Martensitic Transformation in Austenitic Stainless Steels

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“The saddest aspect of life right now is that science gathers knowledge faster than society gathers wisdom.”

-Isaac Asimov
ABSTRACT

Martensitic transformation is very important in austenitic stainless steels where the transformation induced plasticity phenomenon provides a combination of good mechanical properties, such as formability and strength. However, the difficulty of predicting the material behaviour is one of the major drawbacks of these steels. In order to model this behaviour it is of great importance to be able to characterize the morphology, crystallography and the amount of different types of martensite. The morphology and crystallography of thermal and deformation induced lath martensite in stainless steels were re-examined by means of optical microscopy and electron backscatter diffraction (EBSD) technique.

The experiments were performed on AISI301, 304 and 204Cu austenitic stainless steels. Plastic deformation was carried out by means of uniaxial tensile tests at the strain rate of $1 \times 10^{-3} \text{s}^{-1}$ to produce strain induced $\alpha'$-martensite at a temperature ranging from 0 to 60ºC. An in-situ measurement of the martensite content was performed during the tensile testing using a Ferritescope to provide the necessary experimental values for modelling.

Optical microscopy revealed the morphology of the strain induced $\alpha'$-martensite as sets of thin parallel needles that go through the parent austenite grain and stop at the grain or annealing twin boundaries. Large amount of $\alpha'$-martensite could be seen at the intersection of shear bands. However, considerable amount of $\alpha'$-martensite was also observed when only one set of bands is activated. EBSD was successfully used to analyze the morphology and crystallography of martensite. The $\alpha'$-martensite maintained the Kurdjumov-Sachs (K-S) orientation relationship with the austenite phase. Although all six possible variants did not appear within a single packet, one or two variants were often favoured out of six related to the specific $\{111\}$ plane. The misorientations between the neighbouring variants were mainly $<111> 60^\circ$ or $<110> 49.5^\circ$.

Keywords: Stainless Steel, SEM, EBSD, $\alpha'$-martensite, Ferritescope
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Chapter 1

INTRODUCTION

Stainless steels have been increasingly used during the past decades. It is not clear when and where the stainless steels were first discovered, but initial attempts are believed to date back to 1821 when a Frenchman named Berthier tried to alloy iron with chromium \[1\]. After development of new methods to produce low carbon steel with chromium by Goldschmidt in 1897 and direct-arc electric furnace technique by Heroult in 1899, French, German, English and later United States metallurgists began studying the low-carbon, chromium-containing ferrous alloys. The early studies were published between 1904 and 1909 by the French metallurgists L. B. Gulliet and A. M. Portevin on martensitic and ferritic steels; containing 0.12 to 1.0% C. Gulliet and the German metallurgist, W. Giesen, also published several studies on Fe-Cr-Ni austenitic alloys \[2\]. At about the same time in Germany, P. Monnartz began to study the role of carbon content on corrosion resistance of iron-chromium steels and revealed that the “stainless” property of these materials was a result of passivity phenomenon \[3\]. The works of H. Brearley in England (martensitic stainless steels), F. Becket and C. Dantsizen in the U.S. (ferritic stainless steels) and E. Maurer and B. Strauss in Germany (austenitic stainless steels) urged the commercial applicability of stainless steels between 1910 and 1915. The development of precipitation-hardenable stainless steels in 1940s was led by United States Steel Co. as a consequence of further research on the role of composition and heat treatment in stainless steels. The raw material shortage, particularly nickel, during World War II and also Cold War politics gave rise to many attempts to replace some or all of nickel with manganese and this led to development of high-manganese austenitic stainless steels. Duplex stainless steels containing both austenite and ferrite were first discovered in the 1930s. The industrial interest in duplex stainless was renewed during 1960s when the results of studies on the superplasticity of fine-grain austenitic-ferritic alloys were published \[4\].

The adoption of new refining techniques in early 1970s revolutionized stainless steels production in which development of argon-oxygen-decarburization (AOD) process was the most significant one. The first commercial AOD vessel for refining
stainless steels came into operation in 1968 \[5\]. The AOD process permitted the quick decarburization without excessive chromium loss to the slag due to the oxidation. The AOD process and related processes also enhanced desulphurization and provided better chemistry control. Nowadays a broad variety of new alloys with vastly improved corrosion resistance and mechanical properties are being produced by means of subsequent developments in smelting and refining techniques, such as vacuum oxygen decarburization (VOD), vacuum induction melting, vacuum arc melting, electroslag remelting, and electron beam melting/remelting.

Stainless steels are usually classified into five families. The first four are named after their characteristic crystallographic microstructures at room temperature which are ferritic, martensitic, austenitic and duplex. The fifth family, the precipitation-hardenable alloys, is named after the heat treatment used rather than the microstructure.

Austenitic stainless steels are well suited to service at either elevated or cryogenic temperatures. The combination of good corrosion resistance, weldability and mechanical properties such as ductility, formability, and toughness even at cryogenic temperatures, in addition to significant hardenability by cold work, makes them the most commonly used grade of stainless steels. They form the largest group of stainless steels in terms of application and variety of alloys.

Austenitic grades contain 16 to 28% ferrite-stabilizing chromium. Sufficient concentration of austenite-stabilizing elements such as nickel, manganese, nitrogen and carbon expands the gamma phase field and preserve the nonmagnetic face-centred-cubic (FCC) austenite phase to room temperature. As stated before, this stable or metastable austenite can be hardened by cold working. However, the deformed microstructures can be quite complex involving deformation induced martensite structure, slip planes and potential mechanical twinning in highly alloyed grades.

The martensitic transformation is believed to be a displacive transformation without diffusion of atoms. Martensite transformation is very important in steels because the structure has the highest strength for a given chemical composition \[6-8\]. The early works of Angel \[9\] showed that that plastic straining induces martensitic transformation in TRIP steels. The transformation induced plasticity phenomenon (TRIP effect) allows combining very good mechanical properties such as high formability and strength. The development of steels with a good compromise between ductility and toughness is very attractive to the industry especially for automotive and home appliances. However, the difficulty of predicting the deformation processes such as sheet forming is one of the major drawbacks of these steels. The wide spreading research in the field of martensite transformation emphasizes the intricacy and importance of this multilateral process which implicates appropriateness of further studies in order to illuminate new aspects of
Chapter 1

its nature and possibility of modelling the process with respect to affecting parameters.

In order to model the martensitic transformation in stainless steels it is of great importance to be able to characterize the different types of martensite and the amount of each and understand how they are affected by alloying elements, deformation and temperature. The aim of the present study was to re-examine the morphology and crystallography of spontaneous (thermal) and deformation induced lath martensite in stainless steels by means of optical microscopy (OM) and electron backscatter diffraction (EBSD) technique.

Various uniaxial tensile tests were performed on AISI301, 304 and 204Cu austenitic stainless steel grades to produce strain induced α'-martensite and the effect of temperature and chemical composition was investigated. The tests were performed at the strain rate of $1 \times 10^{-3} \text{s}^{-1}$ at a temperature ranging from 0 to 60ºC. An in-situ measurement of the martensite content was performed during the tensile testing using a Ferritescope to provide the necessary experimental values for comparison between the ferritescope readings and actual martensite content. The experimental data was used as input to Olson and Cohen strain induced martensite model.
Chapter 2

GENERAL ASPECTS

2.1 Crystallography of Martensitic Transformation

Two types of martensite can form in stainless steels: ferromagnetic, body-centred cubic (BCC) \(\alpha'\)-martensite and non-ferromagnetic, hexagonal closed-packed (HCP) \(\varepsilon\)-martensite. \(\alpha'\)-martensite in steels generally has a BCC or body-centred tetragonal (BCT) structure but in case of stainless steels due to the relatively low content of interstitials, it is normally referred to as BCC rather than BCT.

In the FCC crystal structure an intrinsic stacking fault changes the regular staking sequence of the \{111\} planes from ABCABCABC to, for instance, ABCACABCA. Therefore even a single stacking fault has a thin layer of HCP phase (CACA) which can be regarded as a nucleus of \(\varepsilon\)-martensite \[10\]. The perfect \(\varepsilon\)-martensite grows if intrinsic stacking faults overlap regularly on every second \{111\} plane \[11-13\]. If two intrinsic stacking fault overlap on the successive \{111\} planes, an extrinsic stacking fault with ABCACBCAB sequence will form which can be regarded as a twin nucleus. The growth of the mechanical twin takes place by proceeding the overlapping of intrinsic stacking faults on successive \{111\}. Irregular overlapping causes a stacking fault bundle which may be regarded as either faulted \(\varepsilon\)-martensite or faulted austenite. Therefore it is difficult to distinguish between microstructure features originating from the formation and overlapping of stacking faults in austenitic stainless steels and a collective term “shear bands” is often used for the planar defects including bundles of overlapping stacking faults, \(\varepsilon\)-martensite and mechanical twins.

The crystallography of the martensite transformation is usually described by the habit plane and orientation relationships between the martensite and the parent austenite phase. The habit plane for martensite changes with chemical composition; nevertheless, steels of vastly different composition can have identical habit plan and other crystallographic characteristics. It has been reported that steels with low
amount of carbon have a \{557\} habit plane, which is similar to a \{111\} plane; whereas high-carbon steels have habit planes of \{259\} or \{225\}. The diffusionless transformation involves the coordinated movement of atoms. Therefore, the parent and product lattices will be closely related. The orientation relationship describes an existing parallelism between planes in the parent and the product phase\(^{[14]}\).

The orientation relationship between the austenitic matrix phase and \(\varepsilon\)-martensite is often reported as: \(^{[15]}\)

\[
\begin{align*}
\{111\}_\gamma && \{0001\}_\varepsilon \\
\langle 1\bar{1}0 \rangle_\gamma && \langle 1\bar{2}10 \rangle_\varepsilon
\end{align*}
\]

(1)

Whereas the orientation relationship for \(\alpha'\)-martensite and parent phase is mostly reported to follow the Kurdjumov-Sachs (K-S) criteria: \(^{[16]}\)

\[
\begin{align*}
\{111\}_\gamma && \{011\}_\alpha' \\
\langle 1\bar{1}0 \rangle_\gamma && \langle 1\bar{1}1 \rangle_\alpha'
\end{align*}
\]

(2)

However broad distribution around the ideal orientations of expected variants or deviation towards Nishayama-Wassermann (N-W) is observed in low carbon and alloy steels \(^{[17-19]}\).

According to K-S orientation relationship, due to the symmetry in cubic systems, 24 equivalent crystallographic variants in \(\alpha'\)-martensite can be developed from an austenite single crystal. Table 2.1 shows 24 orientation variants which satisfy the K-S relationship and the 10 different misorientation angles that can be formed between the variants \(^{[17]}\).
Table 2.1—The 24 K-S orientation variants and misorientation angle from V1.

<table>
<thead>
<tr>
<th>Variant no.</th>
<th>Plane parallel</th>
<th>Direction parallel</th>
<th>Misorientation angle from V1 (°)</th>
<th>CSL*</th>
</tr>
</thead>
<tbody>
<tr>
<td>V1</td>
<td>(111)γ∥(011)α’</td>
<td>[101]γ∥[111]α’</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>V2</td>
<td>[101]γ∥[111]α’</td>
<td>60.00</td>
<td>Σ3</td>
<td></td>
</tr>
<tr>
<td>V3</td>
<td>[011]γ∥[111]α’</td>
<td>60.00</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>V4</td>
<td>[011]γ∥[111]α’</td>
<td>10.53</td>
<td>Σ1</td>
<td></td>
</tr>
<tr>
<td>V5</td>
<td>[110]γ∥[111]α’</td>
<td>60.00</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>V6</td>
<td>[110]γ∥[111]α’</td>
<td>49.47</td>
<td>Σ11</td>
<td></td>
</tr>
<tr>
<td>V7</td>
<td>(111)γ∥(011)α’</td>
<td>[101]γ∥[111]α’</td>
<td>49.47</td>
<td>Σ19b</td>
</tr>
<tr>
<td>V8</td>
<td>[101]γ∥[111]α’</td>
<td>10.53</td>
<td>Σ1</td>
<td></td>
</tr>
<tr>
<td>V9</td>
<td>[110]γ∥[111]α’</td>
<td>50.51</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>V10</td>
<td>[110]γ∥[111]α’</td>
<td>50.51</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>V11</td>
<td>[011]γ∥[111]α’</td>
<td>14.88</td>
<td>Σ1</td>
<td></td>
</tr>
<tr>
<td>V12</td>
<td>[011]γ∥[111]α’</td>
<td>57.21</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>V13</td>
<td>(111)γ∥(011)α’</td>
<td>[011]γ∥[111]α’</td>
<td>14.88</td>
<td>Σ1</td>
</tr>
<tr>
<td>V14</td>
<td>[011]γ∥[111]α’</td>
<td>50.51</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>V15</td>
<td>[101]γ∥[111]α’</td>
<td>57.21</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>V16</td>
<td>[101]γ∥[111]α’</td>
<td>20.61</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>V17</td>
<td>[110]γ∥[111]α’</td>
<td>51.73</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>V18</td>
<td>[110]γ∥[111]α’</td>
<td>47.11</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>V19</td>
<td>(111)γ∥(011)α’</td>
<td>[110]γ∥[111]α’</td>
<td>50.51</td>
<td>-</td>
</tr>
<tr>
<td>V20</td>
<td>[110]γ∥[111]α’</td>
<td>57.21</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>V21</td>
<td>[011]γ∥[111]α’</td>
<td>20.61</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>V22</td>
<td>[011]γ∥[111]α’</td>
<td>47.11</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>V23</td>
<td>[101]γ∥[111]α’</td>
<td>57.21</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>V24</td>
<td>[101]γ∥[111]α’</td>
<td>21.06</td>
<td>-</td>
<td></td>
</tr>
</tbody>
</table>

* Coincidence site lattice is indicated when Brandon’s criterion [20] is satisfied.

2.2 Morphology

The mechanical properties and formability of austenitic stainless steels is remarkably affected by martensitic transformation and its morphology during straining [21, 22]. A variety of martensite morphologies has been observed in ferrous alloys including lath, butterfly, lenticular and thin plate [22, 23], but in case of stainless steels, due to low carbon content, lath martensite appears more frequently. The morphology and crystallography of the lath martensite has been greatly investigated by optical microscopy and transmission electron microscopy (TEM) [24-28].
Although the small size of martensite laths makes the clear observation of individual laths in optical micrographs very difficult, the lath martensite shows a characteristic microstructure at a coarse scale, since they have a tendency to align themselves parallel to one another in the large area of the parent grain.

The martensite lath is a single crystal of martensite with a high density of lattice defects. General view is that an austenite grain breaks down to several packets (the group of laths with the same habit plane) each containing parallel blocks (the group of narrow, ruler shaped [27] laths of the same orientation or variant) [29, 30]. Figure 2.1 illustrates a typical lath martensite structure with laths, blocks and packets within a single grain of austenite [17].

According to Table 2.1, most of the misorientations at the packet and block boundaries that satisfy the K-S orientation relationship exceed 15°, which is generally the criterion between the low and high angle boundaries. Even the minimum misorientation, 10.53°, is relatively large, whereas the misorientations along the individual laths are low angle boundaries within a range of few degrees.

The laths in a single packet have similar habit plane and different crystallographic orientations. Orientation relationship between an identical (111) austenite plane and six K-S Variants (V1-V6) in a given packet is showed in Figure 2.2.
K-S orientation relationship gives four separate groups of $\alpha'$-martensite variant pairs (in a given packet) with different misorientation angles (Table 2.2), whereas the variants with the same habit plane in N-W OR are related by $[011]/60^\circ$ rotation.

**Table 2.2-Misorientation between variant pairs in a given packet [19].**

<table>
<thead>
<tr>
<th>Group</th>
<th>Variant pair</th>
<th>Misorientation axis/angle</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>V1-V4, V3-V6, V5-V2</td>
<td>$[011]/10.5^\circ$</td>
</tr>
<tr>
<td>2</td>
<td>V1-V6, V3-V2, V5-V4</td>
<td>$[011]/49.5^\circ$</td>
</tr>
<tr>
<td>3</td>
<td>V1-V3, V3-V5, V5-V1, V2-V4, V4-V6, V6-V2</td>
<td>$[011]/60.0^\circ$</td>
</tr>
<tr>
<td>4</td>
<td>V1-V2, V3-V4, V5-V6</td>
<td>$[111]/60.0^\circ$ twin related $\equiv[011]/70.5^\circ (\Sigma 3)$</td>
</tr>
</tbody>
</table>
2.3 Thermodynamics

Several attempts have been made to associate the $M_s$ temperature to the chemical composition by empirical equations. The Eichelmann and Hull’s equation is one of the most commonly used: \[^{[31]}\]

\[
M_s(°C) = 1302 - 42(%Cr) - 61(%Ni) - 33(%Mn) - 28(%Si) - 1667(%[C+N]) \quad \ldots \ldots
\tag{3}
\]

As this equations shows, many stable and metastable austenitic steels will partially transform to $\alpha'$-martensite when cooled to a cryogenic temperature. The impoverishment of chromium, carbon and other alloying elements close to the grain boundaries due to precipitation of $M_{23}C_6$ increases the $M_s$ temperature. Consequently, the sensitization enhances the ability of $\alpha'$-martensite formation adjacent to grain boundaries during cooling \[^{[32]}\].

The martensitic transformation in stable austenitic grades happens only during cooling to cryogenic temperatures whereas the strain induced martensite is more frequently seen at room temperature in less stable grades. This transformation and the amount of $\alpha'$ and $\varepsilon$-martensite is affected by e.g. temperature, chemical composition, stacking fault energy (SFE) of austenite, deformation degree, deformation rate, and also stress state during deformation \[^{[33]}\].

Thermodynamics of strain induced martensite are illustrated in Figure 2.3 \[^{[34]}\], which schematically shows that if adequate mechanical driving force ($U'$) is applied on the austenite, the martensitic transformation can take place at a temperature ($T_1$) higher than $M_s$. Whereas the spontaneous transformation occurs only at $M_s$ when the chemical driving force reaches the critical value ($\Delta G_{M_s}^{\gamma\rightarrow\alpha'}$):

\[
\Delta G_{T_1}^{\gamma\rightarrow\alpha'} + U' = \Delta G_{M_s}^{\gamma\rightarrow\alpha'} \quad \tag{4}
\]
It has been shown that the mechanical driving force results from the applied stress which aids the chemical driving force. Patel and Cohen \cite{55} suggested that the mechanical driving force can be calculated using the crystallographic theory of martensitic transformation as function of stress and orientation. Thus, according to Fig. 2.3 the corresponding stress level increases linearly with temperature. This linear relation is believed to be valid until the stress level reaches the austenite yield strength.

In 1970 Bolling and Richman defined the $M_s^a$ as the temperature below which the yielding can take place by martensite transformation, whereas at temperatures above $M_s^a$ the transformation takes place after the plastic deformation of austenite. The idea of stress-assisted and strain-induced martensitic transformation was later established in 1972 by Olson and Cohen \cite{36} in order to clarify the transformation mechanism below and above the $M_s^a$. They defined the $M_s^a$ temperature as the maximum temperature at which, the stress-assisted martensitic transformation takes place by means of the “elastic” stress and the critical stress to initiate the transformation within this range increases linearly with temperature. At temperatures above $M_s^a$ when the martensitic transformation takes place after plastic flow of the austenite, the transformation is referred to as strain-induced transformation. Olson and Cohen suggested that the plastic strain of the austenite contributes to the transformation by the generation of energetically favourable nucleation sites through shear band intersections. In this temperature regime the transformation critical stress decreases significantly, and the $M_d$ temperature is
defined as the upper limit for strain-induced transformation to reflect the stability of austenitic phase. The discussion presented above is illustrated in Figure 2.4.

Figure 2.4-Schematic illustration of the critical stress to initiate martensite transformation as function of temperature [36].

It is well established that the austenite stability and the extent of the strain-induced \( \alpha' \)-martensite transformation, is significantly affected by chemical composition, temperature and strain rate.

Numerous studies have been made to relate the \( M_d \) temperature to chemical composition in which one of the most used formulas is attributed to Angel [9],

\[
M_d(30/50)(^\circ C) = 413 - 13.7(%Cr) - 9.5(%Ni) - 8.1(%Mn) - 18.5(%Mo) - 9.2(%Si) - 462(%[C + N]) \ldots \quad (5)
\]

where \( M_d(30/50)(^\circ C) \) represents the temperature at which 50 vol. % \( \alpha' \)-martensite is formed after a true tensile strain of 30 %.

Another most often used equation is due to Nohara et al. [37]. They conducted the following equation by modifying Angel’s equation and including the effect of grain size:

\[
M_d(30/50)(^\circ C) = 551 - 13.7(%Cr) - 29(%[Ni + Cu]) - 8.1(%Mn) - 18.5(%Mo) - 9.2(%Si) - 462(%[C + N]) - 68(%Nb) - 1.42(GS - 8) \quad (6)
\]

where GS is the grain size according to ASTM.

Normally, in solution-treated condition, the \( M_s \) temperature is below room temperature for most of the austenitic grades, but \( M_d \) temperature is usually above room temperature.
The austenite stability and the extent of the strain-induced \( \alpha' \)-martensite transformation has been explained in terms of the variation in the chemical free-energy difference between the austenite and \( \alpha' \)-martensite phases, referred to as the chemical driving force. However, the stacking fault energy play also an important role in determining the austenite stability, since it controls the generation of energetically favourable nucleation sites for the \( \alpha' \)-martensite through formation of the shear bands. Several authors have shown that the SFE is strongly dependent on the chemical composition \([38-40]\) and temperature \([41-43]\). Although there is no empirical equation for \( \varepsilon \)-martensite formation like that for \( \alpha' \)-martensite, it is generally accepted that the decrease in stacking fault energy of the austenite increases the \( \varepsilon \)-martensite formation, whereas an increase of the stacking fault energy changes the dominant deformation mode from the formation of \( \varepsilon \)-martensite to twinning, and then to slip. This is illustrated in figure 2.5 as the schematic diagram defined for an austenitic Fe-Mn-Cr-C alloy by Remy and Pineau (1977) \([44]\).

![Schematic diagram of deformation structure of an austenitic Fe-Mn-Cr-C alloy](image)

**Figure 2.5**-Deformation structure of an austenitic Fe-Mn-Cr-C alloy as a function of both temperature and stacking fault energy \([44]\).

The \( \alpha' \) and \( \varepsilon \)-martensite form almost simultaneously which makes investigation of their formation mechanism rather difficult. According to Mangonon and Thomas \([15]\) the amount of \( \varepsilon \)-martensite formed during deformation of an AISI304 reaches a maximum and decreases afterwards, while the amount of \( \alpha' \)-martensite continuously increases with deformation. Seetharaman and Krishnan \([45]\) also showed that \( \varepsilon \)-martensite formation precedes the formation of \( \alpha' \)-martensite during deformation of AISI316 steel at low temperatures. Therefore it has been suggested that \( \varepsilon \)-martensite is formed from austenite and it is then transformed to \( \alpha' \)-
martensite. It is also found that α'-martensite can be directly formed from austenite.

The deformation induced martensite in stainless steels may revert to austenite during annealing at lower temperatures and shorter times than those required for the recrystallization of deformed stainless steels without the formation of martensite \[46\]. Burstein et al. \[47\] found that it is possible to remove the strain induced martensite in AISI304L by proper electrochemical treatment in aqueous solutions at much lower temperatures than conventional annealing heat treatments.

2.4 Previous Works

The conventional methods of studying the martensitic transformation have been X-ray diffraction (XRD) and electron diffraction in TEM \[48-53\]. X-ray diffraction has been proved to be a very simple and useful method to investigate the crystallography; however morphological study of the microstructure is not possible. On the other side, although TEM establishes a mutual relation between crystallographic information and microstructure, the observed area is usually limited to about \(10^{-10}\)m².

In the last decade, Scanning electron microscope (SEM) and electron backscattering diffraction (EBSD) technique in SEM have been effective tools for characterization of materials \[54, 55\]. More accurate techniques such as Kikuchi diffraction pattern, taken from local area on TEM and EBSD \[56\], and the computer assisted orientation mapping (OIM) in SEM \[57\] have also been used for orientation and crystallography measurements of lath martensite.

Nowadays, new technical developments especially that of high-resolution field emission SEM (FESEM) have coped with the many challenges for critical applications \[58\]. FESEM/EBSD has been used to analyze the structures full of lattice defects, such as ultrafine-grained structures in steels produced by severe plastic deformation \[59, 60\] and also rapid automated pattern acquisition and crystallographic studies of complex microstructures and martensite in steels \[61-63\]. Although one of the difficulties to obtain a sound reconstruction of microstructures by EBSD is the presence of the complex microstructures with more than one phase or phases containing a high density of lattice defects and intrinsic dislocations \[64\].

The strength and toughness of stainless steels seem to be strongly affected by martensite morphology e.g. packet and block sizes (effective grain size in lath martensite structure) \[65, 66\], yet only a few investigations have been made using EBSD to analyse the microstructure of martensite \[67, 68\].
Chapter 3

THEORETICAL ASPECTS

3.1 Quantitative Measurement Methods

Various techniques have been used for quantitative measurements of $\alpha'$-martensite, in which X-ray diffraction and techniques based on the ferromagnetism of $\alpha'$-martensite phase are the most common methods. Density measurements are also used for quantitative phase analysis of $\alpha'$-martensite \cite{69}.

X-ray diffraction method is based on the difference between the crystal structures of the austenite and $\alpha'$-martensite. The texture in the sheet materials, especially after plastic deformation, decreases the accuracy of X-ray diffraction. However this effect can be reduced by averaging intensities of several diffraction peaks \cite{70}. The X-ray diffraction is also expensive and time consuming.

The magnetic techniques are based on measurement of magnetic force acting on the specimen placed in a magnetic field and measurement of magnetization \cite{9, 71-74}. In most of these techniques as well as X-ray diffraction method a small specimen must be cut from the test material.

One in-situ method is the Ferritescope which is easy and non-destructive. Ferritescope is usually used to measure the delta-ferrite content of austenitic stainless steel weldments. The Ferritescope measurements are based on the magnetic induction method. An exciter coil generates a magnetic field which interacts with the magnetic components of the sample. The changes in the magnetic field are proportional to the magnetic constituents of the structure which induce a voltage in a secondary coil, Figure 3.1. This voltage is then measured and calibrated with a set of standard samples. In this way all magnetic components of the structure are recognized including $\alpha$-ferrite, $\delta$-ferrite, and $\alpha'$-martensite.
Theoretical Aspects

Figure 3.1—Schematic illustration of magnetic induction method used in Ferritescope.

The following factors affect the ferrite content measurement with the Ferritescope:

a) Curvature of the measuring object
b) Thickness of the measuring object
c) Layer thickness
d) Distance of the measuring position to the edge

In addition to this, the magnetic permeability of $\alpha'$-martensite is a function of strain \cite{72} which causes an error in the measured values. Therefore, Ferritescope reading must be converted to actual $\alpha'$-martensite content by using a calibration curve. Several calibration curves have been determined by using different measurement techniques. A good comparison of different methods for measuring strain induced $\alpha'$-martensite content in austenitic steels is done by J. Talonen, et al. \cite{73}. According to that, the Ferritescope readings, $F$, can be converted to the actual $\alpha'$-martensite contents $C_{\alpha'}$ with the following equation:

$$C_{\alpha'} (\text{mass\%}) = 1.7 \times F$$ \hspace{1cm} (7)

Moreover, susceptibility of a material changes when subjected to a mechanical stress; this effect is called the Villari effect or inverse-magnetostrictive effect. Therefore, the compensation due to this effect must be considered when the measurements are done with the samples under stress.
Chapter 3

3.2 Kinetics of Strain Induced $\alpha'$-Martensite Transformation

The kinetics of deformation-induced martensite transformation has been of a great importance in the development of high strength, transformation-induced plasticity (TRIP) steels, which achieve high work-hardening rates through such a transformation. The increasing use of finite element method (FEM) in the engineering application, for instance in the automotive industry, demands accurate constitutive equation which can simulate the material behaviour. However, prediction of material behaviour and mechanical response in the metastable steels is rather difficult because of the complex interaction between the external conditions and microstructural evolution.

The constitutive equations and macroscopic models as well as micromechanical and mesoscopic modelling for metastable steels have been extensively investigated in the literature.

One of the best known models to predict the kinetics of strain induced martensite transformation was developed by Olson and Cohen [76]. They assumed that the shear band intersection is the dominant mechanism of strain induced nucleation. By considering the course of shear band formation, the probability of shear band intersections, and the probability of an intersection generating a martensitic embryo, they derived the following equation for the volume fraction of martensite vs. plastic strain:

$$f^{\alpha'} = 1 - \exp\{-\beta[1 - \exp(-\alpha\varepsilon)]^n\} \quad (8)$$

Where $\alpha$ defines the course of shear bands formation with strain and is temperature sensitive through its dependence of stacking fault energy. The $\beta$ parameter is proportional to the probability that an intersection will form an embryo, and is temperature dependent through its relation to the FCC-BCC chemical driving force. The calculated transformation curves give a sigmoidal shape with saturation levels below 100 pct. It is suggested then that the temperature dependence of the transformation kinetics may be minimized by decreasing the FCC, BCC and HCP entropy differences through proper compositional control [76].
Chapter 4

EXPERIMENTAL WORK

4.1 Materials and Sample Preparations

The test materials were commercial austenitic stainless steel grades AISI304 (EN 1.4301) and AISI301 (EN 1.4310) and AISI204Cu provided by Outokumpu Stainless Steels. The chemical compositions of the steels are given in Table 4.1. All sheets were 1 mm thick.

Table 4.1—Chemical composition of the investigated grades.

<table>
<thead>
<tr>
<th>AISI Grade</th>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>P</th>
<th>Cr</th>
<th>Ni</th>
<th>Mo</th>
<th>Nb</th>
<th>Cu</th>
<th>Co</th>
<th>N</th>
</tr>
</thead>
<tbody>
<tr>
<td>301</td>
<td>0.105</td>
<td>0.9</td>
<td>1.14</td>
<td>0.025</td>
<td>16.88</td>
<td>7.15</td>
<td>0.24</td>
<td>0.002</td>
<td>0.21</td>
<td>0.14</td>
<td>0.023</td>
</tr>
<tr>
<td>304</td>
<td>0.052</td>
<td>0.38</td>
<td>1.15</td>
<td>0.032</td>
<td>18.1</td>
<td>9.09</td>
<td>0.12</td>
<td>0.007</td>
<td>0.17</td>
<td>0.1</td>
<td>0.043</td>
</tr>
<tr>
<td>204Cu</td>
<td>0.075</td>
<td>0.4</td>
<td>9.01</td>
<td>0.034</td>
<td>15.02</td>
<td>1.17</td>
<td>0.03</td>
<td>0.001</td>
<td>1.67</td>
<td>0.046</td>
<td>0.12</td>
</tr>
</tbody>
</table>

The effect of sample preparation method (mechanical polishing vs. electrolytic polishing) was investigated. The specimens for the optical microscopy and SEM/EBSD were cut out of the test samples and the surface was studied. In order to avoid cold-working effect, the samples were first grinded by 600 grit sandpaper and then electrolytically polished at room temperature. The electropolishing was done by Struers’ Lectropol in a mixture of 90% acetic acid and 10% perchloric acid for 2 min at a voltage of 20 V. The specimens were immediately rinsed with water and placed in an ultrasonic ethanol bath for 2 min to obtain the required surface finish.

In spite of the fact that stainless steels are reasonably easy to polish, etching is usually a more difficult step. The corrosion resistance of stainless steels and the potential microstructure complexity of these alloys make selection of best etchant a more difficult problem than for carbon and alloy steels. A series of microetchants was tested for different grades and structures in this study. Table 4.2 shows the etchants used in this work.
Table 4.2—The microetchants for stainless steels tested in this study.

<table>
<thead>
<tr>
<th>Etchant</th>
<th>Composition</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aqueous nitric acid electrolytic etch</td>
<td>60 mL HNO₃ and 40 mL H₂O</td>
</tr>
<tr>
<td>Aqueous oxalic acid electrolytic etch</td>
<td>10 g oxalic acid and 100 mL H₂O</td>
</tr>
<tr>
<td>V2A, Kungsvatten</td>
<td>50 mL H₂O, 50 mL HCl, 5 mL HNO₃</td>
</tr>
<tr>
<td>15-10-10 Acetic glyceregia, Tint etch</td>
<td>10 mL HNO₃, 10 mL acetic acid, 15 mL HCl, 2 drops glycerol (Add HNO₃ last)</td>
</tr>
<tr>
<td>Beraha’s tint etch</td>
<td>1 part HCl, 2 parts H₂O, 1 g K₂S₂O₅ per 100 mL of solution</td>
</tr>
</tbody>
</table>

4.2 In-Situ Martensite Measurements during Tensile Testing

The plastic deformation of the test materials was done by means of uniaxial tensile test. The experimental equipment for martensite measurements during tensile loading included a tensile machine INSTRON 8500 PLUS, an extensometer for strain measurements, a thermocouple for temperature measurements and a Ferritescope, FERITSCOPE® MP30, for monitoring of the martensite content. Figure 4.1 shows the setup with attached extensometer, welded thermocouple and the Ferritescope probe for martensite measurements.

**Figure 4.1**—Specimen with attached extensometer, Ferritescope probe and welded thermocouple.
The thermocouple was calibrated for the temperature region -50 to 100°C. The Ferritescope was calibrated with δ-ferrite standard samples. The tensile specimens were cut and trimmed according to the geometry described in Figure 4.2. The specimens were austenitized at 1050°C for 5 minutes and quenched in water. In order to remove the stains from the surface, the samples were pickled in a mixture of 3M HNO₃ and 2M HF at 60 ±1°C for 1.5-2 minutes.

Susceptibility of a material changes when subjected to a mechanical stress; this effect is called the Villari-effect or the inverse-magnetostrictive effect. In order to determine the true martensite content, the compensation due to this effect has to be done. More precisely, necessary information to the martensite content determination is provided during the unloading and the specimens are not allowed to fracture. Therefore, all the tensile tests including martensite measurements preceded by tensile tests at which the specimens were strained to fracture for determination of the fracture strain, A50. The fracture strain is determined at each temperature. A conservative strain value at which unloading takes place in the tensile tests including martensite measurement is then determined by multiplying A50 with 0.8-0.9. Further tensile tests were carried out and stopped at 10, 20 and 30% nominal strain for each grade.

In order to utilize the measured data, some numerical treatment is necessary to obtain the true stress, true strain and particularly the correct martensite content.
The effect of thickness and inverse-magnetostrictive effect (Villari effect) on the result were compensated for. (Appendix 1)

All the tensile tests were carried out at the nominal strain rate of $1 \times 10^{-3} \text{s}^{-1}$.

### 4.2.1 Testing at Various Temperatures

The effect of temperature was studied by performing the test at three different temperatures: room temperature, above and below room temperature. For the tests below room temperature, the specimens were first cooled down to -30°C in an ethanol bath and the test started when the temperature reached 0°C. When testing was performed at temperature above room temperature, a hot-air pistol was used to heat the samples. The specimens were first heated up to 80°C and when the temperature decreased to 60°C, the tensile tests started. The adiabatic heating during the deformation was monitored by means of the thermocouple welded on the specimen. The above methods for cooling and heating can be used to establish the initial temperature and for isothermal tests other techniques have to be used.

### 4.3 EBSD Analysis

The EBSD scans were performed in a LEO 1530 field emission gun-scanning electron microscope (FEG-SEM) equipped with the Channel 5 system (HKL technology, Hobro, Denmark). The SEM was operating at 15kV and 10 mm working distance, with the sample tilted by 70 deg. a 0.5 and 0.1 μm step size were used. The morphology and crystallography of spontaneous martensite (thermal) and deformation induced α’-martensite was investigated at different strain levels.
Chapter 5

RESULTS AND DISCUSSION

5.1 Effect of Sample Preparation Method

Electrolytic polishing or electropolishing is widely used in metallography of stainless steels. When electropolishing is correctly performed a high quality, scratch free surface will be produced and the deformation from cutting and grinding is removed. Generally a high quality, deformation free surface will be obtained by electropolishing; though, inclusion attack may be encountered, and second phases may be attacked preferentially.

Figure 5.1 shows the difference between the microstructures of two grades in as-received condition, when the samples are mechanically and electrolytically polished. As it can be seen in Figure 5.1, the microstructure of the surface of austenitic grades is very sensitive to the sample preparation method. The grinding and mechanical polishing causes the artefacts, which make the study of structures, phases and transformation difficult. Therefore, the electropolishing is strongly recommended.
Figure 5.1-The microstructures of two austenitic grades in as-received condition (a. AISI204Cu, b. AISI304), when the samples are mechanically polished (left) and electropolished (right).

5.2 Etching

Etching of the austenitic grades to examine the grain structure is difficult with most standard reagents. To measure the grain size, when a more accurate value is required than can be obtained by comparison chart rating, all grain boundaries must be revealed and most of the standard reagents reveal only some of the grain boundaries. Tint etching reveals all of the grains by colour contrast. However it requires a high quality polish for good results. An alternative technique is electrolytically etching. Electrolytically etching methods provide greater control of the etching process and are highly reproducible.
5.2.1 Aqueous Nitric Acid Electrolytic Etching

Aqueous nitric acid electrolytic etch was used with stainless steel cathode at 1.1 V dc, 0.075-0.14 A/cm², and 25 mm spacing for 120 s.

The etchant revealed austenite grain boundaries (but not twins) in AISI304 and AISI301 grades (Figure 5.2) but it had a poor attack on manganese containing AISI204Cu grade.

Figure 5.2-Grain boundaries were revealed in (a) AISI304 (twins were not attacked) and (b) AISI301 grades by aqueous nitric acid electrolytic etch.

5.2.2 Aqueous Oxalic Acid Electrolytic Etching

Oxalic acid electrolytic etching is a popular electrolytic etch for austenitic grades. The etching was performed at 2.7 V dc and 25 mm spacing with stainless steel cathode. The grain boundaries were revealed in AISI301 and AISI304 grades after 120 sec, and the twins were also attacked. (Figure 5.3).
Results and Discussion

Figure 5.3-Grain boundaries and twins were attacked in (a) AISI304 (b) AISI301 grades by oxalic acid electrolytic etching.

The etchant revealed (dissolves) the carbides at the grain boundaries after 30-60 sec in AISI204Cu grade when sensitized (Figure 5.4), but it had a poor attack on this grade in solution treated condition.

Figure 5.4-Carbides at the grain boundaries and twins were attacked in sensitized AISI204Cu grade by oxalic acid electrolytic etching.

When martensite was present in the structure the martensite and austenite phases have responded differently to the oxalic acid electrolytic etching. Topographical features of the surface revealed the microstructure morphology. Clear distinction between shear bands and \(\alpha'\)-martensite, however, was rather difficult. Figure 5.5 shows the microstructure of AISI301 steel after austenitization and quenching in liquid nitrogen.
Figure 5.5-Shear bands and $\alpha'$-martensite were attacked in quenched AISI301 grade by Oxalic acid electrolytic etching.

5.2.3 V2A Kungsvatten

The etching by V2A reagent was carried out at 60ºC. The sample was placed in the etchant with the polished side of the sample facing upward until hydrogen bubbles appeared on the surface. The sample was then shaken so that the bubbles disappeared; the etching was repeated 2 times. To obtain a smooth etching, a few drops of etchant could be dropped on the polished surface.

The grain boundaries were partially attacked and the result of etching was not satisfactory. (Figure 5.6) However, this etchant is appropriate for quickly etching the microstructure.

Figure 5.6-The grain boundaries were partially attacked in AISI304 grade by V2A.
5.2.4 Acetic Glyceregia 15-10-10

This etchant was very strong and aggressive and suitable for the grades which are difficult to etch. The grains and twins were revealed by colour difference in AISI204Cu grade within a few seconds. See Figure 5.7.

The etchant must be used fresh and never be stored (It should be discarded when reagent is orange colour). The etching must be done with care under a hood.

![Figure 5.7-Grain boundaries and twins were revealed in AISI204Cu grade by tint etching in 15-10-10 acetic glyceregia reagent.](image)

5.2.5 Beraha’s Tint Etching

1 g K$_2$S$_2$O$_5$ per 100 mL of solution was added last and the etchant was active only 2 hours after adding K$_2$S$_2$O$_5$. The specimens were immersed in the etchant at room temperature until surface became reddish.

The grain boundaries and twins were revealed by colour difference after 20 sec in 204Cu grade, and carbides were attacked if present (Figure 5.8). The etching time for grain boundary etching in AISI301 and AISI304 grades was about 120 sec.
Figure 5.8-Grain boundaries and twins were revealed by colour difference in (a) AISI204Cu and (b) AISI304 by etching with Beraha’s tint reagent.

The Beraha’s tint etchant was also found to be suitable for etching of all grades after martensitic transformation either by quenching or deformation, when α’-martensite is present. Martensite was revealed by brown colour after 20 sec (See Figure 5.9). The etchant at longer immersion time attacked the prior austenite grain boundaries while martensite turns to black.

Figure 5.9-Martensite was revealed brown by etching in Beraha’s tint reagent in AISI301grade: (a) quenched in liquid nitrogen and (b) 60% strained by tensile at room temperature.
5.3 Heat Treatments

Figure 5.10(a) shows the AISI301 microstructure at the as-received condition (cold rolled). The grains in the as-received sheets are stretched along the rolling direction and approximately 35% of the structure is consisting of α’-martensite. In order to optimize the austenitization heat treatments, three samples were heat treated at 1050°C for 5, 10 and 30 minutes and subsequently quenched in brine. (Figure 5.10 (b), (c) and (d)). Figure 5.10 shows that austenitization for 10 an 30 minutes causes abnormal grain growth in which some martensite formed during quenching. The formation of martensite in these grains might be due to the accommodation of residual thermal stresses in them. Therefore all of the tensile specimens in this study were heat treated at 1050°C for 5 minutes.

Figure 5.10-Microstructure of AISI301 in (a) As-received condition (cold rolled) and austenitized at 1050°C for (b) 5 minutes (c) 10 minutes and (d) 30 minutes.
Chapter 5

The same austenitization cycle (1050°C, 5 min) was carried out for the AISI204Cu and AISI304 grades. Figure 5.11(a) shows the as-received microstructure of AISI204Cu, where the carbides are present at the grain boundaries; and Figure 5.11(b) shows the microstructure after solution treatment.

The microstructure of AISI304 sheet in as-received condition and after solution treatment is depicted in Figure 5.12 (a) and (b) respectively.

![Figure 5.11-Microstructure of AISI204Cu sheet (a) as-received, carbides are present at the grain boundaries (b) solution treated.](image1)

![Figure 5.12-Microstructure of AISI304 sheet (a) as-received (b) solution treated.](image2)

The grain sizes were measured by mean linear intercept method after austenitization. Table 5.1 shows the number of intercepts counted per millimetre, average intercept distance, and the calculated grain size number according to ASTM standard.
Table 5.1 – The grain size after austenitization.

<table>
<thead>
<tr>
<th>Grade</th>
<th>Intercept count. n/l (mm(^2))</th>
<th>Average intercept distance (μm)</th>
<th>ASTM grain size number G</th>
</tr>
</thead>
<tbody>
<tr>
<td>AISI301</td>
<td>38,6</td>
<td>25,9</td>
<td>7,3</td>
</tr>
<tr>
<td>AISI304</td>
<td>35,2</td>
<td>28,4</td>
<td>7,0</td>
</tr>
<tr>
<td>AISI204Cu</td>
<td>27,8</td>
<td>36,0</td>
<td>6,3</td>
</tr>
</tbody>
</table>

5.4 Optical Microscopy

Comprehensive optical microscopy was performed on the samples of the three grades after quenching in liquid nitrogen and also on the strained specimens, deformed up to various strains at different temperatures. A selection of micrographs is discussed in this section to indicate general aspects of the observed martensite morphology. Quantitative image analysis was also carried out on OM micrographs to clarify the relation between actual martensite content and Ferritescope readings which is presented in section 5.5.

![Figure 5.13](image)

Figure 5.13 – The light optical micrograph of AISI301 steel after austenitization and quenching in brine and liquid nitrogen.

The AISI301 grade exhibited a considerable amount of martensitic transformation upon quenching in liquid nitrogen whereas no martensite was observed in AISI304 and AISI204Cu steels. Figure 5.13 shows the light optical micrograph of an
AISI301 sample after austenitization and subsequent quenching in brine (25 °C) followed by quenching in liquid nitrogen (-196°C). The morphology of the product martensite phase consists of thin needles which align themselves in parallel bands, but packets of martensite can also be observed outside the parallel bands. Although reconstruction of parent austenite grains is rather difficult, one or two sets of parallel bands seem to be transformed in each austenite grain.

Figure 5.14 (a) to (d) shows the microstructure evolution of the AISI301 steel, deformed at room temperature, up to true plastic strains of 0.093, 0.180, 0.256 and 0.403 respectively. It seems that the martensitic transformation is mostly confined within the shear bands or at the intersections and the martensite content increases with proceeding deformation.

Figure 5.14-Evolution of deformation microstructure in AISI301 tensile strained at room temperature to true plastic strain of (a) ε= 0.093, (b) ε=0.180, (c) ε=0.256, (d) ε=0.403.

The optical micrographs of the AISI204Cu steel, deformed to different plastic strains are shown in Figure 5.15. Similarly, the morphology of the α’-martensite consists of sets of thin parallel needles that go through the parent austenite grain.
Results and Discussion

and stop at the grain or annealing twin boundaries. Large amount of $\alpha'$-martensite can be seen at the intersection of shear bands. This is in agreement with previous optical and electron microscopy studies, which report the presence of $\alpha'$-martensite at the intersection of shear bands or $\epsilon$ bands \cite{73, 76-77}. However considerable amount of $\alpha'$-martensite is also observed when only one set of bands is activated. The amount of retained austenite decreases as the deformation proceeds.

Figure 5.15-Evolution of deformation microstructure in AISI204Cu tensile strained at room temperature to true plastic strain of (a) $\varepsilon=0.093$, (b) $\varepsilon=0.180$, (c) $\varepsilon=0.256$, (d) $\varepsilon=0.467$

The AISI304 grade shows the highest austenite stability among the investigated grades. Figure 5.16 shows the microstructure of 304 steel, strained up to true plastic strain of $\varepsilon=0.403$. Figure 5.16 shows that when the test was carried out at room temperature, the martensite has nucleated mostly at the intersections of shear bands. Whereas when the test was initiated at a lower temperature, more martensite is formed within the shear bands.
5.5 Image Analysis

Extensive metallography by optical microscopy was performed on the strained samples and the volume fractions of $\alpha'$-martensite were measured by quantitative image analysis. Figure 5.17 indicates the relation between Ferritescope readings and image analysis results. Each data point represents the $\alpha'$-martensite volume fraction evaluated with quantitative image analysis measurements from an individual test sample, after straining to the requested plastic strain. The straight line in the Figure 5.17 represents the result of an investigation by the Talonen et al. [75] in which the Ferritescope readings, can be converted to the actual $\alpha'$-martensite content by the correction factor of 1.7 (Equation 7).

The result of quantitative image analysis in this study indicates a higher $\alpha'$-martensite volume fractions than calculated by the Talonen et al. formula. The low spatial resolution of optical microscopy, uneven etching effect as well as false indication of shear bands and grain boundaries as $\alpha'$-martensite phase, may lead to higher estimation of actual $\alpha'$-martensite content in the optical microscopy image analysis. However the results of the image analysis seem to have a good correlation with the results of the comparison made by Talonen et al. Therefore, the correction factor of 1.7 was taken for the conversion of Ferritescope readings to the actual $\alpha'$-martensite content in this study.
Figure 5.17 - The Ferritescope reading and actual $\alpha'$-martensite contents by LOM.

5.6 Temperature Mapping

The changes in the temperature were recorded during all of the tensile tests. Figures 5.18 to 5.20 show the temperature profiles for different grades when the tests were started at 0°C, room temperature, and 60°C respectively.

Figure 5.18 - Measured temperatures during tensile testing of 3 grades when the tests were started at 0°C.
Figure 5.19- Measured temperatures during tensile testing of 3 grades when the tests were started at room temperature.

The increase in temperature can be observed in all of the tests. This is mainly due to the conversion of mechanical work to heat. The measured heating of the samples was higher in less stable grades, AISI301 and AISI204Cu. The fluctuations in temperature were also observed to be increasing by strain in these grades. This is due to the exothermic nature of the strain induced $\alpha'$-martensite transformation. The more stable AISI304 steel exhibited a different behaviour than the AISI301 and AISI204Cu steels: The increase in temperature in the AISI304 steel was less than that in the other two steels and almost no fluctuation in temperature was measured. This is due to the fact that the role of heat generation (related to the martensitic transformation) in this grade is negligible, in comparison with the less stable grades. Furthermore, some of the mechanical energy may be stored in the microstructure.
Results and Discussion

Figure 5.20-Measured temperatures during tensile testing of 3 grades when the tests were started at 60°C.

It is noteworthy that the strain tests were carried out at $10^3 \text{ s}^{-1}$ in a non-isolated environment. Consequently, the heat transfer to the environment dramatically changes the rate of heat accumulation. For instance, Figure 5.20 shows that the temperature initially decreases and then increases when the test were started at 60°C, indicating the contrary effect of heat transfer to the environment and heat generation in the sample.

5.7 In-Situ Martensite Measurements

The strain-induced $\alpha'$-martensite volume percentage in the AISI301 and 204Cu steels tensile strained at various temperatures is plotted as a function of true plastic strain in Figures 5.21 and 5.22. The effects of tensile sample thickness and inverse-magnetostrictive effect (Villari effect) on the Ferritescope readings were compensated for and the correction factor of 1.7 for the convection of Ferritescope readings to the $\alpha'$-martensite content was used \cite{75}. Each colour in the diagram represents the in-situ $\alpha'$-martensite content measured from an individual test sample during straining up to the requested plastic strain.
Figure 5.21- Strain induced $\alpha'$-martensite content as a function of true plastic strain in AISI301 steel when the tests were started at 0, 25 and 60°C.

Figure 5.22- Strain induced $\alpha'$-martensite content as a function of true plastic strain in AISI204Cu steel when the tests were started at 0, 25 and 60°C.

Figure 5.23 shows the volume percent of $\alpha'$-martensite as a function of plastic strain in the more stable AISI304 steel, tensile strained at different temperatures.
Results and Discussion

![Figure 5.23](image)

**Figure 5.23**-Strain induced $\alpha'$-martensite content as a function of true plastic strain in AISI304 steel when the tests were started at 0, 25 and 60°C.

The results plotted in Figures 5.21 to 5.23 show the strong temperature and composition dependence of austenite stability. The AISI301 steel exhibits the least austenite stability in comparison to the AISI204Cu and 304 steels, with extensive strain induced $\alpha'$-martensite transformation at all the test temperatures. Significant $\alpha'$-martensite transformation was also observed in AISI204Cu steel, whereas in the more stable AISI304 steel, the transformation only occurred at high strain levels at low temperatures. The martensitic transformation was considerably suppressed by increasing temperature in all of the investigated steels.

### 5.8 Effect of Chemical Composition

As stated before, a considerable amount of martensitic transformation was observed in the AISI301 grade upon quenching in liquid nitrogen whereas no martensite was observed in AISI304 and AISI204Cu steels. Table 5.2 shows the calculated $M_s$ according to Eichelmann (Equation 3) and $M_d(30/50)$ according to Angel and Nohara’s formulae (Equations 5 and 6). In contradiction to what observed, Eichelmann formula predicts a $M_s$ temperature about -33.8°C for the AISI204Cu steel. It should be noticed that the Eichelmann formula does not involve copper element, while this grade contains about 1.7% Cu. Determination of $M_d(30/50)$ temperature with high degree of precision requires performing more isothermal deformation tests at various temperatures which was not the case in this study. Nevertheless, the calculated $M_d(30/50)$ temperatures are presented for comparison.
Table 5.2—Calculated $M_s$ and $M_d(30/50)$ by the empirical equations.

<table>
<thead>
<tr>
<th>AISI Grade</th>
<th>$M_s$ (°C)</th>
<th>$M_d(30/50)$ (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Eichelmann</td>
<td></td>
<td></td>
</tr>
<tr>
<td>301</td>
<td>-119,3</td>
<td>32,7</td>
</tr>
<tr>
<td>304</td>
<td>-219,6</td>
<td>19,8</td>
</tr>
<tr>
<td>204Cu</td>
<td>-33,8</td>
<td>28,8</td>
</tr>
</tbody>
</table>

5.9 Modelling

Least squares method was used for fitting of the Olson-Cohen model, Equation 8, to the experimental data. It should be mentioned that the Olson and Cohen model is an isothermal model, which was not the case in this study. However, still the experimental values were fitted fairly well with this model. The lines shown in Figure 5.24 to 5.26 represent the fitting results, where a constant value of 4.5 was used for the exponent $n$.

![Graph showing volume fraction of strain induced $\alpha'$-martensite $f_{\alpha'}$ as a function of true plastic strain in AISI301 steel tensile tested at strain rate $1 \times 10^{-3}$ s$^{-1}$ when the tests were started at different temperatures.]

Figure 5.24—Volume fraction of strain induced $\alpha'$-martensite $f_{\alpha'}$ as a function of true plastic strain in AISI301 steel tensile tested at strain rate $1 \times 10^{-3}$ s$^{-1}$ when the tests were started at different temperatures.

The parameters $\alpha$ and $\beta$ obtained by the fitting are shown in Tables 5.3 to 5.5.
Table 5.3—α and β parameters of Olson and Cohen’s kinetic model for AISI301 steel, tensile tested at strain rate $1 \times 10^{-3} \text{ s}^{-1}$ determined by least squares fitting.

<table>
<thead>
<tr>
<th>Initial temperature</th>
<th>0°C</th>
<th>25°C</th>
<th>60°C</th>
</tr>
</thead>
<tbody>
<tr>
<td>α</td>
<td>7.806</td>
<td>4.81</td>
<td>2.472</td>
</tr>
<tr>
<td>β</td>
<td>1.848</td>
<td>2.765</td>
<td>6.222</td>
</tr>
</tbody>
</table>

It was found that α decreased by increasing temperature, supporting the Olson-Cohen hypothesis that formation of shear bands is reduced by increasing temperature, through its dependence to the stacking fault energy. On the other hand, in contrast to what suggested by Olson and Cohen, β increased with increasing temperature and showed the highest value at the highest temperature. According to the Olson-Cohen model, the saturation level is determined by β, while the rate of approach to saturation is controlled by both α and β. In other words the saturation values pay an important role in fitting of the β parameter. It should be noticed that in many cases in the present work, the saturation doesn't seem to be reached within the material tensile strength at the test conditions (temperature and strain rate).

Figure 5.25—Volume fraction of strain induced α’-martensite $f_{\alpha'}$ as a function of true plastic strain in AISI204Cu steel tensile tested at strain rate $1 \times 10^{-3} \text{ s}^{-1}$ when the tests were started at different temperatures.
Table 5.4 – α and β parameters of Olson and Cohen’s kinetic model for AISI204Cu steel, tensile tested at strain rate $1 \times 10^{-3}$ s$^{-1}$ determined by least squares fitting.

<table>
<thead>
<tr>
<th>Initial temperature</th>
<th>$0^\circ$C</th>
<th>$25^\circ$C</th>
<th>$60^\circ$C</th>
</tr>
</thead>
<tbody>
<tr>
<td>α</td>
<td>7.3</td>
<td>5.095</td>
<td>2.617</td>
</tr>
<tr>
<td>β</td>
<td>0.9556</td>
<td>1.093</td>
<td>2.322</td>
</tr>
</tbody>
</table>

Figure 5.26- Volume fraction of strain induced $\alpha'$-martensite $f^{\alpha'}$ as a function of true plastic strain in AISI304 steel tensile tested at strain rate $1 \times 10^{-3}$ s$^{-1}$ when the tests were started at different temperatures.

Table 5.5 – α and β parameters of Olson and Cohen’s kinetic model for AISI304 steel, tensile tested at strain rate $1 \times 10^{-3}$ s$^{-1}$ determined by least squares fitting.

<table>
<thead>
<tr>
<th>Initial temperature</th>
<th>$0^\circ$C</th>
<th>$25^\circ$C</th>
<th>$60^\circ$C</th>
</tr>
</thead>
<tbody>
<tr>
<td>α</td>
<td>4.588</td>
<td>2.125</td>
<td>1.038</td>
</tr>
<tr>
<td>β</td>
<td>0.1958</td>
<td>0.5526</td>
<td>4.85</td>
</tr>
</tbody>
</table>
5.10 EBSD Analysis

5.10.1 Thermal $\alpha'$-Martensite

Figure 5.27(a) presents a phase map combined with a band contrast map of AISI304 steel quenched in liquid nitrogen. The phase map (where the martensite and austenite are coloured red and green respectively) shows that almost no martensite has formed spontaneously during quenching in liquid nitrogen. This is in agreement with previous studies and optical metallography in this study, which indicates that the $M_s$ temperature for this grade is below the boiling point of liquid nitrogen. The inverse pole Figure (IPF) colour map of the same area, Figure 5.27(b), shows the orientation of each indexed diffraction pattern by giving it a unique colour. Therefore, different grains and annealing twins are easily distinguished.

![Figure 5.27](image)

Figure 5.27- (a) Phase map combined with band contrast map (b) IPF orientation map of AISI304 steel after quenching in liquid nitrogen.

Unlike the AISI304 steel, the AISI301 grade exhibits a considerable amount of martensite transformation upon quenching in liquid nitrogen. Figure 5.28 shows a Kikuchi pattern quality (KPQ) map obtained from the EBSD measurements of the AISI301 steel, after austenitization and quenching in liquid nitrogen. The image quality map is based on the average intensity of Kikuchi bands against the overall pattern intensity (Band contrast) and is very sensitive to microstructure features. Local strains at deformed regions and pattern overlapping at grain boundaries usually give a low band contrast value because it is difficult to obtain clear electron backscatter pattern. Black-to-white pixel gray levels in Figure 5.28 indicate increasing KPQ values. Therefore, the prior austenite grain boundaries and the lath
Chapter 5

martensite with high density of dislocations are revealed darker within the austenite grains.

![Kikuchi pattern quality (KPQ) map of AISI301 steel after austenitization and quenching in liquid nitrogen.](image)

**Figure 5.28** - Kikuchi pattern quality (KPQ) map of AISI301 steel after austenitization and quenching in liquid nitrogen.

Figure 5.29 shows the phase map combined with the band contrast map of the same area as in Figure 5.28. The morphology and distribution of the martensite formed during quenching are shown in this figure, where the martensite and austenite are coloured red and green respectively. It is observed that the martensite phase has extensively formed within the two twin boundaries and in the other regions it has nucleated at grain boundaries or twins and spread within the parent γ grain. The statistical analysis, after performing the noise reduction routine for zero solution points (measurements which could not be indexed), shows that about 40% martensite has formed in this sample.
Phase boundaries with up to 5° deviation from the Kurdjumov-Sachs orientation relationship (Equation 2) are shown in yellow. The frequency of deviation from the Kurdjumov-Sachs orientation relationship is also illustrated in Figure 5.30. The accumulative analysis of the same data shows that 90% of the phase boundaries consist of K-S defined boundaries up to 5° deviation and the rest is mostly at the grain boundaries where the martensite phase in one grain is adjacent to the austenite from the other grains.
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Figure 5.30- The deviation from the Kurdjumov-Sachs orientation relationship at phase boundaries.

Figure 5.31 shows an Inverse Pole Figure (IPF) colour map only for the martensite phase (the austenite phase is showed by the gray level in band contrast). The colours in this figure correspond to the crystallographic orientation normal to the observed plane, as indicated by stereographic triangle in the inset. The detailed morphology and crystallographic features of the lath martensite structure can be easily distinguished in this mode. The boundaries in Figure 5.31 are drawn when the misorientation between adjacent points is greater than 10º, since according to K-S orientation relationship all the packet and block boundaries should have misorientations larger than 10º (Table 2.1).
**Figure 5.31**-IPF colour map of $\alpha'$-martensite, austenite phase is showed in gray. The black boundaries show misorientations above 10°.

Fig. 5.32 shows the point to point misorientation profile between points A and B. Figure 5.32 shows that the corresponding block boundary has a misorientation of approximately 50°, which corresponds to the one of the calculated misorientation between the variants. However, the misorientation within each block is rather small, and shows some picks close to 5°. Therefore, it is concluded that these areas have blocks with only one variant. Although the misorientations of 5° to 10° for the ideal misorientation relationship of $<011>$10.53° is often reported in literature between two variants [17, 28].
Figure 5.32-Point to point misorientation between points A and B in Figure 5.31.

5.10.2 Deformation Induced $\alpha'$-Martensite

Figure 5.33 shows the SEM image of AISI204Cu steel deformed up to 30% nominal strain at room temperature. Since the martensite and austenite phases have responded differently to the applied electropolishing, topographical features of the surface reveal the microstructure morphology in the SEM secondary electron mode. The austenite grain boundaries can also be distinguished in this mode.

Figure 5.33-The Secondary electron mode SEM image of AISI204Cu steel deformed up to 30% nominal strain at room temperature.
Results and Discussion

Figure 5.34 shows the phase maps combined with band contrast maps of individual AISI204Cu steel samples at 20%, 30% and 60% nominal plastic strain. The tensile direction is parallel to the horizontal direction in all images. The morphology and distribution of the martensite is depicted in the phase maps, where the martensite and austenite are coloured red and green respectively. It is interesting to notice that, the $\alpha'$-martensite is mostly limited within the shear bands however it has also formed when only one set of bands is activated and not necessarily at their intersections.

**Figure 5.34** - Phase map combined with the band contrast map of the AISI204Cu steel at (a) 20% (b) 30% and (c) 60% nominal plastic strain.
It is known that the presence of the complex microstructures with more than one phase or phases containing a high density of lattice defects and intrinsic dislocations causes improper identification of the diffraction patterns and indexing errors using EBSD. The fraction of non indexed points for these samples was 7.4%, 14% and 30% at 20%, 30% and 60% plastic strain respectively. Table 5.6 summarizes the volume fraction of non-indexed points, martensite of the samples for each EBSD scan, and the fraction of martensite calculated from Ferritescope measurements for the same tensile specimen. The lower levels of martensite within the EBSD measurements can be attributed to poor indexing of regions containing martensite phase with high density of lattice defects. Figure 5.35(a) and (b) shows the phase map of the AISI304 and 301 steels at 50% strain respectively.

Table 5.6-Volume fraction of the non-indexed points and $\alpha'$-martensite content measured with EBSD; last column contains data from Ferritescope measurements on the corresponding tensile specimens.

<table>
<thead>
<tr>
<th>AISI Grade</th>
<th>Plastic strain, %</th>
<th>Non-indexed, %</th>
<th>$\alpha'$-martensite, vol, %</th>
<th>$\alpha'$-martensite, vol, % (Ferritescope)</th>
</tr>
</thead>
<tbody>
<tr>
<td>204Cu</td>
<td>20</td>
<td>7.37</td>
<td>6.56</td>
<td>8.23</td>
</tr>
<tr>
<td>204Cu</td>
<td>30</td>
<td>13.99</td>
<td>11.1</td>
<td>20.39</td>
</tr>
<tr>
<td>204Cu</td>
<td>60</td>
<td>29.54</td>
<td>34.3</td>
<td>48.04</td>
</tr>
<tr>
<td>301</td>
<td>50</td>
<td>30.55</td>
<td>46.37</td>
<td>77.03</td>
</tr>
<tr>
<td>304</td>
<td>50</td>
<td>5.77</td>
<td>1.01</td>
<td>4.53</td>
</tr>
</tbody>
</table>
Information about the parent austenite grains orientations can also be deducted from the EBSD. The Schmid factor is calculated for the orientation at each point for the austenite phase and the data displayed as a gray colour map in Figure 5.36. The Schmid factor gives an indication of how easy it is for slip to occur for a particular slip system. Black to white pixel gray levels indicate increasing Schmid factor, when the slip system is defined as \{111\} and the direction is of type \langle110\rangle. The load direction is parallel to the horizontal direction. It can be seen that in light gray grains more slip systems are more activated and consequently more martensite is formed alongside the shear bands and their intersections. However, a more detailed analysis of martensitic transformation with respect to the parent austenite texture requires EBSD maps covering more grains.
Figure 5.36 - An example of EBSD scan of the AISI204cu steel at (a) 20% and (b) 30% plastic strain. Black to white pixel gray levels indicate increasing Schmid factor in austenite; martensite is coloured red.

Large orientation gradients are observed in the austenite grains even though the point to point misorientations are always below 2°. The orientation gradients in the parent grains can readily be seen in Fig. 5.35 as the contrast changes within the austenite grains.

Because of the large orientation gradients and the non-indexed points, the orientation relations between the different phases cannot be determined with a high degree of precision. However, after performing a standard noise reduction with medium level of extrapolation, the volume fraction of the non-indexed points were lowered to below 1 percent and the $\alpha'/\gamma$ and $\alpha'/\alpha'$ orientation relationship analysis was facilitated.

Figure 5.37 shows the phase map combined with the band contrast map of the AISI204Cu steel at 60% nominal strain after noise reduction, where the martensite and austenite are shown in red and green respectively. Phase boundaries up to 5° deviation from the Kurdjumov-Sachs orientation relationship (Equation 2) are shown in yellow. It can be seen that despite the high plastic deformation and local distortions, the phase boundary misorientations matches the K-S OR fairly well.
Results and Discussion

Figure 5.37-The phase map combined with the band contrast map of AISI204Cu steel at 60% nominal strain, yellow lines shows the phase boundaries up to 5° deviation from the K-S OR.

Figure 5.38 shows the IPF colour map of the martensite for the same area as Figure 5.37. By looking at the phase map, a few $\alpha'$-martensite regions seem to cross the austenite grain boundaries. However, the IPF map shows that on each side of the boundaries, $\alpha'$-martensite exhibits a different orientation.
It is noteworthy that K-S orientation relationship gives four separate groups of martensite variant pairs (in a given packet) with different misorientation angles (Table 2.2), whereas the variants in N-W OR are related by [011]/60° rotation. Therefore, comparing the measured and calculated misorientation between different variants in a given packet can be used as an indication to weather the orientations is closer to K-S or N-W orientation relationship. Figure 5.39 represents the IPF colour map of lath martensite in AISI301steel at 50% strain. The colours in this Figure correspond to the crystallographic orientation of α’-martensite normal to the observed plane, as indicated by stereographic triangle in the inset; the tensile direction is parallel to the horizontal direction. The austenite phase is indicated by gray scale band contrast map. The four misorientation groups between adjacent α’-martensite variants in a packet are included in the boundary analysis with axis/angle deviation maximum to 5°.
Figure 5.39-IPF colour map of $\alpha'$-martensite, austenite phase is showed in gray.

The $\langle 111 \rangle$ 60° misorientations are shown by red, $\langle 110 \rangle$ 49.5° by yellow, $\langle 011 \rangle$ 60° by lime green, and $\langle 011 \rangle$ 10.5° by aqua lines. The numbers in the brackets shows the percent of misorientation relationship to the total misorientations. The $\langle 111 \rangle$ 60° and $\langle 110 \rangle$ 49.5° boundaries consist 9.73 and 1.22% of the total misorientations respectively. It should be noticed that the four
groups of misorientations which are included in the analysis are misorientations between the adjacent \( \alpha' \)-martensite variants in a single packet, with the same habit plane. Apparently within a band, one or two variants are often formed out of six possible variants with the same \{111\} plane and the misorientations between the neighbouring variants are mainly \(<111> 60^\circ\) or \(<110> 49.5^\circ\).
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SUMMARY AND CONCLUSIONS

The volume fraction, morphology and crystallography of spontaneous (thermal) and deformation induced lath martensite in three austenitic stainless steels were examined by means of optical microscopy, Ferritescope and FE-SEM/EBSD. The following conclusions can be drawn:

1. Sample preparation is a very important step in the metallography of austenitic stainless steels. Electrolytic polishing or electropolishing is strongly recommended to obtain a high quality, deformation free surface. Though, inclusion attack may be encountered, and second phases may be attacked preferentially.

2. When etching is performed correctly, optical microscopy is a rather quick and useful tool for microstructure analysis of the austenitic stainless steels in order to achieve the prior austenite grain size and twinning. Moreover, the morphology and volume fraction of the lath martensite which forms spontaneously and during plastic deformation can readily be identified.

3. Ferritescope can be used as an easy and non-destructive, in situ method for quantitative measurement of $\alpha'$-martensite content. The Ferritescope reading must be converted to actual $\alpha'$-martensite content by using a calibration curve. The results of this study confirms that the correction factor of 1.7$^{[75]}$ can be successfully used for this conversion.

4. Strong temperature and composition dependence of austenite stability was observed. The AISI301 steel exhibited the least austenite stability in comparison with the AISI204Cu and 304 steels. Extensive martensitic transformation was observed in AISI301 type at all the test temperatures whereas in the more stable AISI304 steel the transformation only occurred at high strain levels and low temperatures. AISI204Cu showed intermediate austenite stability regarding the strain–induced martensitic transformation within the investigated grades.
5. The experimental in-situ $\alpha'$-martensite measurements by Ferritescope were fitted fairly well with the Olson and Cohen model $^{[6]}$. The values of fitting parameters, especially $\beta$, extensively depend on the martensitic transformation saturation, which in most cases is beyond the tensile strength of the test material.

6. LOM reveals that the morphology of the strain induced $\alpha'$-martensite consists of sets of thin parallel needles that go through the parent austenite grain and stop at the grain or annealing twin boundaries. Large amount of $\alpha'$-martensite can be seen at the intersection of shear bands. However, considerable amount of $\alpha'$-martensite is also observed when only one set of bands is activated. Therefore, the volume fraction of $\alpha'$-martensite formed is more related to the number of activated shear bands rather than their intersections, as it is assumed in the Olson and Cohen model.

7. EBSD proved to be a useful tool for analyzing the complex microstructure of spontaneous and strain induced lath martensite in stainless steels despite a high density of lattice defects in the martensite crystal and local texture. Crystallographic and morphological features of the lath martensite were identified of the order of prior austenite grain size or larger. However, because of high fraction of non-indexed pixels, the EBSD analysis may not be used to evaluate the volume fraction of martensite at high strain levels.

8. It was showed that the misorientations at phase boundaries are mostly corresponding to K-S OR when the $\alpha'$-martensite is formed upon quenching or deformation.

9. Within a band, one or two variants are often formed out of six possible variants with the same $\{111\}$ plane and the misorientations between the neighbouring variants are mostly $<111> 60^\circ$ or $<110> 49.5^\circ$. 
**Future Work**

Variant analysis of the lath martensite could be carried out by means of pole figures and inverse pole figures (IPF) on the EBSD data. The packet and block boundaries might be determined from the orientation analysis. The possible variant selection with respect to size of the packet, prior austenite grain size, plastic deformation, chemical composition, and temperature could also be investigated.

Proper indexing of ε-martensite could be achieved by optimizing the EBSD acquisition process. The transformation sequence and the $\gamma/\varepsilon$ and $\varepsilon/\alpha'$ orientation relationships may be investigated.
ACKNOWLEDGMENTS

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Reza Naraghi

Stockholm, August 2009
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Appendix 1

Guidelines for Data Processing

Abstract

All the martensite measurements during tensile testing are saved in ASCII text files which could be read in for example Notepad. These files have the “RAW” and “DAT” extensions. In order to utilize the measured data some numerical treatment is necessary to obtain the true stress, true strain and particularly the correct martensite content. At the moment, an Excel macro, “OMM_Data_Processing_34” is available to do so and it exports the true stresses, true strains, corrected martensite contents, and temperature. Furthermore, some illustrating graphs are drawn. The Excel macro is almost self explanatory; nonetheless some of the calculation steps and functions are described below.

Loading the data

First of all it should be noticed that in order to use the macros, the Macros & ActiveX controls of Excel must be enabled. The compatibility of the decimal and thousand separators in the Microsoft Excel with the saved dat-file must be checked before loading the data. It is possible to use .(dot) instead of ,(comma) as the decimal separator during saving the measured data in tensile tests operating software, “Tensile 2.19”. This option can be found in Setup> Service menu of that software.
Appendix 1

One can also change the Microsoft Excel decimal separator in the following ways depending on the software version.

Microsoft Excel 2007  Excel options> Advanced> Editing options
Microsoft Excel 2003  Tools> Options> International

After loading the measured dat-file, a window according to Figure 1 appears. In the “testing parameters” panel, the initial and final dimensions of sample as well as the deformation velocity and the starting temperature can be seen and modified.

In the “correction values” panel, the thickness correction parameters \( (a_0 \text{ and } a_1) \) and the Villari constant \( (b_v^e) \) are already set to their default values. The Villari-constant will be calculated and can be replaced afterwards. The thickness correction parameters also can be changed directly in the data processing excel sheet. The Villari-effect constant and the thickness corrections are described in detail in their corresponding sections.

Figure 1. Load data window.

Villari-Effect

Susceptibility of a material changes when subjected to a mechanical stress; this effect is called the Villari effect or inverse-magnetostrictive effect.

The data required for calculating the Villari constant are measured during unloading the sample.
In the Villari worksheet, see Figure 2, the first and second column are the measured Force[kN] and martensite content, $M\sigma [%]$, during unloading. The third column is true stress, $\sigma[\text{Mpa}]$, calculated by the following equation:

$$\sigma[\text{Mpa}] = \text{Force[kN]} \times 1000 / A_{\text{end}} [\text{mm}^2]$$

(1)

where the $A_{\text{end}}$ is the cross section area after unloading which is calculated by $w_{\text{end}} [\text{mm}] \times t_{\text{end}} [\text{mm}]$. The 4th column is CF-1 which is defined as:

$$CF - 1 = \frac{M_{\text{max}}}{M_\sigma} - 1$$

(2)

where $M_\sigma$ is the measured martensite content and the $M_{\text{max}}$ is the maximum martensite value read after unloading.

The "least squares" method is used to calculate a straight line that best fits the CF-1 values plotted vs. $\sigma[\text{Mpa}]$ and the Villari constant is thus calculated as the slope of the regression line. Figure 2.

$$\text{Regress.} = b_0 \times \sigma[\text{Mpa}]$$

(3)

By clicking on “Calculate VC” button, the Villari constant will be calculated according to the mentioned method; however the “set VC manually” button facilitates adjusting the regression line manually.

Click on “accept VC” when satisfied.

**Figure 2.** Villari-effect worksheet.
Appendix 1

Process data

Click on “process data” button in the data processing worksheet after calculating the Villari-constant. A window like Figure 3 appears. The yield stress is automatically calculated as the 0.2% strain but here it can be adjusted furthermore manually. Click on the “more” button and increase the number of plotted points if needed to have better clarification. It should be mentioned that all of the data points below yield stress (starting point of plastic deformation) will not be processed and only the plastic deformation region is considered in the following data processing.

![Figure 3. Selecting the yield stress.](image)

After selecting the yield point the same window appears to select the end point of data.

The first column in processed data table is $\varphi_1$ or the true plastic strain. Equations 4 and 5 show the relation between the engineering strain, $e$, and the true strain, $\varepsilon$:

\[
e = \frac{l - L}{L} \quad (4)
\]

\[
\varepsilon = \ln(1 + e) \quad (5)
\]
Similarly the true plastic strain, $\varphi_1$, is calculated by the following equation:

$$\varphi_1 = \ln \left[ 1 + \left( \frac{RDL - RDL_{corr}}{RDL_{corr}} \right) \right] \quad (6)$$

where the $RDL_{corr}$ is the extensometer gauge length at the yield point.

True stress, $k_f$, is calculated by the Equation 7:

$$k_f = \frac{\text{Force} [kN] \times 1000}{w_0 [mm] \times t_0 [mm] \times \exp (\varphi_1)} \quad (7)$$

$w_0$ and $t_0$ are initial width and thickness respectively.

The third column in the processed data table is Thickness [mm]. The thickness is not measured during the test but it can be calculated by the equation below:

$$Thckn.[mm] = t_0 [mm] \times \exp \left( \frac{-\varphi_1}{1 + R} \right) \quad (8)$$

where

$$R = \frac{\ln \left( \frac{w_0}{w_{end}} \right)}{\ln \left( \frac{t_0}{t_{end}} \right)} \quad (9)$$

The 4th column shows the $M_{Vill.}$ [%] which is the calculated martensite content by considering the Villari-effect:

$$M_{Vill.} = M_{measured} \left( 1 + b_v \times k_f \right) \quad (10)$$

**Thickness correction**

The Thickness of measured object affects the values measured with the Ferritescope. The effect of thickness can be corrected by multiplying the measured values with the corresponding correction factor, see Figure 3.

![Figure 3. Correction factor, $C_t$, vs. thickness of the measured object [1].](image)
Appendix 1

Figure 1 is provided in the FERITSCOPE® MP30 Operating manual as the correction factor for influence of the thickness of the measuring object. In this Excel macro, the above thickness correction factor, $C_t$, is estimated by the Equation 8:

$$C_t = 1 + \left[ a_0 - \left( \frac{a_1 \ln (M_{meas} \times \text{Thickn.}^2)}{\text{Thickn.}} \right) \right]$$  \hspace{1cm} (11)

where the $a_0 = 0.10435$ and $a_1 = 0.01976$ are set as the default values but can be adjusted in the “correction values” panel during loading the dat-file or later on at the “Correction constants” cells above processed data table.

As a consequence the corrected martensite content, $M_{corr.} \ [%]$, considering both Villari effect and thickness effect is calculated by the Equation 4:

$$M_{corr.} = C_t \times M_{Vill.}$$  \hspace{1cm} (12)

Summary

The thickness correction factors as well as Villari-effect are considered in this version. However it has been found that the Ferritescope underestimates the actual martensite content, therefore the evaluated martensite content must be multiplied by 1.71, which is a factor established by Talonen, J., et al. \cite{2}. This correction is not made in this version of Excel macro and is left for the user.

References