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Slow strain rate tensile test results of new multiphase 17%Cr stainless steel under hydrogen cathodic charging

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ABSTRACT

Supermartensitic stainless steels were developed to allow high corrosion resistance in seawater environments along with high mechanical strength. These materials derived from conventional 12–13% Cr steels, but contained an extra-low carbon content (<0.03%), Ni and Mo addition. Recently, the increase of Cr and other ferritizing elements increased the amounts of ferrite phase in the microstructure. Depending on the final heat treatment, the microstructure may also contain austenite within the martensite islands, and intermetallic phases. In this work, the hydrogen embrittlement of an experimental 17%Cr steel with complex microstructure was studied by means of slow strain rate tensile tests in 3.5% NaCl solution at room temperature The material was processed by different quenching and tempering heat treatments. Cathodic polarization (−1.2 V_{SCE}) was imposed to introduce hydrogen during the tests. The hydrogen embrittlement was evaluated by the comparison of the tensile properties with and without cathodic charging. The microstructure, fracture surfaces, hardness, and toughness were also analyzed to enhance the discussion about the hydrogen embrittlement of the steel.

Introduction

Supermartensitic stainless steels (SMSS) were developed to allow high corrosion resistance in seawater environments along with high mechanical strength. The first grades derived from conventional 12–13% Cr steels, but contained an extra-low carbon content (<0.03%), and nickel addition to obtain austenite at high temperature, and martensite after cooling. Different Mo additions (up to 2%) were introduced to some steels in order to improve corrosion resistance and mechanical strength. The increase of alpha gene elements had to be counterbalanced by Ni addition. However, the increase of the overall alloying elements may also introduce austenite in the microstructure of some steels. In the heat affected zone (HAZ) and weld metal of welded joints delta ferrite elongated islands can also be formed and retained in room temperature [1].

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More recently, seeking for higher corrosion resistance, steelmakers studied new grades of steels with higher Cr addition (15 and 17%) [2]. The increase of Cr and other alpha gene elements increased the amounts of ferrite phase in the microstructure. Depending on the final heat treatment, the microstructure may also contain austenite within the martensite islands.

A first application of supermartensitic and super-ferritic-martensitic stainless steels is in oil country tubular goods (OCTG) for off-shore oil and gas production [2,3]. The materials can suffer hydrogen attack due to the H_2S and acidity of the fluids transported, corrosion processes, and also cathodic protection.

In this work, a 17%Cr experimental alloy was investigated. Different microstructures were produced by quenching and tempering treatments. The susceptibility to hydrogen embrittlement was evaluated by slow strain rate tensile tests under cathodic hydrogenation charging in 3.5%NaCl solution. The microstructure and mechanical properties were also characterized. The goal of the paper was to determine the best and worse heat treatments for the material studied.

Experimental

The 17%Cr steel studied in this work was issued from a seamless tube with composition shown in Table 1. The pitting resistance equivalent value (PRE = %Cr+3.3(%Mo)+1.65(%W)+16(%N) of the alloy is 27.4, which is similar to AISI 317 L austenitic and lean duplex UNS S32304 steels.

The tubes were cut to produce the specimens for Charpy impact tests and slow strain rate tensile tests (SSRT). After a rough machining the specimens were heat treated, and then final machining operations were performed.

Table 2 shows the specimens identification adopted in this work and the 6 heat treatments tested. All specimens were soaked at 1000 °C for 1 h and oil quenched. Specimen Q was only quenched, specimens QT-300, QT-400, QT-500 and QT-650 were quenched and tempered, and specimen DT1 was quenched and double tempered.

The specimens for Charpy tests were standard size (10 × 10 × 55 mm) with a V-notch. The tests were performed at room temperature and −46 °C in an Universal impact machine with 300 J capacity.

The specimens for SSRT tensile tests had 4.0 mm of diameter and 28.0 mm of gauge length. Before SSRT tests, all specimens were pre-charged for 24 h in a 3.5%NaCl solution at room temperature with a potential of −1.2 V_{SCE}. Thie choice for this pre-charging time was based on previous works on non asutenitic steels [4–6]. The open circuit potential (OCP) of the different specimens were in the −0.3 to −0.2 V_{SCE} range, which means that the cathodic potentials applied were between −0.9 and −1.0 V_{SCE}. The same cathodic potential (−1.2V_{SCE}) and solution (3.5%NaCl) were maintained during the SSRT tests. The strain rate applied was 10^{-6} s^{-1}. The main result of the SSRT test is the σ_N *versus* ε_N curve, were σ_N is the nominal stress and ε_N is the nominal strain. The parameters of hydrogen embrittlement (HE) susceptibility ($K_{EL.}$, K_{ENERGY} and K_{UTS}) were obtained from the comparison of the total elongation (%El.), total area under the curve σ *versus* ε (AREA σxε) and ultimate tensile strength (UTS) in air (inert media) and in solution with hydrogen charging:

$$K_{EL} = \frac{\%El\cdot_H}{\%El_{AIR}} \quad (1)$$

$$K_{ENERGY} = \frac{AREA(\sigma x \varepsilon)_H}{AREA(\sigma x \varepsilon)_{AIR}} \quad (2)$$

$$K_{UTS} = \frac{UTS_H}{UTS_{AIR}} \quad (3)$$

Three tests per heat treatment condition were performed and average values of K_{EL} K_{ENERGY} and K_{UTS} are presented.

Microstructural characterization was performed by X-ray diffraction, optical and scanning electron microscopy (SEM). X-ray diffraction analyses were carried out in the as quenched specimen and also in precipitates from specimens QT-500 and QT-650 extracted by electrolytic method. X-ray diffractograms were obtained with Cu radiation and 0.02° step.

Austenite quantification was performed by magnetic measurements in a Vibrating Sample Magnetometer. The magnetization saturation (m_s) of specimens quenched and tempered in the 300–650 °C range were measured from the magnetization curves obtained at room temperature with maximum field 1.5 T. The austenite volume fraction was calculated by the equations:

$$C_{M/\delta} = \frac{m_S}{m_{S(i)}} \quad (4)$$

$$1 = C_{M/\delta} + C_\gamma \quad (5)$$

Where $C_{M\text{-}\delta}$ and $C\gamma$ are the volume fractions of martensite/ferrite (M/δ) and austenite (γ), m_s is the magnetization saturation of the specimen analyzed, and $m_{s(i)}$ is the intrinsic saturation magnetization of martensite/ferrite. Despite the different chemical composition of ferrite and martensite, it was considered that these two magnetic phases have the same $m_{s(i)}$, which corresponds to the m_s of a specimen without austenite.

After the SSRT, the surface fractures were observed in the scanning electron microscope (SEM).

Results

Fig. 1 shows the microstructure of the specimen Q (quenched), with the martensite and ferrite phases. The ferrite volume fraction determined by quantitative metallography is

Table 1 – Chemical composition of the 17Cr steel studied.

%C	%Cr	%Ni	%Mo	%Mn	%Cu	%Si	W	Nb	N
0.027	16.4	3.62	2.42	0.300	0.941	0.256	1.890	0.100	0.025