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AN OVERVIEW ON THE TRIBOLOGICAL BEHAVIOUR OF NITRO-CARBURISED STEELS FOR VARIOUS INDUSTRIAL APPLICATIONS

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Abstract: *The term "tool steel" is used to describe a wide family of highly alloyed steels possessing special properties rendering them - from a techno-economic point of view - suitable for manufacturing of severely loaded engineering parts. Depending on its chemical composition, a tool steel grade can exhibit: (a) Extreme toughness and shock resistance, required in applications such as punches, shear knives and air-operated chisels; (b) Excellent machinability, required in the manufacture of dies; (c) High hardenability and dimensional stability under heat treatments; (d) Excellent abrasion resistance, required in applications such as brick moulds; (e) Heat resistance, required in applications such as casting and forging dies, blades for hot shearing and hot extrusion tools. However, in many applications additional surface modification of the component is often necessary, in order to enhance its lifetime when operating under sliding and/or fatigue conditions and/or in corrosive environment. Liquid nitrocarburizing is an industrial surface treatment process employed for the improvement of wear/friction and fatigue resistance of steels, since it is associated with high dimensional accuracy required in high precision tooling. Compared to conventional gas-nitriding, the liquid nitrocarburizing technique, especially Tufftriding, which involves treatment of metallic components in molten cyanide salt baths at 580 °C, is faster and produces nitride layers of higher wear/corrosion resistance and enhanced fatigue strength and toughness. The present sudy is focused on elucidating the wear mechanisms taking place during sliding friction of five (5) steel grades subjected to Trufftriding. For this purpose, three sliding friction experimental configurations were used and the friction coefficients, real-time-recorded during testing ere correlated to the post-testing microscopic observations of the worn surfaces. Finally, the experimental findings were evaluated with respect to the initial microstructure of the tool steels in their as-received and/or heat-treated state.*

Keywords: *tool steel nitrocarburising, Tufftriding, case depth, white layer, diffusion depth, friction coefficient, wear coefficient.*

1. INTRODUCTION

The term "tool steel" is used to describe a wide family of highly alloyed steels possessing special properties that render them suitable for manufacturing of severely loaded engineering components, e.g. extrusion dies and screws, guideways, shearing dies, etc. In general, a tool steel grade is delivered by the manufacturer in as-annealed state and after its proper heat treatment it can achieve hardness values up to 65 HRC, according to the requirements of each particular application. In many cases, these requirements could be further augmented, demanding thus, the enhancement of the components' surface properties, mainly, its wear, fatigue and corrosion/oxidation resistance. Such a characteristic example is the case of PVC plastic moulds, where the metallic parts are subjected to chloride attack, under alternate frictional conditions. Among other surface modification techniques, liquid nitrocarburising (Tufftriding) is an industrial thermochemical process, well-established for the improvement of the surface properties of steels and cast-iron, since it is associated with high dimensional accuracy required in high precision tooling and is less expensive and conceptually simpler than other diffusion-based or nitriding techniques [1-5].

During liquid nitrocarburising, the metallic components are immersed in molten cyanide salt baths at 580 °C, a temperature lower than the eutectoid points of both the Fe-N phase diagram (591 °C), as well as the Fe-C one (727 °C). The simultaneous interstitial diffusion of nitrogen and carbon into the ferrite lattice results in the "nitrocarburised case", with a depth varying from several tens to hundreds of micrometers, depending on the initial microstructure of the treated steel grade [6].

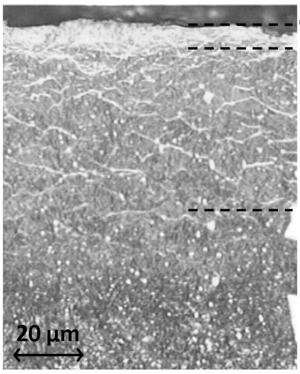


Figure 1. Typical microstructure layers of a nitrocarburised tool steel

The nitrocarburised case consists of two successive layers (Fig. 1):

(a) The outermost one, called "white layer", is grown from the diffusion of the bath's elements (C, N) in the ferrite and their simultaneous chemical reaction with the main element of the base material (Fe); thus, this compound layer is mainly composed of ε-carbonitride $Fe_{2\text{-}3}(C,N)$. The carbonitride's layer favours the wear resistance of the base material.

(b) The layer underneath, called "diffusion zone", is characterised by the nitrogen-only diffusion, since the ferrite is normally at its equilibrium concentration with respect to carbon [7]; thus, this internal layer is mainly composed of α-(Fe,N) solid solution [8,9]. Additionally, complex nitrides and/or carbonitrides, such as γ'-Fe_4N and ξ-Fe_2N, can be precipitated in both the white layer and the diffusion zone. The interstitially diffused nitrogen at this zone inserts compressive stresses that improve the steel's fatigue resistance.

The present study is focused on the comparison of the tribological performance of five (5) representative steel grades after heat treatment and/or nitrocarburising under industrial conditions. For this purpose, three sliding friction experimental configurations were used and the non-surface-treated state of the materials was evaluated as a reference to assess the wear resistance improvement due to nitrocarburising.

2. EXPERIMENTAL DETAILS

Five (5) highly alloyed steel grades (A, B, C, D and E) have been examined in soft annealed state and/or after their proper heat treatments, according to their supplier's recommendations, to a series of targeted hardness values. Their nominal chemical compositions are presented in Table 1, together with their state and the hardness achieved. The first four (A, B, C, and D) correspond to tool steel grades commonly used for cold or hot working applications, whilst the last one (E) to a precipitation hardening (PH) stainless steel. As some of these materials do not directly correspond to a standardised grade, a general steel coding (A, B, C, D and E) with respect to the chemical composition is adopted.

Before any heat and/or surface treatment, specimens from grades A, B and E were sectioned from cylindrical bars of 40 mm diameter to disks of 10 mm thickness and subsequently polished to 1.0 μm average surface roughness (*Ra*). In the case of steels A, C and D, specimens were also shaped from cylindrical bars to the testing-proper form of pins with 8 mm diameter and 50 mm length.

The specimens in their final shape have been heat treated (or not) to the targeted hardness values presented in Table 1. After heat treatment, half part of each steel's quality was kept as reference material after further tribological testing and the other half was subsequently liquid nitrocarburised under the same industrially-relevant conditions.

The surface treatment (Tufftriding) of the specimens was carried out in three steps:

(a) preheating,
(b) immersion in molten salt bath, consisting of 60 % KCN, 24 % KCl and 16 % K_2CO_3 by weight, at 580 °C for 4 h and
(c) controlled cooling.

The tribological tests were carried out in three different experimental setups (I, II and III), by applying various testing parameters' values, as summarised in Table 2.

Tests for the experimental setups (I) and (II) were performed using a point contact apparatus (Centre Suiss D' Electronique et de

Table 1. Nominal chemical compositions (wt%) of the examined steel grades

<table>
<tr><th>Steel</th><th>C</th><th>Si</th><th>Mn</th><th>Cr</th><th>Mo</th><th>V</th><th>W</th><th>Ni</th><th>Al</th><th>Fe</th><th>State</th><th>Mean Hardness</th></tr>
<tr><td rowspan="2">A</td><td rowspan="2">0.39</td><td rowspan="2">1.00</td><td rowspan="2">0.4</td><td rowspan="2">5.2</td><td rowspan="2">1.4</td><td rowspan="2">0.9</td><td rowspan="2">-</td><td rowspan="2">-</td><td rowspan="2">-</td><td rowspan="2">bal.</td><td>Annealed</td><td>180 HB</td></tr>
<tr><td>Heat Treated</td><td>40 HRC
45 HRC
50 HRC</td></tr>
<tr><td>B</td><td>0.90</td><td>0.90</td><td>0.5</td><td>7.8</td><td>2.5</td><td>0.5</td><td>-</td><td>-</td><td>-</td><td>bal.</td><td>Heat Treated</td><td>40 HRC
45 HRC
50 HRC
60 HRC</td></tr>
<tr><td>C</td><td>2.05</td><td>0.30</td><td>0.8</td><td>12.5</td><td>-</td><td>-</td><td>1.3</td><td>-</td><td>-</td><td>bal.</td><td>Annealed</td><td>240 HB</td></tr>
<tr><td>D</td><td>0.60</td><td>0.35</td><td>0.8</td><td>4.5</td><td>0.5</td><td>0.2</td><td>-</td><td>-</td><td>-</td><td>bal.</td><td>Annealed</td><td>200 HB</td></tr>
<tr><td rowspan="2">E</td><td rowspan="2">0.03</td><td rowspan="2">0.30</td><td rowspan="2">0.3</td><td rowspan="2">12.0</td><td rowspan="2">1.4</td><td rowspan="2">-</td><td rowspan="2">-</td><td rowspan="2">9.2</td><td rowspan="2">1.6</td><td rowspan="2">bal.</td><td>Solution-treated</td><td>325 HV</td></tr>
<tr><td>Age-hardened</td><td>550 HV</td></tr>
</table>

Table 2. Experimental plan for the comparative study of the tribological behaviour of the steels examined

EXPERIMENTAL SETUP			TESTING PARAMETERS		
	Examined steel's shape	Contact area	Counterbody	Applied load [MPa]	Sliding speed [m/s]
(I) Ball-on-disk	Disk	Point (non-conformal)	Al_2O_3 ball (Ø6)/ Hardness: 15.7 GPa	1 2 5 10	0.20
(II) Pin-on-disk	Disk	Point (non-conformal)	75 % WC-15 % TiC-10 % Co insert/ Hardness: 1500 HV	5 6 8 10	0.60
(III) Pin-on-disk	Pin	Plane (conformal)	AISI D6 tool steel disk/ Heat treated to 65 HRC	53 159 265	2.68

Microtechnique, CSEM), described in our previous work [10], whilst those for the experimental setup (III) were performed using a pin-on-disk tester (Plint-Cameron), described in [11].

In the case of point contact wear tests (setups I and II), the wear volume was estimated after each test by measuring with a stylus profilometer (Taylor-Hobson) the track cross-sectional area at different locations and by multiplying the average track area by the circumference of each slide circle. In the case of plane contact (setup III), the volume loss was determined from the length's decrease of the examined pin.

Microstructure characterisation of the materials under study was carried out using an Olympus BX60 metallurgical microscope, after the typical metallographic preparation of the respective cross-sections. In-depth Vickers microhardness measurements were carried out on a SHIMADZU-M apparatus, applying a load of 0.3 kg. Detailed analysis of the worn surfaces was conducted with the aid of JEOL JSM 5900 LV and JEOL JSM 6300 LV Scanning Electron Microscopes (SEM), both equipped with X-ray microanalysis.

3. RESUTLS AND DISCUSSION

3.1 Microstructure characterisation

The initial microstructure of the five steel grades examined, together with the in-depth microhardness distribution of all the nitrocarburised qualities examined is presented in Figure 2. A first remark, common for the four tool steel grades A, B, C and D, is that as they contain significant amounts of carbide-forming elements, e.g. Cr, Mo, V and W, their initial microstructure is characterised by the kind, the size and the dispersion of carbides within the metallic matrix:

- Steel (A) is characterised by the fine dispersion of small-size spherical V carbides.
- Steel (B) is characterised by the presence of coarse and irregularly-shaped eutectic Cr carbides.
- Steel (C), containing much higher Cr, W and C percentages, is characterised by the coexistence of large-sized and irregularly-shaped chromium carbides that have been identified to be Cr_7C_3 [12], as well as finer W carbides.
- Steel (D), containing much lower Cr and C compared to (C), forms a network of uniformly dispersed fine carbides.

For the above steel grades, heat treatment was found not to affect the kind, the shape or the distribution of these carbides.

In the case of PH stainless steel (E) supplied in solution-treated state, the microstructure is characterised as martensitic of the Fe-Ni system. After ageing treatment under the conditions in this work, hardening of the steel was achieved via the precipitation of the hard intermetallic compound NiAl [13].

After Tufftriding for 4 hours, in all cases the top "white layer" formed had a thickness of about 10 – 15 μm (Fig. 1, corresponding to nitrocarburised (B) steel grade) and is composed, mainly of ε-carbonitride of the type $Fe_{2\text{-}3}(C,N)$. The microstructure features observed within the diffusion zone underneath depend strongly on the initial microstructure of each particular steel and have been described in details in our previous works [14-17]. A common observation for all the cases is that the nitrides precipitations are distributed mainly along the steels' prior grain boundaries. The thickness of the diffusion zone as defined by microscopic observations, as well as in-depth microhardness measurements, was found to depend not only on the chemical composition of the steel but also on its previous heat treatment. It is interesting to note (Fig. 2) that for the same steel grade, by increasing the hardening level of the bulk material the maximum hardness and the depth of the diffusion zone also increase. For example, in the case of steel (A) in its soft-annealing state the maximum microhardness after nitrocarburising was ~800 HV0.3. The same steel submitted to heat hardening up to 50 HRC prior to nitrocarburisation, exhibited a maximum microhardness value in the nitrocarburised case of ~1000 HV0.3. The

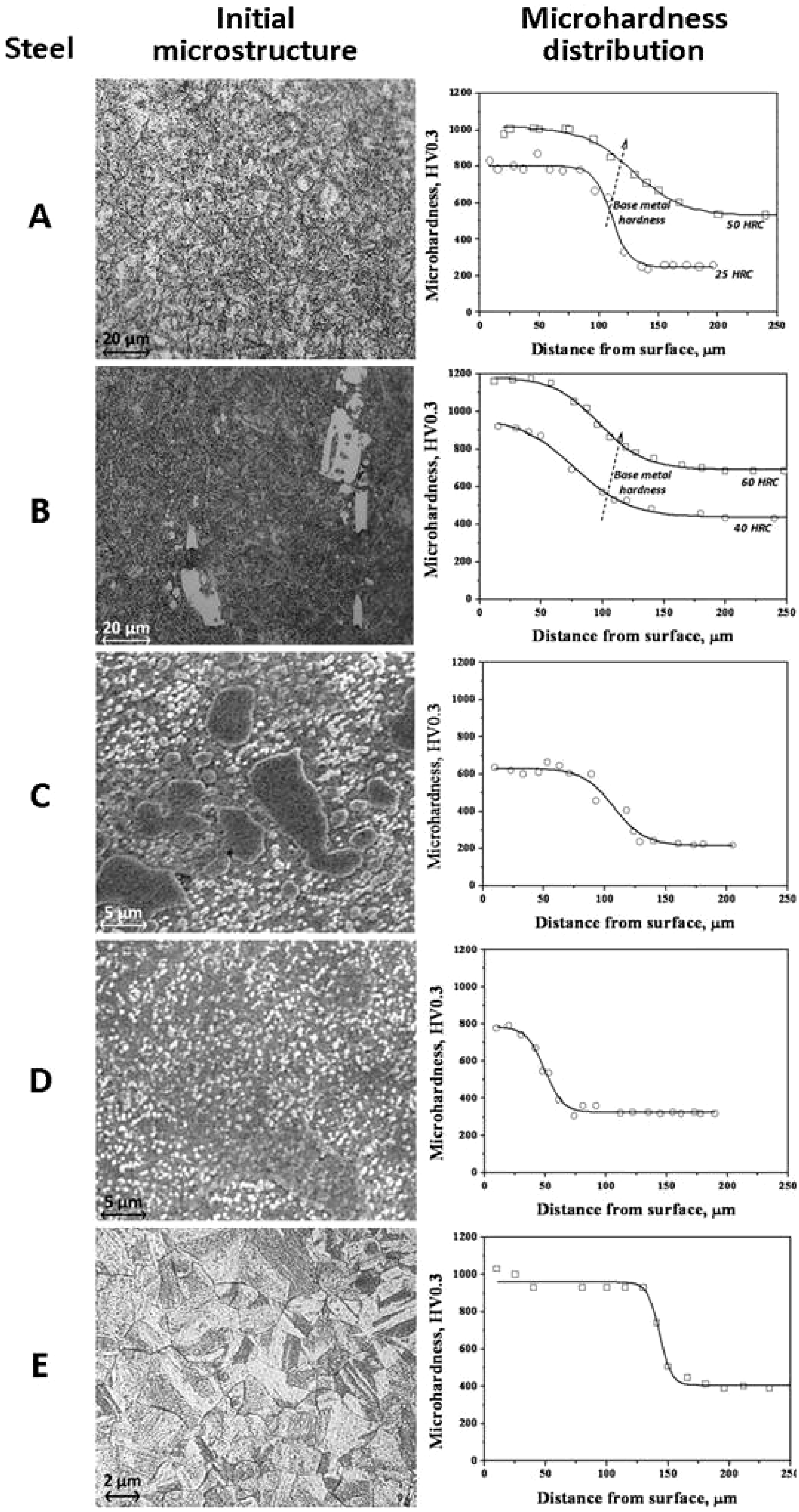


Figure 2. Initial microstructure and in-depth microhardness distribution after nitrocarburising of the five (5) steel grades examined

excess hardness achieved following nitrogen diffusion and solution or nitride precipitation could be correlated with the outward carbon diffusion from high carbon concentration regions of the matrix into the compound layer [7], due possibly to the additive effect of the metastable tempered martensite due to non-equilibrium carbon concentration in martensitic areas.

3.2 Non-conformal point contact

The (A), (B) and (E) steel grades in all hardening states, before and after nitrocarburising were tested in configurations (I) and (II) that ensured point contact at stationary conditions. Nitrocarburising was applied after the final heat treatment for steels A and B and age hardening for steel E.

The friction coefficient values recorded during steady-state sliding are presented in Figure 3. For both tool steel grades (A) and (B) the friction coefficient was constant, irrespective of the normal load applied and the state of the tested material (heat treated or nitrocarburised). For the steel (A)/Al_2O_3 ball tribosystem, the dry sliding friction coefficient was 0.95 (Fig. 3a), whilst that of steel (B) tested in the same configuration (I) was 0.78 (Fig. 3b). Deviations from this generic trend in the case of heat-only-treated steel (B) tested under low load, were correlated to the presence of large chromium carbides within the metallic matrix and have been discussed in details previously [15]. For the steel (B)/WC-based insert tribosystem, the dry sliding friction coefficient was slightly lower: 0.72 (Fig. 3b) [16]. In the case of stainless steel (E) tested in configuration (II), the sliding friction coefficient was also independent of the applied load, whilst nitrocarburising led to a slight shifting towards higher values: from 0.62 to 0.71 (Fig. 3c) [16].

As previously, for all the four tribosystems examined, the wear coefficient was practically constant regardless of the normal load applied (Fig. 4). Previous heat treatment, aiming to harden the three grades examined, was found to have a significant influence only in the case of age-hardened steel (E). Similarly, after nitrocarburising the wear coefficient tended to the same value for each steel grade, irrespectively

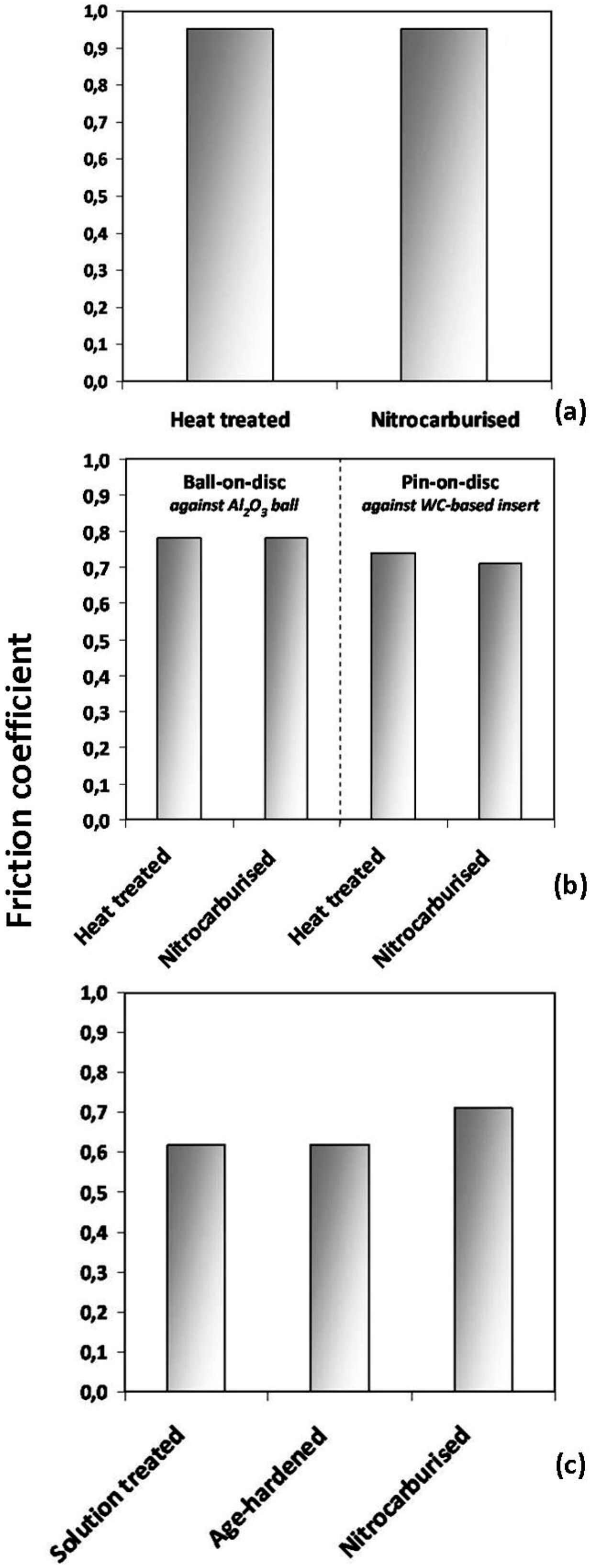


Figure 3. Friction coefficient values for various tribosystems: (a) steel (A) against Al_2O_3 ball, (b) steel (B) against Al_2O_3 ball and against WC-based insert and (c) steel (E) against WC-based insert

of the normal load applied and/or the initial bulk hardness of the base material. As is clearly shown in Figure 4, the particular surface treatment led to a drastic reduction of the wear coefficient by an order of magnitude compared to the respective value of the same steel in its heat-only-treated state.

For all the grades and for both testing configurations, the predominant wear mechanisms identified on the worn surface of heat-only-treated steels were those of micro-ploughing and oxidation (Fig. 5). In the case of surface-treated steels tested under the same conditions and duration, the top “white layer“ has not been totally removed and only shallow polishing lines can be observed within the wear track, together with sparse, random small exfoliation regions (Fig. 6).

3.3 Conformal plane contact

The (A), (C) and (D) steel grades in their soft-annealing state and after nitrocarburising were tested in configuration (III) that ensured plane contact.

The evolution of the wear coefficient of the three grades against AISI D6 hardened to 65 HRC is presented in Figure 7. A common remark for the non-treated grades is that for low pressure applied, the wear coefficient presents a minimum value. There seems to be a first threshold, above which it tends to be constant, independent of the pressure applied. Above a second pressure threshold, seizure occurs, leading to failure of the relative motion of the pairs of the tribosystem, an effect attributed to interfacial friction [17] and the

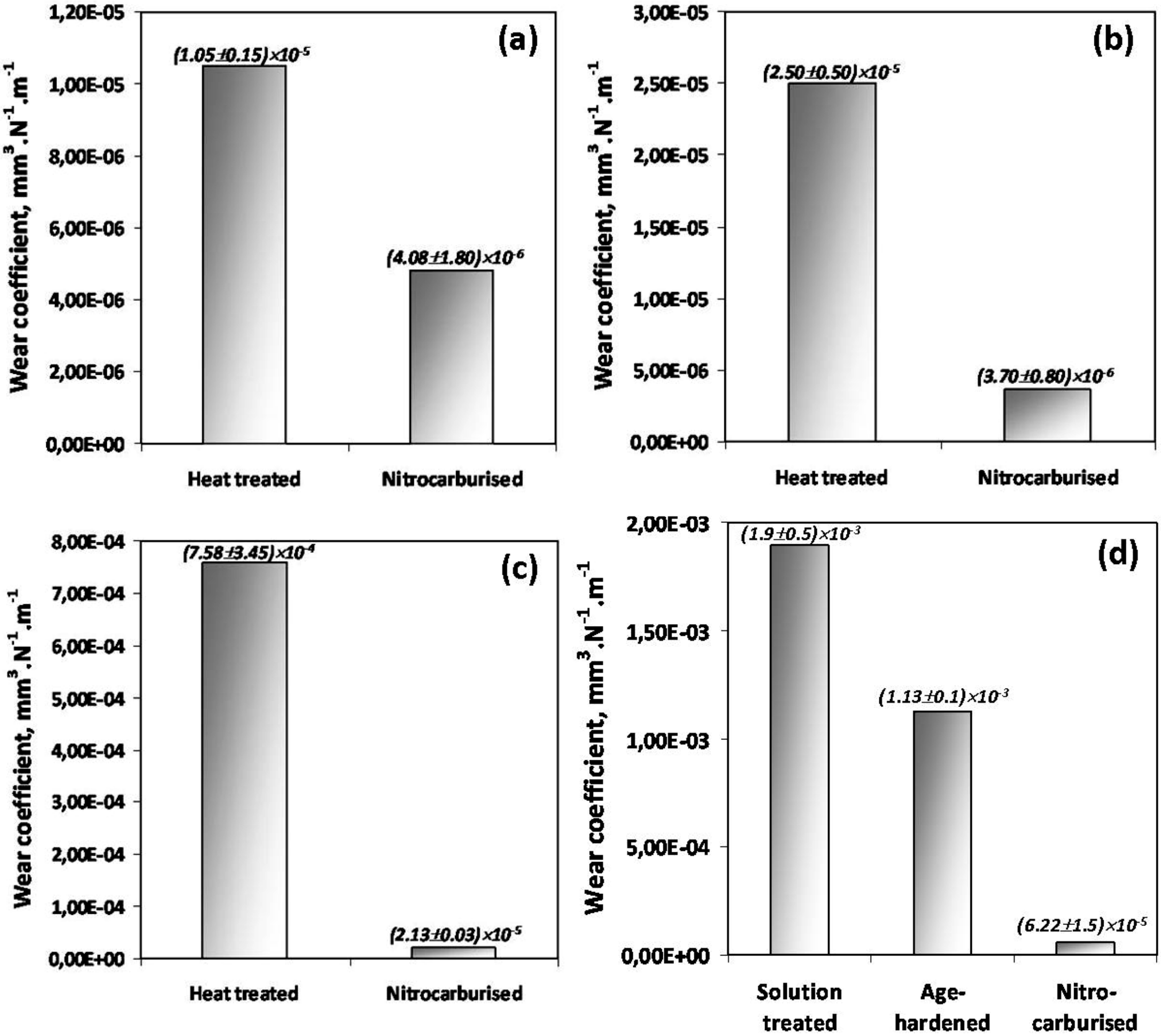


Figure 4. Wear coefficient values for various tribosystems: (a) steel (A) against Al_2O_3 ball, (b) steel (B) against Al_2O_3 ball, (C) steel (B) against WC-based insert and (c) steel (E) against WC-based insert

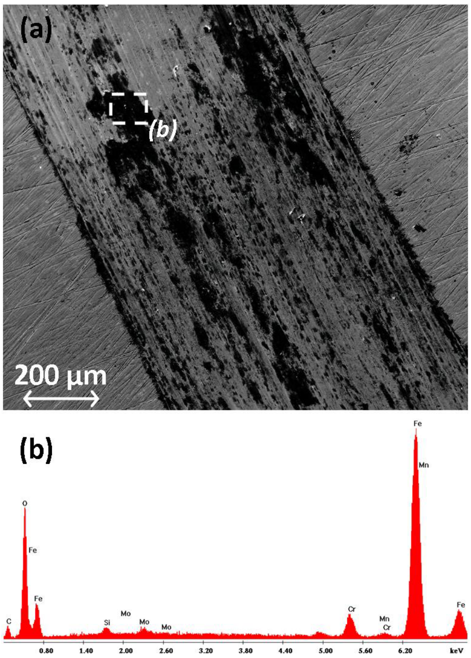


Figure 5. (a) Characteristic SEM micrograph of the worn surface of heat-only-treated steel (A), tested in configuration (I) and (b) EDS spectrum of the area marked in (a)

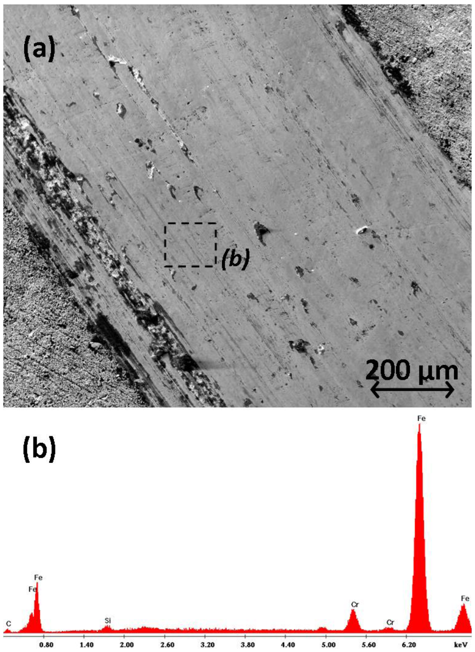


Figure 6. (a) Characteristic SEM micrograph of the worn surface of nitrocarburised steel (A), tested in configuration (II) and (b) EDS spectrum of the area marked in (a)

metallurgical phenomena implied, mainly recrystallisation and interdiffusion between metals in contact that are enhanced by the temperature increase during sliding [18]. In the present study and for the range of pressure values applied, seizure was recorded only in the case of steel (D), which was of the same chemical composition with that of the counterbody (Fig. 7a). For the dissimilar steel grades (A) and (D), this second threshold seems to have been shifted toward higher pressure values. In the case of nitrocarburised steels, a similar pattern of the evolution of wear coefficient is indicated and the respective plateaus were found to be one order of magnitude lower than those of the respective steel in soft-annealed state (Fig 7b), whilst no seizure was reported. Studies on the detrimental phenomenon of seizure are mainly focused on the interpretation of the frictional forces developed at the contact area, which contribute to the rupture of the oxide layer and promote metal-to-metal contact at elevated temperatures. However, it seems that also the wear coefficient evolution tracking could provide evidence of a forthcoming failure. A schematic approach of the above is presented in Figure 8.

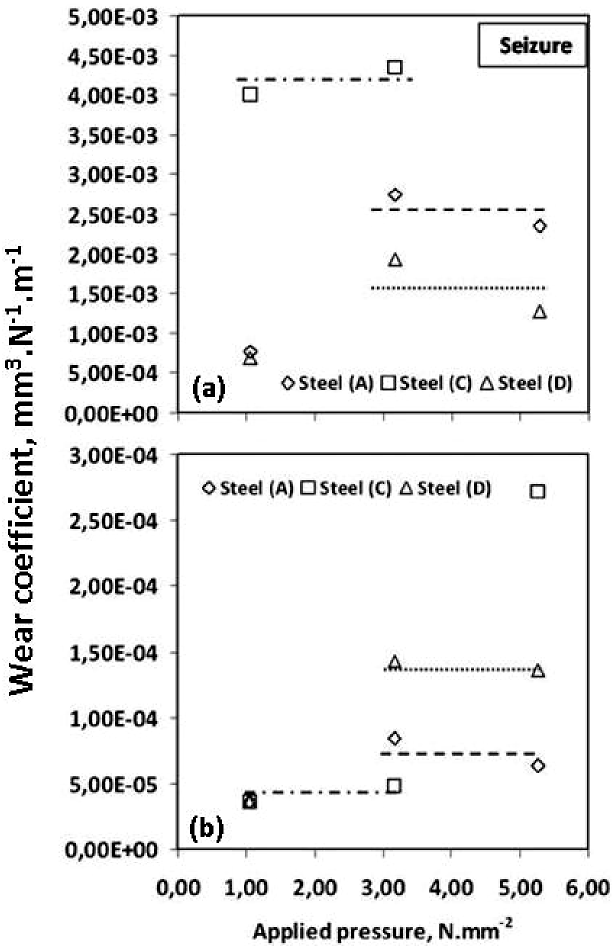


Figure 7. Evolution of wear coefficient of the three steel grades examined in conformal configuration (III) versus the applied pressure: (a) in soft-annealing state, (b) after nitrocarburising

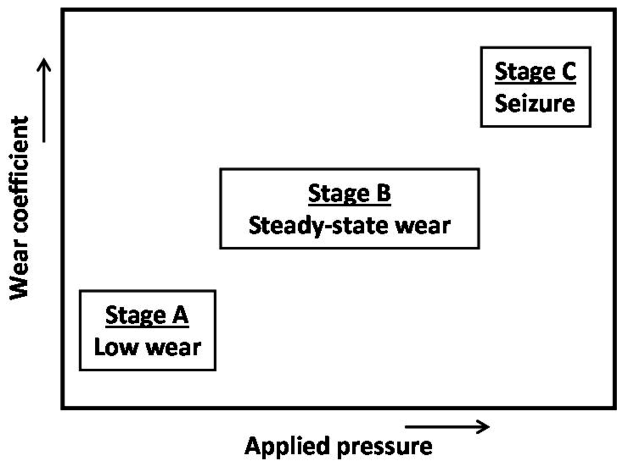


Figure 8. Schematic of the wear coefficient evolution versus the applied pressure, for steel-on-steel plane contact sliding

A detailed study on the surface wear and the subsurface degradation mechanisms of the three particular steel grades has been reported in [14]. As a recapitulated example, characteristic SEM micrographs of the worn surfaces are presented in Figure 9.

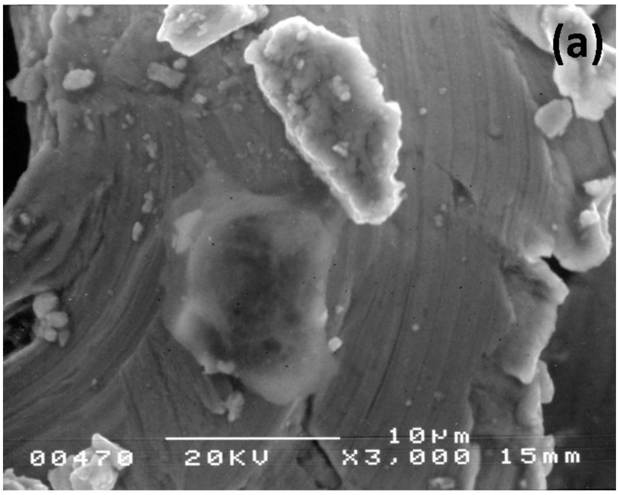
For the three steels in soft-annealed state, extensive plastic deformation and oxidation were observed on the worn surface (Fig. 9a); whilst in the case of nitrocarburised state, the extensive cracking of the compound layer (Fig. 9b) promoted the sub-surface degradation and internal oxidation of the diffusion zone.

4. CONCLUSION

Five different highly alloyed steels, - four tool steel and one stainless steel grade - have been tested under non-conformal and conformal sliding conditions. Prior to tribological characterisation, the materials have been heat-treated and nitrocarburised. Microstructure characterisation and in-depth microhardness measurements revealed the increase of case depth and maximum hardness values, with the increase of hardness levels achieved by prior heat treatment.

In the case of non-conformal point contact against ceramic counterbodies:

- The friction coefficient depends only on the steel grade and the particular counterbody and it tends to a constant value, regardless of the applied load and/or its heat- or surface treatment. Only in the case of steel (E), a slight increase of the friction coefficient after nitrocarburising has been recorded.
- The wear coefficient depends also strongly on the steel grade, while the steel's particular microstructure influences its wear micro-mechanisms.
- In all cases, nitrocarburising leads to a reduction of the wear coefficient by an order of magnitude.
- Ploughing and extensive oxidation of the contact area are the predominant mechanisms of the heat-treated grades; whilst those of the nitrocarburised steels are polishing and random exfoliation.



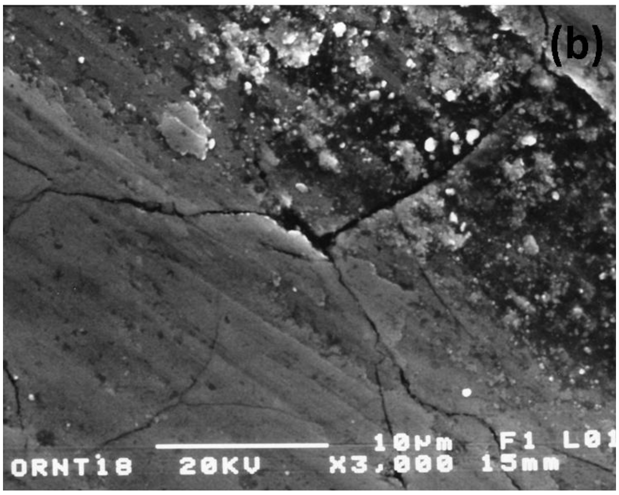


Figure 9. Characteristic micrographs of the contact surfaces after steel-on-steel plane contact sliding tests: (a) worn surface of soft-annealed steel (C) showing deformation patterns, (b) worn surface of nitrocarburised steel (C); branched cracks appeared as a precursor of exfoliation and spalling

In the case of conformal plane contact against hardened steel:

- For each steel grade, there is an applied pressure range at which the wear coefficient remains practically constant. For higher pressure values, seizure occurs.
- Seizure failure is promoted when similar metallic pairs are in sliding contact, occurring even at lower pressure values.
- Nitrocarburising “displaces” constant wear coefficient range towards higher pressure values, resulting in seizure retardation.
- Plastic deformation and oxidation of surface is predominant in the case of soft-annealed grades; whilst cracking of the compound layer and sub-surface oxidation are the main causes for the degradation of the nitrocarburised grades.

These comparative experimental results, showed a strong evidence of the feasibility to propose a holistic approach on the tribological behaviour of nitrocarburised tool steels, thus it would be worth to be verified for further tribo-pairs.

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