

Development of a Method for the Reconstruction of Three-Dimensional Dendritic Structures in Steel

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ABSTRACT: The shape of growing dendrites during the solidification of cast steel depends on many different parameters, as for example the casting technique, the applied cooling conditions or the casting geometry. Alloying elements such as carbon, silicon or manganese also have a major influence on the solidification behaviour and therefore on the morphology of the growing dendrites. In this paper, a method is presented which enables the reconstruction of three-dimensional dendritic structures by using concentration maps of such elements. For this purpose, a sample of a conventionally produced St52 continuous casting steel has been investigated. To determine the required two-dimensional maps, an electron probe microanalyzer (EPMA or "microprobe") with wavelength dispersive spectrometers (WDS) has been used. After the EPMA measurements, a commercial software tool has been utilized to process the concentration maps into three-dimensional structures.

1. INTRODUCTION

Nowadays, several investigation methods are available to reconstruct three-dimensional shapes. Their application depends on the required purpose. For instance, in the field of metallurgy computed tomography (CT) is a common method to visualize the structure of metals [1]. To gain accurate results by using this method for volumetric analysis, sufficient density differences inside the sample are required. CT also allows the examination of the surface topography of a sample. As an example for such measurements, the surface of a partial solidified dendritic structure after decanting the remaining interdendritic melt is illustrated in Fig. 1. Another method for producing metallic structures is to use a set of light microscopy images from polished surface areas. These images must be taken from the same lateral sample position, but at different depths [2, 3]. It is obvious that this method only works with adequate differences in contrast between dendritic and interdendritic areas. Many non-ferrous alloys provide such contrast after solidification, but occasionally this can also be achieved by etching the sample's surface. An example for the reconstruction of a non-ferrous dendritic structure is shown in Fig. 2.

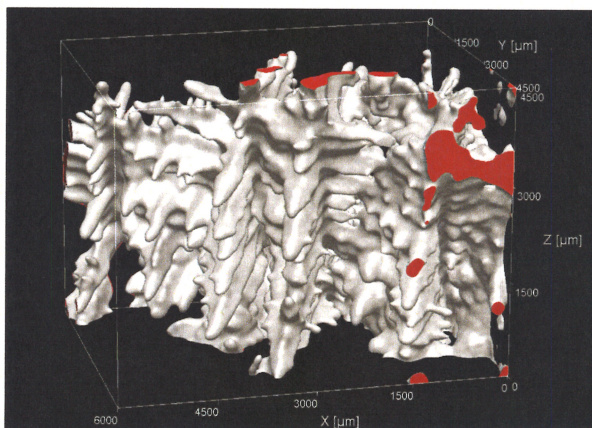


Fig. 1: 3D-reconstruction of steel dendrites (computed tomography measured at the ÖGI, Leoben)

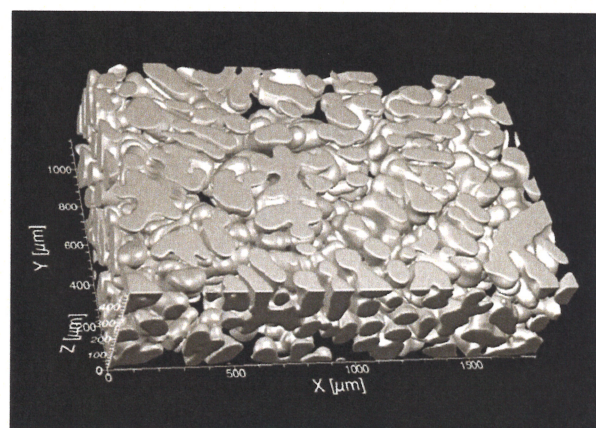


Fig. 2: 3D-reconstruction of dendrites in an AlCu-alloy, generated from light microscopy images (taken from [4])

Both these methods have a main disadvantage: only the shape of the final solidified structure can be reconstructed. This means that it is not possible to visualize the changing shape caused by the growth during the solidification process. To gain information about the changing structure with ongoing solidification, it is necessary to focus on a detectable

parameter which changes in relation to the volume fraction of solid. It is beneficial if this parameter can be measured at room temperature after solidification has finished. For the reconstruction method presented in this paper the selected characteristic is the concentration of the alloying element manganese (Mn) inside a solidified metal sample.

When the solidification process starts by cooling down the liquid steel, dendritic crystals which initially form have a different composition than the melt. The amount of a certain alloying element inside such a crystal depends on its specific segregation behaviour in the presence of iron and other elements. Hence, it can be enriched or depleted inside the solid phase in relation to its average content in the melt [5]. For instance, the concentration of Mn increases with advancing solidification. Considering that the change in concentration of a certain alloying element is more or less proportional to the increasing fraction of solid and the diffusion tendency in the solid phase is low, the concentration distribution at room temperature within a dendrite reflects the distribution during growth of the solid phase. Therefore, the determination of these concentrations allows an inference to the growth of the solid structure.

The idea behind the reconstruction method presented here is to measure the concentration distribution of a certain element at a defined sample position. The sample is sectioned several times and at each layer the concentrations are measured at the same horizontal position again. Afterwards, these layers can be merged to a three-dimensional structure by connecting concentrations which have a predefined value. A similar method is used at the microscale size, where the sample material is removed by a focussed ion beam (FIB) and the concentrations are measured by an energy dispersive spectrometer (EDS) [6].

2. EXPERIMENTAL METHODS

2.1 Embedding the sample

The sample used for the investigations was taken from an industrially produced St52 continuous casting slab. The position close to the slab's surface (shown in Fig. 3) was chosen to achieve an oriented columnar dendritic structure. The final size of the investigated sample area was about 3 mm x 3 mm in square.

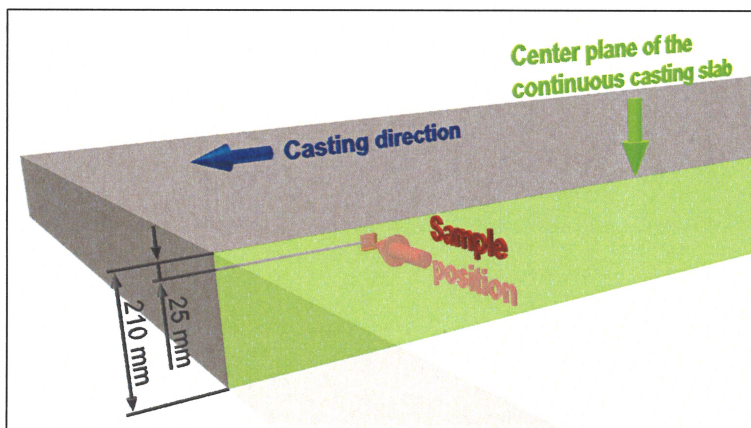


Fig. 3: Position of the investigated sample inside a continuous casting slab



Fig. 4: Embedded St52-sample (sample: grey square in the centre)

For microprobe analysis it is always necessary to guarantee the electric conductivity between the sample holder and the mounted specimen. For example, non-conductive samples and embedding materials (e.g. minerals, conventional plastics) are covered with graphite inside a vacuum chamber to achieve this. Another possibility for conductive samples (e.g. metals) is to embed them directly into a conductive material, like a metallic alloy. In the recent study a eutectic tin-bismuth-alloy with a melting point below 150 °C was used for this purpose [7]. By applying this method, the electric conductivity required for the measurements is ensured inside the complete volume of both, sample and embedding mass, and not only on its upper surface. This is a great advantage, especially if the surface layer is removed several times between the measurements. A further advantage is that the metallic alloy does not tend to evaporate and to contaminate the vacuum chamber of the

microprobe like conventional embedding plastics, if the beam passes it during the measurement. The resistance of the embedding material against the electron beam is very important, because during the measurement procedure presented in this paper the embedding area surrounding the sample was also analysed. The low X-ray intensities detected inside this area produced a sharp boarder to the comparatively high intensities inside the sample. Therefore an exact alignment of the measured maps was possible by orienting them along these visible boarder lines. Fig. 4 shows the investigated St52-sample which is completely embedded in the SnBi-alloy.

2.2 Polishing the sample

To achieve sufficient results with quantitative microprobe measurements, it was necessary that the examined surface area was well polished. To prepare the sample for the measurements described in this paper, the sample was positioned in a semi-automatic polishing machine where the material was removed in two steps: For the first step a mono-crystalline 3 μm diamond suspension on a satin woven natural silk cloth was used. This suspension allows fast material removal. To finish the surface afterwards, the second polishing step was performed with a mono-crystalline 1 μm diamond suspension on a porous neoprene cloth. During the whole procedure sufficient alcohol-based lubricant liquid was added to cover the used polishing cloths with a thin liquid film. The removed layer thickness from the surface of the sample was 25 μm ($\pm 3 \mu\text{m}$). It was checked by control surveys with a digital indicating calliper which allows a theoretical measurement resolution of 1 μm . Altogether, the concentration of manganese in five different layers was measured. Each layer was prepared in the described manner.

2.3 Microprobe measurements

All concentration measurements presented in this paper were carried out by the electron probe microanalysis (EPMA). This is an efficient method, which allows the non-destructive determination of chemical composition at solid materials like minerals, metals, biogenic matter, etc.. At the EPMA, a high energetic focussed electron beam impacts the sample surface and generates X-rays from a small volume below the interaction spot. These X-rays are characteristic for the elements inside the investigated specimen. The higher the measured X-ray intensity for a certain element, the larger its content within the sample. It is also possible to get a quantitative result for the chemical concentration of an element. For this purpose, the measured intensity must be compared with the value from a standard material which is well known in its composition. Then C_{Sample} is calculated with Eq. 1 [8]:

$$C_{Sample} = C_{Standard} \cdot \frac{I_{Sample}}{I_{Standard}} \cdot ZAF \quad (1)$$

In the above-mentioned equation, C_{Sample} is the unknown concentration of a certain element at the investigated sample position, $C_{Standard}$ is the known concentration of the same element inside the standard, I_{Sample} stands for the measured X-ray intensity of this element in the sample and $I_{Standard}$ for its measured X-ray intensity in the standard. The ZAF - factor takes into account the influence of the atomic numbers on the calculated concentration value as well as effects of absorption and fluorescence.

The investigation of the St52 sample was conducted using a JEOL 8200 microprobe with the basic settings from Tab. 1 below. It takes approximately 16 hours to measure the concentrations of manganese with these settings.

Analysed element:	Mn
Analysing crystal:	PETJ
Acceleration voltage:	15 kV
Beam current:	250 nA
Preset probe diameter:	0 μm (focussed)
Dwell time per measurement point:	120 ms
Interval between measured points:	5 μm
Number of measured points:	660 x 660

Tab. 1: Basic settings of the JEOL 8200 microprobe

For the measurements wavelength dispersive X-ray spectrometers (WDS) were used. They contain a set of special analysing crystals. Depending on their position these crystals are able to diffract X-rays specific to several selected elements, which are counted with a detector afterwards. To adapt the spectrometer for each desired chemical element the angle of X-ray incidence has to be changed. Therefore it is possible to move both, crystal and detector, inside of the spectrometer on a circular path. The result of the WDS-measurement at the microprobe is an intensity map. Every pixel of this map represents a square area of $5\ \mu\text{m} \times 5\ \mu\text{m}$ with a corresponding value for the measured X-ray intensity. To obtain values for the concentration of a specific element, it is necessary to quantify every single pixel with a standard as shown in Eq. 1. Normally this can be achieved directly at the microprobe by using its software; the quantified map is then called a “concentration map”. For the reconstruction method presented here, five of these concentration maps (taken from different depths in the sample) were the input for the three-dimensional reconstruction of the dendritic structure. In every pixel of those maps the value for the manganese concentration was clearly defined. Fig. 5 shows one of the original concentration maps for Mn, measured with WDS using the JEOL 8200 microprobe.

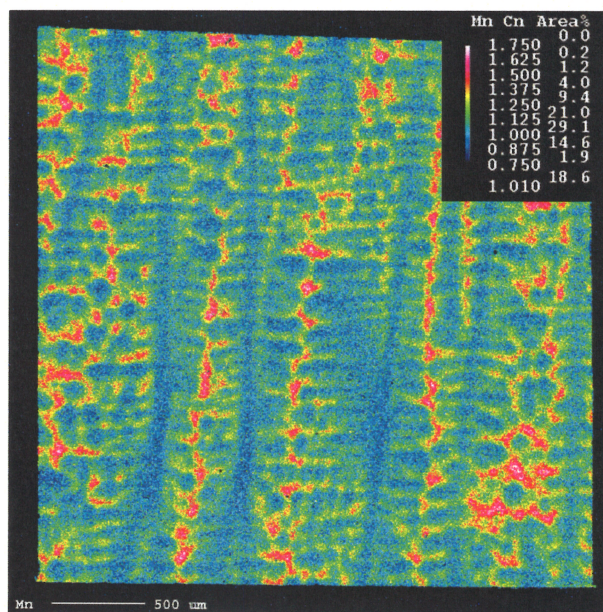


Fig. 5: Typical concentration map of Mn in steel measured with WDS

3. PROCESSING OF THE CONCENTRATION MAPS

The commercial software package MATLAB[®] was used to process the concentration maps with the following procedure.

3.1 Image filtering

As visible in Fig. 5, large jumps in concentration between adjacent pixels can appear regionally which make the reconstruction of smooth iso-concentration lines or faces impossible. Therefore it is necessary to smooth these undesirable concentration jumps before reconstructing the three-dimensional structure. This smoothing procedure, also called “image filtering”, was achieved in two steps.

For instance, if the electron beam passes a non-metallic inclusion inside the investigated sample, the detected X-ray intensity can be up to ten times higher than the expected intensity found in the surrounding area. Or in contrast, if there is a hole in the investigated surface, it may lead to an abrupt decay of intensity at this location. Therefore the concentration values calculated from the measured intensities may become extremely high or low, in the inclusion or hole respectively. To remove such intensities or concentration peaks also known as “salt and pepper noise”, a median filter was applied on the present concentration maps. This filter is defined in Eq. 2, which is adapted from [9]:

$$C'(x, y) = \text{median}_{(i, j) \in R_{x, y}} \{C(x+i, y+j)\} \quad (2)$$

In Eq. 2, $C'(x, y)$ is the new concentration value for a specific pixel at the Cartesian map coordinates x and y . It is calculated as the median of the original concentration values $C(x+i, y+j)$ at the map coordinates $x+i$ and $y+j$. The local coordinates i and j only exist inside the predefined filter area $R_{x,y}$, which surrounds the pixel.

The application of the median filter only removed undesired peak values effectively. The remaining image was hardly influenced and differences between concentration values at single adjacent pixels still existed. Therefore a Gaussian filter was applied to the median filtered maps in a second step. It led to a blurring effect whereby smooth transitions in concentration were achieved. The Gaussian filter is expressed by the following two equations [10]:

$$C'(x, y) = \sum_{(i,j) \in R_{x,y}} C(x+i, y+j) \cdot G(i, j) \quad (3)$$

and

$$G(i, j) = e^{-\frac{i^2+j^2}{2\sigma^2}} \quad (4)$$

In Eq. 3 above, $C'(x, y)$ is the new concentration value for a specific pixel at the Cartesian map coordinates x and y . $R_{x,y}$ is the filter area surrounding this pixel and $C(x+i, y+j)$ are the original concentration values at the map coordinates $x+i$ and $y+j$. The two-dimensional Gaussian weighting function $G(i, j)$ with the local coordinates i and j is only valid inside of $R_{x,y}$. In Eq. 4, the standard deviation σ defines the width of $G(i, j)$. To process the present concentration maps, the size of $R_{x,y}$ was chosen with 9 x 9 pixels in square for the median as well as for the Gaussian filter.

3.2 Creating the three-dimensional dendritic structure

After image filtering, all concentration values for the five maps were stored in a three-dimensional matrix which included five layers. Each single map was allocated to one of these layers. It was necessary to shift and rotate them into the right position before, so that they lay exactly one upon each other. The required alignment of the maps was achieved by orientate them along their outlines. The outlines were visible due to the large difference in the concentration of manganese between the sample and the embedding material. Finally, to get the desired dendritic structure based on the concentration matrix, all entries which contain a preset concentration value of Mn were connected with iso-surfaces. Depending on the change of this value, it is possible to illustrate different stages of microsegregation during the dendritic growth, as shown in Fig. 6 below. The entire structure depicted has overall dimensions of 2500 x 2500 x 100 μm . It gives a first approximation to the shape of the growing dendrites with advancing solidification. However, to get precise three-dimensional reconstructions of dendritic structures for different fractions of solid the effect of curvature on the microsegregation pattern has to be considered. This is an ongoing task.

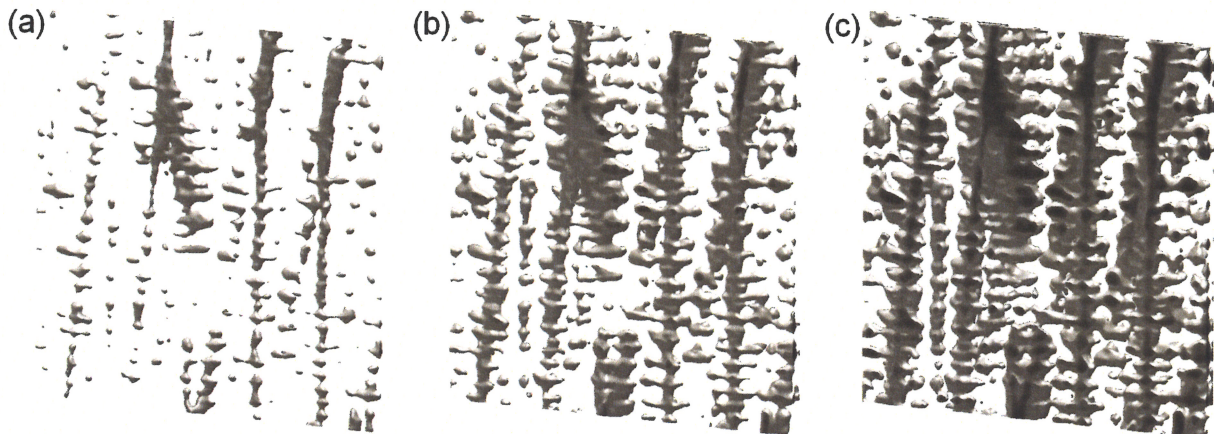


Fig. 6: Different fractions of solid depending on the predefined concentration values of manganese; (a) 0.95 % Mn, (b) 1.00 % Mn, (c) 1.05 % Mn

4. CONCLUSIONS

A method to reconstruct the three-dimensional shape of dendritic structures is presented by using serial two-dimensional microprobe concentration maps. These maps, which show the quantitative distribution of manganese at the investigated surface area, are generated using an electron probe microanalyzer (EPMA) equipped with wavelength dispersive spectrometers (WDS). The WDS-device allows the accurate quantification of element concentrations at each measured point. After the microprobe measurements, different image filters are applied to the created maps to prepare them for further three-dimensional reconstructions. Then the filtered maps are merged in an array. Inside this array all pixels which have the same predefined concentration value are connected with an iso-surface. Because the concentration of alloying elements (e.g. manganese) changes during solidification, the surface approximates the dendritic morphology at a certain solidification stage. Hence, it is possible to visualize the growth of the dendritic structure by increasing the predefined concentration value.

5. ACKNOWLEDGMENT

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