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Automated measurement of sintering degree in optical microscopy through image analysis of particle joins

Frida Nellros [a], Matthew J. Thurley [a,*], Håkan Jonsson [a], Charlotte Andersson [b], Seija P.E. Forsmo [c]

[a] Luleå University of Technology, Department of Computer Science, Electrical & Space Engineering, Luleå SE-97187, Sweden
[b] R&D, Luossavaara-Kiirunavaara AB, SE-98381 Malmberget, Sweden
[c] MK3, Luossavaara-Kiirunavaara AB, SE-98381 Malmberget, Sweden

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ABSTRACT

In general terms, sintering describes the bonding of particles into a more coherent structure, where joins form between packed particles, usually as a result of heating. Characterization of sintering is an important topic in the fields of metallurgy, steel, iron ore pellets, ceramics, and snow for understanding material properties and material strength. Characterization using image analysis has been applied in a number of these fields but is either semi-automatic, requiring human interaction in the analysis, or based on statistical sampling and stereology to characterize the sample. This paper presents a novel fully automatic image analysis algorithm to analyze and determine the degree of sintering based on analysis of the particle joins and structure. Quantitative image analysis of the sintering degree is demonstrated for samples of iron ore pellets but could be readily applied to other packed particle materials. Microscope images of polished cross-sections of iron ore pellets have been imaged in their entirety and automated analysis of hundreds of images has been performed. Joins between particles have been identified based on morphological image processing and features have been calculated based on the geometric properties and curvature of these joins. The features have been analyzed and determined to hold discriminative power by displaying properties consistent with sintering theory and results from traditional pellet diameter measurements on the heated samples, and a statistical evaluation using the Welch *t*-test.

1. Introduction

Characterization of sintering is an important topic in numerous fields, including; metallurgy, ceramics, steel, iron ore, and snow. Sintering affects material properties, particularly strength, and describes the bonding of particles into a coherent structure typically via thermal treatment.

In the mining of iron ore, refining and concentration is performed to produce high-grade iron ore products that provide beneficial production conditions in steel making. One such product is iron ore pellets, where the refined and ground ore is balled into spheroids of about 1 cm^3 and then heat treated. The reason for this process is that the very strong pellets are more efficient to use than the finely ground powder they are made of, since they can be stacked in a pile and do not crumble at very high temperatures in the blast furnace, thus enabling a continuously high air flow through the entire reduction to pure iron [1].

The final strength of the pellet depends highly on the sintering of the iron-oxide hematite particles that take place during the heat treatment. In the case where the ore consists of magnetite iron-oxide, the nature of the sintering is dependent on the initial process of oxidizing the magnetite to hematite. Getting these processes to run in a balanced way is the key to creating a high quality end product [2]. To evaluate macrobehavior during transportation and blast furnace reduction properties, pelletizing companies usually have large lab facilities that can work in micro and pilot scale. In these labs, standardized tests are performed to measure such things as cold compression strength and reducibility [1].

In the pelletizing labs, sintering degree is never measured but related properties, such as porosity and expansion during heat treatment and shrinkage during cooling [1,3,4], can be measured. However, to really understand what happens and why, one must study the microstructure. Up to now this has very much been a handcraft work, an art-form of the trained expert eye such as the PhD thesis by Niiniskorpi [2]. Microscope operators and researchers who have been working at the same pellet plant for decades can look into the oculars and evaluate the quality of the product

* Corresponding author.
E-mail addresses: mjt@ltu.se (M.J. Thurley), hakan.jonsson@ltu.se (H. Jonsson).

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that they see, having years of experience to judge from. However, these experts are increasingly rare with people changing jobs more frequently, and the need for quantitative, comparable measurements have risen. Furthermore, evaluating large amounts of pellets in a microscope is tiring for the human operator and the demand for reliable statistics and large amounts of data has increased.

In ceramics the topic of image analysis as a way of measuring sintering has been thoroughly explored. Chermant et al. [5] have been working with characterization of sintering through morphological analysis since the beginning of the 1980s [5–7]. Starting with metallurgical powders of convex particles in 1981, Chermant et al. have developed image analysis methods based on the stereology principles as developed by strong names such as DeHoff et al. [8], Serra [9]. The motivation of stereology is to assume randomness and by letting a grid, plane or line interact with a sample in 1D or 2D being able to unfold its corresponding 3D/volume properties through mathematical relationships. One typical example of a stereometric approach is the classical method within materials science called point counting.

The sintering of a metal powder is studied by Chermant et al. [5] in a time lapse sequence. Chermant et al. [5] performed a horizontal line scanning analysis on thresholded images to compute the connectivity, concavity and convexity numbers of particles. These three features were derived from the count of tangents in convex and concave parts of extreme points on the particle perimeters. Furthermore, necks that form between the particles were identified by an algorithm using the distance transform but this approach was only possible because particles were convex. The results were compared only to the conventional method of studying porosity and the conclusion was that sintering is possible to characterize in the stereometric measurements. Early publications following Chermant et al. [5] focus on validating that the stereometric analysis is indeed possible through automatic image analysis. Further methodology development investigate the full potential of the stereometric unfolding into 3D features such as integral mean curvature [6], i.e. the property corresponding to total curvature of pore-solid interface per unit volume, computed from a rescaled connectivity number.

More recent publications go into more advanced theory, such as relating the results to grain growth theory, performing simulations and investigation of sample homogeneity [7]. Focus lies on the sintering analysis and not full automation, and “images still need additional treatment” [7] which can be suspected to mean manual intervention – in the preceding steps.

In steel and iron ore sinter cakes, image analysis has been used to study the porosity fraction and shape factors as an indicator of sintering degree [10,11]. Descriptions of the methodology are quite minimalistic and seem to contain some manual steps; e.g. “The void fraction and perimeter of voids was measured using commercial software for each binarized image” [10]. Kasai et al. [10] reported that the void fraction and specific surface area are correlated with strength of the sinter cake, and Molinari et al. [11] used image analysis methods to determine correct sintering time for steels with different boron contents. Later publications exist but seem mostly to vary the steel properties and not the image analysis methods [12].

The possible use of measuring curvature for quantifying sintering is also suggested for 3D data on snow. By consecutive slicing and imaging, Brzoska et al. [13] created 3D data of a wet snow sample in Brzoska et al. [13]. After segmenting the data into snow and pores, a method to measure local curvature from snow surface normal vectors and porosity medial axis was developed [13]. This technique has been further developed by Flin et al. [14] and explored using μXRT data to study curvature evolution over time and to model grain growth and the sintering process [15].

Only one previous publication on studies of sintering in iron ore pellets through image analysis have come to the author's knowledge. de Oliveira Simões et al. [16] claim to automatically identify and measure the necks and their number of connections and compare this to the pellets compressive strength. However, no method description on how this is performed in the Zeiss AxioVision software is given, and a visual inspection of the example segmentation shows clear ambiguities. Only three images at selected positions in each sample are analyzed. Results thus contain several unanswered questions and lack statistical validation but show an optimistic trend that there seems to be significant information in the analyzed measurements.

This paper presents an automated method for measuring sintering that can distinguish iron ore pellets subjected to different heat treatments through image analysis of cross-sections imaged in an optical microscope. One typical application of such a system would be lab scale evaluation of the sintering ability of new ores. The presented method studies necks and curvatures, however, not from the same stereological approach of Chermant et al. [5] but by directly identifying particle joins and analyzing their geometric properties. Data collection is semi-automated through Zeiss AxioVisions MosaiX module [17] and the image analysis is fully automated in Matlab® [18].

The results of this paper may interest several categories of readers such as both an image analysis audience and material scientists. Therefore the introduction is followed by a theory section that provides a short introduction to and review of both computational and material principles that have been applied. The method section has been supplemented with Appendix A, which contains a descriptive pseudo-code summary of the presented algorithm.

2. Theory

2.1. Sintering

Sintering is the bonding of particles into a more coherent structure (usually occurring under thermal treatment), where bridges start to form between particles at close range [19,20]. Fig. 1a depicts a simplified two particle model illustrating the initial stage for two adjacent spheres. Continuing the process, necks start to merge and grains fuse, see Fig. 1b, which lead to a denser material interlaced with fewer but larger pores. Angularities become more round as particles diffuse and swell. Finally, pores entirely contained within particles, referred to as “closed porosity”, disappear.

The optimum level of sintering for pellets is when necks have formed and increased the structural integrity, but when porosity is still uniformly high and allows a good gas flow throughout all of the pellet. Wynnyckyj and Fahidy [3] concludes that there is a drastic increase of crushing strength when shrinkage starts to be observed in the heated pellet, different from the steady and continuous growth of strength that can be measured initially. This most likely coincides with when neck growth turns into particle merging, as illustrated in Fig. 1b.

2.2. Image analysis

The images are analyzed mainly through mathematical morphology techniques. These techniques are particularly useful for identifying and analyzing spatial structures in image data. Morphological image processing operations apply a structuring element, which is a spatial probe similar to a neighborhood operation, to the image. A short presentation of the operations used in this work is provided below, but or a more elaborate