MEASUREMENT OF SENSITIZED STRUCTURES OBTAINED BY BRIEF TIME THERMAL TREATMENT IN AUSTENITIC STAINLESS STEEL

First Author's Name: Lopes, Jadir Antônio Moreira
Institution and address: CDTN-CNEN
e-mail: jaml@cdtn.br

Second Author's Name: Cândido, Luiz Cláudio
Institution and address: REDEMAT-UFOP
e-mail: candido@em.ufop.br

Abstract. Sensitization is detrimental to the corrosion resistance of austenitic stainless steels in a variety of environments. This transformation occurs when these materials are subjected to temperatures between 450 and 800°C. Welding process normaly are the principal cause of sensitization in structural parts. The metallurgical structure resultant of these process is similar to thermal treatment of brief time. In this paper was applied a image analitical program to quantify strutures resultant of oxalic test in sample of steel AISI 304 heat treated between 1 and 60 min at 675°C.

Keywords*: sensitization, austenitic stainless steel, electrochemical technical, quantitative metallography*

1. Introduction

Austenitic stainless steels have good corrosion resistance as well as good mechanical characteristics and are utilized in various fields, from high pressure chambers in chemical and nuclear industries to housewares. Their corrosion resistance is due the existence of a passive film very stable and adherent to the surface that grow in some ambient and their stability determine the corrosion resistance of this materials. Chromium is the chemical element responsible by passive behavior, that start when the Cr content is about 11%, and increasing with the Cr content increase [Fontana, Peacock,1970]. Under some specific conditions, these films aren't stable and this can nucleate localized corrosion. The localized corrosion can be by pitting, crevice, intergranular and stress corrosion cracking. Intergranular and intergranular stress corrosion are highly influenced by the metallurgical characteristic of the materials [Sieradzki, 1987]. The heating of austenitic stainless steels between 450° and 800°C [Berry, White, Boyd, 1987] can transform these materials in sensitive to intergranular corrosion because metallic carbides ($M_{23}C_6$, when Cr content is about 70% de M) grow in the grain boundaries [Joshi, Stein, 1972].This phenomena is knew like sensitization and characterized by the depletion of the matrix in protector element, Cr, facilitating localized attacks.

The sensitized state, can be measured by X-ray spectroscopy, metallographic observations or electrochemical techniques. Chromium depletion change the regions near the grain boundaries in an alloy of low content of Cr and corrosion susceptible. The wide of this regions are nanometrical turning the corrosive process highly deleterious, because it form cracking fine and deep of difficult detection, characterizing that is conventionally knew like intergranular corrosion.

The susceptibility tests to intergranular corrosion of the austenitic stainless steels use techniques that make visible or quantifiable the change that occur in the sensitization process. The standard [ASTM A 262 – 93] describe procedures destructive or no, applicable to the detection and measurement of susceptibility to intergranular attack of the austenitic stainless steels. Among these procedures, the Practice A, that consist in an electrochemical attack and classification of the structures revealed, characterize by to be non destructive and qualitative. This paper was realized with the objective of quantify these structures. For this, sensitized strucutures was produced in different levels and made the quantification of the resultant structures by quantitative metallography.

2. Materials and methods

The material composition, a plate with 1,5 mm thick of stainless steel AISI 304 is given in Tab. 1. To guarantee a more fast carbide precipitation, to obtain several degrees of sensitization in brief time of thermal treatment, the used material was one of high carbon content.

Table 1. Chemical composition of the stainless steel AISI 304 (%w).

C	Cr	Ni	Mn	Mo
0.06	17.0	8.7	1.2	0.024

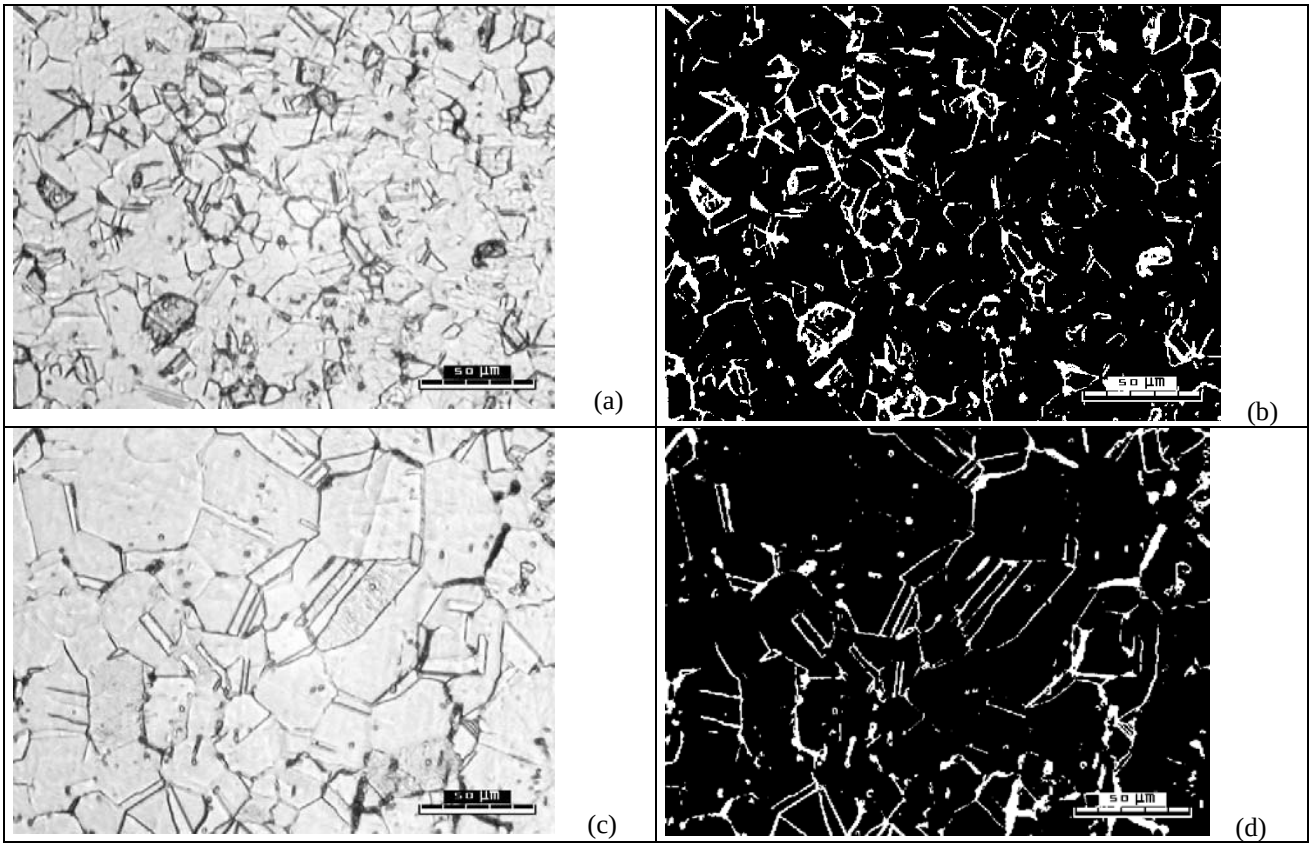
Samples of 15 mm diameter was thermal treated in lead at temperature of 675 ± 5°C, temperature in that carbide precipitation kinetic is fastest [Steigerwald, 1987]. The treatments times were 1, 2, 3, 5, 10, 15, 20, 30 e 60 minutes. The use of lead to make the thermal treatment allowed that was possible utilize short times without the periods of heating and cooling concealed the transformation that occurred in the temperature established. Records showed that the time used in heating/cooling was approximately 2 seconds, that represent a value less than 4% of the shortest treatment time.

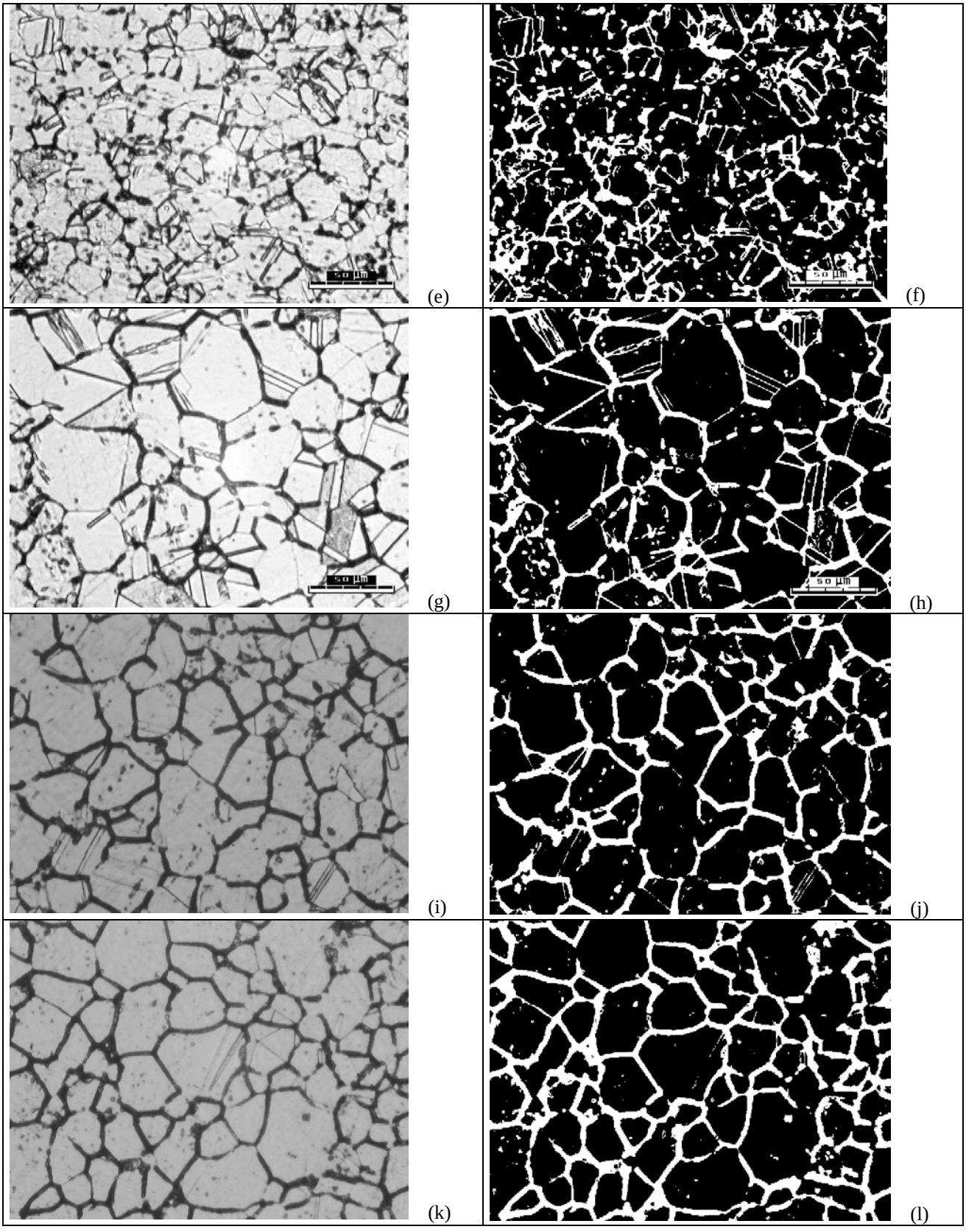
The thermal treated samples were mounted in Bakelite with a conductor terminal welded in one face. According ASTM A 262 – 93, Practice A, its were mechanical polished until 1 μm diamond paste. The samples were submitted to attack in a solution of 10% oxalic acid in a equipment LECTROPOL with a current density of 1 A/cm^2. Its were examined and photographed in a optical microscopy LEITZ, model ORTHOLUX II BK, where was adapted a digital camera CREATIVE, coupled to a computer to automatic register of the digital micrographies.

To quantify the degree of sensitization, the used parameter was percent of area that was attacked. These values was obtained with the application of a microstructural analyzer software QUANTIKOV [Pinto, 1966], in two field by sample. This software facilitate the work of quantification because it allow that, by change the image, the measured characteristic was evidenced.

3. Results and discussion

The resultant structures of the thermal treatments, revealed by the procedure of the oxalic acid electrochemical attack, realized according Practice A of the standard ASTM 262 – 93, and the modified image used to quantification of the carbides occupied areas was showed in the Fig. 1 a-t. The micrographies were in order of increased treatment time.




50 µm
(e)
50 µm
(f)
50 µm
(g)
50 µm
(h)
(i)
(j)
(k)
(l)

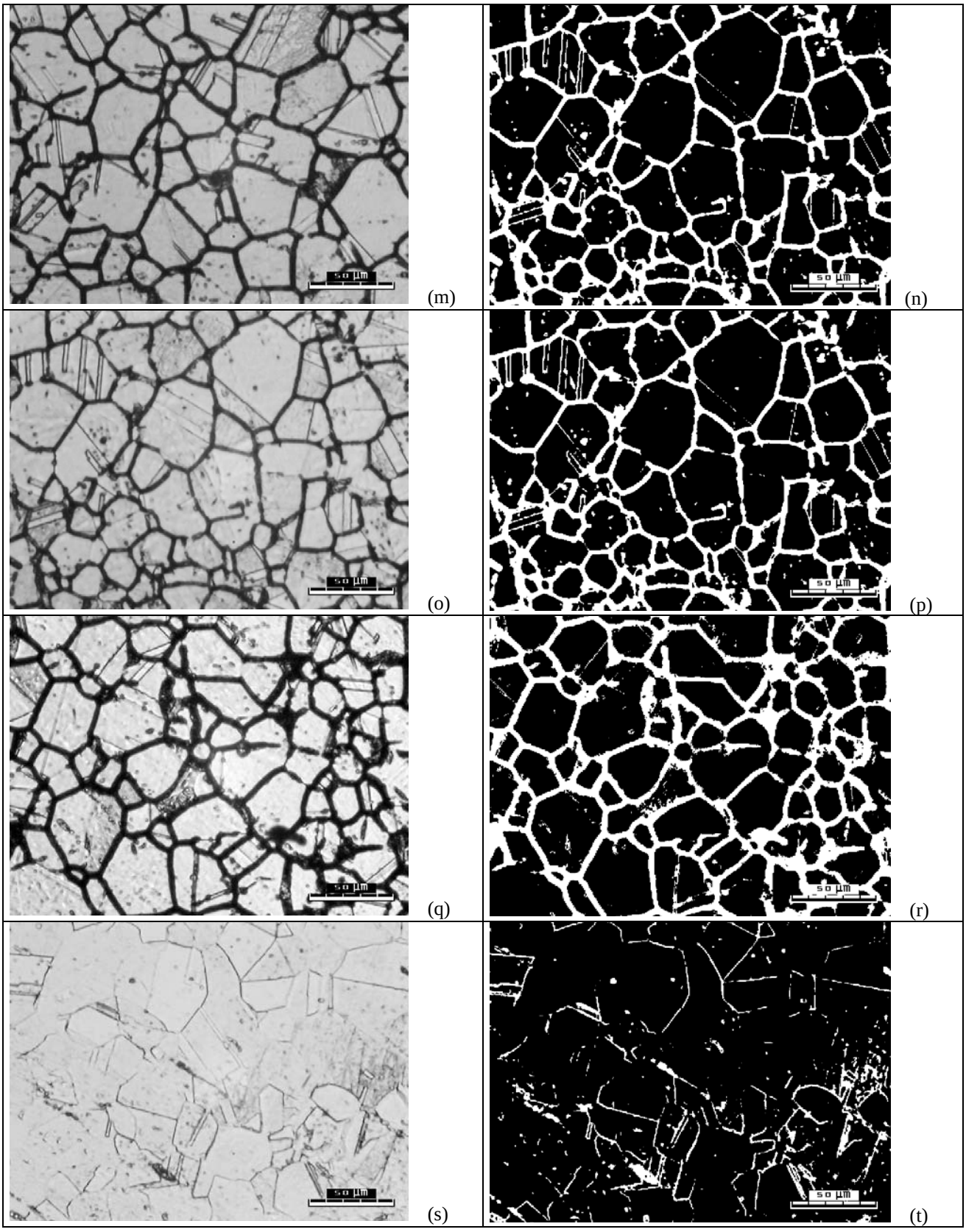


Figure 1 – Microstructures revealed by the oxalic acid attack and its worked image to quantification made by the analyzer QUANTIKOV, presented according treatment time; (a/b = 1min, c/d = 2min, e/f = 3min, g/h = 5min, i/j = 10min, k/l = 15min, m/n = 20min, o/p = 30min, q/r = 60min, e s/t = without treatment), 400X.

Discern by micrographies, that treatment up 20 min the most grains are completely enveloped by carbides, showed a high degree of sensitization. This confirmed that, the treatments are adequate to obtain represented metallurgical structures since annealed to complete sensitization. These results are in according with [Bennett, Pickering, 1988] that developed a sensitization kinetic model to apply at AISI 304 steel. By this model, times between 30 a 45 min at temperature of 650°C promote complete sensitization. The parameter used to describe quantitatively the amount of carbides in the structure was the ratio processed area/total area. This parameter showed good because, by the analyzer, processed area is the white field and so this ratio measure the relative area occupied by the carbides. The results of these quantification are presented in Tab. 2.

Table 2 – Ratio of processed area/total area of AISI 304 steel thermal treated at 675°C by different times. Oxalic acid 10% attack.

Tempo (min)	1	2	3	5	10	15	20	30	60
% area	7.9	9.3	13.3	13.9	14.6	18.4	20.4	20.7	29.9

Analyzing these results it´s possible see that quantitative metallography, image analyzer and the used parameter are apt to show the state produced by treatments. The amount of precipitate carbide in grain boundaries increase with time and these answer show this fact. Similar measurements of sensitized structures were made by Matula at alli (2001), using a different method of quantitative metallography. In this paper the sensitization index was the ratio number of grain boundaries with carbide intercepted by a test line/total number of intercepted grain boundaries. The thermal treatments was long time (up 100 h at 650°C). The values obtained became between 0%, no one carbide intercepted, to 64% to totally sensitized structure. The saturation value was obtained by time equal 500 h at 650°C. These results are compatible with this work.

The results have showed that initially exist a fast increase in amount of precipitated carbide whose rate decrease with time, a typically kinetic of phenomena commanded by nucleation and growing. More long time treatment probably show the saturation.

4. Conclusions

The analyze of the results show that:

- Quantitative metallography can to be used to quantify sensitized structures revealed by the oxalic acid attack, and the software used has potential to make these measurements.
- The used parameter (%area) wasn´t totally just to make this quantification and must be better worked.

5. References

ASTM A 262-93. Standard Practices for Detecting Susceptibility to Intergranular Attack in Austenitic Stainless Steels.

BENNETT, B. W., PICKERING, H. W., A Finite Difference Model for the Kinetics of Sensitization, Acta Metall., Vol. 36, N°. 3, 1988.

BERRY, W. E., WHITE, E. L., BOYD, W. K., Stress Corrosion Cracking of Sensitized Stainless Steel in Oxygenated High Temperature Water, Corrosion-Nace Vol. 29, Nº 12, pp. 451-469, December, 1973.

FONTANA, M. G., PEACOCK, J. H.; Materials for Corrosion Environments, Brasunas, A. de S., Nace Basic Corrosion Course, Houston, Texas. NACE, 1970, pp. 16-4.

JOSHI, A., STEIN, D. F., Chemistry of Grain Boundaries and Its Relation to Intergranular Corrosion of Austenitic Stainless Steel, Corrosion-Nace, Vol. 28 Nº 9, pp. 321-330, September, 1972.

MATULA, M., at alli, Intergranular corrosion of AISI 316L steel, Materials Characterization, Nº 46, pp. 203-210, 2001.

PINTO, L. C. M., Quantikov - Um Analisador Microestrutural para o Ambiente Windows, 1996, 160, Tese de Doutorado, USP, São Paulo, 1996.

STEIGERWALD, R., Metallurgically Influenced Corrosion, Metals Handbook, Vol. 13, 9a Edition, ASM International, 1987.

6. Responsibility notice

The author(s) is (are) the only responsible for the printed material included in this paper.