Microstructural Characterization of
Austenitic Metastable Steel

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Abstract

In this work the microstructure of the austenitic metastable steel Sandvik Nanoflex™ is investigated. This Sandvik Nanoflex™ is a high alloyed stainless steel. This led to some difficulties in microstructural examinations. Therefore a good sample preparation procedure needed to be set up. A method with which the samples could be analyzed proved to be SEM (Scanning Electron Microscopy) with the additional technique of OIM (Orientation Imaging Microscopy).

The sample preparation method includes a step of diamond sawing followed by extensive grinding and polishing and finally an electrolytic polishing treatment is applied. With this procedure phase determination within the samples cross section is made possible by the use of OIM. Also a preferred crystallographic orientation (texture) was detected within the parent austenitic phase. However no relation could be found between this preferred orientation and the transformation of austenite into martensite. The obtained results have been verified and correlations between the microstructural images obtained by optical techniques and OIM measurements were found. Electrolytic etching of the material did not lead to useful optical images.
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1 Introduction

Almost everything around us these days is produced in some way or another. Many different materials are used. Some obvious examples are wood, plastics, textiles and metals. In this case we are only interested in a certain type of metal. The metal that’s been investigated is used by Philips DAP (Domestic Appliances and Personal Care) in the production of their electrical shavers. These electrical shavers have to be capable of withstanding a lot. First of all, people shave themselves usually wet, this means that a corrosion resistant metal is needed. Other important properties of the metal are that it has to be easily deformable during fabrication, and when finished it has to be very hard, to prevent deformation of the final product. A metal that fulfils these needs is the Sandvik Nanoflex™. This metal is being used by Philips in the production of shaver cups, cutters and springs for several years. A shaver, cup and cutter are depicted in figure 1.1. The metal has good qualities for these applications. However as in every industry, improvements are to be made to stay ahead of the competition. This means that the shavers need to get better and better. The smoother and quicker the shave, the better. This will subsequently lead to different demands from the materials used. At present the shaver cups have to be thinner, in order to obtain a smoother shave. However making thinner cups is not an easy task. During metal forming several things occur. The metals are deformed plastically. Plastic deformation always involves a part of elastic deformation as well, which will spring back when the applied load is removed.

This is general for all metals, however when manufacturing Sandvik Nanovlex™ several more things will occur. In this work a certain aspect is investigated, which deals with phase transformations within the metal. It is important to understand the material behavior during the deformation and transformation in order to be able to do numerical simulations and predict this behavior. Therefore experiments have been conducted. The goal of the experiments was to discover a way for analyzing the different phases of the steel within the material and from these results establish some predictions about the formation of these phases. This is why the microstructures have been investigated thoroughly.

First the characteristics of Sandvik Nanoflex™ will be elaborated and the phases of interest of steel will be explained, with their differences. Hereafter the experimental techniques with which the analysis were carried out will be discussed. Then the sample preparation will be brought to light and the results obtained will be explained. This procedure of sample preparation had to be developed from scratch for there was no previous experience with these types of stainless steels within the Mechanics of Materials group at the TU/e yet. Then some recommendations will be made for future research and finally the conclusions will be drawn.

Figure 1.1 Electrical shaver from Philips and two of its components. On the left the final product, in the middle a shaver cup and on the right a cutter.
2 Material Sandvik Nanoflex™

The investigated material is the so-called Sandvik Nanoflex™ steel. This is a high alloy metastable austenitic stainless steel. The chemical composition is shown in table 1. Compared to more common steels, this steel contains relatively high amounts of chrome, nickel, molybdenum and even some titanium. This causes difficulties in examination resulting from the imperfections in the iron lattice due to interstitial atoms. The different elements can bond with each other, which makes it hard to determine the specific influence of each alloying element on the thermo-mechanical properties of the material. This means that for example the influence due to the presence of chromium on the eutectoid temperature of a steel, cannot be superpositioned on the eutectoid temperature of Sandvik Nanoflex™.

Table 1: Chemical composition of Sandvik Nanoflex™ [1]

<table>
<thead>
<tr>
<th>Element</th>
<th>C+N</th>
<th>Cr</th>
<th>Ni</th>
<th>Mo</th>
<th>Ti</th>
<th>Al</th>
<th>Si</th>
<th>Cu</th>
</tr>
</thead>
<tbody>
<tr>
<td>Weight percentage</td>
<td>≤0.05</td>
<td>12.0</td>
<td>9.0</td>
<td>4.0</td>
<td>0.9</td>
<td>0.40</td>
<td>≤0.5</td>
<td>2.0</td>
</tr>
</tbody>
</table>

2.1 Phase Transformations

As mentioned before Nanoflex™ is a metastable steel. Metastable implies that the metal is in a quasi stable state of equilibrium. It is not in its most preferred phase at the given temperature. However the available energy is too low to instigate a quick transformation. Energy can be supplied to the metal in different ways. There is of course the thermal trigger to start the transformation, but it is also possible to initiate the transformation by applying an external mechanical load on the material. This is shown in figure 2.1.1. Steels that do transform when deformed mechanically are labeled TRIP-steels (TRansformation Induced Plasticity).

![Figure 2.1.1 Schematic diagram showing the free energy change for a martensitic transformation.](image)

At $T_0$ the two phases are in thermodynamic equilibrium. No transformation occurs because of the strong nucleation barrier to the formation of martensite. This barrier can be overcome by significant supercooling. The temperature at which transformation starts upon cooling is designated $T_S$ and is located considerably lower than $T_0$. At this point there is enough chemical free energy present to overcome the barrier and to trigger the transformation, due to thermodynamic causes. In the major part
of the region between $T_s$ and $T_0$, for example at $T_1$, it is possible to achieve the required free energy needed for transformation, by applying an additional external stress. This mechanical work is denoted as $U[3]$.

The phase transformation that is of interest in this research is the transformation from austenite to martensite within the Sandvik Nanoflex™. During this transformation the FCC (Face Centered Cubic) crystal lattice of austenite experiences a polymorphic transformation to the BCT (Body Centered Tetragonal) crystal lattice of martensite, which is distinctly different from BCC (Body Centered Cubic) ferrite. This BCT martensite is simply a body-centered cube that has been elongated along one of its dimensions [2]. During this transformation no long-range diffusion occurs. The transformation is so quick that it is considered time independent; the martensite grains nucleate and grow at a very high speed.

An important characteristic of the austenite to martensite transformation is that the atoms will maintain their relative relationships during the transformation. This transformation involves a contraction of approximately 20% of the parent cell along one of its axes and an elongation of about 12% along its other two axes. This deformation is better known as the Bain distortion. Analysis has shown that this Bain distortion involves the smallest amount of atomic displacements and is therefore favored above other possible ways to create the martensitic crystal [3].

### 2.2 Properties of Austenite versus Martensite

Austenite and martensite differ significantly in their physical and mechanical properties. As mentioned already there is a difference in the crystals of both phases. Austenite has an FCC lattice structure and martensite has BCT. Other characteristics of martensite are that it is the strongest and hardest of all possible phases in steel, however it is also the most brittle one, it has in fact negligible ductility. There are actually more possible phases besides austenite and martensite for steels in general, such phases are pearlite, spheroidite, cementite, ferrite and bainite. However in Sandvik Nanoflex™ only austenite and martensite are present due to the chemical composition and the specific thermal treatment. The ductility and toughness of martensite may be enhanced at the cost of hardness by tempering the material. The strength and hardness of martensite are not thought to be related to its microstructure. These properties are attributed to the presence of many strong barriers to dislocation motion in the martensitic crystal structure. These barriers may be related to the effectiveness in hindering dislocation motion by interstitial carbon atoms, as well as by fine twin structures or by high dislocation density [3].

Austenite on the other hand is very ductile and its hardness is low compared to martensite. This means that metal in the austenitic state is easy to form. The martensite on the other hand is very hard and will practically not deform, which is ideal for the application in shavers.

Another distinct difference between austenite and martensite is that martensite is ferromagnetic whereas austenite is paramagnetic. This means that martensitic steel will be attracted by a permanent magnet and austenitic steel will not [1].
3 Scanning Electron Microscopy

Material analysis can be carried out in different ways. In the current work microscopic techniques were used. The instrument which proved most useful for the current research is the scanning electron microscope, or short SEM. The general principle of a SEM will now be elaborated. Then a special technique called orientation imaging microscopy, short OIM, will be explained.

A SEM consists of several essential features, these are [5]:
- an electron source
- a lens system
  - condenser lens
  - objective lens
- a detector
- a display system

A block diagram of a SEM is given in figure 3.1.

Figure 3.1 Block diagram of a SEM, showing the general layout of the essential components [10].

The electron gun is the source for electrons. A SEM works with a primary accelerating voltage of 1-30 kV. The electrons are focused on the specimen by condenser and objective lenses. When the electrons hit the surface of the specimen, several effects occur. These effects can be measured quantitatively with a SEM. The incident electrons from the electron gun will cause different types of electrons to leave the specimen, as shown in figure 3.2. These different types of electrons all contain specific information about the specimen.
Figure 3.2 Reactions of electrons with solids.

From the secondary electrons information about the topography of the specimen can be obtained. Using X-ray spectrometry the elements present in the material can be determined. Backscattered electrons provide information about the crystallographic structure of the specimen. The energy that is lost when the electrons interact with the specimen is transformed to heat and will be conducted through the material. The transmitted electrons are not of interest in this case. Transmitted electrons can be detected with Transmission Electron Microscopy (TEM) or Scanning Transmission Electron Microscopy (STEM). The elastically scattered electrons can be used for dark field imaging, whereas the transmitted, undeviated beam can be used for bright field imaging. The inelastically scattered electrons can be used in TEM to create Kikuchi-diagrams [7].

The electrons can be detected by a suitable detector. There are different detectors for different types of electrons. Finally the detected signal is sent to a display system such as a computer, on which the results can be analyzed.

3.1 Orientation Imaging Microscopy

An additional technique which can be used in combination with the SEM is the orientation imaging microscopy (OIM). This technique makes use of the backscattered electrons. These electrons can be detected by an EBSD (Electron BackScatter Diffraction) detector. This way the orientation and crystallographic structure of individual grains in a sample can be studied.

When irradiating the specimen straight from the top, only a small amount of backscattered electrons will be released. By tilting the sample over 70° the amount of emitted backscattered electrons increases. The EBSD detector has to be placed close to the specimen in order to achieve good results.

When the electrons hit the phosphor screen of the detector, patterns are created that can be related to the crystallographic planes. These patterns are called Kikuchi patterns or Electron BackScatter Patterns (EBSP). An example of such a pattern is shown in figure 3.1.1.
From such a pattern several properties of the measured crystal can be deduced. The width and intensity of the bands are directly related to the spacing between the atoms in the crystallographic planes, the angles between the bands are directly related to the angles between the planes. The symmetry of the crystal lattice is also reflected in the pattern. The crossings of these bands are called poles. With the help of the bands and poles the identification of the crystal orientation is possible [4].

Behind the phosphor screen a CCD (Charged Coupled Device) camera is set up which transmits the patterns to the computer. This CCD camera does not have a photographic film in its focus point but rather a chip, which consists of a series of capacitors that are coupled by electronic switches. The advantages of a CCD are that it is much more sensitive to light than the conventional film and the signal is already digital which enables quick computer analysis.

The image is obtained from a number of lattice planes (approximately 8) in the specimen. By the use of Bragg’s law (formula 3.1) the originally three dimensional distribution of low-loss electrons is transformed to a set of two dimensional planes, along which the electrons propagate (figure 3.1.2). Figure 3.1.2 also shows how the different crystal planes of a sample can be measured.

\[ 2d_{hkl} \sin(\Theta_{hkl}) = n\lambda \]  

(3.1)

In which \( d_{hkl} \) is the lattice spacing, \( \Theta_{hkl} \) the scattering angle, \( n \) the order of diffraction and \( \lambda \) the wavelength of the electron (for 30 kV around 0.007 nm). Here the indices \( hkl \) represent a set of lattice planes. The two planes in figure 3.1.2 cut a detection screen positioned next to the sample resulting in a Kikuchi-band.

By using dedicated software such as OIM Data Analysis 4.5 from EDAX these bands and their angles can be translated to different crystal structures and orientations. The program will choose between the given possible phases, in this case between Iron gamma (=austenite) and Martensite. A crystallographic orientation and microstructure map is produced by scanning the sample. From this scan the grain size, the crystallographic lattice structure and grain orientation can be derived.
4 Sample Preparation

Samples that can be investigated in the SEM and subjected to OIM need to be sufficiently small because there is only a small compartment in the machine in which to place the samples. We had a large piece of metal sheet, roughly about 3 by 0.4 meters. From this sheet samples had to be prepared.

The metal provided by Philips rested at Drachten for several months. Then it was transported to Eindhoven and rested there for a while. The samples were to be cut out and eventually, the final samples were cut when the metal had been in Eindhoven for about 3 months. The metal, as mentioned previously, is very unstable. When it is not at a sufficiently high temperature, where the austenitic phase is preferred, transformation can occur. This is due to the thermal effect that is discussed in chapter 2.1. Since the metal could not be stored under optimal conditions it is probable that deformation, or actually transformation that resulted in permanent deformation of the metal sheet, had occurred. The sheet of metal was no longer even straight; permanent deformation in the form of a wavelike pattern could be seen. This however does not mean that no research can be conducted. Although a certain amount of transformation may have already occurred in the material a good and reliable preparation and analyzing method can still be developed.

It was of special interest to investigate the difference between stress-assisted and strain-induced transformation. Therefore some bending experiments were carried out on the material with a special bending machine of the TU/e, which is capable of applying pure bending. Due to the difficulties met in sample preparation and analysis this difference could not further be examined. The details of the conducted bending experiments are therefore not included in this report. Moreover the results might have been distorted due to the state of the material (plausible thermodynamic transformation during several months).

4.1 Polishing Methods

In order to be able to perform SEM scans the surface quality of the specimens needs to be enhanced, by reducing the roughness. Therefore grinding and polishing protocols needed to be set up. To enable a polishing procedure, the samples had to be put in a holder. This was done by putting the samples in a small cup and then adding epoxy, which is a mix of Specifix-20 Curing Agent and Specifix resin 1:7. This epoxy is used because the hardening involves only a small temperature difference. The temperature does not go above 40°C. In a first attempt it was tried to polish the samples on silicon carbide paper from Struers to a #4000 (#4000 implies that the largest grinding grains on the paper are 5 μm) and finishing with a 1 μm diamond suspension polishing step with lubricant red on an MD-MOL. This gave nearly no results in OIM, the Kikuchi patterns were hardly visible. Then a method that was used to analyze martensite and austenite in a Fe-30% Ni was tried [9]. The main difference is that the last step done by electrolytic polishing with A2. The chemical composition of A2 is listed in appendix A. This gave better results however the results were not yet satisfactory, still many measured points gave no solution. Upon further research and testing the conclusion was drawn that electrolytic polishing with A3 gave the best results, as might have been expected from the Struers manual [6]. The chemical composition of A3 is listed in appendix A as well.

The effect of both A2 and A3 was tested on large areas of the material, that is in plane of the metal sheet. A good voltage for electrolytic polishing was found to be 30 V. The goal of the current research was however to investigate the microstructure in the thickness direction of the metal sheet, and not in plane. This meant another complication, which was how to remove the metal strip from the epoxy upon polishing. When this epoxy is not removed the specimen would charge up in the SEM and no measurements could be carried out. Therefore the samples were put in clay, which could be removed before polishing. After polishing the strips were sawed out of the polymer manually. Finally the procedure stated below was used and this gave good results.

Procedure:
- Put the sample in a small amount of clay
- Put the sample and clay in a cup and add Specifix-20 Curing Agent and Specifix resin 1:7
- Let the epoxy harden for at least 12 hours
- Remove sample, clay and epoxy from the cup
- Label the samples
• Remove clay and abundant polymer

• Grind and polish the samples in several steps with Struers grinding tables: 
  #240, #500, #800, #1200, #2400, #4000, 1 μm diamond suspension on an MD-MOL with 
  lubricant red, 0.04 μm OP-A suspension 1:3 on an MD-CHEM
• Perform electrolytic polishing with A3 for 15 seconds 30 V flow rate 11 with a cap of 1 cm² 
  on a LectroPol-5
• Saw the strips out of its polymer embedding carefully

4.2 First Attempt

The SEM that’s been used for all the OIM measurements is the (XL-30) FEG (Field Emission Gun) 
SEM Sirion from FEI. The settings for SEM were: 30 kV accelerating voltage, 15 mm working 
distance, scanning step size of 1 μm with spot size 6.

Small samples were needed (their length could not exceed 3 cm), so cutting of the material was 
required. However cutting, always leads to plastic deformation within the specimen. At first it was 
assumed that this deformation zone would be relatively small and that the affected area would be 
removed during grinding and polishing. Therefore it was expected that this cutting procedure would 
give satisfying results. Results obtained are shown in figures 4.2.1 and 4.2.2.

Figure 4.2.1 Scan of a sample’s cross section that was not bended and cut on the H.M. JBS-1250/3.0, 
then prepared. Austenite=red 56%, Martensite=green 44%.
Figure 4.2.2 Scan of a sample’s cross section that was bended to 180° and then saved on the Mössner Rekord SM/420, then prepared. Austenite=red 70%, Martensite=green 30%.

Both results were out of the expected range, especially the high content of martensite in the sample that had not been bended in figure 4.2.1 (44% of martensite). Expected was an amount of not more than 10%. What is also striking is that the amount of martensite in the bended (deformed) sample appeared to be considerably smaller. This is not as would have been predicted. For the deformation caused by bending should have increased the nucleation and growth of martensitic grains.

Reasons for these phenomena were to be found. Several possible causes were investigated, these are: the influence of the accelerating voltage; the way in which the sample is scanned and finally the cutting procedure.
4.3 Influence of the Accelerating Voltage

The accelerating voltage at which the electrons are fired at the sample influences the depth to which they penetrate. In order to check if the applied voltage of 30 kV might have led to the unexpected results, one sample was scanned at the same region on 30, 20 and 10 kV. The results are shown in figure 4.3.1.

Figure 4.3.1 The same region of one sample scanned at different voltages; scan (a) was done at 30 kV, scan (b) at 20 kV and (c) at 10 kV. The lack of the black lines (caused by scratches on the surface) in image (c) is due to the auto-contrast option used in order to increase the number of good measured points. Red= austenite, green= martensite.

When looking at these three images one can clearly recognize the similarities. In fact there are hardly any differences in the measured regions of austenite. This cancels out the possibility that the accelerating voltage being too high caused the large amount of martensite detected.

4.4 Influence of the Scanning Direction

Another possible explanation might be that due to the roughness of the sample, different things could be observed from different angles. This could have influenced the measurements causing a seemingly high percentage of martensite. Therefore a sample was rotated 180° and then scanned again over the same area. The results are shown in figure 4.4.1 and 4.4.2, where the great coherence between the pictures can be observed.
Figure 4.4.1 Scan of a sample’s cross section. Austenite=red and Martensite=green. Notice the two large black spots, caused by an impurity on the surface such as a small particle of dirt.

Figure 4.4.2 Scan of the same sample as the one in figure 4.4.1 covering partially the same area. Austenite=red and Martensite=green. Notice again the two dust particles and observe the great coherence in the pictures.

The images show that looking from either site of the specimen does not explain the high martensite contents measured. A small miscalculation though can be due to imperfections in the tilt angle. This is also the reason why the figures 4.4.1 and 4.4.2 show some differences. The distortion in vertical direction is caused by an inaccuracy in the tilt angle. This inaccuracy is caused by the installation of the sample into the chamber of the SEM. The holder of the sample could not be perfectly aligned because
it is a thin sheet of metal which is difficult to insert perfectly straight. This problem can be solved by measuring the angles in the holder and stage of the SEM together optically and then using this angle for alignment in the SEM and OIM analysis.

### 4.5 Influence of the Cutting Procedure

When holding a permanent magnet at a surface of the material there was hardly any attraction. Closer examination showed however, that when the magnet was near the cut edges, that there was a magnetic attraction, indicating that there was a substantial amount of martensite present. This can be explained by the shear effects that are involved into the cutting. Since it was not possible to completely avoid these cut effects an attempt was made to minimize them. Therefore the sample was first roughly cut on a large cutting machine (H.M. JBS-1250/3.0) (which was previously used for all the straight cuts), where now a strip of stronger material was placed on top of the Nanoflex™ sheet to distribute the pressing force evenly. Then on a small manual cutter from Gerver smaller strips were obtained. The metal was both supported on the top and bottom to furthermore reduce the cut effects. This way the metal sheet was cut to a strip of about 2.5 cm wide.

Still with a simple tool such as a magnet it was easily observed that a significant amount of martensite had formed on the cut edges. This is why the strips that were carefully cut were now sawed to their final size of approximately 1 cm high. The strips were sawed with a diamond circular blade on the Labcut 1010 with cooling fluid. Again with a simple magnet it could be checked that now the edges that were sawed showed much less magnetic response. Therefore it can be concluded that cutting, with its shear effects in the cutting zone, is a bigger problem than an abrasive method such as sawing. A result of an OIM scan of a sawed sample is depicted in figure 4.5.1.

![Figure 4.5.1 Scan of a sample’s cross section that was sawed to its final size and subsequently extensively grinded and polished. Austenite=red (91%) and Martensite=green(9%).](image)

Eventually a cause for the high martensite content was discovered. The cutting procedures used led to the formation of martensite. This also explains why there was even a slightly greater amount of martensite in the undeformed samples, compared to the samples deformed by bending, as discussed in paragraph 4.2 and shown in figures 4.2.1 and 4.2.2. The undeformed samples were cut and therefore a substantial cut zone was created while the bended samples were sawed roughly on the Mössner Rekord SM/420. An additional reason why the roughly sawed sample showed less martensite is because it was grinded more since the edge effect of the sawing was optically visible and this was of course grinded away. Both samples showed a martensite content which was greater than expected though. This meant that both techniques were inadequate. Diamond sawing definitely is a better way to slice the material in small samples.
5 Verification

Since now a procedure had been found that produced the results within the expected range, a good sample preparation protocol for the Sandvik Nanoflex™ was set up. Due to the time constraints there was no time left to investigate the strain-induced and stress-assisted phase transformation within the material. This can be done in future projects continuing upon the current results. What could still be done is to verify some of the obtained results and explain what can be retrieved from the measurements and where possibilities lie for more understanding.

Measurements are only useful when they can be trusted. Therefore verification of the obtained results is required. First, the process of indexing the patterns and the determination of phases will be discussed. Then it will be verified whether the obtained results are as would be expected, this is done by comparisons of OIM results with SEM and optical microscopy images.

5.1 Confidence Index (CI)

The Kikuchi patterns that are measured are characteristic for crystal structures. In this case we deal with FCC (austenite) and BCT (martensite). The program OIM Data Analyses that is used for analysis of the data uses a voting scheme. This program operates by giving votes for different possible crystal orientations that are compared to the measured bands in the Kikuchi pattern. Which ever of these possible crystal orientations is awarded with the most votes will be the accepted crystal orientation. This means that now both the crystal orientation and the phase are determined. Because a voting scheme is used this indicates that usually it cannot be stated for sure what orientation and phase is present. The one that fits best will be the result. Because several different choices compete with each other it is important to know whether the first choice is reliable or that maybe the second choice might be just as probable. Therefore a Confidence Index (CI) is also calculated. This index is calculated by subtracting the number of votes for the first and second choice and dividing this by the total possible number of votes from the detected bands. A map can be made of the confidence indexes as well, see figure 5.1.1.

![Image: Confidence Index (CI) map of the same sample and area as figure 4.5.1. The cold colors (blue, green) correspond to a low CI and the warmer ones (orange, red) to high CI values. Detailed information about the color coding is available in appendix B.](image)

The image shows that the overall CI is quite high. The average CI is actually 0.62 from a scale of 0 to 1. However the martensitic areas show a blue color. This means that the CI here is rather low. The question may arise whether this means that these areas are then maybe not martensite but might just as well be austenite, since the first and second choice are obviously very competitive. This is however not
the case. The first and second votes are in fact both for martensite in practically all cases, the orientations merely differ. This means that the phase determination is reliable, however the particular orientation of martensitic grains is not that sure. An example of these votes is listed in appendix C.

5.2 Fit Parameter

When indexing the patterns also a so-called fit parameter is determined. This is done by calculating where the bands on the Kikuchi pattern should appear based on the orientation obtained from the voting procedure. The fit parameter defines the average angular deviation between the recalculated bands and the detected bands. A map of this fit parameter is shown below in figure 5.2.1.

The values for the fit parameter range from 0.038° to 3.977°. The green color however indicates a maximum fit of 1.613° and the blue is below 1°. This means that the fit between the recalculated and detected patterns is quite good. There are two more remarks that can be made about this image. The martensite in general shows a worse fit than the austenite. This is due to the irregularities of the martensite surface and also due to the distortion which is more abundant in the martensitic grains. What can also be seen is that on the top and bottom edge the fits, as well as the confidence indexes, deteriorate. This can be explained by the roundness of the edges of the sample. This will be further discussed at the end of this chapter.

5.3 Comparison with SEM Images

In order to determine whether what is measured by OIM would also be expected from other methods of microstructural investigation, OIM results are compared to both SEM scans and optical microscopy images. A SEM image is made of the same area on which an OIM scan was carried out. The results are shown in figure 5.3.1.
By taking the impurities as reference points the different areas of both images can be compared. When looking at the scratch in the right image one sees smooth bands above and below it. While at the scratch there is a slight increase of the spike shaped contours. These spike shaped contours may indicate the presence of martensite [2]. If indeed the spikes are characteristic for martensite this should show a concentration of martensite at the scratch. And consequently on the smoother surface, austenite would be expected to be the dominant phase. Looking at the phase map tells us that as expected there are two bands, one above and one below the scratch.

Looking at the dirt particle on the left of the image on the right of figure 5.3.1, a large smooth surface surrounds it and there is a sort of bulb just right of it. Comparing this with the OIM scan, again the correlation appears to be quite good. The other dirt particle, the most right one, seems to be enclosed by a smooth surface, except below it. Comparing this SEM image with the OIM result shows us that there actually is martensite on the left and right of this black spot.

From these comparisons it can be concluded that the more irregular surfaces and spike forms present are usually an indication of the presence of martensite at that point. At smoother surfaces austenite will generally be the dominant phase. Looking at the SEM image or an optical image for that matter, will not be sufficient to exactly determine the amount of martensite and austenite, but it can give a good indication.

### 5.4 Comparison with Optical Images

The rougher parts on the SEM image, as in figure 5.3.1 often show distinct spiked shapes. When looking at the material with an optical microscope, these shapes can also be distinguished. An optical image made with the Axioptan 2 from Zeiss is shown in figure 5.4.1.
Figure 5.4.1 Optical image of a sample’s cross section showing spike shapes, which indicate the presence of martensite.

This means that a quick check with an optical microscope can give an indication about the amount of martensite present. For detailed information OIM scans are required. Interesting regions on the surface can already be spotted optically.

It was tried to verify the results by external electrolytic etching of the material with oxalic acid. (10% solution for 60 seconds at 15 V, as is a standard Struers etching method for stainless steels in the LectroPol-5). This etching should reveal the grain boundaries of single grains and especially a difference in height would be expected between the austenite and martensite. A result of an etched sample is depicted in figure 5.4.2. Here the PLμ 2300 from Sensofar is used, this is an optical imaging profiler.
Figure 5.4.2 Optical image of a sample’s cross section etched with oxalic acid, dimensions of the image are 255 μm by 185 μm.

From this figure however not much information can be obtained. There is a difference in height created by etching, but the different phases in the metal can not be distinguished. Oxalic etching of this material might be examined in future work, on the other hand, different etching methods could also be tried. Methods that have showed useful for this material are the chemical etchants Lichtenegger and Blöch and Fry [8].
6 Results and Further Possibilities of OIM Analysis

When looking at all the results in general, one could notice the horizontal bands of martensite. These seem to occur in the middle of the cross section. The bands are observed in optical microscope images as well. An image made with the Axioplan 2 is shown in figure 6.1.

![Optical microscopy image of a cross section of Sandvik Nanoflex™ sheet metal.](image)

Figure 6.1 Optical microscopy image of a cross section of Sandvik Nanoflex™ sheet metal.

The sample that is shown in figure 6.1 is polished according to the procedure described in section 4.1. As mentioned the spike like patterns indicate the presence of martensite. In figure 6.1 the horizontal bands are quite obvious.

On the OIM scans however the martensite does not seem to be that well concentrated to the middle all the time. For example in figure 6.2 the martensite band seems to be near the top instead. The reason for this is that the electrolytic polishing has more effect on the edges. The edges are polished more thorough due to the flow of A3 around it. In fact the thickness of the sheet decreased from 450 \( \mu m \) to 380 \( \mu m \) in the specimen of figure 6.1 due to electrolytic polishing. This also resulted in the edges being not very sharp, but rather round. That this effect is quite substantial can also be seen from figure 6.1.
In OIM the sample is tilted over 70° this means that one of the rounded edges will still emit low-loss backscatter electrons which are caught by the detector, whereas the other edge will not. This could explain why the band of martensite does not show up that well at the middle as was expected. To check this assumption a sample is scanned from one side and then rotated over 180°. The result is shown in figure 6.3.
From figure 6.3 it is obvious that on both the left and right pictures the martensite is concentrated from the middle to near the top of the sample. However the sample was rotated 180°, this implies that the middle of the scanned area is not the middle of the sample itself. The first reason for this as mentioned is the rounding of the edges. Another reason is that the tilt angle is not exactly determined. The program works with a tilt angle to recalculate the areas to the original flat surface. This also explains the change in dimensions of different areas for the left and right picture. The percentage of martensite measured is therefore also influenced by this factor. Therefore it is very important to know the tilt angles precisely for exact measurements of martensite content.

To summarize this means that the band of martensite which seemed to be so near the top in figure 6.2 is actually closer to the middle than it seems. This is in agreement with what would be expected from an optical image as is shown in figure 6.1. The martensite is indeed concentrated towards the middle. Other studies on thermally induced nucleation of martensite in the Sandvik Nanoflex™ showed this phenomenon of martensite band formation as well [12].

The reasons for this concentration of martensite in horizontal bands near the middle are however still not fully understood. Possibly the hot and cold rolling during fabrication could be a cause. Another possible explanation might lay in the local chemical composition of the material. Nickel tends to preserve the austenitic lattice structure at room temperature [13]. It may be interesting to investigate the distribution of the elements, especially nickel, across the cross section of the material. Other explanations could lay in the orientation of the parent austenite in which the martensite forms. Also the typical grain sizes of the parent austenite may have a relation to faster transformation.

Besides the by now familiar phase plots that have already been shown in previous chapters there is much more information that can be obtained from OIM measurements. Figure 6.4 shows on the left one of the phase plots and on the right an image quality map.

![Figure 6.4](image.png)

**Figure 6.4 On the left a phase plot showing austenite (red) and martensite (green) distribution, 90% and 10% respectively. On the right is an image quality map (IQ map) of the same region.**

The image quality (IQ) parameter describes the quality of an electron backscatter diffraction pattern. The image quality depends on the material and its condition, it is not an absolute value but rather a function of the technique and materials used to index the pattern. Crystal lattice distortions within the diffracting volume will produce lower image quality. A low image quality is shown dark. As can be
seen from figure 6.4 the dark areas in the IQ map correspond with grains of martensite. The lower image quality in these martenritic areas can be explained by the larger distortion within these grains compared to austenitic ones. From this IQ map the individual grains within the sample can be distinguished. These are best visible for the austenite grains, since the image quality is good in the austenitic regions. In an IQ map the grain boundaries are visible. When the electron beam is situated near a grain boundary the diffraction volume may contain both crystal lattices separated by the boundary. Thus, the diffraction pattern will be composed of a mix of both patterns leading to a lower quality pattern [11]. Since the IQ is in general quite good this tells us that the preparation method of polishing and grinding is adequate for preparing Sandvik Nanoflex™ for SEM and OIM measurements.

It is difficult to say anything about the distribution of grain sizes in this picture. The sizes of the individual grains may be somewhat distorted resulting from the inaccuracy in tilt angle, but nevertheless it is hard to distinguish a distribution in size. The average grain diameter here is $3 \mu m$.

### 6.1 Orientation Analysis

Of course Orientation Imaging Microscopy, as the name would suggest, can be used for analysis of the crystal orientations present in samples. Another important one is that an Orientation Distribution Function (short ODF) can be calculated. This ODF gives information about the texture of the material. A pole figure shows only a part of the ODF, it is only possible for very simplistic textures to derive the texture of a sample from just a pole figure [7]. From the distribution of crystal orientations a pole figure can be made. For the current material a measured pole figure is depicted in figure 6.1.1.

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**Figure 6.1.1** Pole figures showing the distributions of the crystal orientations.

A pole figure contains information about the coherence in crystal orientation. As can be seen in figure 6.1.1 the martensite grains do not possess a specific preferred orientation whereas for the austenite a pattern is distinctly visible. The lack of coherence in orientation for the martensite grains may be explained by the unreliability of orientation determination in OIM as mentioned previously. However there may also be no distinct preferred orientation at all. The orientation determination of austenite was however much more reliable and therefore the pole figure of austenite is also reliable and there indeed is a preferred orientation in the austenite grains. It is also possible to depict this information in a density pole figure. An example is shown in figure 6.1.2.

---

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Figure 6.1.2 Density pole figure of austenite without any symmetry enforced on the left and with Orthotropic (rolled sheet) symmetry enforced on the right.

This orientation distribution is consistent over the entire cross section. The top, middle and bottom parts of the cross section show the same patterns as is shown in appendix D. Because these pole figures do not show a difference in the middle of the sheet cross section, it is not possible to ascribe the bands of martensite in the middle to a different orientation of the parent austenite.
7 Recommendations for Future Research

The Sandvik Nanoflex™ is still far from being fully understood. In order to improve our understanding future research is required. From the current project many lessons can be learned. The results tell us that several things should be kept in mind when doing analysis of this material. In a future research it should be possible to investigate the stress-assisted and strain-induced transformation of austenite into martensite. This means that bending experiments will have to be conducted.

In the first place it is very important to know the condition in which your material is. The material used in this project was way past its “expiration” date. Many things may have occurred during the months it rested. These effects can not be fully determined and should therefore be avoided at all cost. Receiving fresh material from Sandvik is essential to ensure trustworthy results.

Then there was the problem with the cut effect. It is possible to use the method used here, which is diamond sawing of the strips. It may however be preferable to investigate the use of spark erosion or laser cutting as well. These techniques have a heat affected zone. It is therefore important to investigate the penetration depth of this first. This can be done by cutting a small strip and then instead of polishing the side in which we are generally interested, grind thoroughly and then polish it perpendicular to this side. Then the martensite content can be measured from the affected edge towards the middle and the size of the affected zone can be determined. This is of course also a good experiment for diamond sawing. Also for diamond sawing of bended strips a solution will need to be found for the mounting of the strips. In case of laser cutting or spark erosion it is easier to cut these bended samples.

Another problem which is still present is that of the rounding of the edges of the strips during electrolytic polishing. This effect should be minimized. A custom cup might lead to a solution but the problem here could be that the strips are too thin and thus the cut in the polishing cap might be that small that no flow will run at all and no polishing will occur. Therefore one can look into the possibilities of applying a second mask around the strip of metal instead.

The grinding and polishing method that was used is quite extensive. This procedure can probably be shortened or simplified. It is possible to experiment with less polishing steps, or it could be tried to polish the samples automatically on an automatic polishing machine such as the Struers TegraPol-21 in combination with a TegraForce-5 and TegraDoser-5.

Another important factor to look at is the mounting of the samples in the stage of the SEM and determining the exact tilt angle of the sample. This can be done in the current setup, but then the entire stage with sample will need to be measured optically in a microscope such as the PLµ 2300 from Sensofar. Then the measured angle can be incorporated with the specimen tilt and the dimensions of the grains measured should then be correct. It is however quite a hassle to remove the entire stage from the SEM. Therefore a different method would be preferable. It is possible to measure the angle this way once and then measuring the thickness of the cross section when tilted in the SEM visually and keep this length as a reference.

Currently no real optical verifications could be performed. Different etchants can be tried as mentioned before, these are Fry and Lichtenegger and Blöch. These can then be optically analyzed to verify what is measured in OIM.

In order to determine why the martensite appears to form particularly in the middle, the chemical distribution of elements along the cross section can be investigated. In this respect special interest should be paid to the distribution of nickel. Also further research into the orientation distribution may lead to more understanding. Full orientation distribution functions may provide new insights as they give more detailed information about the texture than pole figures.

Finally, remember to have a magnet with you all the time just in case. It is a quick check and will prevent you from doing useless and time consuming scans.
8 Conclusions

The current work has brought some new insights into the possibilities of examining the microstructure of Sandvik Nanoflex™. OIM has proven to be a technique with many opportunities for further research. A way to determine the different phases within the steel is established.

During the project several time consuming problems were encountered and dealt with. This has lead to a limitation of the research. The effect of stress and strain on the transformation of austenite into martensite could not be investigated. A problem encountered was that in early analysis the martensite content was higher than would be expected from literature. The reason for this high content proved to be the transformation of austenite into martensite caused by shear effects that are involved in the cutting procedures used. By selecting new cutting methods the results improved significantly.

One of the main results of the present work is that a procedure for the sample preparation of this stainless steel for OIM analysis is set up. The procedure consists of a sawing step, followed by different grinding and polishing steps and finally electrolytic polishing. The electrolytic polishing proved vital in order to obtain good electron backscatter patterns (EBSP). Without electrolytic polishing poor images were obtained due to a small amount of backscatter electrons properly detected. In general the austenitic areas gave better patterns than the martensitic ones. This is due to the hardness of martensite which complicates the preparation of these surfaces. The identification of the different phases was however reliable. The presence of martensite was also shown optically and these results correlated well with the OIM measurements. The orientations of the different grains were however not that easy to determine. The orientations of the austenitic grains are reliably determined, the determination of martensitic grain orientations is however unreliable. In the austenitic regions a preferred orientation was noticed, however no relation between this preferred orientation and the transformation could be found.

The conducted research has not only given some answers and solutions, but also resulted in new questions and openings for future work. In the future several things can be examined. Besides the stress and strain related transformation of austenite into martensite, one should look for the best suited way to cut the material to properly sized samples. In this respect it should be investigated how big the affected zones of different methods such as laser cutting or spark erosion are compared to diamond sawing. Another thing that should be investigated is the preferred nucleation of martensite near the middle of the sample in horizontal bands across the cross section as they were detected both optically as with OIM. Could this be related somehow to the crystallographic orientations or chemical compositions? Also optical verifications are still required; in this respect different, possibly chemical, etchants can be tried.

The Sandvik Nanoflex™ proved to be very unstable, a transformation had actually occurred while merely resting at room temperature. This also leads to the conclusion that fresh material is needed for more reliable experiments.
9 References

4. *SEM TECHNICAL NOTE, Electron backscatter patterns (EBSP)*, Philips Electron Optics
13. Key to Steel, *Austenitic Steels*
   http://www.key-to-steel.com/Articles/Art104.htm Visited May 2006

9.1 Consulted Literature


Struers.com, *Metallographic preparation of stainless steel*

SEM.com, *Scanning Electron Microscopy*

FEIcompany.com, *All you wanted to know about Electron Microscopy...*
10 Appendices

10.1 Appendix A

Chemical compositions of the used electrolytes [14]

A2

78 ml Perchloric Acid
90 ml Distilled Water
730 ml Ethanol
100 ml Butylcellosolve

A3

60 ml Percholoric Acid
600 ml Methanol
360 ml Butylcellosolve
2 ml Vogel’s Sparbeize
### 10.2 Appendix B

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### 10.3 Appendix C

Voting records for two martensite patterns and one for austenite.

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10.4 Appendix D

Pole figures of different selected areas of a sample’s cross section.

Figure 10.4.1 Pole figures of the entire measured area

Figure 10.4.2 Pole figures of the top of the measured area
Figure 10.4.3 Pole figures of the middle of the measured area

Figure 10.4.4 Pole figures of the bottom of the measured area