Effect of abrasive particle size on friction and wear behaviour of various microstructures of 25CD4 steel

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Abstract. Many parameters, such as normal load and material bulk hardness, control the wear and friction behaviours of materials. Nonetheless, the investigation of the coupled contributions of microstructure and abrasive particle size are still lacking. A contribution is proposed by using steel pins with various microstructures with a similar macro-hardness (around 410HV) and chemical composition. A quenched martensitic microstructure, a tempered martensitic microstructure and three ferrite-martensite dual-phase microstructures, with a similar martensite volume fraction (around 67%) and different martensite colony morphologies, are established. Friction tests are performed between these pins and abrasive papers with different sizes ranging from 15μm to 200μm. Compared to single-phase microstructures (quenched and tempered martensitic microstructures) and whatever the abrasive particle size, ferrite-martensite dual-phase microstructures reduce the friction coefficient and provide better wear resistance. For the ferrite-martensite dual-phase microstructures and unlike fine and fibrous martensite colonies, coarse and granular martensite colonies minimize the friction coefficient. In addition, characterized by a change of wear mechanisms between abrasion and adhesion, an intermediate abrasive particle around 35 μm minimizes the friction coefficient. This study also reveals that the wear rate increases with the abrasive particle size which is associated to an increase of the attack angle of abrasive grains.

1. Introduction

There is an increasing interest to reduce friction and wear in industrial applications. Excessive wear of mechanical parts requires their replacement. In addition, considerable energy losses are consumed to overcome friction which represent major economic losses [1]. For instance, a better understanding of the effect of roughness on friction and wear behaviour could contribute to reduce friction and wear [2-6]. Previous works aim to study the effect of the abrasive particle size on the tribological behaviour but without considering the material properties [2-4]. The bulk hardness is mainly used to control friction and wear resistance [7-8]. Indeed, Archard claims that as the hardness increases, wear rate decreases. However, other parameters such as the chemical compositions and the microstructures are also strongly linked to the tribological behaviour [5-6][9-16]. Compared to bainitic, pearlitic and ferritic microstructures, it is well known that steels with a martensitic microstructure, characterized by a high hardness, are mainly used to assure good wear resistance. Nonetheless, efforts are devoted to find microstructures which present better combinations between toughness and ductility and therefore less brittle structures. For example, investigations are done on tempered martensitic microstructures and
ferrite-martensite dual-phase microstructures [12-15]. Mainly used in the automotive industry because of its very interesting mechanical properties, steels with dual-phase microstructures present a good combination between ductility with a soft ferrite matrix and strength with hard martensite colonies. So far, tribological behaviours of various microstructures are compared but without fixing the hardness and/or the chemical composition. In addition, these investigations on the tribological behaviour are focused to the wear without considering the friction behaviour. Therefore, this work proposes a contribution in studying the effect of microstructures with the same hardness level and chemical composition on friction and wear mechanisms. In addition, the coupled effects of microstructure and surface roughness on the tribological behaviour of the studied low alloy steel are studied.

2. Material and methods
This work aims to investigate the tribological behaviour of a low alloy steel. Table 1 shows the chemical composition of this steel. Five cylindrical pins, with 10mm in diameter and length, are cut from a rod of this steel.

| Table 1. Chemical composition of the low alloy steel. |
|---------|-----|-----|-----|-----|-----|-----|-----|
| C       | Si  | Mn  | P   | S   | Cr  | Mo  |
| 0.22-0.29 | ≤0.40 | 0.40-0.90 | ≤0.035 | ≤0.035 | 0.9-1.20 | 0.15-0.30 |

These steel pins undergo various heat treatments to generate various microstructures. These microstructures include three martensite-ferrite dual-phase microstructures (DP1, DP2 and DP3), a quenched martensitic microstructure (QM) and a tempered martensitic microstructure (TM). The procedures of the performed heat-treatments are presented in Figure 1.

Figure 1(a-c) show the different heat treatments used to develop ferrite-martensite dual-phase microstructures with various martensite colony morphologies. A step quench, an intercritical annealing and an intermediate quench heat treatments are performed to generate these microstructures. In Figure 1a, it is schematically drawn that the first dual-phase microstructure DP1 is obtained by using a step quench starting by an autenitization (at 900°C during 30 minutes) followed by an intercritical annealing (at 725°C during 3 minutes) and a water quench. In Figure 1b, it is schematically drawn that the second dual-phase microstructure DP2 is obtained by using an intercritical annealing starting by an autenitization (at 900°C during 30 minutes) and a slow cooling. Then, the specimen is intercritical annealed (at 790°C during 17 minutes) and then water quenched. In Figure 1c, it is schematically drawn that the third dual-phase microstructure DP3 is obtained by using an intermediate quench starting by an autenitisation (at 900°C during 30 minutes) and a water quench. Then, the specimen is intercritical annealed (at 790°C during 12 minutes 30) and then water quenched.

Figure 1d shows the heat treatment used to generate a quenched martensite microstructure QM. This heat treatment starts by an austenitization (at 1100°C during 2 hours) followed by a water quench.

Finally, Figure 1e shows the heat treatment used to generate a tempered martensitic microstructure TM. This heat treatment starts by an austenitization (at 900°C during 2 hours) followed by a water quench and then a tempering (at 400°C during 2 hours).
Figure 1. The schematic drawings of the heat treatments of the (a) DP1, (b) DP2, (c) DP3, (d) QM and (e) TM microstructures.

To characterize the microstructures, the heat-treated pins are ground mechanically. First, the bulk material structure is reached by using grinding papers of 80 grit to remove 1mm of material from the surface. Second, the surfaces are ground mechanically on SiC grinding papers (whose grit is ranging from 320 to 4000). Third, the surface pins are polished with diamond paste of 1μm. Finally, the pins are etched with a 1% Nital solution during 10s after having cleaned by ultrasound with ethanol during 10min. For the ferrite-martensite dual-phase microstructures, the martensite volume fractions are determined by image processing using ImageJ. Thus, the martensite volume fraction is averaged from ten micrographs done with a Field Emission Electronic Gun-Scanning Electron Microscope (FEG-SEM) ZEISS Sigma at 15keV.

Under a load of 10kg, a vickers indenter is used to characterize the macro-hardness of each microstructure. From different locations, three measurements are averaged.
Before the friction tests, the heat-treated pins are ground mechanically on SiC grinding papers with a grit ranging from 320 to 4000 grit and cleaned with ethanol by ultrasound for 10 min. Thus, the surface pins present the same initial roughness.

Then, dry friction tests are performed between the steel pin and an emery paper according to a pin-on-plate configuration and a linear reciprocating motion. The used emery papers embedded hard abrasive Struers silicon carbide particles (with particle sizes of 15μm, 35μm and 200μm). The variation of the abrasive particle size aims to study the roughness effect on the friction and wear behaviour of the heat-treated pins.

These friction tests are performed at room temperature (23°C) in air, at a relative humidity of 50-60%, under a pressure of 1.6MPa, a linear velocity of 0.06m/s and a frequency of 2Hz. The total sliding distance is 18m and the stroke length is 15mm. During these friction tests, average friction coefficients and wear rates are determined from three measurements. The friction coefficient is calculated as the ratio between the tangential force $F_T$ and the normal force $F_N$ as function of the test time. By using a load cell and a piezoelectric transfer transducer, $F_T$ and $F_N$ are measured each 0.1s. For each configuration, an average friction coefficient is determined for the steady-state. Then, after having ultrasonic cleaned the heat-treated steel pins with ethanol for 10 min, the pin mass is measured before and after the friction test. The wear rate is calculated from these pin mass losses measured by using a direct reading balance (with a minimum scale of 0.1 mg). The wear rate $Q$ is calculated as the ratio between the cumulative lost volume $V$ and the total sliding distance $d$ as shown in the following equation:

$$Q = \frac{V}{d} \quad (with \quad d = 18m) \quad (1)$$

Finally, the initial microstructures, the worn surfaces of steel pins and abrasive papers are observed to analyze the generated wear mechanisms. Scanning electron microscope (SEM) is used at 15kV for the initial microstructures and the worn steel pin surfaces and at 2kV for the abrasive paper surfaces.

3. Results and discussion

3.1. Effect of heat treatment on hardness and microstructure

Figure 2 shows the obtained microstructures and the associated hardness. The micrographs in Figure 2(a-c) exhibit three ferrite-martensite dual-phase microstructures (DP1, DP2 and DP3). Figure 2a, 2b and 2c show dual-phase microstructures with respectively coarse and granular martensite colonies, smaller and granular martensite colonies and then fine and fibrous martensite colonies. The variation of the martensite colony morphologies depends on the initial microstructure before the intercritical annealing in the two phase region composed of ferrite and austenite.

In the case of the first dual-phase microstructure DP1 in Figure 2a, after an austenitization, the microstructure presents austenite. Then, the intercritical annealing results in transforming austenite into austenite and ferrite. Indeed, ferrite nucleates and grows at the prior austenite grain boundaries. The final water quench leads to transform the unchanged austenite into martensite and ferrite remains unchanged. The generated microstructure presents coarse martensite colonies surrounded by ferrite.

In the case of the second dual-phase microstructure DP2 in Figure 2b, the slow cooling after the autenitization generates a microstructure of hypoeutectoid ferrite and pearlite. Then, the intercritical annealing results in the nucleation of austenite at the interfaces between ferrite and carbide inside the pearlite colonies. Austenite grows into the carbides which dissolve. The final water quench leads to a microstructure composed of globular martensite colonies situated at the ferrite boundaries. In this case, the martensite colonies are smaller than for DP1.

In the case of the third dual-phase microstructure DP3 in Figure 2c, after an austenitization and a water quench, a martensitic microstructure is generated. Then, the intercritical annealing results in increasing the number of nucleation sites of autenite and ferrite at the boundaries of the prior austenite grains and martensite laths. The final water quench leads to a microstructure composed of fibrous and fine martensite colonies in a ferrite matrix.
In addition, it is shown that these ferrite-martensite dual-phase microstructures present almost the same martensite volume fraction around 67±2%. Indeed, DP1, DP2 and DP3 have respectively a martensite volume fraction of 67±2%, 69±3% and 65±3%.

The micrograph in Figure 2d exhibits a microstructure composed of martensite in the shape of laths grouped into packets. This kind of microstructure is typical of a quenched martensitic microstructure QM of low and median carbon steels. During this heat treatment, the transformations of the austenite into martensite and a small amount of retained austenite occur. Nonetheless, the retained austenite is not observed in the micrographs which should be in too small amount.

The micrograph in Figure 2e exhibits a tempered martensitic microstructure TM. Indeed, the tempering heat treatment, preceded by an austenitization followed by a water quench, results in transforming a brittle and hard martensitic microstructure into a microstructure composed of martensite in the shape of laths, impoverished in carbon, and fine carbides [17]. It is shown that the prior austenite grain size increases with the time and/or the temperature of austenitization [18]. Thus, because of a lower time and temperature of austenitization, the TM microstructure presents smaller prior austenite grain sizes than the QM microstructure.

![Micrographs of different microstructures](image_url)
3.2. Effect of abrasive particle size on tribological behaviour

Figure 3a shows the evolution of friction coefficient as function of abrasive particle size for the different microstructures. In Figure 3a, it is highlighted that an intermediate abrasive particle size around 35µm minimizes the friction coefficient. This minimization is due to a transition of wear mechanisms between adhesive and abrasive as shown in some works [2,5]. Bowden and Tabor [20] define the coefficient of friction $\mu$ as the sum of an adhesion component $\mu_a$ and a plowing component $\mu_p$. The adhesion component $\mu_a$ is related to the real contact area whereas the plowing component $\mu_p$ is related to the attack angle of the asperities. This intermediate abrasive particle size leads to minimize these two components. As the abrasive particle size decreases from 35µm, the friction coefficient increases because of the real contact area increase due to the predominance of adhesion wear mechanisms. Then, as the abrasive particle size increases from 35 µm, abrasive wear mechanisms become predominant. Thus, there is an increase of the pressure and plastic deformation at each abrasive grain which results in increasing the friction coefficient [3,19,22-26].

Figure 3b shows the evolution of wear rate as function of abrasive particle size for the different microstructures. In the case of the QM, TM and DP3 microstructures, it is shown that the wear rate increases with the abrasive particle size and thus the attack angle of the abrasive particles. It is explained that as the size and the penetration depth of abrasive particles increase, the predominant wear mechanisms are plowing, followed by wedge formation and then cutting [7,9] which generate an increase of the wear rate. More particularly, for DP1 and DP2, by considering the error bars, when the abrasive particle size increases, the wear rate increases and then remains constant for an abrasive particle size higher than 35µm.

**Figure 2.** Scanning electronic micrographs (SEM) of the microstructures of the (a) DP1, (b) DP2, (c) DP3, (d) QM and (e) TM.
Figure 3. Evolution of (a) friction coefficient and (b) wear rate as function of abrasive particle size.

Figure 4 shows the SEM observations of the worn surfaces of the pin, the abrasive paper and the steel wear debris in the case of the QM microstructure. These surfaces are obtained after friction tests performed between a pin with a QM microstructure and an abrasive paper with particle sizes of 15µm, 35µm and 200µm.

The observations of the worn pin surfaces reveal parallel grooves in which the material is displaced to the edges. Thus, these surfaces reveal severe plastic deformations produce by the hard abrasive particles. The edges may be removed after several passages of abrasive particles on the pin surface and generate wear debris. The presence of wear debris on the worn surfaces of the abrasive paper and the pin reveal the cutting process. Thus, the predominant wear mechanisms are ploughing with severe plastic deformation and cutting.

Then, compared to fine abrasive particles, the coarse abrasive particles generate wider and deeper grooves and therefore a higher wear rate. Indeed, the wear rate increases because of the increases of the steel wear debris size and the cutting process [2-3]. In the case of coarse abrasive particles, the worn abrasive paper exhibits abrasive wear mechanisms characterized by abrasive grain fractures. As the abrasive particle size increases from 35µm, the later phenomenon leads to the increase of the friction coefficient [2,5]. In the case of small abrasive particles, the worn abrasive paper reveals accumulations of steel wear debris around the abrasive particles and thus clogging. As mentionned by De Pellegrin et al. [21], as the particle size decreases, the space between abrasive grains decreases. This phenomenon is due to a facilitated accumulation of the metallic debris in smaller volumes. As the abrasive particle size decreases from 35µm, this clogging phenomenon generates adhesion and a higher friction coefficient. Thus, in the case of the intermediate abrasive particle size of 35µm, the worn abrasive paper presents a combination between adhesive and abrasive wear mechanisms which contributes to minimize the friction coefficient.
Figure 4. In the case of the QM microstructure, scanning electron micrographs (SEM) of the worn surfaces of the (a) pins, (b) abrasive papers and (c) wear debris.

3.3. Effect of microstructure on tribological behaviour

Figure 3a and 3b reveals that dual-phase microstructures DP1, DP2 and DP3, present a lower friction coefficient and wear rate than single-phase microstructures such as the QM and TM microstructures. Because of a higher carbon content, the martensite of the DP microstructures are harder than the QM and TM microstructures. Indeed, it is well known that the carbon content controls the hardness of the phases. In the case of the ferrite–martensite dual phase steels, the martensite phase, which is a metastable iron phase supersaturated in carbon, is significantly harder than the ferrite phase. On the other hand, the ferrite phase is able to dissolve a maximum carbon amount of only 0.02%wt. Based on a mixture rule as mentioned in some works [28-29], the dual-phase steel carbon concentration C is estimated by
considering the carbon concentration $C_m$ of the isolated martensite, the carbon concentration $C_f$ of the ferrite, the martensite volume fraction $V_m$ and the ferrite volume fraction $V_f$ as followed:

$$C = C_f V_f + C_m V_m = C_f (1 - V_m) + C_m V_m$$

Thus, the carbon concentration of the isolated martensite can be characterized by the following equation:

$$C_m = C_f + \frac{C - C_f}{V_m}$$

Thus, for a martensite volume fraction $V_m$ around 67% for the three dual-phase microstructures studied, the carbon concentration $C_m$ is estimated around 0.36% which leads to a harder martensite than for the martensite of the single-phase microstructures. Therefore, in the case of DP microstructures, the martensite carries much higher pressure and exhibits lower penetration depth of the abrasive grains than the martensite of the single-phase microstructures. Thus, in the case of DP microstructures, the depth penetration of the abrasive grains on the martensite are lower. In addition, the microstructures impacts the evolution of the wear rate as function of the abrasive particle size. Indeed, for the QM, TM and DP3 microstructures, the wear rate increases with the abrasive particle size. On the other hand, for the DP1 and DP2 microstructures, when the abrasive particle size increases, the wear rate increases and then remains constant for an abrasive particle size of 35μm. It is known that for abrasive particle sizes higher than a critical particle size usually around 100μm, the wear rate remains constant [27]. Nonetheless, this critical particle size depends on the microstructure. The critical particle size for the QM, TM and DP3 is higher than for the DP1 and DP2 microstructures.

Figure 5 shows the SEM observations of the worn surface of the steel pin, the abrasive paper and the wear debris after a friction test carried out with an abrasive paper with a particle size of 200μm in the case of the QM and the DP microstructures.

As regards the QM microstructure, the pin presents a predominance of plowing with severe plastic deformation and cutting. On the used worn abrasive paper, adhesion of wear debris on the abrasive particle tops appear and contribute to increase the contact area and therefore the friction coefficient [2,5]. On the other hand, with coarse and granular martensite colonies, the DP1 microstructure presents a predominance of cutting followed by plowing. The DP1 microstructure also generates smaller wear debris which leads to decrease the wear rate.

Among the dual-phase microstructures, it is shown that coarse and granular martensite colonies presents lower friction coefficient than fine and fibrous martensite colonies. The coarse and granular martensite (DP1) supports the majority of load and leads to reduce the plastic deformation compared to fine and fibrous martensite (DP3) [12]. On the other hand, the effects of martensite colony morphologies on wear rate are related to the abrasive particle size.

Among the single-phase microstructures, the TM microstructure has a higher wear rate and a lower friction coefficient than the QM microstructure. Indeed, in the case of the TM microstructure, the presence of martensite laths impoverished in carbon, the carbides and the finer prior austenite grain size than the QM microstructure generates a friction coefficient decrease and a wear rate increase.

![SEM observations of the worn surface of the steel pin, the abrasive paper and the wear debris](image)
4. Conclusions

For a similar hardness level (around $410H_V$) and from the same low alloy steel, various microstructures are generated by using various heat treatments. The main aim is to analyze the combined effects of the abrasive particle size and the microstructure on the friction and wear behaviours. Therefore, dry friction tests are performed between these heat-treated pins and an abrasive paper with various particles sizes. By using coarse and fine abrasive particles, the effect of roughness on the tribological behaviour are investigated. The main findings are:

- Compared to single-phase microstructures (quenched martensitic and tempered martensitic microstructures) and whatever the abrasive particle size, ferrite-martensite dual-phase microstructures minimize the friction coefficient and enhance the wear resistance.
- Among the dual-phase microstructures and whatever the abrasive particle size, the morphologies of the martensite colonies influence the friction behaviour. Indeed, coarse and granular martensite colonies minimize the friction coefficient whereas fine and fibrous martensite colonies increase the friction coefficient.
- The friction coefficient is minimized for an intermediate abrasive particle size around $35\mu m$. This minimization is explained by a transition of wear mechanisms between abrasion and adhesion for this intermediate abrasive particle size. However, the wear rate and the wear debris size increase with the size and the attack angle of the abrasive particles. Nonetheless, the wear rate becomes constant for a critical abrasive particle size which is closely linked to the microstructures. Indeed, the dual-phase microstructures with granular martensite colony present a lower critical abrasive particle size than the others microstructures.

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