Original Article

Eddy current characterization of cold-worked AISI 321 stainless steel

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\textbf{A B S T R A C T}

Austenitic stainless steels are alloys with desirable features especially in corrosive environments. However, some processes, such as cold-work, can change their original properties owing to the formation of \(\alpha\)-martensite during a strain-induced process. Many techniques have been employed to detect and quantify this metallurgical phase and the non-destructive methods are particularly interesting for quality control in industrial plants. In this work, the use of impedance phase angles from the eddy current testing is proposed to quantify the martensite volume fraction. Methods such as optical microscopy, X-ray diffraction, and vibrating sample magnetometry were also employed to characterize the samples microstructure. Upon magnetic measurements, it was found that the phase angle and martensite volume fraction are related by an exponential fit.© 2018 Brazilian Metallurgical, Materials and Mining Association. Published by Elsevier Editora Ltda. This is an open access article under the CC BY-NC-ND license (http://creativecommons.org/licenses/by-nc-nd/4.0/).

\section{1. Introduction}

Austenitic stainless steels (ASS) have been extensively investigated owing to the strain-induced formation of \(\alpha\)-martensite from austenite (\(\gamma\)) \cite{1,2,3}. The susceptibility of this transformation is strongly affected by the stacking fault energy (SFE), which depends on the chemical composition, temperature, and strain rate \cite{4}. The austenite phase is face-centered cubic (fcc), and the \(\alpha\)′-martensite is body-centered cubic (bcc). When a stacking fault is created, a small region of the fcc structure turns into bcc \(\alpha\)′-martensite. Moreover, it is worth pointing out that \(\gamma\) is paramagnetic and \(\alpha\)′ is ferromagnetic. These crystallographic and magnetic differences enable the use of destructive
and non-destructive methods such as vibrating sample magnetometry (VSM) [5], magnetic induction measurements [6], scanning electron microscopy (SEM) [7], X-ray diffraction (XRD) [8] and Magnetic Barkhausen Noise (MBN) [9], to evaluate and quantify the phenomenon mentioned before. The eddy current testing is another efficient non-destructive technique as demonstrated in some investigations. In one of them, Khan et al. [10] applied the eddy current testing to the AISI 321 and AISI 304 steels. Using one inspection frequency, the approach was able to clearly separate signals on the impedance plane between 20% and 60% reduction, indicating that martensite transformation occurred in that range of strain, which changed the electrical conductivity and magnetic permeability of the alloy. In another, more detailed study, Surkiliabad et al. [11] investigated the AISI 304L steel by changing the inspection frequency from 0.05 to 10 kHz. Their results showed that between 10% and 50% reduction, appreciable signal differences were detected. Furthermore, a correlation between the amplitude of the eddy current signal and the martensite volume fraction was performed, giving an exponential relationship.

The aim of this work is to propose an approach using the eddy current testing to estimate the volume fraction of martensite in a non-destructive way. The main contribution is the use of the phase angle of the impedance to predict the martensite volume fraction. Previous works [6,11,12] established a relationship between the martensite volume fraction and the amplitude of the impedance, which is strongly affected by lift-off variations and surface conditions. Secondary contributions are related to using ferrofluid to reveal the α’-martensite phase and VSM to perform the phase quantification. The use of ferrofluid ensures that only a magnetic phase will be shown, and VSM quantification method is more reliable than X-ray diffraction because the latter suffers from surface conditions and the presence of texture [13].

2. Methods

Nine rectangular strips with dimensions of 80 × 70 × 9.5 mm² were cut from an AISI 321 stainless steel plate with the chemical composition showed in Table 1. The cutting process was performed with the length of the samples parallel to the original rolling direction of the plate, and all samples were solution-treated for 30 min at 1100 °C with no protective atmosphere.

The deformation process was performed in a rolling mill in several steps, each one with a mean reduction of 0.1 mm. Between each step, the samples were cooled in water at 25 °C, and the thickness was measured with a caliper rule at three different points. The true strain (ε₁) and final thickness (t₂) of each sample are shown in Table 2. The overall reduction, until sample #12, was 9.0 mm in 100 rolling steps.

Table 2 - Specimens produced by cold rolling.

<table>
<thead>
<tr>
<th>Sample</th>
<th>True strain ε₁</th>
<th>Final thickness t₂ (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.00</td>
<td>9.5</td>
</tr>
<tr>
<td>2</td>
<td>0.05</td>
<td>9.0</td>
</tr>
<tr>
<td>3</td>
<td>0.10</td>
<td>8.7</td>
</tr>
<tr>
<td>4</td>
<td>0.15</td>
<td>8.2</td>
</tr>
<tr>
<td>5</td>
<td>0.20</td>
<td>7.8</td>
</tr>
<tr>
<td>6</td>
<td>0.27</td>
<td>7.3</td>
</tr>
<tr>
<td>7</td>
<td>0.40</td>
<td>6.4</td>
</tr>
<tr>
<td>8</td>
<td>0.55</td>
<td>5.4</td>
</tr>
<tr>
<td>9</td>
<td>1.00</td>
<td>3.5</td>
</tr>
<tr>
<td>10</td>
<td>1.98</td>
<td>1.3</td>
</tr>
<tr>
<td>11</td>
<td>2.47</td>
<td>0.8</td>
</tr>
<tr>
<td>12</td>
<td>2.94</td>
<td>0.5</td>
</tr>
</tbody>
</table>

After the cold-rolling steps, the samples were cut with a diamond disc at low rpm for light optical microscopy (LOM) measurements. Fig. 1 shows where the samples were taken. To reveal the microstructure, electrolytic etching with 60% nitric acid solution was performed. In addition, a magnetic etch with a ferrofluid was performed to reveal the α’-martensite phase.

The martensite volume fraction was estimated by vibrating sample magnetometry (VSM). A magnetic field from 0 G to 17 kG was applied to the rod-shaped samples of 3.0 mm diameter and 2.6 mm height, cut by electrical discharge machining. Using the method cited by Tavares et al. [14], which uses Eq. (1), the volume fraction of martensite (Cₘ₃) could be estimated.

\[ C_{\alpha'} = \frac{m_s}{m_{si}} \]

where \( m_s \) is the magnetization saturation under each deformation condition and \( m_{si} \) is the intrinsic magnetization.

![Sample obtained for light optical microscopy analyses. The striped face was observed.](image)

Table 1 - Chemical composition of the AISI 321 stainless steel (%wt) – Fe balance.

<table>
<thead>
<tr>
<th>C</th>
<th>Mn</th>
<th>Si</th>
<th>P</th>
<th>S</th>
<th>Cr</th>
<th>Ni</th>
<th>Mo</th>
<th>Al</th>
<th>Cu</th>
<th>N</th>
<th>Ti</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.009</td>
<td>0.82</td>
<td>0.48</td>
<td>0.036</td>
<td>0.002</td>
<td>17.05</td>
<td>9.04</td>
<td>0.038</td>
<td>0.006</td>
<td>0.054</td>
<td>0.009</td>
<td>0.122</td>
</tr>
</tbody>
</table>
saturation of martensite phase. To determine $m_s$, the last points of the magnetization saturation curve are fitted by a linear fit and the value is found by extrapolation when the applied magnetic field is null.

The same process is used to determine $m_{si}$ from a fully martensitic sample. This condition was ensured by XRD with a CoKα source ($\lambda = 1.79$ Å), scan interval from 45° to 105°, and angular pass of 0.01°. Quantification by XRD was also performed for comparison with the VSM results.

To perform the eddy current testing (ECT), an OmniScan MX ECA equipment was used with an absolute probe operating at 8 kHz. Calibration with a standard block was performed to achieve a clear distinction between the magnetic and non-magnetic responses. In this process, phase angle responses ranging between 90° and 180° on the impedance plane and between 180° and 270° were selected for the magnetic materials and nonmagnetic materials, respectively. Fig. 2 shows the experimental setup for the calibration, and all parameters used are listed in Table 3.

3. Results and discussion

Fig. 3 shows the LOM images of the undeformed sample etched with nitric acid and a ferrofluid. It is relevant to emphasize that the use of magnetic etching (ferrofluid) is important in this work to reveal the α′-martensite phase without shear bands, because the classical light optical microscopy images [15] may be indistinguishable as the morphology of α′-martensite and shear bands are similar. Thus, in Fig. 3, it is possible to see both α′-martensite laths and austenitic grains. The martensite phase is the dark one because the ferrofluid particles are attracted to these sites and scatter light. Martensite formation was not expected at this stage, as this sample is an undeformed one. However, this sample was grinded for metallography, which provided enough energy to induce the $\gamma \rightarrow \alpha'$ transformation. This result agrees with the findings reported by others [16].

The microstructure of the specimen deformed at 0.27 of true strain can be seen in Fig. 4. The grain boundaries as well as the dark regions inside the grains indicate martensite formation. Morphologically, this phase consists of parallel laths, as seen in the region defined by the circle in the figure, because martensite nucleation occurs at the shear bands and their intersections, which are formed owing to the plastic strain. The shear bands consist of planar defects that result from a stacking fault on the (111) planes of the austenite phase according to the literature [17,18].

| Table 3 – Parameters used for the calibration of the eddy current equipment. |
|-------------------|---------|
| Probe drive       | 4 V     |
| Gain              | 36 dB   |
| Vertical gain     | 2 dB    |
| Rotation          | 85°     |

Fig. 3 – Undeformed sample etched with nitric acid and a ferrofluid. The dark areas represent the α′-martensite phase.

Fig. 4 – Light optical microscopy image of the sample deformed at 0.27 of true strain. The circular region highlights the martensite formation.
The microstructure of the sample deformed at 0.55 of true strain can be seen in Fig. 5. Comparison with Fig. 4 shows that there are more elongated grains parallel to the rolling direction because of the higher deformation level. Dark areas indicating the presence of the martensite phase are also visible.

The patterns obtained by X-ray diffraction are shown in Fig. 6. Peaks of austenite and martensite are present in the undeformed sample. As discussed above, martensite formation was not expected for the 0.00\(\varepsilon\) sample. This XRD result confirms that grinding the sample surface resulted in sufficient strain to cause the \(\gamma \rightarrow \alpha'\) transformation. The patterns of the other samples show that as the deformation level increases, the peak intensity of \(\alpha'\)-martensite increases whereas that of the \(\gamma\)-austenite peaks decreases. At 1.00\(\varepsilon\), there is no austenite peak, and only the \(\alpha'\)-martensite phase is present.

The quantification of the martensite volume fraction from the VSM results, using Eq. (1), and XRD can be seen in Fig. 7. The intrinsic magnetization saturation of martensite (\(m_0\)) was 133.28 emu/g. Note that the results for each method of quantification are quite different because XRD obtains information from a small layer of the test piece, which is strongly influenced by grinding, polishing, and texture effects [13]. Moreover, according to Ref. [15], the amount of strain-induced martensite decreases from the top to the center of the specimen. As the cylindrical samples for VSM were obtained at the center of the samples, the surface conditions were eliminated and larger amounts of material were taken, yielding results that are more reliable. The values remained different until 0.55\(\varepsilon\), where the steel is virtually saturated with martensite. Beyond 0.55\(\varepsilon\), no more martensite is formed, and both methods give the same results.

Fig. 7 also shows that in the VSM results, at the early stages of strain (0.00\(\varepsilon\) and 0.05\(\varepsilon\)) the martensite fraction is too low, between 0% and 5%. At 0.10\(\varepsilon\), the volume fraction is around 19%. The complete transformation occurs between 0.10\(\varepsilon\) and 0.50\(\varepsilon\), where the specimen is completely martensitic. This result is similar to that from previous works [8,10,11], where both methods showed that the \(\alpha'\)-transformation started at 10% of cold-reduction and ended at 60% of cold-reduction.

Considering the mentioned XRD limitation and the relevant results achieved by VSM in the martensite evaluation, it was defined that the VSM quantification results were more appropriated to be used as reference to correlate the martensite content with the eddy current signal.

Eddy current testing is based on electromagnetic induction where a coil of conductive wire is excited with an alternating electrical current. The coil impedance variation is monitored.
Fig. 8 – Impedance plane in the eddy current testing. The colored arrows represent the phasor for each specimen, and $\theta_1$ and $\theta_2$ are phase angles.

and measured by instrumentation. The conventional analysis of the testing is performed through the impedance plane which is composed of a real part denoted as the electrical resistance ($R$) and an imaginary part denoted as the inductive reactance ($X$).

Fig. 8 shows the impedance plane obtained with the eddy current testing. The blue curves represent the inspection made for each specimen. The beginning and the end of each curve can define a phasor, represented as the colored arrows on the graph. For instance, the green arrow corresponds to the phasor for the sample deformed at 0.20$\varepsilon_t$ (#5). This phasor can be defined by an amplitude and a phase angle. Although in the eddy current testing the phasor amplitude and phase angle are both important parameters, only the phase angle was monitored in this work, as it is less impaired by surface conditions [11]. An example on how to get the phase angle is given by the $\theta_1$ and $\theta_2$ angles in Fig. 8, for specimens deformed at 0.20$\varepsilon_t$ (#5) and 0.00$\varepsilon_t$ (#1), respectively. According to the calibration made before the inspection and described in Section 2, magnetic materials will show a phase angle between 90° and 180°, as $\theta_1$, while nonmagnetic materials will show a phase angle between 180° and 270°, as $\theta_2$.

In Fig. 9, the phase angles for the specimens are plotted as a function of the strain. The dashed line indicates the phase angle that determines whether the material is magnetic or nonmagnetic. The plot shows that samples with 0.00$\varepsilon_t$ (#1) and 0.05$\varepsilon_t$ (#2) exhibit nonmagnetic behavior. Although martensite is present on the surface of the first sample (showed by XRD and LOM), it was not enough to significantly change the magnetic property of the sample. There is around 5% of martensite in the 0.05$\varepsilon_t$ sample, but it also did not result in a change in the magnetic behavior. The change in phase angle for the latter sample is only due to changes in the electrical resistance from the formation of dislocations, stacking faults, voids, and a small amount of $\alpha'$ phase, which act as barriers to the flux of electrons.

The phase angle of the 0.10$\varepsilon_t$ (#3) shows that the sample exhibits a magnetic response. For higher strain levels, the phase angle shows that all the specimens are magnetic, and the phasor approaches the vertical axis ($X$) in the impedance plane, indicating that the material can concentrate more magnetic field as the strain increases. This behavior is only possible because a magnetic phase ($\alpha'$) is being formed, which increases the magnetic permeability of the material. Beyond 0.55$\varepsilon_t$ (#8), the values of the phase angle are indistinct, suggesting that no more martensite transformation occurs. In other words, the martensite volume fraction reached a maximum. Those results are in a good agreement with the VSM quantification showed in Fig. 7 and denote that the martensite formation increases to around 0.5$\varepsilon_t$, and beyond this strain, the alloy is essentially 100% martensitic.

The results from the VSM quantification and the eddy current signals were used to plot the graph in Fig. 10. This plot was made with four points, considering specimens #1, #5, 

Fig. 9 – Phase angle for each strain. Note that as the strain increases, the phase angle decreases.

Fig. 10 – Relationship between the $\alpha'$-martensite fraction and the reciprocal of the phase angle. The martensite volume fraction was measured by VSM.
Table 4 – Coefficients and correlation coefficient.

<table>
<thead>
<tr>
<th></th>
<th>a</th>
<th>b</th>
<th>c</th>
<th>R²</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>0.50797</td>
<td>−0.00189</td>
<td>−5.61458</td>
<td>0.99</td>
</tr>
</tbody>
</table>

#4, #6, and #8, related to strains of 0.00ɛt, 0.15ɛt, 0.27ɛt, and 0.55ɛt, respectively. The graph shows the relationship between the fraction of austenite transformed into martensite (%ATM) and the phase angle from the impedance phase. The horizontal axis is the reciprocal of the phase angle (RPA), used to simplify the graph interpretation. The equation that best fits these points is an exponential function given by Eq. (2), where a, b, and c are constants. The values of the constants as well as the correlation coefficient are given in Table 4.

\[
\begin{align*}
\%\text{ATM} &= a \exp \left( \frac{\text{RPA}}{b} \right) + c \\
0 \leq \%\text{ATM} &\leq 100%
\end{align*}
\]

Eq. (2) allows using the phase angle values to determine the martensite volume fraction. Therefore, it is possible to determine the martensite volume fraction directly from the eddy current, and some corresponding measurements are plotted in Fig. 11. This figure also shows the results obtained by XRD and VSM quantification for comparison with the eddy current results. The ECT measurement results agree very well with the VSM results, which are more reliable than the XRD results, as explained before.

It is worth mentioning that the calibration obtained in this work, which is in good agreement with the VSM results, demonstrates the possibility to use eddy current for the quality control of parts treated thermally or mechanically in real time. This result is particularly interesting as the eddy current approach is a non-destructive and portable method, in contrast to VSM. In this work, the calibration curve was made for the AISI 321 steel with the chemical composition given in Table 1.

4. Conclusions

Magnetic etching brought some benefits to optical microscopy as it allowed revealing only the α'-martensite phase without other features such as shear bands, which can disturb the interpretation. Therefore, samples that were only grinded for light optical microscopy showed the presence of martensite on their surface. This result was confirmed by XRD.

As XRD is highly influenced by surface conditions, quantification by magnetization saturation with VSM was preferred as it provides information from the center of specimens and is therefore more accurate. The VSM results showed that at 0.10ɛt, the alloy is around 19% martensitic and at 0.55ɛt, the alloy essentially only consists of the martensitic phase.

The phase angle of the impedance was an effective approach to separate the different stages of strain based on the different amounts of martensite phase present in the sample. Indistinct signals were obtained for samples that were saturated with martensite, according to the VSM and XRD results.

An exponential fit of the VSM results of martensite quantification and the phase angle impedance was a good approach to yield a calibration curve for the studied alloy. It is an important result that demonstrates the usefulness of eddy current for real-time evaluation in industrial environments.

Conflicts of interest

The author declares no conflicts of interest

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