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Research Article

Keywords: X52 HSLA steel, Heat treatment, Dual-phase, Friction, Wear, Phase transformation

Posted Date: July 23rd, 2021

DOI: https://doi.org/10.21203/rs.3.rs-720895/v1

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Microstructural and Tribological characterization of API X52 dual-phase steel

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Abstract

The effect of the morphology and the martensite volume fraction on the microhardness, the tensile, the friction and the wear behavior of API X52 dual phase (DP) steel has been investigated. Three different heat treatments were used to develop dual phase steel with different morphologies and with different amounts of martensite: Intermediate Quenching Treatment/Water (IQ); Step Quenching Treatment (SQ) and direct quenching (DQ). Tribological tests are conducted on DP steels using a ball-on-disc configuration under normal load of 5 N and at a sliding speed of 4 cm/s were used to study the friction and wear behavior of treated samples. Results show that the ferrite–martensite morphology has a great influence on the mechanical properties of dual phase steel. The steel subjected to (IQ) treatment attain superior mechanical properties compared to the SQ and the DQ treatments. On the other hand, it is also found that the friction coefficient and the wear rate (volume loss) decrease when the hardness and the martensite volume fraction increase. The steel with fine fibrous martensite provide good wear resistance.

Keywords X52 HSLA steel · Heat treatment ·Dual-phase · Friction · Wear · Phase transformation

INTRODUCTION

Dual Phase (DP) steels, composed of a soft ferrite matrix and dispersed hard martensite islands, are widely used as a structural material in the automotive industry because they offer a good combination of high strength and ductility [1]. This microstructure can be developed via heat treatment. Low alloy hypoeutectoïd steel is intercritically annealed between A_{c1} and A_{c3} temperatures, where a certain amount of austenite is formed with subsequent cooling to transform the remaining austenite to martensite [2]. Dual-phase steels exhibit continuous yielding behavior, low yield strength to tensile strength ratio, and high initial work hardening [3]. Since the first discoveries of Dual-Phase steels in the 1960s [4], much research has been concentrated to develop and improve these steels. Some researchers have tried to reduce the tensile strength during plastic deformation by reducing the strength difference between soft ferrite and hard martensite through micro addition of Ti and Cu [5]. Others have work on the coupling between heat treatments and plastic deformation during rolling to optimize microstructure and properties [6]. Finally, others have explored a new method of rapid heating annealing to investigate the possibility of producing a Dual-Phase steel (DP) with high-performance by controlling the decomposition of austenite during cooling [7]. A number of researchers investigated the impact of martensite morphology and volume fraction on the tensile of dual-Phase steels. Pierman et al. [8] has found that the effect of martensite morphology on the mechanical properties of Dual Phase steels is more important than the effect of volume fraction of martensite. Other researchers have demonstrated that IQ treatment provided the best mechanical properties in dual phase steels [9, 10]. Some studies focus on the effect of martensite morphology and volume fraction on friction coefficient and wear rate of dual-Phase steels. Some researchers have shown that the wear rate increases when the martensite volume fraction decreases [11-14]. Wei et al. [15] investigate on the effect of ferrite–martensite morphology on the scratch and abrasion resistance of ferrite–martensite dual phase.

The objective of this work is to transform an API X52 steel into a dual phase microstructure with different morphologies of martensite and different volume fraction martensite values via three different heat treatments:

- Intermediate Quenching Treatment/Water (IQ)
- Step Quenching Treatment (SQ)
- And Direct Quenching (DQ).

In this study, we evaluate the effects of the martensite morphology of IQ, SQ and DQ treatments and it's volume fraction on the microhardness, the tensile, the friction and the wear of API X52 dual Phase steel.

2 Materials and Methods

2.1 Material

The used material in this study is an X52 HSLA steel with a ferrite-pearlite fine-grained microstructure. The chemical composition of this steel is given in Table 1. Specimens are cut from coils for welding pipelines. Specimens used for the friction tests were cut to dimensions of 20 mm × 20 mm × 10 mm, for tensile tests Specimens (according to EN 10002-1, ASTM E8).

Table 1 Chemical composition of X52 HSLA steel

Elements	C	Mn	Si	S	P	Nb	V	Ti	Al
Composition (%)	0,12	1,22	0,23	0,001	0,011	0,03	0,03	0,002	0,034

2.2 Heat treatments

Before heat treatments, all the samples are polished with 180 to 1000 grit silicon carbide abrasive paper (SiC). The objective of this polishing process is the disposal of oxides and surface defects.

Three types of heat treatment (as seen in Figure were performed to generate DP steels with different morphologies: Intercritical annealing from an almost fully Martensitic starting state followed by Quenching (IQ) ; full austenisation, then intercritical annealing to form ferrite structures followed by Quenching (SQ) and Intercritical annealing, directly from the Ferrite/pearlite starting microstructure followed by Quenching (DQ). The detailed heat treatments are described below:

- IQ: first full austenisation at 940°C for 30 min followed by water quenching; Then intercritical annealing at 760°C and 800°C for 30 min followed by water quenching, as shown In Fig.1a.
- SQ: full austenisation at 940℃ for 30 min followed by intercritical annealing at 760°C and 800℃ for 30min followed by water quenching, as shown in Fig. 1b.
- DQ : intercritical annealing directly from the Ferrite/pearlite starting microstructure at 760 °C and 800 °C for 30 min and water quenching, as shown in Fig. 1c.

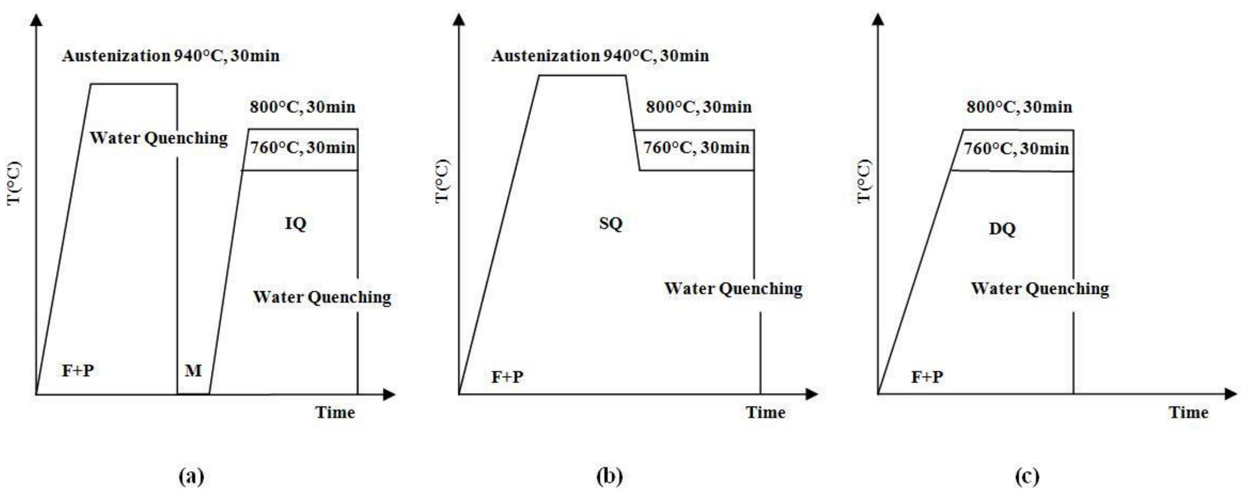

Fig. 1 Schematic drawing of the heat treatment procedure: (a) IQ, (b) SQ and (c) DQ

2.3 Metallographic preparation

The samples were polished with silicon carbide abrasive paper (SiC) of 3000 grit, and etched with 4% Nital solution for metallographic examination. The etched samples were examined using light microscopy and the phase proportions were determined by a manual point-counting technique.

2.4 Microhardness and tensile tests

The microhardness measurements are carried out using a Vickers indenter under 200 g load. The microhardness values reported are the average of three measurements from different locations for each sample. Tensile testing of flat specimens was conducted at room temperature on a computer-controlled Mohr Federhaff Sachsenhausen System Machine.

2.5 Wear and friction test

The friction and wear tests were performed by using a CSM Instrument ball-on-disc type tribometer. The tested samples were rubbed against a hardened 100Cr6 steel ball (65 HRC) with 6 mm diameter. The wear tests were carried out under dry sliding condition with 5N applied normal load and sliding speed of 4 cm/s. The total sliding distance was 20 m. All the experiments were carried out in an ambient atmosphere at temperature of 25 °C and with relative humidity of 50%. The friction evolution was directly recorded during the test. A simplified schematic view of the ball-on-disc test configuration is shown in Figure 2.To evaluate volume loss recorded during the wear tests, an optical profilometer (Altisurf 500) equipped with a 3D image processing software (Altimap topography XT) and a 2D roughness profile was used to measure surface roughness and the wear volume loss

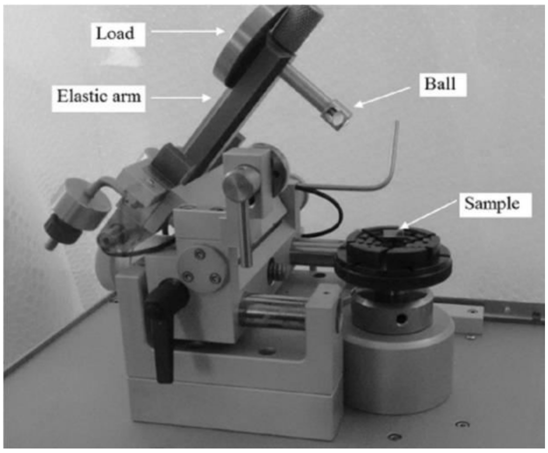

Fig. 2 View of the used ball-on-disc tribometer

3 Results and Discussions

3.1 Microstructures

The obtained microstructures by the applied heat treatments shown that in all cases the intercritical annealing conduct to a dual phase microstructures consisting of ferrite (white regions) and martensite (black regions). No other phases were present (Figure 3). The martensite and ferrite fractions depend on the annealing temperature (760 or 800 °C). Figure (3) shows a dual-phase microstructure obtained by the IQ treatment, consisting of a fine and fibrous martensite uniformly distributed within the ferrite matrix. According to literature [16], this morphology is obtained from the nucleation of austenite along the lath boundaries of prior martensite during the annealing heat treatment. The microstructure of the sample subjected to the SQ treatment shows Ferrite and Martensite band morphology with a uniform distribution of phases. The microstructure is austenitic before entering into in the two-phase ($\alpha + \gamma$) region. Upon lowering the temperature to the two-phase region, ferrite nucleates at the grain boundaries of austenite grows within the austenite from the nucleation of austenite along the lath boundaries of prior martensite. The microstructure of the sample subjected to the SQ treatment shows Ferrite and Martensite band morphology with a uniform distribution of phases. The microstructure is austenitic before entering into in the two-phase ($\alpha + \gamma$) region. Upon lowering the temperature to the two-phase region, ferrite nucleates at the grain boundaries of austenite and grows within the austenite grains [17].The microstructure of the sample subjected to the DQ treatment shows a dual-phase structure characterized by a microstructure which consists of hard martensite dispersed in a ductile ferritic matrix, obtained by the DQ treatment from intercritical temperatures 760 and 800 °C. When heating in the ($\alpha + \gamma$), the ferrite remains essentially unchanged, and the pearlite change to carbon-rich austenite. As long as martensite transforms without diffusion, it inherits the amount of carbon in austenite fixed by the intercritical temperature. So the volume fraction of the martensite increases with the intercritical annealing temperature.

3.2 Tensile Properties

The yield strength (YS), highest ultimate tensile strength (UTS), total elongation and the microhardness of the specimens before and after heat treatments IQ, SQ and DQ are presented in Table 3, The variation of the mechanical properties of dual phase steel were strongly influenced by morphology and the volume fraction of the dispersed martensitic phase. A comparison of tensile properties between IQ, SQ and DQ specimens, show that IQ specimen had the highest value of UTS, yield strength and total elongation compared to SQ and DQ specimens. More the proportion of martensite increases, the more the mechanical properties improved. This agrees with Sodjit and Uthaisangsuk [18], C. Dulucheanu et al.[19], Bayram et al. [20] and Y. Tomita [21] Ashrafi et al. [22]. The IQ Show a excellent combination of strength and ductility, this is due to the fine and fibrous martensite morphology.

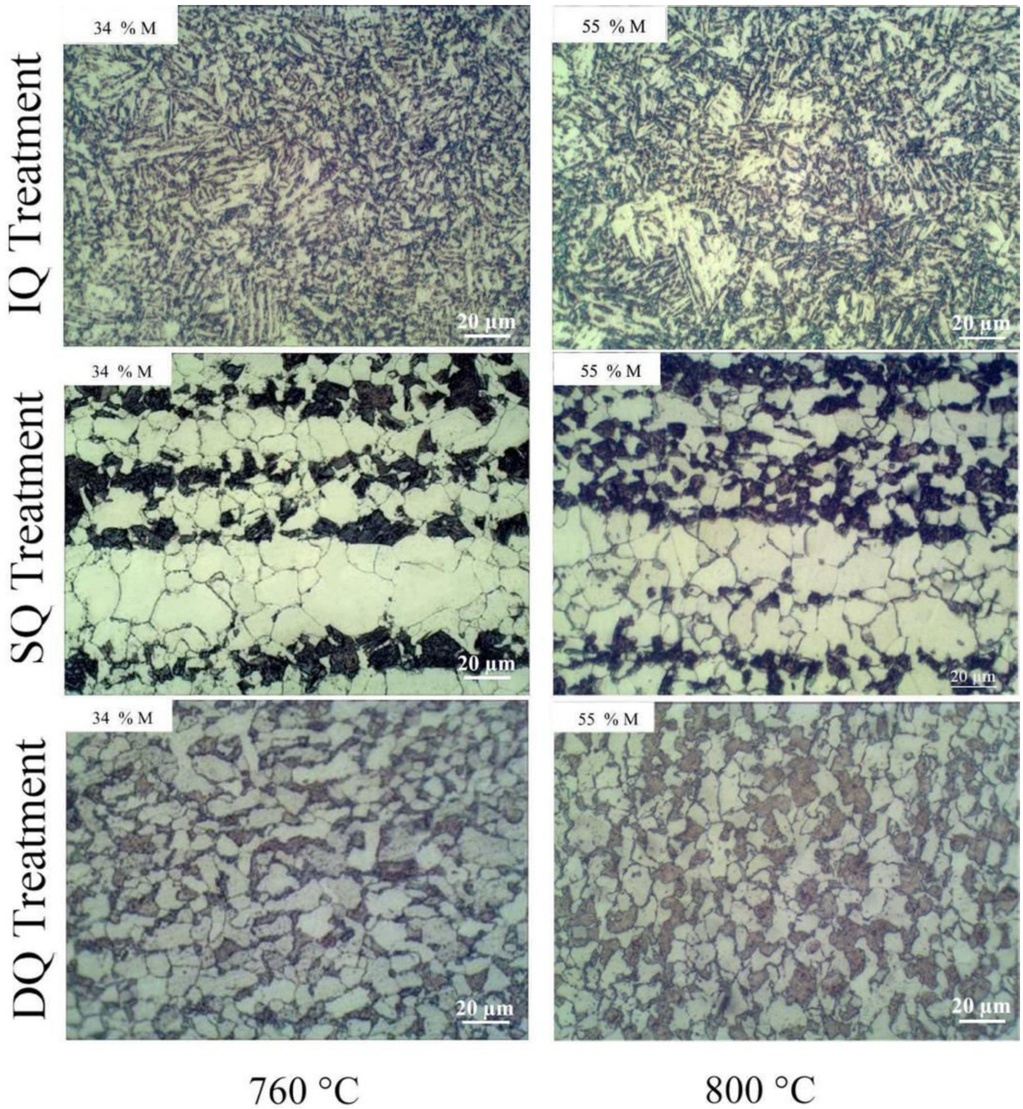

Fig. 3 Optical micrographs of dual phase microstructures of IQ, SQ and DQ treatments at 760 and 800 °C

Table 2 Mechanical properties values obtained in this work

Steel	Specimen	AT (°C)	VFM [%]	UTS [MPa]	YS [MPa]	TE [%]	Microhardness [HV]
	Initial values	/	/	516	377	27.5	179
	IQ	800	55	845	525	25	210
API X52		760	34	735	470	31	190
	SQ	800	55	790	500	11	187
		760	34	680	440	16	153
	DQ	800	55	750	485	17	190
		760	34	670	405	22	210

3.3 Wear and friction tests

3.3.1 Friction coefficient evaluation

The tested surfaces result in coefficient values between 0.30 and 0.55 for all tested samples. In each case, the evolution of the tangential force as a function of the cycle's number can be decomposed into two phases whatever the heat treatment applied (Figure 4 and 5):

- A first phase when the friction coefficient increases up to a maximum value, ranging from 0.30 and 0.55. This phase is associated with the formation of a transferred layer and the appearance of wear particles on ball tracks [23].
- A second phase where the coefficient of friction stabilizes between 0.44 and 0.48. It seems that this phase is associated with an increased abrasive action of the wear particles [24].

The friction coefficient and volume loss values obtained in this work are grouped in table 3.

Table 3 Friction coefficient and volume loss values as function of VFM and its morphology

Specimen		AT (°C)	VFM (%)	Load (N)	Speed (cm/s)	Friction Coefficient	Volume loss (µm3/µm)	Microhardness (HV)
	IQ	800	55			0.3	5741	210
		760	34			0.33	10214	190
X52	SQ	800	55			0.47	15115	187
		760	34	5	4	0.55	22619	153
	DQ	800	55			0.4	11080	190
		760	34			0.45	11853	210

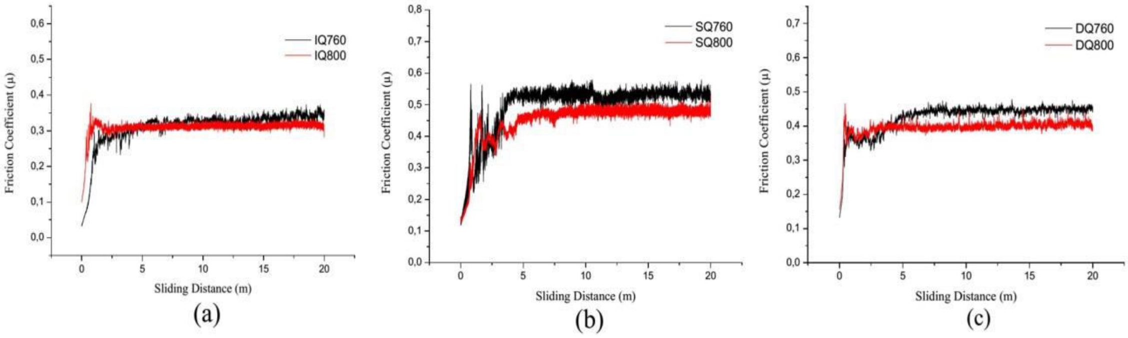

Fig. 4 Variation of friction coefficient with sliding distance for a load of 5N (a) IQ 760 and IQ 800, (b) SQ 760 and SQ 800, (c) DQ 760 and DQ 800

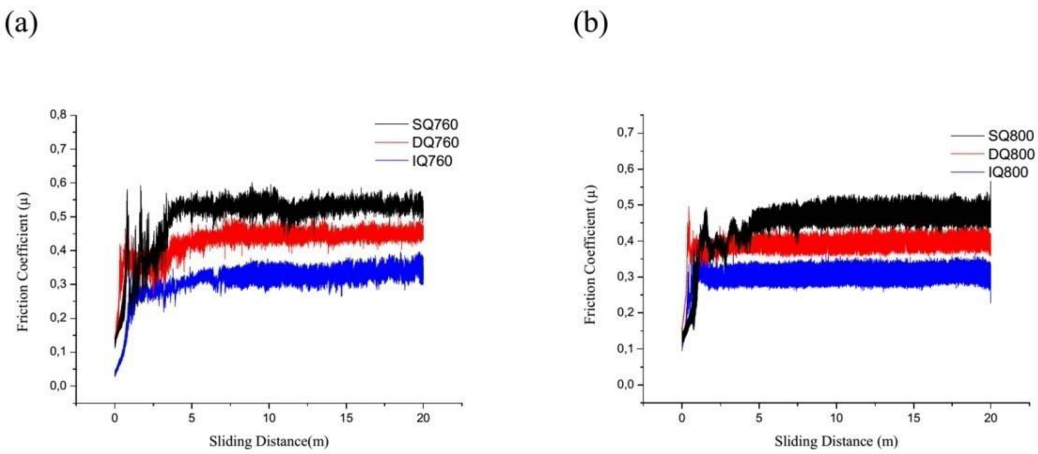

Fig. 5 Variation of friction coefficient with sliding distance for a load of 5N (a) IQ 760, SQ 760 and DQ 760, (b) IQ800, SQ 800 and DQ 800

Martensite Volume fraction effect

From figure 4 and table 3 it can be seen that the friction coefficient decrease when the martensite volume fraction increase. The martensite of dual-phase microstructures withstands a higher pressure and exhibits lower penetration depth of the indenter and lower plastic deformations, due to higher hardness of this phase, which leads to a decrease in the surface of contact and the reduction of the possibility of formation and propagation of crack during delamination. On the other hand, the morphologies of the martensite colonies influence the friction behaviour. This agrees with Saghafian et al. [25] and Tyagi et al. [13] results, who have also reported that the the friction coefficient of DP steels decreases when the volume fraction of martensite (VFM) increase.

Martensite Morphology effect

From figure 6 and table 3 it is observed that the IQ specimen with fine fibrous martensite had the lowest friction coefficient. This probably due to the fine and fibrous martensite uniformly distributed within the ferrite matrix. This agrees with Ashrafi et al. [22] results, who found that the average friction coefficient increases in the order of IQ < DQ < SQ.

3.3.2 Wear

Wear volume is calculated from the wear track, the test was repeated in three different zones, the average values obtained are being taken. A first observation of the volume loss curve as a function of the sliding distance obtained in figure 6 let us do the following remarks:

- The maximum volume loss corresponds to the SQ760 treatment.
- The volume loss recorded on the IQ800 samples is less than that registered on SQ and DQ.
- The volume loss recorded on the IQ800 samples is five times less than the SQ760 samples.

The difference in volume loss between the different samples is due to the volume fraction of the martensite, or to its morphology:

Martensite Volume fraction effect

For the same heat treatment (SQ), the volume loss recorded at the temperature intercritical 800 °C, is less than that of a 760 °C, this is not surprising since the volume fraction of martensite of SQ800 is more important than SQ760.

- The volume loss is 30000 $\mu m^3/\mu m$ for SQ760 and 15000 $\mu m^3/\mu m$ for SQ800 respectively. We can easily observe that the volume loss recorded on the SQ800 samples is two times less than that of SQ760 samples.
- The volume loss is 10000 $\mu m^3/\mu m$ for IQ760 and 5000 $\mu m^3/\mu m$ for IQ800 respectively. These values indicate that the volume loss recorded on the IQ800 samples is two times less than that of IQ760 samples.

On the other hand for the DQ samples, the values are close; the volume loss is 10000 $\mu m^3/\mu m$ and 14000 $\mu m^3/\mu m$ for DQ800 and DQ760 respectively.

Martensite Morphology effect

- The volume loss recorded on the IQ760 samples is three times less than the SQ760 samples.
- The volume loss recorded on the IQ800 samples is three times less than the SQ800 samples.
- The wear rate is affected by the morphology and the proportion of martensite.
- On note that the influence of the morphology is more important than the proportion.

From figure 6, it is clearly seen that the IQ specimen show greater wear resistance than SQ and DQ specimens. The better wear resistance of IQ steel compared with that for other samples due to fine and fibrous martensite morphology and the volume fraction of the dispersed martensitic phase (higher values of microhardness) and UTS [22]. This agrees with Saghafian et al. [25] and Tyagi et al. [13] results, who have also reported that the wear resistance of DP steels increases with the increasing volume fraction of martensite (VFM).

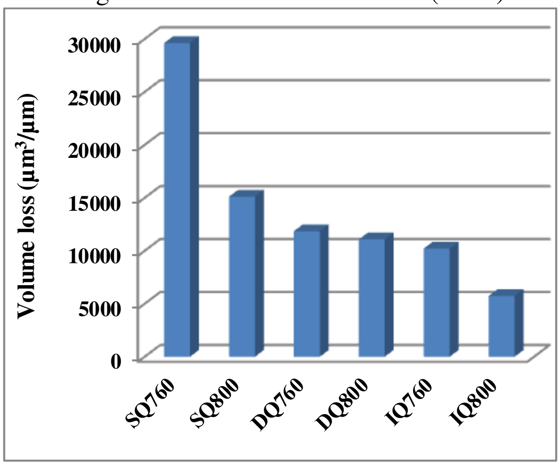

Fig. 6 Wear rate of different samples at 5N load

4 Conclusions

Three different morphologies of martensite and two different volume fraction martensite values (34% and 55%) in the dual- phase microstructure were obtained by changing heat treatment cycles, fine and fibrous martensite uniformly distributed in the ferrite matrix (IQ), blocky, and banded martensite–ferrite microstructure (SQ), and fine martensite islands distributed in the grain boundaries of polygonal ferrite matrix (DQ).

The variation of the mechanical properties of DP steel was strongly influenced by morphology and the volume fraction of the dispersed martensitic phase. The IQ specimen had the highest value of UTS, yield strength and total elongation. The good mechanical properties of IQ specimen was attributed to the presence of fine and fibrous martensite morphology.

Wear test results show that the IQ specimen had the highest wear resistance and the lowest friction coefficient, whereas the SQ specimen show the lowest wear resistance and highest value of friction coefficient, this is due to fine and fibrous martensite morphology and the volume fraction of the dispersed martensitic phase (higher values of microhardness) and UTS.

Acknowledgement

The authors gratefully acknowledge the financial support of the Directorate General for Scientific Research and Technological Development (DGRSDT).

Declarations:

Funding

- The authors did not receive support from any organization for the submitted work.

Conflicts of interest/Competing interests

- The authors have no conflicts of interest to declare that are relevant to the content of this article.

Availability of data and material

- The data used in this work are available.

Code availability

- The software applications used (tribometer and roughness) are specific to the equipment manufacturers.

Ethics approval

- The manuscript is not submitted to another journal.
- The submitted work is original and has not been published elsewhere in any form or language (partially or in full).
- All results are presented clearly, honestly, and without fabrication, falsification or any inappropriate data manipulation.
- We ensure that no data, text or theory of others is presented as if it were the property of the author.

Consent to participate

- Non applicable.

Consent for publication

- The authors have no conflicts of interest to declare that are relevant to the content of this article.

Authors' contributions

- All authors participated in the experimental procedures, the preparation and writing of the article.

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