

EFFECT OF STRESS CORROSION ON MECHANICAL PROPERTIES OF STAINLESS STEELS

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ABSTRACT

Presently, it is known that most of the time the approach to endless research results is unacceptable for use in real stainless steel materials. In man made structures, imperfections appear during the production stage at nano, & micro scales, which change throughout its useful life, creating failures, some of which, can be disastrous. Fractography studies show the non nature of fracture designs. The fracturing of stainless steel materials is an interesting problem from a scientific and a technological point of view, which has created a lot of research concerning the formation and propagation of cracks. It is well established that fracture forms may be treated from the point of view of fractal geometry. Unfortunately, there were also many accidents leading to human and material losses due to failure of such structures. Some of them were due to imperfect designs, but it has also been discovered that the weaknesses in stainless steel materials like determined cracks might have started cracking, as a result leading to fracture. The surface of stainless steel materials depends a lot on properties such as, adhesion, resistance, and absorbency, etc. It is true that surfaces exposed to the environment and interactions between both of them are responsible for the behavior of mechanical properties of stainless steel materials. Data that might be gained from the surface may be related to microstructure and mechanical properties of stainless steel materials. It must also be remembered that when a metal piece is under stress, corrosion concentrates causing transgranular or intergranular cracks.

Keywords: micro-structure, stainless steel, stress corrosion, mechanical properties.

Introduction

In the case of surface crack, its investigation is usually carried out through metallography, which may tell us about, among others things, the derivation of the fracture, the direction of its propagation and the type of load that initiated it. The surfaces of cracks are usually a set of repeated forms, for example, cracks, fractures, edges, holes and intergranular defects, among others. Quantitative fractography aims at translating such features to a parametrical shape, and that is where fractal geometry comes in.

Fractal geometry looks for regularity in the connection of an object and its parts at several scales that is, it studies invariable geometrical characteristics, regardless of scale changes (Burdekin, 1971). In other words, fractal geometry studies non differentiable or non continuous geometrical shapes at any scale. From a mathematical point of view, a fractal is a subset of a metric space for which its dimension is strictly higher than its topological dimension. In general, fractals have some type of similarity and it may be deliberated that they are made of small parts similar to the whole. This similarity may be strictly geometrical or only estimated or statistical. Quantitatively explored for the first time, the fractal character of crack surfaces subject to several thermal treatments and reported a connection between fractal dimension and tenacity of the crack, their property estimated through impact energy. These outcomes, though questioned in the beginning, started a new age in the fractography disciplines. From then, analysis of self attraction of crack surface is a very active study field, which has had the influence of modern and sophisticated statistical and mathematical methods.

There are several methods for analyzing self affinity of crack surfaces, most of them use profiles removed from surfaces through the use of experimental techniques (Folias, 1965). Highly refined experiments, focused on many materials, do not approve connection between the fractal dimension and mechanical properties. It has been proven that rugosity exponent ζ is the proper parameter by which to describe crack surfaces in fast dynamic conditions analyzed mainly through scanning electron microscope SEM. Some studies proposed a worldwide rugosity exponent $\zeta = 0.78$, regardless of microstructure and properties. Such general was questioned by the discovery of another self affine system categorized by a rugosity exponent $\zeta = 0.5$ for crack surfaces generated in slow crack propagation situations or examined at nano-metric scales, using atomic force microscope AFM. Existence of both systems in numerous materials has also been reported. Such systems irritated in a crack length, which seems to be independent of dynamic conditions. Efforts to relate such crack length with micro-structural parameters of some materials continue to be carried out (Mandelbrot, 1982).

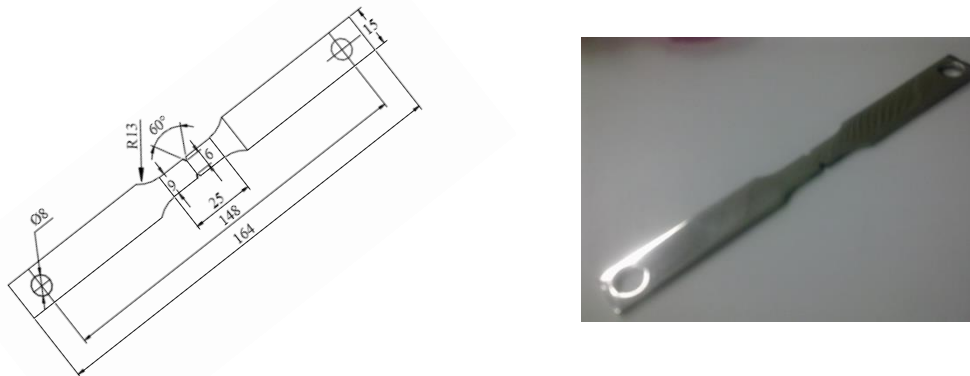
The development of this research on behavior of unprotected 304 stainless steel exposed to corrosion which the steel had no coating technique applied using zinc as the main protective element, caused from observing occurrence of cracks on structures exposed to a marine environment as well as the analysis of significances understood by such fact, regarding marine corrosion.

Study is shown from dimensioning of test specimens lab tests with their chemical analysis and, finally, a fractography of the consistent test specimen, obtaining its fractal dimension based on the theoretical development shown.

Test Specimens' Dimensioning

In accordance with ASTM A-370 standard specifications and complying with such parameters geometrical connections between length, width and height, Figure 1 shows the type of test specimen used. As well it shows proportions for dimensioning of the test specimen used in the lab, gained in accordance with ASTM standards. Test specimens were made of 304 stainless steel ($C = 0.29$ max., $Mn = 1.20$, $Si = 0.40$ max., $P = 0.40$ max., $S = 0.05$ max). In accordance with ASTM specifications, test specimen were made with 304 stainless steel, chemical composition: $C = 0.29$ max., $Mn = 1.20$, $Si = 0.40$ max., $P = 0.40$ max., $S = 0.05$ max. Existing guidelines were considered for tensile tests on steel products.

Figure 1: The basic geometry and dimensions of the tension specimens.



Laboratory Tests And Chemical Analysis

Tensile tests were carried out in a 250 KN Universal Mechanical Testing machine. Conditions for the test were testing speed 0.60 mm/min. sampling frequency 5.0 points/s; downwards.

Traction analyzes for unprotected 304 stainless steel were carried out on three types of sample, taking into reason the base material, and other two samples were applied under real marine corrosion, throughout 6six months and one year aging stages. Based on features and components of the metal used, the sample was roughed down and polished in order to remove scratches. The procedure used was the traditional one, consisting in roughing down the surface of the sample and sanding it with an ultimate approximation to a flat surface free from scratches with a nourished spinning wheel. The sample was nourished in a dish with a solution of Nital dissolved in water for 10 seconds. Nourishing time was wisely controlled. Action of nourishing stopped when the sample was put under a water stream, cleaned with alcohol and a drier was used for finishing.

Mechanical Properties Of 304 Stainless Steel

A fractographic analysis begins with a final observation of the crack's surface structures.

Sign of nucleation of fracture, mechanism and direction of propagation causes may be obtained, and an estimation of acting loads' magnitude may eventually be obtained. And Figure 2 shows the results of the stress & strain graph for newly made material not exposed to marine conditions for which a maximum 369.18522 MPa load and a 255.1545 MPa yield strength were obtained. Figure 3 shows its fractography. For stainless steel aged more than six months at sea, a 351.50238 MPa maximum load and a 238.676 MPa yield strength were found, Figure 2 Fractography in Figure 3 shows nucleated cracks and a behavior yielding to crack. For stainless steel aged more than one year, a 315.883 MPa maximum load and a 226.24 MPa yield strength were gained, Figure 2 Fractography shown in Figure 3 shows crack branching due to tensile testing since material loses ductility due to corrosion. Figure 2 shows the results of the mechanical properties of steel in accordance with the tensile test. It should be observed that results attained from tensile test for newly made 304 stainless steel test specimen are very similar to those shown by those studies.

Stress under which the material changes its behavior from elastic to plastic is not easily detected, so for this reason the yield strength has been determined drawing a line parallel to the initial section of the stress strain curve, but displaced 0,002 mm/mm 0,2 % from the origin, crossing the curve of the diagram. The intersection point is the yield point.

To obtain fractal dimension of micro-cracks found in the material, the theoretical description of a fractal's behavior in unprotected 304 stainless steel exposed to corrosion is shown (Cherepanov, 1995).

Figure 2: Yield strength for test specimen.

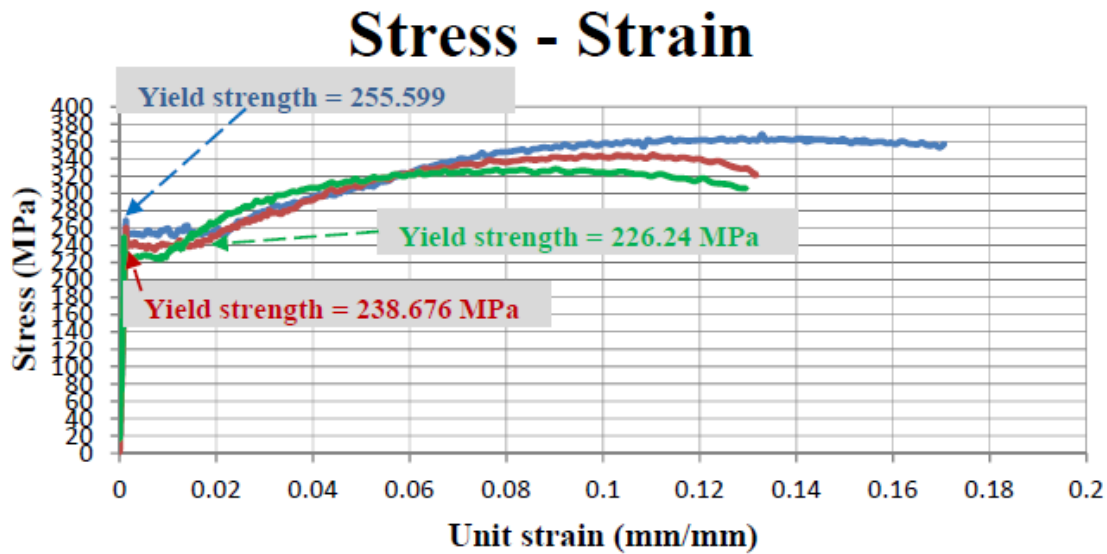
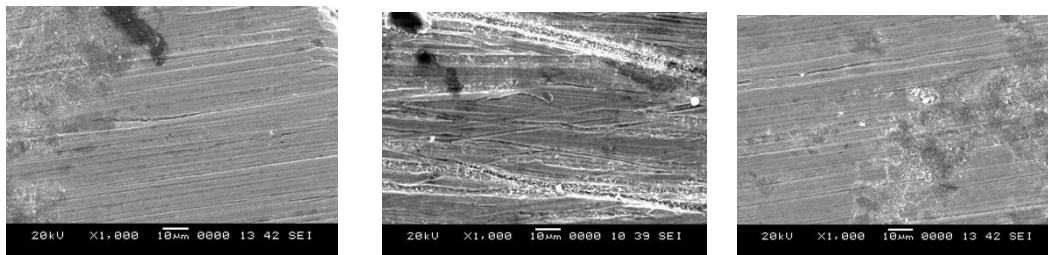


Figure 3: fractography of test specimen.

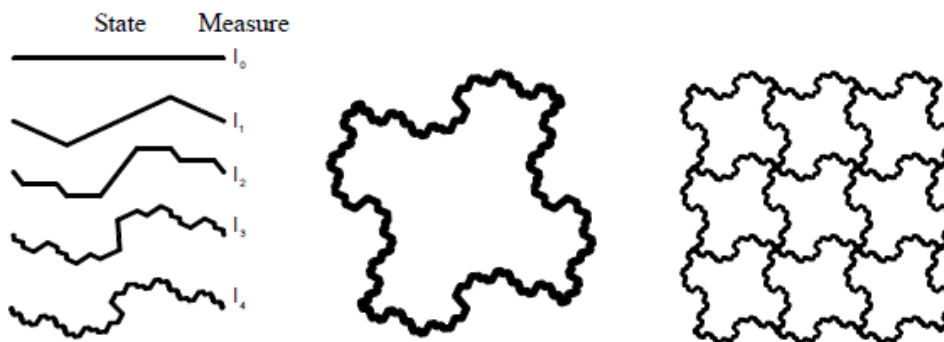


A Fractal Theoretic Explanation

Explanation of creating curve. Let I_0 be a unitary length line section, contained in a closed intermission, that is, $I_0 \subset [a, b]$. Let I_1 be a set with sectioned behavior, containing in three sections of a straight line which create based on initial point a of I_0 , two scalene triangles reflected regarding the middle point c of I_0 , attained as follows, the first half of section I_1 is replaced or removed by the sides of triangle which create an angle with I_0 . This process is repeated for the second half, but with the sides reflected from middle point c .

This process is known as generator which is called state one. Structure of set I_2 is made applying the generator to every section of I_1 , which is called state two. Therefore, set I_k is produced applying generator I_1 on every section of I_{k-1} , which is called state k , Figure 4.

Figure 4: Construction of the fractal curve I . In every state I_k generator I_1 is applied on every section of the curve. Fractal curve I on the plane. Fill the plane with the curve fractal.



Some particular characteristics obtainable in the structure of this type of fractal should be noticed when generator is created by combination or triangular layouts in size and form.

- States I_{k-1} and I_k are different from each other in the sequence shown by curves $\forall k \rightarrow \infty$,
- set I has a fine structure that is it contains every detail in every chance small scale.
- although the generator is composed by two triangular figures corresponding to logic, the geometry of $I_k \forall k \rightarrow \infty$ is too irregular to be described in classical geometric terms.

Figure 4 shows the curve constructed on the plane through reflection of I_k on every side.

Curve I is characterized by being of a similar scale since, based on an alteration $F: \mathfrak{R}^n \rightarrow \mathfrak{R}^n$ with $\lambda_i > 0 \forall i \exists n, a, b \in \mathfrak{R}^n$ such that $|F_i(a)-F_i(b)| = \lambda_i |a-b|$. Similarity of scale is present for triangles created with I_0 , by generator I_1 . In a like method, it also has the property of being affine, since based on transformation F already defined, $F(a) = T(a) + \alpha$ with T a non singular linear transformation and $\alpha \in \mathfrak{R}^n$. It must be remembered that attraction is considered as a cut off alteration or resistant to cutting, and is a diminishing or expanding effect, not necessarily in the same direction. A curve type I_k contains the scale principle if all its relative figures are linked to each other by a scale law.

Let I be a Borel's set such that $I = \{I_1, I_2, I_3\}$, where I_j is a limited succession of line sections creating the generator $\forall j = 1, 2, 3$, in order that $I_i = \{U^j_{j=1} I_i \forall i = 1, 2, \dots\}$ is a countable sequence of sets. Therefore, measurement μ , of sections I_i is defined.

When a geometrical gap is of the fractal type, produced by a natural process, a uniform reticulate should be made. Let $(\chi, P(\chi), \mu)$ be a space with measures such that the sample space is $\chi = [0, 1]$ and $P(\chi)$ is a set of subsets of χ , where the measurement is μ . Since the system is dynamic, $\chi \subseteq \mathfrak{R}^p$ is the phase space. Let us consider a χ reticulate covered by p -dimensional boxes with radius δ_n , where $B_{\delta_n}(t)$ is the next box having the section of straight line or point t . Succession of next boxes has radius $\delta_n \rightarrow 0$ as $n \rightarrow \infty$.

Let us suppose that there is subset $I \neq \emptyset$ from euclidian space n -dimensional, \mathfrak{R}^n , and that $|I| = \sup\{|a-b| \text{ such that } a, b \in I\}$. If $\{I_i\}$ is a countable set of group with radius δ covering I , as a result, there exists a subset II of \mathfrak{R}^n such that $II \subset (U^{\infty}_{i=1} I_i)$ with $|I_i| \in [0, \delta] \forall i$. Thus, $\{I_i\}$ is next δ of I . If there is a $k > 0$, then for every one $\delta > 0$ a function to minimize total covering II may be defined (Chaaban, 1991).

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Meshing And Definition Of Fractal Outline

Let $N(t^*, \Delta t^*)$ be the number of squares contained by reticulate, and $N(t, \Delta t)$ the number of squares intersected by the fractal curve. $D_{\theta^{\circ}}(I)$ shall be the fractal dimension, L the total length of the object, and l the length of every section. Then, L/l measure defines the number of sectors contained by every side of the intersected reticulate. These scale properties resemble to a disjointed fractal, and the multifractal theory is applied. Based on the above:

$$N(t^*, \Delta t^*) \cong \Delta t^* \rho(t^*) f(t^*)$$

where $t \in [t^*, t^* + \Delta t^*]$. $P(t^*)$ is the probability of distribution of connection points $t \in [t^*, t^* + \Delta t^*]$ and $f(t^*)$ the fractal dimension of such points. Since a random generation of $f(t)$, the original curve is rotated to different angles, preferably constant, in order to calculate $D_{\theta^{\circ}}$ for every case.

In order to rotate the original curve a certain number of times, let us consider mapping $M_n: \chi \rightarrow \mathfrak{R}$, where $M_n(t) = -\log \mu[B_{\delta_n}(t)]$, if $\mu[B_{\delta_n}(t)] > 0$ then $C_n(t)$ is a rescaled version of $M_n(t)$, where C_n describes the local behavior of μ measurement.

Behavior Form

The fractal behavior of a geometrical disjointedness takes us to the concept of diagonal self-affinity diagonal. In order to define the form of the fractal generator, let us begin drawing straight lines from its base to the points where such curves are all along its length, and so obtain a fractioned curve. It is important to draw horizontal lines in case there is a change in its path behavior, in order to identify the affinity along such path.

Horizontal lines identifying the start of the generator must show the feature of proportionality d such that:

$$d = L \operatorname{sen} \beta \text{ where } \operatorname{sen} \beta = L / d \text{ and } \cos \beta = k / L$$

L is the length of the generator, l the length of every section, and L/l is the number of sectors contained by the generator. In order to detect the ideal behavior of the path, the scale connection is defined as the average of the lengths of connecting generators that is, $(L_1 + L_2) / 2$; Figure 2.

The scale factor of every rotation undergone by the generator or fractal curve, s , is defined as:

$$s = \log_{10}(N) \forall N \rightarrow \infty \quad (6)$$

where the value of N is the average of the highest number of every rotation. Based on the above, we may get the scale factor of the generator, defined as the inversion of s which, applied, generates the geometrical structure (Budiman, 1997).

Calculation Of Fractal Measurement Of The Crack For Six Months Test Specimen

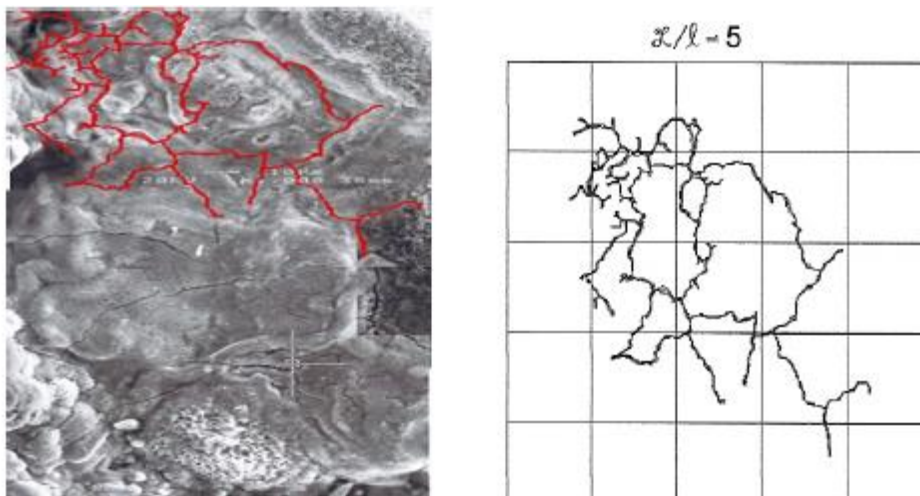
In order to calculate the fractal dimension for cracks generated on test specimen exposed to marine corrosion, both for six and twelve months, the theory existing above was applied to disjointed fractals, or fractals generated by natural processes. The original crack was rotated 45°, 90°, 135° and 360° or 0° degrees in order to determine in a more reliable way the fractal dimension, D_f . The process was to choose a picture for the six months test specimen and netting it in order to calculate its fractal dimension, which is shown in Figures 5.

In order to obtain fractal dimension D_f , an average of calculated measurements for each of the four rotations applied to the fractal curve was obtained. Fractal dimension for a crack on stainless steel exposed six months to marine environments is:

$$D_f = (D_{0^\circ} + D_{45^\circ} + D_{90^\circ} + D_{135^\circ})/4 = (1.34950 + 1.3429 + 1.3398 + 1.3432)/4 = 1.34385$$

In demand to confirm that fractal dimension calculated under the theory developed above is valid, the commercial software was used. The fractal dimension obtained with such program is $D_f = 1.34029$. As may be seen, the results are similar (Roylance, 2001).

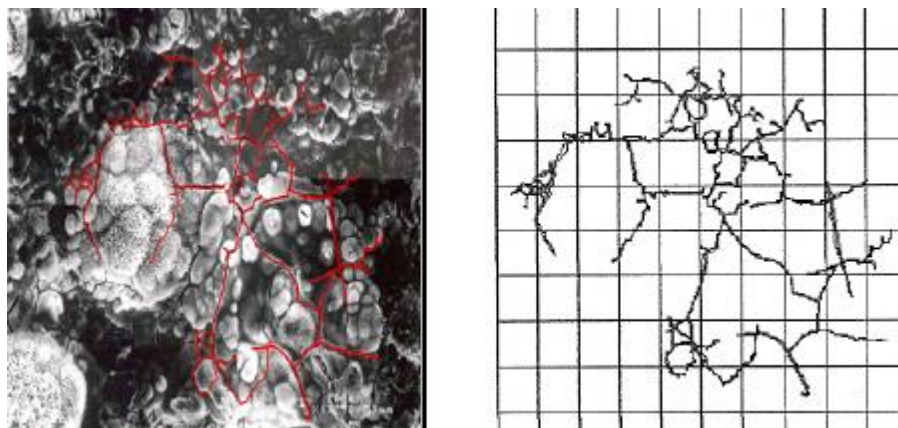
Figure 5: Fractal behavior of the crack for six months test specimen & web of fractal with five sectors and 0 degree rotation.



Calculation Of Fractal Measurement Of The Crack For One Year Test Specimen

Figure 6 shows fractal behavior of the crack, and shows its digitalization. To attain fractal dimension of the one year test specimen in marine environments, the same calculation procedure used for the six month test specimen was done (Gonzalez, 2000).

Figure 6: Fractal behavior of the crack for one year test specimen & web of fractal with ten sectors.



Fractal measurement for a crack in 304 stainless steel emersed to marine environments throughout a year is as follows:

$$D_f = (D_{0^\circ} + D_{45^\circ} + D_{90^\circ} + D_{135^\circ})/4 = (1.3946 + 1.3943 + 1.4 + 1.3943)/4 = 1.3958$$

That is, fractal dimension of the cracks in 304 stainless steel emersed to marine environments during a year has a 1.3958 value. In accordance with commercial software, its fractal dimension is 1.40040.

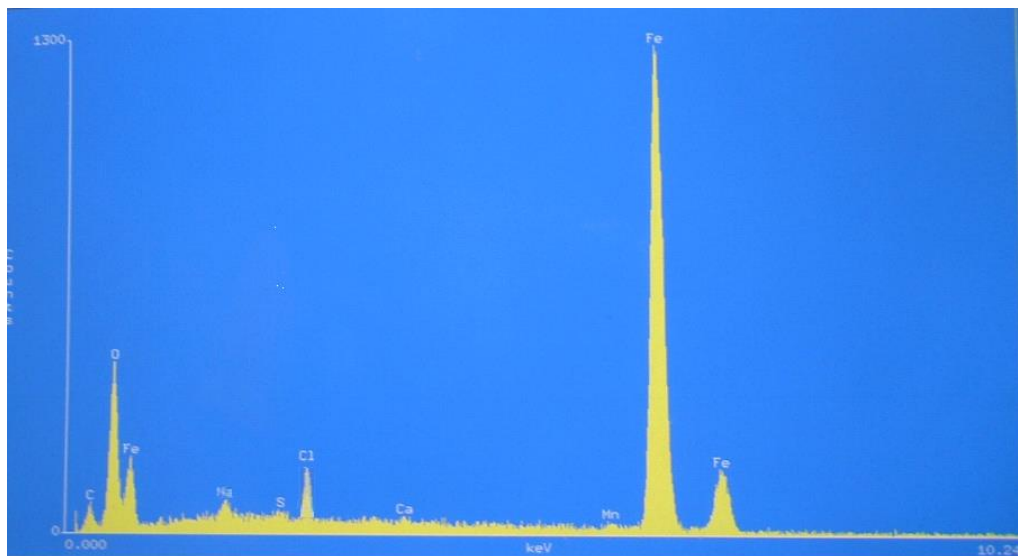
Micro Examination

The X-ray micro-analysis study on test specimen aged twelve months at sea shows presence of carbon, oxygen, iron, silica, sulphur, chloride, sodium, calcium and magnesium as the most representative elements as in Figure 7. Chloride linked to sodium; basic elements of sea water are present as sodium chloride. Magnesium is present in constant relation with chloride which, when combined with other elements, creates magnesium chloride and magnesium sulfate. Sulphur is present in sulphates. All these elements have made possible quickening of test specimen corrosion (ASTM A-370).

Results & Discussion

Newly made 304 stainless steel showed a ductile crack surface, which caused concentration of stress and a limited increase of plastic deformation (Daguier, 1996). The elements showed uniform corrosion for both cases of sea exposition, which caused a generalized thinning on the entire surface. They also showed pitting corrosion and a brittle and leaky surface.

Figure 7: Analysis spectrum through electron microscopy.



A loss of ductility of 11.96 % six months and 43.87 % twelve months were noted after sea exposition, regarding the newly made test specimen. These small values do not allow creation of a function for loss of ductility due to permanence of steel in sea coasts. This loss of ductility is mainly recognized to the presence of hydrogen, an element present in sea water and organic compounds on the site of marine exposition, initiating it to be absorbed and transported to the inside, towards failures of the element, therefore causing separation of grain boundaries.

In the case of tensile tests, breaking had to be realized in order to calculate the fractal dimension of crack generated. The consistent stress strain graphical appearance shows difference in the fluency strength in each description, due to the reduction of the area based on type of uniform corrosion initiated (Daguier, 1997).

Once the crack was found, it was digitalized and the description format to obtain its fractal type was applied. Then, it was proved that such generator was applicable to fractal behavior of the crack. This being so, the crack was interlocked, sectioned and rotated at many angles for calculation of its fractal dimension (Bouchaud, 1997). Fractal dimension calculated shows a ± 0.0036 difference regarding commercial software, which may be due to the box counting algorithm. Fractal dimension obtained for the test specimen unprotected twelve months at sea shows a fractal dimension higher than the six month test specimen, since the crack has more branches.

Conclusions

Regarding description of probes to the environment, it may be stated that due to interaction of an alloy with the environment to which it was exposed, corrosion products were generated on it, which changed failure mechanism of probes subject to tensile analyze. Probes exposed on both stages have important stress concentrations on areas where corrosion developed. This was observed in lab examines, since failure appeared in such areas.

Regarding theoretical development of fractals obtained, it has been found from line a section creating a generating curve categorized by sets of Borel's theory, which is possible since generation of fractal curve is carried out through natural processes generating geometrical cutoffs of any typical fractal. Classification of fractal is carried out on a reticulation and its value is based on count of segments for every box in such reticulation. The original crack was rotated 45°, 90°, 135° and 360° or 0° degrees in order to determine in a more reliable method the fractal dimension, D_f . Self affinity of the fractal is also tested.

Fractal dimension obtained after six months of exposition is lower regarding that obtained at twelve months, which shows that experience time is directly proportional to cracks generated due to corrosion products and that crack consequence effects the consistent value.

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