

How to Measure Phase Distribution and Degree of Sintering in Iron Ore Pellets

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Abstract—Iron ore pellets are made from refined and ground ore that is balled into spheroids and then heat treated. Pellet properties are measured through standardized physical, chemical and metallurgical tests to guarantee a high quality end product. However to better understand the oxidation and sintering of iron ore pellets one has to study the microstructures. This paper provides a proof of concept that optical microscopy combined with image analysis methods can be used to quantitatively measure phase distribution and degree of sintering in iron ore pellets.

I. INTRODUCTION

The iron ore extracted from LKAB's underground mines consists mainly of the mineral magnetite. Magnetite, as the name implies is the most magnetic of all the naturally occurring minerals and has a black color with a brownish tint. About 80% of LKAB production consists of iron ore pellets, which are made from refined and ground ore that is balled into spheroids with a diameter of about 10 mm and then heat treated. This process turns finely ground powder into strong pellets that can retain structural integrity even at very high temperatures in the blast furnace.

The first step in the process is oxidation of magnetite to hematite. This might seem a bit odd since the pellets are later reduced in the blast furnace. However the oxidation causes structural changes on a molecular level, which make the hematite pellets easier to reduce to iron than the raw magnetite ore. The oxidation reaction is also strongly exothermic and provides more than two thirds of the energy needed in the plants for the second step: the sintering.

Sintering is the process of particle bonding that usually occur under thermal treatment. First bridges start to form between particles at close range. Then necks start to merge and grains fuse, which leads to a denser material interlaced with fewer but larger pores. Finally even closed or intra-particle porosity disappears. The optimal level of sintering for pellets is when necks have formed to increase the structural integrity but porosity is still evenly distributed to allow high gas flow through the pellet

Pellet properties are measured through standardized physical and chemical tests to guarantee a high quality end product. However to better understand the oxidation and sintering of iron ore pellets one has to study the microstructures. This is a complicated task usually performed manually by experts in optical microscopy. The evaluation is tedious and takes a lot of time per sample and years of experience to perform well. However the

demand for reliable and quantitative measurements of large amount of data has increased rapidly.

In this study we will focus on two areas: methods to measure oxidation i.e. the phase distribution between magnetite and hematite and methods to measure degree of sintering

This work is a part of the Hjalmar Lundbohm Research Centre-funded (HLRC) project "Pellet Image Analysis". The aim is to develop fully automated image analysis techniques for quantitative characterization of sintering related features in iron ore pellets.

II. MATERIAL AND METHODS

A. Sample preparation

In this study pellets both taken from production and from different experimental conditions were used. All sample pellets were cast into epoxy, vacuum impregnated, cut in half and polished to give a good, reflecting cross section to study with the microscope

B. Image acquisition

Images were acquired with a Zeiss Axio Imager .M2m upright microscope system. Since, depending on the magnification, several hundreds to over thousand images were required to cover the sample the module MosaiX in the software AxioVision was used to create the desired scanning pattern. In 20x magnification for one pellet with a diameter of 10 mm more than 1000 12-bit grayscale images of size 1388x1040 pixels were acquired (Fig. 1). The spatial resolution was 0.32 μm per pixel. Automatic focusing at each stage position had to be used to ensure good focus for the entire sample.

C. Image processing

The AxioVision software provided a method for vignette removal using automatically acquired out of focus images and a method to stitch the images together. However the vignette removal proved unstable and the stitched images could not be exported in full bit depth, so instead all images were saved in the Zeiss' proprietary ZVI image format and processed with the image processing software Fiji [1].

First the images were imported with the Bio-Formats plugin [2] and then saved as individual tif-files. For illumination correction 10-15 images collected from regions with epoxy was used. First the images were filtered with a median filter of suitable size to remove speckles and scratches. Second the images were put into a stack and

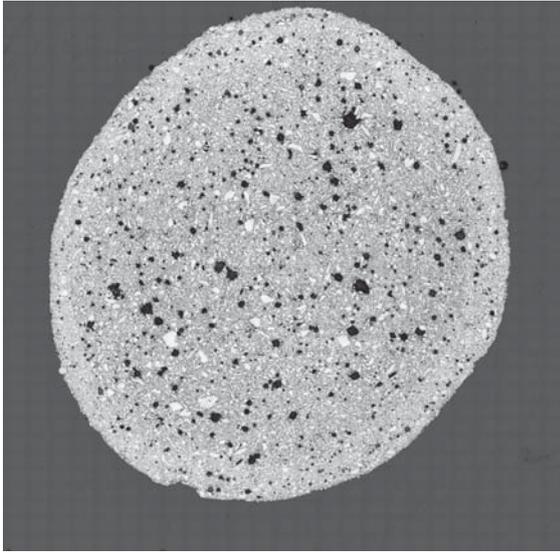


Fig. 1. An image of an iron ore pellet with the size of 31365x30993 pixels stitched together from 825 images without illumination correction. Vignetting is clearly visible in the individual images.

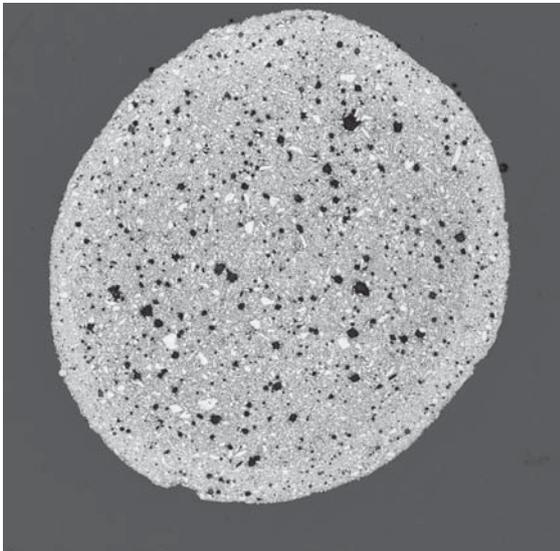


Fig. 2. An image of an iron ore pellet with the size of 31365x30993 pixels stitched together from 825 images with illumination correction.

a median projection was performed. The mean intensity for the resulting images was used to normalize and calculate an illumination correction map. All images were corrected before they were stitched together with the Grid/Collection Stitching plugin [3] (Fig. 2).

D. Pixel classification

In optical microscopy magnetite and hematite can manually be identified since magnetite is generally a darker grey with a brownish tint, whereas hematite is almost white in comparison. In the paper by Nellros [4] a global thresholding approach was used to define hematite, magnetite and pores & additives. Since the edges of hematite grains had the same grayscale value as magnetite these areas were erroneously classified as magnetite. To compensate for this a class for pseudo-magnetite was

defined as an edge with a set width that surrounded hematite grains and borders of pores & additives. This area was subtracted from the magnetite class and added to hematite.

In this study we have used the software Ilastik [5] to generate a pixel classifier for the three classes: 1) Hematite, 2) Magnetite and 3) Pores, Additives and Epoxy. This software allows the user to choose filters from a filter bank that enhances several features, such as edges and texture, in a range of sizes. The filters are applied to a representative sample set of images and the user can select the number of classes to identify and then interactively label pixels that belong to different classes. The software will, based on the calculated features, compute a classifier that divides the pixels into groups with minimal overlap. This classifier can be exported to other softwares, such as CellProfiler, to generate probability maps for the different classes. Ilastik can also perform the classification and export the results as probability maps or simple segmentations.

In this study the classifier was trained in collaboration with application experts and the exported segmentations were manually inspected and approved.

E. Measurements of phase distribution

Once the classifier was trained the software Ilastik was used to generate a simple segmentation where every pixel was labelled with one of the three classes: 1) Hematite, 2) Magnetite and 3) Pores, Additives and Epoxy. Since the pellet was surrounded by epoxy in all samples the third class could be used to delineate the area corresponding to pellet and create a binary mask. The other two classes were also used to create binary masks for areas corresponding to hematite and magnetite respectively.

Since the pellet is spherical it is subjected to several gradients during production where heat and oxygen levels depend on the distance from the surface. Hence the measurements of phase distribution should also take the distance from the surface into account. To achieve this a distance transform was computed for the binary mask of the pellet. This generated an image with a maximum value between 10 000 and 25 000 depending on the size of the pellet. A histogram was computed for the distance map of the pellet to be used for normalization of the phase distribution. The distance map was then masked with the binary masks for each class to generate three additional histograms. These histograms described how many pixels corresponding to the different classes were present at different distances from the surface. Since there are more pixels with a shorter distance to the surfaces the histogram for the unmasked distance map was used to normalize histograms and then plot the distribution of hematite, magnetite and pores & additives as a function of distance from the surface.

F. Measurements of degree of sintering

For measurements of degree of sintering the classes for hematite and magnetite generated by the simple segmentation were combined into one class and used to generate

a binary mask. When sintering occurs, small particles are forming bridges and are clumped together. When particles merge, their combined area will stay the same, however, their joined perimeter will decrease. This effect could be measured by computing a distance transform of the binary mask and generate a histogram of the distance map. When histograms from pellets with different degrees of sintering were compared, more pixels corresponding to shorter distances to the background were seen in pellets with low degree of sintering, whereas more pixels corresponding to longer distances to the background were seen in pellets with high degree of sintering.

III. RESULTS

This study is a first step to reach the long term goal to develop fully automated image analysis techniques for quantitative characterization of sintering related features in iron ore pellets. Hence, the result section will focus on the workflow developed to do the measurements and not the result of the actual measurements.

A. Image acquisition

The image acquisition could be automated to a large extent with the AxioVision software. The manual selection of region of interest and initial focus set up required about 15 minutes followed by one to two hours of automatic scanning depending on pellet size and magnification. Automatic focus correction had to be applied to each stage position to ensure good focus for the entire pellet, which increased the time for image acquisition by almost a factor of two.

The microscope was adjusted and tuned to provide even illumination of the sample, still vignetting was clearly visible in the images (Fig. 1).

B. Image Processing

The AxioVision software provided a method for illumination correction and image stitching. However, since the illumination correction proved unpredictable and the stitched images could not be exported in full bit depth, these steps were done in the software Fiji (Fig. 2).

C. Pixel Classification

The software Ilastik [5] was used to generate a pixel classifier for the three selected classes: 1) Hematite, 2) Magnetite and 3) Pores, Additives and Epoxy. A subset of 10 images could be used for training the classifier and the classifier could subsequently be used to segment images of pellets acquired with the same settings and magnification. The segmentations were manually inspected by an application expert and were approved as being accurate. The edges of hematite grains were correctly classified and there was no need to introduce a pseudo-magnetite class [4].

D. Measurements of phase distribution

Phase distribution was measured for several pellets with different degrees of oxidation. Here the measurement for a pellet with incomplete oxidation is presented (Fig. 3). The darker area in the middle of the pellet is magnetite

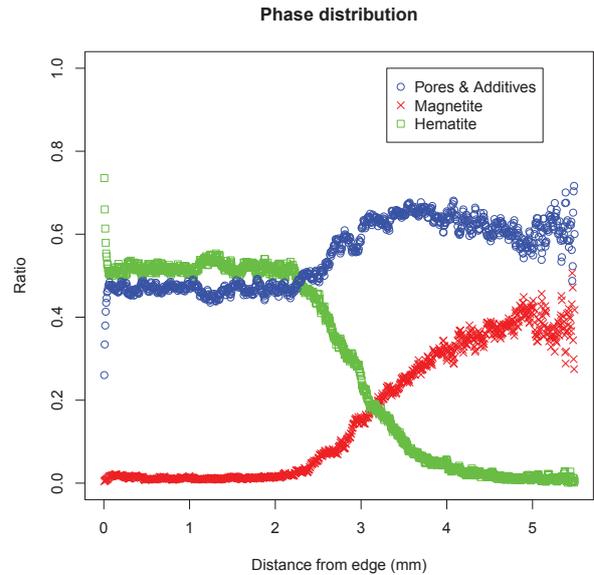


Fig. 3. Measurements of phase distribution for a pellet with incomplete oxidation. The amount of magnetite increases with distance to the surface, whereas hematite decreases.

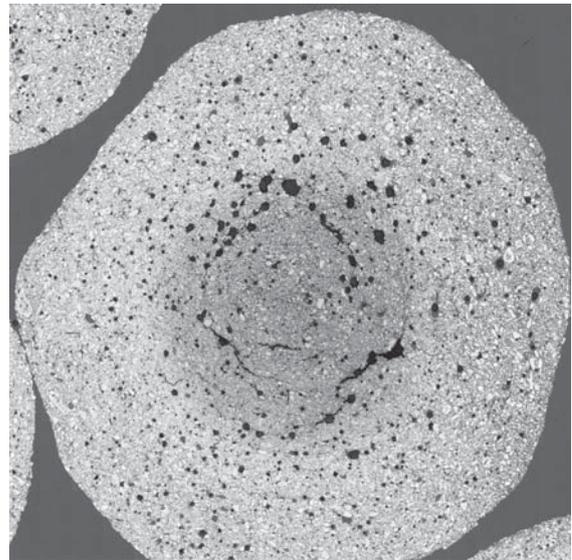


Fig. 4. Pellet with variation in oxidation. The darker area in the middle of the pellet is magnetite and the outer part is hematite. Cracks and bigger pores can be seen in the inner region.

and the outer part is hematite (Fig. 4). The amount of magnetite increases with distance to the surface, whereas hematite decreases. The oxidation front at 3 mm agrees with manual measurements. The increase of cracks and pores are also consistent with manual evaluation.

E. Measurements of degree of sintering

Sintering is the process of particles bonding together. When two particles fuse, the area in a cross section of the particles will remain the same while the perimeter will decrease. The measurement proposed here is based on the distribution of distances to the edge for the pixels in the particles. Pixels with short distances correspond to

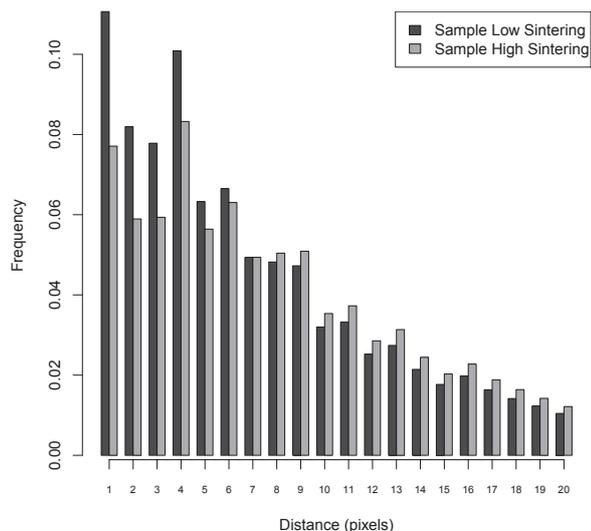


Fig. 5. Measurements of degree of sintering for two pellets manually graded as high and low. The frequencies for pixels with distances shorter than 6 pixels were higher for the sample with lower degree of sintering.

pixels close to the perimeter of the particle. For a pellet with higher degree of sintering the number of pixels with short distances decrease.

Here the measurements for two pellets with different degree of sintering are presented (Fig. 5). The frequencies for pixels with distances shorter than 6 pixels were higher for the pellet with lower degree of sintering. For longer distances the pellet with higher degree of sintering had higher frequencies.

The degree of sintering was also graded manually by visual inspection and pellets could be ordered in relation to other pellets. The measurements for degree of sintering agreed with the manual grading and relative ordering of samples. The measured degree of sintering was also consistent with experimental conditions.

IV. DISCUSSION

This paper provides a proof of concept that optical microscopy combined with image analysis methods can be used to measure phase distribution and degree of sintering in iron ore pellets. The measurements agree with visual inspection and manual grading. However a larger sample size of pellets and a more detailed and quantitative manual ground truth is needed to explore the discriminative power of the measurement methods.

Sample preparation and image acquisition proved to be crucial steps for the measurements. Difficulties in polishing of the samples may leave scratches and also result in out of focus images if the surface is not flat enough. Scratches may also influence the sintering measurements since scratches may give the appearance of a larger particle being split into smaller.

The pixel classification improved the segmentation considerably compared to global thresholding techniques and the knowledge from the applications experts could be incorporated in the training. The use of distance transform was also found to be an efficient way to extract spatial information for both the phase distribution and the degree of sintering.

The next step will be to use these methods to analyze a larger set of pellets. This will also give more data to validate the measurement methods and compare the results with other measurement techniques.

ACKNOWLEDGMENT

The author would like to thank Frida Nellros, Charlotte Andersson and Magnus Stafstedt for their work with preparation, image acquisition and manual grading of samples.

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