A heat treatment for removal of microstructure deformation histories in steels

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1 Introduction

The automotive industry is deeply interested in replacing the conventional high strength low alloy steels (HSS) with advanced high strength steels (AHSS), for weight minimization purposes. However, these materials have frequently been observed to fail by ductile fracture [1]. These failures cannot be captured with the current continuum damage models (CDM’s).

Improvement of these models require more advanced experimental methodologies for quantification of local damage evolution, which is responsible of ductile fracture. Among these experimental methodologies, the indentation based approach has drawn the most attention both due to practical aspects (e.g. ease of experimentation and industry relevant test setup) and also technical aspects (e.g. ‘local’ results, hardness and modulus of elasticity being measured simultaneously). In this methodology, degradation of the hardness or modulus of elasticity as a result of deformation is compared to the same parameters of the undamaged material to obtain the necessary damage parameter for the improved damage-induced CDM’s. This is illustrated for hardness in Figure 1.

![Figure 1: Hardness versus local effective plastic strain (a) and damage versus local effective plastic strain (b). The damage parameter is calculated via \( D_H = 1 - \frac{H}{H_D} \) [2].](image)

However, recent experimental results showed that this methodology has significant limitations and reproducibility problems [3]. From a hardness measurement point of view, damage evolution is strongly coupled to strain hardening (and microstructure evolution) and a drop in hardness (as a function of deformation) is only rarely seen. This can be seen in Figure 2. Furthermore, these experiments showed that even when a hardness drop is seen, this may be due to several other microstructural effects active simultaneously (e.g. strain hardening, grain shape and texture [4]).
Figure 2: Hardness versus local Von Mises strain for DP600 steel. No drop in hardness as a function of deformation is seen [3].

From a modulus of elasticity measurement point of view, the measurements are even less reproducible due to the pile-up of material, especially for indents on severely hardened parts of the specimens. This is illustrated in Figure 3. It is noted that also here several microstructural effects play a role simultaneously.

Figure 3: Modulus of elasticity versus local Von Mises strain for IF steel. The two results differ significantly, indicating reproducibility problems [3].

An innovative idea to overcome these problems is to remove the deformation history of the material (while preserving the microvoids), by designing a suitable heat treatment. If the complete deformation history of the material (for example due to a tensile test) can be re-
moved such that a new, completely homogeneous microstructure is obtained (e.g. in terms of phases available, grain size and texture), damage due to the initial tensile deformation would be the only factor causing the degradation of hardness or modulus of elasticity as obtained by indentation tests. This approach would lead to ‘cleaner’ determination of the damage parameter for CDM’s, also due to the well-defined undamaged reference state, as shown in Figure 4.

![Diagram](image)

**Figure 4:** *Hardness versus position for an as-fractured sample (A) and heat treated sample (B).* The triangles represent indents. The dashed lines are the hardness of the undamaged reference case, which is unknown for sample A. Sample A has strain hardened regions, whereas sample B is free of strain hardening due to a suitable heat treatment.

**Project goal**

The objective in this report is to obtain a suitable heat treatment resulting in a homogeneous microstructure, such that the complete deformation history has been removed. Section 2 provides a background on the physical phenomena associated with a (changing) microstructure under temperature and load. Section 3 presents the experimental methodology used including specimen preparation, image acquisition and analysis. Section 4 covers the results and a discussion. Finally, section 5 contains conclusions and recommendations.
2 Background

The indentation based approach to quantify local damage evolution has significant limitations and reproducibility problems. The hardness-based and modulus-based damage characterization methodologies are unreliable, because they both are influenced by strain hardening, which in turn intrinsically is coupled to the damage evolution. Also, hardness and modulus of elasticity depend on grain shape (i.e. equiaxed, having approximately equal dimensions in all directions, or elongated grains).

![Figure 5](image_url)

Figure 5: Regions of compression and tension located around an edge dislocation [5].

2.1 Strain hardening

Strain hardening is the phenomenon whereby a ductile metal becomes harder and stronger as it is plastically deformed. This phenomenon is explained on the basis of dislocation-dislocation strain field interactions. When metals are plastically deformed, some fraction of the deformation is retained internally and the remainder is dissipated as heat. The major portion of this stored energy is as strain energy associated with dislocations. For edge dislocations this is illustrated in Figure 5. Some atomic lattice distortion exists around the dislocation line because of the presence of an extra half-plane of atoms. As a consequence, there are regions in which compressive and tensile lattice strains are imposed on the neighboring atoms. The strain fields surrounding dislocations in close proximity to one another may interact such that forces are imposed on each dislocation by the combined interactions of all its neighboring dislocations. This interaction is depicted in Figure 6.

The dislocation density in a metal increases with deformation, due to dislocation multiplication or the formation of new dislocations. Consequently, the dislocations are positioned closer together. On the average, dislocation-dislocation strain interactions are repulsive.
The net result is that the motion of a dislocation is hindered by the presence of other dislocations. As the dislocation density increases, this resistance to dislocation motion becomes more pronounced. Thus, the imposed stress necessary to deform a metal increases with increasing strain hardening, making it harder and stronger.

Therefore, strain hardening is causing a problem in the indentation based approach. The desired drop in hardness to obtain the damage parameters should be caused only by damage. However, strain hardening obscures this drop in that it increases hardness upon deformation. This can be seen in Figure 2.

### 2.2 Grain deformation

Besides strain hardening, the grain shape is of large influence as well. Grains can be equiaxed or elongated, being small or large. For polycrystalline materials, the presence of grain boundaries forms barriers for dislocation movement.
A fine-grained metal is harder and stronger than one that is coarse-grained, since the former has a greater total grain boundary area to impede dislocation motion. Deformation causes grains that initially have an equiaxed shape to elongate in the direction of the load. This is illustrated in Figure 7. If the deformation is such that necking occurs and is continued until fracture, the grains become even more elongated and are confined in a small region close to the neck. This is a very hard and strong region, because of the huge total grain boundary area.

Grain size and shape affect the indentation experiments. This can be seen as follows: indentation causes a stress field around the indenter tip, which acts as a source for dislocations. If grains are smaller and elongated, these dislocations will encounter more grain boundaries to which they pile-up, which results in a larger hardness.

2.3 Heat treatments

The idea behind applying heat treatments is to remove the deformation history of the material, that is to remove the strain hardening effect and to obtain a homogeneous microstructure that consists of equiaxed grains all over the specimen. This requires clever use of recovery, recrystallization and phase transformations in material specific heat treatments.

Recovery allows partial annihilation of the effect of the strain hardening. During recovery, some of the stored internal strain energy is relieved by virtue of dislocation motion, as a result of enhanced atomic diffusion at an elevated temperature. There is some reduction in the number of dislocations, and dislocation configurations are produced having (much) lower strain energies.

Both recrystallization and phase transformations are based on two kinetic concepts: nucleation and grain growth. An important quantity in the kinetics of nucleation and grain growth is temperature.

Nuclei form preferentially at structural inhomogeneities, such as external surfaces, grain boundaries and dislocations. This is due to a reduced activation energy of nuclei at these inhomogeneities. Since the region near the neck contains a larger amount of total grain boundary area and has a greater dislocation density than regions further away from the neck, more nuclei will form close to the neck. It is a challenge in this project to have nuclei form homogeneously distributed over the sample volume, such that these nuclei can grow equal in size and equiaxed in shape.

A grain starts growing as soon as a stable nucleus has formed. However, the mechanism of grain growth differs for recrystallization and phase transformations. In case of recrystallization, grain growth is governed by the migration of grain boundaries. Boundary motion is the short-range diffusion of atoms from one side of the boundary to the other. Large
grains grow at the expense of small grains that shrink until the parent material is completely consumed. Thus, the average grain size increases with time, and at any particular instant there will exist a range of grain sizes.

Figure 8: Grain size as a function of temperature [5].

Grain structures during recovery, recrystallization and grain growth are shown schematically in Figure 8.

In case of phase transformations, grain growth is governed by long-range atomic diffusion, which involves several steps, like diffusion through the parent phase, across a phase boundary, and then into the nucleus. The growth process will cease in any region where grains of the new phase meet. For the case that indeed many nuclei will form in the neck, these nuclei cannot grow as large as in the region further away from the neck where less nuclei form.

Figure 9: Schematic plot showing curves for nucleation rate, growth rate, and overall transformation rate versus temperature [5]. Here, $T_m$ is the equilibrium solidification temperature.

The dependence of nucleation and grain growth on temperature is depicted in Figure 9.
If no recovery would be applied prior to recrystallization or phase transformations, no homogeneous microstructure would be achieved. Close to the neck more nuclei would form and the grains would remain relatively small. However, applying a long recovery will relieve a large amount of internal strain energy, which may enable nucleation to initiate distributed over the specimen. Furthermore, sufficient time is required for equilibrium to be acquired in phase transformations.

Therefore, the strategy adopted in this project to find a suitable heat treatment starts with a long recovery for which the recovery temperature is determined to remove the gradient in nucleation as much as possible, as has been explained above. Then four different routes are examined, which are illustrated in Figure 10.

All routes continue with recrystallization, of which the temperature is also determined by preliminary tests. Route 1 then finishes with furnace cooling. Route 2 proceeds with austenization. The time for austenization is long enough such that the specimen can fully austenize. Subsequently, the specimen is cooled down to a temperature at which coarse grains of ferrite and pearlite form and is hold at that temperature for a sufficient amount of time. Finally, the temperature decreases to room temperature through furnace
cooling. Route 3 and 4 are almost similar to route 2. Route 3 differs in that the specimen is austenized three times. In route 4 the time held at the specific temperatures is much longer. Following one of the presented routes a homogeneous microstructure without deformation history is expected to be acquired.
3 Experimental methodology

3.1 Specimens

In this project two steels are examined: Dual Phase 600 steel (DP600 steel) and Interstitial-Free steel (IF steel). The chemical compositions of both steels are provided in Table 1. To find the parameters in the damage model, the specimens have to be deformed up to necking and fracture to obtain the complete damage evolution. This is carried out by uniaxial tensile tests.

<table>
<thead>
<tr>
<th>Steel</th>
<th>C</th>
<th>Mn</th>
<th>Si</th>
<th>Al</th>
<th>N</th>
<th>P</th>
<th>S</th>
<th>Nb</th>
</tr>
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<tbody>
<tr>
<td>DP600</td>
<td>0.092</td>
<td>1.680</td>
<td>0.241</td>
<td>0.032</td>
<td>0.025e^{−5}</td>
<td>0.016</td>
<td>0.004</td>
<td>0.002</td>
</tr>
<tr>
<td>IF</td>
<td>0.004</td>
<td>0.168</td>
<td>0.013</td>
<td>0.025</td>
<td>0.030e^{−5}</td>
<td>0.007</td>
<td>0.007</td>
<td>-</td>
</tr>
</tbody>
</table>

<table>
<thead>
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<th>V</th>
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<th>Cr</th>
<th>Ni</th>
<th>Mo</th>
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</thead>
<tbody>
<tr>
<td>DP600</td>
<td>0.007</td>
<td>0.002</td>
<td>0.009</td>
<td>0.003</td>
<td>0.567</td>
<td>0.022</td>
<td>0.002</td>
<td>0.005e^{−5}</td>
</tr>
<tr>
<td>IF</td>
<td>-</td>
<td>-</td>
<td>0.005</td>
<td>0.001</td>
<td>0.016</td>
<td>0.023</td>
<td>0.001</td>
<td>-</td>
</tr>
</tbody>
</table>

Table 1: Chemical compositions of DP600 steel and IF steel in percentages\(^1\).

In previous research ‘dog bone’ specimens, shown in Figure 11(a), were used. A disadvantage of this geometry is that the location of fracture may be different for each specimen, as necking prior to fracture initiates at random spots across the region in the center with a constant cross-sectional area.

![Figure 11(a)](image1)

![Figure 11(b)](image2)

Figure 11: Common tensile test specimen geometries.

To avoid this problem and obtain a constant location of necking and fracture, the geometry of the specimen is determined such that the smallest cross-sectional area is in the center of the specimen, as illustrated in Figure 11(b). Localization will always initiate at this point. The specimens are cut by Electrical Discharge Machining (EDM).

\(^1\) provided by Corus
3.2 Tensile tests

To deform the specimens to the point of necking and fracture, the Kammrath & Weiss tensile stage is used with a 2 kN load cell, which is shown in Figure 12.

![Kammrath & Weiss tensile stage with a 2 kN load cell.](image)

The IF steel specimens were loaded with a forward velocity of 20 $\mu$m/s. The DP600 steel specimens were subjected to a forward velocity of 3 $\mu$m/s.

![Typical engineering stress strain responses when fracturing specimens. The circles indicate the strains at which specimens have been obtained that exhibit necking, but no fracture.](image)

Typical engineering stress strain responses for IF steel and DP600 steel are shown in Figure 13(a) and 13(b), respectively. Both steels exhibit ductile fracture, however, as can be seen from the figures, IF steel behaves more ductile. One can also see that a larger stress is required for the DP600 steel specimen to start necking. In Figure 14(a) the DP600
specimens that exhibit necking prior to fracture are illustrated. The fractured samples are illustrated in Figure 14(b).

![Figure 14: Specimens prior to fracture (a) and fractured specimens (b). In (a) all specimens are DP600 and show a clear necking. In (b) the upper specimen is IF steel and the lower specimen is DP600 steel, which exhibits less ductile fracture.](image)

### 3.3 Specimen preparation

In order to analyse the microstructure, before and after heat treatments, a specimen preparation protocol is necessary for each tested steel. In this section the different steps in the specimen preparation (i.e. grinding, polishing and etching) are described.

![Figure 15: Struers Knuth-Rotor 2 grinding machine.](image)
3.3.1 Grinding and polishing

To grind the specimens, the Struers Knuth-Rotor 2 grinding machines are used, of which one is illustrated in Figure 15.

Struers Waterproof Silicon Carbide papers are used as grinding papers with roughnesses 220, 400, 1200, 2400, 4000. The specimens with neck are ground as shown in Figure 16(a) to avoid temperature gradients in the heat treatments. Following the heat treatments, the rectangular specimens are also ground up to the centerline. The fractured specimens are ground up to the centerline in order to examine the microstructure from the neck to the hole, as illustrated in Figure 16(b).

![Figure 16](image)

Figure 16: The specimens with neck are ground in the shape of the dashed rectangle (a). The fractured specimens are ground up to the centerline of the specimen (b).

After grinding, the specimens are mechanically polished with the Struers DAP-V polishing machine, which is shown in Figure 17.

![Figure 17](image)

Figure 17: Struers DAP-V polishing machine.

For polishing a Struers MDNap sheet is used in combination with Struers DP-Spray containing 1 \( \mu \text{m} \) diamond particles. The polished surface is cleared with ethanol to obtain a smooth and shiny surface. This cleaning is recommended to remove the diamond particles.
3.3.2 Etching

The final step in the specimen preparation is etching to reveal the microstructure. The etching should be done sufficiently to obtain a nice contrast between the different grains, which is very important for the image analysis later on.

To etch DP600 steel, Nital 3% etchant is used that consists of 100 ml ethanol and 3 ml nitric acid. The time required to etch fractured, non-fractured and heat treated DP600 steel specimens ranges between 30 seconds and 1 minute.

An etchant that etches IF steel sufficiently and homogeneously has not been found in this project. Four options were applied, each leading to unsatisfactory results: (1) Etching with Nital 3% resulted in preferential etching of the ferrite grains. (2) Slight electro polishing and etching with Nital 3% also led to preferential etching. Another disadvantage of this option is the rather difficult and time consuming proces of finding the right parameters to set for electro polishing. (3) Etching with Marshall’s reagent did not attack the grain boundaries, revealing no microstructure. (4) Grinding and etching simultaneously with OPS resulted in not enough contrast.

Because of the insufficient etching of IF steel\(^2\), further analysis is only performed on DP600 steel.

Figure 18: Carbolite HVT 12/60/700.

\(^2\)and due to time constraints within the project
3.4 Heat treatments

The heat treatments are performed with the Carbolite HVT 12/60/700, shown in Figure 18. This high vacuum furnace enables a low oxygen level, resulting in less oxidation of the specimens at high temperatures. Furthermore, with this equipment a temperature trajectory can be programmed.

3.5 Microstructural characterization

In order to characterize the as-received, fractured and heat treated microstructures quantitatively, an adequate analysis methodology has to be used. In this section, the methods to obtain the images will be described together with their advantages and disadvantages concerning the image quality. Furthermore, the methodology used to conduct image analysis will be explained.

3.5.1 Image software

To conduct the image analysis, explained in section 3.5.2, use has been made of ImageJ, which is a public domain Java-based image processing program. It should be underlined that the acquired images of the microstructure should reveal sufficient contrast, allowing a clear distinction to be made between different gray values of the grains and grain boundaries. This remark will become more clear in section 3.5.3.

Figure 19: Locations along the centerline of the specimen of which to take images of the cross-sectional area.

—in Appendix A one can find a detailed description on how to use ImageJ in the microstructural characterization. For clarity, this description is accompanied with an example.
3.5.2 Image analysis

The measure used to characterize the microstructure of the specimens is grain size. To check whether a homogeneous microstructure is achieved or not, images are taken three times at four different locations along the centerline of the cross-sectional area of the specimen, which is illustrated in Figure 19.

3.5.3 Image acquisition

The images of the microstructure can be obtained through electron microscopy and optical microscopy. Each method has its own advantages and disadvantages. The requirement to properly perform thresholding in the image analysis (step 9 in Appendix A) is a clear distinction between different gray values of the grains and grain boundaries. In the ideal case, the ferrite, martensite and grain boundaries each have their own gray value.

Electron microscopy

The Philips XL30 ESEM-FEG microscope has been used, which is shown in Figure 20(a).

![Electron Microscope Images](image)

(a) (b)

Figure 20: The Philips XL30 ESEM-FEG microscope (a) and the Zeiss Axioplan2 microscope in combination with the Zeiss Axiocam(b).

The images automatically have a gray scale. An advantage is the fact that electron microscopy can achieve a high resolution, resulting in a lot of detail on a small scale. However, in contradiction to optical microscopy, it is not possible to obtain such a contrast that a clear distinction between different gray values of the grains and grain boundaries exists, which is shown in Figure 21(a).
This is a severe disadvantage when one intends to use ImageJ, because it is impossible to threshold properly. This disadvantage can also be seen from Figure 21(b). The histogram exhibits the undesired shape of a normal distribution, which means that the ferrite grains consist more or less of an equal amount of bright and dark pixels. The same holds for the martensite grains.

Figure 22: *Image of DP600 steel obtained through optical microscopy (a) and a histogram of the present gray values (b).*

Optical microscopy

The Zeiss Axioplan2 microscope in combination with the Zeiss Axiocam has been used,
which is illustrated in Figure 20(b). It is possible to acquire colored (RGB) or gray scale images. It is suggested not to use RGB images, because it saves a lot of memory when using gray scale images. An advantage of optical microscopy is the possibility to obtain such a contrast that a clear distinction between different gray values of the grains and grain boundaries exists. This is shown in Figure 22(a). The ferrite grains are the bright pixels and the dark pixels represent the martensite and grain boundaries. Figure 22(b) shows the corresponding histogram that exhibits a desired shape. A disadvantage is the fact that optical microscopy cannot achieve a high resolution, resulting in less detail on a small scale.
4 Results and discussion

4.1 Heat treatments

The appropriate recovery temperature to remove the gradient in nucleation is found to be approximately 500°C. At this temperature no changes are detected in the deformed microstructure, indicating the start of recrystallization. The recrystallization temperature is found to be approximately 700°C.

<table>
<thead>
<tr>
<th>#</th>
<th>Heat treatment</th>
<th>Temperature [°C]</th>
<th>Time [h]</th>
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</thead>
<tbody>
<tr>
<td>1</td>
<td>Recovery</td>
<td>500</td>
<td>24</td>
</tr>
<tr>
<td></td>
<td>Recrystallization</td>
<td>700</td>
<td>24</td>
</tr>
<tr>
<td></td>
<td>Furnace cool</td>
<td></td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>Recovery</td>
<td>500</td>
<td>24</td>
</tr>
<tr>
<td></td>
<td>Recrystallization</td>
<td>700</td>
<td>24</td>
</tr>
<tr>
<td></td>
<td>Austenization</td>
<td>1000</td>
<td>24</td>
</tr>
<tr>
<td></td>
<td>Below eutectoid</td>
<td>600</td>
<td>24</td>
</tr>
<tr>
<td></td>
<td>Furnace cool</td>
<td></td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>Recovery</td>
<td>500</td>
<td>24</td>
</tr>
<tr>
<td></td>
<td>Recrystallization</td>
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</tr>
<tr>
<td></td>
<td>Furnace cool</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Table 2: Heat treatments.

The heat treatments shown in Figure 10 are also given in Table 2 in more detail. All steps in the heat treatments last for 24 hours, except for heat treatment 4, in which the third, fourth and fifth step last for 72 hours.
4.2 Analysis

Heat treatment 3 led to the best results for a homogeneous microstructure. The images at the different locations are shown in Figure 23.

From these images it can be seen that a homogeneous microstructure has been obtained all over the specimen. The white areas represent ferrite, whereas the dark areas represent grain boundaries and pearlite. The fraction versus grain size is shown in Figure 24 for the four locations. It can be seen that more or less the same trend in grain sizes appears for all locations, which supports the conclusion of a homogeneous microstructure.
Figure 24: Fraction versus grain size for the four locations ranging from far away from the neck (a) to in the neck (d).
5 Conclusion and recommendations

It can be concluded that a suitable heat treatment has been found for DP600 that results in a homogeneous microstructure. The heat treatment in which the specimen has been austenized for three times led to the best results. A few recommendations can be made for future research:

- It still has to be verified that the damage distribution and morphology remain unaltered by the heat treatment.
- If the damage is preserved, then indentation measurements should be repeated to see if the limitations and reproducibility problems are solved.
- The current heat treatments are based on time temperature transformation (TTT) diagrams. If the damage is not preserved, then the heat treatments could be based on continuous cooling transformation (CCT) diagrams.
- Different types of cooling could be investigated, such as quenching and air cooling.
- It still has to be found how to etch IF steel correctly.
- A suitable heat treatment for IF steel has to be searched for.
References


A Quantitative image analysis

The image analysis with the use of ImageJ will be elaborated stepwise. In each step a picture is provided what to click to perform that step. Furthermore, an example will be given throughout the steps.

1. Load the image.

2. Select a region of the image that is in focus. Regions that are not focus cause less distinction between grains and grain boundaries.
3. Copy this selection.

4. Close the figure.

5. Paste the selection, automatically a new window is opened. Now the focused selection is filtered and ready to be processed.

6. Adjust the brightness and contrast to enhance the image quality.
7. Remove the pixels that have outlying gray values within the ferrite grains. This will improve the thresholding in step 9. Choose the dark outliers to be removed.

8. Remove the pixels that have outlying gray values within the martensite and grain boundaries. This will enhance the contrast between the grains and grain boundaries. Choose the bright outliers to be removed.
9. Adjust the threshold. The result is a binary image consisting of black and white pixels. Threshold the ferrite, which will appear in red, so that these grains will become black and can be counted and measured in step 12. To properly perform this step, it is required that a clear distinction can be made between different gray values of the grains and grain boundaries. If this is not the case, it will be difficult to find the right threshold values. Areas that are not ferrite will also become red.

10. It is possible that white pixels appear within the ferrite grains, which are black. This can be due to pixels that were not removed in step 7. One can use the ‘Fill Holes’ option to get rid of these white pixels. However, when using this option, one should
verify that white areas representing martensite and grain boundaries are not filled! An alternative is to use the ‘Remove Outliers’ option again to get rid of the white pixels within the ferrite grains.

11. To have better results in the last step, one might want to get rid of the possible spray pattern of black pixels around the black ferrite grains. To achieve this the ‘Remove Outliers’ option can be used. It is also possible to skip this step, because in the next step the size of the particles that have to be taken into account must be set. Hence, the spray pattern, which consists of small particles of black pixels, can be omitted.
12. Before analyzing the black particles, which represent the ferrite grains, circularity has to be ticked in ‘Set Measurements’. Now the particles can be measured. The output can be saved in an Excel file for later analysis.

For more information on specific image processing the reader is referred to the ImageJ documentation.