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

M. Hauser, M. Wendler, S. Ghosh Chowdhury, A. Weiß & J. Mola

To cite this article: M. Hauser, M. Wendler, S. Ghosh Chowdhury, A. Weiß & J. Mola (2015) Quantification of deformation induced $\alpha'$-martensite in Fe–19Cr–3Mn–4Ni–0.15C–0.15N austenitic steel by in situ magnetic measurements, Materials Science and Technology, 31:12, 1473-1478, DOI: 10.1179/1743284714Y.0000000731

To link to this article: https://doi.org/10.1179/1743284714Y.0000000731

Published online: 08 Dec 2014.
Quantification of deformation induced α’-martensite in Fe–19Cr–3Mn–4Ni–0.15C–0.15N austenitic steel by in situ magnetic measurements

M. Hauser*1, M. Wendler1, S. Ghosh Chowdhury2, A. Weiß1 and J. Mola1

An in situ magnetic device was employed to quantify the deformation induced martensite in a Fe–19Cr–3Mn–4Ni–0.15C–0.15N (wt-%) steel during tensile testing in the temperature range of −40 to 22°C. The new device consists of an electromagnetic field which serves to magnetise the martensite phase as it forms during tensile loading and a second coil to detect the effective electrical potential difference induced by the magnetisation of tensile specimens. 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 procedure was found to require only the quantification of deformation induced martensite content in a tensile specimen strained until fracture using an ex situ magnetic saturation unit.

Keywords: Austenitic stainless steel, Martensitic transformation, Deformation induced martensite, in situ magnetic measurement, TRIP effect

Introduction

Metastable austenitic Fe–Cr–Mn–Ni steels undergo martensitic transformation during uniaxial static tensile loading which can lead to the transformation induced plasticity (TRIP) effect.1–3 Mechanisms for the α’-martensite formation have been examined extensively and well reported in the literature.4–9 The α’-martensite commonly forms at the intersections of shear bands in the austenite which may consist of stacking fault bundles, ε-martensite, and mechanical twins.9,10 The amount of deformation induced martensite depends on the chemical composition, deformation temperature, grain size and the type of loading. Common characterisation methods employed for the investigation of the α’-martensite are transmission electron microscopy (TEM), X-ray diffraction, electron backscatter diffraction (EBSD), and optical microscopy.11–17 The paramagnetism of the austenite phase and the ferromagnetism of the α’-martensite phase in Fe–Cr–Mn–Ni metastable austenitic steels enable the determination of deformation-induced α’-martensite content with methods based on magnetic property measurements. The most common magnetic methods for the quantification of α’-martensite are magnetic saturation and Feritscope measurements.18,19 Magnetic saturation measurements are performed ex situ and require interruption of deformation in order to determine the transformed fractions. Feritscope measurements, on the other hand, can be performed in situ, for instance during tensile deformation. Nevertheless, Feritscope measurements are highly sensitive to surface conditions such as roughness and surface oxides on the sample and its geometry.20,21

The present work reports on a method of in situ quantification of deformation induced α’-martensite which shares the advantages of magnetic saturation and Feritscope measurements. The system devised enables the reliable in situ quantification of the martensite formation rate and the precise determination of the stress required to trigger the deformation induced martensite formation which are relevant to the analysis of strengthening and plasticity effects caused by the deformation induced α’-martensite formation.

Experimental methods

The steel used for investigation was melted in a vacuum induction furnace under a nitrogen partial pressure of 450 mbar before being cast into a water-cooled copper mould with a dimension of 230×63×695 mm. The chemical composition of the cast steel is given in Table 1. To avoid pore formation in ingots, the nitrogen partial pressure was raised to 1500 mbar during casting.

To ensure the absence of machining induced martensite near the surface of tensile specimens, the solution heat treatment was performed after machining of tensile specimens. The solution heat treatment aimed at the dissolution of carbides and nitrides likely existing in
the as cast microstructure. It also led to the partial homogenisation of the steel in the austenitic phase field. The solution heat treatment consisted of holding the steel at 1150°C for 30 min under an argon atmosphere.

Using a Zwick 1476-type universal testing machine, tensile specimens were tested at an initial strain rate of $4 \times 10^{-4}$ s$^{-1}$. With the aid of a thermal chamber which surrounded the tensile specimen and the sample holding jaws, different temperatures in the range of $-40$ to $22$°C could be adjusted. Interrupted tensile tests at various temperatures were performed up to stress levels of 700, 900 and 1100 MPa.

An in situ magnetic measurement system was devised to determine the $\alpha'$-martensite content formed during tensile tests. The experimental setup is shown in Fig. 1. The magnetic measurement system consisted of two coils. The first coil served to generate an electromagnetic field which magnetised the martensite phase as it formed during tensile loading. The magnetisation of martensite phase in tensile specimens induced an electrical potential difference (voltage) in the second coil which could be recorded. To avoid interactions between the magnetic field and the surrounding components in the thermal chamber, different temperatures in the range of $-40$ to $22$°C could be adjusted. Interrupted tensile tests at various temperatures were performed up to stress levels of 700, 900 and 1100 MPa.

Table 1 Chemical composition of investigated cast steel in wt-%

<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>

Results and discussion

Figure 2 shows engineering stress–strain curves for specimens cut from tensile specimens after saturation magnetisation. The ferromagnetic phase fraction was calculated after an internal correction for the chemical composition. The correction took into account the influence of alloying elements on the magnetic moment of pure iron. The measurement accuracy with this method is within $\pm 1\%$.

For comparison with the $\alpha'$-martensite contents quantified by ex situ magnetic saturation device, X-ray diffraction (XRD) quantification of $\alpha'$-martensite was performed. XRD measurements with Co $K_{\alpha}$ radiation were performed in a Seifert-FPM-type diffractometer equipped with a Meteor detector.

Magnetic saturation measurements prior to tensile tests enabled the quantification of delta ferrite contents retained after solution annealing. The delta ferrite content in solution annealed tensile specimens was also determined by quantitative metallography. These measurements closely reproduced the delta ferrite contents based on ex situ magnetic saturation measurements.

The specimens for light optical microscope (LOM) examinations were ground under a stream of hot water to minimise the deformation induced formation of martensite. The specimens were finally electro-polished and etched with nitric acid.
Ex situ magnetic saturation measurements are marked on each micrograph. As the maximum stress increases, the content of deformation induced α'-martensite increases too.

Using the in situ magnetic measurement system shown in Fig. 1, the electrical potential difference in the second coil was recorded during tensile tests. The induced voltage during tensile tests at various temperatures is shown as a function of strain in Fig. 4. 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 influence of delta ferrite in tensile specimens was eliminated by subtracting the voltage before loading from the values measured during tensile tests. This ensured that the voltage was zero at the beginning of tensile tests.

In order to convert the induced voltage obtained in situ as shown in Fig. 4 to the α'-martensite fraction, ex situ magnetic saturation measurements of specimens tensile tested up to various stress levels were performed. Subsequently, for each deformation temperature, the martensite content was correlated with the induced voltage. This correlation is shown in Fig. 5. The correlation between the martensite fraction and the induced voltage at each temperature can be expressed with a linear relationship the slope of which (proportionality constant) depends on the deformation temperature. The temperature dependence of the above correlation is due to the temperature dependence of the magnetic moment of the experimental steel in the α'-martensitic state. The linearity of the relationship between the α'-martensite content and the induced voltage 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 without the need for interrupted tensile tests. For instance, the same specimen used for the in-situ magnetic measurements until fracture can be used for the determination of deformation-induced α'-martensite content using an ex situ magnetic saturation device.
In order to compare the martensite fractions quantified with our magnetic device with an alternative non-magnetic method, XRD of specimens tensile strained to various stress levels at −40°C was performed. The XRD traces are shown in the Fig. 6. The α′-martensite contents quantified from the XRD traces are summarised in Table 2 and compared with the fractions based on ex situ magnetic saturation measurements. In contrast to the ex situ magnetic saturation results which show a continuous increase in the fraction of α′-martensite, the XRD based fractions of α′-martensite do not produce a meaningful trend. For instance, the martensite fraction after straining up to 900 MPa was quantified to be larger than that after tensile testing until fracture. Because of the effort made to prepare the surface of specimens under identical conditions, the unclear trend might have arisen because of the coarse grain size of cast specimens and the problem of preferred orientation of grains in the examined area. The preferred orientation can be understood by considering the XRD trace of the specimen tested up to a stress level of 700 MPa where the (002)c peak appearing at about 60° has a higher intensity than the (111)c peak appearing at about 51°.

The orientation dependence of austenite to α′-martensite transformation during tensile deformation has been well documented in the literature for various materials including austenitic stainless steels. The preferred orientation of grains in the limited area of exposure during XRD measurements and the orientation dependence of martensitic transformation are thought to be responsible for the difference in the α′-martensite fractions based on XRD and ex situ magnetic saturation measurements. Owing to the collection of information from the bulk, the values quantified by the latter technique are less influenced by the preferred orientation of grains in coarse grained as cast specimens. This justifies the use of ex situ magnetic saturation measurement results to establish the correlation between α′-martensite content and the voltage induced in the in situ magnetic device (Fig. 5).

In order to correlate the stress–strain curve with the deformation induced α′-martensite fraction, the evolution of martensite fraction during tensile test at −40°C obtained from in situ magnetic measurements is superimposed on the stress-strain curve (Fig. 7). The evolution of α′-martensite fraction with strain resembles the available literature data on the kinetics of deformation-induced martensite formation in metastable austenitic alloys. The increase in the work hardening rate caused by the deformation induced formation of α′-martensite is known to cause a first inflection point in the stress–strain curve of metastable austenitic steels. Taking the stress at which almost 1% α′-martensite has formed by deformation as the triggering stress for the deformation-induced α′-martensite formation (σA), it does not match the first inflection point (first IP) of the stress–strain curve. As marked in Fig. 6, the first inflection point during tensile deformation at −40°C occurs only after the formation of almost 3 to 5% martensite in the microstructure. In other words, the first inflection point gives a slightly overestimated approximation of the triggering stress. Between the first and second inflection points, the martensite formation rate remains almost constant.

Similar type of in situ magnetic measurements to study the evolution of martensite fraction during tensile deformation have been carried out by Maxwell et al.

<table>
<thead>
<tr>
<th>Specimen</th>
<th>MSAT</th>
<th>XRD</th>
</tr>
</thead>
<tbody>
<tr>
<td>700 MPa</td>
<td>22</td>
<td>53</td>
</tr>
<tr>
<td>900 MPa</td>
<td>40</td>
<td>68</td>
</tr>
<tr>
<td>1100 MPa</td>
<td>52</td>
<td>52</td>
</tr>
<tr>
<td>Fracture</td>
<td>56</td>
<td>65</td>
</tr>
</tbody>
</table>

The orientation dependence of austenite to α′-martensite transformation during tensile deformation has been well documented in the literature for various materials including austenitic stainless steels. The preferred orientation of grains in the limited area of exposure during XRD measurements and the orientation dependence of martensitic transformation are thought to be responsible for the difference in the α′-martensite fractions based on XRD and ex situ magnetic saturation measurements. Owing to the collection of information from the bulk, the values quantified by the latter technique are less influenced by the preferred orientation of grains in coarse grained as cast specimens. This justifies the use of ex situ magnetic saturation measurement results to establish the correlation between α′-martensite content and the voltage induced in the in situ magnetic device (Fig. 5).
The correlation between the martensite content and the flow curves for the high Ni steels used in their experiments was however unclear. This is most likely caused by the ferromagnetism of the austenite phase in the presence of high Ni contents. The Curie temperature for a binary Fe–30Ni alloy is for instance of the order of 30 to 47°C.30-32 Determination of the evolution of deformation induced α'-martensite fraction at temperatures just below the Curie temperature, where the temperature dependence of magnetic properties is large, then requires the knowledge of the magnetic moment of austenite in such alloys. This was confirmed when a Fe–29.5Ni binary alloy was tested with the magnetic measurement system devised in the present work. As demonstrated in Fig. 8, the voltage in the second coil decreases as deformation induced α'-martensite forms during tensile testing at –45°C of a Fe–29.5Ni binary alloy. The in situ quantification of the α'-martensite fraction during tensile testing of metastable austenitic alloys is therefore best suited for application to steels in which the austenite phase remains paramagnetic at deformation temperatures of interest.

Conclusions

The present paper describes an in situ experimental setup to quantify the deformation-induced α'-martensite content during tensile straining of metastable austenitic steels which are paramagnetic in the austenitic state but transform to ferromagnetic α'-martensite upon tensile deformation. This becomes particularly important in the study of the temperature dependence of the TRIP effect. The low sensitivity to surface conditions, geometry, and preferred orientation of coarse-grained tensile specimens are the main advantages of the designed magnetic measurement system over Fertiscope and XRD methods. Interrupted tensile tests implied a linear relationship between the induced voltage and the α'-martensite content determined by ex situ magnetic saturation measurements. This simplifies the conversion of voltage data to the α'-martensite fraction since the proportionality constant for each temperature may be obtained from ex situ magnetic saturation measurement of the same specimen used for in situ magnetic measurement after deformation until fracture.

Acknowledgements

The authors would like to thank all of the staff involved in the Collaborative Research Centre 799 and the German Research Foundation (DFG) for the financial support of this work. Thanks are also due to Prof. Ferenc Tranta (University of Miskolc, Hungary) whose prototype magnetic measurement device provided inspiration for the presented magnetic measurement system.

References


21. R. Magnabosco: ‘Comparative study of ferrite quantification methods applied to duplex stainless steels’, Proc. 7th European Stainless Steel Conf., Como, Italy, September 2011, AIM.


