

The effect of plasma nitriding and post oxidation on fretting wear behaviour of a high strength alloy steel

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Abstract. The fretting wear performance of the non-nitrided, nitrided and nitrided-post oxidized high strength alloy steel, W460 were investigated in the gross slip regime at ambient condition. Fretting wear tests were performed with an applied normal load of 250 and 650 N at a displacement amplitude of 100 μm using a cylinder-on-flat configuration. X-ray analysis (XRD) revealed the formation of the iron-nitrided Fe_3N and Fe_4N during plasma nitriding and iron oxide phases of hematite (Fe_2O_3) and magnetite (Fe_3O_4) during post-oxidation of the cylindrical steel samples. The steady state tangential force coefficient decreases when the nitrided and post-oxidized samples were fretted against the non-nitrided steel material when compared to the non-nitrided steel contact pair. The steady state tangential force coefficient decreased with an increase in applied normal load across all of the fretting conditions. The total dissipated energy and the total wear volume increased with an increase in applied normal load with total wear volume of the non-nitrided vs nitrided and non-nitrided vs nitrided post-oxidized sample pairs, showing a reduction in the wear volume of approximately 50% compared to the non-nitrided vs non-nitrided combination under the fretting conditions examined. The worn surface morphology of the fretted samples examined using a scanning electron microscope showed the presence of loose wear debris in the wear track, fragmented wear debris, delamination cracks, delamination with large discontinuities, plate-like wear debris, oxide patches and formation of large cavities.

Keywords: Fretting wear, high strength steel, plasma nitriding, plasma nitriding and post oxidation

1. Introduction

Surface strengthening of metallic materials is achieved by various methods: modifying the surface chemistry, introducing compressive residual stresses and surface hardening. Various mechanical and thermo-chemical surface modification techniques have been developed to strengthen the surfaces of engineering materials, as many machine components experience fatigue loading in service, and mostly, fail due to fatigue related mechanisms. To overcome the tribological issues of any metallic material, surface strengthening of metallic materials are continually being developed for use with specific applications such as increased surface hardness, reduced coefficient of friction and improved mechanical and tribological properties [1]. One of the most economical ways of improving the tribological and corrosion properties of high strength steels is through the use of plasma nitriding, a thermochemical process which involves the diffusion of nitrogen atoms into the steel surface at elevated temperature to



produce a compound layer and diffusion layer. A post-nitriding oxidation treatment forms superficial oxide layer over the compound layer on the surface of the metallic materials. Plasma nitriding and post-nitriding oxidation treatments find increasing application on low alloy steels, stainless steels, cast iron and tool steels due to the ability to use a lower process temperature, shorter duration of the treatment, minimal distortion to the surface and low use of energy compared to conventional surface modifications [2-3].

Fretting is characterised by small amplitude relative oscillatory motion in the order of a few tens of microns, occurring between the two normally loaded contacting solid surfaces as a result of cyclic loading and is predominant in quasi-static loaded assemblies of machine elements and engineering structures. Although the fretting wear is complicated in nature, the two most important parameters affecting the fretting mechanism are the displacement amplitude and applied normal load [4]. Mashreghi et al. [5] investigated the effect of plasma nitriding temperature and duration on the microstructure, surface hardness, wear and corrosion resistance of a quenched and tempered cold work tool steel. It was reported that plasma nitriding resulted in a significant improvement in the surface hardness, wear and corrosion resistance of the tool steel and the samples with a higher surface hardness and thicker compound layer with epsilon iron nitride ($\epsilon\text{-Fe}_3\text{N}$) phase showed better wear and corrosion resistance. It was stated by Abedi et al. [6] that plasma nitriding and plasma nitriding along with post oxidation surface treatment on AISI 316 austenitic stainless steels reduced the friction coefficient and surface roughness whereas as the oxidation treatment time increases, the friction coefficient and surface roughness increased and the wear resistance decreased. The wear resistance decreased after the oxidation treatment at high applied normal loads and the authors described that this may be attributed to the reduction in the nitride layer thickness [6]. Ramesh and Gnanamoorthy [7] investigated the influence of post-oxidation on the fretting wear behaviour of liquid nitrided structural steel, En 24 against the hardened and tempered En 31 steel and conveyed that the sample liquid nitrided and post-oxidized for 60 minutes showed a lower coefficient of friction and reduced wear volume and hence exhibited a higher resistance to fretting wear compared to the sample liquid nitrided and liquid nitrided and post-oxidized for 15 minutes due to a thicker oxide layer.

BÖHLER W460 is a recently developed ultra high strength steel, manufactured by a double vacuum melted production route, leading to an ultra clean steel with excellent mechanical properties which finds usage in motorsport and advanced engineering applications, particularly highly fatigue-loaded components such as camshafts, gears, connecting rods, crankshafts, bolts and drive shafts, all of which are susceptible to fretting wear and fretting-fatigue failure when the contact conditions prevail. The high range of tempering temperatures for W460 allows for a comprehensive range of surface treatments. However, very few research articles have been published on the friction and wear characteristics of surface modified metallic materials, especially on high strength steels under fretting conditions. In this work, the fretting wear performance of the non-nitrided (quenched and tempered), nitrided and nitrided post-oxidized W460 steel were studied using a cylinder-on-flat samples configuration under various applied normal loads and constant displacement amplitude in dry sliding conditions. The basis for this investigation is the need for high strength materials with enhanced mechanical and tribological properties, providing mechanical integrity for longer life in the engineering applications. Creating an understanding of the behaviour of the material pair at the ambient temperature will allow new materials to be used in highly fatigue loaded components and will provide a platform to better understand the role of contact conditions on the mechanisms controlling the fretting wear process.

2. Methodology

Fretting wear studies were performed on the non-nitrided, nitrided and nitrided post-oxidized BÖHLER W460 high strength chromium–molybdenum–vanadium steel at ambient temperature using a crossed cylinder-on-flat sample configuration. BÖHLER W460 steel contains 4.55 Cr, 3 Mo, 0.75 V, 0.5 C, 0.2 Si, 0.45 Mn and balance Fe by weight percentage. The flat quenched and tempered W460 samples were fretted against the cylindrical non-nitrided, nitrided and nitrided post-oxidized W460 samples. Initially, the test materials were cut into sample blanks providing an allowance for heat treatment on all

the sides. The heat treatment consisted of a preheat at 560 °C for 120 mins, 650 °C for 30 mins and 860 °C for 30 mins followed by normalising at 1050 °C for 30 mins after which samples were oil quenched; subsequently, samples were tempered at three different tempering temperatures with air cooling after each successive temperature, 550 °C for 180 mins, 580 °C for 180 mins and 580 °C for 120 mins. The flat and cylindrical samples were further machined into the required dimensions from the blanks once the heat treatment process was completed, removing the decarburised layer formed by the heat treatment process from the sample surface. The machined cylindrical samples were subjected to nitriding and nitriding post-oxidation treatment. The cylinder-on-flat test sample configuration used for the fretting studies, which results in a 10 mm line contact, is shown in Figure 1. The cylindrical samples were manufactured with a radius of 6 mm and the flat samples had a width of 10 mm. The surface hardness of the quenched and tempered W460 material was measured as 605 ± 15 HV30. The elastic modulus and Poisson's ratio of the W460 material were 206 GPa and 0.28 respectively. X-ray analysis (XRD) revealed the formation of the iron-nitrided Fe_3N and Fe_4N during plasma nitriding and iron oxide phases of hematite (Fe_2O_3) and magnetite (Fe_3O_4) during post-oxidation of the cylindrical steel sample.

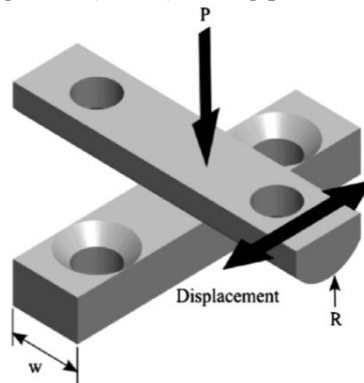


Figure 1. Cylinder-on-flat sample configuration used in the fretting experiments, where $W = 10$ mm, $R = 6$ mm, P is the applied normal load.

A detailed description of the fretting wear test setup is given elsewhere [8, 9] although a brief explanation of the test rig is presented here for ease of understanding. Initially all the fretting samples were demagnetised, thoroughly degreased using detergent, rinsed with industrial methylated spirit (IMS) and acetone and finally dried using a hot air dryer to remove the surfactant present in the samples before the start of the experiment. On the stationary lower specimen mounting block (LSMB) the flat sample was mounted, while on the moving upper specimen mounting block (USMB) the cylindrical sample was mounted. The fretting studies were conducted with applied normal loads of 250 and 650 N, with a displacement amplitude of 100 μm , at a constant frequency of 20 Hz, with relative humidity of 35 ± 5 % at ambient temperature. Table 1 shows the fretting wear test parameters used in the current study. The main components of fretting wear test rig are upper and lower specimen mounting blocks, electromagnetic vibrator, sensors and data acquisition systems. The relative displacement between the flat and cylindrical samples is produced by an electromagnetic vibrator which develops a force that is axially guided by a leaf spring. The relative displacement between the upper and lower specimen mounting block was measured using a linear variable differential transformer (LVDT). The tangential force at the contact interface are measured using the piezoelectric load cell. Normal loads were exerted on the sample by dead weights using the lever arm to the USMB. The tangential force (Q) and relative displacement (δ) during the running condition was continuously logged using the control and data acquisition system programmed in LabVIEW. Both the load sensor and displacement sensor were calibrated externally and in-situ under static conditions. To assess the surface topography of the flat and cylindrical samples a Taylor-Hobson Talysurf CL1 1000 tactile profilometer with a fine diamond stylus was used to scan the wear scar individually. Prior to scanning, the samples were rinsed with acetone to remove the loose wear debris that was not adhered to the surface. The worn surface morphology of the fretted flat and cylindrical surfaces was characterised using a Philips XL 30 SEM scanning electron

microscope to reveal more information about the nature of contact and associated damage mechanisms exhibited at the contact interface.

Table 1. Summary of the fretting wear test parameters.

Material combination (Flat/Round)	W460 – W460, W460 – W460N, W460 – W460NO
Normal load, P (N)	250 and 650
Displacement amplitude, δ (μm)	100
Number of cycles, N	100×10^3
Radius of the cylindrical sample, R (mm)	6
Frequency, f (Hz)	20
Temperature, T ($^{\circ}\text{C}$)	Ambient, 120 and 150

3. Results and discussion

The tangential force coefficient, or coefficient of friction, is defined as the ratio of tangential force amplitude (Q) to the applied normal load (P) under fretting sliding conditions. As pure sliding motion occurs between the two contacting surface in the reciprocating sliding wear process, the coefficient of friction is used to describe the friction at the interfaces whereas in fretting wear process as there is no pure sliding motion between the two members at the contact interface, tangential force coefficient is used to define friction at the contacts. The variation of the tangential force coefficient with number of cycles elapsed for the non-nitrided, nitrided and nitrided post-oxidized W460 high strength alloy steel under the applied normal loads of 250 and 650 N with displacement amplitude of 100 μm is shown in Figure 2. During the initial stages of the fretting process, the tangential force coefficient increases with the number of cycles for all the conditions examined. The tangential force coefficient for the quenched and tempered material pair (non-nitrided) is low at the beginning of the test and after the completion of a few hundred cycles, the tangential force coefficient increases rapidly and reaches a more steady state value under the conditions investigated. The initial increase in the tangential force coefficient is mainly attributed to the strong adhesion at the contact interface after the removal of the initial protective oxide films by the mechanical action. Once the initial oxide layer is interrupted there is no further formation of the oxide layer and the metal surface remains reactive resulting in high adhesion at the contact interface. This phenomenon not only results in high tangential frictional forces but in some conditions (displacement amplitudes) it can also lead to seizure [10]. Seizure is usually defined as the arrest of the relative motion between the contacting bodies as a result of the adhesive interaction of the rubbing surfaces as well as the debris entrapment. The probability of seizure reduces at high displacement amplitude due to larger relative motion between the contacting bodies as they are accommodated by elastic deformation and large variation in the tangential force coefficient with applied normal load and cycles elapsed has been observed. The tangential force coefficient of the nitrided and nitrided post-oxidized material pair at an applied normal load of 250 N and displacement amplitude of 100 μm increases initially as the cycles progressed ($< 15,000$) and then fluctuates for a period of cycles ($\sim 15,000$ to $55,000$) and reaches a more steady state value ($> 55,000$) under the conditions investigated. At an applied normal load of 650 N, the nitrided and nitrided post-oxidized material pair showed a sharp drop in the tangential force coefficient during the initial cycles with a gradual increase in the tangential force coefficient with an increase in number of cycles and as the fretting cycles progressed the tangential force coefficient fluctuates.

The steady state tangential force coefficient under fretting conditions represents the friction coefficient between the materials where the various parameters influencing the friction process attain a steady state value after initial perturbations. A steady state condition depends on the development of the wear debris, deformation and ejection of the wear debris from the contact interface by wearing down of tangentially interlocked protrusions and depressions. The average value of the tangential force

coefficient between 40×10^3 to 100×10^3 cycles was calculated for all the conditions examined to enable further comparison of the behaviour of the material pairs under different fretting conditions and is presented in Figure 3 (a). The steady state tangential force coefficient decreases with an increase in applied normal loads across all of the fretting conditions. The lower value of the steady state tangential force coefficient in the nitrided and nitrided post-oxidized W460 samples are attributed to the presence of the white iron nitride Fe_3N and Fe_4N layer on the surface formed during plasma nitriding and iron oxide phases of hematite (Fe_2O_3) and magnetite (Fe_3O_4) during post-oxidation. Figure 3 (b) shows the influence of the applied normal load and displacement amplitude on the total energy dissipated, it can be seen that the total energy dissipation increases with an increase in the normal load for the material pairs and displacement amplitude examined. The increase in total energy dissipated with applied normal load is due to the shearing of asperities at the contact interface and the frictional energy needed for the removal of the material at high loads. There is very little change in the total energy dissipation due to nitriding and post-oxidization of W460 steel. Figure 4 shows the influence of the applied normal load and displacement amplitude on the total wear volume and the individual wear volume of the flat and the cylindrical samples examined. The total wear volume decreases with the nitrided and nitrided post-oxidized samples compared to the non-nitrided test due to (i) the presence of a dense compound layer of iron nitride and iron oxide phases on the surface, (ii) an increased surface hardness that allows

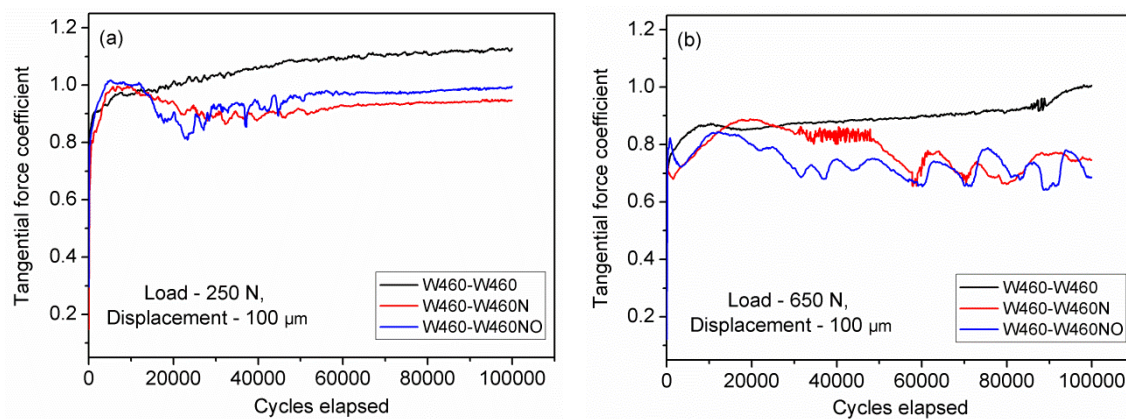


Figure 2. Variation of tangential force coefficient with cycles elapsed for different material contact pairs at (a) 250 N, 100 μm and (b) 650 N, 100 μm.

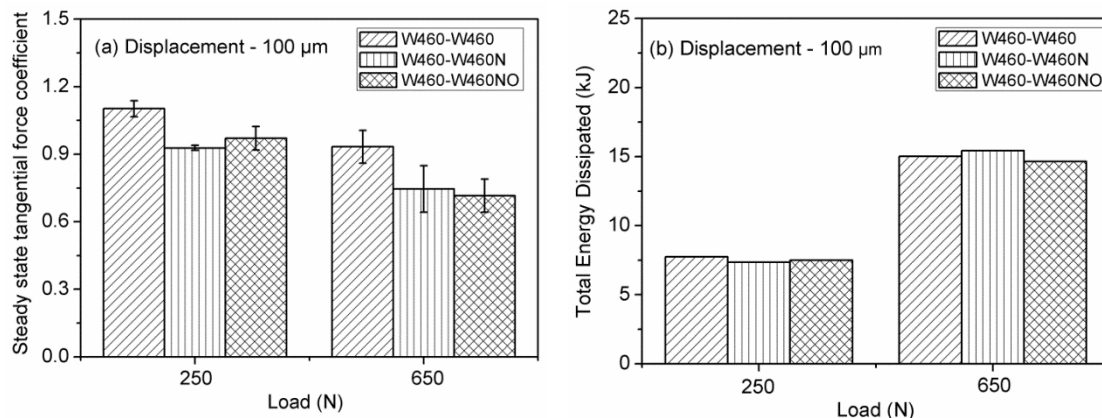


Figure 3. Influence of normal load on (a) steady state tangential force coefficient and (b) total energy dissipated.

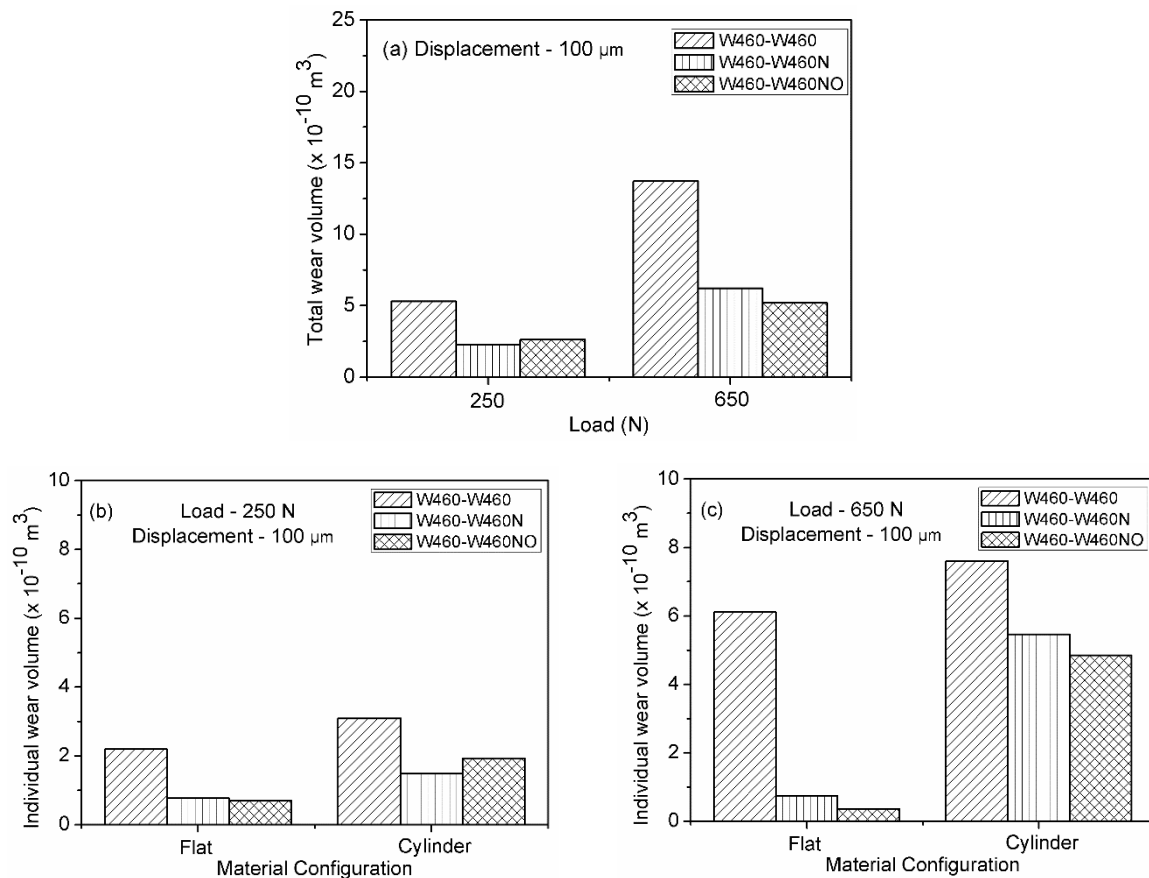


Figure 4. Influence of applied normal loads and displacement amplitude on (a) total wear volume, (b) individual wear at 250 N, 100 μm and (c) individual wear at 650 N, 100 μm .

only limited plastic deformation on the surface and (iii) a reduction in the tangential force coefficient. Similar behaviour of a decrease in the wear volume of liquid nitrided steels were reported by Ramesh and Gnanamoorthy [11] and Kato et al. [12], they suggested that the improvement in the fretting and sliding wear resistance of the nitrided steels are attributed to the presence of a thin mono-phase epsilon iron nitride layer on En24 steel [11] and En41B steel [12]. It is also reported by Ramesh and Gnanamoorthy [11] that the load at which the transition between the slip regimes occurs depends on the surface composition of the contact pair and not on the hardness of the material pair studied. At an applied normal load of 650 N, the total wear volume of the nitrided post-oxidized samples was lower than the nitrided and non-nitrided samples which is attributed to the reduction in the nitride layer thickness and prolonged oxidation time; similar behaviour was reported by Abedi et al. [6]. The oxide film formed by the post-oxidation treatment may act as a solid lubricant reducing the tangential force coefficient. At high load the post-oxidized sample had a lower tangential force coefficient and wear compared to the non-nitrided and nitrided samples, whereas at low load the tangential force coefficient and wear volume are greater. The individual wear volume of the flat samples were lower than the round W460 samples for all the conditions examined. As the applied normal load increases for the same displacement amplitude (i.e., 100 μm), the wear volume increases as the fretting contact area increases thereby increasing the total energy required to wear the fretted surface. Figure 5 shows the wear depth profile of the flat sample with an applied normal load of 250 and 650 N. The profile of the wear scars was trough-like in nature, showing U-shape for the non-nitrided samples and a W-shaped for nitrided and nitrided post-oxidized samples, indicating bulk material removal. The magnitude of the fretting damage increases with increase in applied normal load for the non-nitrided material pair as illustrated in Figures 5 (a and b), whereas there is not much increase in the fretting damage for the nitrided and nitrided

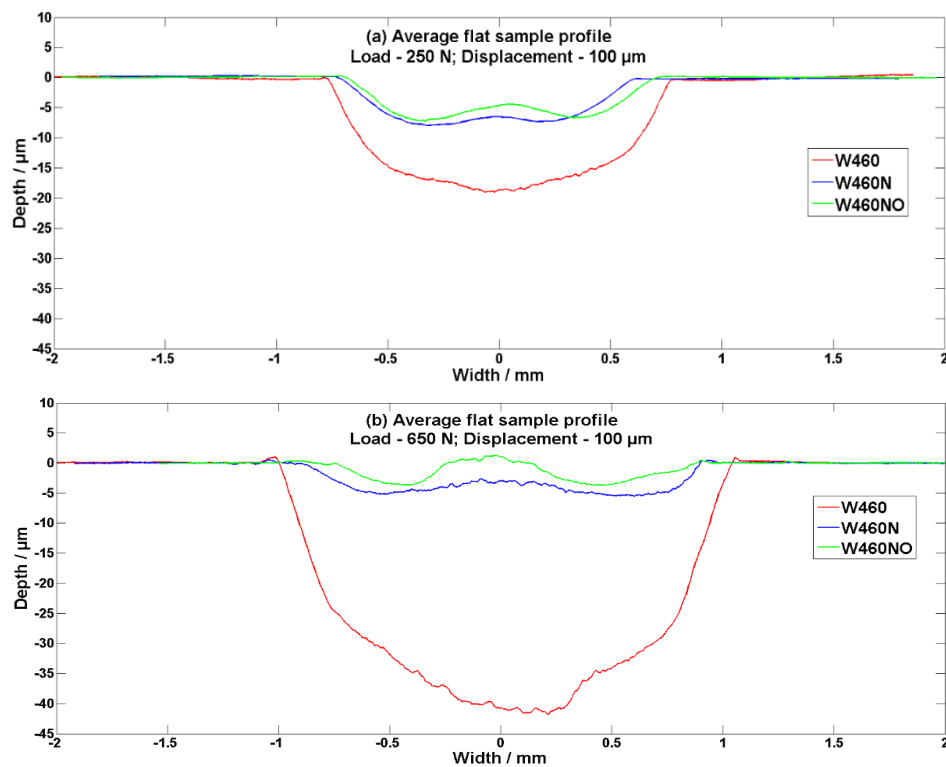


Figure 5. Wear depth profile of the flat sample for the test conducted at 100 μm displacement amplitude for different applied normal loads (a) 250 N and (b) 650 N.

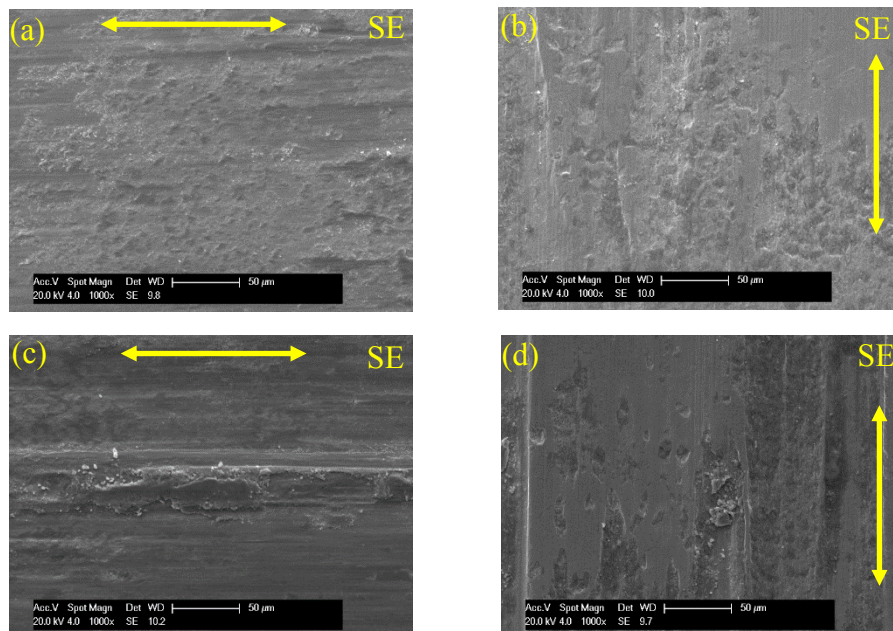


Figure 6. Worn surface morphology of the fretted sample at 250 N, 100 μm (i) W460 – W460N and (ii) W460 – W460NO; (a, c) – flat W460 sample, (b) – round W460N sample and (d) – round W460NO sample.

post-oxidized samples which are attributed to the presence of the iron-nitride and iron oxide phases. At the edge of the contact, an increase in the height of the wear depth was observed indicating either plastic deformation or embedding of wear debris taking place during the tests for all the conditions examined. Figure 6 shows the worn surface morphology of the W460 fretted against W460N and W460NO at 250 N, 100 μm . The worn surface morphology of the fretted samples showed the presence of loose wear debris in the wear track, fragmented wear debris, delamination cracks, delamination with large discontinuities, plate-like wear debris, oxide patches and formation of large cavities. Abrasion was found to be the most predominant mechanism of failure in non-nitrided, nitrided and nitrided-post oxidized W460 high strength alloy steel under the fretting conditions investigated.

4. Conclusions

The fretting wear performance of the non-nitrided, nitrided and nitrided-post oxidized W460 high strength alloy steel was investigated in the gross slip regime using a cylinder-on-flat configuration. For the conditions studied, the steady state tangential force coefficient of the nitrided and nitrided post-oxidized W460 samples was lower than the non-nitrided W460 samples. The wear volume of the nitrided and nitrided post-oxidized W460 samples was reduced by almost 50 percent compared to the non-nitrided samples due to the presence of the iron-nitrided Fe_3N and Fe_4N during plasma nitriding and iron oxide phases of hematite (Fe_2O_3) and magnetite (Fe_3O_4) during post-oxidation of W460. As the applied normal load increased for the same displacement amplitude, the wear volume increased as the fretting contact area increased thereby increasing the total energy required to wear the fretted surface. The worn surface morphology of the fretted samples showed the presence of loose wear debris in the wear track, fragmented wear debris, delamination cracks, delamination with large discontinuities, plate-like wear debris, oxide patches and formation of large cavities. Abrasion was found to be the most predominant mechanism of failure in non-nitrided, nitrided and nitrided-post oxidized W460 high strength alloy steel under the fretting conditions investigated.

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