



# STUDY OF THE SURFACE MORPHOLOGY OF A CARBON STEEL AFTER TESTINGS CORROSIVE-CAVITATIVE-EROSIVE WEAR IN AQUEOUS MEDIUM WITH SALT (NaCl), CO<sub>2</sub> AND SOLID PARTICULATES (SiO<sub>2</sub>) FRACTIONS\*

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## **Abstract**

Wear mechanisms of materials that constitute the equipments and operating parts in the pre-salt area during the production of petroleum and gas can include corrosion, corrosion-cavitation and corrosion-cavitation-erosion. In the laboratory, it is realizable, but at times it is inaccurate to reproduce or visualize some of these mechanisms due to the real complexities caused by hydrodynamic flows, and the physico-chemical and mechanical non-linearities. The main objective of this work was to study coupons of low carbon steel under laboratory conditions, such as corrosive, cavitative-corrosive (CO<sub>2</sub>) and corrosive-erosive (CO<sub>2</sub> + SiO<sub>2</sub>), in a stirring aqueous saline solution (0 and 5.0 m/s) at two levels of temperature, with bubbling of gas (5.0 L/min) and contaminated with or without (2.5 % mass), by solids particles of SiO<sub>2</sub>. The surface of the coupons subjected to upstream flow (0° in a cylindrical generatrix of coupon), and downstream flows (180°) were analyzed by profilometry. The measurements of roughness and waviness of all coupons were statistically analysed using Statgraphics® Centurion XVI, at a confidence level of 95% and significant differences observed in some matrices were discussed.

**Keywords:** Tribochemistry; Corrosion; Erosion; Cavitation; Surface texture.

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## 1 INTRODUCTION

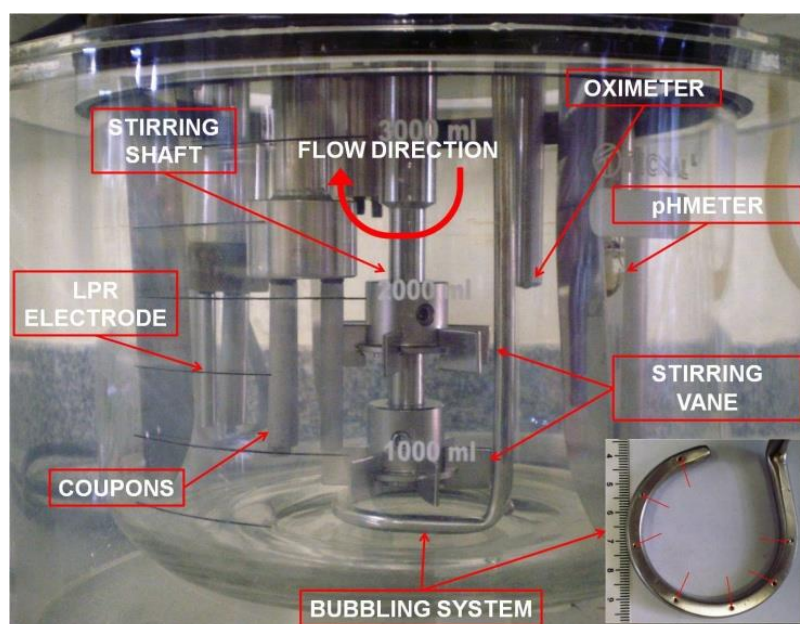
The production of petroleum in the offshore fields as well as the transportation of the petroleum, water, particulate matter and gas, including those associated with the pre-salt layer, has demanded detailed study about the main wear mechanisms of the materials including those defined as corrosion, corrosion-cavitation and corrosion-cavitation-erosion. It is a complex study. It could be associated with the presence of O<sub>2</sub>, H<sub>2</sub>S, CO<sub>2</sub>, bubble formation and erosion by hard particles, as discussed by Nunes da Silva [1]. Besides, this complexity may be increased by the action of microorganisms on the severity of wear rate as well as by the particulated matters such as CaCO<sub>3</sub>, SiO<sub>2</sub>, iron oxides, sulfides and so. Geometric features of particles, flow rates and pressure fields have been studied and modeled to correlate them with valves, equipment and damage to oil pipelines, such as pites, scars, wrinkles/waves, residual stresses, striations, residual stresses, fatigue, as related in several investigations [2-5].

The erosion is considered as a purely mechanical phenomenon, in which the metal is removed or mechanically destroyed, so rendering physical changes. The erosion-cavitation-corrosion is considered as a tribochemical phenomenon.

As a result of the proper erosion and/or cavitation, the surface is almost free from possible corrosion products, due to the bombardment of the particles within the fluid (solid particles in a liquid, liquid droplets in a gas, solid particles in a gas) on the solid surface, as a result of extremely high speeds of the fluid flow (> 10 m/s). All types of equipments that are exposed to fluids in motion are subjected to erosion-cavitation-corrosion [6,7]. The control and management of the erosion-corrosion synergism are required for operational safety and the reduction of production costs.

Some of the relevant aspects of tribochemical wear of a low-carbon steel has been investigated at the Federal University of Rio Grande do Norte, Brazil, and this work is a little part of this investigation.

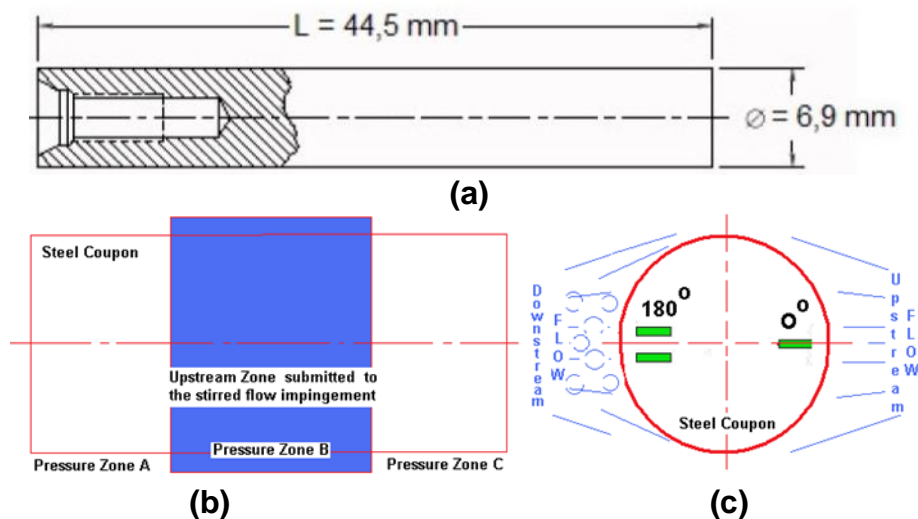
## 2 MATERIAL AND METHODS



**Figure 1.** Electrochemical Cell equipped with a CO<sub>2</sub> bubbling system (detail at right), a stirring for the saline solution, a linear polarization resistance (LPR) electrodes, coupons, oximeter and pHmeter.

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The electrochemical cell, Figure 1, was developed and tested by LabCorr-UFRN to investigate ferrous materials used in pipelines and analogous mechanical structural parts that are exposed to aqueous saline solutions in the presence of petroleum and gas. By the first time, it was included or not 2.5% SiO<sub>2</sub> in the aqueous solution trapped in this cell during some tests for analyze detectable changes in the fluid flow concerning to its erosive role and tribochemical action as wear rate inhibitor or activator. The physical dimensions of this particles ranging from 100 to 500 m in size. A total of 84 cylindrical coupons were manufactured using AISI 1018 steel (Table 1(a) and reference electrode Table 1(b)) in accordance with the NACE RP 0775 Standard (1999), Figure 2(a). The samples were treated for 24 hours in a saline solution (10,000 ppm of Cl<sup>-</sup>) at two temperatures by testing, 30°C or 70°C, respectively. The saline solution was stirring (5.0 m/s) or unstirring to cause, or not, a forced turbulent flow and bubbling of CO<sub>2</sub> gas (5.0 L/min), in order to add some of the different environmental combinations. The overall methodology, the cell and each experimental step of this work are carried out according to Nunes da Silva [1]. Visual Inspection, periodic (R<sub>sm</sub> and W<sub>sm</sub>) and stochastic (Ra, Rq and Rz, Wa, Wq and Wz) parameters of the surface roughness and waviness, respectively, were evaluated in a pair of diametrically opposite (0° and 180°, Figure 2(c)) generatrices of each cylindrical coupon. A set of three cylindrical zones A, B, C, Figure 2(b), was stratified conceptually. They can be visualized as a function of the surface response to the multiphase flow pressure field, temperature and ionic concentration fluctuations. These scenarios concern to the influence of the stirring or unstirring flow distributed in the upstream and downstream zones of the flow around each zone of the tested coupons. The Multitrend software V3.09® (CorrOcean ASA) monitors the rate of corrosion [8] as a function of time. The profilometry and microhardness resulting of the machining process of coupons are presented in Table 2, for additional informations see [8].



**Figure 2.** (a) Dimensions of the Cylindrical coupon for testing;(b)Side view of the coupon and the three main flow pressure zones; (c) Top view of coupon and the two main generatrices at 0o (upstream) and 180o (downstream).

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**Table 1.** Chemical composition (% mass) of (a) AISI 1018 steel (coupons) and (b) AISI 304 L (reference electrode)

(a)	C	Cu	Cr	S	P	Mn	Mo	Ni	Si	Fe
	0,18	-	-	0,05	0,04	0,85	-	-	-	98,88
(b)	C	Mn	Si	P	S	Cr	Ni	Mo	Al	Fe
	0,05	1,95	0,45	0,04	0,03	18,20	8,22	0,41	0,01	70,64

**Table 2.** Coupon characterization “as acquired”.

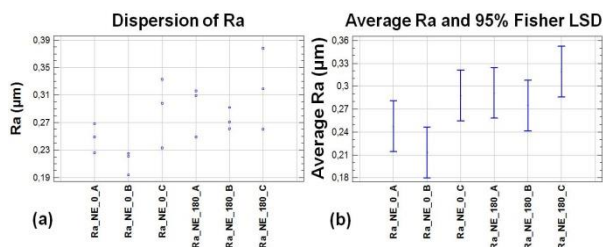
HV <sub>0,05</sub>	190 - 263	220 - 275	195 - 290	
HV <sub>0,10</sub>	230 - 280	230 - 280	245 - 310	
HV <sub>0,20</sub>	235 - 270	230 - 290	240 - 290	
Rsm (µm)	35 - 42	30 - 43	35 - 44	0°
Ra (µm)	0,23 - 0,27	0,19 - 0,23	0,23 - 0,34	
Rq (µm)	0,29 - 0,35	0,25 - 0,30	0,31 - 0,45	
Rz (µm)	1,6 - 2,5	1,3 - 2,1	1,9 - 2,5	
	A	B	C	
Rz (µm)	2,1 - 2,6	1,9 - 2,3	1,8 - 3,0	
Rq (µm)	0,33 - 0,40	0,33 - 0,37	0,35 - 0,49	
Ra (µm)	0,25 - 0,32	0,26 - 0,29	0,26 - 0,37	
Rsm (µm)	40 - 46	39 - 54	36 - 70	180°
HV <sub>0,20</sub>	240 - 295	220 - 300	215 - 255	
HV <sub>0,10</sub>	220 - 305	255 - 290	220 - 270	
HV <sub>0,05</sub>	240 - 295	215 - 270	240 - 270	

The software Statgraphics® Centurion XVI was used for the statistical comparison between sets of data at 95% confidence level.

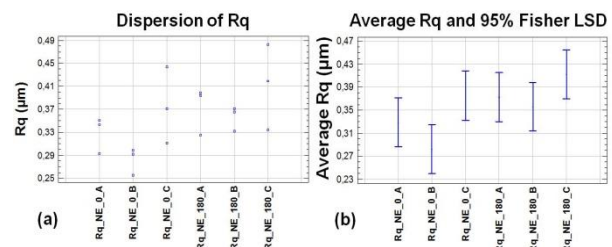
### 3 RESULTS AND DISCUSSION

#### 3.1 Coupon as Acquired

A profilometric analysis between the zones A, B and C in their upstream and downstream generatrices of the coupon in the condition "as acquired" revealed that the parameters of roughness and waviness (Rz, Rsm, and Wsm) did not present statistically significant difference at 95% confidence level. By the other hand, Figures 3 and 4 show the statistically significant differences for the roughness Ra and Rq, as well as the dispersion of the measured values. These results show that there is a difference in some parameters of the surface roughness and waviness revealing heterogeneity due to the manufacturing process.



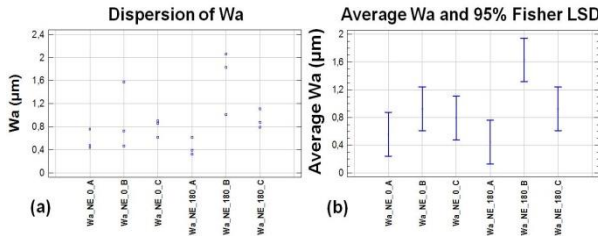
**Figure 3.** Coupon as acquired, results of Ra in the A, B, C generatrix zones: (a) Ra dispersion (b) Ra average.



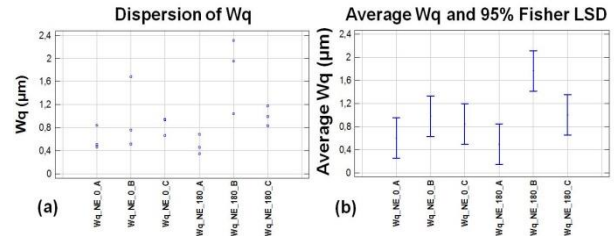
**Figure 4.** Coupon as acquired, results of Rq in the A, B, C generatrix zones: (a) Rq dispersion (b) Rq average.

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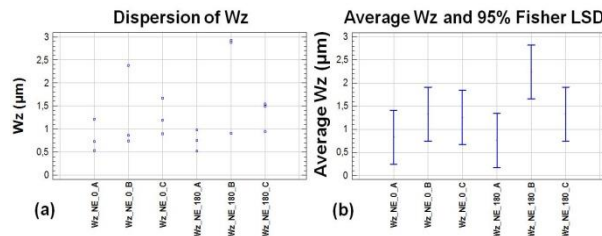
In Figures 5 to 7, it is shown that the parameters for waviness,  $W_a$ ,  $W_q$  and  $W_z$ , showed statistically significant differences at 95% of confidence level, when compared to the areas of the same generatrix and also between areas of different generatrices. The dispersion of values in some regions showed higher values than the others.



**Figure 5.** Coupon as acquired, results of  $W_a$  in the A, B, C generatrix zones: **(a)**  $W_a$  dispersion **(b)**  $W_a$  average.



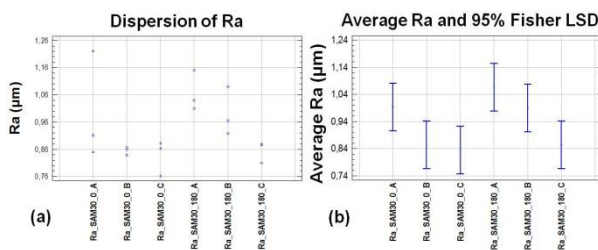
**Figure 6.** Coupon as acquired, results of  $W_q$  in the A, B, C generatrix zones: **(a)**  $W_q$  dispersion **(b)**  $W_q$  average.



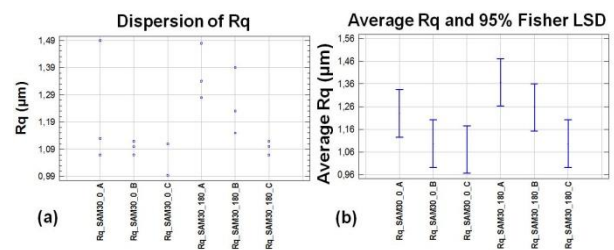
**Figure 7.** Coupon as acquired, results of  $W_z$  in the A, B, C generatrix zones: **(a)**  $W_z$  dispersion **(b)**  $W_z$  average.

### 3.2 Test without Fluid Mechanical Stirring at 30°C (SAM30)

The results of surface roughness for condition without mechanical stirring and bubbling of gas at 30°C, (Figures 8 to 11) reveal that the parameters  $R_a$ ,  $R_q$  and  $R_{sm}$  present statistical differences, both between the areas as well as between the generatrices. The parameter  $R_z$  presented statistical difference only between generatrices. From the results, it was observed that the dispersion values were higher.

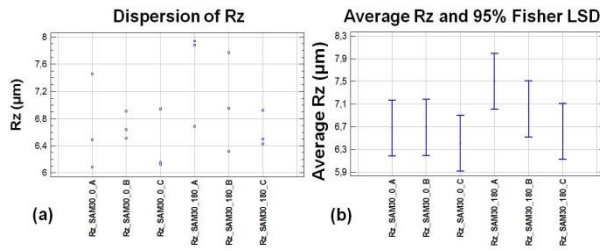


**Figure 8.** Coupon tested at 30°C without stirring, results of  $R_a$  in the A, B, C generatrix zones: **(a)**  $R_a$  dispersion **(b)**  $R_a$  average.

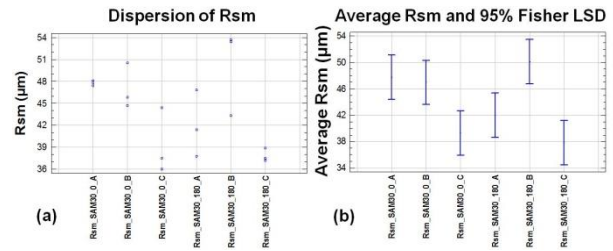


**Figure 9.** Coupon tested at 30°C without stirring, results of  $R_q$  in the A, B, C generatrix zones: **(a)**  $R_q$  dispersion **(b)**  $R_q$  average.

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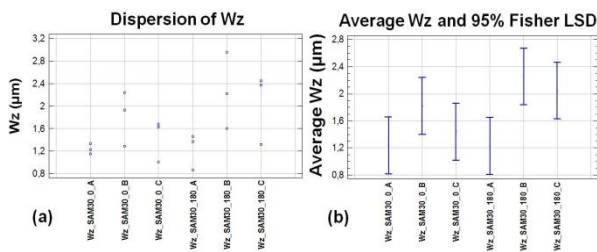


**Figure 10.** Coupon tested at 30°C without stirring, results of Rz in the A, B, C generatrix zones: **(a)** Rz dispersion **(b)** Rz average.

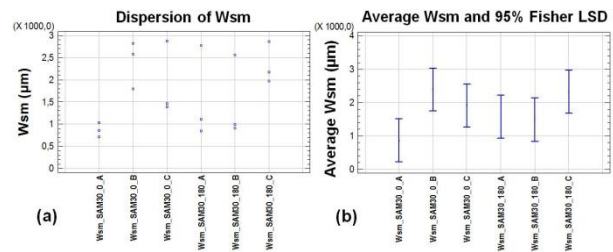


**Figure 11.** Coupon tested at 30°C without stirring, results of Rsm in the A, B, C generatrix zones: **(a)** Rsm dispersion **(b)** Rsm average.

The parameters Wa and Wq did not show statistical significant difference in condition SAM30. On the other hand, the parameters Wz and Wsm showed differences between areas and also between generatrices (Figures 12 and 13) of the tested sample. It was observed that there was a higher dispersion in the values obtained for the waviness parameters Wz and Wsm.



**Figure 12.** Coupon tested at 30°C without stirring, results of Wz in the A, B, C generatrix zones: **(a)** Wz dispersion **(b)** Wz average.

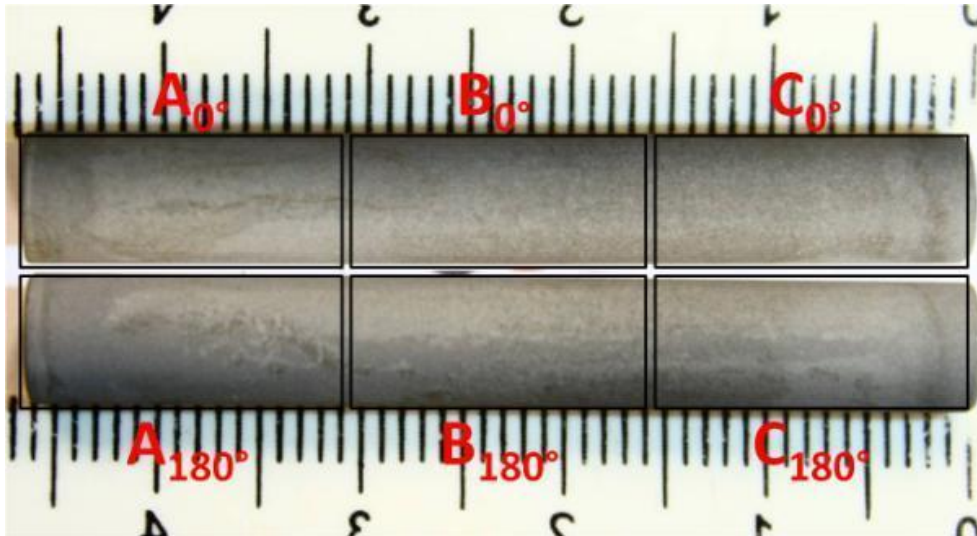


**Figure 13.** Coupon tested at 30°C without stirring, results of Wsm in the A, B, C generatrix zones: **(a)** Wsm dispersion **(b)** Wsm average.

The results presented (Figures 8 to 13) have demonstrated that, in spite of the absence of a fluid in motion, the surface showed a certain degree of heterogeneity, a fact confirmed by means of statistical differences.

In Figure 14, it can be seen that the generatrices upstream (0°) and downstream (180°) of one coupon after testing during 24 hours without mechanical stirring and bubbling of gas at 30°C. The mechanism of corrosive wear, under this condition, may have been influenced by factors such as the physico-chemical reactions of oxidation of iron, concentration of the chemicals and temperature.

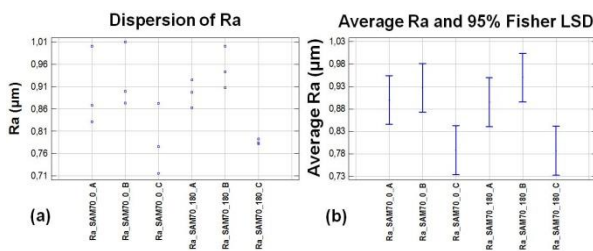
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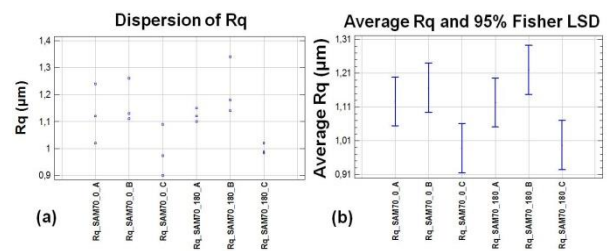
**Figure 14.** Zones A, B and C of a coupon after testing at 30°C without stirring in the saline bath.

### 3.3 Test without Fluid Mechanical Stirring at 70°C (SAM70)

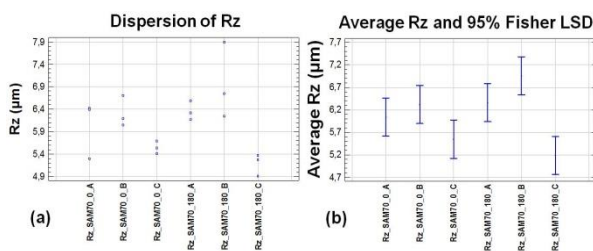
Figures 15, 16, 17 and 18 include the results of surface roughness. They were computed at 95% confidence limit for the test without mechanical stirring and bubbling of gas at 70°C, as well as the respective dispersion of the measured values. Statistical differences between the zones A, B and C as well as between the generatrices, 0° and 180°, for the parameters Ra, Rq, Rz and Rsm are also observed. These results probably were influenced by the convective flow lines present in the heated multiphasic fluid into the cell. Higher variations in Ra, Rq, Rz and Rsm in most of the zones upstream and downstream of the coupon can also be observed.



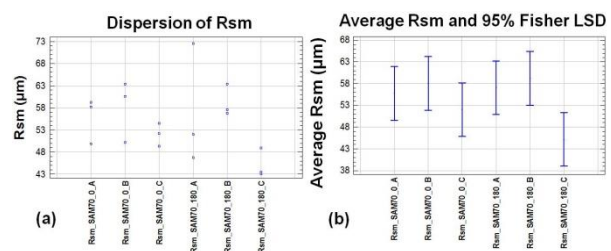
**Figure 15.** Coupon tested at 70°C without stirring, results of Ra in the A, B, C generatrix zones: **(a)** Ra dispersion **(b)** Ra average.



**Figure 16.** Coupon tested at 70°C without stirring, results of Rq in the A, B, C generatrix zones: **(a)** Rq dispersion **(b)** Rq average.



**Figure 17.** Coupon tested at 70°C without stirring, results of Rz in the A, B, C generatrix zones: **(a)** Rz dispersion **(b)** Rz average.

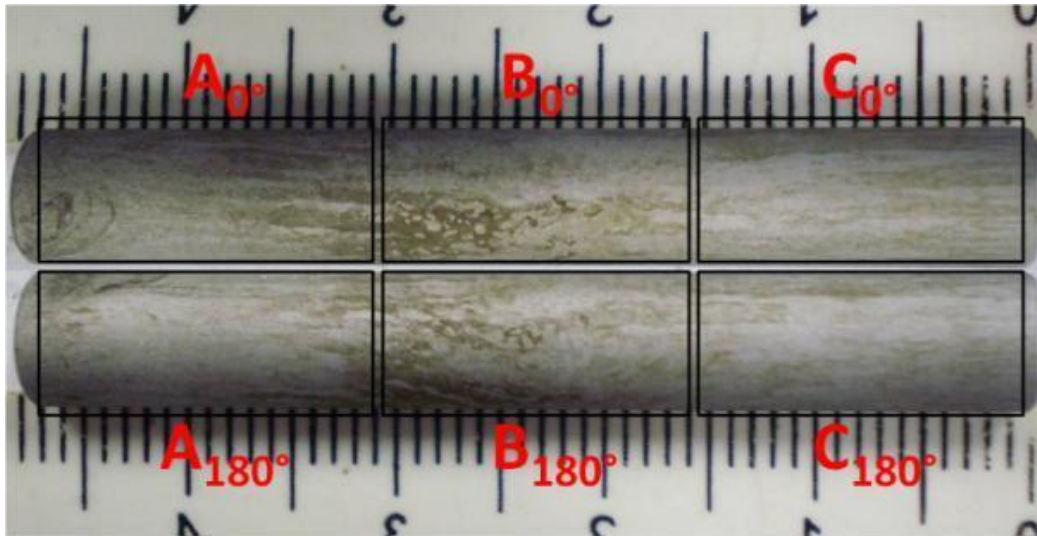


**Figure 18.** Coupon tested at 70°C without stirring, results of Rsm in the A, B, C generatrix zones: **(a)** Rsm dispersion **(b)** Rsm average.

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The parameters of waviness  $W_a$ ,  $W_q$ ,  $W_z$  and  $W_{sm}$  did not show statistically significant difference according to the comparison performed by Statgraphics® Centurion XVI at 95% confidence limit, there is a certain degree of homogeneity in superficial waviness of the coupon after the test in the condition SAM70.

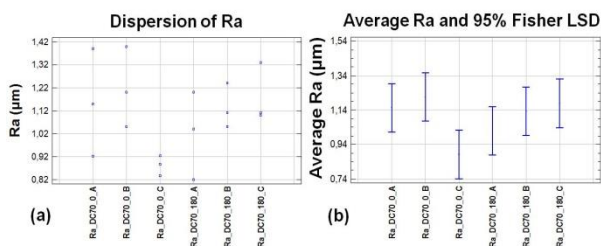
The Figure 19 shows the generatrices upstream ( $0^\circ$ ) and downstream ( $180^\circ$ ) of one of the coupons after 24 hours test, without mechanical stirring and bubbling of gas at  $70^\circ\text{C}$ . The mechanism of corrosive wear, in this condition, may have been influenced by factors such as the physico-chemical reactions of oxidation of iron, concentration of chemicals and, mainly, the convection currents caused by heating.



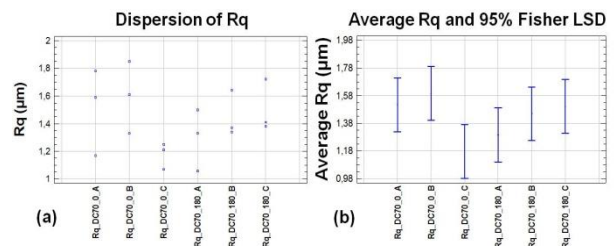
**Figure 19.** Zones A, B and C of a coupon after testing at  $70^\circ\text{C}$  without stirring in the saline bath.

### 3.4 Test with Fluid Mechanical Stirring at $70^\circ\text{C}$ (DC70)

The parameters of surface roughness  $R_a$ ,  $R_q$  and  $R_{sm}$  showed statistically differences in the comparison between zones, but in comparison between generatrices, only  $R_a$  and  $R_{sm}$  had differences (Figures 20, 21 and 22) for the condition of corrosive wear-cavitative at  $70^\circ\text{C}$ , with respect to variations of the values was noted that these were higher in almost all areas. The parameter  $R_z$  showed no statistically significant difference for the same condition. These results confirm the hypothesis that the mechanisms that operate in generating the upstream generatrix differ from that of downstream of coupon.



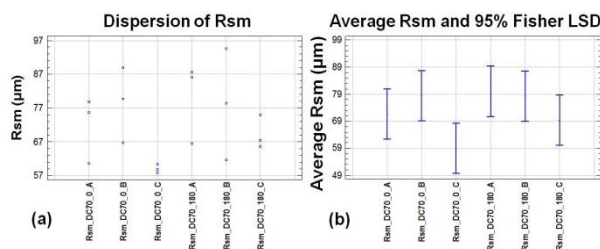
**Figure 20.** Coupon tested at  $70^\circ\text{C}$  with stirring, results of  $R_a$  in the A, B, C generatrix zones: **(a)**  $R_a$  dispersion **(b)**  $R_a$  average.



**Figure 21.** Coupon tested at  $70^\circ\text{C}$  with stirring, results of  $R_q$  in the A, B, C generatrix zones: **(a)**  $R_q$  dispersion **(b)**  $R_q$  average.

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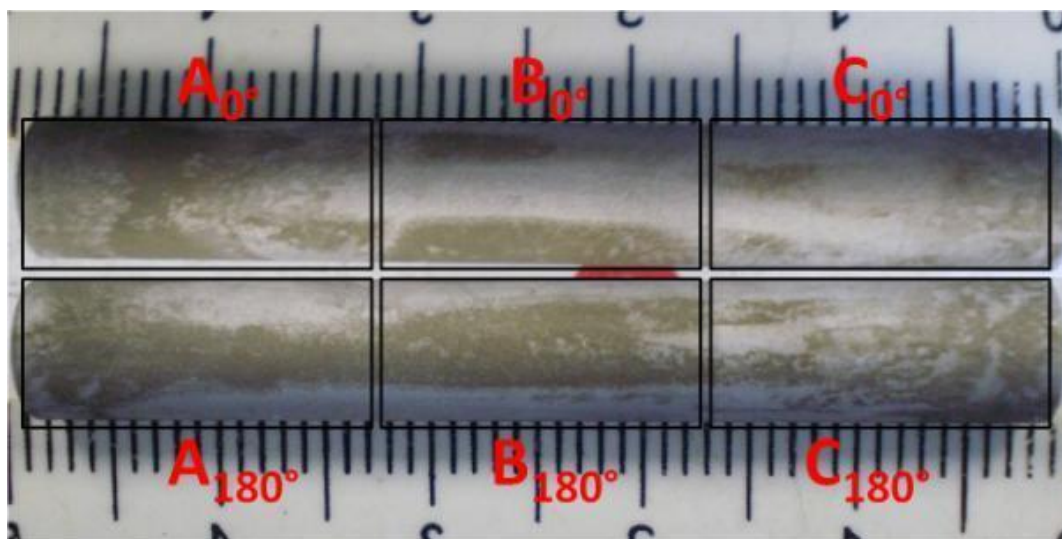




**Figure 22.** Coupon tested at 70°C with stirring, results of Rsm in the A, B, C generatrix zones: **(a)** Rsm dispersion **(b)** Rsm average.

The parameters of waviness  $W_a$ ,  $W_q$ ,  $W_z$  and  $W_{sm}$ , did not show statistically significant difference according to the comparison performed by Statgraphics® Centurion XVI at 95% of confidence limit, there is a certain degree of homogeneity in surface waviness of coupon after the test under the condition DC70.

The Figure 23 shows the photo of a generatrices upstream (0°) and downstream (180°) of one coupon after testing during 24 hours under corrosive-cavitative wear condition at 70°C. Under this condition, thermal and reaction chemical rates, besides the concentration of chemicals and multiphasic fluid hydrodynamics participated more or less intensely on the synergic mechanism of corrosive-cavitative wear. The upstream generatrix has the influence of a laminar Couette flow, while the downstream generatrix has a strong influence on the formation of Taylor vortices associated to the stirring flow condition and also the presence of CO<sub>2</sub> bubbles.



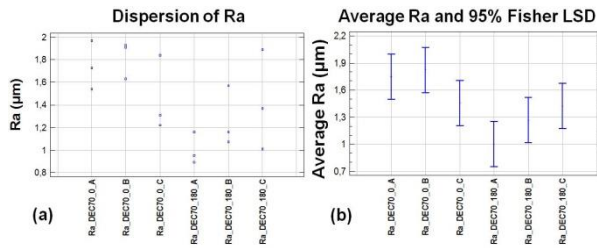
**Figure 23.** Zones A, B and C of a coupon after testing at 70°C with stirring in the saline bath.

### 3.5 Test with Fluid Mechanical Stirring and SiO<sub>2</sub> Particles at 70°C (DEC70)

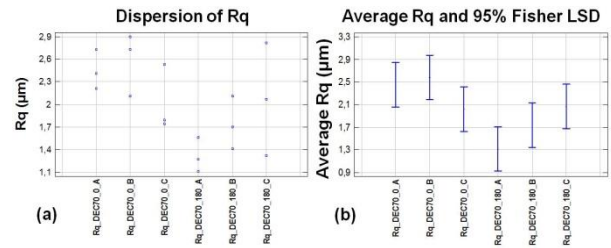
Figures 24, 25, 26 and 27 contain graphs of the surface roughness parameters,  $R_a$ ,  $R_q$ ,  $R_z$  and  $R_{sm}$ , respectively, on the condition of erosive-corrosive wear at 70°C. They show statistically significant differences between the generatrices, upstream and downstream of the coupon, as well as higher dispersion in some zones.

As cited by Ruzic *et al.* [9], the surface roughness increased the location of the crack in passivated films, improves the level of the local turbulence promoting the separation of the passivated films and the lifting off the metal surface, i.e., this mechanism may occur in regions with formation of Taylor vortices (downstream).

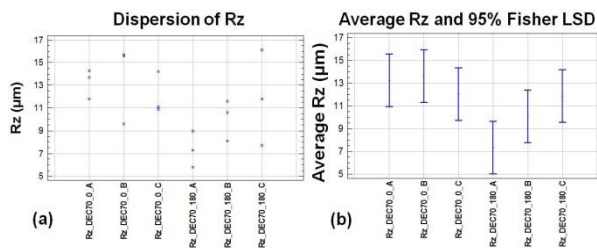
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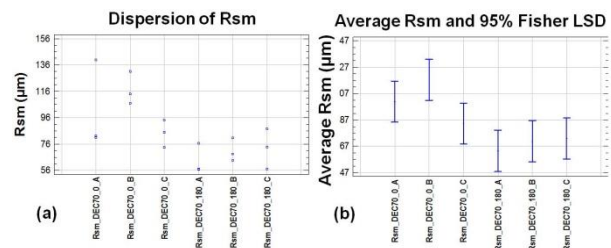
**Figure 24.** Coupon tested at 70°C with stirring and SiO<sub>2</sub> particles in the saline bath, results of Ra in the A, B, C generatrix zones: **(a)** Ra dispersion **(b)** Ra average.



**Figure 25.** Coupon tested at 70°C with stirring and SiO<sub>2</sub> particles in the saline bath, results of Rq in the A, B, C generatrix zones: **(a)** Rq dispersion **(b)** Rq average.

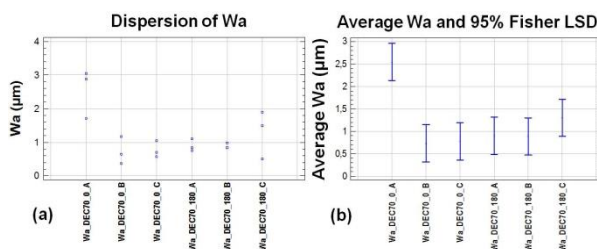


**Figure 26.** Coupon tested at 70°C with stirring and SiO<sub>2</sub> particles in the saline bath, results of Rz in the A, B, C generatrix zones: **(a)** Rz dispersion **(b)** Rz average.

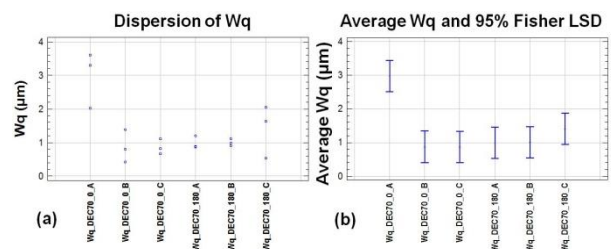


**Figure 27.** Coupon tested at 70°C with stirring and SiO<sub>2</sub> particles in the saline bath, results of Rsm in the A, B, C generatrix zones: **(a)** Rsm dispersion **(b)** Rsm average.

In Figures 28 to 31 are presented the measurements of the surface waviness parameters Wa, Wq, Wz and Wsm for the wear condition DEC70, as well as the respective dispersion of these values. Statistically significant differences were observed between the zones intragroups (same generatrix) and intergroups (zones of different generatrices). Analogously, the statistical analysis for roughness parameters also confirm that the active damage mechanisms in the diametrically opposite generatrices differ due to (1) upstream jet impingement and (2) the formation of bubbles associated with downstream vortices. The dispersions of the values obtained were lower in some areas for this test condition.

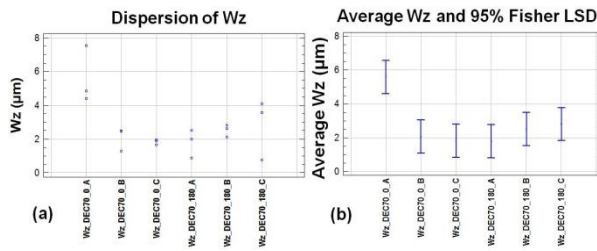


**Figure 28.** Coupon tested at 70°C with stirring and SiO<sub>2</sub> particles in the saline bath, results of Wa in the A, B, C generatrix zones: **(a)** Wa dispersion **(b)** Wa average.

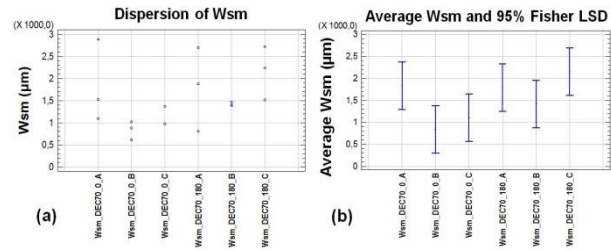


**Figure 29.** Coupon tested at 70°C with stirring and SiO<sub>2</sub> particles in the saline bath, results of Wq in the A, B, C generatrix zones: **(a)** Wq dispersion **(b)** Wq average.

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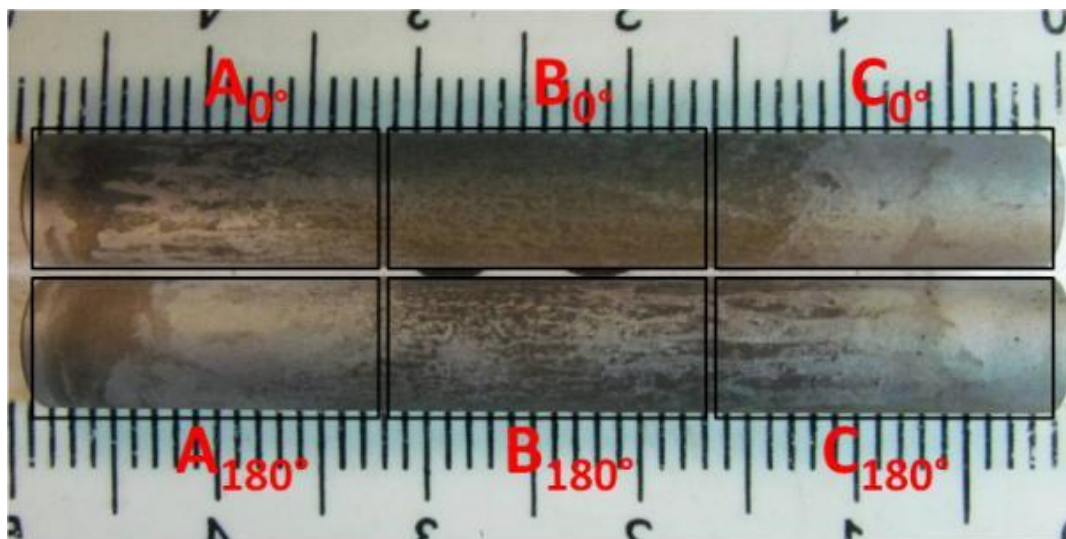


**Figure 30.** Coupon tested at 70°C with stirring and SiO<sub>2</sub> particles in the saline bath, results of Wz in the A, B, C generatrix zones: **(a)** Wz dispersion **(b)** Wz average.



**Figure 31.** Coupon tested at 70°C with stirring and SiO<sub>2</sub> particles in the saline bath, results of Wsm in the A, B, C generatrix zones: **(a)** Wsm dispersion **(b)** Wsm average.

Figure 32 shows the generatrices of upstream (0°) and downstream (180°) in one of the coupons after 24 hours test under the condition of erosive-corrosive wear at 70°C. Under this condition, the damage factors (temperature, concentration and reaction rate) formerly mentioned are competing with the action promoted by solid particles (SiO<sub>2</sub>) dispersed in the flow. Now, the upstream generatrix has the influence of the laminar Couette flow with a smaller Reynolds number. It is associated with the presence of silica constituting a slurry erosion. In the downstream geratrix, Taylor vortices also were influenced by particulate matter and CO<sub>2</sub> bubbles dispersed in the flow, although the formation of these bubbles is more difficult due to smaller Reynolds numbers associated to the SiO<sub>2</sub> particulates.



**Figure 32.** Zones A, B and C of a coupon after testing at 70°C with stirring and SiO<sub>2</sub> particles in the saline bath.

#### 4 CONCLUSION

The results of the statistical analysis using the statistical software Statgraphics® Centurion XVI at 95% confidence interval of the periodic and non-periodic parameters of the roughness and surface waviness of the middle and extremes zones of a same generatrix and the generatrices diametrically opposites of a cylindrical coupon, demonstrated some significant differences, consistent with the wear mechanism and morphology of the damages in these regions.

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The hydrodynamics of the fluidic suspension around the coupon and the particulates present there in limited areas in each coupon suggest different mechanisms of wear, from the analysis of the results from RPL, mass variation, surface texture and micro-hardness [8].

In both corrosive-cavitative wear tests, as in erosive-corrosive, the fluid flow within the cell showed traces of laminar Couette flow and presence of Taylor vortices, characterized morphological and physically through visual inspection and by the response of the texture changes of the tested coupons, suggesting since a transition until a competition between erosive-cavitative wear and corrosion mechanisms. The surface roughness and waviness showed be a statistically significant tool for evaluate differences in these different situations of the tests.

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