Effect of Variation of Martensite with a Constant Carbon Content on Mechanical Behavior and Sliding Wear of Dual Phase Steels

Mehran Zamani1 · Hamid M. Ghasemi1 · Hamed Mirzadeh1

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Abstract
In a dual phase steel, an increase in the martensite volume fraction leads to a decrease in its carbon content, and hence, elucidating the effect of martensite content on the mechanical and tribological behaviors has not been possible. For the first time, the present work dealt with this subject by maintaining a constant amount of carbon in different volume fractions of martensite. Tribological properties was evaluated via a pin-on-disk sliding tribometer. The study of wear tracks and debris revealed oxidative wear as the dominant wear mechanism. Increasing normal load from 10 to 30 N resulted in a decrease in the specific wear rate, which was related to the high work-hardening capacity. This resulted in higher surface hardness, accommodating the formation of a more durable tribofilm on the surface, and a lower coefficient of friction. However, the increase in normal load from 30 to 60 N resulted in an increase in the specific wear rate and reduction of oxide coverage on the sliding surface due to high surface stresses. The worn debris were mainly identified as metallic in the running-in stage and Fe3O4 oxide in the steady-state stage.

1 School of Metallurgy and Materials Engineering, College of Engineering, University of Tehran, P.O. Box 11155-4563, Tehran, Iran
Graphical Abstract

Keywords Dual phase steels · Wear resistance · Mechanical properties · Strain-hardening rate · Archard law

1 Introduction

Dual phase (DP) steels, a main group of advanced high strength steels [1], can be considered as wear-resistant materials for application in mining, pipeline transportation, mineral processing, etc. [2]. This comes from the co-presence of martensite with a high hardness and appreciable work-hardening capacity of DP steels [3–7]. Generally, wear resistance of DP steels can be affected by their microstructural, mechanical properties and the tribological conditions [8, 9]. The martensite phase is the main parameter that controls the properties of DP steels, including mechanical, microstructural, and tribological ones, where the amount of martensite [10–16], its carbon content [17], and its morphological aspects [18–22] are governing parameters. Regarding the effect of martensite volume fraction, Aksoy et al. [10] reported that the wear resistance of the AISI 1020 steel (against a hardened and tempered cold-working steel as the counterface material) decreased with increasing martensite volume fraction which resulted in reduction of martensite hardness due to decline of its carbon content. However, Tyagi et al. [11] (0.42 wt% C steel against hardened En-32 steel), Modi [12] (0.19 wt% C steel against abrasive SiC particles), and Abouei et al. [13] (0.2 wt% C steel against hardened and tempered AISI 52,100 steel) have reported an enhancement of wear resistance with increasing the amount of martensite. Tekeli et al. [14] investigated the effect of intercritical annealing on graphite-contained low-carbon steel. Their report indicated that the best wear resistance obtained at a low intercritical annealing temperature inasmuch as lower amount of martensite enjoys higher hardness because of the presence of higher amount of carbon content. Interestingly, based on the multi-pass dual-indenter (MPDI) scratch test, Xu et al. [15] found an optimal martensite fraction to reach the minimum scratch depth. Recently, Penfornis et al. [9] evaluated tribological behavior of dual phase steel via nanoindentation and scratch tests. This report indicated the effect of variation of martensite on the wear behavior and friction coefficient in a microscopic scale. It also elaborately illustrated that increasing of martensite volume fraction...
reduces its carbon content, thereby reducing the hardness of the phase.

Previous works have provided valuable information about mechanical and tribological behavior of dual phase steel. However, none of them indicated the effect of martensite volume fraction precisely due to the reduction of its carbon content. In other words, each of previous works obtained their dual phase specimens from one specific kind of steel, therefore, the reduction of carbon content of martensite by increasing its volume fraction was inevitable. This research addressed the problem for the first time. Hence, an unambiguous study of the effect of martensite fraction with a constant amount of carbon on mechanical behavior and sliding wear of dual phase steels is the aim of the present work.

2 Experimental Details

2.1 Processing of DP Steels

Carbon steels with 0.04, 0.12, 0.19, and 0.27 wt% carbon (Table 1) were received as annealed condition. In order to reveal the effect of temperature on martensite volume fraction ($V_M$) of each steel, they were intercritically annealed at different temperatures for a soaking time of 10 min followed by quenching in water. The resulting microstructures were examined by image analysis techniques and the volume fraction of martensite were determined. The results are shown in Fig. 1. The carbon content of 0.27 wt% in the martensite phase of the alloys was considered as the desired value. Based on the lever rule, it can be deduced that the carbon content of martensite ($C_M$) can be determined by $C_M = C_{Steel}/V_M$ [17]. As a result, martensite volume fractions of 0.15, 0.44, 0.70, and 1.00 for steels with 0.04, 0.12, 0.19, and 0.27 wt% of carbon, respectively, resulted in a constant carbon content of 0.27 wt % in the martensite. These points are also shown on Fig. 1.

2.2 Microstructural Characterization

The microstructures were etched by the LePera’s and Nital solutions, and then, revealed by an Olympus Vanox optical microscope and a FEI Nova 450 FE-SEM. A Bruker detector for energy-dispersive spectroscopy (EDS) was used for elemental analysis investigations. X-ray diffraction (Philips PW-3710 diffractometer with Cu-Kα radiation) was used for phase identification.

2.3 Evaluation of Mechanical Properties

JIS Z 2201 standard for tensile test specimens was used. A SANTAM universal testing machine was used for tensile tests at room temperature with an initial strain rate of 0.001 s$^{-1}$. The reproducibility of results was evaluated by the repetition of the tests. The hardness and microindentation hardness measurements were based on the Vickers hardness under loads of 30 kg and 10 g, respectively. The work-hardening rate ($\theta = d\sigma/d\varepsilon$) was evaluated based on the Crussard–Jaoul analysis [23], where $\sigma$ and $\varepsilon$ are the true stress and true strain, respectively. Moreover, the yield ratio (YS/UTS) and work-hardening exponent ($n$-value) [24] were also used for evaluation of work-hardening behavior, where YS and UTS are the yield stress and the ultimate tensile strength, respectively.

2.4 Evaluation of Wear Behavior

Tribological behavior of the DP samples was evaluated based on the pin-on-disk tribometer. Disk specimens were prepared from the as-received sheets (Table 1), and then, intercritically annealed as described above to obtain various
volume fractions of martensite with carbon content of 0.27 wt%. The pins and the discs were ultrasonically cleaned in an acetone bath to eliminate any contaminations. Dry sliding wear tests were carried out using a pin-on-disc tribometer against a hardened AISI 52,100 steel pin (with a diameter of 5 mm and hardness of 763 HV30) with a tip radius of 5 mm as the counterpart material. The tests were performed under normal loads of 10, 30, and 60 N at a sliding speed of 0.5 m/s for a sliding distance of 1000 m. Each test has been repeated at least three times. After each test, weight loss was determined using a balance with an accuracy of 0.1 mg.

3 Results and Discussion

3.1 Mechanical Properties

The literature data [25, 26] on the effect of carbon on the hardness of martensite are shown in Fig. 2. The data could be modeled by an exponential relation, where for a carbon content of 0.27 wt%, a hardness of about 524 HV was obtained. The experimental microindentation hardness of the martensite phase of four processed DP steels was in the range of 554–585 HV0.01 as shown in this figure, which were close to the calculated value.

The microstructures of as-received and DP steels are shown in Fig. 3. It can be seen that the as-received steels contained microstructures comprising of ferrite grains and pearlite colonies arranged alternatively in the form of banded structure [27]. Moreover, by increasing the carbon content of as-received steels, the amount of pearlite was increased (from Fig. 3a–d). After the intercritical annealing, ferritic-martensitic DP microstructures were achieved. As the carbon content of DP steels increased, the amount of martensite was also increased as mentioned in the experimental section (Figs. 3e–h). In the case of 0.27 wt% C steel, a fully martensitic microstructure was obtained, where the martensite laths were observed in the provided SEM image (Fig. 3k).

Figure 4 shows the tensile stress–strain curves of the as-received steels. The figure reveals that the yield stress and tensile strength were increased via increasing carbon content, which could be related to the increase in the amount of pearlite (Fig. 3). Figure 5a shows the tensile stress–strain curves of the DP steels. It can be seen that by increasing the amount of martensite (i.e., with constant carbon content of 0.27 wt%), the strength level was increased tremendously. This could be verified for the yield stress and tensile strength (Fig. 5a) and hardness (Fig. 5b). However, the total elongation declines, which was indicative of the deterioration of ductile properties. Figure 5a also reveals that at 1 martensite volume percent of 70% (that is higher than 50%), the strength level increased significantly, which could be related to the fact that the hard martensite phase became the matrix of the alloy (Fig. 3). These effects are much larger when compared to the case of increasing martensite content in a given steel composition. For example, as with 0.12 wt% carbon steel shown in Fig. 5c, the carbon content of martensite declined by increasing the amount of martensite [10, 17].

The work-hardening behavior of the DP steels is shown in Fig. 6a. It can be seen that by increasing the amount of martensite from 15 to 44%, while there was a drop in the strain, the work-hardening rate increased significantly. For the martensite volume fraction of 70%, the level of work-hardening rate was comparable to that of the sample with 44% martensite but it dropped rapidly to a lower strain. In the case of 100% martensitic sample, a poor work-hardening behavior was evident. The results of Fig. 6a indicated that the sample with 44 vol% martensite exhibited the best work-hardening behavior due to higher ln(θ) at the higher strains. Figure 6b shows that the yield ratio (YS/UTS) of the DP steel with 44% martensite was lowest, which also revealed the best work-hardening behavior in accordance with Fig. 6a. However, Fig. 6b shows that the work-hardening exponent (n-value) continuously decreased with an increase in the amount of martensite, indicating a decline in the uniform elongation [24].

3.2 Tribological Properties

Figure 7a and b show the wear rate and specific wear rate of the steels at various loads and martensite volume fractions. It can be seen that by increasing the amount of martensite, the wear rate of DP samples was decreased under various
normal loads, which could be related to the increase in hardness in the alloys as shown in Fig. 5b. This can be also verified based on the observed wear tracks shown in Fig. 8a–c. It can be seen that by increasing the amount of martensite and increasing the hardness, the width of wear track decreased significantly under a normal load of 10 N due to a lower penetration of the pin counterface material, which revealed a notable increase in the deformation resistance of the alloys.

Figure 7a shows the enhancement of wear resistance of the DP alloys with increasing the amount of martensite, which agreed with the reported works by Tyagi et al. [11], Modi [12], and Abouei et al. [13]. However, a constant amount of carbon (i.e., 0.27 wt%) in the various volume of martensite considered in the present work helped to unambiguously characterize the effect of martensite content. It should be noted that for DP steels, Aksoy et al. [10] reported that in a block-on-ring test, a decrease in
wear resistance with increasing martensite volume fraction was observed. However, Xu et al. [15] showed that there was an optimum martensite fraction with a higher scratch wear resistance than a fully martensitic sample at relatively high stress. Owing to the fact that none of these reports showed any control on the carbon content of their dual phase samples, the various amount of carbon in martensite and different contact geometries could affect the wear behavior.

In the backscattered SEM images of the wear tracks (Fig. 8a–c), the darker regions suggested that a lower atomic numbers compound was formed on the wear tracks. The EDS point analysis from these regions and elemental x-ray maps of the wear surface of the sample with 15 vol% martensite
are shown in Fig. 10. It can be seen that the bright and dark regions contained metallic and oxide phases, respectively. Figure 8 reveals that a considerable region of the wear tracks in all samples were covered with an iron rich oxide layers which could indicate an oxidative wear behavior.

Figure 7a reveals that for a given martensite fraction, by increasing the normal load, the wear rate was increased, which could be expected [11]. The specific wear rates are also summarized in Fig. 7b, which indicated that the specific wear rate was sensitive to the normal load in DP samples. According to Fig. 7b, by increasing the normal load from 10 to 30 N, the specific wear rate of dual phase samples fell considerably while the specific wear rate of the martensitic sample (i.e., 100% martensite) was approximately constant. This could be related to the work-hardening capability of the sliding surfaces. The effect of work-hardening on enhancement of wear resistance has been well-established [28, 29]. It has been reported that in materials with high work-hardening capacity, the surface hardness on wear track is increased, and therefore, the overall scratch resistance of the material is improved [29]. Deformation of the sub-surface of wear tracks in Fig. 9 is an evidence that they were affected by work-hardening phenomenon. It can be seen that the reduction of specific wear rate by increasing the normal load from 10 to 30 N was more pronounced for the sample with 44% martensite, which showed a better work-hardening behavior, i.e., a higher ln(θ) at a higher strain, compared to the other samples (Fig. 6). This could also help to support a more durable tribolayer. Figure 11 shows wear tracks and friction coefficients of 44 vol% martensite sample at different normal loads. It can be seen that the increase in normal load from 10 to 30 N resulted in more coverage of oxide film on the surface which could cause less metallic contact, less adhesion between the mating surfaces, lower friction and also a lower amplitude of friction oscillation. There are other reports [30–32] which indicate that the formation of durable Oxide film on the sliding surface can improve wear resistance behavior of different materials.

By increasing the normal load from 30 to 60 N, the specific wear rate of dual phase samples was slightly increased (Fig. 7b) and the coverage of oxide film on the wear surface decreased (Fig. 11). According to these results, it seems that the dual phase samples might be work-hardened efficiently at 30 N and higher normal loads. Under a normal load of 60 N, although a higher surface temperature and, therefore,

![Fig. 6 Analysis of work-hardening behavior of DP samples based on: a Crussard–Jaoul analysis and b Dependency of yield ratio and work-hardening exponent on the martensite volume fraction](image)

![Fig. 7 Variation of a wear rate with amount of martensite of DP samples under various loads, and b specific wear rate versus normal load](image)
a higher oxidation rate was achieved, however, the higher stress could increase the rate of oxide fracture and removal rate of the oxide layers. This resulted in a decrease in the oxide coverage (Fig. 11), a more metallic contact, higher adhesion between surfaces leading to a higher amplitude of friction and slightly higher specific wear rate of the 44% martensite sample (Fig. 7). The higher work-hardening of the sample under a higher normal load of 60 N could result in an increase in surface hardness and smaller real area of contact and a lower coefficient of friction.

The variation of coefficient of friction of 44% martensite sample under a normal load of 60 N in the early stage of sliding is shown in Fig. 12a, which reveals the presence of two regimes: the running-in and steady-state stages. It can be seen that the friction amplitude of the running-in stage was higher than the steady-state stage which could be related to a higher adhesion and more metallic contact under the running-in stage. During the running-in stage, it is liable that the work-hardening could increase deformation resistance of the sliding surface, which led to a better support of oxides in the following steady-state stage. Therefore, the durability of oxide film resulted in less adhesion between the mating surfaces and reduction of the friction amplitude and coefficient of friction of the steady-state stage. Figure 12b and c show the worn debris of the running-in and steady-state stage of 44% martensite sample at 60 N. It can be seen that the debris in running-in stage were much larger compared with the steady-state stage. The XRD patterns of the debris in Fig. 12d indicate that debris in running-in and steady-state regions were mainly metallic and oxidative (i.e., Fe₃O₄ shown in Fig. 12d) in nature, respectively. Due to the large metallic worn debris and high amplitude of friction in the running-in stage, it was deduced that at the beginning of the sliding, the wear mechanism is mainly metallic adhesive wear. However, the work-hardening and formation of durable oxide film on the sliding surface, resulted in the reduction of metallic contact and changed the dominant wear mechanism from adhesive (severe wear) to oxidative wear (mild wear).

4 Conclusions

The effect of 15, 44, 70, and 100 vol% martensite with a constant amount of 0.27 wt% carbon on mechanical and tribological behaviors of DP steels was studied. The following conclusions can be drawn from this study:

(1) A pronounced effect of martensite content on tensile properties and wear resistance was observed, which was related to the constancy of carbon content in the martensite constituent.
(2) The wear resistance of DP samples with constant amount of carbon in martensite was enhanced by increasing the amount of martensite.

(3) Increasing the normal load from 10 to 30 N increased the wear rate, however, decreased the specific wear rate in DP steels. This could be related to the high work-hardening capacity and higher subsurface strength, ability to form a more effective tribofilm, lower adhesion between mating surfaces and a lower coefficient of friction. However, an increase in normal load from 30 to 60 N resulted in an increase in specific wear rate due to a lower coverage of oxide film on the sliding surfaces.

(4) The coefficient of friction and amplitude of friction oscillation at the running-in stage were much higher compared with the steady-state stage. The worn debris in the running-in and steady-state stages were mainly identified as metallic and Fe$_3$O$_4$ oxide types, respectively. Based on the frictional behavior and XRD patterns of wear debris, the dominant wear mechanism of the running-in and steady-state stages were identified as adhesive and oxidative wear, respectively.
Fig. 10  EDS elemental X-ray maps and point analysis obtained from the wear track of a DP sample with 15% martensite.
Fig. 11  Coefficient of friction and backscattered SEM image of sliding surface of a DP sample with 44% martensite under three different loads.
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