An Analysis of the Structure of Iron Ore Sinter Cake

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Physical properties of sinter cake such as strength and permeability are strongly related to its structure. In this investigation, the 3-dimensional structure of voids in sinter cakes prepared from raw mix with a range of coke contents was quantified using image analysis. The physical properties were then compared with the measured structural parameters.

The results obtained are of a preliminary nature and the interpretations are not conclusive. However, a possible way to evaluate sinter strength was identified which utilizes the measured values of void fraction and specific surface area of the sinter cake. The work also showed that when a sinter cake is considered to be a packed bed, the "apparent" diameters of the particles used for estimating gas flow resistance across the bed and heat exchange rates between gas and solid are different for the respective transport phenomena.

A theoretical analysis of the magnitudes of forces acting on granules was made to identify the causes of the rearrangement of a bed during sintering.

KEY WORDS: sinter; iron ore; structure; permeability; image analysis; sinter cake; void fraction; heat exchange rate; pore.

1. Introduction

Structure of sintering bed of iron ore, characterized, for example, by the void fraction and surface shape of the solid, is an important factor which directly affects permeability of the bed and strength of the sinter product. The structure is influenced by the properties of the raw materials and the operating conditions of the sintering process. An analysis of the change in the bed structure during sintering is extremely complex because the bed structure itself influences gas flow rate through the bed which is one of the key process parameters.

It is necessary to quantify the permeability and heat exchange characteristics of a sintering bed for making more accurate mathematical models of the sintering process. Generally, Ergun's equation and Ranz-type equation have been adopted to describe the permeability and heat transfer, respectively. Mean particle size and void fraction are included in the permeation resistance coefficients in Ergun's equation as representative structural characteristics. The Ranz's equation makes use of the particle diameter and, for a packed bed, an effective mass flow rate of fluid equal to 9 times the superficial velocity. An alternative approach when using the Ranz equation for packed beds is to modify it to include the void fraction. Kunii and Suzuki's equation has also been used to describe heat transfer in sintering beds. This equation makes use of the particle diameter, void fraction and a channeling factor to characterize the bed structure. The channeling factor is defined as the ratio of the average channel length to the particle diameter and is also a parameter showing the frequency of causing paths of flow.

All these approaches have been limited due to the structural changes in sintering bed from a packed bed, through a semi-molten state to a solid cake state permeated by voids. In addition, the driving forces to this structural change and the influences of properties and quantity of melt formed in the sintering bed have not been investigated in detail. Analysis of the mechanical strength of sinter product based on strength theory of porous media have been made, however, the relation between the strength and the structure of sinter cake is not clarified yet. The reason is due to the difficulty of direct observation of the bed structure.

Recently, non-destructive methods was reported on the structure of sinter cake obtained using X-ray CT. The resolution of the X-ray CTS is not sufficient for detailed analysis at present but improvement in the capacity of the hardware is expected in the near future. On the other hand, image analysis has been used for quantitative analysis of mineral phases in sinter. The remarkable advance made in image analysis has been in the measuring precision and speed. However, it is difficult to obtain 3-dimensional information on sinter and bed structure because the measuring objects are limited to flat sections and the textures and structures are complex.

In the present study, several sinter cakes, which were prepared with various coke contents in the raw mix, were subjected to image analysis by slicing the cakes thinly and examining the flat surfaces of the plates. Some results on the structural characteristics of the cake and on their relations to strength, permeability and effective heat exchange area between gas and solid were obtained. In attempt to understand the formation of the structure of sinter cake, driving
forces which produce the changes in bed structure were analyzed quantitatively.

2. Theory of Measurement of Structural Properties

Consider a cylindrical bed having cross-sectional area of \( A \) and height of \( L \). Let the distance from top of the bed be \( z \). Consider the bed to be made up of \( n \) horizontal slices each \( J_z \) thick. Let \( i \) be the number of a slice from the top of the bed; \( i \) varies from 0 at the top to \( n=\frac{L}{J_z} \) at the bottom of the bed. The internal surface area, \( S \), of the bed can be calculated from the perimeters of voids \( L_p(z) \) and \( L_p(i) \) at the position \( z \) and \( i \), respectively, as follows:

\[
S = \int_0^L L_p(z)dz = \lim_{n \to \infty} \sum_{i=1}^n (L_p(i)J_z) = L \lim_{n \to \infty} \sum_{i=1}^n (L_p(i)/n) = \frac{L \bar{L}_p}{\bar{L}} \quad \text{..................(1)}
\]

where, \( \bar{L}_p \) : the average perimeter of voids in the cake.

Eq. (1) shows that the surface area is proportional to the perimeter of voids, the proportionality constant, \( L \), being the height of the bed. Therefore, surface area per unit volume of cake, \( A_s \), is given by:

\[
A_s = \frac{S}{A/L} = \frac{\bar{L}_p}{A} \quad \text{..................(2)}
\]

The specific surface area of cake \( A_p \) is given by:

\[
A_p = \frac{S}{A/L(1-S_p)} = \frac{\bar{L}_p}{A(1-S_p)} \quad \text{..................(3)}
\]

where, \( S_p \) : the average fractional area of voids and is equal to the void fraction, \( \varepsilon \), in the bed.\(^{(10)}\)

Therefore, the surface area, specific surface area and void fraction of the bed can be obtained substituting representative values of \( \bar{L}_p \) and \( S_p \) obtained by image analysis into the above equations.

3. Experimental

3.1. Sample Preparation

In order to prepare several sinter cakes having different structures, the coke content of raw mix was varied over 5 steps from 4.0 to 5.0 mass\% (named T-1 to T-5, respectively). An Australian hematite ore (T:Fe: 59.1, CaO: 0.04, SiO\(_2\): 7.35, Al\(_2\)O\(_3\): 2.84, Loss on ignition: 5.1 in mass\%), Japanese limestone (T:Fe: 0.22, CaO: 54.6, SiO\(_2\): 0.56, Al\(_2\)O\(_3\): 0.28, Loss on ignition: 43.2 in mass\%), serpentinite and metallurgical coke were used as raw materials. Return sinter fines were prepared by primary sintering of the same raw mix. The target composition of the sinter was CaO: 11.7, SiO\(_2\): 6.5, Al\(_2\)O\(_3\): 2.3, MgO: 1.7 in mass\%, and its basicity (CaO/SiO\(_2\)) was 1.8. The sintering tests were conducted in a cylindrical pot at the Central Research Laboratories, BHP, Australia. The sintering conditions are listed in Table 1. The bulk density of the charged bed and the sintering time for each test are shown in Table 2.

![Fig. 1. Method of preparing sinter samples for image analysis.](image)

After firing, the sinter cakes were carefully removed from the pot. A cube of sinter of side 100 mm was cut from the center of each cake as illustrated in Fig. 1. The cubes were mounted in white-colored epoxy resin then cut horizontally to form thin plates. The resin was colored to make the boundary between the resin (void) and sinter clear. The average thickness of the plates was 2.1 mm. A portion of cake of thickness 0.9 mm was lost in each cut. The remainder of each cake was given a shutter treatment by dropping it 4 times from the height of 2 m. Finally, 6.65 kg of the sinter (+8.0 mm) was subjected to an ISOM tumble test.
plates for each sinter cake from the upper to lower slices.

The images were first treated by software to resolve
the resin as voids from the sinter, then they were
binarized. The void fraction and perimeter of voids
were measured using commercial software for each
binarized image. Then, channels less than 1 mm
wide between large voids were blocked off using the
software and the circle equivalent diameters and shape
factors (4πA/πL²): where, A and L are the sectional
area and perimeter of void, respectively, were
determined for the pores.

4. Experimental Results and Discussion

4.1. Relation between Cake Content of Raw Mix and
Strength of Sinter

The strength of the sinter measured by the ISO
tumble test method is shown in Table 2 and Fig. 2.
The relation between coke content and sintering time
is also indicated in Fig. 2. The strength of sinter
increases with an increase in coke content but flattens
at high coke levels. The sintering time has a mini-
imum value at about 4.2 mass% of coke and is pro-
longed with an increase in coke content. This trend
agrees with the results reported previously. [2]

4.2. Structure of Sinter Cake

The void fraction and specific surface area ob-
tained for each cake are given in Table 3. Each
value is the average of about 40 measurement fields
of about 69 mm x 69 mm. The calculated shape fac-
tors and average circle equivalent diameters of pores,
and D, are also presented in the Table. D, is the
diameter of a sphere having the same specific surface
area as the cake and is equal to 6/\(\Delta_{s}\).

The shape factor of pores increases with an in-
crease in coke content. This suggests that surface in
a cake becomes smoother due to increase in the
amount of heat of combustion which leads to an in-
creased amount of melt and decrease in its viscosity.

Fig. 3 shows the distribution of void fraction in the
vertical direction for the cake T-3 which is typical of
the results. The area fraction of voids fluctuates con-
siderably in the vertical direction. However, some
regularity is apparent in the change and this has a
periodicity (or wave length) of about 15 mm. This
trends was clearer for the cakes sintered with higher
coke content.

The value of the measured area fraction of voids
will approach the value of the average void fraction
with an increase in the area of the object field for
porous solids and packed beds due to the averaging
effect. Therefore, in order to investigate local fluc-
tuations of void fraction it is necessary to restrict the
size of the object area to near that of the crosssec-
tional area of the corresponding unit cell of the
porous solids and bed. This can be demonstrated by
examining some simple packing models.

Consider a regularly packed lattice, such as simple
cubic, body-centered cubic (BCC) or face-centered
cubic (FCC), consisting of spheres having the max-
imum diameter, \(d_p\), the closest spheres touch each
other. The distribution of void fraction in a vertical

\[
\text{Table 3. Characteristics of sinter cake structure obtained by image analyzer.}
\]

<table>
<thead>
<tr>
<th>Cake</th>
<th>Void fraction (v)</th>
<th>Surface area in cake (A_s) ((m^2/m^3-cake))</th>
<th>Specific surface area of cake (A_s) ((m^2/m^3-solid))</th>
<th>(D_s) ((mm))</th>
<th>Average diameter of pore ((mm))</th>
<th>SD (\pm)</th>
<th>Average shape factor (\tau)</th>
</tr>
</thead>
<tbody>
<tr>
<td>T-1</td>
<td>0.433</td>
<td>300</td>
<td>548</td>
<td>10.9</td>
<td>8.13</td>
<td>0.676</td>
<td>0.298</td>
</tr>
<tr>
<td>T-2</td>
<td>0.437</td>
<td>271</td>
<td>481</td>
<td>12.5</td>
<td>7.42</td>
<td>0.712</td>
<td>0.305</td>
</tr>
<tr>
<td>T-3</td>
<td>0.432</td>
<td>248</td>
<td>437</td>
<td>13.7</td>
<td>8.01</td>
<td>0.658</td>
<td>0.347</td>
</tr>
<tr>
<td>T-4</td>
<td>0.432</td>
<td>252</td>
<td>444</td>
<td>13.5</td>
<td>7.92</td>
<td>0.810</td>
<td>0.357</td>
</tr>
<tr>
<td>T-5</td>
<td>0.439</td>
<td>249</td>
<td>433</td>
<td>13.9</td>
<td>8.02</td>
<td>0.814</td>
<td>0.362</td>
</tr>
</tbody>
</table>

\(\Delta_s\): Diameter of a sphere having same specific surface area as sinter cake
\(\pm\): Standard deviation of diameters of pores
\(\tau\): Average value of pores with sizes in the range from 2.5 to 20 mm in diameter

Fig. 2. Variation of tumble index and sintering time with the coke content of sinter mixes.

Fig. 3. Change of void fraction in vertical direction of cake for Run T-3.
plane along the side of a unit cell for each cell type is shown in Fig. 4. From the figure, it can be seen that the void fractions change periodically with a wave length equal to the side of unit cell \((d_p)\) for a simple cubic lattice, and equal to the half side of unit cell, \(d_p/\sqrt{3}\) and \(d_p/\sqrt{2}\), for BCC and FCC lattices, respectively. The wave length is constant for these ideal lattice arrangements if the object area is of the same order as the sectional area of each unit cell.

The wave length of the cake T-3 is about 15 mm and, assuming the unit cell of the cake is simple cubic or BCC, the side of each unit cell is 15 or 30 mm, respectively. Accordingly, distribution of the void fraction of all sample cakes in the vertical direction was re-measured using a field of 30 mm \(\times\) 30 mm.

Fig. 5 demonstrates the distribution of void fraction measured for each sample. The wave lengths of the fluctuation can be estimated as about 9 mm for T-1, 11 mm for T-2, 13 mm for T-3, 19 mm for T-4 and 30 mm or greater for T-5. Assuming that the structural characteristics of each cake are basically similar, this suggests that the diameter of “apparent” particles composing the sinter cake becomes greater with an increase in coke content in the raw mix. A higher maximum bed temperature and longer holding time at high temperatures are normally observed when coke content is increased. Therefore, it seems that the amount of melt formed during sintering increases with an increase in coke content and the coalescence of granules is promoted as a result. \(D_v\) increases with an increase in coke content, from 11 to 14 mm, but the increase is not as great as suggested by the increase in wave length of the void fraction. Presumably, this is because sinter cake is not a packed bed of particles but rather a continuous solid containing pores and the analogy can not be rigorously applied.

4.3. Relation between Sinter Strength and Structure

The strength of porous medium can often be estimated by the strength of its matrix and the porosity. A model\(^{19}\) for estimating sinter strength has been proposed which uses strengths of various matrixes and the porosity. The sinter used for the tumble test in the present experiments was the product obtained by dropping then screening the cake. In order to predict the strength of the product from the structure of cake or to improve the quality of the product by controlling the structure of cake, it is necessary to know whether or not each void in the cake is preserved in the product. Previous work has shown that there is indeed a positive correlation between the void fraction of sinter product and of sinter cake\(^{11}\) (when hollows on the surface of sinter product are included as voids) and that the voids in a cake are almost entirely preserved in the product sinter.\(^{14}\) There does appear to be negative correlation between the void fraction of cake and tumble index of the sinter product obtained in this study as shown in Fig. 6 though the results are not conclusive.

Void structure in sinter is not uniform and the magnitude of the void fraction does not completely characterize the structure of sinter. A more complete characterization is provided by considering void
fraction and the surface area of the sinter together. These may be combined in a single parameter, specific surface area. When tumble index is plotted against the specific surface area as in Fig. 7, a better correlation than with void fraction is obtained. A similar correlation, obtained from a study of plant sinters, was recently reported by Busby and Fray.  

4.4. Relation between Specific Surface Area of Sinter Cake and Effective Area for Heat Exchange, and Permeability

As mentioned before, the permeability and specific surface area of a sintering bed are important factors in mathematical models of sintering process since these determine the gas flow rate through the bed and heat exchange rate between the gas and solid, respectively. Previous investigators have estimated these factors using experimental data under various assumptions. However, the reliability of the values is not sufficient.

The proportion of closed pores in the total pores measured in this experiment is thought to be very small, so the influence of the closed pore has been ignored in the following discussion. The specific surface area of the sample cake were in the range 430 to 550 m²/m³-solid and the corresponding diameter of apparent particles (Dₐ) was from 11 to 14 mm. These values agree well with the "apparent" particle diameter calculated for the sintered zone from the cooling profile of sintering beds.

Ergun's equation has usually been used as the permeation equation for sintering beds. The Ergun's equation can be written as follows:

\[ \frac{dP}{L} = k_1 \varepsilon + k_2 \varepsilon^2 \Delta \]  

where, the permeation resistance coefficients \( k_1 \) and \( k_2 \) are given by:

\[ k_1 = 150(1-\varepsilon)^2/(\varepsilon d_p^2) \]  

and

\[ k_2 = 1.75(1-\varepsilon)/(\varepsilon d_p) \]  

The values of \( k_1 \) and \( k_2 \) obtained by substituting void fraction and \( D_p \) for each cake into \( \varepsilon \) and \( d_p \) in the Eqs. (5) and (6) are given in Table 4. Since values of \( k_1 \) and \( k_2 \) decrease with an increase in coke content, it can be concluded that the permeability of the sintered zone improved with an increase in coke content. There is a region in Fig. 2 where the sintering time increases as the coke content increases. This may be caused by the increases in maximum temperature and thickness of the high temperature zone. Therefore, it is possible that the influence of an improvement in permeability of the sintered zone on the permeability of the whole bed during sintering is relatively small.

The values of \( k_1 \) and \( k_2 \) obtained by parameter fitting method using the mathematical model reported by Yoshinaga and Kubo and those obtained by measurement of pressure drop by Shibata et al. are also listed in Table 4. The former values are in reasonable agreement with those obtained in this study. However, the latter values are considerably larger. Shibata et al.'s values are likely to be more realistic because they were obtained by the direct measurement. Comparing the values with those of this study, \( k_1 \) is about 100 times and \( k_2 \) is about 15 times larger. If the void fraction of the sinter cake is assumed to be 0.45, which is nearly the same value for the sinter cakes used in the present study, the "apparent" particle diameter can be calculated as about 1 mm. This is approximately 1/10 of the value of \( D_p \) obtained in the present study. This difference can not be explained by the difference in sintering conditions.

The closed pores which do not take part in gas flow could be a factor making for error in the determination of void from perimeter measurements. However, the proportion of closed pores is small and, besides, \( D_p \) would become larger in that case since neglect of the closed pores makes the perimeter of voids smaller. Sinter cake has voids of irregular shape and a channeling structure. Accordingly, the structure is significantly different from that of a packed bed. If pressure drop in channels is dominant, \( d_p \) calculated using Eqs. (4) to (6) (in which a packed bed structure is assumed), will be smaller than the "apparent" diameter determined by image analysis. Therefore, it is not surprising that the "apparent" particle diameters used for estimating pressure drop and the heat exchange rate between gas and solid are different in the two cases.

5. The Driving Forces of Structural Change in Sintering Beds

The structural change in a sintering bed is usually
observed as a contraction of bed height and increases in void fraction and "apparent" particle diameter. These may be caused by the disappearance of substances due to various reactions and by the rearrangement of particles due to melt formation, the flow of gases and the gravitational force. The principal reactions are dehydration, decomposition of limestone and coke combustion.

The driving forces of the structural change in sintering bed have been examined from a fundamental perspective in this study.

The following forces act on particles in sintering bed:

i) Compressive force due to gravity: \( F_s \) (N/m²)

ii) Capillary bonding force between particles wetted by melt: \( F_p \) (N/m²)

iii) Frictional force developed by gas flow through the bed (pressure drop): \( F_f \) (N/m²)

The compressive force, \( F_s \), at the distance of \( z \) from the top of a bed is given by:

\[
F_s = \rho g z \cos \theta
\]

The capillary bonding force between two particles, \( H_p \), in pendular state as illustrated in Fig. 8 is given by:

\[
H_p = \gamma d_p \sin \beta [\sin (\beta + \theta) + (d_p/4)(1/R_1 - 1/R_2) \sin \beta]
\]

where, \( R_1 = [d_p(1 - \cos \beta) + 1]/2 \cos (\beta + \theta) \)

\( R_2 = (d_p/2) \sin \beta + R_2 \sin (\beta + \theta) - 1 \)

For the purpose of comparing the relative magnitude of these forces, \( d_p \) was taken as 0.002 m, \( \gamma \) as 0.61 N/m, which is the surface tension of the melt in CaO (20 mass%-Fe₂O₃) system at 1250°C and \( \beta \) as 60°. Both \( \theta \) and \( l \) are assumed to be zero. This assumption is adequate because \( H_p \) is not significantly dependent on the volume of melt when the amount of the melt is small. \( H_p \) is calculated to be 2.55 x 10⁻³ N using these values. The capillary bonding force per unit cross-sectional area, \( F_{cp} \), is given by:

\[
F_{cp} = 9.8((1 - l)/4\pi k)kH_p
\]

\[
= 660(1 - l)/4\pi k
\]

where, \( k \): the co-ordination number and is ideally given by \( k = \pi/4 \).

The bonding force becomes about 3 times as great as that calculated from Eq. (9) when the particles are in the capillary state.

The friction force, \( F_f \), in a packed bed for a uniform gas flow is equal to the pressure drop across the bed. The \( F_f \) was estimated using pressure drop calculated by applying Ergun's equation for the height corresponding to diameter of particles on the assumption that the particles are rigid bodies. The \( d_p \) for the calculation of \( F_f \) should change with a change in the bed structure. In this calculation, however, \( d_p \) was taken as a constant 0.002 m in order to estimate the magnitude of \( F_f \) at an early stage of sintering. The value of \( F_f \) will decrease as sintering progresses because the apparent particle size and the void fraction of the bed will increase.

The value of each force, calculated as above, is shown as a function of the void fraction in Fig. 9 and as a function of superficial velocity converted to the value at N.T.P. in Fig. 10. It is found that \( F_f \) is relatively small compared with the other two forces. Therefore, \( F_s \) and \( F_p \) will be dominant except in the region where the gas velocity is extremely high, such as in channels. \( F_s \) depends on the distance from the top of bed and value becomes several times larger than \( F_p \) in the lower part of the bed. Since \( F_s \) acts consistently downwards and \( F_p \) has no preferred direction, the structural change that occurs during sintering can be understood to be the result of the
balance of these two forces. In other words, $F_c$ compresses the bed uniformly, while $F_p$ promotes coalescence of particles and formation of large voids; and $F_f$ acts locally in a supplementary manner to enlarge channeling spots.

6. Conclusion

An image analysis study was conducted on sliced samples of several sinter cakes obtained by varying the coke content in the raw mix. The values of void fraction and specific surface area, determined from the slices, were compared with the strength of sinter products and the "apparent" particle diameters used for estimating the heat exchange rate between gas and solid and pressure drop. As a result, evidence was found that the strength is correlated with void fraction and specific surface area of the cake. Since the structure of sinter cake is different from that of an ideal packed bed, it is not surprising that "apparent" particle diameter required for estimating the heat exchange rate is considerably different from that for the pressure drop. The results indicate that difference between these two diameters might be a factor of about 10. This value is based on a comparison of sinter test results conducted under different sets of condition and, therefore, should be regarded as an estimate only.

The driving forces of structural change in a sintering bed are of three types; viz., compressive, capillary and friction forces. A comparison of the magnitudes of these forces showed that the structural changes in the sintering bed proceed mainly as a result of the balance of the compressive and capillary forces.

Nomenclature

$D_s$: Diameter of sphere having same specific surface area as sinter cake (m)
$d_p$: Diameter of sphere or "apparent" particles (m)
$g_s$: Gravitational acceleration [9.807] (m/s²)
$L$: Height of bed (m)
$H_f$: Capillary bonding force between two particles (N)
$\Delta P$: Pressure drop across bed (Pa)
$u, u_0$: Velocity and superficial velocity of gas, respectively (m/s)
$\gamma$: Surface tension of melt (N/m)
$\varepsilon$: Void fraction of bed (–)
$\mu$: Viscosity of gas (Pa·s)
$\rho_s, \rho_g$: Bulk density of bed and density of gas, respectively (kg/m³, bed, kg/m³)

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