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Influence of heat treatments on the microstructure and degree of sensitization of base metal and weld of AISI 430 stainless steel

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ABSTRACT

AISI 430 is a non-stabilized ferritic stainless steel grade with carbon content lower than 0.12%.After hot and cold rolling this material is annealed. The slow cooling after soaking at temperatures between 900°C and 1000°C promotes the formation of a high quantity of carbides and nitrides, while the rapid cooling partially suppresses the formation of these precipitates, but introduces martensite in the microstructure. Intergranular martensite can also be produced in the weld metal and in the heat affected zone (HAZ) of welds of non-stabilized ferritic stainless steels. In this work, several heat treatments between 900°C and 1000°C, with different cooling rates, were performed in a commercial sheet of AISI 430 grade. Also, an autogenous welding was produced with GTAW process, and post weld heat treatment at 700°C was carried out. The different microstructures produced were analyzed by optical and scanning electron microscopy (SEM). The degree of sensitization was measured by double loop electrochemical potentiodynamic tests (DL-EPR). The pitting corrosion resistance was evaluated by cyclic polarization tests in 3.5%NaCl solution. Hardness and toughness tests were also performed in selected heat treatment conditions. The results indicate that the slow cooling results in a higher degree of sensitization than observed in the material rapid cooled from the annealing temperature. The ferritic martensitic structure produced by water cooling has higher pitting potential and lower degree of sensitization, but is brittle at room temperature. A subsequent tempering treatment between 600 and 800°C can increase the toughness, but the corrosion resistance may decrease due to carbides precipitation.The heat affected zone of AISI 430 welds contains intergranular martensite, which is brittle and susceptible to corrosion attack. Post weld heat treatment at 700°C decomposed the martensite into ferrite and carbides and improved the corrosion resistance.

Key-words: ferritic stainless steels, microstructure, DL-EPR test.

1. INTRODUCTION

AISI 430 steel is one of the most popular ferritic stainless steels. Although more modern ferritic stainless steels have been developed, the production of AISI 430 is still elevated due to its low cost and good corrosion properties.

The influence of heat treatments on microstructure, corrosion resistance and mechanical properties of stainless steels is a key issue. Depending on the final heat treatment, the mechanical properties and corrosion properties may vary significantly. Frequently, the best heat treatment for corrosion resistance is not the best for the desired mechanical properties.

On the other hand, welding always produces important changes on the microstructure of weld metal and heat affected zone (HAZ) which affects corrosion resistance and mechanical properties. In the case of ferritic stainless steels, the main change produced in fusion welding processes is the pronounced grain growth in the weld metal and HAZ [1]. Besides this, in non-stabilized steels, such as AISI 430, intergranular martensite may form and intergranular precipitation of $M_{23}C_6$ carbides and $M_{23}(C,N)$ carbonitrides may occur in the coarse grain heat affected zone (CGHAZ)[1-4].

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Krafft [5] reported a failure case occurring in the weld metal (WM) and heat affected zone (HAZ) of an AISI 430 component of a heat recovery steam generator. The post weld heat treatment (PWHT) applied to the welded joint was non-uniform and insufficient to promote the proper tempering of martensite.

In this work, the effect of heat treatments on the microstructure and corrosion resistance of an AISI 430 steel was studied by means of double loop electrochemical potentiodynamic reactivation (DL-EPR) tests and pitting corrosion tests. The effect of a post weld heat treatment (PWHT) on microstructure and corrosion resistance of an autogenous welded joint produced by gas tungsten arc welding (GTAW) was also investigated.

2. MATERIALS AND METHODS

The material studied was a hot rolled and annealed sheet of 3.0 mm of thickness of AISI 430 steel with composition shown in Table 1. Specimens of the base metal with dimensions (15 x 10 x 3) mm³ were cut for heat treatment and corrosion tests.These specimens were submitted to isothermal heat treatments for 1 hour in the temperatures 900^{o}C, 950^{o}C and 1000^{o}C. Three cooling media were used: water (fast cooling), air (moderate) and furnace (slow cooling).

Some specimens were heat treated in selected conditions and machined to the dimensions of sub-size Charpy test specimens with (55 x 10 x 2.5) mm³ and V-notch. Charpy impact tests were carried out at room temperature.

Table 1: Chemical composition of the base material.

Cr	Ni	Si	P	C	N	S	Fe
16.14	0.19	0.31	0.025	0.048	0.045	0.001	Bal.

Two portions of the rolled sheetcut and machined to dimensions (100 x 100 x 2.5) mm^3 were autogenous welded with automatic GTAW process with 99.9% Ar as gas protection. The parameters were adjusted to obtain full penetration. The heat input was 0.8kJ/mm. Specimens of the welded joint, including the weld metal (WM) and the HAZ were cut for post weld heat treatment (PWHT) and subsequent analysis by electrochemical corrosion tests and microscopy. The PWHT was carried at 700^{o}C for 1 hour with water cooling.

The degree of sensitization was measured by double loop electrochemical potentiodynamic tests (DL-EPR) [6-7]. A three electrodes cell, with working electrode (WE) of the material analyzed, saturated calomel electrode as reference and Pt wire as counter-electrode were used. WE's were prepared with the specimen to be analyzed embedded in epoxy resin among with a cooper wire for electric contact. The surface of WEs were prepared by grinding with sand paper grit 100, 200, 300 and 400. The area exposed to the rest solution was delimitated with enamel. The test solution was 0.25MH_2SO_4 and 0.01M KCSN. After 1 hour of stabilization of the open circuit potential (E_{OCP}) the anodic polarization with sweep rate 1 mV/s initiated. The sweeping was reverted to the cathodic direction at 0.300 V_{SCE}.

After the DL-EPR tests, the pitting corrosion resistance of some selected conditions were evaluated by cyclic polarization tests in 3.5%NaCl solution at room temperature. The tests were also conducted in a three electrode cell, but WE's were grinded and polished with diamond paste. After the stabilization of the E_{OCP}, the working electrode was polarized in the anodic direction with sweep rate 1mV/s. The scanning was reverted to the cathodic direction when the current density reached 5.10^{-3} A/cm^2.

Each corrosion test was repeated 3 times. Average values and standard deviations are presented in the results.

The microstructural investigation was performed by optical microscopy (OM) and scanning electron microscopy (SEM) with the specimens etched with Villela's reagent (95 ml ethanol, 5 ml HCl and 1g of picric acid).

3. RESULTS AND DISCUSSION

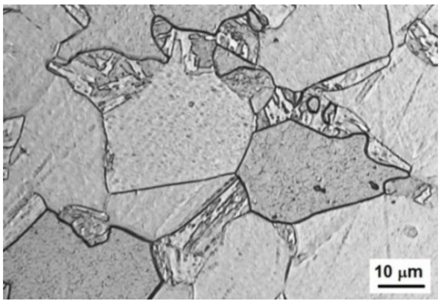
3.1. Heat treatments in the base material

Figs.1(a-c) compare the microstructures of specimens treated at 950^{o}C and cooled in water, air and furnace. The slow cooling after soaking at temperatures between 900^{o}C and 1000^{o}C promotes the formation of a high quantity of chromium carbides and nitrides (Fig.1(a)), while the rapid cooling partially suppresses the formation of these precipitates, but introduces martensite in the microstructure (Figs. 1(b) and (c)). The martensite volume fraction of specimen treated at 950°C and water quenched was (0.33 ± 0.04).

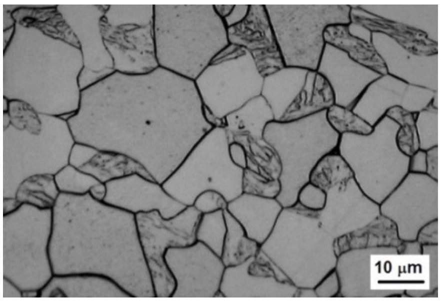
Carbon and nitrogen diffusion is so fast in the ferritic phase that is not possible to suppress completely the carbides and nitrides precipitation, but they are too fine to be observed by optical microscopy [8]. Fig. 2(a) shows the intra and intergranular precipitation observed by SEM. The EDS analysis confirms that these particles are chromium rich carbides (Fig.2(b)).

Air cooling produces a microstructure with ferrite and martensite partially decomposed, with some precipitates, as show in Fig. 3.

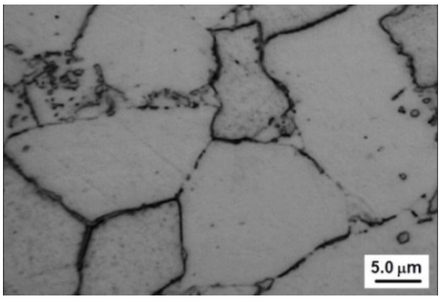
Fig. 4 presents the DL-EPR curve of specimen heat treated at 950°C and furnace cooled. The main result of the DL-EPR test is the degree of sensitization (DOS) given by the ratio Ir/Ia, where Ir is the reactivation peak of current, and Ia is the activation peak of current. Fig. 5 shows how the DOS varies with heat treatment temperature and cooling media. Slow cooling, which produces a microstructure of ferrite and precipitates (carbides and nitrides) gives the highest DOS, i.e. the higher susceptibility to corrosion due to chromium depletion. The ferritic-martensitic microstructure with low density of carbides and nitrides gives the lower DOS.



(a)

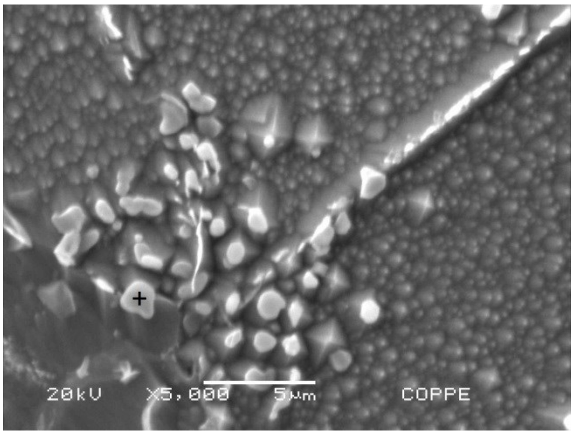


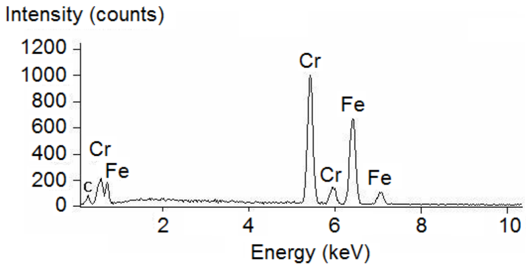
(b)



(c)

Figure 1: Microstructures of specimens treated at 950°C and cooled in (a) water, (b) air and (c) furnace.





(a) (b)

Figure 2: Details of precipitates in specimen treated at 950°C and cooled in furnace: (a)SEM image; (b) EDS spectra.

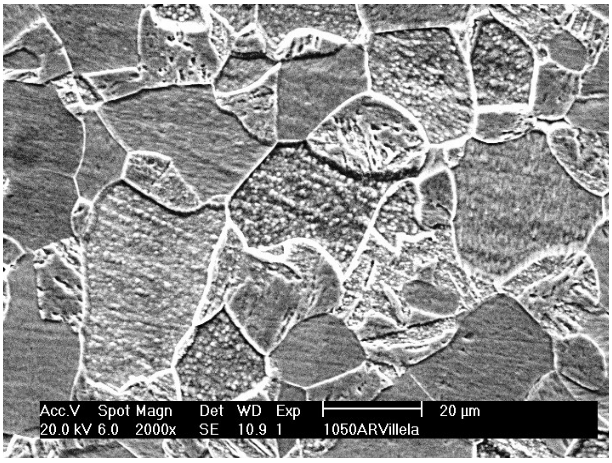


Figure 3: Microstructure of specimen treated at 1000°C and air cooled.

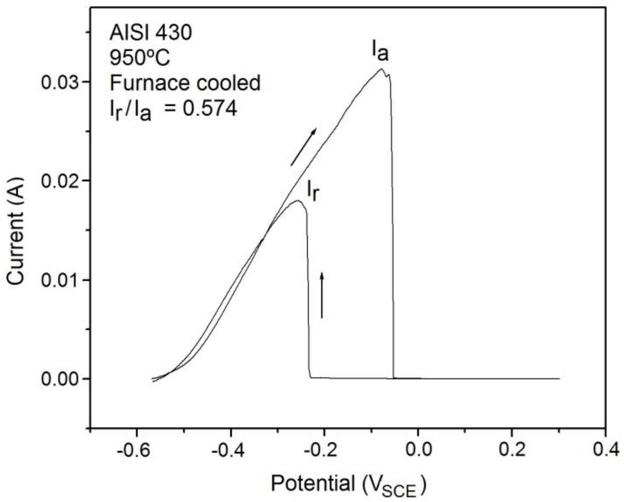


Figure 4: DL-EPR curve of specimen het treated at 950°C and furnace cooled.

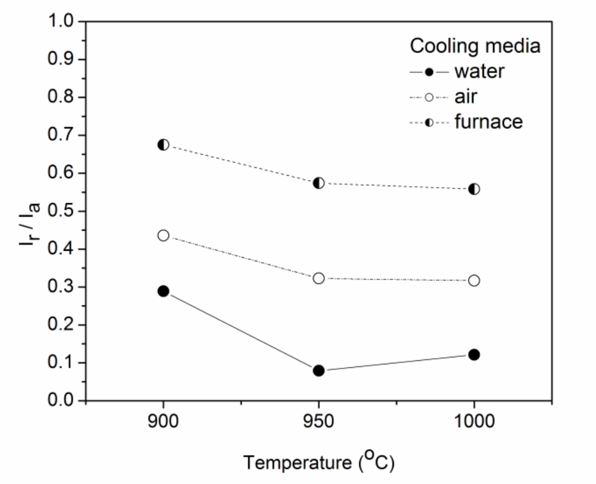


Figure 5: Variation of the degree of sensitization (Ir/Ia) with heat treatment temperature and cooling media.

Table 2 shows the pitting potentials measured in polarization test in 3.5%NaCl solution of specimens treated at 900°C, 950°C and 1000°C and water cooled. The specimen treated at 950°C and water cooled is also the one with higher pitting resistance.

Table 2: Pitting potentials measured in cyclic polarization tests in 3.5%NaCl.

HEAT TREATMENT	PITTING POTENTIAL (V_{SCE})
Water quenching from 900°C	0.595 ± 0.090
Water quenching from 950°C	0.757 ± 0.062
Water quenching from 1000°C	0.634 ± 0.070

Table 3 shows the Charpy impact energy of specimens quenched from 950°C, with and without subsequent tempering at 600°C, 700°C and 800°C for 1 h. The DL-EPR tests and pitting corrosion tests suggest that the microstructure of ferrite and martensite with low density of precipitates is the most favorable to a better corrosion resistance of AISI 430 steel. However, the martensite is brittle and makes the hardness increase. As a result, the toughness of the steel quenched in water from 950°C is very low, although it can be improved by tempering in the 600 – 800°C range.

Table 3: Impact toughness and hardness of specimens quenched from 950°C and tempered.

HEAT TREATMENT	IMPACT TOUGHNESS(J)	VICKERS HARDNESS (HV30)
Quenched (950°C/1h-water)	3.0 ± 0.5	251 ± 4
Quenched and tempered 600°C	18.0 ± 0.5	231 ± 3
Quenched and tempered 700°C	19.0 ± 0.5	195 ± 4
Quenched and tempered 800°C	22.0 ± 0.5	177 ± 5

3.2. Weld Metal (WM) and Heat Affected Zone (HAZ)

Fig. 6 exhibits the macrostructure of the welded joint, where the pronounced grain growth can be observed. Figs. 7(a-b) show the microstructure of the coarse grain HAZ in the as welded condition. Intergranular martensite is clearly shown. In some very coarse grains, such as observed in Fig. 7(b), intragranular carbides and nitrides are noted. The intergranular martensite contains less Cr than the ferritic matrix, and, as a consequence, this phase is preferentially attacked in the electrochemical corrosion tests (see Figs 8(a-b)).

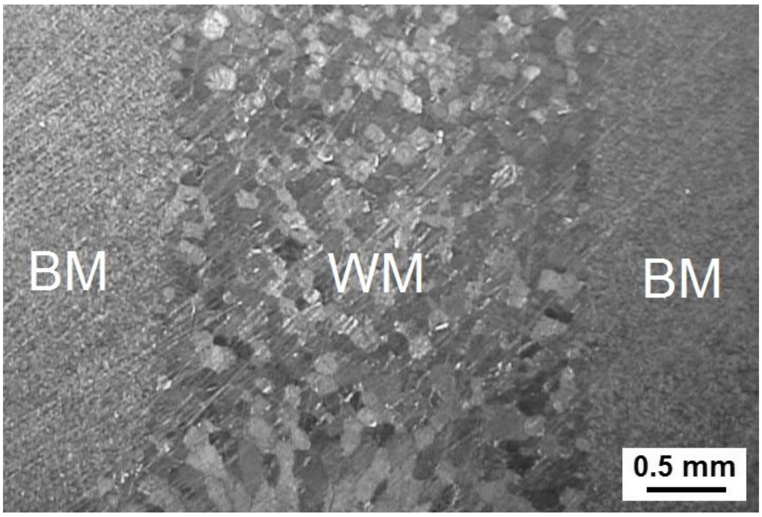
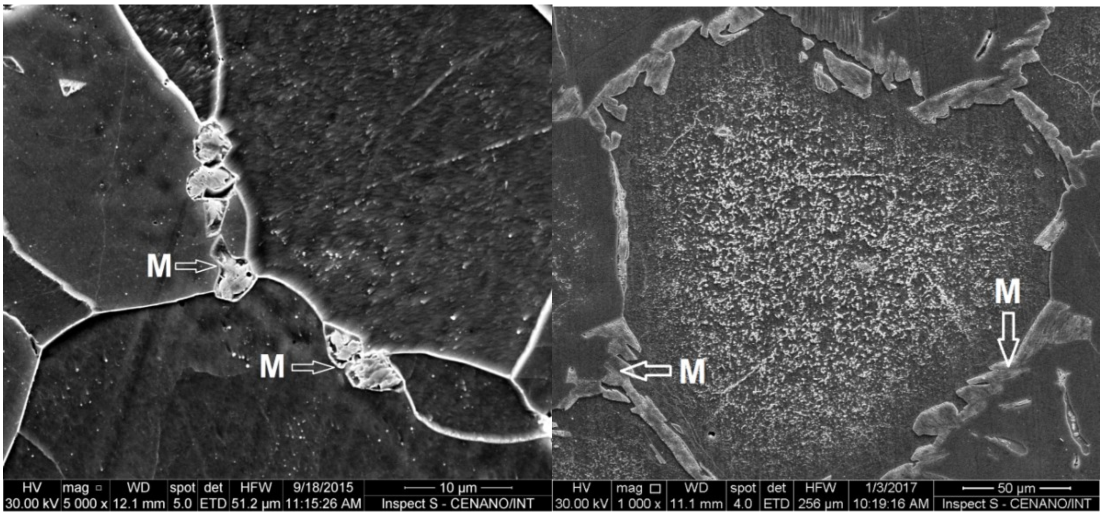
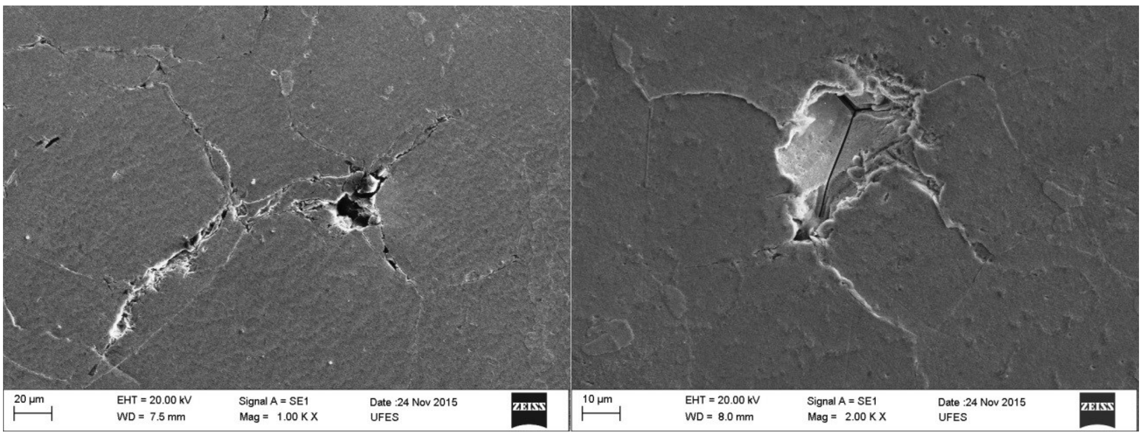


Figure 6: Macrostructure of welded joint. WM = Weld Metal; BM = Base Metal.



(a) (b)

Figure 7: Microstructure of the coarse grain HAZ, as welded. (Intergranular martensite indicated as “M”).



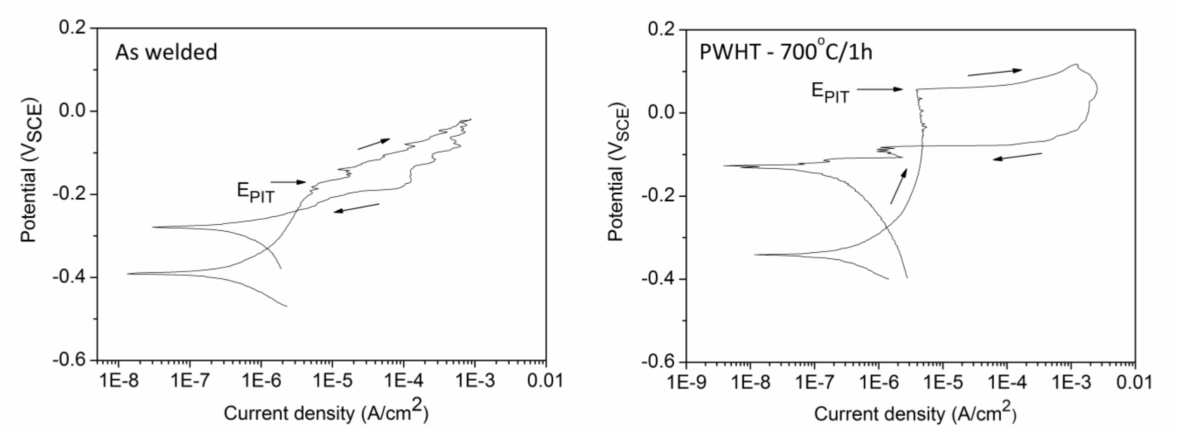
(a) (b)

Figure 8: Preferential attack of intergranular martensite after pitting corrosion test of the as welded AISI 430.

Figs. 9(a-b) compare the pitting potentials curves of the welded joint before and after PWHT

(700°C/1h). Table 4 shows the DOS and E_{PIT} parameters of the weld metal plus HAZ before and after the PWHT at 700°C. It is observed an important decrease of DOS and a small increase of the pitting potential with the PWHT. The main microstructural change observed is the chromium carbide precipitation (tempering reactions) in the martensite. This is clearly observed in the HAZ and base metal, as shown in Figs.10(a-b).

The effects of PWHT at 700°C on the DL-EPR results of the base metal, however, may be different from that observed in the WM and HAZ. If the base metal was previously quenched from 950°C and has the microstructure of ferrite, martensite and few carbides/nitrides, the tempering at 700°C promotes the increase of the DOS due to additional chromium carbides precipitation. Even considering that a healing effect due to chromium diffusion is reported [8], in this work the tempering at 700°C of the base metal increased the DOS from 0.079 to 0.250 (curves not shown). Thus, the tempering of martensite at 700°C for 1h is beneficial to the corrosion resistance of the HAZ, but has a negative effect to the corrosion resistance of the base metal if it was welded in a water quenched condition. It must be pointed out that the grains in the HAZ are very coarse and the un-tempered martensite is concentrated in the grain boundaries. On the other hand, the martensite volume fraction in the base metal water quenched from 950°C (0.33 ± 0.04) was much higher than the amount found in the HAZ. These differences may explain why the intergranular martensite of the HAZ has a more deleterious effect in the corrosion resistance than the martensite obtained by quenching treatment in the base metal, and why the PWHT is so important to the HAZ.

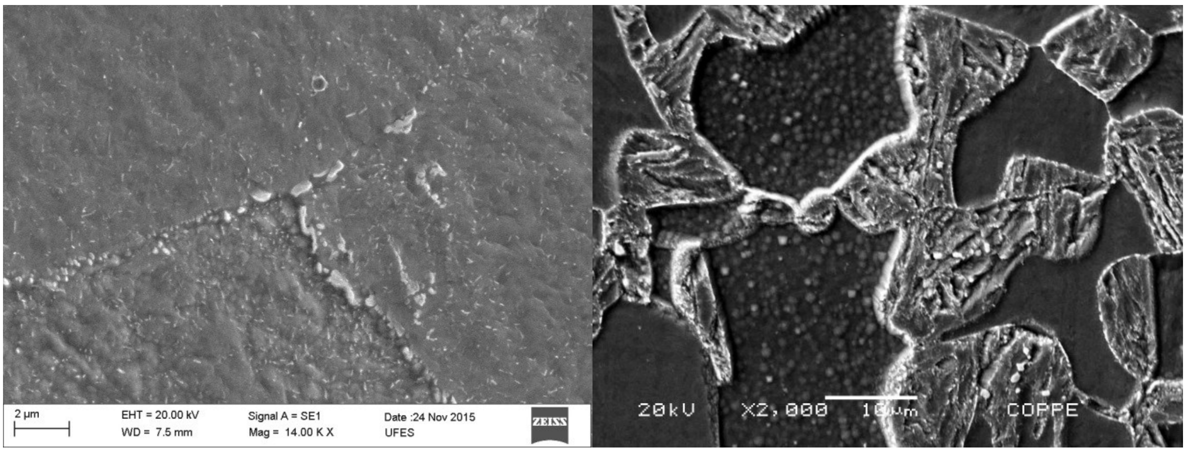


(a) (b)

Figure 9: Polarization curves of HAZ and WM: (a) as welded; (b) after PWHT at 700°C for 1 hour.

Table 4: Pitting potentials (EPIT) and DOS of WM and HAZ before and after PWHT at 700°C for 1 h.

MATERIAL	DOS (Ir/Ia)	E_{PIT} (V_{SCE})
HAZ+WM untreated	0.33 ± 0.08	- 0.140
HAZ+WM with PWHT	0.13 ± 0.05	0.060



(a) (b)

Figure 10: Microstructures resulted from tempering at 700°C for 1h: (a) HAZ and (b) base metal quenched from 950°C.

4. CONCLUSIONS

This work investigated the influence of heat treatments on the microstructure and degree of sensitization (DOS) of base and weld metal of AISI 430 stainless steel.

A microstructure of ferrite and martensite (~33%) is produced in the rolled sheet of AISI 430 by water quenching from 950^{o}C. This treatment and microstructure gives the higher corrosion resistance to the hot rolled base metal if compared to air and furnace cooling. However, the martensite turns the material very brittle, as concluded by Charpy impact tests at room temperature.

A heat treatment in the 600 - 800^{o}C after quenching from 950^{o}C increases the toughness of the base metal due to the tempering of martensite. However, the degree of sensitization may increase due to the intense carbide precipitation, even if some healing effect due to Cr diffusion occurs.

The microstructure of the heat affected zone consisted of coarse ferritic grains with intergranular martensite. The intergranular martensite is preferentially attacked because is Cr-depleted. A PWHT promotes tempering of the intergranular martensite and increases the corrosion resistance of the welded joint.

5. ACKNOWLEDGEMENTS

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