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Influence of heat treatments on the intergranular corrosion resistance of the AISI 347 cast and weld metal for high temperature services

A. Yae Kina^a, V.M. Souza^a, S.S.M. Tavares^{a,*}, J.A. Souza^a, H.F.G. de Abreu^b

^a Universidade Federal Fluminense, Departamento de Engenharia Mecânica/PGMEC, Rua Passo da Pátria 156, CEP 24210-240 Niterói, RJ, Brazil

^b Universidade Federal do Ceará, Departamento de Engenharia Metalúrgica e de Materiais, Brazil

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ABSTRACT

Stabilized austenitic stainless steels such as AISI 347 can be selected to high temperature services due to the high creep resistance of austenite phase. Niobium is added to form fine NbC carbides, which increase the creep resistance and prevents intergranular corrosion. AISI 347 wires and electrodes are frequently used as feed metal to weld AISI 321 and 347 wrought plates. In the present work, we studied the intergranular corrosion resistance of cast structures of AISI 347 steel using electrochemical potentiodynamic reactivation tests. The study applies to cast and weld metal structures subjected to high temperature usage. The solution and stabilization treatments were evaluated. It was found that the solution treatment at 1100 °C followed by stabilization in the NbC precipitation range of temperature is of fundamental importance to avoid sensitization.

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1. Introduction

Austenitic stainless steels can be selected to high temperature services due to the creep properties of the fcc austenitic structure and the oxidation resistance provided by chromium. Stabilized austenitic stainless steels such as AISI 347 and 321 present other two advantages related to the Nb and Ti additions. These elements are added to form TiC and NbC carbides and stabilize the steel against sensitization in the 450 °C–850 °C range (Pickering, 1983). The fine carbides and carbonitrides particles also increase the strength and creep resistance of the steel (Padilha et al., 2003).

In the case of the AISI 347 grade, the best corrosion resistance properties are achieved when all carbon of the steel is combined to niobium to form NbC. Nitrogen can also be dissolved in this particles forming Nb(C,N) (Pickering, 1983). For

this reason the Nb content must be 10 times the (C + N) weight present in the alloy.

When the AISI 347 is selected to services at temperatures in the 450 °C–800 °C range, stabilized steels must be heat treated to provide the maximum NbC precipitation. The kinetics of NbC precipitation is described by a TTT curve in which the nose is at higher temperature than the TTT diagram of chromium carbides (Padilha et al., 2003). If a solution-treated AISI 347 steel is put into service at a temperature into the sensitization interval (450 °C–800 °C) then Cr carbides will likely form, since there is free carbon in solid solution. This will happen before NbC precipitation, resulting in a poor corrosion resistance. Stabilization treatment is so necessary to promote the NbC (or TiC) precipitation.

Welding of stabilized steels AISI 321 and 347 with GTAW process are often performed using AWS 347 wires. Cast com-

* Corresponding author.

E-mail address: ssmtavares@terra.com.br (S.S.M. Tavares).

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Table 1 – Chemical composition (wt%) of the AISI 347 steel studied

C	0.045
Si	0.870
Mn	1.460
S	0.012
P	0.016
Cr	19.100
Nb	0.550
Mo	0.030
Cu	0.070
Ni	9.650

ponents of AISI 347 are also used. This work is focused on the study of the influence of solution and stabilization treatments on the intergranular corrosion resistance of these cast structures for high temperature services (~600 °C).

2. Experimental

The material studied was an AWS ER 347 wire of 2.5 mm diameter and chemical composition shown in Table 1. Samples with around 7 g were melted in an arc furnace. The arc furnace basically is a GTAW machine where the fusion is carried out in sealed chamber with pure argon atmosphere. Solidification occurs in a water-cooled cooper crucible, after this the samples were heat treated in vacuum. The heat treatments applied were solution treatment and stabilization. Solution treatments were carried out at 1100 °C by 1 h and 5 h, followed by water quenching. Stabilization treatments were performed at 850 °C, 875 °C, 900 °C, 925 °C, 950 °C and 975 °C by 2 h. A group of samples were stabilized without previous solution treatment and another group was solution treated and stabilized. The samples were aged at 600 °C for 24 h and 48 h. Fig. 1 shows a schematic sequence of the heat treatments performed.

The intergranular corrosion resistances of the samples were investigated by double-loop electrochemical potenti-

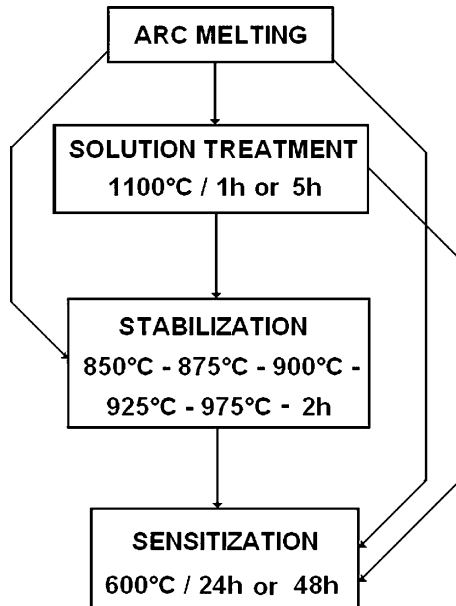


Fig. 1 – Fluxogram of preparation of samples.

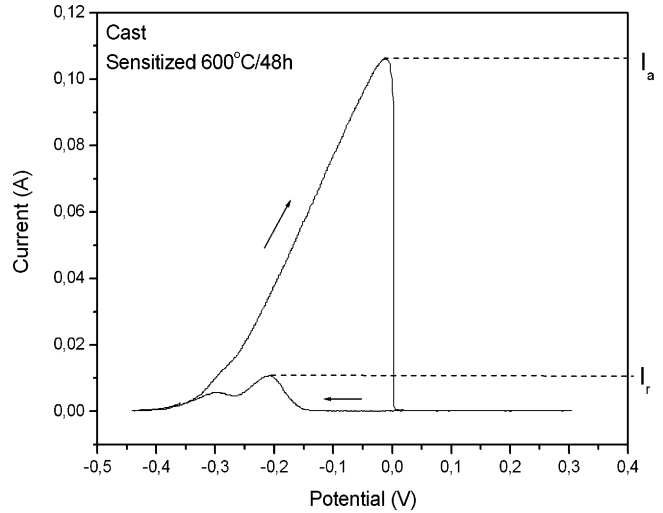


Fig. 2 – DL-EPR curves of the sample cast and aged at 600 °C for 24 h showing I_r and I_a obtained from the curves.

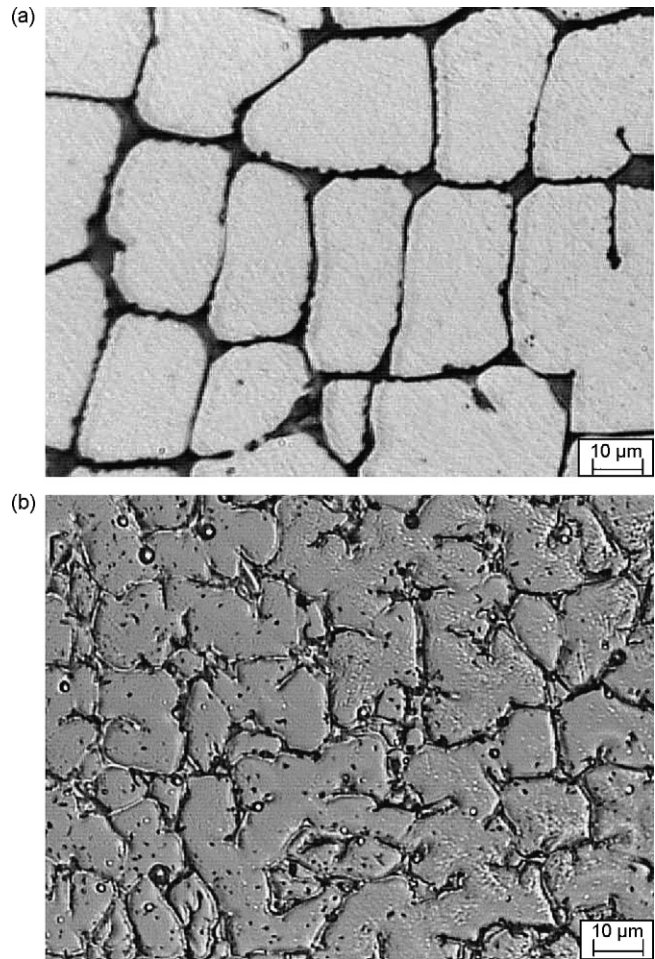


Fig. 3 – Microstructures of the material in the as cast condition: (a) Behara's etching; (b) electrolytic etching in 10% oxalic acid solution.

dynamic tests (DL-EPR) (Lopez et al., 1997). These tests were conducted in a conventional three-electrode cell using a Pt foil as the auxiliary electrode and a saturated calomel electrode (SCE) as the reference one. The working electrode was constructed using the AISI 347 samples embedded in epoxy resin grinded with grid 400 emery paper, degreased with alcohol and cleaned in water. The tests were initiated when steady-state open circuit potential (E_{oc}) was developed (about 30 min) followed by the potential sweep in the anodic direction at 1 mV s^{-1} until the potential of 0.3 V (vs. SCE) was reached, then the scan was reversed in the cathodic direction until the E_{oc} . The electrolyte was 0.5 M H_2SO_4 + 0.01 M KSCN solution. The loss of corrosion resistance due to the chromium-depleted regions, i.e. the degree of sensitization, was evaluated from the ratio I_r/I_a , where I_a is the activation peak current of the anodic scan and I_r is the reactivation peak current in the cathodic scan, as shown in Fig. 2.

The microstructures were investigated by optical and scanning electron microscopy with energy dispersive spectrometer (EDS). Microstructures were revealed by electrolytic etch (ASTM A262-Practice A; ASTM, 1993) and Behara's etch (20 ml of HCl, 80 ml of H_2O and 0.3 g of potassium metabisulfite).

3. Results and discussion

Fig. 3(a) and (b) shows the microstructure of the as cast sample containing austenite and $14.9\% \pm 3.5\%$ of delta ferrite. The microanalysis of the delta ferrite and austenite phases (Fig. 4(a and b)) show that niobium is concentrated in the ferrite phase. A small peak of this element is observed in the ferrite phase analysis, despite of its low percentage present in the steel (0.550%).

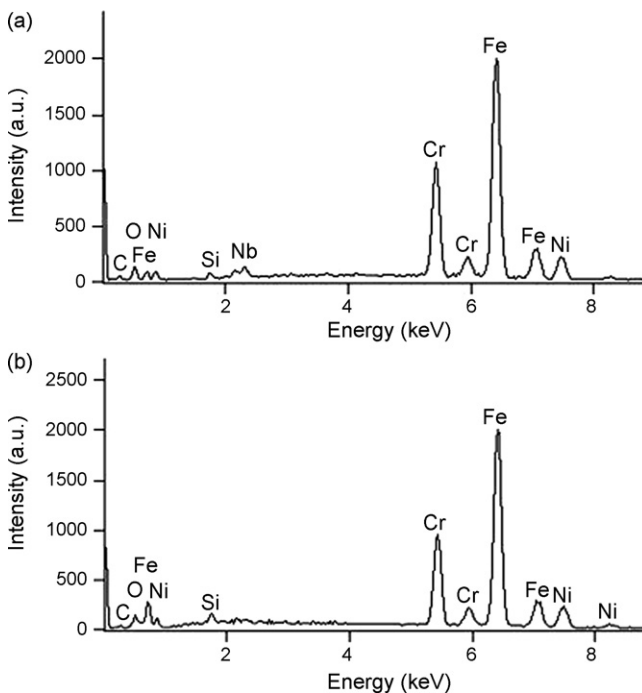


Fig. 4 – EDS spectra of the as cast sample: (a) delta ferrite; (b) austenite.

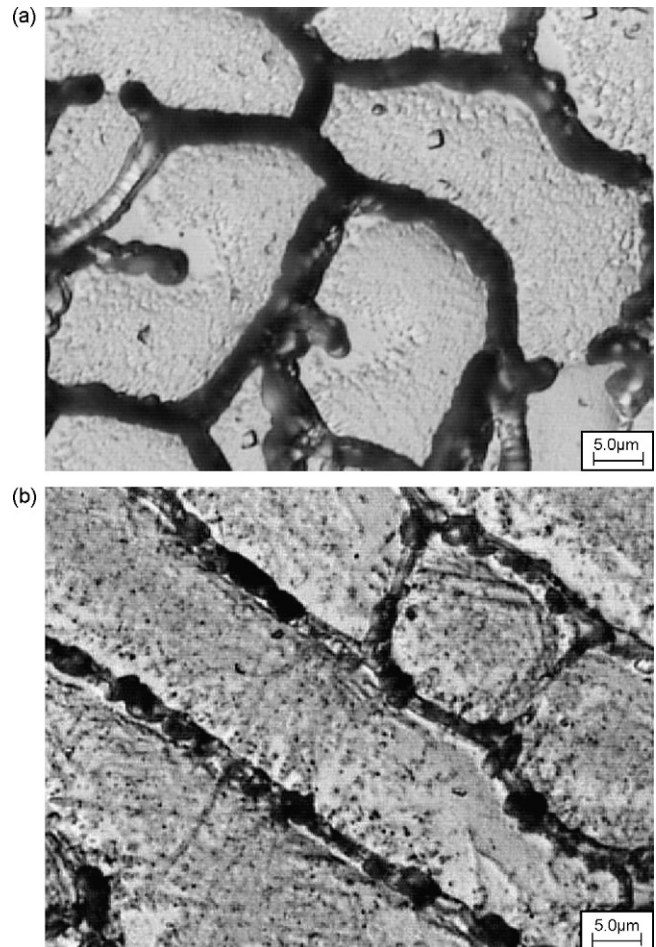


Fig. 5 – Microstructures revealed with electrolytic etching in 10% acid oxalic solution: (a) sample sensitized at 600 °C for 24 h; (b) sample stabilized at 900 °C and sensitized at 600 °C for 48 h.

The DL-EPR curve in the as cast condition does not show a reactivation peak ($I_r/I_a = 0.0$). However, if this structure is aged at 600 °C sensitization occurs, as can be seen by microscopy (Fig. 5(a)) and also by DL-EPR test (Fig. 2). Fig. 5(b) shows the micrograph of the sample stabilized at 850 °C and aged at 600 °C for 48 h. At this condition, fine precipitates are observed in the austenite, but there are also many holes in the austenite/ferrite boundaries, indicating that the material became sensitized. Indeed, the DL-EPR curves of the samples stabilized at 850 °C and 900 °C show pronounced reactivation peaks (Fig. 6(a and b)). The same behavior is observed for other stabilization temperatures. It is interesting to note that, as general behavior, the reactivation loop is composed by two peaks. This can be attributed to some composition heterogeneities of the austenite phase in the cast structure.

Stabilization treatments become more effective when the material is previously solution treated at 1100 °C. Fig. 7 compares the behavior of the degree of sensitization (I_r/I_a ratio) as function of the stabilization temperature with and without previous solution treatment at 1100 °C for 1 h. This treatment reduces the delta ferrite content to $10.8\% \pm 1.2\%$ and, consequently, dissolves more niobium into the austenite, pro-

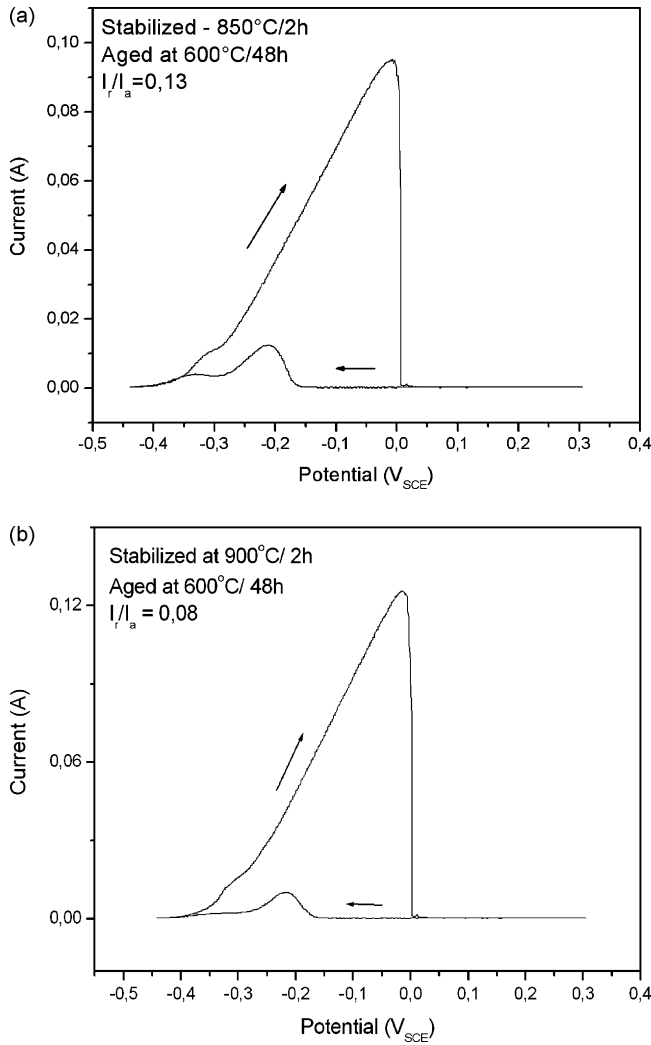


Fig. 6 – DL-EPR curves: (a) sample stabilized at 850 °C and sensitized at 600 °C for 24 h; (b) sample stabilized at 900 °C and sensitized at 600 °C for 48 h.

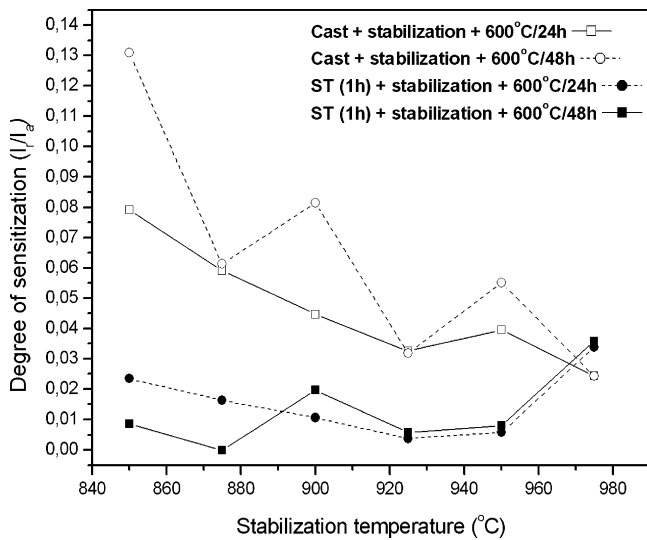


Fig. 7 – Degree of sensitization (I_r/I_a ratio) as function of the stabilization temperature with and without previous solution treatment at 1100 °C for 1 h.

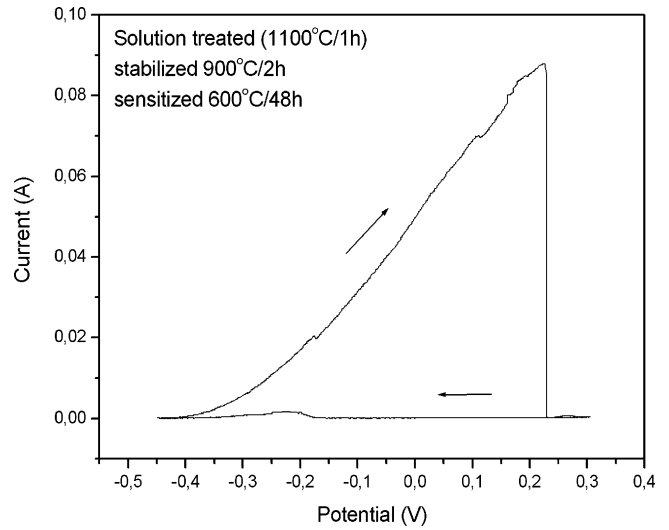


Fig. 8 – DL-EPR curves of sample solution treated at 1100 °C for 1 h, stabilized at 900 °C and sensitized at 600 °C for 48 h.

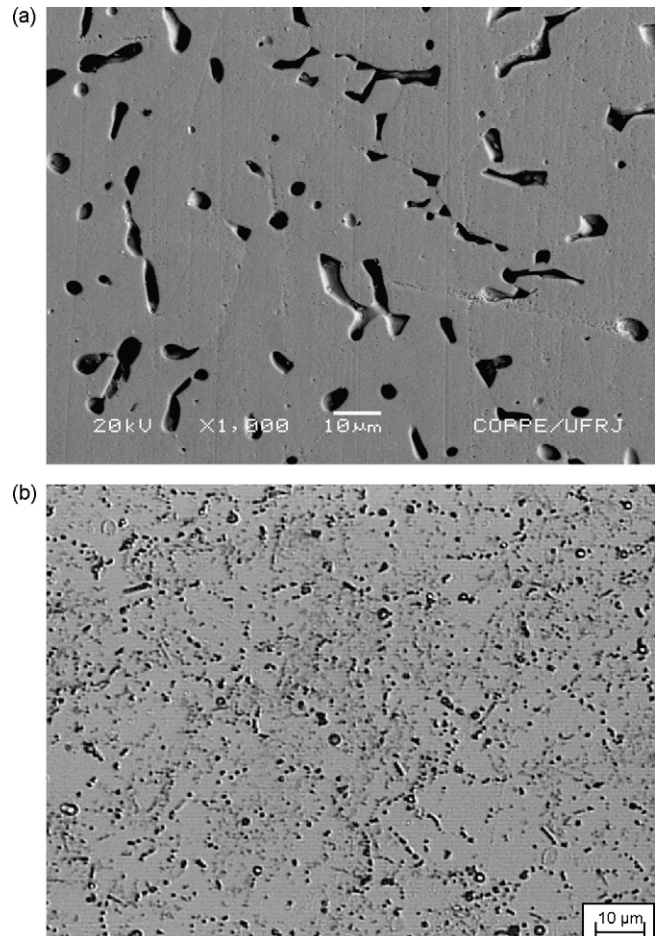


Fig. 9 – Microstructures of samples solution treated at 1100 °C for 1 h (a) and 5 h (b).

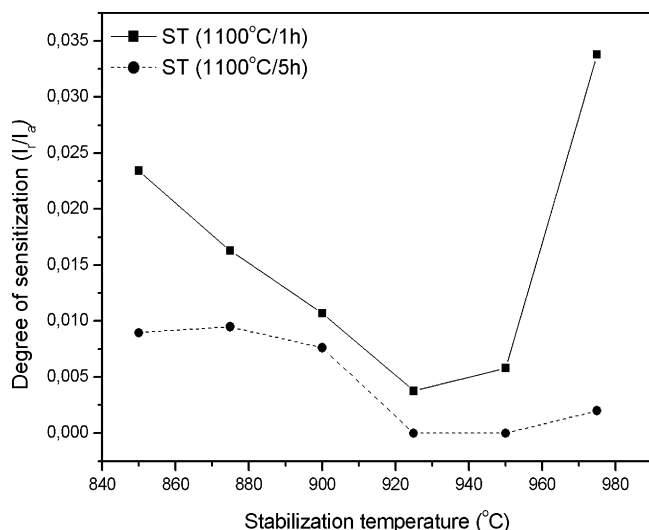


Fig. 10 – Degree of sensitization (I_T/I_a ratio) as function of the stabilization temperature for samples previously solution treated at 1100 °C for 1 h and 5 h. The samples were stabilized and treated at 600 °C for 24 h.

viding a better response to subsequent stabilization treatment.

However, the samples solution treated at 1100 °C for 1 h and stabilized still present a small reactivation peak in the DL–EPR test (see Fig. 8, for instance). The increase of the time at 1100 °C in the solution treatment, from 1 h to 5 h, reduces the ferrite content, as seen by comparing Fig. 9(a) and (b). The delta ferrite content after the solution treatment for 5 h decreases to $6.1\% \pm 1.0\%$. This also leads to a better Nb redistribution, improving the response to the stabilization treatment. As consequence, the material shows a further decrease of the degree of sensitization, as shown in Fig. 10.

Fig. 10 also shows that the stabilization temperatures which provide the lower I_T/I_a ratio are 925 °C and 950 °C. Besides of this, at these temperatures the NbC particles are relatively coarse, which is recommended to avoid re-heating cracks in this class of steel (Pickering, 1983; Younger and Baker, 1961).

The sequence of solution treatment and stabilization is sometimes of difficult application. However, this procedure

is the most indicated to avoid sensitization of welded parts and cast components during high temperature services. The sequence of solution treatment and stabilization can be carried out directly, just as one treatment with two temperatures steps: the material is treated at 1100 °C for 1 h or, preferentially, 5 h, and then cooled to the stabilization temperature (925 °C or 950 °C), where it is maintained by a couple of hours.

4. Conclusions

The intergranular corrosion resistance of cast components and weld metal of AISI 347 steel is improved by solution treatment at 1100 °C followed by stabilization in 925 °C–950 °C range. Stabilization without solution treatment results in very poor intergranular corrosion resistance in welded and or cast components. The stabilization treatment dissolves part of the delta ferrite and redistributes the niobium in the austenite phase. The increase of the time of solution treatment from 1 h to 5 h promotes higher ferrite dissolution and enhances niobium redistribution, resulting in a decrease of the degree of sensitization (I_T/I_a ratio) to very low values after stabilization. Solution treatment and stabilization are post-weld heat treatments specially indicated to multipass welds and cast components made of stabilized austenitic stainless steels designed to high temperature services (450 °C–800 °C).

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