiles were measured by scanning electron microscope (SEM) equipped with energy-dispersive X-ray spectroscopy (EDS) detector (FEG 2000, 13 KV). The results were the average of 16 scan frames. The elastic moduli of the case hardened layers were studied by nanoindentation with a load of 2500 μN (Hysitron).

Pin-on-disk sliding tests (sliding velocity = 0.1 m/s, normal load = 10N) were done to determine the friction coefficients of the treated samples. The wear and surface fatigue resistance were evaluated with an inclined impact-sliding wear tester.

![Setup illustration of the inclined impact-sliding wear tester](a)

![Normal load curve for one of the test cycles](b)

Figure 1. (a) Setup illustration of the inclined impact-sliding wear tester; (b) normal load curve for one of the test cycles.

An illustration of the setup of the inclined impact-sliding wear tester is shown in Figure 1(a). A steel ball with a diameter of 9.5 mm was used as a counterface material. The applied load curve is shown in Figure 1(b), where it can be seen that the load quickly increased to 140 N and reached the maximum force of 300 N before decreasing back to zero in each cycle. The tests were carried out in an ambient...
environment without lubrication. According to the Hertzian calculation, when F = 300 N, the maximum contact pressure should be around 3.60 GPa. This stress range is close to the calculated maximum stress possible at the die working corner (about 4.0 GPa) [8], while the maximum contact stress for the Pin-on-disk (POD) sliding test is only about 1.2 GPa and the purpose of the POD test was to measure coefficient of friction in this study. The period for each inclined reciprocating sliding test cycle was 0.5 s; the numbers of test cycles were 500, 1000, 1500, and 2500 for the TI sample, and 100, 250, 500, and 1000 for the TS sample. The way of choosing test cycles was the following: firstly, both the samples were tested with 500 and 1000 cycles; after observing the surface damage conditions, to increase the test cycles was decided for the TI sample and to decrease the test cycles for the TS sample. For all the tests, the balls as the counterface materials were made of SAE 52100 hardened steel (60–62 HRC).

3. Results and discussion

3.1. Tribological performance under inclined sliding wear tests

Figure 2 shows the wear volume loss and average wear volume loss per test cycle at different numbers of test cycles. Obviously, the accumulated wear volume loss increased with test cycles for both TI and TS samples. However, the average wear volume loss per test cycle is different for different samples. For the TI sample, the average wear volume loss gradually decreased from 5.92E-6 to 2.5E-6 mm³/cycle as the number of test cycles increased from 500 to 2500; that is, the wear rate decreased as the test continued. On the other hand, the average wear volume loss of the TS sample increased sharply to 1.6E-5 mm³/cycle at 250 test cycles and then decreased quickly to 9.38E-6 mm³/cycle at 500 test cycles, indicating that there must be a change in the wear mechanisms.

Figure 2. Wear volume loss and average wear volume loss per test cycle for the TI and TS samples at different test cycles.

The SEM morphologies of the wear track on the TS sample after 500 test cycles are presented in Figure 3; B, C, and D represent the head, middle, and tail areas of the wear track, respectively. In Figure 3(b), a large area of chipping/peeling is revealed. The inset in Figure 3(e) shows a detailed image of area E in Figure 3(b), in which materials transfer and wear debris pile-up can be seen. It can also be observed that numerous surface fatigue cracks initiated and propagated across the whole wear track.

In the middle area, as shown in Figure 3(c), severe material transferring from the ball and wear debris pile-up led to form a loose transfer layer on the surface of the wear track. The inset in Figure 3(f) shows this kind of transfer layer at a higher magnification. The small stars in Figure 3(f) represent the locations of EDS point analysis, which was applied to distinguish the compound layer, the diffusion zone and the materials transfer. The results are summarized in Table 1. It should be noted that only iron, nitrogen and oxygen were counted during the EDS analysis. Therefore, the nitrogen concentration for the nitrided layer was higher (up to 30 at. % for the compound layer and 15 at. % for the diffusion zone) than the commonly-expected value, and cannot be viewed as standard reference values. But the relative
differences were sufficient to distinguish the compound layer, the diffusion zone and the materials transfer. The red star shows relatively low O and N concentrations, indicating that the compound layer has already been peeled away. The green and blue stars show relatively high O concentration without the existence of N. This indicated the materials transfer layer at the green star and the wear debris pile-up at the blue star.

In the tail area of the wear track, only slight abrasive wear was observed without surface fatigue cracks.

Figure 3. SEM observation of the wear track of the TS sample after 500 test cycles: (a) overview of the wear track; (b) head area of the wear track, the inset shows the detailed structure of the worn surface in the head area; (c) middle area of the wear track, the inset shows the detailed structure of the worn surface in the middle area and the arrangement of EDS point analysis; (d) tail area of the wear track.

The SEM observations revealed the change of wear mechanisms for the TS sample during the inclined impact-sliding wear test. At the beginning (50 to 100 cycles), the surface fatigue cracks were initiated and propagated in the head and middle areas. The cracking patterns were similar to those observed in scratch tests. When the crack density reached a critical value, the compound layer was chipped and peeled away. This process occurred first in the middle area (at around 200 to 250 cycles) and then in the head area (at around 500 cycles). This could explain the sharp increase of average wear volume loss during the 250 test cycles. Then, severe materials transfer from the ball and wear debris pile-up in the area of chipping and peeling could occur. This process reduced the wear volume loss measured. Meanwhile, chipping and peeling of the compound layer still happened in other areas. Thus, the wear volume loss still increased but at a much lower rate, as shown in Figure 2.

Table 1. EDS analysis results for the middle area of the TS sample’s wear track after 500 test cycles
|       | Fe (at.%) | N (at.%) | O (at.%) |
|-------|-----------|----------|----------|
| Red   | 80.88     | 9.11     | 10.01    |
| Green | 72.72     | NA       | 27.28    |
| Blue  | 57.61     | NA       | 42.39    |

Note: The values should only be used for comparison with each other to distinguish the compound layer, the diffusion zone and the materials transfer.

Figure 4 shows the detailed SEM images of the worn surface of the Ti sample after 2500 cycles of the test; B, C, and D represent the head, middle, and tail areas of the wear track, respectively. The head and middle areas of the wear track appear to have numerous surface fatigue cracks, large areas of material transfer, and a few very small chipping areas, as shown in Figure 4(b, c). The tail area of the wear track appears to have a little material transfer in the areas where graphite was originally located, as shown in Figure 4(d). Slight abrasive wear was also observed in this area without the existence of surface fatigue cracks.

![Figure 4. The worn surface of the Ti sample after 2500 test cycles: (a) overview of the wear track at ×30 magnification; (b) head area, (c) middle area, and (d) tail area at ×200 magnification. The dashed curve indicates the boundary of the wear scar.](attachment:figure4.png)
of this porous layer. Thus, the average wear volume loss, that is, the wear rate, was relatively high. Later on (500 cycles to 2500 cycles), surface fatigue cracks initiated and propagated in the head and middle areas, but no chipping or peeling occurred. In this period, the wear volume loss was mainly contributed by the abrasive wear of the inner compound layer with more compact structure and higher hardness. Thus the average wear volume loss per cycle, that is, the wear rate, gradually decreased. On the other hand, we also noticed that small areas of chipping and peeling started to occur after 2500 test cycles, which implies that severe surface fatigue failure may happen later (perhaps after 3000 or 5000 test cycles).

Figure 5. Worn surface of the wear track of the TI sample after 500 test cycles, showing the porous surface layer of the TI sample

3.2 Influence of the friction coefficient.

The coefficients of friction of the samples under pin-on-disk tests are plotted in Figure 6. For the treated steel sample, after an initial running-in stage with a coefficient of friction value of 0.58, the coefficient of friction reached a steady-state value of $0.74 \pm 0.2$. The low friction coefficient of the running-in stage was achieved through the existence of an iron oxide layer, which was confirmed by the EDS analysis.

Figure 6. Recorded friction coefficient curves of the treated samples.

Compared with the TS sample, the treated iron sample had a much lower friction coefficient of $0.34 \pm 0.2$. The surface-distributed graphite, which could act as a solid lubricant, would be responsible for the reduction of the friction coefficient.

Since the TI sample had a much lower friction coefficient, the sliding energy density applied on the surface of the TI sample during the inclined impact-sliding wear test should be smaller. This led to the
delayed initiation and propagation of surface fatigue cracks and thus increased the resistance to surface fatigue failure. The lower friction coefficient could also lead to smaller tangential stresses during the wear test, which was beneficial for improving the resistance to surface fatigue failure.

3.3 Influence of hardness gradient and mismatch of elastic modulus

Besides the influence of the coefficient of friction, the difference in elastic modulus and hardness gradient between the compound layer and the diffusion zone of the nitriding cases could also play an important role. The sectional nitrogen concentration profiles (which would be the main cause of hardness gradient and mismatch of elastic modulus) were measured. Here the subject interested was the difference of iron nitrides in the two samples, thus only Fe and N were counted when measured the nitrogen concentration profiles. The nitrogen concentration presented thereby was thus too high. Therefore, the values could only be considered for relative comparison. The difference of the nitrogen concentration profiles are revealed in Figure 7.

Figure 7 demonstrates the sectional nitrogen concentration profiles below the surfaces of nitrided samples. It is clear that at the boundary between the compound layer and diffusion zone (at 10–15 µm from surface), the TI sample had a much smaller nitrogen concentration gradient. Moreover, at the top of the diffusion zone (which was covering a range of 15–50 µm starting from the boundary), the TI sample had a higher nitrogen concentration.

![Figure 7](image)

Figure 7. Sectional nitrogen concentration profiles of the nitrided samples (Y scale should be considered as arbitrary).

The Vickers hardness profiles below the surfaces of the samples are plotted in Figure 8. Each of these values is the average of five measurements. It is clear that, compared with sample TS, sample TI had a higher hardness in the top area of the diffusion zone with a lower hardness-depth gradient at the boundary between the compound layer and the diffusion zone.

Nanoindentation was utilized to study the elastic modulus of the compound layer and the diffusion zone of the TI and TS samples, respectively. The indentations were made at 5 µm and 30 µm below the surface to characterize the compound layer and the diffusion zone. It is clear that the compound layers of both samples had similar elastic modulus while their diffusion zones had significantly different elastic modulus. The mismatches of the elastic modulus of the compound layer and diffusion zone were 66.2 ± 3.2 GPa for the TS sample and 31.5 ± 2.7 GPa for the TI sample.
Figure 8. Cross-sectional hardness profiles of the TI and TS samples.

Figure 9. Nanoindentation load–displacement curves for the compound layers of the TI sample (TI-CL) and TS sample (TS-CL) at 5 µm below the surfaces, as well as for the diffusion zones of the TI sample (TI-DZ) and TS sample (TS-DZ) at 30 µm below the surfaces.

The large mismatch of the elastic modulus and the high hardness gradient at the boundary between the compound layer and the diffusion zone of the TS sample had a critical influence on the elastic/plastic deformation behaviours of the compound layer and the diffusion zone. When the tested sample was subjected to extremely high contact, such as 3.6 GPa during the inclined impact-sliding wear test in this study, the corresponding strain (elastic plus plastic) of the compound layer and the diffusion zone differed considerably. This led to a concentration of applied normal stress at the interface nearby area. Combined with the tangential stress caused by the sliding friction, the total stress was high enough to initiate surface fatigue cracks very quickly. The high combined stress also accelerated the propagation of the surface fatigue cracks and thus caused severe surface fatigue failure like chipping and peeling. On the other hand, the elastic modulus mismatch and hardness gradient of the TI sample were much smaller. Therefore, the corresponding strain and stress concentration were significantly smaller during the wear test. In addition, the tangential stress caused by the sliding friction was much smaller, leading to a smaller combined stress. The lower combined stress would cause the delayed initiation and propagation of surface fatigue cracks.

From above analysis, reducing the sliding friction of the surface and decreasing hardness gradient and mismatch of elastic modulus between the compound and diffusion layers are beneficial for increasing the surface fatigue resistance of the hardened case.

4. Conclusions

During the inclined impact-sliding wear test, numerous surface fatigue cracks formed on the nitrided samples. This was due to the extremely high contact stresses and sliding energies applied on the surfaces of the treated samples during the wear test.

On contrast to the plasma nitrided steel sample, the plasma nitrided iron sample showed higher surface fatigue resistance, resulting in delayed surface fatigue failure, which was usually initiated with chipping and peeling.

The superior surface fatigue resistance of the nitrided iron sample was attributed to the smaller elastic modulus mismatch and hardness gradient at the boundary between the compound layer and the diffusion zone. The lower coefficient of friction also contributed to the improvement in the surface fatigue resistance.

The cast iron and steel samples studied in this project were treated at the same way. The plasmas nitriding process parameters may need to be modified so that it can provide an improved wear property.
for the cast steel to withstand the extremely high stressed condition (by reducing the sliding friction of surface, and lowering hardness gradient and elastic modulus mismatch between different layers, for instance).

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