Structure Analysis of High Strength Coke Using X-ray CT

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(Received on December 10, 2018; accepted on June 11, 2019)

Development of coke production technology which can maintain high coke strength with use of certain amount of high rank coal is needed. Accordingly, it is important to investigate influence of coke pore structure on coke strength and dominant factors of coke strength. In this study, results of three-dimensional analysis such as components of coke structure and Mises stress of two types of high strength coke will be presented. Three-dimensional composition of coke was successfully visualized and quantified by three-valuation method. Moreover, three-dimensional stress analysis was conducted to investigate the relationship between coke structure and coke strength.

KEY WORDS: coke strength; pore structure; X-ray CT; 3-D stress analysis.

1. Introduction

Coke is needed to act as reductant, spacer and to provide heat for the blast furnace. Especially, the strength of coke is important physical property for support the iron ore charged and for passing gases through melting zone in blast furnace. A variety of studies have been investigated to use certain amount of low quality coal for producing high strength coke. Coke is a porous material that consists of matrix and pores. The microstructure of coke is known to influence its strength.1,2) Accordingly, it is essential to understand dominant factors of coke strength and influence of coke pore structure on coke strength. Various researches of evaluating coke strength by using quantitative analytical method have been done.1,3–7) Anraku et al. established one of stress simulation method by using 3-D images of coke which were taken by X-ray CT.9) Hiraki et al.10) investigated relationship between coke microstructure and coke strength by using numerical analysis of three-dimensional micro X-ray CT as well. They compared a high strength coke with too low strength coke for practical use. In this study, components of coke structure, shortly pore, matrix and highly dense matrix (include mineral matter), were discriminated by using three-valued analysis of three-dimensional micro X-ray CT. Moreover, Mises stress analysis was conducted on two types of high strength coke to be compared.

2. Experimental

Two coke samples: coke A and coke B were produced from different composition of caking coals. Table 1 shows coal characteristics and mixing rate of the coals used for two coke samples. Two samples were prepared by heated in a test coke oven (430 mm in width, 600 mm in length and 600 mm in height) under same conditions as shown in Table 2.

Table 1. Coal characteristics and mixing rate of the coals used for coke samples.

<table>
<thead>
<tr>
<th>Coal characteristics</th>
<th>Blend of coal percentage</th>
</tr>
</thead>
<tbody>
<tr>
<td>Coal characteristics</td>
<td>[wt.%]</td>
</tr>
<tr>
<td>Volatile Matter</td>
<td>Mean random reflectance</td>
</tr>
<tr>
<td>[wt.%]</td>
<td>of the vitrinite in oil</td>
</tr>
<tr>
<td>Coal A</td>
<td>21.5</td>
</tr>
<tr>
<td>Coal B</td>
<td>23.2</td>
</tr>
<tr>
<td>Coal C</td>
<td>25.3</td>
</tr>
<tr>
<td>Coal D</td>
<td>34.5</td>
</tr>
<tr>
<td>Coal E</td>
<td>34.4</td>
</tr>
</tbody>
</table>

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DOI: https://doi.org/10.2355/isijinternational.ISIJINT-2018-805
Table 3 shows properties of the coke samples. International Standard specifies methods of ISO1014: 1985 was applied to calculate the porosity of the coke samples. Briefly mentioned, The true density value of coke, crushed to 212 μm, in relation to water and the apparent density value of cokes, i.e. the ratio of the mass of a volume of dry coke to the mass of an equal volume of water were used to calculate the porosity of the bulk cokes. As coke A has higher strength than coke B, coke A and B were named “HS (Higher Strength) coke” and “LS (Lower Strength) coke” respectively. Two specimens were prepared for CT scanning for each coke sample.

Coke specimens were scanned by Micro Focus X-ray CT System (inspeXio SMX-100XT, Shimadzu). Scanning area was $12.8 \times 12.8 \times 12.4$ mm$^3$ and resolution was 0.025 mm/voxel for each specimen. X-ray CT images of the samples were composited to gray-scale voxel images ($5 \times 5 \times 5$ mm$^3$ in size) for coke structure analysis and stress analysis (brighter means denser). Pore (black area), matrix (gray area) and highly dense matrix (white area) in coke structure were discriminated by means of three-valued image processing by utilizing difference of their brightness as shown in Fig. 1. The adaptive histogram-based method incorporating the information of pore and ash content measurement was used to determine the threshold value dividing each segment. Difference of true density between ash contents and carbonaceous material is not consider for determining the threshold value in this study.

Pore shape parameters in three axial directions were calculated by use of pore structure tomographic images ($2.5$ mm$x2.5$ mm) from X-ray CT. The parameters were circularity and sphericity, which were defined as below.

Circularity is defined as the ratio of the area of an object to the area of a circle with the same perimeter.

$$\text{Circularity} = \frac{4 \pi \cdot \text{area}}{(\text{perimeter})^2}$$

A circularity value of 1.0 indicates a perfect circle. As the value approaches 0.0, it indicates an increasingly elongated polygon. Sphericity is defined as one of indicator which measures degree of spherical shape of objects. It is defined as the ratio of surface of a sphere having the same volume as the particle to the surface of that particle.

Table 4. The Young’s modulus and Poisson’s ratio for each component of the model.

<table>
<thead>
<tr>
<th></th>
<th>Young’s modulus [GPa]</th>
<th>Poisson ratio [-]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Matrix</td>
<td>29.4</td>
<td>0.2</td>
</tr>
<tr>
<td>High-descend matrix</td>
<td>68.7</td>
<td>0.2</td>
</tr>
</tbody>
</table>

Fig. 1. Three-valued image processing of voxel image of coke specimen.

Fig. 2. Setting three axis on voxel image of coke specimen. (Online version in color.)

Fig. 3. Calculation scheme for Mises stress visual analysis.
sphericity = \left(\frac{\pi}{6}\right) \cdot \left(\frac{8V}{A^2}\right)^{2/3}

where \(V\) is the volume of the object and \(A\) is its surface area. A sphere has a sphericity of 1.0 and other objects have less sphericity.

Synopsys’ Simpleware™ ScanIP software was used for image processing, segmentation and model reconstruction, with the Simpleware SOLID module used for stress analysis. As the heating direction was not clear for each original specimen (20×20×20 mm³), X-, Y- and Z-axis were set at random (Fig. 2) and partial images (2.5×2.5×2.5 mm³) of coke specimens were randomly selected as shown in Fig. 2.

Pore segment was removed from the coke structure. 3.5 million grid points were generated on the coke structures of matrix and highly dense matrix for each voxel image and subsequently parameters such as Young’s modulus were provided to the coke structure as well. The Young’s modulus and Poisson’s ratio for each component of the model were shown respectively in Table 4.

The stretching was applied by uniformly moving a surface in the uniaxial tensile direction (z-direction). One side of z-axis is fixed but left free on the two sides orthogonal to the z-axis. One point tetrahedron element formulation was adopted to simulate coke matrix with highly dense material. The tetrahedron meshes are suitable for static analysis and works well with complex structures. To simplify the analysis in this study, shared nodes were used at the interface between two physical contact bodies and provide a means of load transfer between parts. Mises stress on each grid point, which generated when tensile load was applied along each axis, was analyzed. Brighter colored part means being subjected to higher stress as shown in Fig. 3.

3. Results and Discussion

3.1. Composition of Coke Structure

Figure 4 shows voxel image of each coke specimen.

<table>
<thead>
<tr>
<th>Sample</th>
<th>pore</th>
<th>matrix</th>
<th>highly dense matrix</th>
</tr>
</thead>
<tbody>
<tr>
<td>(a) HS Coke N=1</td>
<td>49.9</td>
<td>38.7</td>
<td>11.4</td>
</tr>
<tr>
<td>(a) HS Coke N=2</td>
<td>50.9</td>
<td>38.4</td>
<td>10.7</td>
</tr>
<tr>
<td>(a) HS Coke average</td>
<td>50.4</td>
<td>38.6</td>
<td>11.1</td>
</tr>
<tr>
<td>(b) LS Coke N=1</td>
<td>54.4</td>
<td>34.5</td>
<td>11.1</td>
</tr>
<tr>
<td>(b) LS Coke N=2</td>
<td>51.3</td>
<td>39.7</td>
<td>9.0</td>
</tr>
<tr>
<td>(b) LS Coke average</td>
<td>52.9</td>
<td>37.1</td>
<td>10.1</td>
</tr>
</tbody>
</table>

Fig. 4. Voxel images of coke specimens.
Results of coke composition analysis are shown in Table 5. HS coke had lower pore proportion and higher matrix proportion, which corresponded to the tendency of DI$_{150}$ (Table 3) of each coke sample.

3.2. Distribution of Pore Size and Pore Shape Parameter Analysis

Distribution of pore size was measured using surface area measuring instrument (N$_2$ Gas flow method). Consequently, there was little difference between HS coke and LS coke samples; their major pore size were 100 to 150 μm as shown in Fig. 5. Table 6 shows pore shape param-

<table>
<thead>
<tr>
<th>Sample</th>
<th>X-direction</th>
<th>Y-direction</th>
<th>Z-direction</th>
<th>Sphericity</th>
</tr>
</thead>
<tbody>
<tr>
<td>(a) HS Coke N=1</td>
<td>0.772</td>
<td>0.780</td>
<td>0.769</td>
<td>0.782</td>
</tr>
<tr>
<td>(a) HS Coke N=2</td>
<td>0.763</td>
<td>0.765</td>
<td>0.764</td>
<td>0.786</td>
</tr>
<tr>
<td>(b) LS Coke N=1</td>
<td>0.771</td>
<td>0.781</td>
<td>0.770</td>
<td>0.785</td>
</tr>
<tr>
<td>(b) LS Coke N=2</td>
<td>0.760</td>
<td>0.766</td>
<td>0.762</td>
<td>0.792</td>
</tr>
</tbody>
</table>

Table 6. Pore shape parameter (Circularity, Sphericity) of the coke samples.

Fig. 6. Variance of the parameters along Z-axis of the coke samples.

Fig. 7. Sphericity distribution of pores of coke samples.

Fig. 8. Statistical distribution of von Mises stress along each axis.
eter of the coke samples. Variance of the parameters along Z-axis of the coke samples is shown in Fig. 6. The pores of sphericity distribution of the coke samples are shown in Fig. 7. The vertical line in Fig. 7 denotes the number of the corresponding pores to a certain sphericity for a given 3D model (2.5×2.5×2.5 mm³). A three-dimensional network model with pores is used for the interpretation of the size distribution in Fig. 5 and the sphericity distribution of the pores in Fig. 7. There was little difference in the parameter among the coke samples in Figs. 6, 7 and Table 3. It is thought that almost the same value was obtained because each sample has sufficient coke strength for use in a blast furnace, and structural defects leading to the destruction are similar in this analyzed cube (2.5×2.5×2.5 mm³) although these samples were classified as “high strength coke” and “low strength coke”.

3.3. Stress Analysis

Figure 8 showed the statistical distribution of von Mises stress along each axis. As there was certain dispersion among the specimens, clear difference between HS coke and LS coke or correspondence between porosity and stress distribution could not be found. There was not clear correlation between pore shape parameter and stress-concentration index. Thus, there is no relationship between coke pore shape and maximum stress concentration in 2.5 mm cube, which means that it is impossible to estimate amount of stress concentration causing pore structure breakage from an average pore structure parameter. But, it is necessary to increase quantity of samples or extend the analysis area to obtain clear results.

Meanwhile, Mises stress of HS coke (N=1) was less than any other specimens as shown in Fig. 8. Focusing on matrix structure, HS coke (N=1) had more highly dense matrix (include mineral matter) parts merged into matrix of coke than other specimens such as LS coke (N=2) as shown in Fig. 9.

The von Mises stress distribution under uniaxial tensile loading (z-axis) was visualized in Fig. 10. The analysis showed that HS coke (N=1) had more highly dense matrix (include mineral matter) parts merged into matrix of coke than other specimens such as LS coke (N=2) as shown in Fig. 9.

![Fig. 9. Comparison of merged mineral (including inert) into matrix of coke structure.](image)

(a) HS coke (N = 1)

(b) LS coke (N = 2)

![Fig. 10. Illustration of Von Mises stress distribution over an area (2.5×2.5 mm²) under uniaxial tensile loading (z-axis).](image)
results revealed that von Mises’ stress is concentrated in the highly dense region in HS coke, but spreads over both matrix and highly dense region in LS coke. The volume distribution of highly dense matrix in HS coke ($N=1$) and LS coke ($N=2$) was shown in Fig. 11. According to the analysis results, it was found that the highly dense particles with a volume larger than $0.5 \times 10^{-3}$ mm$^3$ are about 13% in HS coke ($N=1$), but only 4.3% in LS coke ($N=2$). This indicates that the highly dense matrix in HS coke has a broader size distribution than that in LS coke. The shape irregularity can be characterized by means of analysis of sphericity distribution, which was shown in Fig. 12. Comparing with the distribution of both HS and LS coke samples, it was observed that there is a slight shift towards lower value in distribution peak of HS coke. This reflects more existence of the highly dense particles with irregular geometry in HS coke than in LS coke.

As separately distributed highly dense matrix (include mineral matter) parts might cause decrease of coke strength by forming non-adhesion grain boundaries \cite{12}, extent of merged mineral (including inert) into matrix could give rise to difference in concentration of stress or coke strength. Further, the highly dense matrix is matrix which is hard to transmit X-ray and shows the presence of inert such as carbonaceous material. There was no significant difference in ash content, pore anisotropy, pore circularity or Mises stress in HS coke and LS coke. However, it is inferred that destruction hardly occurs in coke structure when highly dense matrix connection is formed widely.

4. Conclusions

In this study, three-dimensional composition of coke was successfully visualized and quantified by three-valuation method. Moreover, three-dimensional stress analysis was conducted to investigate the relationship between coke structure and coke strength. The results are summarized as follows:

(1) Coke which has higher strength had lower pore proportion and higher matrix proportion from the result of three-dimensional composition analysis.

(2) There was little difference in pore size distribution between the two cokes. Their major pore size was 100 to 150 μm.

(3) The result of three-dimensional stress analysis indicated that extent of merged highly dense matrix (include mineral matter) into matrix could give rise to difference in concentration of stress or coke strength.

REFERENCES