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Analogue iron ore sinter tablet structure using high resolution X-ray computed tomography

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ABSTRACT

During iron ore sintering large structural changes occur as liquid is formed in the flame front. The transformation of the structure into the agglomerated product is important for its physical and metallurgical properties. To quantify the change in sinter structure, analogue sinter tablets were made under tightly controlled conditions in a rapid heating furnace. The effect of maximum temperature, partial pressure of oxygen and ore type was investigated using a factorial design methodology. The internal structure of these tablets was measured in three dimensions using high resolution X-ray computed tomography (4.5 μm voxel size), to determine the size, shape and connections of the pores. Most of the open pore volume was contained in a single continuous interconnected pore network. Analysis showed maximum temperature, ore type and the interaction between ore type and maximum temperature to have a statistically significant impact on tablet volume. A similar analysis showed maximum temperature, ore type and the interaction between maximum temperature and partial pressure of oxygen (pO_2) to have a statistically significant impact on total porosity. Greater melt volumes increased the size, sphericity and total volume of pores.

1. Introduction

The quality of iron ore sinter is important for efficient, stable and productive operation of the blast furnace. The transformation of the green granulated sinter bed into the fired sinter cake in the flame front is thought to control the quality of the product sinter. Previous work has shown that both the mineralogy and the structure of iron ore sinter has an important impact on its quality [1, 2]. The structure of sinter is strongly influenced by the melt that is formed in the flame front. This melt is mobile and very wetting [3], so it rapidly coats surfaces and is drawn into pores in ore particles, where it partially assimilates the ore into the melt. Voids form at sites of melt formation (near coke and flux particles) and develop a structure of continuous interconnected gas channels and a continuous matrix of unassimilated nuclear ore particles surrounded by melt.

The mineralogy of sinter is commonly measured and related to the quality of the sinter produced. The optimum mineralogy is reported to consist of unreacted hematite nucleuses surrounded by acicular silicoferrite of calcium and aluminium (SFCA) and a low fraction of magnetite [4, 5]. At higher temperatures and lower pO2, SFCA is decomposed to magnetite and gangue [6, 7] forming a less desirable product [8]. Magnetite concentration is commonly used as a measure of heat imparted to the sinter and is an indication of quality [9].

The micro-structure of iron ore sinter has been investigated previously using pure bonding phase melt tablets [10] and analogue sinter tablets [11]. The adhering fines composition in these studies replicates what was measured in the −0.5 mm size fraction attached to the outside of granules in an industrial sinter strand. The basicity of this material was 2.0. By replicating this adhering fines material, the authors studied how it is transformed during sintering, with and without interaction with the nuclear ore particles. These studies were carried out in air, so did not take into consideration the variation of the partial pressure of oxygen (pO_2) that occurs in the industrial sintering system. Optical microscopy techniques were used to quantify the size and shape distribution of pores in the samples. Using optical microscopy has the disadvantage of only being able to view cross sections of the sample. This limits the area of the sample that can be analysed and does not provide information on the connections between pores in the sample. Using high resolution X-ray computed tomography (HRXCT) overcomes these drawbacks, as it is able to characterise the way pores throughout the sample are connected in three dimensions. The HRXCT technique can characterise the distribution of pores throughout the samples based on whether they are connected to the outside surface of the sample (open pores) or if they are entirely surrounded by solid (closed pores). It can also be used to quantify the sphericity of the

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pores. This is an improvement on the 2-D circle factor of the pores used in [10, 11]. An increase in the sphericity of all pores (open and closed) in the melt occurs as the degree of coalescence of the sinter increases. This is driven by the surface tension of the melt minimising the surface energy of the bubbles in the melt [12]. In this way, sphericity indicates how far the sintering reactions and reshaping has progressed. For analogue sinter tablets that all start out having the same volume, a good indicator of the degree of coalescence on the macro scale is to measure final volume of the tablets. Tablets that have undergone more coalescence have a lower final volume.

X-ray CT is a non-destructive technique that has been used to investigate the structure of many materials to determine the shape and connections of pores in 3-dimensions. Early uses of this technique for iron ore sintering focused on the formation of the sinter cake structure [13–17]. Hot stage X-ray CT techniques used by a number of these researchers showed how the gas channels in the sinter cake form across a range of conditions (alumina concentration, raw materials segregation, bed bulk density, natural gas injection and others). By using large samples, the resolution of the scans was limited to around 0.25 mm voxel size hence the fine detail (<~1 mm in size) of the sinter product could not be resolved. More recent work by Shatokha et al. [18] focused on the structure of the iron ore sinter product using higher resolution scans (20 μm voxel size). Shatokha segmented the porosity into open and closed pores. A relationship was shown between basicity and open porosity, however no correlation was found between basicity and closed porosity.

Variability in the structure of industrial and sinter pot test sinters comes from the uneven heat distribution down the sinter bed and from the heterogeneous distribution of coke and flux particles. Also the local pO_2 changes as the flame front approaches and passes. These factors influence the volume and distribution of the melt, in turn influencing the structure. X-ray CT scans of industrial and pot grate sinters using synchrotron radiation showed high variability in the structure of similar samples [19]. To reduce the variability in the sinter being studied and to increase the resolution of the scans (4.5 μm voxel size), analogue sinter tablets were created in a rapid heating furnace (RHF) and scanned using HRXCT. The aim of this study was to use HRXCT to characterise analogue sinter as part of a research program aimed at understanding the impact of structure on sinter quality. A full factorial experimental design methodology [20] was used varying maximum temperature, pO2 and ore type as independent variables and measuring total tablet volume and porosity as dependent variables. Mineralogy and FactSage calculations were used to elucidate reasons for the results.

Specific aims are to:

- Measure analogue sinter tablet total volume and assess statistical significance of sintering factors on total volume
- Measure analogue sinter tablet porosity and assess statistical significance of sintering factors on porosity
- Segment porosity into open and closed pores and compare pore volumes
- Relate predicted liquid fraction to measured porosity and pore sphericity
- Compare measured magnetite formation with FactSage analysis

2. Experimental

2.1. Raw materials

Analogue sinter tablets were created from natural ore nuclear particles (NP) and a mixture of laboratory grade chemical reagents to simulate adhering fines. The NP used were a tight size fraction (−1 + 0.71 mm) collected by wet sieving from a representative sample of iron ore fines. After wet sieving the NP were dried for at least 10 h at 105 °C. The adhering fines were fine laboratory reagent powders (<5 μm) mixed in a large batch under acetone. Table 1 shows the chemistry of the adhering fines and nuclear particles. The same adhering fines chemistry and ratio of adhering fines to nuclear particles as previous studies was used [10, 11]. XRF was used to measure ore chemistry and adhering fines chemistry was created by weighing pure chemical powders. The basicity of the combined NP and adhering fines was 0.93 and 0.99 for ore A and B respectively.

Table 1
Chemistry of ores and adhering fines (mass %, dry basis).

	Fe	FeO	SiO2	Al_2O_3	CaO	MgO	LOI 1000
Ore A	58.70	0.00	4.59	1.11	0.04	0.06	10.15
Ore B	61.60	0.41	4.13	2.17	0.06	0.04	5.24
Adhering fines	51.67	0.00	4.54	1.92	10.53	0.88	8.26[a]

[a] LOI (Loss on ignition) from decomposition of $CaCO_3$ and $MgCO_3$ (calculated).

The gas atmosphere inside the furnace was controlled using air with $pO_2 = 0.21$ atm and bottled N_2 and O_2 mixture with pO_2 5×10^{-3} atm. Bottled gas was prepared to ISO standard 6142. The thickness of the nickel foil sleeve on the tablets was 25 μm and the purity was 99.9% Ni.

2.2. Green tablets

Apparatus: Green sinter tablets were prepared using

- A three decimal place electronic balance for weighing the tablet materials
- A 6.0 mm internal diameter stainless steel die mould
- Shimadzu AGS-D Autograph Universal Tester with 10kN load cell
- Other standard laboratory equipment

Procedure: The method of creating the unfired tablets was the same as that used by previous researchers [21]. To prepare an unfired analogue sinter tablet 0.36 g of ore nuclear particles and 0.24 g of adhering fines mixture were weighed into separate vials. Using a glass dropper, a single drop of water (60 μL) was added to the vial containing the nuclear particles. The vial was then shaken to mix the water and ore. Adhering fines were then added to the wet ore and mixed with a spatula for approximately one minute. The mixture was then transferred to the mould then loaded at 5 mm/min using the Shimadzu to a maximum load of 900 N.

2.3. Furnace

Apparatus: Tablets were sintered in an Ulvac Riko MR-39H/D furnace. This furnace shown in Fig. 1 has two infra-red lamps and gold mirrors that focus infra-red light onto a platinum crucible in the centre of the furnace. The crucible was supported from below by a type B control

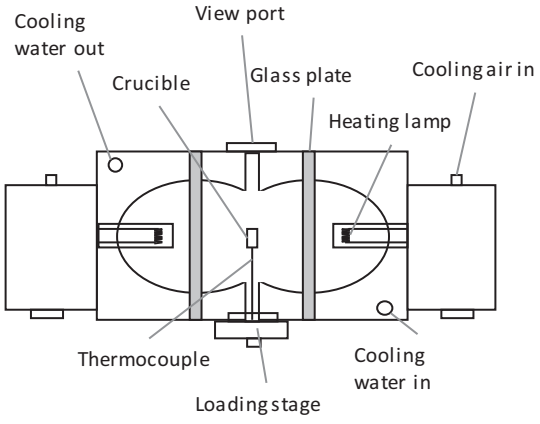

Fig. 1. Diagram of rapid heating furnace.