

## Optimization of process parameters for recycling of mill scale using Taguchi experimental design<sup>†</sup>

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### Abstract

The present work submits an investigation about the optimum process parameters and quality improvement of mill scale recycling. With increasing concerns on environmental issues, the recycling of materials of all types has become an important issue. In this paper, an optimization method is developed to improve quality in mill scale recycling. The optimum configuration of process parameters to achieve high metallization efficiency was determined by experiments. The Taguchi method, the signal-to-noise (S/N) ratio, the analysis of variance (ANOVA) and response surface optimization are employed to find the main effects and to determine their optimum process parameters. The significant process parameters were identified and their effects on mill scale recycling were studied. Finally, a confirmation experiment with the optimal levels of the process parameters was carried out to demonstrate the effectiveness of the Taguchi method.

*Keywords:* Taguchi method; Mill scale recycling; Chebyshev orthogonal polynomial; Successive approximation; Missing value

### 1. Introduction

Mill scale refers to a form of byproduct that is mixed with moisture (that is, a coolant) in various steel-making processes. Although this material holds a high level content of iron of 70%-75%, it is sold very inexpensively as an industrial waste in the form of iron oxide, rather than collected through a recycling process. Most of this material is reused either as a material in the manufacture of iron ore pellets for a melting furnace or as the coolant of a ladle converter. Recently, with respect to the development of a waste recycling method and the rise in the raw material price of steel products, the recycling process that collects pig iron by producing direct reduction iron (DRI) [1, 2] using high purity iron mill scale has become a subject of much interest.

Mill scale recycling consists of two steps. The first step involves the binder of the mill scale mixing process and the strength evaluation of the mixing ratio, and the second step requires economical processing cost in metallization.

To determine the optimum condition of process parameters to achieve efficient mill scale recycling with respect to the above perspectives, we used the Taguchi method [3-6]. The two major tools used in the Taguchi method are (1) the signal-to-noise ratio (S/N ratio), which measures the quality, and (2) orthogonal array, which is used to study many design variables simultaneously. Rao [7] first proposed the application of the orthogonal array in experiments. Since Yokoyama and Phadke, many other methods have been developed based on the Taguchi method [3, 8-13]. In a significant portion of the literature in this area, the value ranges of the design variables are set as large as possible, and the values of the highly effective design variables increased. However, the experiments of the mill scale recycling process in this paper are extremely time-consuming and costly, also missing values [14, 15] occur for some combinations of design variables. Thus, a common orthogonal array is not appropriate for the experiment design in this paper; we propose an orthogonal array that can determine the sensitivity of process parameters effectively even if there are missing values. The properties of this method are listed as follows:

- (1) Evaluation of the binder and mixing ratio for the mixing process of the mill scale.




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Table 1. Chemical compositions of mill scale.

Component	Continuous casting	Rolling	Desclaer
T-Fe	73.77%	73.18%	72.65%
M-Fe	0.2%	0.1%	0.35%
FeO	64.16%	63.82%	59.49%
Fe <sub>2</sub> O <sub>3</sub>	34.19%	33.56%	37.25%
Sample			

(2) Determination of the optimum condition based on the process parameters that yields 95% metallization efficiency.

## 2. Pre-processing of mill scale

Pig iron is manufactured as a lump by the mill scale recycling process using the direct reduction method. In this reduction method, the mill scale is mixed with the reducing agent. The target metallization efficiency of the mill scale in this process should be such that it yields an iron (Fe) purity of 95%. This process is further explained in the following paragraphs.

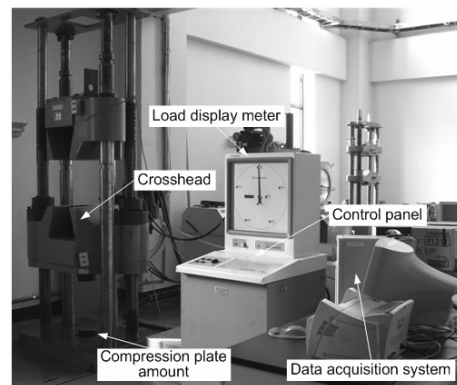
### 2.1 Mixing process of mill scale

If iron is heated to a high temperature, iron oxide is produced as the oxygen in the air combines with the atoms on the surface of the iron. This material is referred to as mill scale. Generally, mill scale is generated in the continuous casting process, rolling furnace and descaler process, and forging process.

Table 1 shows the chemical forming process that generates mill scale. Carbon and hydrogen gas are used as the reducing agents of mill scale. In this paper, metallurgical cokes were used because they have a relatively a better reducing ratio with respect to costs than coal, which is classified as carbon. The binder plays a very important role in improving the strength of the forming material. The binder types are matrix, film and chemical binders. In this paper, we used molasses for the matrix binder and bentonite for the film binder.

### 2.2 Forming method

The forming methods of common mill scale can be divided into the palletizing method and briquetting method, according to the distribution of the grain size. The grain size has a great effect on the mechanical strength of the forming material. More than 30% of the mill scale investigated in this paper had grain size of 1mm or greater. Hence, we used the briquetting method suitable in forming a material consisting of rough particles. While considering the cost of the mixing materials needed in production and reduction, we decided to use the forming agent of rectangular parallelepiped shape of dimensions 20 mm×30 mm×30 mm. For the specimen production



(a)



(b)

Fig. 1. (a) Experimental layout; (b) Compressive strength testing.

process, the samples were prepared by measuring, mixing, discharging, molding, drying and sintering in a muffle box furnace. All the samples were sintered in air at 150 °C, 500 °C and 800 °C (30 minutes) with slow heating and cooling.

### 2.3 Strength evaluation of forming agent

Fig. 1 shows a multi-functional material tester (Shimadzu, UH-100A) used to test the forming material strength of a sintered specimen. The most important part in the mixing process of mill scale is the evaluation of the forming material strength to determine the mixing ratio of the binder.

Table 2 shows the 12 test conditions that were applied to obtain the binder mixing ratio. Fig. 2 shows the graph of the compression strength versus sintered temperature for the forming agents of the test cases in Table 2. When only bentonite was used, the strength greatly increased as the sintered temperature increased, whereas when only molasses was used, the strength increased as the sintered temperature reduced.

Fig. 3 shows the compressive strength of the forming agent according to the mixing ratio of the binder. The compressive strengths of bentonite and molasses increased proportionately to the mixing ratio. The minimum compressive strength required for the forming agent was 0.7 kgf/mm<sup>2</sup>. To satisfy this compressive strength, 3% of bentonite and 5% of molasses were mixed at a mixing ratio of no less than 5%. According to the test result, the mixing ratio of the maximum compressive strength was determined to be 3.5% for bentonite and 1.5% for molasses.

Table 2. Test cases of binder mixing ratio.

Exp.	Bentoite (g)	Molasses (g)	Binder ratio (%)	Mill scale (g)	Cokes (g)
1	4.0	0	1.5	220	46
2	8.0	0	3.0	220	46
3	13.3	0	5.0	220	46
4	0	4.0	1.5	220	46
5	0	8.0	3.0	220	46
6	0	13.3	5.0	220	46
7	2.7	5.3	3.0	220	46
8	5.3	2.7	3.0	220	46
9	4.0	4.0	3.0	220	46
10	4.0	9.3	5.0	220	46
11	9.3	4.0	5.0	220	46
12	6.7	6.7	5.0	220	46

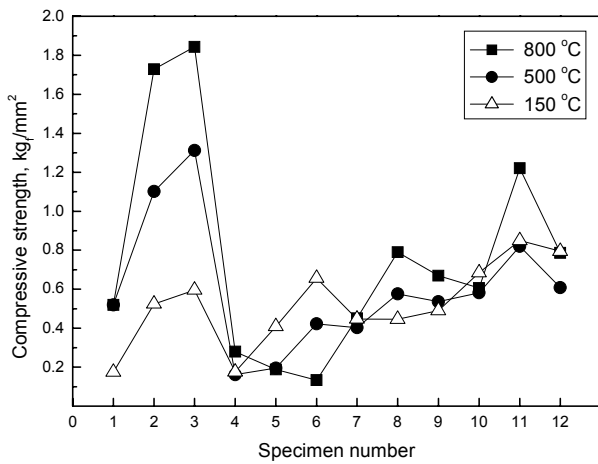


Fig. 2. Relationship between compressive strength and sintering temperature.

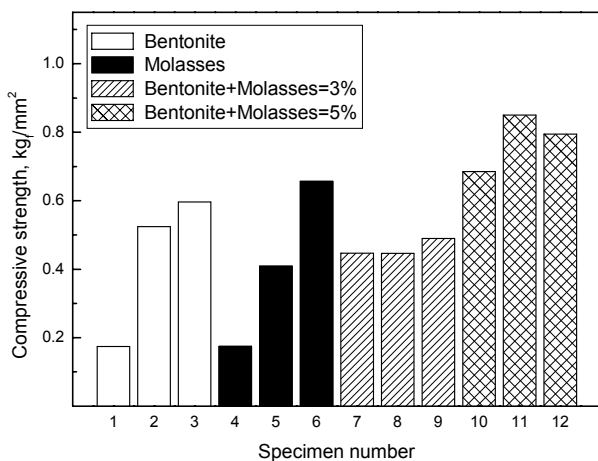


Fig. 3. Compressive strength of cold forming specimens for binder mixing ratio.

### 3. Experimental results and discussion

#### 3.1 Taguchi optimization technique with missing data

To obtain the sensitivity of the process parameters and the optimum condition in the manufacture of pig iron by using mill scale, we used the Taguchi method. The Taguchi method is one of the most efficient DOE methods. The signal-to-noise (S/N) ratio, a measure that simultaneously considers the average and variations of responses, is based on the analysis of variance (ANOVA) technique.

Taguchi's approach is a method for improving the quality of a product through minimizing the effect of variation without eliminating the causes [16, 17]. Reducing the variation may be the same as increasing the S/N ratio. The S/N ratio can be defined as nominal-the-best, smaller-the-better or larger-the-better according to the characteristics of the problem. Since the metallization and compressive strength of mill scale in this paper hold the larger-the-better characteristics, the S/N ratio is defined as

$$(S/N)_L = -10 \log \left( \frac{1}{n} \sum_{i=1}^n \frac{1}{y_i^2} \right) \quad (1)$$

where  $n$  is the number of all data points and  $y_i$  is the value of the  $i$ th data point. The optimum level and relative effect of various process parameters are calculated using the S/N ratio and the ANOVA analyses. The optimum condition can be realized by maximizing the S/N ratio. The test result is analyzed by the verification experiment for the optimum condition. For the process parameter optimization of mill scale, the experimental schedule was built using a Taguchi L18 orthogonal array.

Actually, there are many factors for the process optimization, for example: the fixed carbon, formed thickness, grain size, binder type, drying and sintering conditions and so on. However, having considered the time consumed and the limit of experiment condition, four factors are chosen as the process parameters:  $x_1$ : fixed carbon,  $x_2$ : formed thickness,  $x_3$ : grain size and  $x_4$ : heat treatment temperature. Table 3 shows the three levels for each process parameter. Table 4 shows the test result of L18, which includes a missing value. The missing values that did not yield the experimental data are shown on the No. 17.

In this paper, the experiment is very costly and requires an amount of time, and there may be a big response variation in the experiment because of the missing value. Although the Fisher-Yates method can be used to estimate the missing value, we used successive approximation, which is generally used [14, 18]. The relevant steps of successive approximation are explained as follows.

**[Step 1]** Take the 0<sup>th</sup> approximate value by substituting the average response value close to the missing value.

**[Step 2]** **Construct** an approximation model by using the factors of large effectiveness other than the missing value.

Table 3. Process parameters and their levels.

Process parameter	Description	Level 1	Level 2	Level 3
$x_1$	C content	20%	25%	30%
$x_2$	Thickness	10 mm	15 mm	20 mm
$x_3$	Granularity	1 mm	2 mm	3 mm
$x_4$	Heat treatment temperature	1100 °C	1200 °C	1300 °C

Table 4. Results of L18 array with observational data.

Exp.	Compressive Strength (kgf/mm <sup>2</sup> )	Total Fe (%)	Metallization (%)
1	12.9	92.6	80.25
2	12.5	92.79	92.01
3	14.6	92.5	91.13
4	10.3	84.82	87.21
5	11.7	90.03	84.25
6	8.6	74.94	61.67
7	15.3	91.35	95.47
8	15.2	91.85	96.11
9	15.4	91.77	96.85
10	14.5	92.30	95.13
11	14.7	92.01	95.52
12	12.4	88.75	85.8
13	13.7	86.27	88.52
14	12.0	87.02	88.34
15	10.4	81.3	64.85
16	12.6	88.87	92.14
[17]	10.3	78.2	93.02
18	10.9	73.35	88.27

[]: Missing value

Estimate the missing value by using the approximation model and take this value as the first approximate value. The approximation model uses the Chebyshev orthogonal polynomial  $P_n(x)$ , where the degree of the design variable  $x$  is  $n$  [19-22]. If these are expressed in the form of a second-order polynomial, and can be expressed as Eq. (2).

$$y = b_0 + b_1(x - \bar{x})^2 + b_2 \left[ (x - \bar{x})^2 - \frac{a^2 - 1}{12} h^2 \right] + b_3 \left[ (x - \bar{x})^3 - \frac{3a^2 - 7}{20} (x - \bar{x}) h^2 \right] + b_n p_n(x) + \dots \quad (2)$$

$$p_0(x) = 1, \quad n = 0$$

$$p_1(x) = x - \bar{x}, \quad n = 1$$

$$p_2(x) = (x - \bar{x})^2 - \frac{a^2 - 1}{12} h^2, \quad n = 2$$

$$p_n(x) = p_{n-1}(x)p_1(x) - \frac{(n-1)^2 \{a^2 - (n-1)^2\} h^2}{4\{(n-1)^2 - 1\}} p_{n-2}(x),$$

$$n = 3, 4, 5, \dots$$

Table 5. Analysis of variance for metallization.

Process parameter	Dimension	DOF	Variance	F-ratio	Effective ratio (%)
$x_1$	1	1	192.73	26.75	12.1
	2	1	63.85	8.86	4
$x_2$	1	1	46.17	6.41	2.9
	2	1	12.27	1.7	0.8
$x_3$	1	1	72.7	10.09	4.6
	2	1	43.25	6	2.7
$x_4$	1	1	936.4	129.94	59.1
	2	1	218.16	30.27	13.8
Error		7	64.86		
Total		17	1650.38		100

where  $\bar{x}$  is the average of the  $a$  levels of design variable and  $h$  is the interval between the design variable levels. Note that the degree  $n$  should be less than  $a$ . The maximum degree for each design variable is  $a - 1$ .  $b_0, b_i$  can be expressed as regression coefficients by Eq. (3).

$$b_0 = T / lm = \bar{y}$$

$$b_i = \sum_{k=1}^a p_i(x_k) y_k / \sum_{k=1}^a p_i^2(x_k), \quad k = 1, 2, \dots, a \quad (3)$$

where  $x_k$  means each level of  $x$  and  $y_k$  means the average of the experiment data at each level. The use of the orthogonal polynomial is advantageous in the ANOVA. In ANOVA of orthogonal polynomials, each effect is estimated independently. The effects can take a linear, quadratic, or higher order ( $n - 1$ ) functional form. Also, since every term in an orthogonal polynomial is independent of each other, the estimates of the regression coefficient can be calculated successively from low order to higher order. This enables the derivation of an efficient approximation model, since the base can be normalized even when the coefficient of the higher order is not known or the coefficient difference becomes large [19, 22].

[Step 3] Repeat Step 2 until the estimate of the missing value converges.

The average of 17 metallization data is 87.34%. Table 5 shows the result of ANOVA with this substituted value. If the approximation model is expressed as a linear quadratic polynomial, Eq. (4) can be obtained.

$$y = -1683.1 + 97.91x_1 - 18.517x_1^2 - 4.514x_2 + 0.13927x_2^2 + 2.691x_3 - 1.0071x_3^2 + 2.6519x_4 - 0.001044x_4^2 \quad (4)$$

By substituting  $x_1$  (level 3),  $x_2$  (level 1),  $x_3$  (level 2) and  $x_4$  (level 3) to Eq. (4) for the experimental conditions of No. 17

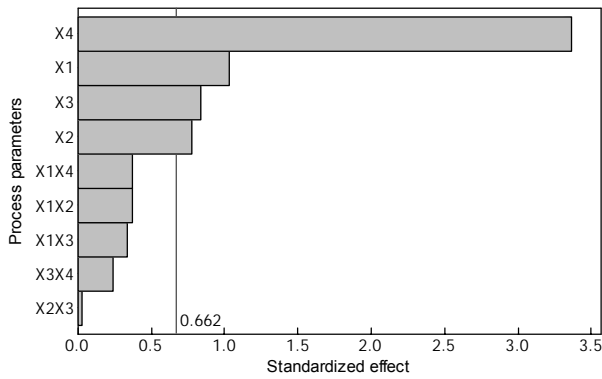


Fig. 4. Pareto chart of the standardized effects for metallization.

in the Table 4, we obtained 97.23%. Therefore, the 1<sup>st</sup> approximate value of the missing value is 97.23%. In the same way by performing the ANOVA using this value, the approximation model for Eq. (5) can be obtained.

$$y = -1586.1 + 95.85x_1 - 17.692x_1^2 - 2.453x_2 + 0.07333x_2^2 + 3.103x_3 - 1.1101x_3^2 + 2.4582x_4 - 0.000962x_4^2 \quad (5)$$

Then, the 2<sup>nd</sup> approximate value of 96.67% from No. 17 is obtained by using Eq. (5). After this process was repeated five times, the approximate value of 93.06% was obtained. Since this value does not deviate from the fourth approximate value of 93.02% and can be smoothly converged, we stopped the experiment at this point. Therefore, we determined the surrogate value of No. 17 to be 93.02%.

Fig. 4 shows the sensitivity of the process parameters in metallization using a Pareto chart. The absolute values of the effects in this chart are shown by reference lines based on Lenth's method [23, 24]. Any effect that extends past this reference line is potentially important.

From the result of the Pareto chart, the following two pieces of information can be obtained. The first information is the heat treatment conditions of the dominant process variables that influence the metallization. The second information is the small influence of reciprocal action. Generally, to improve the strength and reduction quality, grain size and fixed carbon quantity can be adjusted in the mixing process of mill scale. Since the porosity of the charging layer increases and the specific surface area increases as the grain size decreases, the speed of the reducing action tends to increase. However, the result of Fig. 4 shows that the adjustment of the heat treatment temperature is effective in maximizing the strength.

Figs. 5, 6 and 7 show the main effect of the S/N ratio on the four process parameters. If the magnitude and the importance of the process parameter sensitivity approaches the fixed carbon quantity ( $x_1$ ) and the heat treatment temperature ( $x_4$ ) increases up to level 3, the characteristics of each response value improve as the forming thickness ( $x_2$ ) and grain size ( $x_3$ ) de-

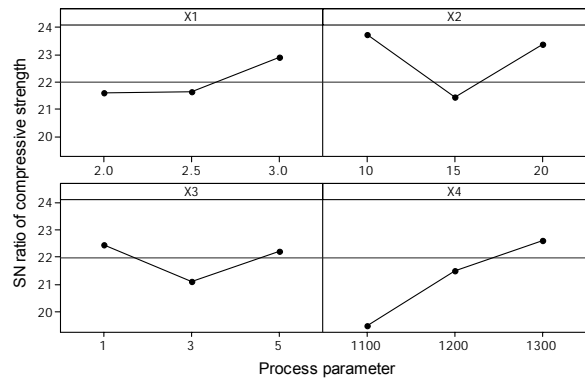


Fig. 5. Effect of the process parameter on compressive strength.

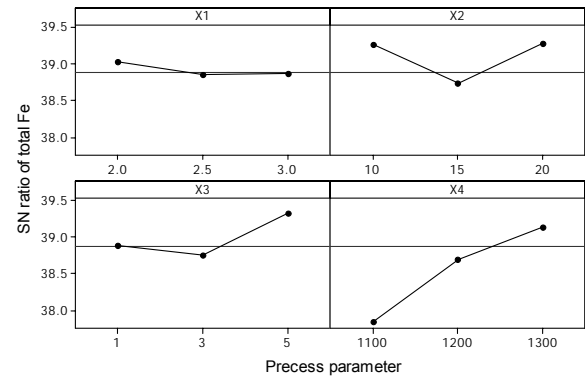


Fig. 6. Effect of the process parameter on total Fe.

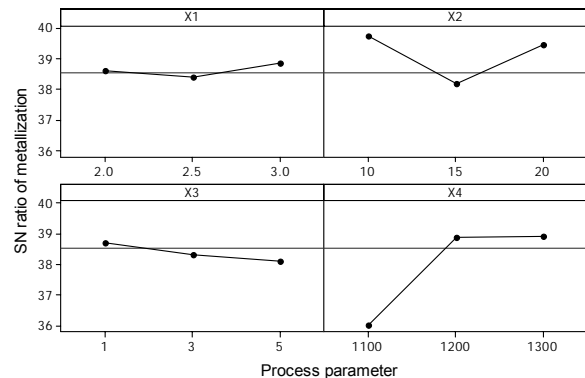


Fig. 7. Effect of the process parameter on metallization.

crease to level 1. Accordingly, the metallization efficiency of pig iron collection increases as the compressive strength increases.

Table 6 calculates the effect of metallization on every level of a process parameter according to the S/N ratio. As for the process parameters that have the most influence on the metallization from the mixing process of mill scale up to the time when the pig iron is obtained from the reducing reaction, the heat treatment temperature ( $x_4$ ) and forming thickness ( $x_2$ ) had the greatest influence in this process. At this time, the average S/N ratio of metallization was 38.538 and the response was

Table 6. Process parameters affecting the metallization.

Process parameter	Effect			Deviation	Percentage contribution (%)	Rank
	Level 1	Level 2	Level 3			
$x_1$	38.59	38.39	38.85	0.46	8.3	4
$x_2$	39.72	38.18	39.45	1.54	27.8	2
$x_3$	38.71	38.32	38.09	0.62	11.2	3
$x_4$	36.02	38.89	38.94	2.92	52.7	1
Total				5.54	100	

Table 7. Comparison between prediction and optimal results.

Result	Optimal value	Predicted value	Error (%)
SN ratio	38.538	39.722	3.07
Mean	85.62	96.85	13.12

85.62%. The optimum conditions of the process parameter can be achieved by the fixed carbon quantity of 30% (level 3), forming thickness of 10 mm (level 1), grain size of 1 mm (level 1), and heat treatment temperature of 1300 °C (level 3).

### 3.2 Optimum conditions and verification experiment

We conducted the verification experiment, determined the optimum levels for the process parameters, and predicted the performance under these levels. If the observed and the projected improvements match, we adopt the suggested optimum conditions. If the predicted response under the optimum conditions does not match the observed response, then it implies that the interactions, missing value, and experiment error are important. Accordingly, we can evaluate how much the treatment of a missing value influences the analysis result. The predicted value of S/N ratio expressed as  $\mu$  can be expressed as follows:

$$\mu = \bar{T} + (x_i^{\text{Level}} - \bar{T}) \quad (6)$$

where  $\bar{T}$  is the total average analytical S/N ratio and  $x_i^{\text{Level}} - \bar{T}$  represents the improvement of each parameter at the optimum level. The additivity [3, 25] of variable effects is good when an appropriate S/N ratio is used. We can determine whether the quality characteristics selected through the verification experiment and S/N ratio hold the additivity characteristic.

Table 7 shows the comparison of the values obtained under the optimum conditions with the predicted values for the metallization efficiency obtained in Table 6. Since the result shows a relatively good match, we can judge that the process holds the additivity characteristic. By performing the verification experiment with the conditions obtained, we analyzed the actual experiment result.

### 3.3 Validation of experimental results

For the optimum condition and candidate condition, Fig. 8

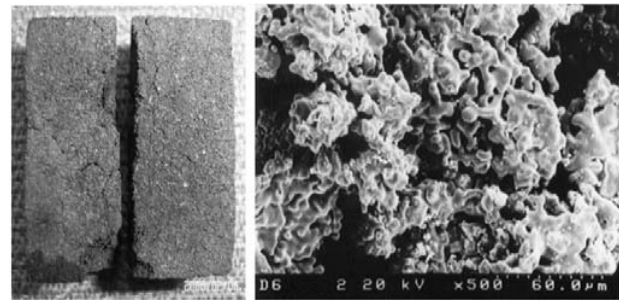
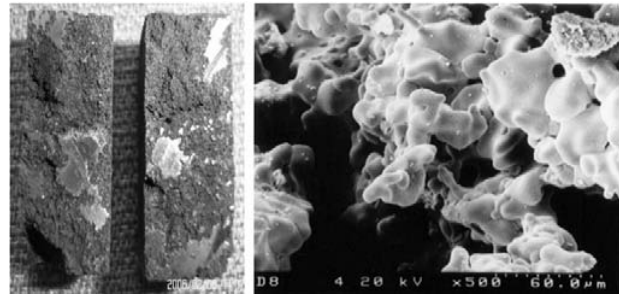
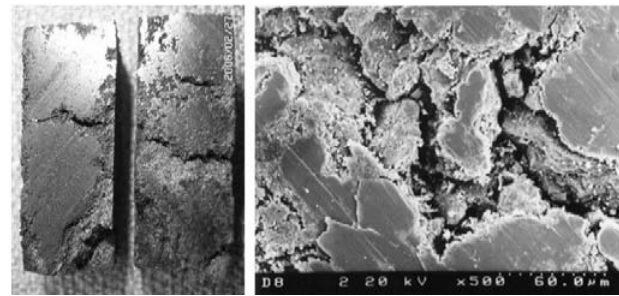
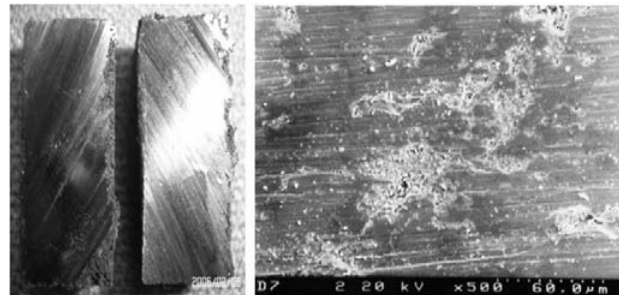
(a)  $x_1$  (20%),  $x_2$  (15 mm),  $x_3$  (3 mm),  $x_4$  (1300°C)(b)  $x_1$  (25%),  $x_2$  (15 mm),  $x_3$  (3 mm),  $x_4$  (1300°C)(c)  $x_1$  (25%),  $x_2$  (10 mm),  $x_3$  (1 mm),  $x_4$  (1300°C)(d)  $x_1$  (30%),  $x_2$  (10 mm),  $x_3$  (1 mm),  $x_4$  (1300°C)

Fig. 8. SEM photographs of direct reduced iron process for test results.

shows the photo of a section of reduction iron, which was obtained by using a scanning electronic microscope. The reducing of most mill scale is generated in the part where the single crystal appears dark, and the iron layer is made in the part that appears partially bright. The iron layer appears at the reducing temperature of 1200 °C or higher.

Fig. 8(d) shows a case where a verification experiment was performed under the optimum condition. The chemical prop-

erties of the direct reducing iron were obtained from the experiment, and the metallization efficiency was 92%. Compared with the predicted optimum result (96.85%), the verified result under predicted optimum conditions has an error of 5.3%, which means there remains a small quantity of unreduced iron oxide. The part where the iron particles appear dark on the photo is the unreduced part. Therefore, it is necessary to consider an additional process parameter related to the speed of the reducing response and to evaluate the metallic characteristics of reduced iron.

#### 4. Conclusions

In this paper, optimization of process parameters with a missing value was presented to improve the productivity of the mill scale recycling process so that high-quality products can be produced at low cost. The result can be summarized as follows.

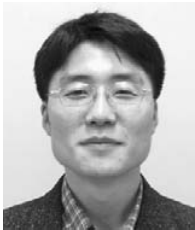
- (1) To collect the pig iron effectively, the mill scale recycling process is applied through a two-step process, where in the first step, the strength analysis of the forming agent is carried out for the mixing process of mill scale, and in the second step, the optimum condition of the process parameters that maximizes the metallization efficiency is determined.
- (2) Every step of the experiment of this recycling process is very costly and time-consuming. In this paper, it is not possible to predict accurate statistical values. Therefore, methods including successive approximations and S/N ratio are utilized to optimize the process parameters. The selected optimum condition matched the result of the actual verification experiment for the error ratio (within 5.3%).

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