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PRELIMINARY REDUCTION OF CHROMIUM ORE USING SI SLUDGE GENERATED IN SILICON WAFER MANUFACTURING PROCESS

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Abstract

In order to promote the recycling of by-product from Si wafer manufacturing process and to develop environment-friend and low cost process for ferrochrome alloy production, a basic study was performed on the preliminary reduction reaction between chromium ore and the Si sludge, comprised of SiC and Si particles, which is recovered from the Si wafer manufacturing process for the semiconductor and solar cell industries. Pellets were first made by mixing chromium ore, Si sludge, and some binders in the designed mixing ratios and were then treated at different temperatures in the 1116 °C–1388 °C range in an ambient atmosphere. Cordierite and SiO₂ were confirmed to be formed in the products after the reduction. Additionally, metal particles were observed in the product with Fe, Cr, and Si components. It is found that temperatures above 1300 °C are necessary for the reduction of the chromium ore by the Si sludge. The reduction ratio for Fe was evaluated quantitatively for our experimental conditions, and the proper mixing ratio was suggested for the pre-reduction of the chromium ore by the Si sludge. This study provides basic information for the production of ferrochrome alloys on the pre-reduction of chromium ore using Si sludge.

Keywords: Chromium ore; Silicon sludge; Silicon carbide; Silicon; Pre-reduction; Ferrochrome.

1. Introduction

In recent years, the use of silicon wafers has been on an increase owing to the development of computer and electronics industries [1]. Furthermore, global warming caused by greenhouse gases is intensifying, and despite the low price of crude oil, technological development for the generation of renewable energy such as solar power and wind power has been continuing worldwide [2]. Different types of solar cells are available, in terms of power generation efficiency and economy, and silicon-based solar cells using silicon wafers account for more than 80% of solar cells [3].

To manufacture the wafers that can be used in the semiconductor and photovoltaic industries, a highpurity silicon ingot must be sliced, and approximately 30% to 50% of the ingot is lost to the sludge in this process. The sludge is divided into two types based on the cutting method. The first method is a slurry method in which a certain ratio of SiC powder serves as an abrasive. A coolant oil (ethylene glycol) serving as a lubricant is continuously supplied to the sawing wire during the cutting process. Thus, the sludge consists of SiC, Si, iron powder, and cooling oil. In the second method a silicon ingot is cut with a sawing wire with electrodeposited diamond particles on the surface. Oil or cooling water is continuously supplied to the sawing wire to remove the heat generated during the cutting and to discharge the kerf generated by the cutting. Hence, the sludge generated in this process does not contain SiC, but contains only oil, water, Si, and iron particles.

The total amount of polycrystalline silicon used in 2014 was reported to be 280,000 tons, and the annual amount of recoverable Si powder is expected to be 100,000 tons. This amount is expected to grow by 25% every year in the future [4], therefore, one may expect an increase in the amount of the Si sludge discharged worldwide. Although attempts have been made to recover Si from the Si sludge by centrifugation and specific gravity separation, proper recycling methods, other than the use of landfills, have not been developed successfully [5-8].

On the other hand, chromium is added to the stainless steel in a large amount. The chromium,

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which is one of important elements in steel products, is used for increasing the hardness, strength, as well as corrosion resistance and abrasion resistance of the steel. At low carbon concentration, the corrosion resistance is increased when the chromium content is more than 12 wt%, and the abrasion resistance is improved at the high carbon concentration. Additionally, the addition of chromium is also known to improve the high-temperature strength [9, 10]. In the steel making process, the chromium component is added via a ferro-chrome alloy (Fe-Cr alloy) [11].

Every year Korea imports 500,000 tons of ferrochrome that is mainly used for stainless steel production. 95% of the global chromium ore mining production originates in South Africa, Zimbabwe and Kazakhstan, and ferrochrome is produced predominantly in South Africa and partially in India and China. These ferrochromes are currently produced by reducing the chromium ore using coal as a reducing agent [12-15]. Unfortunately large amount of the greenhouse carbon dioxide gas is generated in this process, thereby causing environmental problems. To achieve a higher energy efficiency, the process for producing ferrochrome is generally composed of two steps; a step of chrome ore reduction partially in the preliminary reduction, followed by a melting and reducing treatment to produce the final ferrochromium alloy.

In this study, basic research was conducted on the alternative process for the production of ferrochrome alloy utilizing Si sludge which is not recycled effectively now. To get some basic information for the development of environmental friendly and cost effective process for the ferrochrome alloy, preliminary reduction treatment of chromium ore was studied carefully with the Si sludge generated in the semiconductor industry and solar cell industry which is not recycled effectively.

2. Theoretical background

Thermodynamic calculations were performed to investigate the feasibility of the chromium ore reduction using the Si sludge generated in the silicon wafer manufacturing process. Si sludge consists mainly of Si and SiC particles, and it is expected that these components will reduce chromium ore via a silicothermic reaction. Chromite (FeO·Cr₂O₃) is the main phase of the chromium ore, which will be described in next section. In this study, thermodynamic calculations were performed for the expected reaction using the FactSage software [16]. The reactions are shown below, and Figure 1 represents the value of the Gibbs free energy change for each reaction calculated using FactSage.

$$\frac{1}{2}FeCr_2O_4 + Si = \frac{1}{2}Fe + Cr + SiO_2$$
(1)

$$\frac{3}{4}FeCr_2O_4 + SiC = \frac{3}{4}Fe + \frac{3}{2}Cr + CO + SiO_2$$
(2)

The results of the thermodynamic calculations in Figure 1 show that all of the above reactions exhibit negative free energy values, indicating that chromite can be reduced by Si and SiC. Therefore, it is expected that chromium ore can be reduced by Si sludge. It can also be expected that chromite will be reduced using Si, rather than SiC below 1550 °C and that it can be reduced using SiC instead Si above 1550 °C.



Figure 1. The free energy change of reactions between Si/SiC and Chromite, calculated by FactSage.

3. Experimental 3.1 Materials

In this study, sludge recovered from P company (Korea) was used as the Si sludge, where Figure 2 shows its XRD pattern of the Si sludge. As shown in the figure, the Si sludge consists mainly of Si and SiC. From the results of chemical analysis by ICP and C/S analysis, it was deduced that sludge was composed of 38.2 wt% Si and 56.7 wt% SiC.



Figure 3 shows the results of the particle size distribution analysis of the Si sludge. The average particle size was $1.573 \mu m$ and the mean volume particle size was $4.660 \mu m$. Other studies reported that Si particles in the sludge were small, whereas SiC particles were large [5-7, 17]. In Figure 3, it is assumed that Si particles are mainly distributed in the small size region and SiC particles are found in the large size region.



Figure 3. Particle distribution of Si sludge.

The XRD pattern for the chromium ore used in this study are shown in Figure 4. It can be seen from the XRD pattern that the chromium ore mainly consists of the chromite phase with the formula Cr_2O_3 ·FeO [18]. Table 1 shows the analysis results for the chromium ore. It consists of about 45 wt% Cr oxide and 26 wt% iron oxide, respectively, as well as of other components, such as Al₂O₃, MgO, TiO₂, and

Table 1. Chemical composition of chromium-ore (wt%).



Figure 4. XRD pattern of chromium-ore.

 SiO_2 . The Cr/Fe metal component ratio of the chromium ore was found to be 1.59.

The XRD pattern in Figure 4 shows the chromite phase, but V. E. Roshchin et al. showed that chromium ore might correspond to the $(Mg^{2+}, Fe^{2+})[Fe^{3+}, Al^{3+}, Cr^{3+}]O_4$, the complex oxide spinel [19]. Figure 5 presents the SEM image and EDS results for the chromium ore used in the present study. The size of chromium ore particle was found to be $300 \sim 500 \,\mu\text{m}$, and magnesium and aluminum were confirmed in the EDS spectra.

Additionally, bentonite, starch, and slaked lime were used as a binder and the basicity adjustment. The slaked lime was produced by heating limestone at 900 °C for 2 hours or more, where it was added in small quantities acting as the binder, and it was also used for adjusting the slag component in the main reduction process after the preliminary reduction.

	Cr ₂ O ₃	FeO	SiO ₂	Al ₂ O ₃	MnO	CaO	MgO	TiO ₂	Na ₂ O
Chromium-ore	45.19	25.65	1.41	16.81	0.28	0.048	8.92	2.17	0.046



Figure 5. SEM image and EDS spectra of chromium-ore.



3.2 Experimental apparatus and procedure

The experimental setup used for the study of the chromium ore pre-reduction behavior is shown in Figure 6. Both ends of the reaction tube $(60\Phi \times 52\Phi \times 1000 \text{ mm}, \text{HB}, \text{Nikkato, Japan})$ with a mullite material were sealed with a silicon rubber stopper, and the experiment was carried out in a high purity Ar gas atmosphere. An alumina boat $(13 \times 11 \times 70 \text{ mm}, \text{Samhwa Ceramic, Korea})$ was used as a crucible and the experimental temperature was controlled by the PID controller between 1116 °C and 1388 °C.

The raw materials were weighed to a total weight of 10 g or 15 g according to the blending ratio, and then treated with a ball mill for 10 to 15 minutes to uniformly mix the powder. During the mixing milling process, distilled water was added in an amount of 5% of the total weight, and the milling was carried out using an alumina ball. Thereafter, a cylinder-shaped green compact with the diameter of about 8 mm and a height of about 10 mm was produced by using a mold. The weight of one pellet was approximately 1.0-1.5 g. Through preliminary experiments, the press pressure in making the compact was determined to be 2,000 psi, and all pellets were produced at the pressure of 2,000 psi. Three to four pellets were prepared for each condition and used in the experiments. The pellets were dried for more than 12 hours in an oven at 80 °C and used in the experiment.

The compacts were placed in an alumina boat and loaded into the center of the furnace. Argon gas was purged into the inside of the reactor for a long period of time and the inside of the reactor was replaced with argon gas. After the furnace was heated to the target temperature, the temperature was lowered after holding for 1 hr. During the experiment, argon gas was continuously flowed into the reaction tube at the flow rate of 300 sccm.

After the pre-reduction experiment, the compacts were recovered, and the change in the weight before

and after the reduction was measured. The compressive strength of the specimen was measured after the pre-reduction experiment. Metal particles were formed in some experiments, and they were separated from powder in those cases. XRD (D/max 2500, Rigaku Japan, 40 kV 200 mA CuK α) analysis was performed on the powder samples before and after the experiment, and the chemical components were analyzed by XRF (Simultix 12, Rigaku Japan), ICP (Spectro ARCOS EOP, Spectro Germany) and chemical analysis. The morphologies of metal particles were examined using field emission scanning electron microscopy (FE-SEM, JEM-7610F, JEOL, Japan) and energy dispersive X-ray spectrometry (EDS) at 10.0 kV.

4. Results and Discussion 4.1 Composition of mixed samples

Compacts were prepared by blending raw materials according to the mixing ratios specified in Table 2, and bentonite, starch, and slaked lime acted as the binder and component adjustment. Table 3 shows the predicted results of the pellets for these mixing ratios. In this calculation, it is assumed that the Si/SiC composition ratio was maintained in the Si sludge, and only the weight ratio of the entire sample was changed. Compositions of chromium ore and bentonite were used according to the values analyzed in this study, and those of starch and lime were calculated using the theoretical values specified in the

Table 2. Mixing ratio of starting materials (wt%).

	Si sludge	Cr-ore	bentonite	starch	slaked lime
А	40	50	5	2	3
В	35	55	5	2	3
С	30	60	5	2	3
D	25	65	5	2	3



Figure 6. Schematic drawing of experimental apparatus.



	1	1	5 1	5	,	<i>,</i>				
	С	Cr ₂ O ₃	Fe ₂ O ₃	SiO ₂	CaO	Al ₂ O ₃	MgO	TiO ₂	Si	SiC
Α	1.06	22.62	14.28	4.28	2.45	9.51	4.6	1.08	16.07	23.86
В	1.14	24.82	15.85	4.34	2.45	10.33	5.04	1.19	14.03	20.82
C	1.22	27.01	17.23	4.4	2.44	11.14	5.47	1.29	12	17.81
D	1.29	29.19	18.6	4.46	2.43	11.95	5.9	1.4	9.97	14.8

Table 3. Expected composition of compacts before treatment (wt%).

literature. The metal Cr/Fe ratio in the pellets was 1.53 in all of A, B, C, and D, and they did not change much with the ratio in the chromium ore.

4.2 FactSage calculation

The calculation using FactSage was performed for the composition in Table 3 in order to get some information on the phases in equilibrium as a reference. This calculation was done for the equilibrium phase and it was performed to obtain reference information even though the results in this experiment were in non-equilibrated condition. Figure 7 shows the calculated results for the D composition. It was expected that various phases would be formed depending on the temperature, and CO was expected to be generated as a vapor phase, but it was not included in Figure 6. The silica, cordierite, anorthite, and pyroxene phases were expected to be formed as solid phases at high temperature, and the liquid phase was expected to be generated from around 1225 °C.



Figure 7. FactSage simulation results on Mixing D in different temperatures.

4.3 Products after reduction treatment

In this study, compacts were reduced at 1116 °C to 1388 °C in the argon atmosphere. The reduced compacts maintained their shape, and the pellets and the alumina boat did not react, so the pellets were easily recovered in most of the cases. The weight of the pellet before and after the treatment was measured, and it was found that the weight decreased after the reduction. A weight reduction of 5%-10%

occurred after the pre-reduction treatment, and the weight decrease for the high temperature treatment was larger than that of the low temperature treatment. This was due to the CO gas generated as a result of the reduction reaction, and the reaction proceeded more actively at high temperature.

Figure 8 shows the results of compressive strength measurement of the compacts recovered after the prereduction treatment at 1300 °C and 1388 °C. The strength increased with the increase of the chromium ore mixing content. For higher temperature runs, the compressive strength was lower than that at low temperature. The compressive strength values for mixing ratios C and D were lower than those of A and B, especially at 1388 °C, and the pellets were strongly deformed, and many pores were observed after the pre-reduction treatment. Therefore, the compressive strength could not be measured accurately, and many shining particles were observed in the recovered powder. Based on these observations, the reduction reaction at 1388 °C appears to be active, resulting in a large amount of CO gas evolution, a large number of pores and a reduced strength.



Figure 8. Comparison of compressive strength of compacts after pre-reduction treatment at different temperatures.

Figure 9 shows the XRD patterns of the samples after the pre-reduction treatment of the pellets prepared with mixing ratios A and B. Figure 10 shows the similar XRD patterns after the reduction treatment of the pellets for mixing ratios C and D together with the cordierite reference pattern [20]. In this study, the pre-reduction process was carried out at 1116 °C as well, but the reaction did not progress significantly,



resulting in only small changes in the XRD pattern; therefore, these XRD patterns are similar and not presented here. The presence of metal particles formed during the preparation of the sample for the XRD measurement made it difficult to perform the XRD measurements. Therefore, the metal particles were removed by sieving and only the powder was used for XRD measurement. Thus, the diffraction peaks for the metal were not included in the XRD patterns. Metal particles were analyzed using FE-SEM as described later.



Figure 9. XRD patterns of Mixing A and B reacted at 1177°C, 1303°C and 1388°C.

In the XRD pattern obtained prior to the prereduction treatment, the peaks of SiC and chromite overlapped for the peaks at 36°, indicating very high strength. We note that in Figure 9, there was no peak at around 10° for the sample at 1177 °C, but this peak was observed for the samples at 1300 °C and 1388 °C. It can, therefore, be concluded that this was the peak of the phase produced by the pre-reduction reaction. The peaks at 10°, 22°, 26°, 28°, and 29° were in accordance with the peaks of the cordierite phase (Mg₂Al₄Si₅O₁₈) shown in Figure 10 (The peak at 22° overlaps with SiO₂). The other peaks also showed good agreement with the peaks of the cordierite

phase. Figure 10 shows the XRD patterns for the mixing ratios C and D at 1300 °C and 1388 °C, when the reduction reaction proceeded relatively well. The red closed marks indicate typical peaks of the cordierite phase, and the blue closed marks indicate the SiO₂ peaks. The peak at 36° for SiC and chromite was the highest for the sample at low temperature, and the peak around 10° for cordierite was highest at 1388 °C. In the patterns for 1300 °C and 1388 °C, the intensity of the peak at 36° decreased from A to D. It is considered that SiC and chromium ore reacted with each other, resulting in the decrease of the peak at 36°. In addition, the intensity for the SiO₂ peak at 22° increased with increasing temperature, most likely due to the formation of SiO_2 as the reduction reaction progressed.



Figure 10. XRD patterns for Composition C and D reacted at different temperatures and reference pattern of Cordierite.

50

2 theta (deg.)

60

10

20

30



80

90

70

4.4 FE-SEM analysis

In the present study, the compact samples mixed at the mixing ratios A-D were reduced in the 1116-1388 °C temperature range. The reduction reaction did not progress much at 1116 °C and 1177 °C, while at 1300 °C and 1388 °C, the reduction progressed actively and shining metal particles were observed. Figure 10 shows a photograph of the metal particles recovered from the sample with the mixing ratio D at 1388 °C, in which some particles were found to be a few mm in size. In other words, it can be confirmed that the chromium ore was reduced by the Si sludge, generating the metal. Kapure *et al.* [21] reported metal and slag being separated as a result of the reduction experiments at 1400 °C in the direct reduction of lowgrade chromium powders using coal.



Figure 11. Metal particles recovered after the prereduction treatment (Mixing D, T=1388°C).

Figure 12 shows the results of the FE-SEM analysis of the sample recovered after the prereduction treatment, and Table 4 gives the results of EDS analysis of the samples with mixing ratios A and C reduced at 1388 °C. EDS 1 was analyzed for the entire range of the SEM images, including the slag and metal parts. It was found that metal part contains only C, Fe, Cr and Si components, while slag part included Mg,A1 and O as well. The white (Point 3) and grey (Point 2) parts in the metal particle in SEM image for sample C can also be seen. Point 2 in sample C showed low Cr content and Point 3 showed high Cr content. Some deviation was found in the components in the metal part. The EDS results showed that the composition of the metal part was 15–80 wt% Fe, 20–70 wt% Cr, 10 to 20 wt% Si,and 3–15 wt% C. These results confirm that the chromium ore is reduced by the Si and SiC components, producing the metal particles.

		С	Fe	Cr	Si	Mg	Al	0	Ca
	1	15.08	13.71	20.02	13.92	4.06	4.73	28.49	23.57
	2	3.67	59.81	24.87	11.65	-	-	-	
A	3	5.98	51.04	33.72	9.26	-	-	-	
	4	6.87	34.86	46.4	9.37	-	-	-	-
	1	12.33	13.38	26.69	12.73	4.32	4.06	24.84	1.76
C	2	13.74	15.38	70.88	-	-	-	-	-
	3	4.17	82.33	-	13.51	-	-	-	-

Table 4. EDS results for Figure 12 (wt%).

4.5 Reduction ratio

In this study, it is necessary to evaluate how much chromium oxide is reduced to chromium metal in order to quantitatively evaluate the extent of the chromium ore reduction by the pre-reduction treatment in compacts with various mixing ratios. While a method based on the weight of oxygen in the sample [22, 23], that requires accurate measurements of the weight change of the sample, is sometimes used for evaluation of the reduction ratio, the analytical results were used instead in this study. However, in the case of chromium, there is no way to distinguish precisely the chromium present as an oxide from the chromium present as a metal by chemical analysis or XRF. Therefore, similarly as the work of Kapure et al. [21] and Pan et al. [24], in this study the reduction of chromium is indirectly evaluated by defining the reduction ratio based on the iron content, as given below



Figure 12. SEM images for recovered samples (Mixing A and C, T=1388°C).



Reduction Ratio (%) = $\frac{(M.Fe)}{(T.Fe)} \times 100$ (7)

Here, (M. Fe) is the metallic iron content in the sample after the pre-reduction treatment, and (T. Fe) is the total iron content in the compacts prior to the reduction treatment. These values were obtained by chemical analysis.

Figure 13 shows the reduction ratio of the pellets with the respective mixing ratios for different prereduction temperatures. The fraction of chromium ore increases and the Si sludge fraction decreases as the mixing ratio changes from A to D. The reduction ratio of A was the lowest, and those of B, C, and D increased. This implies that the use of a large Si sludge amount does not increase the reduction ratio, instead an appropriate amount should be used. Si sludge contains SiC more than Si, and, as observed from the thermodynamic calculation results shown in Figure 1, the reduction by Si occurs more easily than that by SiC. Therefore, it is concluded that a large amount of SiC does not significantly contribute to the chromium ore reduction. The effect of the reduction temperature on the reduction rate is quite significant, as reduction rate was higher at 1303 °C than that at 1177 °C. In this study, the reduction rate at 1388 °C is expected to be higher than that at 1303 °C. However, in the sample at 1388 °C, most of the metal particles were separated from the slag part and thereby analyzed to a very low value. Because of this, the reduction rate for 1388 °C was not presented in Figure 13. Pan et al. [24] reported that the reduction rate varies from 20.8% to 33.8% depending on the temperature in the solid phase chromium ore reduction by coal. They reported the reduction rate of 32.6% for the reduction of 90 minutes at 1300 °C, which is slightly higher than the reduction rate of this study. Based on the reduction rate results obtained in this study and the analysis of the experimental results, it was deduced that the temperature of 1300 °C or higher, and the suitable mixing ratios of C or D are the optimum mixing conditions.



Figure 13. Reduction ratio for different experimental conditions.

This study is expected to be applied for the basic research on the preliminary reduction process using low-cost Si sludge in the manufacturing of the ferrochrome (Fe-Cr) alloy, which is an important raw material in the steel industry. This process is expected to be environmentally advantageous and economical due to the efficient recycling of Si sludge, and the reduction of a large amount of global warming gas generating in the production of ferrochrome alloy by coal in the conventional process.

5. Conclusion

The preliminary reduction reaction in compacts made by mixing Si sludge and chromium ore was studied in a basic study using Si sludge composed of SiC and Si fine powders generated in the Si wafer manufacturing process for semiconductors and solar cells.

Si sludge composed of SiC and Si was mixed with chromium ore to produce compacts. It was confirmed that chromium ore was reduced to produce Fe-Cr-Si metal by the pre-reduction treatment of the compacts at various temperatures. Si sludge, chromium ore and binder were used to produce compacts, and the optimum condition for producing the compacts were derived. Cordierite (Mg₂Al₄Si₅O₁₈) phase was mainly formed by the reaction of Si sludge and chromium ore, and SiO₂ was generated as well. Based on the results of visual inspection, XRD and SEM analysis for the various mixing ratios, it is concluded that the optimum mixing ratio by weight is Si sludge 25%-30%, chromium ore 60%-65%, bentonite 5%, starch 2%, and slaked lime 3%. Temperature has a strong effect on the preliminary reduction of the chromium ore, and must be higher than 1300 °C. In the experiments at the temperatures above 1300 °C, shining particles with metallic appearance were observed in the pre-reduced products. Metal particles were formed by the pre-reduction reaction in the experiments at 1388 °C. The reduction ratio of Fe was quantitatively evaluated as an alternative indicator for the reduction ratio of chromium.

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PRELIMINARNA REDUKCIJA RUDE HROMA IZ SILICIJUMSKE ŠLJAKE NASTALE TOKOM PROCESA PROIZVODNJE SILICIJUMSKE PLOČICE

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Apstrakt

Da bi se promovisala reciklaža nusprodukta koji nastaje tokom procesa prizvodnje silicijumske pločice i da bi se razvio ekološki prihvatljiv i ekonomski isplativ proces proizvodnje legure hroma sa gvožđem, sprovedeno je ispitivanje reakcije preliminarne redukcije između rude hroma i Si šljake koja se sastoji od SiC i Si čestica, dobijene tokom procesa proizvodnje silicijumskih pločica za poluprovodnike i solarne ćelije. Korišćeni pelet se sastoja od rude hroma i Si šljake koja je sadržala veziva pomešana u određenoj razmeri, a koji je zatim tretiran na temperaturama u rasponu od 1116°C -1388°C. Utvrđeno je da se nakon redukcije u produktu javljaju i korderit i SiO₂. Pored toga, u produktu se mogu naći i čestice metala Fe, Cr i Si. Ustanovljeno je i da je temperatura iznad 1300°C potrebna da bi došlo do redukcije hroma Si šljakem. Redukciona razmera Fe, koja je bila potrebna za eksperiment, utvrđena je kvantitativno, a zatim je određen odgovarajući odnos potreban za pre-redukciju hroma iz Si šljake. Ovaj rad pruža osnovne informacije za proizvodnju fero-hrom legure pre-redukcijom rude hroma Si šljakom.

Ključne reči: Ruda hroma; Silicijumska šljaka; Silicijum karbid; Silicijum; Pre-redukcija; Fero-hrom.

