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Quantitative image analysis of bubble cavities in iron ore green pellets

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ABSTRACT

Scanning electron microscopy and image analysis was used for quantitative analysis of bubble cavities in iron ore green pellets. Two types of pellets prepared with and without addition of flotation reagent prior to balling were studied. The bubble cavity porosity amounted to 2.8% in the pellets prepared without addition of flotation reagent prior to balling. When flotation reagent was added prior to balling, the bubble cavity porosity increased by a factor of 2.4 and the median bubble diameter was decreased slightly. It was also shown that mercury intrusion porosimetry is not suitable for determination of the distribution of bubble cavities. Finally, our data suggested that the difference in total porosity determined by mercury intrusion porosimetry and pycnometry between the two types of pellets was due to the bubble cavities.

1. Introduction

Iron ore pellets are one of the main feed sources for blast furnaces or direct reduction processes for iron making or steel making industries. Iron ore fines are agglomerated into green pellets by first mixing magnetite concentrate, water, additives and binder, i.e. bentonite. In the next step, green pellets are formed in balling drum or disk pelletizer and then the pellets are dried and indurate in straight grate or grate kiln plants to form the final pellet product [1,2]. The properties of green pellets depend on the constituent minerals, chemical composition, particle size distribution, moisture content, binder type and dosage and additives.

Porosity and pore structure are important features of green pellets that affect permeability and durability of the final pellets. Thermal conductivity and diffusivity increase with porosity up to a certain level, but beyond this level, both conductivity and diffusivity decrease [3]. Oxidation time of magnetite is reported to be shorter with increasing porosity [4]. Therefore, controlling porosity in green pellets, both for quality assessment and development purposes, is of importance to the iron ore pelletizing industry.

One particular type of porosity is that introduced by bubble entrapment. This phenomenon causes the formation of large spherical cavities after drying and was found to be enhanced with increasing amount of the flotation collector reagent employed during separation of apatite from magnetite [5]. Air bubbles might also become entrapped by moistened particle or sprayed water during balling in the rotating drum. The resulting entrapped bubbles can act as crack initiators and deteriorate the compressive strength of the green pellets. Hence, quantitative data related to this fraction of porosity would be valuable.

Measurement of the total porosity in green pellets can be carried out by a combination of gas displacement pycnometry and analysis of the envelope density (density that can be obtained by shrinking films around the individual object in order to occupy the objects completely within the pellets) with silica sand [6] or by mercury porosimetry. The latter technique can in addition give information about the size distribution of the pore throats and under certain circumstances of the pore chambers using the intrusion and extraction measurements, respectively [7,8]. However, mercury is not environmentally viable and many companies have given up this characterization technique.

Image analysis of SEM micrographs may be used for quantitative determination of morphological features in iron ore pellets. However, the porosity of iron ore green pellets after drying is complex and has two different origins: the continuous porosity between the grains packed during balling and large cavities caused by bubble entrapment as discussed above. If one wants to quantitatively determine the latter by image analysis, the two types of porosity must be distinguished. Since morphological opening is useful to smooth irregular borders and fill or remove isolated pixels from images [9,10], it has a strong potential to isolate the large individual cavities from the smaller voids of the continuous porosity.

In the present work, we show for the first time how image analysis of scanning electron micrographs can be used for quantitative determination of bubble cavities in iron ore pellets. The results obtained from 2D SEM micrographs image analysis were translated to 3D by stereology. As, flotation reagent enhances air bubble entrapment, pellets prepared with and without addition of flotation reagent prior to balling were

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characterized and the results were compared with mercury porosimetry and pycnometry data.

2. Materials and methods

2.1. Materials

Magnetite concentrate enriched by flotation was collected from the Kiruna concentrating plant, Sweden. The concentrate was stored in barrels for several years prior to pelletization. Bentonite was used as a binder. The used flotation collector reagent is an anionic collector consisting of a main collector, co-collector and foam regulator and contains 95–98% surface active compounds and 2–5% maleic acid and glycol derivates.

2.2. Micro-balling of iron ore green pellets

Green pellets were prepared by micro balling as described in detail by Forsmo [11]. In short, the pellet feed was prepared by mixing 7 kg of magnetite concentrate with 0.5% bentonite in a laboratory mixer (Eirich R02, Germany) and the moisture content was adjusted to 9.4% during mixing. Directly after mixing, small granules (3.5 to 5 mm) were prepared by spreading some of the feed in a 0.8 m drum rotating at a speed of 37 rpm while tempered water (tempered water is the water in the temperature range from 29 °C to 43 °C) was sprayed to initiate growth of agglomerates. In the next step, 150 g of granules were returned to the drum, now rotating at a speed of 47 rpm. The granules were grown to green pellets by successively adding feed and spraying water in the drum. The final moisture content was 9.2% and green pellets with a size between 10 and 12.5 mm were selected by screening. Two batches of green pellets were prepared. In one batch, no flotation collector reagent was added to the feed. In the other batch, 60 g/t of flotation reagent was added to the feed prior to pelletization. The pellet batch made with addition of flotation collector reagent is denoted FLOT, while the other batch used as a reference, in which no flotation reagent was added, is denoted REF in the following. The samples were dried overnight at 105 °C.

2.3. Sample preparation for SEM imaging of the cross section

Three dried pellets from each batch were mounted in low viscosity epoxy resin (Struers EpoFix) using vacuum impregnation prior to SEM observation. In order to improve epoxy impregnation of the core of the pellets, slightly less than half of each dry pellet was removed by manual grinding prior to embedding. Metallographic polishing was carried out in a semi automated polishing machine using 9 μm, 3 μm and 1 μm diamond suspensions consecutively. Polishing was carried out in such a way that the polished surface propagated close to the center of the original pellet.

2.4. Image acquisition

Images were acquired by scanning electron microscopy (JSM-6460lv, JEOL, Japan) utilizing the backscattered electron compositional (BEC) signal at 15 kV and 100× magnification. Each image was recorded with a resolution of 960 pixels by 1280 pixels, resulting in 1.0 pixel per 1.0 μm. Images were acquired manually with virtually no overlapping and the entire cross-section of each pellet was captured. This was achieved by first calibrating scan rotation and lateral displacement for the particular instrumental conditions e.g. operating voltage, focus, working distance and magnification. To minimize the intensity gradient in the images, the polished section of the pellet was kept parallel to the backscattered electron detector. Similar levels of brightness and contrast were maintained for each image acquisition by adjusting the dynamic range of the gray levels (0, 255) of the histogram if necessary.

2.5. Image processing

2.5.1. Stitching

The acquired images of the entire cross-section of each pellet for each sample, about 120 images in total, were assembled together into a single image using the "montage" function in the image processing toolbox in Matlab R2009a. Fig. 1(a) and (b) shows the assembled cross-sections of one pellet in the FLOT and REF series, respectively. Fig. 1(c) and (d) shows the corresponding cross-sections after image processing. In these images, white represents pores. To better illustrate the different steps of image processing described below, Fig. 2 shows an enlargement of the area delimited in red in Fig. 1(a).

2.5.2. Noise reduction

The microstructure shown in Fig. 2(a) is subjected to artifacts (e.g. noise from the microscope detector, floating grains in the pores and formation of bubbles during epoxy impregnation) which must be eliminated before pore structure analysis. Median filtering was performed to remove extreme pixel values from the image.

2.5.3. Thresholding

A typical histogram of the cross-section of the pellet shown in Fig. 1(a) is presented in Fig. 3. It corresponds to the area delimited in red. The first peak at low gray scale values is due to the dark epoxy, which fills the pores, and the second, at high gray scale values, corresponds to magnetite. Silicates result in gray levels located in between both peaks. The threshold value was determined by trial and error until all pore regions were converted to black and the mineral regions to white, as shown in Fig. 2(b). The optimum threshold value was 71.5 and this value is indicated by a dashed line in Fig. 3.

2.5.4. Complementation and filling

The binary image obtained after thresholding contains floating grains inside the pores, see Fig. 2(b). The floating grains were removed using the Matlab function 'imfill' and the result is illustrated in Fig. 2(c).

2.5.5. Morphological opening

Next step was morphological opening, i.e. an erosion step followed by dilation. A disk-shaped structuring element of 10 pixels in radius was employed. The final binary image is shown in Fig. 2(d).

2.6. Image analysis

After cutting and polishing a pellet, only profiles of the randomly sectioned pores are visible on the two dimensional flat cross-section. A number of parameters of these profiles were measured in the images shown in Fig. 1(c) and (d) utilizing image analysis. A Matlab code was developed to count and label individual pore profiles with corresponding area and equivalent diameter of a circle with the same area using the Matlab function 'regionprops'.

The 2D data obtained by image analysis were unfolded to three dimensional data using stereology assuming a spherical shape for the pores. In a first step, the total pore profile area density for each size class was converted to area numerical density (number of pore profiles per unit area) by dividing each value with the equivalent area of a circle corresponding to the average equivalent diameter of the size class. Subsequently, the Wicksell–Saltykov [12,13] unfolding procedure was followed to calculate the volume numerical density (number of spherical pores per unit volume). Finally, this set of data was converted to total pore volume density by multiplying the volume numerical density by the volume of a sphere derived from the equivalent diameter of each size class and the cumulative curves were plotted.