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Aço inoxidável austeno-ferrítico contendo nióbio

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Resumo

Os aços inoxidáveis austeno-ferríticos apresentam melhor combinação de propriedades mecânicas e resistência à corrosão que os inoxidáveis austeníticos e ferríticos. A microestrutura desses aços depende da composição química e tratamentos térmicos. Nos aços inoxidáveis austeno-ferríticos, a solidificação inicia a 1450ºC com a formação de ferrita, austenita a 1300ºC e fase sigma na faixa de 600 a 950ºC. Esta última compromete a resistência à corrosão e a tenacidade desses aços. Conforme a literatura, o nióbio tem grande influência na transformação de fases dos aços inoxidáveis austeno-ferríticos. Essa pesquisa avaliou o efeito do nióbio na microestrutura, dureza e resistência de transferência de carga de um aço inoxidável austeno-ferrítico. As amostras foram solubilizadas a 1050ºC e envelhecidas a 850ºC, para promover a formação da fase sigma. Os ensaios de corrosão foram realizados em meio de saliva artificial. Os resultados mostram que a adição de 0.5% Nb no aço provoca a formação da fase de Laves. Essa fase, associada à fase sigma, aumenta a dureza do aço, reduzindo, porém, os valores da resistência de transferência de carga.

Palavras-chave: Aço inoxidável austeno-ferrítico, fase sigma, fase de Laves, resistência de transferência de carga.

Abstract

The austenitic-ferritic stainless steels present a better combination of mechanical properties and stress corrosion resistance than the ferritic or austenitic ones. The microstructures of these steels depend on the chemical compositions and heat treatments. In these steels, solidification starts at about 1450ºC with the formation of ferrite, austenite at about 1300ºC and sigma phase in the range of 600 to 950ºC. The latter undertakes the corrosion resistance and the toughness of these steels. According to literature, niobium has a great influence in the transformation phase of austenitic-ferritic stainless steels. This study evaluated the effect of niobium in the microstructure, microhardness and charge transfer resistance of one austenitic-ferritic stainless steel. The samples were annealed at 1050ºC and aged at 850ºC to promote formation of the sigma phase. The corrosion testes were carried out in artificial saliva solution. The addition of 0.5% Nb in the steel led to the formation of the Laves phase.This phase, associated with the sigma phase, increases the hardness of the steel, although with a reduction in the values of the charge transfer resistance.

Keywords: *Austenitic-ferritic stainless steels, sigma phase, Laves phase, charge transfer resistance.*

1. Introduction

In austenitic-ferritic stainless steels, the microstructure is basically determined by the chromium and nickel contents, which act as stabilizers of the ferritic or austenitic phases. Currently, these steels are being used in replacement of austenitic stainless steels in several industrial applications, where requirements for corrosion resistance and mechanical strength are greater (Senatore, 2007).

The properties of these steels are obtained with control of the microstructure, chemical composition and thermo-mechanical treatments. Another important factor is that in austenitic-ferritic steels, the nitrogen partially replaces nickel, with improvement in the mechanical properties and cost reduction (Speidel, 2006).

In these steels, the solidification starts at about 1450°C with the formation of ferrite (α), which gives origin to the austenite (γ) at about 1300°C. The M_7C_3 carbides precipitate at temperatures in the range of 950 to 1050°C in the grain contours γ/α. Below 950°C, the $M_{23}C_6$ carbides are formed (Levey, 1995). The σ phase preferentially nucleates in interface α/γ, incoherent with the matrix in the range of 600 to 950°C, and decreases the toughness of the cast materials (Maehara, 1983; Reis & Balancin, 2008). The transformation can occur through a eutectoid reaction forming sigma plus austenite with lower contents of chromium and molybdenum (Li, 1994). Although considered to be detrimental to toughness, the high hardness of the sigma phase improves the wear resistance of these steels.

In this context, the proposal of this subject was to evaluate the niobium effect on the microstructure, microhardness and phase transformation in the SEW 410 Nr.14517 austenitic-ferritic stainless steel. Niobium is widely used in the steels, due to the alphagenic effect and grain refinement. A content of 0.2% by weight is required to stabilize the carbon, according to the stoichiometry of niobium carbide (NbC and Nb_4C_3). A value of 0.5% was chosen because it is usually the maximum content for the residual elements in the standard chemical composition (Rossitti, 2000). Finally, the samples were annealed at 1050°C and aged at 850°C to simulate the sigma phase transformation during cooling.

2. Materials and methods

The SEW 410 Nr. 14517 and SEW 410 Nr. 14517 alloys containing 0.2 and 0.5% niobium were elaborated in an electric induction furnace with a system for vacuum degasification having a capacity of 1000 kg. The liquid metal was poured out in sand molds agglomerated with urethane phenolic resin (ASTM A781/781M Standard).

The chemical analyses were conducted in an optical emission spectrometer and represent the mean of two results per sample (Table 1). The cast blocks were annealed at 1050°C for one hour. Samples were cut in the form of discs with diameter 12 x thickness 4mm. Considering the phase transformation diagrams for austenitic-ferritic stainless steels, the samples were heated at 850°C for 15, 30 and 60 minutes and cooled in air (Magnabosco, 2009).

After the heat treatments, the samples were prepared according to conventional metallographic methods. Then, they were immersed in Behara reagent (125 ml H_2O, 25 ml HCl, 3g ammonium bifluoride and 0.2 g potassium metabisulfide). The quantity of the austenitic phase was determined through optical microscopy, the ferritic phase with use of ferritoscope FMP-30 (Helmut Fischer) and the sigma phase through calculation, according the Equation 1.

The values of microhardness Vickers were obtained by five measurements made in each sample with load of 100 g. The means of these measurements are given in Tables 2, 3 and 4. The electrochemical tests were performed in as lightly aggressive environment such as artificial saliva solution (Lippo, 1998).

In the setting up of the electrochemical cell, austenitic-ferritic stainless steel was used as the work electrode; saturated calomel as the reference electrode; and platinum as the auxiliary electrode. The tests were conducted at room temperature and the charge transfer resistance values are shown in Table 5.

3. Results

Table 1 gives the chemical compositions of the austenitic-ferritic stainless steels SEW 410 Nr. 14517 elaborated for this study.

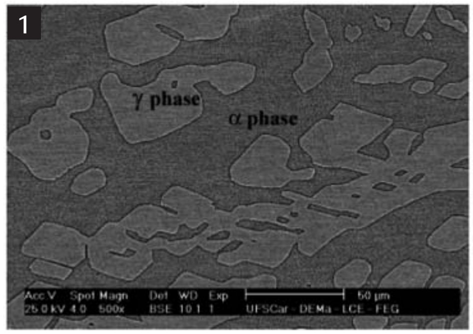
Figures 1 to 8, represent the microstructural characteristics of austenitic-ferritic stainless steel without and with niobium after annealed at 1050 °C and annealed plus heating at 850°C.

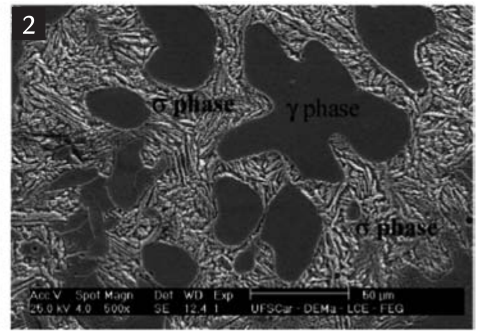
Table 2 gives the percentages of the phases and microhardness values of the SEW 410 Nr.14517 without niobium, after annealing at 1050°C and heating at 850°C. Annealed samples were used to measurements for zero minutes at 850°C. The quantities of magnetic ferritic phase were measured in the ferritoscope, while the sigma values were determined through the equation:

$$\%\ \text{Sigma Phase} = 100 - (\%\text{austenitic phase} + \%\ \text{ferritic phase}) \quad (1)$$

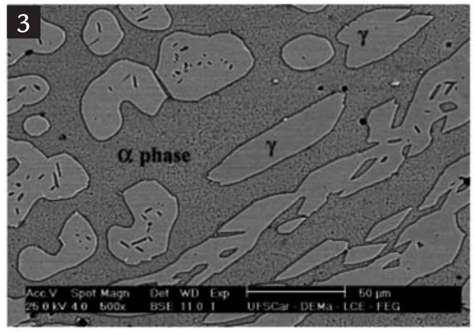
	C_{max}	Cr	Ni	Mo	Cu	Mn_{max}	Si_{max}	N	P_{max}	S_{max}	Nb
SEW Standard	**0.03**	**24.0 26.5**	**5.0 7.0**	**2.5 3.5**	**2.8 3.5**	**2.0**	**1.0**	**0.12 0.25**	**0.03**	**0.02**	**-**
SEW 0.0Nb	0.03	26.0	6.4	3.2	3.0	1.5	0.8	0.22	0.03	0.01	-
SEW 0.2Nb	0.03	26.0	6.5	3.2	2.9	1.3	0.7	0.21	0.03	0.01	0.2
SEW 0.5Nb	0.03	26.0	6.3	3.2	3.0	1.4	0.8	0.21	0.03	0.01	0.5

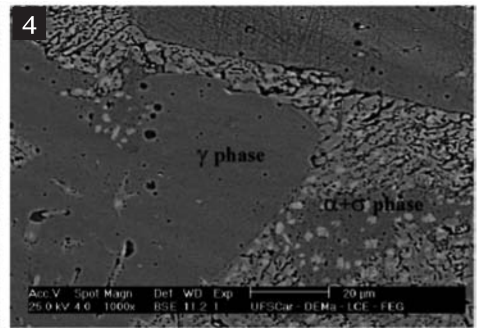
Table 1
Nominal chemical compositions of austenitic-ferritic stainless steel SEW 410 Nr. 14517 and containing niobium (% in weight).



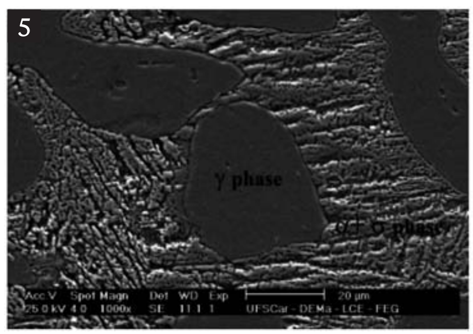


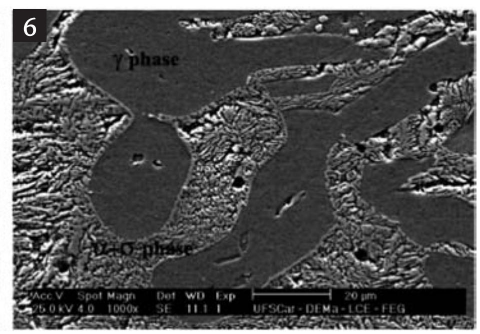
Figures (1) and (2)
Steel without niobium: annealed sample shows elongated austenite grains in the ferritic matrix (1). Practically total transformation of ferrite to sigma after annealed and aged at 850°C/60 minutes (2).



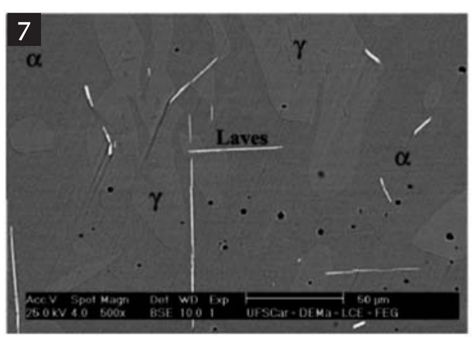


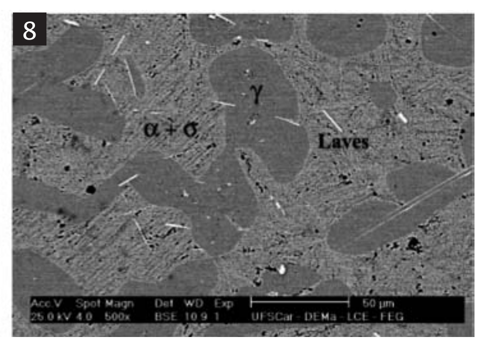
Figures (3) and (4)
Steel modified with 0.2% niobium: annealed sample with austenite grains in the ferritic matrix (3). Figure 4 shows sigma phase after aged at 850°C/15 minutes.





Figures (5) and (6)
Sigma phase increases with the time of aged at 850°C (30 and 60 minutes). There is not Laves phase in this steel.





Figures (7) and (8)
Steel modified with 0.5% niobium: annealed sample (7) and annealed plus aged at 850°C/30 minutes (8). The Laves phase appears as needles in both conditions.

Table 2
Phases percentages of the stainless steel SEW 410 and Vickers microhardness.

Time (min)	Ferrite (%)	Austenite (%)	Sigma (%)	Ferrite+ σ (HV)	Austenite (HV)
0*	46±2	54± 2	0,00	290 ± 10	255 ± 08
15	23±2	52± 2	25± 2	520 ± 20	264 ± 12
30	09±1	51± 2	40± 2	563 ± 15	263 ± 07
60	1±0,1	53± 3	46±3	650 ± 12	258 ± 10

* annealed sample.

Tables 3 and 4 give the percentages and microhardness of the phases of the SEW 410 Nr.14517 with 0.2 and 0.5% Nb, after annealing at 1050ºC and heating at 850ºC.

Table 5 gives the charge transfer resistance values of the SEW 410 Nr.14517 stainless steel without niobium and modified with 0.2 and 0.5% Nb, after annealed and heating at 850ºC.

Figures 9 and 10 show the charge transfer resistance with relation to the niobium and heat treatments effects in the stainless steels.

Time (min)	Ferrite (%)	Austenite (%)	Sigma (%)	Ferrite+ σ (HV)	Austenite (HV)
0*	45±2	55± 3	0.00	305 ± 10	260 ± 03
15	32±2	53±3	15±3	340 ± 20	262 ± 05
30	16±3	55±4	29±4	576 ± 15	283 ± 04
60	07±1	52±4	41±4	692 ±12	292 ± 03

* annealed sample.

Table 3
Phases percentages of the stainless steel SEW 410 0.2 Nb and Vickers microhardness.

Time (min)	Ferrite (%)	Austenite (%)	Sigma (%)	Ferrite+ σ (HV)	Austenite (HV)
0*	47±2	52 ±2	0,00	314 ± 15	263 ± 05
15	29±1	53± 3	18±3	450 ± 20	276 ± 06
30	05±1	52 ±3	43± 5	576 ± 15	290 ± 04
60	1±0,1	54± 5	45 ±5	609 ± 12	292± 04

* annealed sample.

Table 4
Phases percentages of the stainless steel SEW 410 0.5 Nb and Vickers microhardness.

	15 min (Kohm/cm²)	30 min (Kohm/cm²)	60 min (Kohm/cm²)
SEW without Nb	3.10 ± 0.01	1.93 ± 0.02	1.10 ± 0.01
SEW 0.2 Nb	2.60 ± 0.02	1.88 ± 0.02	1.05 ± 0.03
SEW 0.5 Nb	2.00 ± 0.02	1.40 ± 0.02	0.98 ± 0.05

Table 5
Charge transfer resistance of the stainless steels in the artificial saliva solution.

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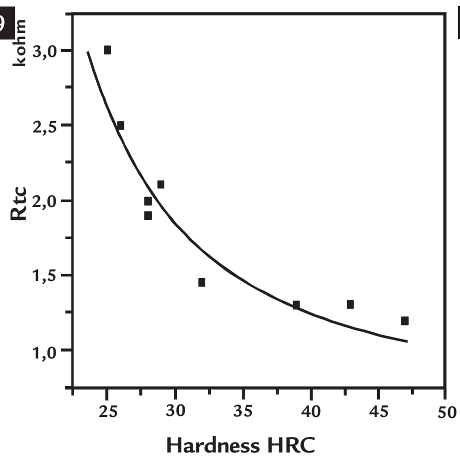

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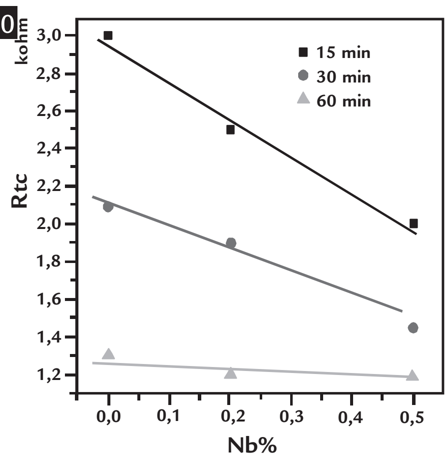

Figures 9 and 10
Charge transfer resistance for the steels regarding the hardness and the niobium containing.

4. Discussion

The chemical compositions shown in Table 1 reveal that the steels are according to SEW 410 Nr.14517 standard. With regard to Tables 2, 3 and 4, the values confirm that after heating at 850ºC, the amount of ferrite is greater in the steel modified with 0.2% of niobium.

In the steel containing 0.5% Nb, there is a precipitation of Laves phase, constituted mainly of iron, niobium and chromium (Figure 4B). Thus, the reduction of the chromium destabilizes the ferritic phase and it favors the sigma transformation, during heating at 850ºC. This explains the higher quantity of sigma phase in the steel modified with 0.5% in relation to containing 0.2% of niobium. Even with heating at 1050ºC during one hour, the Laves phase didn't solubilize.

With relation to the microstructure of the steels after heating at 850ºC, sigma phase increases with ferritic phase reduction. Observed is an increase in sigma phase with the time. After 60 minutes at 850ºC, the transformation of the ferrite phase is practically total, except in the steel containing 0.2% Nb. This explains the alphagenic effect of the niobioum (Lyakishev, 1984).

There is a tendency of the ferrite transforms to eutectoid austenite plus sigma, with the increase of the heating time at 850ºC (Li, 1994). In the steel containing 0.5% Nb, Laves phase appears associated to sigma. The EDS chemical analysis presents 39Cr28Fe26Nb3Mo2Ni2Cu, in the needles shown in Figures (7) and (8). With respect to sigma phase, the EDS chemical analysis presents 56Fe33Cr5Ni4Mo2Cu.

Regarding the average amounts of austenitic phases, the values were determined by optical microscopy. The ferritic phase's quantity was measured by ferritoscope, while sigma was calculated by percentage difference between the austenitic and ferritic phases. The increase of the sigma phase provoked a higher hardness of the steels, as the results shown in Tables (2), (3) and (4). With respect to microhardness measures, the austenitic phase is easily visible in an optical microscope, unlike sigma, which is associated with the ferritic phase. In this case, the measurements were obtained from regions with ferritic phase associated with sigma.

According to load transfer resistance in neutral pH environment, the values in Table (5) decrease with the sigma phase increasing, as the results obtained in solutions containing sodium chloride (ITMAN, 2010). Sigma phase is rich in chromium and occurs preferentially at the interface α/γ with the depletion of the ferritic phase. The growth into the ferritic matrix, with chromium and molybdenum reduction around the sigma, is one of the most common explanations for the corrosion resistance reduction of these steels (Maehara, 1983; Gunn, 2001).

Considering the same content of niobium, the charge transfer resistance reduces with heating time increasing. The results show a negative contribution to the corrosion resistance with niobium increasing, although the hardness of the sigma phase can favor the use of these steels, where the wear is a factor to be minimized.

5. Conclusions

The results of this research show:

- Alphagenic effect of niobium in solution.
- Decreasing of the charge transfer resistance, with niobium and aging time increasing.
- Decreasing of the charge transfer resistance with sigma and Laves phases.
- Ferrite becomes practically sigma plus secondary austenite after 60 minutes at 850ºC.
- Increasing in the hardness of the matrix with sigma phase.
- Niobium increasing favors the formation of Laves phase.
- Laves phase is not solubilized at 1050ºC and destabilizes the ferritic phase.

6. Acknowledgement

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