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RESEARCH

Microstructural and Tribological Characterization of API X52 Dual-phase Steel

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A B S T R A C T

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.

1. INTRODUCTION

Dual Phase (DP) steels, composed of a soft ferrite matrix and dispersed hard martensite islands developed via heat treatment, are widely used as a structural material in the automotive industry because they offer a good combination of high strength and ductility [1]. 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]. R.O. Rocha et al. [6] studied the effects of some intercritical annealing parameters on the microstructure and mechanical properties of a cold rolled DP steel. Other researchers have worked on the effect of welds in the life cycle of DP steels [7], or on the effect of laser welding parameters [8]. 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 [9]. A number of researchers investigated the impact of martensite morphology and volume fraction on the tensile of dual-Phase steels. Pierman and al. [10] 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 [11,12]. 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 [13-16]. Wei Xu and al. [17] 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 Materials

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.

Element	C	Mn	Si	Nb	V	Ti	Fe
Composition [wt. %]	0,12	1,22	0,23	0,03	0,03	0,002	Balance

Figure 1 shows the microstructure of as-received X52 steel, which consists of ferrite (white regions) and pearlite (black regions).

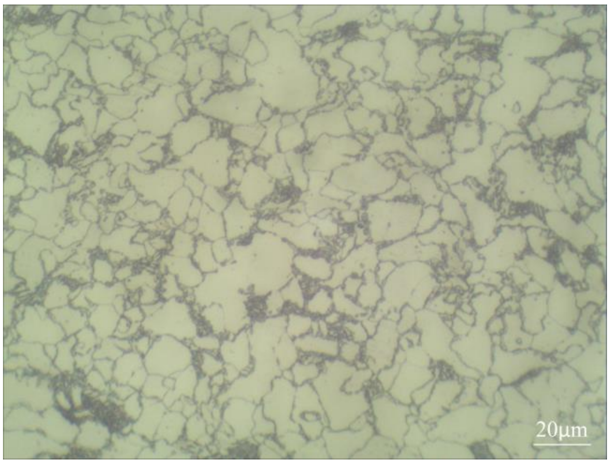


Fig. 1. Ferrite/perlite microstructure of X52 steel as received.

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 2) 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.2a.
- SQ: full austenisation at 940°C for 30 min followed by intercritical annealing at 760°C and 800°C for 30min followed by water quenching, as shown in Fig. 2b.

- 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. 2c.

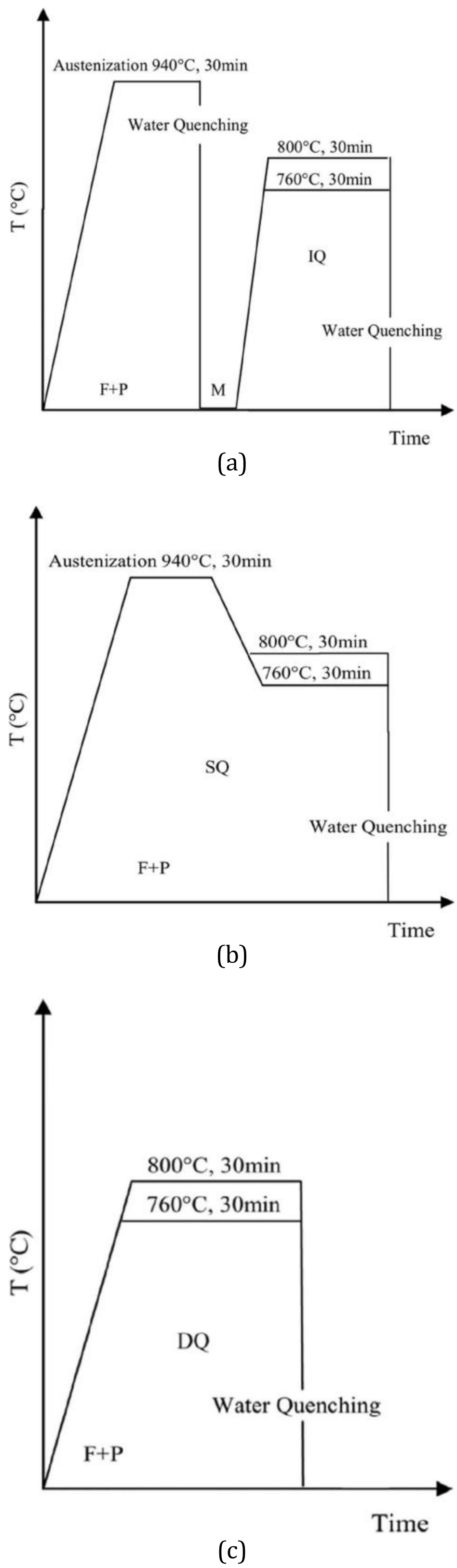


Fig. 2. Schematic drawing of the heat treatment procedure: (a) IQ, (b) SQ and (c) DQ.

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 with rotary movement. 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 3.

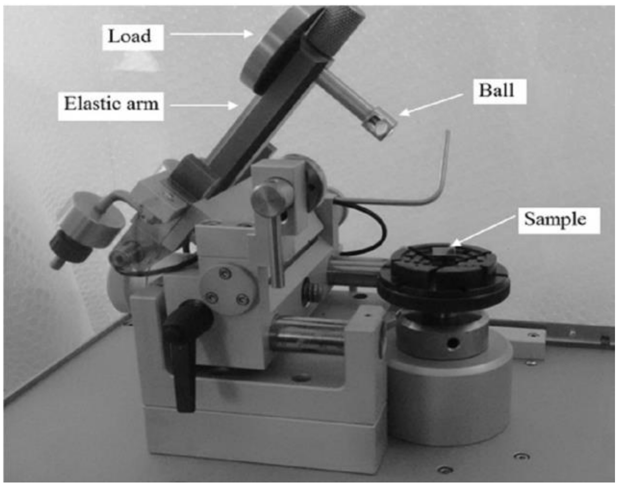


Fig. 3. View of the used ball-on-disc tribometer.

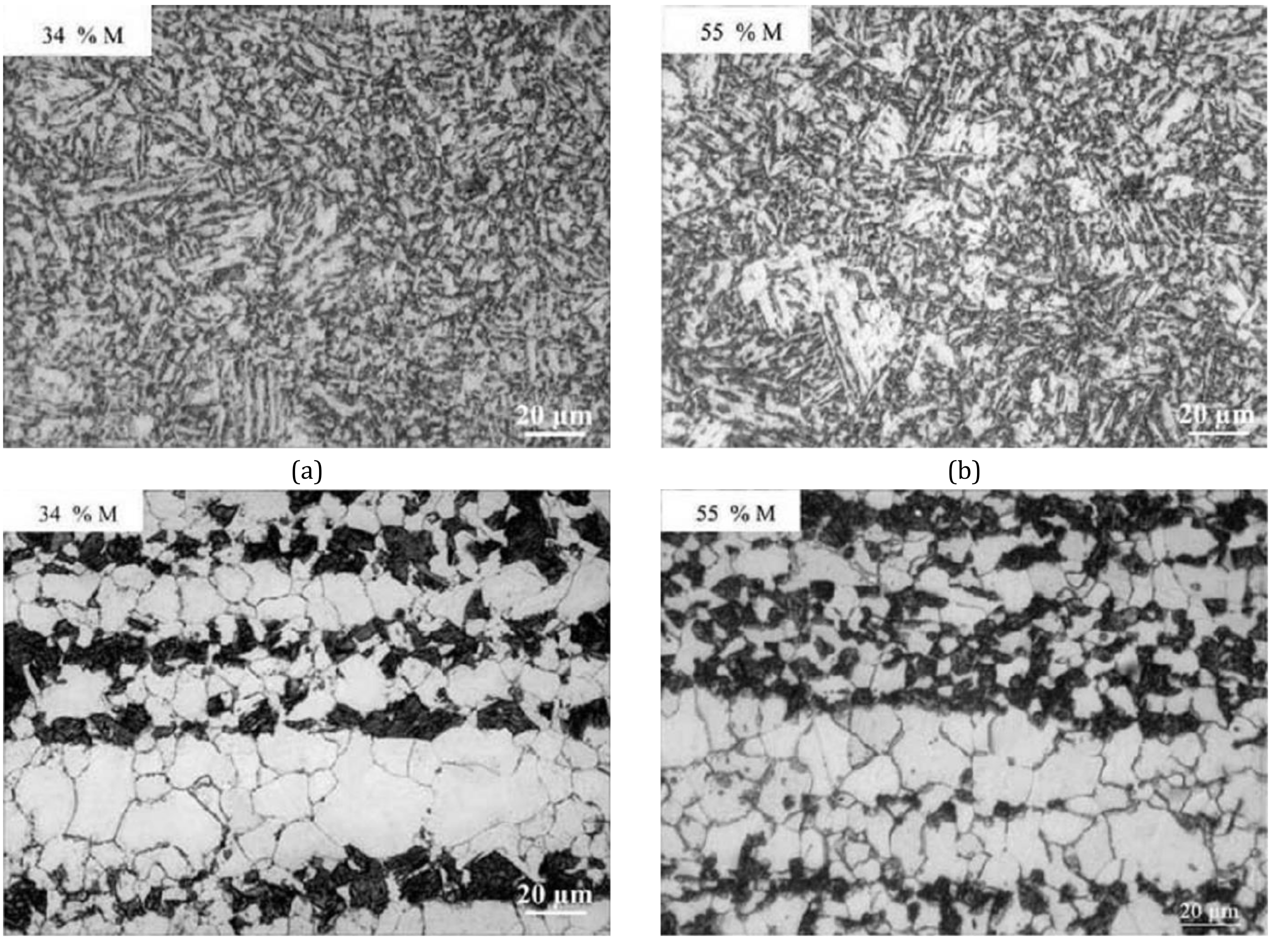
To evaluate volume loss recorded during the wear tests, an optical profilometer (Altisurf 500) equipped with a 3D image processing software (Altimap topography XT) and a 2D roughness profile was used to measure surface roughness and the wear volume loss.

3. RESULTS AND DISCUSSIONS

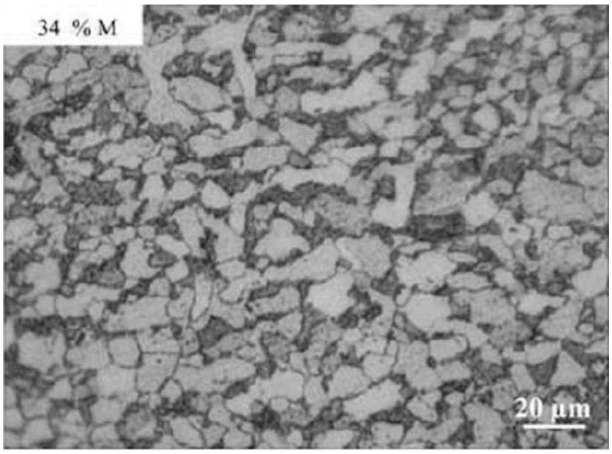
3.1 Microstructures

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 4). The martensite and ferrite fractions depend on the annealing temperature (760 °C or 800 °C).

Figure (4) shows a dual-phase microstructure, obtained by the IQ treatment, consisting of fine and fibrous martensite uniformly distributed within the ferrite matrix. According to literature [18], this morphology is obtained from the nucleation of austenite along the lath boundaries of prior martensite during the intercritical annealing. The microstructure of the sample subjected to the SQ treatment shows Ferrite and Martensite band morphology with non-uniform distribution of phases. The microstructure is austenite before entering into the two-phase (α + γ) region. Upon lowering the temperature to the two-phase region, ferrite nucleates at the grain boundaries of austenite and grows within the austenite grains [19]. The microstructure of the sample subjected to the DQ treatment shows a dual-phase structure characterized by a microstructure which consists of fine martensite islands distributed in the grain boundaries of polygonal ferrite matrix, obtained from intercritical temperatures 760 and 800 °C. When heating in the (α + γ) region, 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.

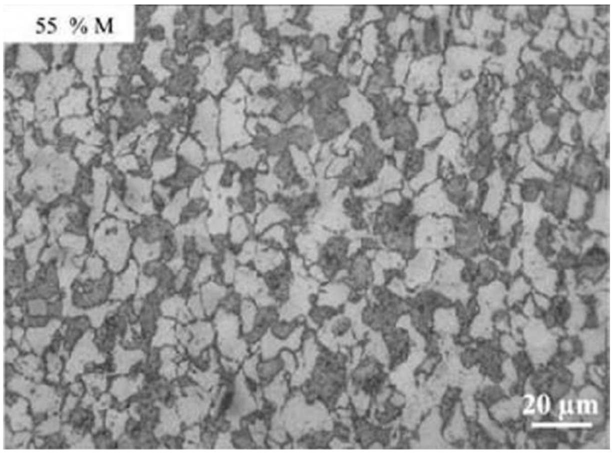


(a) (b) (c) (d)



(e)

760 °C



(f)

800 °C

Fig. 4. Optical micrographs of dual phase microstructures of: (a,b) IQ treatments at 760 and 800 °C, (c,d) SQ treatments at 760 and 800 °C and (e,f) DQ treatments at 760 and 800 °C.

3.2 Microhardness and tensile tests

The yield strength (YS), highest ultimate tensile strength (UTS), total elongation (TE) and the microhardness values of the specimens obtained in this work after IQ, SQ and DQ heat treatments and those of other searchers are grouped in table 2. The variation of the mechanical properties of dual phase steel were strongly influenced by morphologies, volume fraction and the distribution of the dispersed martensitic phase. A comparison of tensile properties between IQ, SQ and DQ specimens, shows that IQ samples have the best mechanical properties, the IQ specimens show an excellent combination of strength and ductility, this is due to the fine and fibrous martensite morphology. The fine size of the ferrite which also contributes to improved mechanical properties.

Table 2. Mechanical properties values obtained in this work compared to the literature.

Steel	Specimen	AT (^{0}C)	VFM (%)	UTS (MPa)	Yield Stress (MPa)	TE (%)	UE (%)	Microhardness ($HV_{0.2}$)	Microhardness ($HV_{0.01}$)	Hardness (HV_{30})	Ref
API X52	IQ	800	55	845	525	25	/	210			This work
		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		200			
		760	34	670	405	22		170			
Low carbon steel (0.18% C)	DQ	720	28	736	441	27.6	/	/		222	[25]
	SQ	720	27	703	459	28.9				202	
	IQ	720	33	757	451	33.5				216	
Low carbon steel (C ≈ 0.1% C)	DQ	760	22.1	638	/	23.3	/	/	185	/	[22]
		780	25.5	673	/	21.0			195		
		800	30.4	699	/	19.3			199		
		820	38.1	718	/	18.6			203		
Low carbon steel (C ≈ 0.1% C)	DQ	750	25	814	337		12.4	/		/	[21]
		770	35	873	342		9.7				
		790	45	933	363		8.2				
		810	60	950	436		6.3				
		830	95	1048	556		4.2				
Low carbon steel (C ≈ 0.1% C)	DQ	820	/	865	545	13.3	/	/		/	[23]
	IQ	820	/	729	445	18					
	SQ	820	/	703	401	11.3					
Low carbon steel (0.1% C)	DQ	780	22.8	637	413	42.5		/		/	[24]
	IQ	780	22.7	655	413	52.9					
	SQ	750	23	568	325	51.9					

AT: Annealing Temperature, **VFM**: Volume fraction of martensite

The DP steels with finely dispersed ferrite present the high dislocation density, which leads to prevent the cleavage crack propagation and increases the toughness and ductility [19,20]. This agrees with Sodjit and Uthaisangsuk [21], C. Dulucheanu et al. [22], Bayram et al. [23] and Y. Tomita [24] Ashrafi et al. [25] who found that the mechanical properties influenced by the morphology of the ferrite and martensite.

The proportion of martensite obtained on API X52 steel relatively higher than that obtained on other steels, can be justified by the presence of Nb and V in X52 steel, which benefit the martensitic transformation [26].

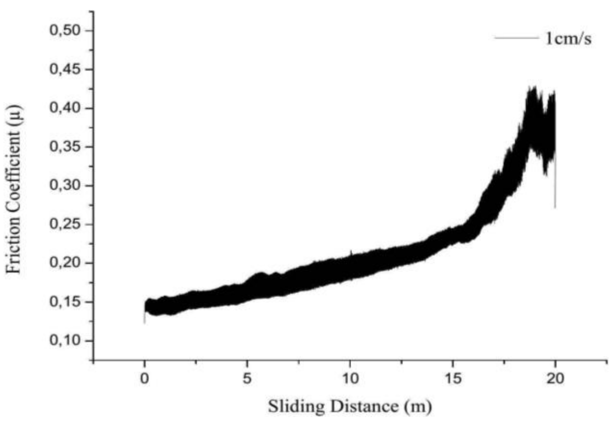
In general, the mechanical properties are very comparable depending on the conditions of the heat treatments followed.

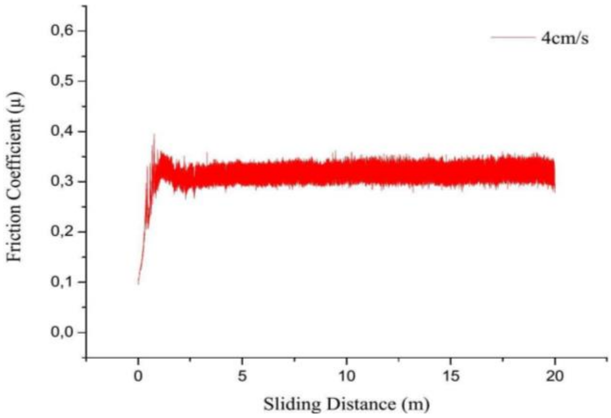
3.3 Wear and friction tests

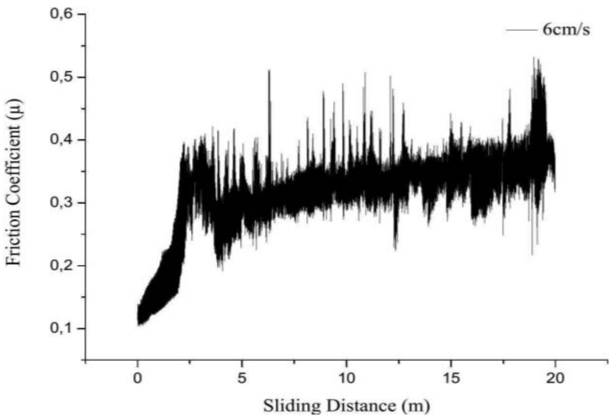
The evaluation of the effect of IQ, SQ and DQ heat treatments on the wear test of X52 steel was made by evaluating the friction coefficient during the test and by recording the volume loss after the wear test.

Friction coefficient evaluation

For the selection of the sliding speed in wear tests, we have conducted a wear test series at 1, 4, 6 and 9 cm/s for a distance of 20 m. Results showed that the best curves of friction coefficient correspond to 4 cm/s sliding speed. The speed of 1 cm/s is relatively low, while the curves corresponding to 6 and 9 cm/s show large fluctuations (Figure 5), which makes estimating the friction coefficient with accuracy very difficult. For the rest, we will consider only the curves made at a speed of 4 cm/s in the discussion of the effect of heat treatments on the friction coefficient and wear.







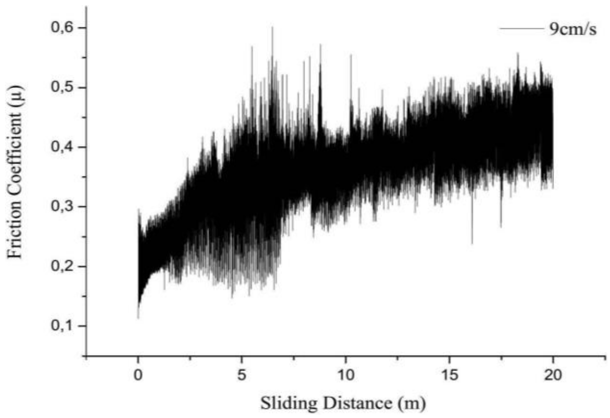
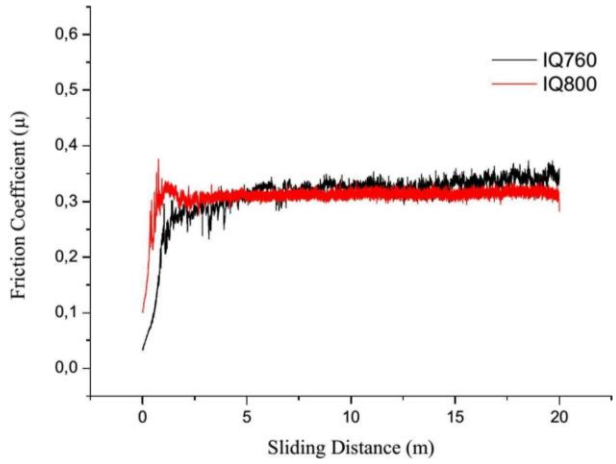


Fig. 5. Wear test series at sliding speed of 1, 4, 6 and 9 cm/s.

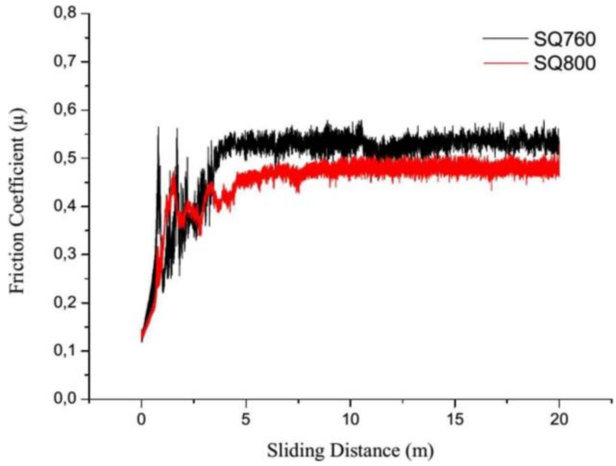
The tested surfaces result in coefficient values between 0.30 and 0.55 for all tested samples. In each case, the evolution of the tangential force as a function of the cycle's number can be decomposed into two phases whatever the heat treatment applied (Figure 6 and 7):

- 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 [27].

- 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 [28].



(a)



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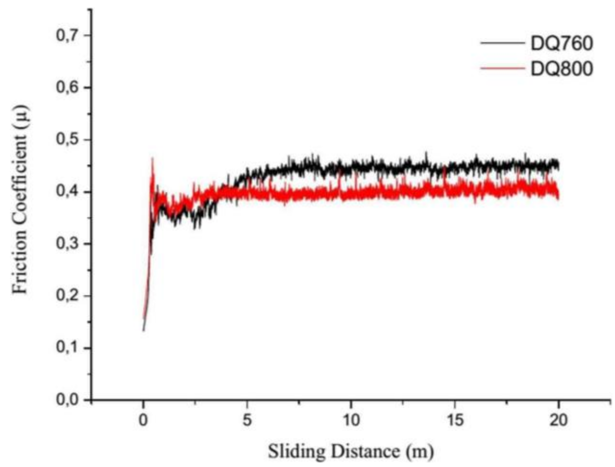
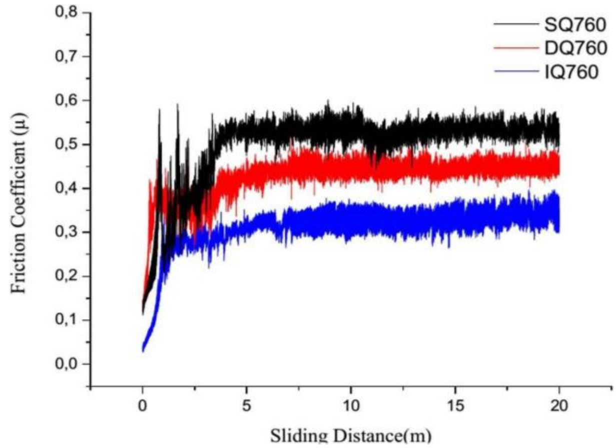
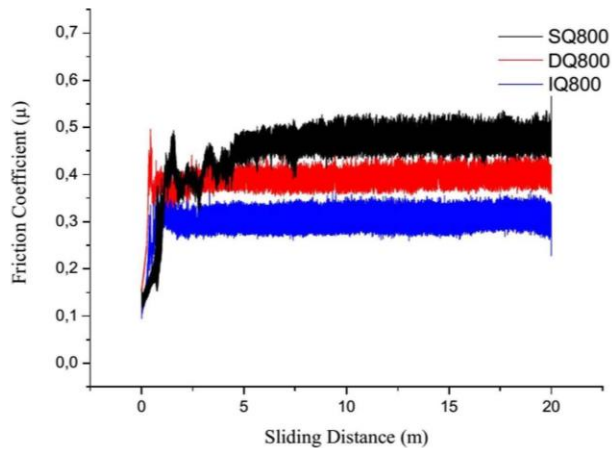


(c)

Fig. 6. Variation of friction coefficient with sliding distance for a load of 5N at 760°C and 800°C: (a) IQ, (b) SQ, (c) DQ.



(a)



(b)

Fig. 7. Variation of friction coefficient with sliding distance for a load of 5N for IQ, DQ and SQ treatments: (a) 760°C, (b) 800°C.

Figure 6 shows the variation of the friction coefficient with the sliding distance at 5N load for IQ, DQ and SQ at 760°C and 800°C.

The friction coefficient decrease when the martensite volume fraction increases. 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, decreasing the surface of contact and reduce the possibility of the formation and propagation of cracks during delamination.

Figure 7 shows the variation of the friction coefficient as a function of the sliding distance for IQ, DQ and SQ specimens. 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 [25].

Volume loss evaluation

The volume loss recorded on the different dual-phase samples after the wear tests under 5N load are presented in Figure 8.

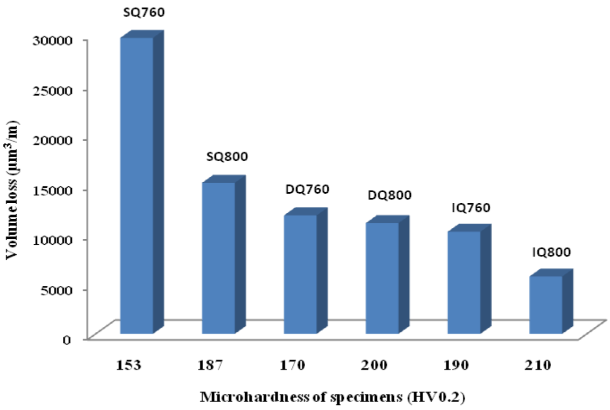


Fig. 8. Wear rate of different samples with different microhardness levels at 5N load.

A first observation of the volume loss curves as a function of the microhardness obtained in figure 8 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.

It is clearly seen that the IQ specimens show greater wear resistance than SQ and DQ specimens. The IQ treatment provided the best combination of strength and ductility due to the finer distribution of martensite and ferrite (high dislocation density) which contribute to enhance the wear resistance compared with that for other samples. However, when the grain size of the ferrite is increased result in increasing of wear volume loss [25,29,30].

The difference in volume loss between the different samples is due either 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 µm³/m for SQ760 and 15000 µm³/ 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 µm³/m for IQ760 and 5000 µm³/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 µm³/m and 14000 µm³/m for DQ800 and DQ760 respectively.

***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.

According to the values of the friction coefficient and the volume loss after dual-phase heat treatment presented in Table 3, we can advance the following observations and findings:

- The friction coefficient and volume loss values of IQ are 0.3 and 5741 for specimens annealed at 800 °C and 0.33 and 10214 for specimens annealed at 760 °C respectively. It can be seen that the friction coefficient decrease when the martensite volume fraction increases. Findings are similar to SQ and DQ specimens. This agrees with H. Saghafian et al. [31] results, who have also reported that the wear resistance of DP steels increases with the increasing volume fraction of martensite (VFM). In fact, they found that the friction coefficient and volume loss values are 0.73 and 4 10^5 µm³/m with 43% VFM, 0.72 and 3 10^5 µm³/m with 53% VFM, 0.71 and 2.5 10^5 µm³/m with 72% VFM and 0.68 and 2 10^5 µm³/m with 81% VFM respectively. The comparison of results values shown in Table 3 confirm the remarks already put forward. Indeed, in almost all cases when the volume fraction of martensite increases, the friction coefficient and volume loss decrease.

- The friction coefficient, and the volume loss values of specimens annealed at 800 °C are 0.3 and 5741 for IQ specimen, 0.47 and 15115 μm³/m for SQ specimen, and 0.4 and 11080 μm³/μm for DQ specimens respectively. It is to be noted that the IQ specimen has the lowest friction coefficient and volume loss than that of SQ and DQ specimens. Findings are similar to specimens annealed at 760 °C. This agrees with Ashrafi et al. [25] results, who found that the average friction coefficient increases in the order of IQ < DQ < SQ. In fact, they found that the friction coefficient and mass loss values are (0.7 to 0.85) and 0.098 mg/m for IQ specimen, 0.8 and 0.15 mg/m for SQ specimen, and 0.8 and 0.116 mg/m for DQ specimens respectively. This also agrees with R. Tyagi et al. [15] who found that wear rate decrease when the martensite volume fraction increase.

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

Steel	Specimen	AT (°C)	VFM (%)	Load (N)	Speed (cm/s)	Friction Coefficient	Volume loss (μm³/m)	Mass loss (mg/m)	Microhardness [$HV_{0.2}$]	Hardness [HV_{30}]	Ref
X52	IQ	800	55	5	4	0.3	$5.74\ 10^3$		210		This work
		760	34			0.33	$1.02\ 10^4$		190		
	SQ	800	55			0.47	$1.51\ 10^4$		187		
		760	34			0.52	$2.26\ 10^4$		153		
	DQ	800	55			0.4	$1.11\ 10^4$		200		
		760	34			0.45	$1.19\ 10^4$		170		
Low carbon steel (0.18% C, 1.25% Mn)	DQ	720	28	80	700	0.8		0.116		222	[25]
	SQ	720	27			0.8		0.151		202	
	IQ	720	33			0.7		0.098		216	
Low carbon steel (C, 0.21)	DQ	780	43	61.3	120	0.73	$4\ 10^5$			211	[31]
			53			0.72	$3\ 10^5$			245	
			72			0.71	$2.5\ 10^5$			285	
			81			0.68	2 105			327	
Low carbon steel (C, 0.42)	DQ	740	42	14.7	115	0.55	$8\ 10^4$			298	[15]
			51			0.54	$7\ 10^4$			336	
			59			0.52	$6\ 10^4$			367	

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).

- IQ specimens have the best mechanical properties (best combination of strength and ductility), this is due to the finer distribution of martensite and ferrite.

- IQ specimens show greater wear resistance, as expressed by its lower friction coefficient and volume loss, putting them usually used in some applications where high wear-resistance (the mining equipments for example) and in the automotive component.

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