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Analysis of the Effect of Heat Treatment Conditions of a Ferritic Stainless Steel on Residual Stresses and Tribological Behavior

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Abstract

In recent years, the scientific community has shown a great interest in the study of the wear performance of metallic materials under different test conditions, together with the measurement of residual stresses. Thus, the objective of the present work was to analyze the effect of heat treatment on residual stresses and tribological behavior of P410D ferritic stainless steel. The results showed that, with the increase in hardness of the material—derived from different heat treatment conditions, the resistance to micro-abrasive wear of P410D ferritic stainless steel increased, characterized by a decrease in wear volume. The residual stresses reported were “*tractive*”; additionally, it was observed that the lowest residual stresses values were related to the lower wear volumes values.

Keywords: stainless steel, heat treatment, residual stress, X-ray diffraction, micro-abrasive wear, tribological behavior

1. Introduction

Stainless steel is an iron-chromium-carbon alloy that has high resistance to oxidation and corrosion in varying environments. In addition to the presence of nickel (Ni), the main alloying element that ensures the chemical resistance to oxidation and corrosion is chromium (Cr), with 11% concentration by mass; such resistances are associated with the formation of a mixed oxides layer and its dissolution in the medium in which it is exposed. Based on the predominant constituent phase in its microstructure, stainless steels are classified mainly into three categories: *austenitic*, *ferritic*, and *martensitic* [1]. Thus, in Fe-Cr phase diagram (**Figure 1** [2]), the “*ferritic stainless steels*” are at the right of the austenitic field [3] and are classified as “*ferritic*

Figure 1.
Fe-Cr phase diagram [2].

stainless steels” the materials with a percentage greater than 12% Cr—by mass [3], possessing body-centered cubic crystalline structure.

In the ferritic microstructure, chromium (Cr) is configured as a substitutional atom, influencing hardening by solid solution [4], where “*hardness*” is a direct indicator of the wear resistance of ferritic stainless steels influenced by the type of heat treatment conducted. Additionally, some ferritic stainless steels can be austenitized at high temperature and cooled in a quench process, to obtain martensitic microstructure, which presents a high density of discordances.

In another scientific segment, the “*X-ray diffraction*” technique is applied for measuring residual stresses in metallic materials. It is conceptualized in the deviation of the propagation trajectory suffered by X-ray waves and is compared with the positions of the atoms of the analyzed material due to the changes caused by residual stresses themselves of the network parameters in the material [5] (**Figure 2**).

In X-ray diffractometer, there is a coordinated movement between the Coolidge tube—generator of the X-rays—and the specimen. The rotational motion of the Coolidge tube defines the “*angle of incidence of the X-rays— θ* ” on the surface of the material and the rotational movement performed by material analyzed defines the “*angle of rotation of the specimen— ψ* ” (**Figure 3**).

As the specimen is rotated by a defined angle ψ , the angle of incidence of the X-rays (θ) is altered accordingly. The angle of rotation of the specimen (ψ) is measured in relation to a normal axis to the atomic planes, defined by “*Miller indexes— hkl* ”, parallel to the surface of the specimen and the angle of incidence of the X-rays (θ) is referenced in relation to the surface of the material.

From there, a graph of $\theta = f(\sin^2\psi)$ is raised, which results in a 1st Degree Equation (Eq. (1)), by which the residual stress is calculated. This technique is called “ *$\sin^2\psi$* ”.

Figure 2.
Physical principle of the “X-ray diffraction” technique.

Figure 3.
“X-ray diffraction”: Definition of the angles “ θ ” and “ ψ ”—“angle of incidence of the X-rays” and “angle of rotation of the specimen”, respectively.

$$\sigma_{res} = m \frac{E}{2(1 + \nu)} \cot (\theta_0) \tag{1}$$

In Eq. (1), “ m ” is the angular coefficient of the generated straight line, “ E ” and “ ν ” are the longitudinal elasticity modulus and the Poisson coefficient of the analyzed material, respectively, and “ θ_0 ” is obtained for $\psi = 0^\circ$. For $m < 0$, the residual stress acting on the material will be “compressive” and, for $m > 0$, the residual stress will be “tractive”.

On the tribological side, “wear” can be defined as “damage on the solid surface, involving progressive loss of mass, due to the relative movement between the surfaces and contact with other material or materials” [6]. Together with the given general definition, each type of wear has a specific setting—“Abrasive wear”, as discussed in this work, is due to hard particles, or hard protuberances, forced against and moving along a solid surface [7].

In industrial sectors where wear causes downtime, there occur decrease in production and involve high maintenance costs, so it is not enough to acquire knowledge only in mechanical and/or metallurgical manufacturing of materials and processes—it is also important to research and to understand the wear processes that act in specific working conditions.

Through analyses of wear craters generated during micro-abrasive wear tests by rotating ball, it is possible to predict or, at least, estimate the abrasive wear behavior of

a material or any mechanical component in real working conditions. Additionally, such analyses can be expanded and better understood along with residual stress measurements conducted by the “X-ray diffraction” technique.

Thus, the objective of this work was to analyze the effect of heat treatment on residual stresses and tribological behavior of a dual-phase stainless steel, under conditions of micro-abrasive wear.

2. Materials, equipment, and scientific methodology

2.1 Materials

Six P410D ferritic stainless steel specimens were used—named “Specimen 1”, “Specimen 2”, ..., “Specimen 5” and “Specimen 6”. The chemical composition of P410D ferritic stainless steel is specified in **Table 1**.

Each specimen had dimensions of, approximately, 27x10x5 [mm], being conditioned under different parameters of heat treatment of quench, in a combustol oven, whose application temperatures were monitored by thermocouples. **Table 2** shows the temperature values adopted for the heat treatments of the specimens.

After heat treatments of quench, the specimens were subjected to metallographic analysis, being, initially, hot embedded, sanded, polished and chemically etched with CATELA—2 g of picric acid, 6 ml of acetic acid, 3 ml of hydrochloric acid, and 100 ml of ethyl alcohol. After, microstructural images were acquired by optical microscopy.

As counter-body, an AISI 52100 steel bearing (quenched and tempered) was used, with diameter $D = 25.4$ mm ($D = 1$ ”—standard size).

Figure 4 presents an image of the AISI 52100 steel bearing microstructure and its chemical composition. Its microstructure is composed only of two phases: the tempered martensitic matrix (with the characteristic shading contrast) and the small M_3C carbide precipitates homogeneously distributed. The darker and lighter areas in the matrix are typical of tempered martensite and show the gradients of etching depending on the orientation of the martensite lenses and the density of the carbide precipitation in different regions (due to small differences in chemical composition).

The abrasive slurry was prepared with black silicon carbide (SiC)—average abrasive particle size of $a_p = 3$ μm —and distilled water. **Figure 5** shows an image of the SiC

Chemical element	Quantity—% mass
Cr	11.1334
Mn	0.5332
Si	0.5260
Ni	0.3046
P	0.0271
C	0.0107
S	0.0005
Fe	Balance
N [ppm]	128

Table 1.
Chemical composition of P410D ferritic stainless steel—% mass.

Specimen	Soaking temperature (°C)
1	822
2	854.1
3	879.5
4	895.4
5	953.3
6	973

Table 2.
Temperature values defined for the heat treatments of P410D ferritic stainless steel specimens.

Chemical Element	Quantity – % mass
C	1.04
Mn	0.35
Si	0.25
Cr	1.45
Fe	Balance

Figure 4.
Microstructure and chemical composition of the AISI 52100 bearing steel sphere—Metallurgical state: Quenched in oil at 860°C and stress-relieved at 200°C for 1 h.

abrasive particles (**Figure 5a**), which was obtained by scanning electron microscopy (SEM), and its abrasive particle size distribution (**Figure 5b**).

Table 3 presents the hardness values of the materials used in this work (specimens, test ball, and black silicon carbide). The numbers of the specimens were established in ascending order, along with the respective hardness values.

2.2 Tribometer

Figure 6 shows the ball-cratering tribometer used in this work. Having a “fixed-ball” mechanical configuration, the test shaft was divided into two distinct parts,

Figure 5. Abrasive particles of black silicon carbide (SiC): (a) image obtained by scanning electron microscopy (SEM) and (b) particle size distribution.

	Material	Hardness—[HV]
Specimen 1	P410D ferritic stainless steel	177
Specimen 2	P410D ferritic stainless steel	200
Specimen 3	P410D ferritic stainless steel	222
Specimen 4	P410D ferritic stainless steel	238
Specimen 5	P410D ferritic stainless steel	297
Specimen 6	P410D ferritic stainless steel	304
Test ball	AISI 52100 steel bearing	856
Abrasive material	Black silicon carbide (SiC)	2380–2630

Table 3. Hardness of the materials used in this work.

called “motor test shaft” and “moving test shaft” (**Figure 6a**). In turn, each of these parts has a face with a concave radius of $R = 12.7\text{ mm}$ ($R = \frac{1}{2}''$), thus, enabling the accommodation of a test sphere of diameter $D = 25.4\text{ mm}$ ($D = 1''$). For the application of the normal force— N , was adopted a “dead weight” mechanical system (**Figure 6b**).

The “motor test shaft” is driven by a direct current electric motor of power $P = 30\text{ W}$ (**Figure 6a**), under a rotating speed of $n = 56\text{ rpm}$.

Finally, the fixation of the specimen is performed by device shown in **Figure 7**.

Figure 8 shows one of the specimens before the tribological tests.

2.3 Research methodology

Table 4 shows the test conditions established for the micro-abrasive wear experiments. A normal force value was defined for the tribological tests, $N = 2\text{ N}$, together with an abrasive slurry concentration of $C = 25\% \text{ SiC} + 75\% \text{ distilled water}$ —in volume.

The test time, for all wear experiments, was established at the value of $t = 20\text{ min}$. With the test ball diameter of $D = 25.4\text{ mm}$ and a ball rotating speed of $n = 56\text{ rpm}$, a sliding distance of $S \approx 90\text{ m}$ was calculated.

Figure 6.
Tribometer “ball-cratering” of “ball-fixed” mechanical configuration: (a) “motor test shaft”, “test ball” and “moving test shaft” mounted on the tribometer; (b) application of normal force by “dead-weight” mechanical system.

Figure 7.
Device for fixing specimen.

For each specimen, three ball-cratering micro-abrasive wear tests were conducted and, during the experiments, the abrasive slurry was continuously dripped between the specimen and the test ball.

Figure 8.
Image of “Specimen 5”, before the ball-cratering wear tests.

Test conditions	
Normal force— N [N]	2
Abrasive slurry concentration— C [% vol.]	25% SiC +75% distilled water
Test time— t [min]	20
Rotation of the test ball— n [rpm]	56
Sliding distance— S [m]	≈ 90
Number of repetitions	3

Table 4.
Test conditions defined for the micro-abrasive wear tests by fixed rotating ball.

All wear craters were generated without removing the specimens from the clamping device available in the equipment since it has the “horizontal” and “vertical” positioning displacements feature.

Diameters of the wear craters (b) developed during micro-abrasive wear tests were measured by optical microscopy. Subsequently, the values of the wear volume (V) of the respective wear craters were calculated using Eq. (2):

$$V \cong \frac{\pi \cdot b^4}{64 \cdot R} \text{ for } b \ll R \quad (2)$$

Where “ R ” is the radius of the test ball.

Finally, the effect of the heat treatment conditions on P410D ferritic stainless steel was validated based on the statistical analysis of the wear craters volumes and on the behavior of the wear volume as a function of the hardness of each specimen— $V = f(H)$, respectively.

3. Results and discussion

3.1 Microstructural analysis

Figure 9 presents the metallurgical microstructures of P410D ferritic stainless steel specimens heat-treated under different soaking temperatures.

Figure 9. Metallurgical microstructures of the P410D ferritic stainless steel specimens after different heat treatment conditions. (a) “Specimen 1”—822°C, (b) “Specimen 2”—854.1°C, (c) “Specimen 3”—879.5°C, (d) “Specimen 4”—895.4°C, (e) “Specimen 5”—953.3°C, and (f) “Specimen 6”—973°C.

It was observed in *Specimen 1* (**Figure 9a**) that the soaking temperature was not high enough for the recrystallization of the ferritic matrix, due to the presence of deformed grains. The presence of recrystallized grains, deformed grains, pores, and martensitic phase can be observed in the *Specimen 2* (**Figure 9b**). In *Specimen 3*—**Figure 9c**, there is a greater presence of recrystallized grains with an increase in the martensitic phase. The *Specimen 4* (**Figure 9d**) presented a higher amount of martensitic phase and grains apparently smaller than the previous samples, with the presence of some deformed grains. In *Specimen 5* (**Figure 9e**), deformed grains are hardly noticed and an increase in the amount of martensite phase was observed. Finally, in *Specimen 6* (**Figure 9f**) occurred the maximum point of martensitic structure formation, with no more deformed grains.

3.2 Residual stresses

Table 5 and Figure 10 presents the residual stress values reported for the P410D ferritic stainless steel specimens, together with the respective hardness values. All residual stress values measured were “tractive” and inversely proportional to the hardness of each specimen.

3.3 Tribological behavior

Figure 11 presents the specimens used for the analysis of results, already with all micro-abrasive wear tests by rotating ball conducted on their surface, and Figure 12 displays images of wear craters produced.

Table 6 shows the arithmetic-mean of the wear volumes (V), as well as the respective values of the standard-deviations, for each one of the specimens; it is noted that, for all specimens, the standard-deviation, in reference to the arithmetic-mean of the values of wear volumes (V), was below 10%.

Figure 13 displays the behavior of the wear volume (V) as a function of the hardness (H) of the specimen— $V = f(H)$.

Specimen	Hardness—[HV]	Residual stress—[MPa]
1	177	+165
2	200	+156
3	222	+128
4	238	+102
5	297	+95
6	304	+87

Table 5.
Values of residual stress measured in the specimens of P410D ferritic stainless steel.

Figure 10.
Residual stress value acting on each specimen of P410D ferritic stainless steel, related to its hardness.

Figure 11.
Wear craters generated on P410D ferritic stainless steel specimens.

It was observed that, with the increase in hardness of the material, the volume of worn material—the volume of the wear crater—decreased, following, in qualitative agreement, the *Archard Equation* (Eq. (3)):

$$\xi = K_W \cdot K_F \int \frac{P \cdot v}{H_{CP}(T)} dT \tag{3}$$

By directing the quantities pertinent of the *Archard Equation* to the “ball-cratering” wear test, they can be defined as:

- ξ is a quantitative quantity, where the higher its value the greater is the severity of the micro-abrasive wear process—or, the greater the volume of worn material;
- K_W and K_F are constant quantities related to materials belonging to the tribological system—specimen, abrasive particles, and test ball—during the micro-abrasive wear process;
- P is the contact pressure reported in the tribological system “specimen—abrasive particles—test ball”, defined by Eq. (4):

$$P = \frac{N}{A} \tag{4}$$

Where A the projected area of the wear crater.

- v is the tangential velocity of the test ball, defined by Eq. (5):

$$v = \pi \cdot D \cdot n \tag{5}$$

- H is the hardness of the specimen, as a function of the temperature (T)— $H = f(T)$.

Figure 12.
Wear craters obtained from “ball-cratering” wear tests conducted in this work: (a) Specimen 2 and (b) Specimen 4.

Specimen	Hardness— H [HV]	Wear volume— V arithmetic- mean [mm ³]	Wear volume— V standard- deviation [mm ³]
1	177	0.0139	0.0009
2	200	0.0119	0.0010
3	222	0.0118	0.0002
4	238	0.0115	0.0010
5	297	0.0111	0.0006
6	304	0.0102	0.0005

Table 6.
Arithmetic-mean of the values of wear volumes (V) with the respective values of standard-deviations.

Analyzing the behavior of the physical parameters of the *Archard Equation* during the micro-abrasive wear process, it is noted that the tangential velocity value of the test ball was the same for all test conditions— $n = 56$ rpm; with this, the tangential velocity of the test ball remained constant for all specimens.

Figure 13.
Behavior of the wear volume (V) as a function of the hardness (H) of the specimen— $V = f(H)$.

Additionally, the temperature of each specimen remained constant at room temperature, resulting in the hardness (H) of the materials analyzed remain unchanged during all ball-cratering wear tests.

However, following the *Archard Equation*, ξ and H are inversely proportional; therefore, the increase in H caused a decrease in ξ , characterizing, consequently, lower severity of wear related to a lower volume of wear (V) generated.

Finally, the only physical quantity that varied in a decreasing way during the ball-cratering wear tests was the contact pressure (P)—since the normal force (N) remained constant, the projected area (A) of each wear crater increased with the progressive increase in the sliding distance.

In fact, in all wear tests, the contact pressure (P) followed, under the qualitative agreement, the approach detailed by Cozza [8, 9] in previous works, where P decreased as a function of the projected area of the wear crater for each specimen. For each hardness value, different values of ξ were obtained, related to the calculated values of wear volumes (V).

Finally, based on **Figure 13**—which exhibited practical results of the variation of wear volume (V) as a function of the hardness of the specimen (H)—and by theoretical complementation departing from *Archard Equation* (Eq. (3)), it can be said that the results generated are within the technical-scientific agreement of micro-abrasive wear.

4. Conclusions

The results obtained in this scientific work showed that the wear volume decreased with an increase in the hardness of P410D ferritic stainless steel specimens—due to the different heat treatment conditions.

Increase in oven temperature led to an increase in solution treatment temperature and hardness of P410D ferritic stainless steel along with a decrease in residual stress.

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