Quantification of deformation-induced $\alpha'$-martensite content in Fe-19Cr-3Mn-4Ni-0.15C-0.17N austenitic stainless steel by in-situ magnetic measurements

Abstract

The paramagnetism of austenite and the ferromagnetism of $\alpha'$-martensite in Fe-Cr-Mn-Ni steels enable the determination of the deformation-induced $\alpha'$-martensite content with the aid of magnetic based methods. For this purpose, an in-situ magnetic measurement system was devised to determine the kinetics of deformation-induced martensite formation during tensile tests at 0°C of a Fe-19Cr-3Mn-4Ni-0.15C-0.17N (wt.-%) steel. The magnetic measurement system consisted of an electromagnetic field which serves to magnetize the martensitic phase as it formed during tensile loading and a second coil which detected the effective electrical potential difference induced by the magnetization of martensite. To implement the in-situ measurement system, a correlation was necessary between the induced electrical potential difference and the deformation-induced martensite fractions during uniaxial static tensile tests. The correlation was found by performing interrupted tensile tests followed by light optical microscopy (LOM) examinations and magnetic saturation measurements (MSAT) in an ex-situ unit in order to quantify the delta ferrite and martensite fractions. The main advantage of the new configuration compared to a conventional Feritscope is that the information’s collected are representative of the entire volume and the results are insensitive to surface conditions such as roughness and the presence of oxides.

Keywords

Austenitic stainless steel, martensitic transformation, deformation-induced martensite, in-situ magnetic measurement, TRIP effect

1. Introduction

Stress- and strain-induced phase transformations and structural changes can occur during deformation of cast austenitic steels. Examples are $\gamma \rightarrow \alpha'$, $\gamma \rightarrow \varepsilon$, and $\gamma \rightarrow \varepsilon \rightarrow \alpha'$ martensitic transformations and deformation twinning [1–3]. To understand the deformation mechanisms in austenitic steels, it is important to describe the conditions favoring each mechanism for instance the temperature-dependent stress values required to trigger the TRIP or TWIP effects. The deformation induced $\alpha'$-martensite commonly forms at the intersections of shear bands in the austenite which may consist of stacking fault bundles, $\varepsilon$-martensite and mechanical twins [4,5]. The amount of deformation-induced martensite depends on the chemical composition, deformation temperature and the type of loading. Common methods for the investigation of the $\alpha'$-martensite include X-ray diffraction (XRD), electron backscatter diffraction (EBSD) and light optical microscopy (LOM) [6–11]. The ferromagnetism of $\alpha'$-martensite and the paramagnetism of austenite in Fe-Cr-Ni-Mn stainless steels enable the determination
of deformation-induced $\alpha'$-martensite content with methods based on magnetic property measurements. Magnetic saturation and Feritscope measurements are the most common magnetic methods for the quantification of $\alpha'$-martensite.

Magnetic saturation measurements are often performed ex-situ and they have a very high accuracy [12,13]. Feritscope measurements can be performed in-situ during deformation but this method is highly influenced by the geometry and surface conditions such as roughness of specimens [14,15]. The present work reports on a method for in-situ quantification of deformation-induced $\alpha'$-martensite which incorporates the advantages of magnetic saturation and Feritscope measurements.

2. Experimental methods

The steel was melted in a vacuum induction furnace under a nitrogen partial pressure of nearly 450 mbar before being cast into a water-cooled copper mould with a dimension of 230 x 35 x 95 mm$^3$. To avoid pore formation, the nitrogen partial pressure was raised to 1500 mbar during casting. The reached chemical composition of the cast steel is given in Table 1.

<table>
<thead>
<tr>
<th>Alloy</th>
<th>C</th>
<th>N</th>
<th>Cr</th>
<th>Mn</th>
<th>Ni</th>
<th>Si</th>
<th>Fe</th>
</tr>
</thead>
<tbody>
<tr>
<td>19NC17-15</td>
<td>0.154</td>
<td>0.167</td>
<td>18.70</td>
<td>2.94</td>
<td>4.22</td>
<td>0.52</td>
<td>bal.</td>
</tr>
</tbody>
</table>

To eliminate any possible presence of strain-induced martensite formed during machining a solution annealing step was performed after machining of tensile specimens. This heat treatment followed the objectives of dissolving carbides and nitrides likely present in the as-cast microstructure and allowing for the partial homogenization of the steel in the austenitic phase field. The solution annealing treatment consisted of holding the steel at 1150 °C for 30 minutes under an argon atmosphere. The specimens were tensile tested at a strain rate of $4 \times 10^{-4}$ s$^{-1}$ using a Zwick 1476-type universal testing machine. With the aid of a thermal chamber which surrounded the tensile specimen and its fixtures the tensile test temperature of 0 °C was adjusted.

An in-situ magnetic measurement system was devised to determine the $\alpha'$-martensite content formed during tensile tests. The experimental setup is shown in Figure 1. The magnetic measurement system consists of two coils. The first coil serves to generate an electromagnetic field which magnetized the martensite phase as it formed during tensile loading. The magnetization of martensite phase in tensile specimens induced an electrical potential difference (voltage) in the second coil which could be recorded. In order to implement the in-situ measurement system, a correlation had to be established between the measured induced voltage and the $\alpha'$-martensite content formed during tensile loading. The correlation procedure consisted of performing interrupted tensile tests followed by magnetic saturation measurements with an ex-situ unit in order to quantify the $\alpha'$-martensite fractions. For the ex-situ quantification of the ferromagnetic phase content in tensile specimens, a Metis MSAT-type magnetic saturation device equipped with a Lakeshore 480 fluxmeter was used. This equipment enables the measurement of magnetic flux density in specimens magnetized until saturation. The ferromagnetic phase fraction of the steel is then calculated after an internal correction for the chemical composition.
The microstructures were examined by light optical microscopy to determine the delta ferrite fraction and then subtract it from the ferromagnetic phase contents determined by the magnetic saturation results. The subtraction gives the martensite content. The specimens were ground and polished under a stream of hot water to avoid deformation-induced martensite formation during preparation. The specimens were finally electro-polished and etched with nitric acid.

3. Results and Discussion

Figure 2 shows engineering stress-strain curves for specimens deformed at 0 °C up to 800 MPa and until fracture. The small deviations in the stress-strain curves of tensile specimens may be associated with compositional inhomogeneities in the cast steels originating from the segregation during solidification. The coarse grain size of steels means that texture differences among tensile specimens may have played a role too. With the aid of light optical microscopy, an average delta ferrite content of approximately 3 vol.% was measured in the solution-annealed specimens. This value closely reproduces with the average ferromagnetic phase content of undeformed tensile specimens determined with ex-situ magnetic saturation measurements.
Fig. 2: Engineering stress-strain curves of tensile specimens tested at 0 °C up to 800 MPa and until fracture.

Figure 3 shows optical micrographs of tensile specimens tested at 0 °C up to 800 MPa and until fracture. The α'-martensite fractions quantified with ex-situ magnetic saturation measurements are marked on each micrograph.

Fig. 3: Light optical micrographs of tensile specimens tested at 0 °C up to (a) 800 MPa and (b) until fracture.

Using the in-situ magnetic measurement system as shown in Figure 1, the electrical potential difference in the second coil was recorded during tensile tests. As long as the deformation temperature, strain rate, and positioning of coils with respect to the specimen remain constant, a direct relationship is expected between the induced voltage and the deformation-induced martensite content. The correlation between the martensite fraction and the induced-voltage was expressed with a linear relationship and is shown in Figure 4. The linearity of the relationship between the α'-martensite content and the induced voltage as demonstrated in [16] favors the applicability of the devised in-situ magnetic saturation system.
since the proportionality constant at each temperature may be simply obtained from a single tensile test until fracture without the need for numerous interrupted tensile tests.

In order to correlate the stress-strain curve with the deformation-induced $\alpha'$-martensite formation, the evolution of martensite fraction during tensile test as obtained from in-situ magnetic measurements is superimposed on the stress-strain curve of the steel (Figure 5). The evolution of $\alpha'$-martensite fraction with strain resembles the available literature data on the kinetics of deformation-induced martensite formation in metastable austenitic alloys [17–19]. The increase in the work hardening rate upon the deformation-induced formation of $\alpha'$-martensite is known to cause a first inflection point (IP) in the stress-strain curve of metastable austenitic steels [20]. The triggering stress for the martensitic transformation does not match with the first inflection point of the tensile test curve. As marked on Figure 5, the first inflection point during tensile deformation at 0 °C occurs only after the formation of almost 4% martensite in the microstructure. Taking the stress at which almost 1% $\alpha'$-martensite has formed by deformation as the triggering stress for the deformation-induced $\alpha'$-martensite formation ($\sigma_\lambda$), the first inflection point gives a slightly overestimated approximation of the triggering stress.
Fig. 5: The evolution of deformation-induced $\alpha'$-martensite fraction during tensile deformation of the 19NC1715 at 0 °C superimposed on the stress-strain curve.

4. Conclusions

An in-situ experimental setup was devised to quantify the deformation-induced $\alpha'$-martensite content during tensile straining of metastable austenitic steels which are paramagnetic in the austenitic state but transform to ferromagnetic $\alpha'$-martensite upon tensile deformation. This is particularly relevant to the study of the temperature dependence of the TRIP effect. The insensitivity to surface conditions and geometry of tensile specimens of the designed magnetic measurement system are the main advantages to the Feritscope. The conversion of voltage data to the $\alpha'$-martensite was done by assuming a linear relationship between them. The proportionality constant was obtained by ex-situ magnetic saturation measurement of $\alpha'$-martensite after tensile straining.

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References


