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Condition for Alkali Molten Decomposition of Zircon

Ri Song Ho, Pak Se Ok and *Chae Yong Suk*

Abstract We had an experiment to find the condition for alkali molten decomposition of zircon concentrate. The most important factors of alkali molten decomposition of zircon concentrate are weight ratio of zircon concentrate and alkali, reaction time, molten temperature and grain size of concentrate, and so on. From the experiment result the optimum condition of the alkali molten decomposition process is follows: weight ratio of zircon and sodium hydrate is 1 ∶ 1.2, temperature of alkali molten decomposition is 700℃, reaction time is 1.5h and the grain size of concentrate is 70μm. The yield of decomposition in optimum condition is more than 98%.

Key words zircon, alkali molten decomposition, optimum condition

Introduction

The great leader Comrade **Kim Il Sung** said as follows.

"**We should further strengthen scientific research and rapidly develop science and technology so that any scientific and technical problems in economic construction can be solved as soon as they arise and thus successfully make the national economy scientific and fully ensure that it becomes Juche-orientated and modern.**"("**KIM IL SUNG** WORKS" Vol. 35 P. 312)

Zirconium and its compounds are important essential materials for the national economy and national defense industry and the demand of it increases day by day. Especially many complex oxides prepared from ZrO_2 are applied more widely in the fields of machine, electron and energy, and so on as the structure and functional ceramic materials[5, 7－9, 12]. The third generation high-temperature solid oxidant fuel cell(SOFC) becomes important development direction of electric energy, in which ZrO_2 is used as solid electrolyte material [10]. Thereby it is very significant in extension of the application field of zirconium to complete the synthesizing process of ZrO_2 [6, 11].

Zirconium oxychloride is raw material of difficult fuctional zirconium compounds and alkali molten decomposition process of zircon is the important step in preparing zirconium oxychloride [13, 14].

Not a little research paper [1－4] associated to alkali molten decomposition of zircon are reported, but according to the kinds of concentrate the reaction indices are quite different.

We had an experiment to establish the condition of alkali molten decomposition of zircon. The most important factors of alkali molten decomposition of zircon are weight ratio of zircon and alkali, reaction time, molten temperature and the grain size of concentrate, and so on.

1. Experiment Method

The purpose of alkali molten decomposition is to separate Si and Zr from zircon concentrate. By alkali molten decomposition of zircon concentrate and next water leaching of melt, Si and Zr can be separated.

$$ZrSiO_4 + 6NaOH = Na_2ZrO_3 + Na_4SiO_4 + 3H_2O \qquad (1)$$

In reaction products sodium silicate is soluble in water and sodium zirconate is dissoluble. During the alkali molten decomposition Ti, Fe, Al react with NaOH to be obtained Na_2TiO_3, $Na_2Fe_2O_4$ and $Na_2Al_2O_4$, respectively, they are soluble in water very well.

The yield(%) of alkali molten decomposition can be calculated as follows.

$$Q = \frac{m}{M} \times 100 \qquad (2)$$

where Q is decomposition yield(%), M weight of sample before decomposition and m weight of residue which decompose in HCl.

2. Experiment Result

2.1. The influence of weight ratio of zircon and alkali

Fig. 1 shows the decomposition yield of zircon according to weight ratio of zircon and alkali in 750℃ of reaction temperature, 1h of reaction time and 70μm of grain size.

As shown in Fig. 1, the decomposition yield of $ZrSiO_4$ is the highest on (1.2～1.3) : 1 of weight ratio.

2.2. The influence of melting temperature

Fig. 2 shows the alkali molten decomposition yield with melting temperature in weight ratio 1.2 : 1, reaction time 1h and grain size 70μm.

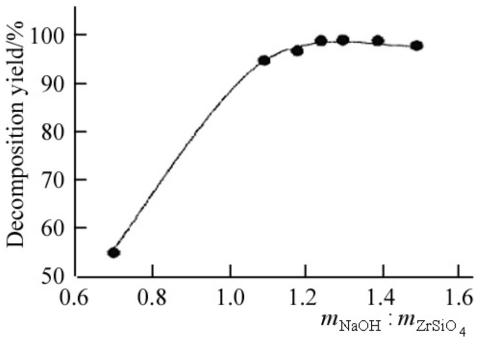

Fig. 1. The influence of weight ratio of zircon and alkali on the decomposition yield

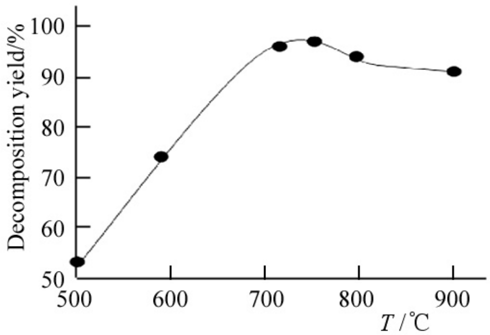

Fig. 2. The influence of melting temperature on the decomposition yield

As shown in Fig. 2, when the reaction temperature is 700～750℃, the decomposition yield is the highest. Because the temperature below 700℃ is too low for zircon to react with alkali and the temperature more than 750℃ volatilizes alkali to make the reaction efficiency reduce.

2.3. The influence of reaction time

In alkali molten decomposition the decomposition yield increased suddenly on the initial stage of reaction, but didn't almost change after 1.5h (Fig. 3).

That is, the optimum reaction time is 1.5h.

2.4. The influence of grain size of zircon

After milling for a certain time sample was assorted in a sieve to prepare, in the reaction condition of $m_{NaOH} : m_{ZrSiO_4}$ 1.2 : 1, the reaction time 1.5h and the reaction temperature 750℃, and in the condition of different grain size the experiment of alkali molten decomposition was done (Fig. 4).

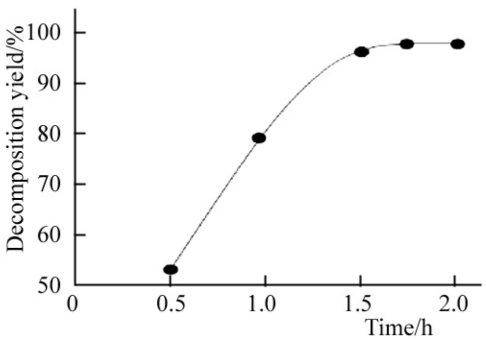

Fig. 3. The influence of reaction time on the decomposition yield

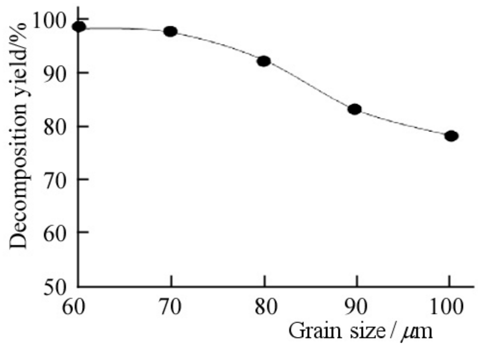

Fig. 4. The influence of grain size of zircon on the decomposition yield

As shown in Fig. 4, when the grain size of zircon is less than 70μm, alkali molten decomposition yield is almost 100% and when more than 70μm the decomposition yield becomes less than 95%.

3. Establishment of Optimum Condition

We confirmed the optimum condition changing the different factors on alkali molten decomposition of zircon by using the method of quality engineering.

The experiment plan was made by using $L_9(3^4)$ type of orthogonal table and the experiment proceeded.

3.1. Apportionment of factor and level

Based on the previous research results [1] we selected four indices affecting on alkali molten decomposition of zircon as factor. And set up the optimum condition obtained from experiment result of elementary divisor as fiducial value (table 1).

Table 1. Control factor and its level

Factor	Unit	Level		
		1	2	3
A : weight ratio of zircon and alkali	—	1 : 1.1	1 : 1.2	1 : 1.3
B : melting temperature	℃	700	750	800
C : reaction time	h	1.0	1.5	2.0
D : grain size	μm	60	70	80

3.2. Experiment value and calculation of SN ratio

Experiment value is decomposition yield. Also the greater decomposition yield, the more increasing characteristic, so SN ratio was be calculated by equation (3).

$$\eta = -10\lg\left[\frac{1}{n}\left(\frac{1}{y_1^2}+\frac{1}{y_2^2}+\cdots+\frac{1}{y_n^2}\right)\right] \tag{3}$$

In the experiment condition given on $L_9(3^4)$ type of orthogonal table we experimented two times in each experiment point to obtain values by averaging experiment values.

Table 2 shows the experiment results and SN ratio.

Table 2. Experiment results and SN ratio

No.	Factor				Yield /%	SN ratio/dB
	A	B	C	D		
1	1	1	1	1	92	39.28
2	1	2	2	2	97	39.74
3	1	3	3	3	94	39.46
4	2	1	2	3	96	39.65
5	2	2	3	1	98	39.82
6	2	3	1	2	93	39.37
7	3	1	3	2	98	39.82
8	3	2	1	3	91	39.18
9	3	3	2	1	97	39.74

3.3. Auxiliary table

Based on table 2, we made an auxiliary table from sum by level of each factor (table 3).

Table 3. Auxiliary table

No.	Factor			
	A	B	C	D
1	118.48	118.75	117.83	118.84
2	118.84	118.74	119.12	118.93
3	118.74	118.57	119.11	118.29
Σ	356.06	356.06	356.06	356.06

3.4. Variance analysis table

Table 4 shows the results that we find variation and variance of each row and calculate the contribution yield of each factor from the auxiliary table.

As shown in Table 4, reaction time has the most influence on the alkali molten decomposition process and reaction temperature has no great effect on like that.

Table 4. Variance analysis table

Factor	Degree of freedom	Variation	Variance	Pure variation	Contribution yield /%
A	2	0.023 0	0.011 5	0.016 2	3.39
B	2	0.006 8	0.003 4	—	
C	2	0.367 0	0.183 5	0.360 2	75.54
D	2	0.080 0	0.040 0	0.073 2	15.35
(e)				0.027 3	5.72
T	8			0.476 8	100

3.5. Discussion of optimum condition and maximum extraction yield

From Table 3 the optimum condition of alkali molten decomposition process of zircon is $A_2B_1C_2D_2$. That is, weight ratio of zircon and sodium hydroxide is 1 : 1.2, alkali molten decomposition temperature is 700℃, reaction time is 1.5h and grain size of concentrate is 70 μm . When we calculate SN ratio from the optimum condition and find maximum decomposition yield, it is (98.17±1.01)%.

On alkali molten decomposition of zircon in optimum condition the decomposition yield is 98.02% and the relative error is 0.15%.

Conclusion

We had an experiment of alkali molten decomposition condition of zircon. The most important factors with alkali molten decomposition of zircon concentrate are weight ratio of alkali and zircon, reaction time and grain size of concentrate and so on.

From the experiment results the optimum condition of alkali molten decomposition process of zircon is as follows: weight ratio of alkali and zircon is 1.2 to 1, temperature of alkali molten decomposition is 700℃, reaction time is 1.5h, the grain size of concentrate is 70μm, and the decomposition yield in optimum condition reaches more than 98%.

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