Characterisation of super duplex stainless steel by optimisation of EBSD parameters

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Abstract. Electron backscatter diffraction (EBSD) was used to characterise the microstructure of a super duplex stainless steel (SDSS) alloy Zeron 100 and the intermetallic phases sigma and chi in this material. The material was analysed in three conditions; as-received, heat-treated at 900 °C for 8 min and heat-treated at 750 °C for 4 h. Heat treatments were performed to introduce the intermetallic phases in the steel. It is difficult to get reliable indexing of the sigma and chi phases due to noisy and overlapping patterns. To get the best results, the Hough transformation parameters were optimised and an averaging technique was used. The optimisation was done by removing some of the reflectors in the sigma- and chi-phases in the original TSL material files used for pattern indexing. By changing Hough parameters, considerable improvements in the indexing of the patterns were observed along with the increase of average confidence index (CI) value for both intermetallic phases. The applied SEM, Zeiss Ultra 55 FESEM was equipped with a NORDIF UF-1100 EBSD detector to acquire and stream patterns to HD. Indexing and data processing were performed by TSL/OIM 7.3. In the present study, at the most four different phases are present in the SDSS.

1. Introduction
Super duplex stainless steels are used in petrochemical industry because of their high corrosion resistance and good mechanical properties. These properties come from high content of alloying elements, especially high chromium content which provides high corrosion resistance. However, the wrong heat treatment for these steel grades can lead to embrittlement of the materials through intermetallic phases such as sigma- and chi-phase. This can happen during fabrication, welding processes and prolonged exposure to high temperatures during their service lives. The embrittlement can lead to catastrophic service failure of components.

A way to investigate the intermetallic phases in the scanning electron microscope (SEM) is to apply the electron backscatter diffraction (EBSD) technique. However, the intermetallic phases, i.e., sigma- and chi-phases, can be challenging to index. The aim of this study is to investigate a super duplex stainless steel alloy, UNS S37260 or Zeron 100, focussing on the intermetallic phases. The project is done together with Equinor ASA.
2. Materials and methods
The material used in this study was the alloy Zeron 100, a super duplex stainless steel in annealed condition. The material was provided by Equinor ASA with composition shown in Table 1.

Table 1. Chemical composition (in wt%) of alloy UNS S32760.

| Alloy element | Cr  | Ni  | Mo  | Cu  | Mn  | W   | Si   | N    | C    | P    | S   |
|---------------|-----|-----|-----|-----|-----|-----|------|------|------|------|-----|
| wt%           | 25.55 | 8.28 | 3.46 | 0.72 | 0.52 | 0.52 | 0.42 | 0.2-0.3 | 0.018 | 0.017 | 0.001 |

Three samples were chosen for investigation: First one as received as reference sample, a second one was heat-treated at 900 °C for 8 min (~5 wt% of sigma-phase), and a third one heat-treated at 750 °C for 4 h (~30 wt% of sigma-phase). The samples were firstly indexed with default Hough-transform parameters and default material files for all the phases. Thereafter, both Hough parameters and material files for the sigma- and chi-phases were changed in TSL OIM 7.3 software.

2.1. Heat treatments
Heat treatments were performed to introduce different amounts of intermetallic sigma- and chi-phases. Heat treatments were done in a Nabertherm P300 muffle furnace and afterwards the samples were quenched in room temperature water. One of the samples was heat-treated at 900 °C for 8 min, which introduces about 5 wt% of sigma-phase and the other at 750 °C for 4 h, which introduced about 35 wt% of intermetallic phases.

2.2. Sample preparation
The samples were grinded using an ATM Saphir 330 at 150 rpm with SiC-paper up to grit 4,000 using water as lubricant. Thereafter, the samples were polished using a Struers DP-U3 polishing machine at 150 rpm with polishing discs having a diamond suspension with particle sizes 3.0 µm and 1.0 µm, and with an ethanol-based lubricant for 15 minutes. After this step, a vibration polishing with at amplitude of 70 % and a weight of 200 grams was performed with a Buehler Vibromet 2 polisher. After each grinding and polishing step, the samples were cleaned with water and ethanol. Lastly, the samples were plasma-cleaned with Fischione Model 1020 for five minutes.

2.3. Scanning electron microscope (SEM)
The SEM used to study and acquire Kikuchi patterns from the material was a Zeiss Ultra 55 FESEM. The parameters used in the SEM are shown in Table 2.

Table 2. Parameters used in Zeiss Ultra 55 FESEM.

| Parameter             | Value          |
|-----------------------|----------------|
| Accelerating voltage  | 20 kV          |
| Working distance      | 25 mm          |
| Magnification         | 200x           |
| Aperture size         | 300 µm         |
| Angle                 | 70°            |
| High current mode     | ON             |
| Dynamical focus       | 10 - 15 %      |
2.4. **Electron backscatter diffraction (EBSD)**

EBSD measurements were taken on three investigated samples. A NORDIF UF-1100 EBSD detector to acquire and stream patterns to HD was employed, and NORDIF 3.0.43 software was used. The parameters are listed in Table 3. The step size used was 0.25 µm and the region of interest varied, with the biggest area being 400 × 400 µm. Thereafter, the analysed area was cropped to get a more detailed look at specific phases. For each measurement, five calibration patterns were collected, one in each corner and one in the middle of the area to optimise the pattern centre. A background image was acquired before acquiring the patterns. This was done to get better signal and avoid background noise.

| Parameter          | Acquisition | Calibration |
|--------------------|-------------|-------------|
| Averaging          | 2           | 5           |
| Speed              | 400 fps     | 500 fps     |
| Resolution         | 120 ×120 px | 160 ×160 px |
| Exposure time      | 2,450 µs    | 7,092 µs    |
| Gain               | 4           | 1           |

2.5. **TSL OIM analysis**

The collected EBSD data were characterised using TSL OIM 7.3 analysis software. At first, default bmt-files for the ferrite, austenite, sigma- and chi-phases were applied. Thereafter, the same acquired patterns were characterised with new bmt-files for sigma- and chi-phases provided by René de Kloe (Ametek). The difference between default and new bmt-material files for the chi-phase lay in the fact that the reflectors [4 2 2] and [6 6 0] were removed in the new, optimised bmt-file. The only change in the sigma bmt-file was the change of symmetry group from tetragonal C4h/[4/m] to tetragonal D4h[4/mmm].

| Parameter          | Before optimisation | After optimisation |
|--------------------|---------------------|--------------------|
| Binned pattern size| 96                  | 96                 |
| Theta step size    | 1°                  | 0.5°               |
| Rho fraction       | 85                  | 88                 |
| Max peak count     | 3                   | 3                  |
| Hough type         | Classic             | Classic            |
| Hough resolution   | Low                 | Low                |
| Convolution mask   | 9 × 9               | 7 × 7              |
| Minimum peak magnitude | 5                | 1                  |
| Maximum peak magnitude  | 15               | 10                 |
| Peak symmetry      | 0.70                | 0.70               |
| Vertical bias      | 0 %                 | 0 %                |
3. Results

3.1. Optimisation of Hough parameters
Optimisation of Hough parameters was done to characterise the sigma- and chi-phases at their best. The optimisation was used on the sample heat-treated at 900 °C for 8 min and the sample head treated at 750 °C for 4 h. Clean up below a confidence index CI = 0.05 was used. The colours of the phases are indicated in Fig. 1.

![Figure 1. Colours for the different phases.](image)

3.2. Material treated at 900 °C for 8 min
The IQ (image quality) map for this sample is shown in Fig. 2. The area of the measurement was 37.75 µm × 32.50 µm. This is obviously a small area and the phase composition is not representative for the entire sample; however, it was used to show the difference in chosen parameters. The step size used to acquire the signals was 0.25 µm. As can be seen, there are some dark areas on the IQ maps. It can also be observed that the maps are not of the optimal quality. The grain boundaries appear dark but relatively clear.

![Figure 2. IQ map of sample heat-treated at 900 °C for 8 min](image)

Figure 3 shows the phase maps of the material heat-treated at 900 °C for 8 min before and after Hough parameter optimisation. Austenite is marked in green, ferrite in red and sigma in yellow. It can be observed that the optimisation leads to less noise in the map and less points are characterised as sigma phase. This can also be seen in Table 5, where a higher fraction of austenite after the optimisation is clearly visible.
Table 5. Phase fraction of the material heat-treated at 900 °C for 8 min before and after Hough parameter optimisation.

| Phase       | α wt% | γ wt% | σ wt% |
|-------------|-------|-------|-------|
| Before optimisation | 45.4  | 45.4  | 7.4   |
| After optimisation  | 44.2  | 50.7  | 5.1   |

Figure 2. Phase map for material heat-treated at 900 °C for 8 min. Left: before optimisation. Right: after optimisation.

Table 6 shows that the CI value for the sigma-phase increases from 0.08 to 0.14. The CI value for ferrite (alpha) also increases after optimisation, while the value for austenite (gamma) decreases slightly. The IQ values for all the phases increase after optimisation. The fit for all the parameters decreases and gets closer to zero.

Table 6. Average IQ, CI and fit values for the material heat-treated at 900 °C for 8 min. before and after optimisation.

| Parameter | γ+α+σ | α | γ | Σ |
|-----------|-------|---|---|---|
| CI, before | 0.64  | 0.66 | 0.70 | 0.08 |
| CI, after  | 0.73  | 0.79 | 0.67 | 0.14 |
| Fit, before | 0.90  | 0.85 | 0.93 | 1.53 |
| Fit, after  | 0.71  | 0.66 | 0.72 | 1.46 |

3.3. Material treated at 750 °C for 4 h
In Fig. 4, the IQ map for this sample is shown. The area of the measurement is 39.40 µm × 33.60 µm. Again, the area is not representative for the phase composition of the entire sample. As can be seen, a large part of the IQ map is covered by dark areas. It can also be observed that image is not of optimal quality. Some lighter areas in the upper left and lower right corners of the map can be noticed, as well as some smaller points in between the darker areas. Grain boundaries appear darker.
Figure 3. IQ map of sample heat-treated at 750 °C for 4 h.

Figure 5 shows the phase maps of the material heat-treated at 750 °C for 4 h before and after the Hough parameter optimisation. Austenite is marked in green, ferrite in red, sigma-phase in yellow, and chi-phase in blue. It can be observed that the optimisation leads to a considerably lower noise level. It can also be observed that most of the noise comes from the chi-phase. After the optimisation, less phase was indexed as chi-phase and more as ferrite (alpha) phase. This is also shown in Table 7. More points were also indexed as sigma-phase after the optimisation.

Figure 4. Phase map for material heat-treated at 750°C for 4 h. Left: before optimisation. Right: after optimisation.

Table 7. Phase fraction of the material heat-treated at 750 °C for 4 h before and after Hough parameter optimisation.

| Phase          | α wt% | γ wt % | σ wt % | χ wt % |
|----------------|-------|--------|--------|--------|
| Before optimisation | 5.6   | 36.5   | 41.4   | 16.5   |
| After optimisation     | 10.9  | 38.0   | 49.1   | 2.0    |
Table 8 shows that the CI value for the sigma-phase increases from 0.08 to 0.14. The same does the CI value for chi-phase, mainly from 0.12 to 0.17. Especially worthy of mentioning is the rise of the CI value for ferrite phase from 0.01 before to 0.45 after optimisation. It can be seen, that after optimisation the fit increases for all the phases except the sigma-phase, with the average increasing from 1.04 to 1.09.

Table 8. Average IQ, CI and fit values for the material heat-treated at 750 °C for 4 h before and after optimisation.

| Parameter | α+γ+σ+χ | α | γ | σ | χ |
|-----------|---------|---|---|---|---|
| CI, before | 0.22 | 0.01 | 0.47 | 0.08 | 0.12 |
| CI, after | 0.30 | 0.45 | 0.48 | 0.14 | 0.17 |
| Fit, before | 1.04 | 0.97 | 0.73 | 1.28 | 1.16 |
| Fit, after | 1.09 | 1.07 | 0.92 | 1.21 | 1.50 |

4. Discussion

4.1. Hough parameter optimisation
Hough parameter optimisation leads to clear changes in the indexing of different phases, especially the sigma- and chi-phases. The IQ, CI and fit values will be discussed as well as the IQ and phase maps for the two relevant samples. The changes introduced by optimisation will be also discussed.

4.2. Effect on phase characterisation
Optimisation of the parameters leads to a clear improvement of the characterisation of the sigma- and chi-phases. Figures 3 and 5 for the material heat-treated at 900 °C and 750 °C respectively, show that the noise level is much higher before optimisation. This is especially shown in Fig. 5. Most of the wrongly indexed pixels in Fig. 3 assigned to sigma-phase have been converted after optimisation either to ferrite or austenite. Also, some pixels assigned previously to ferrite have been indexed after optimisation as austenite. This conversion is a result of the clean-up process.

Figure 3 shows the difficulties of characterising the chi-phase. Presumably, there are extra difficulties when there are two phases, which are difficult to be distinguished. This is also illustrated by the optimisation because the areas previously indexed as the chi-phase were after optimisation indexed as sigma-phase (Fig. 5 and Table 7). The chi-phase is reported to have a BCC-structure [1], while the sigma-phase has a tetragonal structure similar to the BCC one. Some of areas previously indexed as chi-phase were converted to ferrite after optimisation. This is because the unit cells of both phases have a lot of common reflectors and the unit cells parameters are almost the same [2]. Optimisation was based on removing reflectors [4 2 2] and [6 6 0] in the chi-phase, which were also used in the ferritic phase.

Optimisation leads to a smaller amount of the sigma-phase in the material heat-treated at 900 °C (Table 5) and a significantly smaller fraction of the chi-phase and a higher amount of the sigma-phase in the material heat-treated at 750 °C (Table 7). Some of the points indexed as the chi-phase in Fig. 5 can be imagined to be ruled out as noise, but some of the points look like clear clusters of the chi-phase. The indexing problem most probably comes from the difficulties of indexing grain boundaries and the overlap of signals acquired from different grains. Grain boundaries were indexed with the help of misorientation angle, so all boundary segments with an angle higher than the defined critical misorientation angle are considered to be grain boundaries [3].

The fraction of austenitic phase increases with the optimisation in both samples (Tables 5 and 7). It is expected that the fraction of austenite remains the same or even increases, compared to the as-received
material, while the ferrite phase fraction decreases after the short term heat treatment. This is because sigma- (and chi-) phase is formed where saturation with molybdenum and chromium exists, i.e., in the ferritic phase. Such a saturation also causes new austenite areas to be formed.

The symmetry group of sigma is changed with the optimisation from tetragonal C4h/[4/m] to tetragonal D4h[4/mmm]. In the literature, the space group for the sigma-phase has been identified as P42/[mnm], which is the same space group as D4h [2]. This corresponds to the optimised case.

4.3. Effect on CI, IQ and fit values
Optimisation of the parameters leads to a clear improvement in the CI, IQ and fit values not only for the sigma- and chi-phases but also for the ferritic and austenitic phases. The overall CI value increases after optimisation both for the material heat-treated at 900 °C (Table 6) and for the material heat-treated at 750 °C (Table 8). For the material heat-treated at 900 °C the average CI value for all the phases increases from 0.64 to 0.73 before and after optimisation, respectively. The reason for such an increase in this value is mostly caused by the increase of the CI value for the ferrite (alpha) from 0.66 to 0.79. Sigma-phase also contributes positively with an increase from 0.08 to 0.14. For the material heat-treated at 750 °C that contains a significantly larger amount of sigma- and chi-phases, the average CI value for all the phases increases from 0.22 to 0.30 after optimisation (Table 8). The CI values for sigma-and chi-phase increase from 0.08 to 0.14 and from 0.12 to 0.17, respectively. Another value to be noticed is the increase of ferrite fraction from 0.01 to 0.45. A reason for such an increase might be caused by the difficulty with differentiating between the ferrite and the chi-phase. After removing the strong reflector from the chi-phase HKL list, the number of votes for the chi phase decrease contrary to the CI for ferrite phase. Such an increase in the CI value means that the second solution for phase identification is less likely after optimisation.

The IQ describes the quality of an electron diffraction pattern [4]. The higher IQ value means that the acquired bands are sharper and easier to index. It can be seen on the IQ maps that ferrite grains are darker comparing to austenite ones (Figs. 2 and 4). Comparing Fig. 4 to Fig. 2, it can be stated there are much larger dark areas in the sample heat-treated at 750 °C for 4 h. Therefore, more intermetallic phases are expected. The darkest areas in Fig. 4 can be identified as sigma-phase. The dark areas in the IQ map is due to low IQ value, which results from band edges blurring.

The perfect value for the fit is zero. That means that the angular deviation between calculated and detected bands is also zero [4]. The average fit after optimisation decreases from 0.90 to 0.71 for the material heat-treated at 900 °C and increases from 1.04 to 1.09 for the material treated at 750 °C. One possible reason for such a change was the use of different convolution mask, mainly instead of 9 × 9 the mask 7 × 7 was applied (Table 4). The default value is 9 × 9, however for the narrower bands it better to use 7 × 7 mask [4]. For the investigated sigma- and chi-phases, the fit value for sigma is decreasing for both samples, and the fit value for chi-phases is increasing only in the sample heat-treated at 750 °C for 4 h.

The aim of this study was to characterise the sigma- and chi-phases in a better way. From the analysis of the CI, IQ and fit values, it can be deduced that the proposed optimisation resulted in a better sigma-phase evaluation.

5. Conclusion
Changing the Hough parameters as well as the bmt material files in TSL/OIM 7.3 for sigma- and chi-phases led to a better indexing of these intermetallic phases in the SDSS UNS S32760 alloy. This was proven by an increase of the average CI values in both samples from 0.64 to 0.73 and from 0.22 to 0.30 in the materials containing around 5 wt% and 30 wt% of sigma-phase, respectively.

Acknowledgements
The authors would like to thank René de Kloe from Ametek BV, The Netherlands for providing bmt-files for TSL OIM 7.3 analysis software.
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