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A study on grain growth using a novel grain size calculation tool

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Abstract

The growth of prior austenite grains (PAG) of low alloyed martensitic steel is proven to be one of the key attributes contributing to the mechanical properties of ultrahigh-strength steels. The mean linear intercept -method (MLI) is traditionally used to acquire average PAG sizes from light optical microscopy images, which are from experimental test samples. The MLI -method is arduous and time-consuming as well as a highly generalizing method, where you lose information about the grain size distribution. Therefore, a more sophisticated and computerised method is in high demand among metallurgists.

A program has been developed that encompasses an importing, digitalizing and calculating tool, which provides grain sizes and their distribution from multiple images. The tool mimics the workflow of manual MLI -method so the user sets the measure lines and marks all the linear intercepts. After this the tool calculates the MLI grain sizes and their 95 % confidence limits. Additionally, the tool provides the size of each intercepted grain and combines them to create a distribution. This information has been used to study the effects of holding temperature and time on grain sizes throughout the test samples in a case where abnormal grain growth at the centreline was expected.

In the present study, PAG sizes were studied before and after deformation at ¼ and ½ thicknesses at various temperatures and holding times using the grain size calculation tool. The average MLI grain sizes show very little differences between temperatures and holding times, so information about grain size distribution is needed. Traditional presentation of the grain size distributions also shows too much variation to interpret the data properly. Instead, using the grain size distribution information and grouping grains to small, medium and large instances gives more profound data, especially in cases where grain size variation is significantly large.

Distribution data from the test series also showed abnormal grain growth at the centreline of the test sample. The grain size calculation tool is used to quantify the effect of temperature and hold time on abnormal grain growth and its root cause is examined briefly.

Keywords: Prior austenite grain size; static recrystallization; grain growth; abnormal grain growth; grain size calculation

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1. Introduction

Modern steel applications are diverse and very demanding, so controlling the properties of steel precisely is of paramount importance. To achieve such control, a good understanding of the nature of steel is required. One important piece in this puzzle is the microstructure of steel. It has a great influence on steel properties, such as yield strength, ductility and wear resistance. In this study, the focus will be on a high-strength carbon steel, specifically the evolution of the prior austenite grain structure in high-temperature deformation followed by isothermal holding.

A common way to depict PAG structure is to simplify a single grain into being circular, which is not a bad assumption when the structure is equiaxed. This is done so that all grains in a PAG structure can be depicted with a single parameter, grain size. Often grain size is averaged to include all grains in the grain structure. Such a simplification is reasonable when the microstructure is relatively equal-sized and can be useful when going through huge amounts of data [1,2]. On the other hand, when dealing with data including high variation on grain sizes,

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such as partial recrystallization or abnormal grain growth, a single averaged grain size value holds little value and at worst can produce misleading results. Instead of averaged grain size, observing variations between grains can be more useful in such cases.

The mean linear intercept method is a common, widely used way to calculate averaged grain sizes [3,4]. Using it manually is quite cumbersome, so an apparent need exists for a computer-aided calculation method. Some image editing softwares, such as ImageJ, include applications to automatically count grain sizes. However, they do not work reliably when grain boundaries after etching are not fully visible, which is common, especially for low impurity steels. For these reasons, developing a specialized software for grain size calculation was deemed necessary.

In this paper, a novel tool has been presented to help the user with the task of grain size calculation. The tool has been originally developed by Seppälä [5]. Included in the grain size calculation tool is a way to determine grain size distribution from the original MLI data, which the authors find to be potentially quite useful. Also, an improved distribution analyzation method is introduced.

The metallurgical purpose of the research is to observe and analyze the static recrystallization (SRX) and ensuing grain growth and also examine the possible abnormal grain growth at the ¼ and ½ thickness of the hot rolled plate using the individual grain size data from the tool.

Previous work has been done on abnormal grain growth by other authors extensively. For example, in [6,7] the authors have explained the phenomenon in their review articles. Raabe has compiled the history, terminology and methods of recovery, recrystallization and grain growth and explained the driving forces behind the abnormal grain growth phenomenon [6]. Whereas, Alaneme has gathered up-to-date knowledge concerning recrystallization process especially in high strength steels, where influencing factors are given to be stored energy, defect structure, strain, temperature and time [7]. Of these, the latter three have been investigated in this publication.

2. Materials and Methods

2.1. Experimental methods

Experimental material of a low-alloyed martensitic ultrahigh-strength steel was continuously cast, hot-rolled and direct-quenched. Two sets of ∅10x12 mm samples intended for Gleeble 3800 -simulations were cut through the hot-rolled plate. All Gleeble simulated samples had a heating rate of 10 °C/s and pre-holding time of 5 min at the target temperature. One set of samples was examined without deformation in a pre-heated state and others had strain of 0.4 with strain rate of 10 /s. Holding time after deformation and before water quenching is varied to obtain changes in static recrystallization and subsequent grain growth (Table 1.). Microstructural images were taken utilizing laser scanning confocal microscope (LSCM) (VK-X200, Keyence Ltd) after picric acid etching. The PAG structures were quantified at the ¼ and ½ thicknesses of Gleeble-simulated samples from LSCM-images. Strain, strain rate and holding times were selected to approximate the process values used in hot rolling to observe incomplete static recrystallization and subsequent grain growth stage. Holding times of 3 s at 950 °C and 2 s at 1000 °C are expected to have about 50 % SRX and holding times of 6 and 12 s, respectively, almost completed SRX. Longer holding times (50, 100 and 150 s) are carried out in order to study the grain growth following SRX.

Table 1. Gleeble 3800 simulation parameters.

Deformation temperature (°C)	Pre-holding time (s)	Strain rate (1/s)	Strain	Holding times (s)
950	300	10	0.4	0, 3, 12, 50, 100, 150
1000	300	10	0.4	0, 2, 6, 50, 100, 150
1050	300	10	0.4	0, 50, 100, 150
1100	300	10	0.4	0, 50, 100, 150
1150	300	10	0.4	0, 50, 100, 150

2.2. Grain size calculation

Traditionally, the PAG structure of steels has been determined using the MLI -method, with horizontal and vertical lines drawn in the images of the microstructure, and the grain boundaries crossed by these lines are marked. Using equations 1 and 2, MLI grain size is calculated. [4]

$$\overline{L} = \frac{\Sigma L}{\Sigma x} \tag{1}$$

where: $\overline{L}$ – mean linear intercept, ΣL – combined length of measure lines converted to real size and Σx –grain boundary interceptions in measure lines.

To convert measure lines into real size, the gauge line that indicates the image magnification is used. This is done using equation 2.

$$\frac{L}{L_{im}} = \frac{L_g}{L_{g,im}} \rightarrow L = \frac{L_g}{L_{g,im}} L_{im} \tag{2}$$

where: L_{im} – length of measure line in an image, L_g – real size length of a gauge line and $L_{g,im}$ – length of a gauge line in an image.

The data obtained is assumed to be normally distributed so the number and length of drawn lines as well as the number of grain boundaries determines the magnitude of the error. [3,4].

2.3. Calculator tool

The grain size calculation tool has been developed with python 2.7, using the TKinter module. It includes tools to import and modify a microscope image, whose grain size can then be calculated.

In essence, the tool workflow is the same as with manual MLI. First the image magnification must be determined from a scale, like the one on the bottom right edge in Fig 1. With the tool this is done by providing the scale line start and end points as well as its real length. The tool handles an image as just a pixel matrix, so the scale length in pixels is obtained from the scale line start and end points. The length of a single pixel is obtained by dividing real length with pixel length.

The next step is defining measure lines. Several methods have been implemented for this. Simplest way is to individually create (or remove) lines on the image by clicking anywhere. This does not really utilize the capabilities of a computer though, so a more automated way was added. It creates the horizontal and vertical lines for the image with either user-defined line spacing or line amount with a click of a button.

Now that the measure lines are added, the only thing left is to count the grain boundary interceptions. At the moment this must be done manually by clicking on each interception. The tool saves the coordinates and attached measure line for each interception mark to use for calculations later.

At this point, the tool has all the information needed to start calculating grain sizes. MLI grain size is calculated exactly as with the manual method, in eq. 1. Grain size distribution can also be calculated from the interception data, which is explained in chapter 3. The data is saved into tables for each measure line individually as well as horizontal, vertical and all lines combined. In the data are included parameters such as average grain sizes, maximum and minimum grain sizes and error estimates. Several images can also be simultaneously saved and combined, which is convenient when there are many images taken from a single area. Also, the microscope images can be saved with the measure lines and grain boundary intercepts added. This is good for checking the validity of chosen intercepts as well as data visualization. An example of an image handled with the grain size calculation tool is seen in Fig 1.

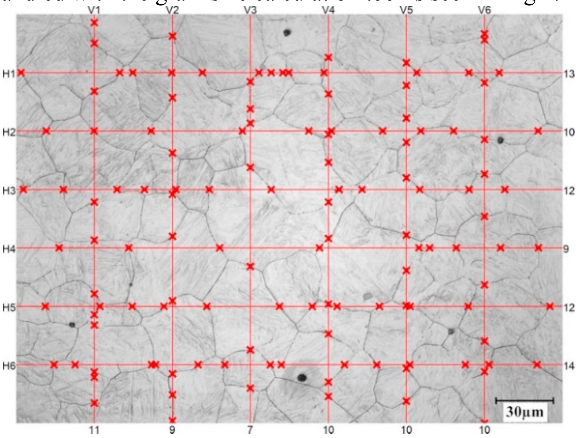


Fig. 1. Laser scanning confocal microscope image from ¼ thickness, 1100 °C temperature and 150 s holding time.

3. Results and Discussion

The basic idea of MLI is that the total length of measure lines is divided by the amount of grain boundary intercepts, which gives the average distance between intercepts. This can be interpreted so that the distance between any two adjacent intercepts always represents a single grain instance. Following this logic, the distribution of grain sizes can be obtained by measuring distances between all adjacent intercepts. Manually this would be a colossal task to perform on even a small set of microstructure images. On the other hand, in the grain size calculation tool the image is digitalized, and each marked intercept is saved as a set of pixel coordinates, which can be used to calculate the grain instance length quite handily. A grain size distribution can be directly created from the grain instance data set. In Fig. 2a and 3a are presented some example distributions obtained this way. MLI grain sizes are included in Fig. 3a to compare with the distribution data.

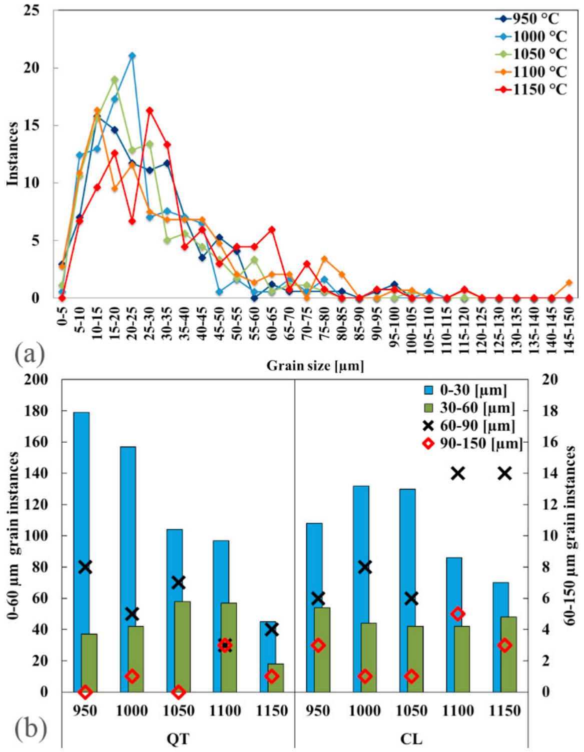


Fig. 2. Grain size distributions for samples with pre-heat treatment for various temperatures (a) conventional distribution graph at ¼ thickness (QT) and (b) Distribution group graph at ¼ thickness and centreline (CL).

The grain size distribution is a big leap ahead from the averaged grain size, but the data produced is quite messy and it is difficult to see clear trends. To clarify the data, the distribution interval was modified into several distribution groups: small, medium, large and abnormally large grains, see Fig. 2b and 3b. The MLI grain size at short holding times was around 30 µm, thus the small group is below, medium is above, and large group is over two times above average grain size and the abnormally large group includes grains beyond that. This way, the data can be analyzed better and the intervals are more meaningful. As an added benefit, more data can be included in a single figure. Grain instance amounts are compared instead of normalized values to better compare instance amounts between different temperatures and hold times.

Initial pre-heated situation before deformation is presented in Fig. 2. Amount of small grains decreases, and medium grains increases gradually when temperature rises. The level of large grains upsurges between 1050 °C and 1100 °C. At the pre-heated state there was only few grains that can be classified as abnormal grains at 1100 °C and 1150 °C temperatures and none of them were bigger than 150 µm.

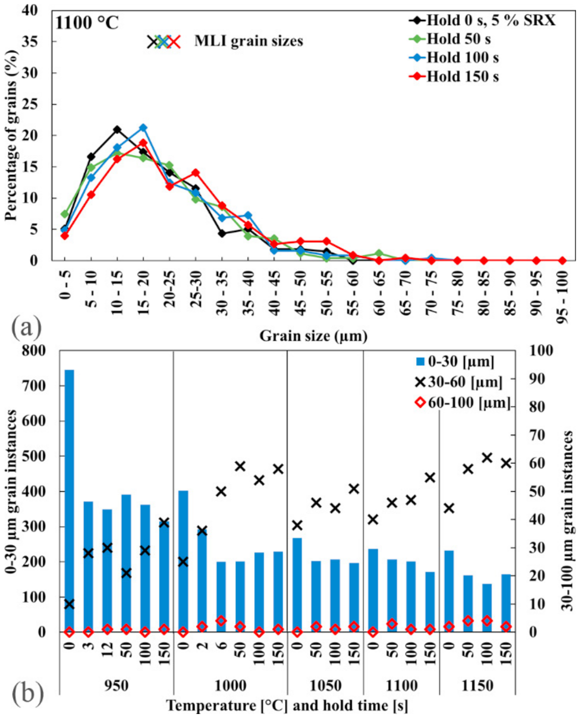
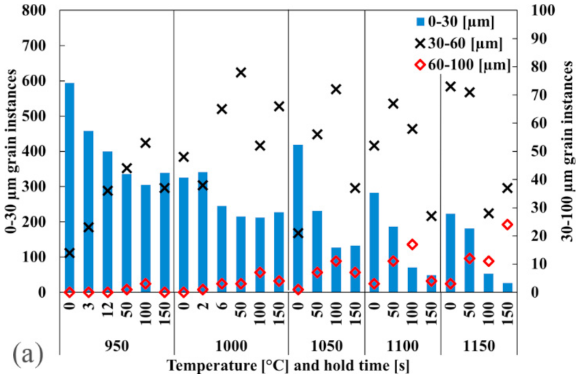


Fig. 3. (a) Average MLI grain sizes and grain size distribution at 1100 °C with different holding times at ¼ thickness. (b) Distribution group graph for different temperatures and holding times at ¼ thickness.

Results after deformation from ¼ thickness are presented in Fig. 3 and from ½ thickness are presented in Fig. 4. The MLI grain sizes and grain size distributions point to a general trend of grain size increasing with temperature and hold time. An increase of either or both temperature and holding time showed an increasing effect to grain growth at the ¼ thickness. Nonetheless, the amount of large over 60 µm grains was very low even with long holding times and high temperatures all grains were smaller than 100 µm. Holding time increased the amount of medium sized grains at all temperatures, but in high temperatures the effect was small after 50 s. The amount of small grains decreased considerably when temperature rises from 950 °C or when holding time is more than 0 s.



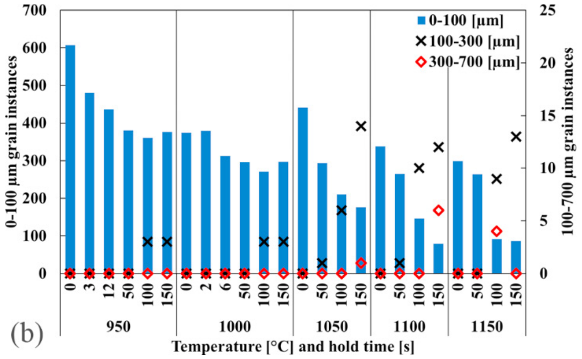


Fig. 4. Distribution group graph for different temperatures and holding times at centreline (a) without abnormal grain instances and (b) including abnormal grain instances.

PAG structure was quite homogenous at ¼ thickness, but abnormal grain growth was observed at ½ thickness, especially at higher temperatures (over 1050 °C) and holding times (over 50 s). Therefore, there was a need to extract the abnormally large grains from data and process them separately, as seen in Fig. 4b. In order to increase datapoints and therefore reliability, the analyzed area was also increased by decreasing the magnification of the microscope.

Mild abnormal growth was observed during preheating, but the effect was significantly greater at deformed samples and even dominant at the centreline. These results suggest that deformation has an empowering effect on abnormal grain growth as amount of strain and stored energy rises.

4. Conclusions

The grain size calculator has been developed and successfully used on a case including prior austenite microstructures with observed abnormal grain growth. The MLI-method is seen to be inadequate when microstructure is heterogenous, like in the present case, and using the developed grain size calculator provides more comprehensive data. The following conclusions can be drawn from the accumulated grain size data:

Pre-heated:

- Temperature rise had an increasing effect on grain size, as expected. The amount of medium and large grains increased, while the amount of small grains decreased.
- Some signs of abnormal grain growth were seen at both the ¼ thickness and centreline at temperatures over 1100 °C. However, this effect was minimal compared to deformation-induced abnormal grain growth at the centreline.

¼ thickness:

- Uniform grain structure after static recrystallization can be seen in all temperatures. Abnormal grain growth was not observed at all.
- The amount of small grains decreases while medium grains increases steadily when holding time increases. The same effect can be seen when temperature increases.

½ thickness:

- Behavior of 0-100 µm grains is similar to ¼ thickness, but noticeable abnormal grain growth can be seen at temperatures 1050 °C and higher.
- Abnormal grain growth plummets the amount of small sized grains for long holding times at over 1050 °C temperatures.

In its current state, the grain size calculator is proven to work consistently and is in use by several metallurgists of the University of Oulu. Future plans for the tool include further automations into the MLI method, free-form measure line drawing as well as multi-phase analyzation.

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