Rietveld and Impedance Analysis of Cold and Hot Rolled Duplex and Lean Duplex Steels for Application in Paper and Pulp Industry

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In this study, X-Ray Diffraction (XRD) and Rietveld Refinement were performed to identify and quantify the ferrite and austenite phase of cold and hot rolled duplex stainless steels (UNS S31803) and lean duplex stainless steels (UNS S32304). Electrochemical impedance spectroscopy (EIS) was applied to evaluate the chemical behavior of duplex and lean duplex stainless steels in white, green, and black liquors of paper and pulp industry. Rietveld analysis results showed a higher austenite content than the standard limit for duplex steels in the hot rolled condition. The hot rolling condition plays a major role in improving corrosion resistance in white liquor mainly for the lean duplex steel.

Keywords: Duplex stainless steels, Rietveld Refinement, Electrochemical impedance spectroscopy, Pulp and paper industry

1. Introduction

Duplex stainless steels (DSSs) presents a mixed microstructure of about equal proportions of γ-austenite (face-centered cubic, FCC) and α-ferrite (body-centered cubic, BCC)1, and have existed for more than 70 years. DSSs can be readily hot rolled or cold-rolled. The microstructure depends on the chemical composition of the alloy and thermomechanical treatment2. The alloy elements can act as ferrite stabilizers (chromium, silicon, molybdenum, titanium and niobium) or austenite stabilizers (nickel, carbon, copper, nitrogen and manganese)3. Hence, the chemical composition of austenite and ferrite are different, resulting in different corrosion resistance in a given environment4. Phase fractions, and phase chemical compositions associated to the pitting resistance equivalent number (PREN = wt.%Cr + 3.3 wt.%Mo + 16 wt.%N)4 of DSSs are affected significantly by solution-treatment parameters (temperature and time)4,5. Tan et al.4 reported that the pitting corrosion resistance of a super duplex stainless steel was determined by the pitting resistance equivalent number of weaker phase. On the other hand, improper heat treatment temperature can result in deleterious secondary phases5. Also, plastic deformation may induce austenite to martensite transformation6.

The optimal phase balance in order to improve mechanical properties and corrosion resistance can be a range between 45% and 60% austenite. The microstructure is obtained by simultaneous control of the chemical composition and thermomechanical history (manufacturing process)6,9.

The Rietveld method together with conventional X-ray diffraction (XRD) techniques was used to quantify the microstructural formation such as austenite; ferrite and sigma-phase in the cast super duplex steel after having been submitted to a series of heat-treated weldments10. The Rietveld method is a technique for refinement of crystal structures that uses data from X-ray or neutron diffraction11. This method is based on a mathematical fit for a real standard (experimental data) with a calculated standard (theoretical model) by minimizing the difference between measured and calculated points, through the method of least squares. The calculated standard is obtained from crystallographic databases such as Inorganic Crystal Structure Database (ICSD), Linus Pauling File (LPF), NIST Atomic Spectra Database, and Cambridge Structural Database (CSD). These databases provide the symmetry of the space group, atomic positions, occupation positions, and network parameters. To implement the method, XRD data are used exactly as obtained using the diffractometer, without any treatment11,12.

A major advantage of this method of quantification is that it uses all the points of an XRD spectrum and considers overlapping peaks, which usually make other methods of XRD quantification unfeasible. The parameters related to the diffractogram scanned data, which vary and are calculated during refinement, are as follows: scale factor, baseline...
(background), peak profile, cell parameters, structure factor, displacement and preferred orientation\(^\text{11}\).

In the pulp and paper industry, carbon steel pulp mill equipment such as digesters and storage vessels have shown general corrosion and stress corrosion cracking. The duplex stainless steels have a higher corrosion and stress corrosion cracking resistance in high pH environments at severe conditions of pressure and temperature in relation to carbon steel and austenitic stainless steel. Therefore digesters and storage vessels are being replaced by duplex stainless steels\(^\text{13}\). The lean duplex steel is a cheaper alternative to duplex steels due to the lower contents of nickel and molybdenum\(^\text{14}\).

The Kraft process used in the paper and pulp industry is divided basically in three steps: firstly, the decomposition of the wood is performed by using active cooking chemicals (white liquor) constituted by sodium hydroxide and sodium sulfite\(^\text{15}\). Secondly, the black liquors consisting of residual inorganic chemicals such as sodium sulfite, sulfate, sodium thiosulfate, sodium carbonate, and sodium hydroxide, and organic extractives in wood are formed due to the fragmentation reactions of lignin\(^\text{16}\). Lastly, there is a chemical recovery by combustion of strong black liquor which converts the recovered inorganic chemicals in melting which is dissolved in water, and the green liquor (sodium carbonate (Na\(_2\)CO\(_3\)) and sodium sulfide (Na\(_2\)S))\(^\text{17}\) are produced. Furthermore the white liquor is considered to be the most aggressive of pulping liquors\(^\text{18}\).

The application of the Rietveld method of quantitative analysis for UNS 31803 and UNS 32304 is not found in literature. This study has also investigated the influence of microstructure of cold and hot rolled duplex steels on electrochemical properties of duplex and lean duplex steels in white, green, and black liquors from Pine and southern hardwood mix of an American pulp and paper industry.

### 2. Materials and Methods

#### 2.1. Material and Sample Preparation

The duplex stainless steels, designated by Unified Numbering System (UNS) are UNS S31803 and UNS S32304 (lean Duplex), were supplied by APERAM South America (Brazil) in hot-rolled and cold-rolled conditions. The steels were examined as-received: hot rolled coils, annealed at 1075 ± 25 ºC, with 4 mm of thickness and cold rolled coils, annealed at 1070 ± 25 ºC, with a thickness of 1.8 mm. The steel sheets were cut in dimensions of 1 cm x 1 cm and the lengths were maintained parallel to the rolling direction. The chemical composition of these DSSs used in this work is shown in Table 1.

#### 2.2. Metallographic Analysis

The samples were immersed in a modified Behara reagent, which consists of 80 mL distilled and deionized water, 20 mL of hydrochloric acid (HCl), and 1 g potassium metabisulphite (K\(_2\)S\(_2\)O\(_3\)); 2 g of ammonium difluoride (NH\(_4\)HF) was added to this stock solution just before the etching\(^\text{19}\). The microstructure analyses were carried out using a Leitz Metalloplan optical microscope (OM).

#### 2.3. X-Ray Diffraction (XRD)

XRD analyses were performed to identify the phases present in the samples, using a Shimadzu 7000 equipment under the following operating conditions: CuKa radiation (35 KV/40 mA), goniometer speed of 0.02° per step 2θ, with counting time of 5 seconds per step and collected from 30° to 100° 2θ. Spectra interpretation was carried out by comparing standards contained in the database (PDF 02 International Centre for Diffraction Data, ICDD, 2003).

The Rietveld refinement was performed using the GSAS interface EXPGUI program\(^\text{20}\). The Thompson-Cox-Hastings pseudo-Voigt profile function was used, and the background was adjusted by Chebyshev polynomial. Scale factor, unit cell, background radiation, profile asymmetry, the full width at half height from the instrumental broadening parameters obtained with a standard, atomic position, isotropic atomic displacements and cations occupational factors were refined. The values for R\(_p\) (profile factor), R\(_{\text{Bragg}}\) (Bragg’s index), R\(_{\text{wp}}\) (weighed profile factor), χ\(^2\) and the graphs obtained at every 3 cycles of refinement were measured to check quality of refinement and to better monitoring the results.

#### 2.3. Electrochemical Analysis

Electrochemical impedance spectroscopy (EIS) was performed using a Gamry potentiostat. A three-electrode electrochemical cell was used with a saturated calomel electrode (SCE) as the reference with Luggin capillary and platinum (Pt) wire as a counter electrode, and the duplex stainless steel as the working electrode. The open circuit potential measurements were performed for 1 h or until to obtain a stable open circuit potential value. The amplitude sine-wave of the applied potential is 10 mV from 100 kHz to 5 mHz with 7 points per decade. The measurements were performed in triplicate to ensure the reproducibility.

### Table 1: Chemical composition of the DSSs investigated (wt%).

| Steel  | Rolling | Cr  | Ni  | Mo  | N   | C   | Si  | Mn  | S    |
|--------|---------|-----|-----|-----|-----|-----|-----|-----|------|
| 31803  | Cold    | 22.43 | 5.34 | 2.67 | 0.11 | 0.012 | 0.29 | 1.85 | 0.0004 |
| 31803  | Hot     | 22.45 | 5.31 | 2.63 | 0.11 | 0.013 | 0.38 | 1.81 | 0.0005 |
| 32304  | Cold    | 22.40 | 4.10 | 0.29 | 0.14 | 0.015 | 0.46 | 1.55 | 0.0002 |
| 32304  | Hot     | 22.87 | 4.20 | 0.27 | 0.15 | 0.011 | 0.20 | 1.45 | 0.0004 |
The experimental impedance spectra were fitted using Z-View software version 3.4d. The electrochemical tests were performed in white, green and black liquors, supplied by Mead Westvaco Corporation - MWV. Parameters were white liquor (WL): 88 g/L effective alkali (EA) as Na₂O, green liquor (GL): 117 g/L total titratable alkali (TTA) as Na₂O and weak black liquor (BL): 15% solids. These electrolytes, such as BL, were originated by southern hardwood mix and GL and WL were originated by Pine mix and hardwood mix.

3. Results and Discussion

3.1. Microstructure Characterization

Figure 1 shows typical microstructures found in as-received duplex stainless (a) UNS 31803 steel cold rolled, (b) UNS 31803 steel hot rolled. Figure 2 shows (a) 2304 steel cold rolled and (b) 2304 steel hot rolled, respectively, containing only austenite (lighter) and ferrite phases (dark). Furthermore secondary phases, such as sigma and chi were not observed. The phases observed are elongated in the rolling direction as reported in literature.

Figure 1: Typical microstructures found in the (a) UNS 31803 cold and (b) UNS 31803 hot rolling containing only austenite (clear) and ferrite (dark). Modified Behara Etching.

Figure 2: Typical microstructures found in the (a) 2304 cold and (d) 2304 hot rolling containing only austenite (clear) and ferrite (dark). Modified Behara Etching.

3.2. Quantitative XRD analysis

Austenite and ferrite phases were identified in duplex and lean duplex steels, and secondary phases such as sigma (σ) or chi (χ) phase were not observed in the steel microstructure. Visual evaluation of the graphic setting for both observed and calculated diffractograms is extremely important in assessing quality of refinement as shown in Figures 3 and 4.

According to the XRD results shown in Table 2, the hot rolled steels exhibited a higher difference between the austenite and ferrite content. Consequently amounts of ferrite and austenite formed during hot working or annealing are dependent of temperature. Thus, hot working temperature must be controlled to obtain the balance between the phases in the duplex stainless steel. The calculated diffractogram should overlap the line that represents the observed diffractogram, and the difference line should be equal to a straight line. Quality of the Rietveld refinement is verified through statistical numerical parameters (indicators) which are used during and after refinement in order to verify if it is occurring satisfactorily. The most commonly used statistical parameters for the GSAS program are R_p (profile factor), R_wp (weighed profile factor), R_Bragg’s (Bragg’s index: this index compares the peaks’ integrated intensities) and χ² (Goodness of Fit = GOF = S).

Figure 3: Observed (pluses) and calculated (line) diffraction pattern for the (a) UNS 31803 cold and (b) UNS 31803 hot rolling. The lower curve shows the difference between observed and calculated patterns.

Figure 4: Observed (pluses) and calculated (line) diffraction pattern for the (a) 2304 cold and (b) 2304 hot rolling. The lower curve shows the difference between observed and calculated patterns.

The χ² parameter values found for the UNS 31803 cold and hot rolled steels and the lean duplex cold and hot rolled steels were 3.5%, 3.4%, 3.6% and 2.4%, respectively (Table 2). The χ² parameter value should be equivalent to 1.0% in a perfect refinement. In practice, values below 5.0% refer to an optimized refinement. The R_Bragg’s, R_p and R_wp (Table 2) for the samples are somewhat higher than those normally obtained for refinement of simple phases. This mismatch is due to the displacement of 2θ and peaks’ symmetry.

According to the XRD results shown in Table 2, the hot rolled steels exhibited a higher difference between the austenite and ferrite content. Consequently amounts of ferrite and austenite formed during hot working or annealing are dependent of temperature. Thus, hot working temperature must be controlled to obtain the balance between the phases in the duplex stainless steel. The hot rolled duplex stainless steel showed a high content of austenite than the standard limit for duplex steels which can be associated with a limitation of Rietveld refinement, because this technique is usually applied for powder diffraction data. Data collection might have promoted errors to justify the higher content of austenite than the standard limit of concentration. In the other hand,
Table 2: Rietveld quantitative analysis results for DSSs samples.

| Samples          | Weight percent | $R_{\text{Bragg}}$ | $R_{\text{wp}}$ | $R_{\text{p}}$ | $\chi^2$ |
|------------------|----------------|-------------------|-----------------|---------------|---------|
| 31803 Cold       | 49.9           | 50.1              | 5.9%            | 8.7%          | 8.7%    | 3.5%    |
| 31803 Hot        | 62.7           | 37.3              | 3.0%            | 9.3%          | 6.1%    | 3.4%    |
| 32304 Cold       | 54.3           | 45.7              | 4.0%            | 10.7%         | 8.2%    | 3.6%    |
| 32304 Hot        | 57.2           | 42.8              | 2.1%            | 8.6%          | 6.3%    | 2.4%    |

Table 3 shows the contents of austenite and ferrite obtained using a conventional technique, the Image pro software by optical microscope. In summary the conventional method after etching with a modified Behara reagent was more reliable to quantify the content of austenite in duplex stainless steels. The main weakness of the Rietveld refinement is that it is usually performed for powder measurements and strengths of this technique are the lower time for analysis than the image analysis and the possibility of phase quantification if the lattice parameters are well defined and the diffraction peaks are not overlapping.

Table 3: Austenite and ferrite contents obtained using Image pro software.

| Samples                  | Weight percent | Austenite (γ) | Ferrite (Fe-α) |
|--------------------------|----------------|---------------|----------------|
| UNS 31803 Cold Rolled    | 48             | 52            |
| UNS 31803 Hot Rolled     | 50             | 50            |
| UNS 32304 Cold Rolled    | 47             | 53            |
| UNS 32304 Hot Rolled     | 46             | 54            |

3.3. Electrochemical Measurements

Figures 5, 6 and 7 show the (a) Nyquist and (b) Bode diagrams of cold and hot rolled duplex stainless steels in white, green, and black liquors. Electrochemical impedance spectroscopy was used to evaluate the polarization resistance ($R_p$) of steels in liquors of pulp and paper industry. The EIS spectra were fitted using a simplified Randles circuit as an equivalent electrical circuit (EEC), which consisted of an electrolyte resistance ($R_e$) in series with a parallel combination of a constant phase element and a polarization resistance as shown in Figure 8. Furthermore, Bode diagrams (Figures 5b, 6 b, and 7b) suggest the presence of one time constant. A second maximum identified in Bode curves of hot rolled UNS 31803 steel in white and green liquors occurred at higher frequencies above $10^4$ Hz and was not associated to a corrosive process. Constant phase elements (CPE) are used to describe the frequency dependence of non-ideal capacitive behavior (Table 4). The dimension of CPE is $s^n/\Omega$, while that of an ideal capacitor (C) is $s/\Omega$. When $n$ is equal to 1, a CPE simplifies to a capacitor; when $n$ equals 0 a CPE represents a pure resistor. Low $\chi^2$ values were considered. The average of polarization resistance between the measurements in each solution is shown in Figure 9.
Furthermore, the steels in black liquor showed a similar electrochemical behavior and exhibited the lowest polarization resistance compared to other environments. This effect could be associated to the presence of solid particles in black liquor which contains 15% of solids and, during the test, the solid particles can deposit on the steel surface generating regions of different potentials, and creating anodic and cathodic areas (and electrochemical cells). The deposition of solids on the steel surface should generate aeration cells where the region under the deposit is the anodic region (lower oxygen concentration). The solid particles deposited on the steel surface also can act as a cathodic region, reducing the active area and enhancing corrosion of steel. In addition, an industrial solution was used with a heterogeneous composition dependent of the type of wood and process conditions which contributes to the variability during the measurements. Literature reports that the organic compounds in black liquor can act as steel corrosion inhibitors. However, in this work, in the black liquor, the steels showed the lowest values of corrosion potential and polarization resistance.

The medium of green liquor contains sodium sulfide and sodium carbonate which precipitated on the steel surface. The carbonate deposits generated aeration electrochemical cells and potential gradients on the steel surface. The surface area under the carbonate can act as an anodic region due to the lowest oxygen concentration. The carbonate precipitation can also reduce the anodic region on the steel surface and acts as a cathodic region where the cathodic reaction occurs. This condition contributes to enhance the corrosion of steels. The values of polarization resistance of steels in green liquor were similar, and higher than in black liquor but lower than in white liquor. In summary, particles in solution that allowed deposition on the surface of the working electrode showed more influence on the polarization resistance than microstructure of duplex.

The medium of white liquor was the less aggressive medium for both steels. In medium of white liquor, no precipitation of solids or compounds occurred on the steel surface. The white liquor contains sodium hydroxide, which

| Table 4: Fitting results EIS Experimental Data in green liquor (GL), white liquor (WL), and black liquor (BL). |
|---------------------------------------------------------------|
| DSS               | OCP (mV SCE) | SD (mV SCE) | CPE (F·s⁻¹·cm⁻²) | n    |
|-------------------|-------------|-------------|------------------|------|
| 31803 Cold Rolled GL | -701.4      | 13.5        | 5.93 x 10⁻⁵       | 0.93 |
| 31803 Hot Rolled GL  | -714.9      | 46.3        | 6.79 x 10⁻⁴       | 0.91 |
| 32304 Cold Rolled GL  | -727.0      | 16.3        | 4.89 x 10⁻⁴       | 0.93 |
| 32304 Hot Rolled GL  | -693.0      | 27.9        | 6.73 x 10⁻⁴       | 0.92 |
| 31803 Cold Rolled WL | -345.0      | 27.6        | 2.53 x 10⁻⁴       | 0.95 |
| 31803 Hot Rolled WL  | -339.4      | 8.3         | 2.18 x 10⁻⁴       | 0.95 |
| 2304 Cold Rolled WL  | -345.0      | 23.6        | 2.76 x 10⁻⁴       | 0.95 |
| 2304 Hot Rolled WL  | -338.6      | 7.7         | 2.68 x 10⁻⁴       | 0.94 |
| 31803 Cold Rolled BL  | -673.2      | 21.7        | 5.77 x 10⁻⁴       | 0.93 |
| 31803 Hot Rolled BL  | -678.1      | 19.5        | 4.52 x 10⁻⁴       | 0.90 |
| 32304 Cold Rolled BL  | -675.2      | 19.5        | 6.41 x 10⁻⁴       | 0.92 |
| 32304 Hot Rolled BL  | -658.2      | 50.9        | 6.18 x 10⁻⁴       | 0.91 |
is beneficial to steel passivation, but contains sodium sulfide which is detrimental on the passivation behavior\textsuperscript{18}.

4. Conclusions

Rietveld analysis was not efficient to determine the amount of austenite and ferrite in duplex and lean duplex steels and the influence of the rolling condition on the phase contents was identified.

The medium of white liquor was the less aggressive medium for both steels. The hot rolling condition plays a major role in improving corrosion resistance in white liquor mainly for the lean duplex steel.

The steels showed the lowest values of corrosion potential and polarization resistance in black liquor. Depositions of solids and carbonates on the steel surface in black and green liquors, respectively, contributed to reduce the corrosion resistance of duplex and lean duplex steels.

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