Note

## High Temperature Reduction of the Stiff Vacuum Extrusion Briquettes under the ITmk3 Conditions

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Stiff vacuum extrusion has been applied for the agglomeration of the iron ore concentrate and fine coal. The products of agglomeration– extrusion briquettes (BREX) were subjected to the high temperature reduction. Under the conditions similar to the ITmk3 process in general. It has been demonstrated that the iron ore and coal BREX could be considered as the alternative to the iron and coal pellets for the high temperature reduction in furnaces like Rotary Hearth Furnace (RHF).

KEY WORDS: extrusion briquette (BREX); stiff vacuum extrusion; ITmk3 process; nuggets; shells; Rotary Hearth Furnace (RHF).

## 1. Introduction

The first commercially acceptable process for the agglomeration of fine iron ores was developed in 1899 by Gröndal and was based on briquetting performed by the equipment similar to that applied for the shaping and hardening of clay bricks.[1)] "Briquettes were made by pressing fine iron ore, mixed with water as a binder, into rectangular shapes about the size of building bricks." The "return" of the brickmaking technology to the agglomeration of fine materials in metallurgy took place only by a century later. This became possible after the development of so called "stiff extrusion" technology (J. C. Steele & Sons, Inc.). The stiff extrusion is taking place under the vacuum and is characterized by the specific combination of the moisture content of the mix and the applied pressure. The working ranges for these parameters are as follows: moisture contents 12–16% (up to 20%); applied pressure for the briquetting does not exceed 3.0–3.5 Mpa.[2)] The products of the extrusion are called BREX (extrusion briquettes). The term was introduced by Aitber Bizhanov in 2012 (priority dated 02.03.2012) and has been registered by Russian Federal Institute of Industrial Property (Certificate # 498006 dated 17.10.2013). The main distinctive features of the stiff extrusion technology were studied and described in details earlier.[3–7)] In particular it has been established that the green BREX do exhibit appropriate mechanical strength and require less amount of the binder to achieve the strength levels required by the metallurgical furnaces. The capacity of the industrial stiff extruders ranges from 15 to 115 tons per hour.

Present paper is devoted to the investigation of the iron ore and coal BREX behavior in the neutral atmosphere at the temperatures range of 1350–1370°C partially simulating the conditions of the reduction of the iron ore and coal pellets in the ITmk3 process developed by «Kobe Steel».[8)]

## 2. Experimental

### 2.1. Materials

For the production of the experimental BREX we have used the hematite iron ore concentrate (63.13% of iron content; $Fe_2O_3$=90.18%, $SiO_2$=4.63%, $Al_2O_3$–3.11%, MnO-0.807%, CaO-0.346%), the coal 1 (bulk density 768 kg/m$^3$; moisture content 7.8%; ash 4%; volatiles 37.7%; 73% particle's size less than 0.6 mm) and the coal 2 (bulk density 800 kg/m$^3$; moisture content 10%; ash 7.1%; volatiles 38.5%; S 0.40%; P 0.021%.; 99% particle's size less than 0.3 mm). The Type I Portland cement (general use) has been used as the binder. Volclay DC-2 Western (Sodium) Bentonite was used as the binder and plasticizer. We have manufactured BREX with the round and oval cross-sections (diameters ½" and 1"; length equal to 1.5–2.0 diameters). The laboratory extruder simulates the processing of the material through the feeder to the pug mill and then to the sealing auger and die, into the vacuum chamber, and then final extrusion. Moisture content was measured using a moisture balance. A calibrated electronic scale with density measuring attachment was used to determine BREX density (Mettler MS603S and Mettler MS-DNY-43).

**Table 1** gives the BREX compositions, shapes and physical properties.

Wet base moisture of the BREX #1 and #2 was 18.5%. For the BREX #3 moisture content was equal to 15.2%. Vacuum applied for the production of the BREX #1 and #2 was at the level of 48.26 mm Hg and for the BREX #3 the value was 38.10 mm Hg.

### 2.2. BREX Mechanical Strength

We have measured the compressive strength of the cured BREX. These values were measured based on the Instron 3345 device (USA). For the measurement procedure we have prepared the required amount of the specially shaped BREX samples and subjected them to the compression. The

**Table 1.** BREX components, shape and physical properties.

| BREX sample | Component, % | | | | Density, g/cm$^3$ | Porosity % | CCS, kgF/cm$^2$ |
|---|---|---|---|---|---|---|---|
| | Iron ore | Coal | Portland cement | Bentonite | | | |
| #1* | 63 | 32 | 5 | – | 2.072 | 34.8 | 59.0 |
| #2 | 63 | 32 | 5 | – | 2.034 | 36.9 | 24.0 |
| #3 | 63.5 | 31.3** | 4.7 | 0.5 | 2.207 | 36.4 | 49.0 |

*oval shape (1.25 cm × 2.5 cm);
**Coal 1

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values of the compressive strength for the BREX were as follows (kgF/cm$^2$): sample #1 - 59.0; sample #2 - 24.0; sample #3 – 49.0. We have observed the influence of the shape of the BREX on the mechanical strength. For the BREX #1 and #2 with the same chemical composition we obtained the significant increase in the compressive strength for the oval shaped BREX when compared with the regular cylindrical form of the BREX.

### 2.3. BREX Porosity

We expected that the increased density of the BREX might prevent their carbothermic reduction. However the results of the porosity study show that for all BREX compositions considered the value of the porosity favors their reducibility. We have applied the Scanning Electronic Microscope LEO 1450 VP (Carl Zeiss, Germany) with the resolution 3.5 nm together with the X-ray computed high-resolution computed tomography system Phoenix V|tome|X S 240 (General Electric, USA). Computed tomography has been used to detect the share of the macro pores (size larger than 100 $\mu$m). For the smaller size pores the SEM had been applied. The approach based on the STIMAN[9] computer software has been used to calculate the porosity detected by SEM. The results of these measurements showed that the total porosity of the BREX #1 was equal to 34.8% (10.9% measured by X-ray computed tomography and 23.9% by SEM) BREX #2 was equal to 36.9% (12.3% measured by X-ray computed tomography and 24.6% by SEM). For the BREX #3 the same value was equal to 36.4% (10.8% and 25.6% correspondingly). The spatial distribution of the pores detected by the X-ray computed tomography is illustrated by the image given in **Fig. 1**.

### 2.4. Reduction Tests

The reduction tests of the iron ore and coal BREX following the conditions of the ITmk3 process we have conducted in the laboratory resistance electric furnaces with controlled atmosphere based on the Electric Furnace SSHVE-1.2, 5/25-I2 (Russia) with vertical arrangement of the graphite heater with the inside diameter equal to 65 mm. We have positioned a working removable Alumina Crucible (98% $Al_2O_3$) in the isothermal zone of the heater. We regulated and stabilized the temperature in the furnace with the help of the thermocouple BP (A) 5/20 located in the isothermal area of the furnace on the outside of the heater. The true value of the temperature we have measured by the thermocouple 2 BP (A) 5/20, located inside the heater and lowered from above to the working Alumina crucible. According to available data on the process of ITmk3 we have selected 1360°C as the working temperature which we maintained with the accuracy of ±10°C (1350–1370°C). Before the test the furnace with the working replaceable Alumina Crucible was pumped out by the forevacuum pump till residual pressure reaches the value of $10^{-1}$Pa and then filled with the argon of high-clean grade. Then we opened the gas discharge to atmosphere and kept the argon consumption through the furnace at the level of 0.5 l/min. We switched on the heating of the furnace, reached and stabilized desired temperature level and dropped the BREX sample through the dosing gateway into the crucible. This very moment was considered as the test start of the test. Thus we have subjected the sample BREX to the rapid heating from the ambient temperature to the working temperature of the furnace. During the every single test we recorded the process on a video using thermal imaging infrared camera «Pyrovision M9000» ("Micron", USA). Sample exposure time was around 15 minutes; if necessary we extended it up to 20 min. After that we depressurized the furnace, extracted the crucible with the products and tempered them in air; installed a new working crucible into the furnace and the new sample BREX into the dosing gateway. We closed the furnace, washed it out by argon and after setting the required temperature we repeated the cycle.

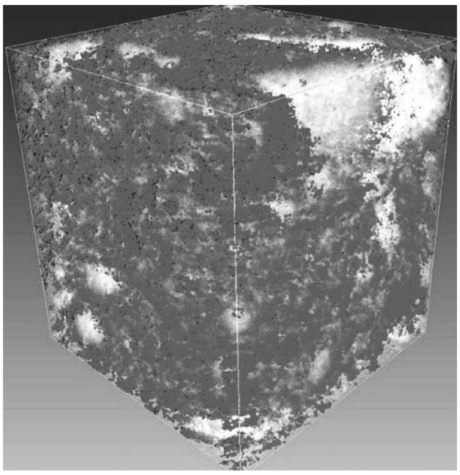

Fig. 1. Spatial distribution of the pores in BREX #2 (X-ray computed tomography; dark and black- pore space; light and transparent –BREX body; resolution 6 $\mu$m).

## 3. Results and Discussions

**Figures 2** and **3** show the photos of the consequent stages of the BREX melting (volatiles release, solid-state reduction, melting and hot metal and slag drop creation) based on the visual data obtained by «PyrovisionM9000».

In all cases, the high density of briquette and large amounts of volatile coal doesn't interfere with the solid-state reduction of iron. Finally the BREX was transformed into the liquid metal (nuggets) and slag drops. In the case of excess of reducing agents we observed the formation of the metallized shells mixed with drops of slag.

Chemical composition of the reduced products (nuggets and shells) is given in **Table 2**. Due to the high content of coal in the BREX the carbon concentration in the metal was at the level of 3.3%–4.8%.

Metal samples (nuggets and shells) were studied using Scanning Electron Microscope Jeol JSM 6490 LV.

All samples but the samples #3 have similar microstructure: small island-type non-metallic inclusions in metal matrix. The smallest size of inclusions is observed in the sample #2 (10–20 $\mu$m), in the sample #1 inclusions have the size 50–60 $\mu$m. There are no any oxides inclusions in the #2 sample. Sample #3 is characterized by nonhomogeneous inclusions of linear form with the width of around 5–10 $\mu$m and length of 200–300 $\mu$m.

As it is seen from Table 2 C and Si content are high in Sample 2. This shows that the reduction material of Sample 2 is excessive compared to Sample 1 and 3. The decrease of

 

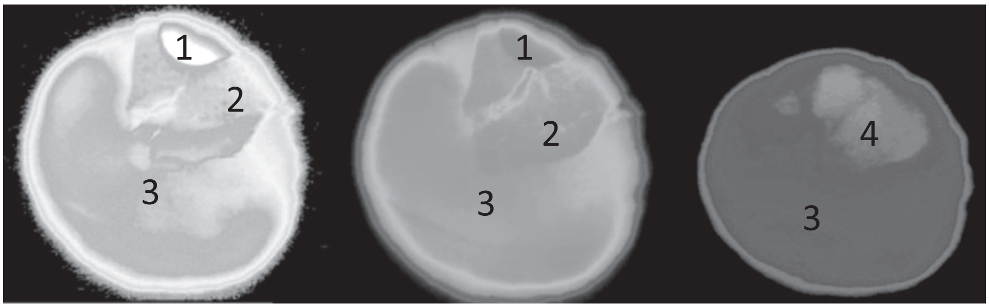


**Fig. 2.** BREX #1 (from left to right: volatiles release, t = 1 min, T = 1 000°C; beginning of the melting, t = 6 min, T = 1 200°C; before complete melting, t = 7 min, T = 1 360°C); 1-thermocouple; 2-BREX; 3-carbonaceous bed zone; 4-melting BREX (solid and liquid).

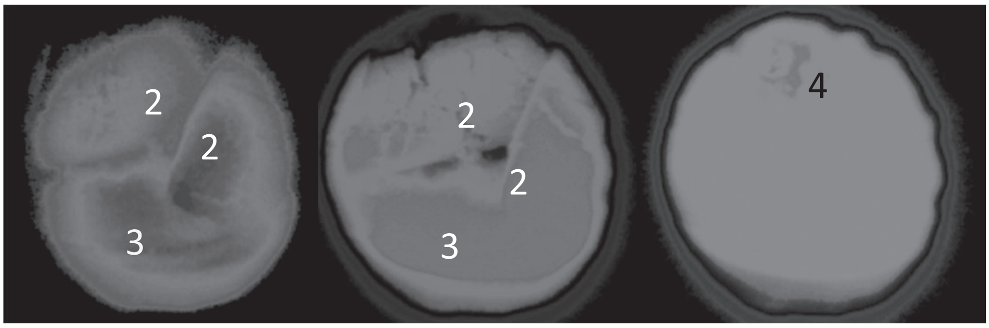


**Fig. 3.** BREX #2 (from left to right: the end of the coal volatiles release, t = 1 min 40 sec., T = 1 000°C; solid state reduction, t = 6 min 37 sec, T = 1 200°C; creation of the nuggets, t = 13 min 20 sec, T = 1 360°C); 2-BREX; 3-carbonaceous bed zone; 4-liquid nugget surrounded by slag.

**Table 2.** Chemical composition of the reduction test products (nuggets and shells).

| Element % | Sample #1 | Sample #2 | Sample #3 |
|---|---|---|---|
| [Fe] | 96.05 | 94.08 | 95.53 |
| [C] | 3.333 | 4.800 | 3.740 |
| [Si] | 0.154 | 0.605 | 0.252 |
| [Mn] | 0.121 | 0.136 | 0.076 |
| [S] | 0.118 | 0.185 | 0.107 |
| [P] | 0.058 | 0.085 | 0.135 |

the reduction material amount in Sample 2 could help BREX strength to be improved. Synchronized thermal analysis methods and Mössbauer spectroscopy has been applied for the study of the phase composition of the BREX (STA 449 C, Germany). A comparison of the peaks of the metallic iron formation shows that the reduction of the metal iron takes place to a larger extent in the BREX samples #1 and #2 (with the coal 2) rather than in the BREX #3 which contains coal#1. Fineness of the coal 2 particles might have influenced on better reducibility.

For a definitive conclusion on the suitability of the BREX, it is necessary to conduct further research on optimization of their size and on the composition of the fluxing oxides in the BREX.

## 4. Conclusions

From the results obtained it follows that:

(1) Iron ore and coal BREX can be considered as the possible alternative to iron ore and coal pellets for the ITmk3 process.

(2) Shape of the BREX cross section can influence on the mechanical strength of the BREX. Surface area of the BREX with oval shape is larger than for the cylindrical BREX of the same weight.

(3) Relatively high density of the BREX contributes to their high thermal shock stability and doesn't interfere with the reducibility.

(4) The porosity of the BREX is sufficient for their good reducibility. Combination of the SEM with the STIMAN software and X-ray computed tomography help to estimate the total porosity of the BREX.

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