

## Studies on the Manufacturing of Carbon Bond Graphite Crucible

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### 카아본 본드형 흑연 도가니 제조에 관한연구

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#### 요 약

Carbon bond 型 黑鉛도가니는 clay bond 型 黑鉛도가니에 比하여 熱傳導率이 크고 熱膨脹率이 적으며, 熱間荷重性이 좋을 뿐 아니라 熱衝擊에도 安全하여 그 使用 壽命이 100餘回나 된다.

본 연구결과 carbon bond 型 黑鉛도가니의 제조에 適合한 原料의 調合比는 鱗狀黑鉛 40, 炭化珪素 15, Ferrosilicon 25, 無機質結合劑 15, tar pitch 11, 氷晶石 3, ferromanganese 2 있으며, 黑鉛도가니 製造에 所要되는 原料들의 相互關係는 黑鉛분이 增加할수록 酸化率 및 氣孔率이 增加하고, 氣孔率의 增加도 黑鉛의 酸化量에 比例하였으며, 炭化珪素의 增加는 酸化率 및 氣孔率은 增加시켰고 ferrosilicon의 增加는 氣孔率 및 酸化率에 別 영향을 미치지 않았고, 유리質의 增加는 酸化率 및 氣孔率을 減少시키나 過量이면 bloating 現象이 일어나 오히려 氣孔率 이 커졌다. 製造된 도가니의 物性은 浮比比重 2.31, 겔보기비중 2.58, 氣孔率 15.2%, 吸水率 6.01%, 壓縮強度 438kg/cm<sup>2</sup>, 引張強度 256kg/cm<sup>2</sup>, 酸化消耗率 3.77% 以下이며 平均使用 壽命은 105回였다.

#### ABSTRACT

This study was focused on the improvement of production techniques of small crucibles in relation with the appropriate selection of raw materials, various batch compositions and physical and chemical characteristics of the crucibles.

Various tests gave the optimum batch composition for the carbon bond graphite crucible as follows:

Pyontaek graphite flake (refractory aggregate):	40Part
Silicon carbide:	15 "
Tar pitch (binder):	11 "
Inorganic additives (to improve the oxidation resistance):	15 "
Cryolite:	3 "
Ferro manganese :	2 "
Ferrosilicon:	25 "

Crucibles pressed with 400kg/cm<sup>2</sup> at 120°C. and fired in reducing atmosphere at 1200°C brought the most favorable results as follows:

Bulk density	: 2.31
Apparent density	: 2.58

Porosity	: 15.2%
Oxidation loss at 1,500°C. for 3 hrs	: below 3.77%
Water absorption	: 6.01%
Compressive strength	: 438kg/cm <sup>2</sup>
Tensile strength	: 256kg/cm <sup>2</sup>

## 1. Introduction

Clay bonded graphite crucible consisted mainly of graphite flake and clay having different thermal expansion characteristics respectively should be pre-heated before melting metallic materials to avoid cracking and partial fracture resulted in the transition of free quartz in the clay refractories when heated suddenly.

Hydrophilic graphite crystal deteriorates the plasticity of the mass clay which should be plastic when added water. Therefore, several days are required for mixing and kneading the mass clay and poor thermal conductivity provides high fuel consumption. And also frequent transformation can be occurred owing to high thermal load characteristics at high temperature.

Present graphite crucibles in the advanced countries contained with binders such as coal tar pitch, pulp waste liquor, and synthetic resins in place of clay. are manufactured by providing these crucibles bonding properties by residual carbon fine particles formed when these organic binders are fired.

Similar thermal expansion coefficient and physical

and chemical properties of carbon fine particles with that of graphite show excellent product qualities in every respect.

This study was carried out to manufacture domestic carbon bonded graphite crucibles.

## 2. Selection of Raw Materials

Analysis of carbon bonded graphite crucible by Heindl<sup>1)</sup>, Eastel<sup>2)</sup>, and Yamawoochi<sup>3)</sup>, and major proportion of Suprex crucibles of Morgan Co. in England show the use of graphite flake, ferro-silicon and silicon carbide as refractory aggregate of the crucible, coal tar pitch, pulp waste liquor, synthetic resins including phenolic resin as a binder, and other particular inorganic materials to prevent the surface oxidation by self-glazing at a required temperature.

Additional additives such as cryolite and ferromanganese were selected in this study to increase the physical properties of the glaze formed at high temperature. Chemical compositions of the selected graphite flake and physical and chemical properties of the graphite ash are shown in Table 1 and 2 respectively

Particle size distribution (Fig. 1) were, measured by

Table 1. Chemical compositions of the graphite

Components	Moisture (%)	Ash (%)	Volatile material (%)	Fixed carbon (%)
Content	0.25	11.95	3.25	84.7

Table 2. Physical and chemical properties of the graphite ash

Components	SiO <sub>2</sub> (%)	Al <sub>2</sub> O <sub>3</sub> (%)	Fe <sub>2</sub> O <sub>3</sub> (%)	CaO(%)	MgO(%)	$\frac{Na_2O}{K_2O}$ (%)	SK
Content	49.13	34.16	12.92	0.77	1.94	1.04	19

sieving method with ASTM standard sieve. Oxidation rate were determined to study the oxidation mechanism of the graphite in the following procedure: Reaction rate of solid and gas is proportional to the surface area of the solid exposed on the gas. Pre-determined amount of sample with a specified range of particle size is

soaked at 900°C. to react in a stream of oxygen in an electric furnace. Oxidation rate is reversely proportional to the residual amount of graphite and the following equation can be obtained:

$$\frac{d(a-x)}{dt} = -k(a-x) \quad (1)$$

where

- a : original amount of graphite, mg
- x : graphite oxidized after t min., mg
- k : constant,

and

t : reaction time, min.

Equation (1) can be rewritten as follows:

$$\log(a-x) - \log a = -\frac{kt}{2.303} \quad (2)$$

$$\text{or } t = -\frac{2.303}{k} \log a + \frac{2.303}{k} \log(a-x) \quad (3)$$

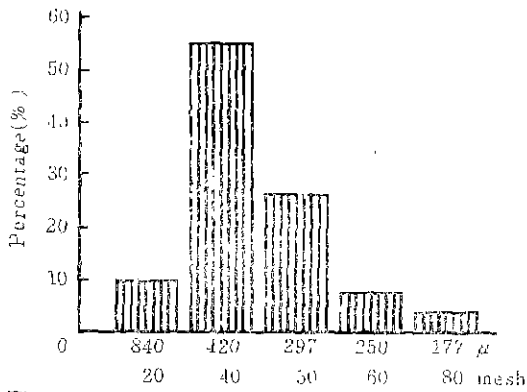


Fig. 1. Size distribution of the graphite flakes.

Table 2. Oxidation rate, K calculated for 10 minute intervals for Pyong taek graphite passing No. 80 sieve and remaining on No. 20 sieve

t (min.)	a-x (mg)	k (min <sup>-1</sup> )
0	1000	
10	392	0.098
20	152	0.101
30	71	0.099
40	43	0.105
50	13	0.094
60	4	0.104
Average		0.101

When t is plotted against log (a-x), a straight line is obtained with the slope of -2.303/k. Fig. 2 and Table 3 were brought by the substitution of the slope with the above equation.

Physical and chemical properties of tar pitch as a binder and borosilicate glass to form surface coating are shown in Table 4 and 5.

Other materials such as ferro-silicon, silicon carbide, ferro-manganese, and cryolite are domestic or

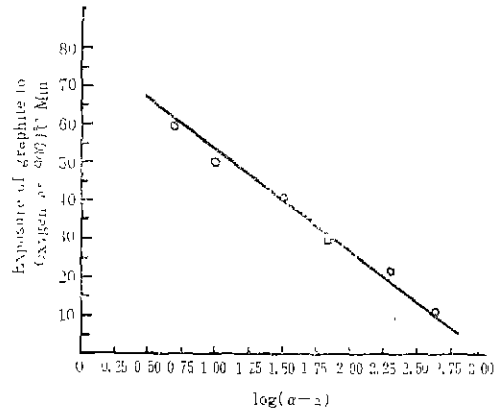


Fig. 2 Oxidation—80+20 sieve fraction of Pyong taek graphite flake at 900°C as a function of time

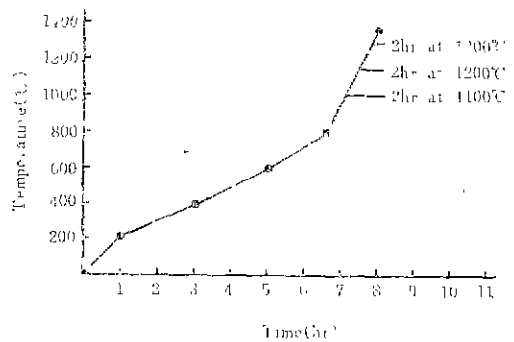


Fig. 3. Firing schedule

Table 4. Composition and physical properties of borosilicate glass

SiO <sub>2</sub> (%)	Al <sub>2</sub> O <sub>3</sub> (%)	Fe <sub>2</sub> O <sub>3</sub> (%)	B <sub>2</sub> O <sub>3</sub> (%)	CaO(%)	MgO(%)	Na <sub>2</sub> O(%)	Sag point(°C)	Softening point(°C)
74.8	3.5	0.2	12.2	0.2	0.5	8.6	580	630

Table 5. Composition and physical properties of tar pitch

Fixed carbon(%)	Benzene insoluble(%)	Fixed carbon Benzene insoluble (%)	ash (%)	Softening point (Hg-method)(°C)
48.02	19.31	28.71	0.10	67.5

foreign-made and their quality and characteristics are so widely known that any particular experiment is not required.

### 3. Batch Composition

Table 6 shows the batch composition of the raw materials.

Table 6. Batch compositions of graphite-refractory

Sample no.	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18
Raw materials.																		
Graphite	60	60	60	60	60	60	60	60	60	60	60	60	50	50	50	50	50	50
Siliconcarbide	35	25	15	5	10	15	20	25	10	15	20	25	35	25	15	5	10	15
Ferrosilicon	10	15	20	25	35	25	15	5	10	15	20	25	10	15	20	25	35	25
Borosilicate	10	15	20	25	10	15	20	25	35	25	15	5	10	15	20	25	10	15
Tar-pitch	13	13	13	13	13	13	13	13	13	13	13	13	12	12	12	12	12	12
Cryolite	3	3	3	3	3	3	3	3	3	3	3	3	3	3	3	3	3	3
Ferromanganese	2	2	2	2	2	2	2	2	2	2	2	2	2	2	2	2	2	2

Sample no.	19	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34	35	36
Raw materials																		
Graphite	50	50	50	50	50	50	40	40	40	40	40	40	40	40	40	40	40	40
Siliconcarbide	20	25	10	15	20	25	35	25	15	5	10	15	20	25	10	15	20	25
Ferrosilicon	15	5	10	15	20	25	10	15	20	25	35	25	15	5	10	15	20	25
Borosilicate	20	25	35	25	15	5	10	15	20	25	10	15	20	25	35	25	15	5
Tar-pitch	12	12	12	12	12	12	11	11	11	11	11	11	11	11	11	11	11	11
Cryolite	3	3	3	3	3	3	3	3	3	3	3	3	3	3	3	3	3	3
Ferromanganese	2	2	2	2	2	2	2	2	2	2	2	2	2	2	2	2	2	2

### 4. Results and Consideration

Particle size of the graphite flake of the raw materials in Table 6 was the same as in Fig. 1. Ferro-silicon and ferro-manganese were ground to pass to 35 to 100 mesh, silicon carbide to pass to 100 mesh, and borosilicate glass and cryolite to pass to 200 mesh. These materials were kneaded by laboratory kneader at room temperature for an hour, mixed and kneaded with tar-pitch at around 120°C. for 2 hours, and then were

pressed 100gr. of the batch mixture in cylindrical mold at 110C. with the pressure of 400kg/cm<sup>2</sup>. These specimens were deeply placed into the refractory setter filled with anthracite coal powder, and then covered to fire at 1,100°, 1,200°, and 1,300°C respectively for two hours in an electric furnace. Firing schedule is shown in Fig. 3.

Table 7 represents physical properties such as apparent density, bulk density, and porosity of the graphite crucible.

Table 7. Physical properties of graphite-refractory

Sample no.	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18
Firing temp.																		
Item																		
1100°C	Apparent density	2.08	2.09	2.12	2.11	2.09	2.12	2.13	2.14	2.16	2.15	2.14	2.16	2.17	2.17	2.18	2.19	2.18
	Bulk density	2.01	2.03	2.03	2.04	2.02	2.01	2.03	2.05	2.07	2.05	2.04	2.02	2.06	2.07	2.07	2.09	2.08
	Porosity (%)	23.2	21.9	21.1	21.4	21.8	21.3	19.9	19.7	18.5	18.8	19.0	18.9	18.7	18.4	18.3	18.5	17.7
1200°C	Apparent density	2.10	2.12	2.15	2.15	2.10	2.14	2.16	2.18	2.19	2.18	2.16	2.16	2.19	2.19	2.20	2.21	2.22
	Bulk density	2.03	2.04	2.04	2.06	2.03	2.02	2.05	2.06	2.08	2.07	2.07	2.05	2.07	2.09	2.08	2.10	2.09
	Porosity (%)	22.2	21.5	21.0	21.0	20.8	20.6	19.6	19.4	18.2	18.3	18.7	18.9	18.5	18.1	17.8	17.6	17.5
1300°C	Apparent density	2.12	2.14	2.14	2.15	2.14	2.25	2.26	2.29	2.29	2.28	2.27	2.27	2.29	2.28	2.31	2.31	2.30
	Bulk density	2.04	2.05	2.03	2.05	2.05	2.02	2.04	2.07	2.09	2.08	2.08	2.0	2.08	2.07	2.09	2.14	2.08
	Porosity (%)	22.0	21.9	22.0	21.0	21.1	20.8	19.8	19.3	18.1	18.0	17.5	19.0	18.1	18.5	17.5	16.6	17.1

Firing temp.	Sample no Item	19	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34	35	36
		1100°C	Apparent density	2.19	2.21	2.23	2.22	2.22	2.21	2.25	2.27	2.28	2.28	2.25	2.26	2.28	2.27	2.28	2.25
	Bulk density	2.09	2.10	2.10	2.10	2.08	2.07	2.12	2.14	2.23	2.24	2.20	2.23	2.24	2.23	2.28	2.21	2.18	2.11
	Porosity (%)	17.6	17.5	17.1	17.4	17.6	17.6	16.8	16.7	16.5	16.6	16.6	16.4	16.5	16.2	16.1	16.4	16.5	16.7
1200°C	Apparent density	2.27	2.29	2.31	2.34	2.35	2.35	2.41	2.48	2.51	2.54	2.57	2.60	2.60	2.59	2.53	2.58	2.51	2.41
	Bulk density	2.10	2.12	2.14	2.12	2.10	2.09	2.20	2.21	2.23	2.26	2.25	2.26	2.27	2.28	2.29	2.27	2.25	2.28
	Porosity (%)	17.4	17.4	16.9	16.8	16.5	16.3	16.1	16.0	15.8	15.6	16.1	16.0	15.8	15.6	15.5	16.3	16.4	16.5
1300°C	Apparent density	2.31	2.31	2.33	2.36	2.32	2.31	2.37	2.39	2.41	2.49	2.52	2.57	2.58	2.57	2.54	2.55	2.56	2.52
	Bulk density	2.11	2.13	2.14	2.14	2.12	2.10	2.24	2.25	2.26	2.28	2.27	2.28	2.28	2.29	2.27	2.29	2.26	2.24
	Porosity (%)	17.3	17.5	16.8	16.7	16.5	16.2	16.0	16.4	15.9	15.5	16.0	15.8	15.5	15.8	16.1	16.4	16.8	17.8

Compressive strength and oxidation loss are shown in Fig. 4

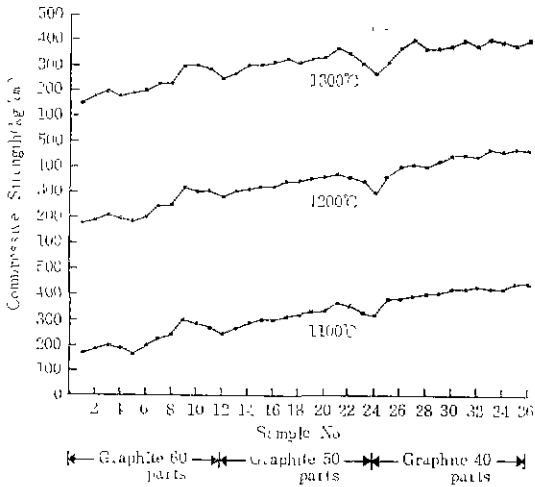


Fig. 4. Compressive strength of the graphite refractory at fired temp.

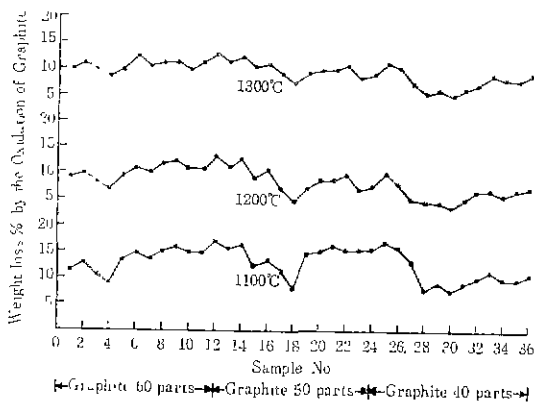


Fig. 5. Weight loss % by the oxidation of graphite at fired temp.

Physical properties such as apparent density and bulk density are increased as the firing temperature is raised, and porosity is decreased. This is resulted in mainly the decrease in the viscosity of fused borosilicate glass which provides the increase in fluidity by cohesion and capillary phenomena and is concentrated to space voids to form resistant coat to oxidation and to close the pores. It is, therefore, considered the decrease in the open pores increases the apparent and bulk density and decreases the porosity.

Compressive strength shows higher value for the specimens fired at 1,200°C. than at 1,100°C., but slight lower value is shown at 1,300°C. Which can be explained in term of the decrease in strength by the formation of the expandable SiO<sub>2</sub> and Fe<sub>2</sub>O<sub>3</sub> produced by the accelerated oxidative decomposition of SiC and ferro-silicon at 1,300°C, which are the reactants of melted glass and SiC or ferro-silicon at 1,000°C.

In addition, physical properties of the specimens fired at 1,300°C. are similar with those of the specimens fired at 1,200°C. and therefore, the optimum firing temperature of the carbon bonded graphite crucible was found to be 1,200°C.

Comparison of oxidation percentage with porosity according to the batch compositions shows the increasing amount of graphite increases the oxidation percentage and porosity and the increase in porosity is found to be proportional to the oxidation amount. Increasing amount of borosilicate glass decreases the oxidation percentage and porosity, however, more than 20 parts brings bloating and increase in porosity while porosity has the tendency to be decreased at higher temperature firing which causes shrinkage of structure by fusion.

Increasing amount of silicon carbide shows the decrease in oxidation percentage and porosity, in which increase in porosity by the expansion of the structure accelerates the oxidation of SiC to SiO<sub>2</sub> and by the increasing temperature and the content of SiC. Bloating can be occurred in the glass by CO<sub>2</sub> formed by decomposition of SiC by alkali contained in it. Increasing amount of ferro-silicon has not considerable effect on oxidation percentage and porosity. However, when added more than 25 parts shrinkage was shown at 1,200°C. and expansion was brought at 1,300°C. Shrinkage was considered to be produced by metallic bond by ferro-silicon around at 1,208°C., melting point of ferro-silicon, above which it is considered that expansion can be shown by the reaction oxygen, and mechanical strength at high temperature will be decreased owing to the decrease in softening point of the refractories.

The lowest value of oxidation loss was obtained with specimen No. 18 and 30, of which batch compositions were selected as that of carbon bonded graphite crucible in this study.

Oxidation mechanism of graphite crucible can be considered as follows: First, surface oxidation of the crucible is caused by its surface condition, gas components in the furnace, and migration of oxidized materials formed on its surface. Second, oxidation proceeds into the body of the crucible. Oxidation is proceeded by the oxygen penetrated into the pores from the surface as the result of controlled diffusion rate of oxygen.

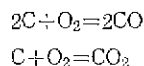
Therefore, when the diffusion rate is accelerated the volume of the oxidized materials is increased to collapse the structure and increase the porosity. When retarded the rate of diffusion the materials densify the structure and prevents the diffusion of oxygen.

The reason of the addition of the glassy material is based on the prevention of oxygen by the formation of glassy coating on the surface of carbon, graphite and silicon carbide around at 1,000°C., however, the glassy material is concentrated to carbon owing to the low viscosity and surface tension of the glass above 1,000°C. and the melting of ferro-silicon increases the viscosity of the glass and forms the oxidation resistant coating on the surface of graphite. The addition of

large amount of the glassy material and ferro-silicon would bring the decreased softening point, poor strength at high temperature, decreased thermal shock characteristics caused by low thermal conductivity, and the acceleration of oxidation caused by breaking the carbon bond between silicon carbide and graphite.

This consideration makes it possible to get the optimum composition in the batch of silicon carbide 15 parts and ferro-silicon 25 parts within the limits of glassy material 15 parts, and the lowest oxidation loss was brought by this batch.

Cryolite was added to increase the physical properties of the solution fused in the graphite crucible at high temperature and the addition of ferro-manganese is based on the study of S. E. Hofmann<sup>4)</sup>, J. R. Arthur<sup>5)</sup>, and B. Neumann<sup>6)</sup>, who concluded that oxidation of manganese effects the negative catalyst in the oxidation of carbon when the free energy of Mn in oxidation is larger than that of the following reaction at 1,200°C.



## 5. Particle Size Control and Its Results

Particle size of the graphite flake was taken as that of Fig. 1. Particle size of ferro-silicon and silicon carbide occupying 40% in the batch was controlled as in Table 8.

Particle size controlled batch was molded and electrically fired at 1,200°C. in reducing atmosphere for two hours. Physical properties was examined for the fired specimen and are shown in Table 9.

Experimental results show the excellent physical properties in the specimen of series No. 30-b-1 which obtained 50 times thermal shock resistance with DIN testing method, presents the favorable thermal shock resistance, increases in the mechanical strength and bulk density caused by the densification of the structure even though poor porosity was brought by the large portion of coarse particles.

## 6. Preparation of Inorganic Bonding Agent and Its Result

E. A. Gulbransen<sup>7)</sup> ascertained the oxidation of graphite as three stages as follows: Adsorption of oxygen is primarily proceeded on a portion of carbon.

Table 8. Control of particle size distribution

Sample no	Ratio of particle size			Particle size percentage of SiC and ferrosilicon				
	Coarse grain	Medium grain	Fine grain	Coarse grain		Medium grain		Fine grain
				20~35mesh	35~60mesh	60~80mesh	80~100 mesh	>100 mesh
30-a-1	5	—	1	41.66%	41.66%	—	—	16.67%
30-a-2	3	—	1	37.50	37.50	—	—	25.00
30-a-3	1	—	1	25.00	25.00	—	—	50.00
30-a-4	1	—	3	12.50	12.50	—	—	75.00
30-a-5	1	—	5	8.34	8.34	—	—	83.32
30-b-1	5	1	1	35.71	35.71	7.14	7.14	14.29
30-b-2	3	1	1	30.00	30.00	10.00	10.00	20.00
30-b-3	1	1	1	16.66	16.66	16.66	16.66	33.33
30-b-4	1	1	3	10.00	10.00	10.00	10.00	60.00
30-b-5	1	1	5	7.14	7.14	7.14	7.14	71.43
30-c-1	5	2	1	31.25	31.25	12.50	12.50	12.50
30-c-2	3	2	1	25.00	25.00	16.67	16.67	16.66
30-c-3	1	2	1	12.50	12.50	25.00	25.00	25.00
30-c-4	1	2	3	8.30	8.30	16.60	16.60	49.80
30-c-5	1	2	5	6.25	6.25	12.50	12.50	62.50

Table 9. Physical properties of fired bodies

Sample no	Item	Apparent density	Bulk density	Porosity (%)	Water absorption (%)	Compressive strength kg/cm <sup>2</sup>	Tensile strength kg/cm <sup>2</sup>
30-a-1		2.27	1.98	16.80	8.21	331	225
30-a-2		2.28	2.01	16.50	7.11	339	230
30-a-3		2.30	2.03	17.21	6.35	350	245
30-a-4		2.31	1.97	16.90	8.10	342	241
30-a-5		2.32	1.96	17.12	9.81	335	230
30-b-1		2.58	2.31	15.21	6.01	438	256
30-b-2		2.56	2.27	16.80	7.21	421	242
30-b-3		2.58	2.19	16.05	8.05	387	237
30-b-4		2.39	2.22	17.11	8.27	345	351
30-b-5		2.29	2.17	18.23	9.31	340	248
30-c-1		2.41	2.21	16.50	7.82	397	232
30-c-2		2.27	2.18	17.23	8.33	379	244
30-c-3		2.29	2.15	18.31	9.05	356	239
30-c-4		2.42	2.21	16.91	9.98	412	241
30-c-5		2.45	2.27	17.73	8.73	408	251

Table 10. Batch compositions of inorganic additives

Component	SiO <sub>2</sub> (%)	Al <sub>2</sub> O <sub>3</sub> (%)	B <sub>2</sub> O <sub>3</sub> (%)	Na <sub>2</sub> O+K <sub>2</sub> O (%)	ZnO(%)	CaO+MgO (%)	MnO <sub>2</sub> (%)	P <sub>2</sub> O <sub>5</sub> (%)	ZrO <sub>2</sub> (%)
A	73.50	3.02	13.58	6.08	1.78	1.07	—	—	—
B	73.50	3.50	12.40	8.60	—	0.70	1.30	—	—
C	67.50	3.00	8.50	7.70	—	1.32	—	12.0	—
D	71.30	2.85	13.57	8.11	—	0.93	—	—	2.24

atom bonded with carbon and then absorbed oxygen reacts with carbon atom followed by desorption of oxygen. Therefore, the factor influencing the rate of oxidation reaction is proportional to the absolute temperature,

the geometrical surface area of the sample, and the diffusion rate of gas.

Oxidation is proceeded by the diffusion of oxygen into the boundary layer of the oxidized glassy material.

formed at high temperature when the surface area and service temperature of the crucible is considered constant, and thickness of the layer influences the rate of oxidation of carbon.

This consideration furnished the glassy coating for the protection of the crucible. Oxidation prevention film can be obtained when oxidized film is adhered on the surface of graphite at room temperature enough to prevent the passage of air and the glassy film safe at high temperature is formed.

Inorganic bonding agent of the intention mentioned above was prepared by the batch in Table 10.

Thus prepared fused samples of four kinds were added 15% each in place of borosilicate for the graphite crucible batch of No. 30 and 30-b-1 (particle size controlled), mixed, and then fired to make specimen A, B, C, and D. Oxidation loss of these specimens are shown in Table 11.

**Table 11.** Weight loss % by the oxidation of graphite

Item Sample No.	Weight loss % by the oxidation		
	1300°C, 2hr	1400°C, 2hr	1500°C, 2hr
30-b-1-A	1.45	2.35	4.21
30-b-1-B	1.57	2.49	3.87
30-b-1-C	1.61	2.73	3.94
30-b-1-D	1.12	2.33	3.77

Comparatively favorable results were obtained for the specimens of 30-b-1-A and 30-b-1-D, however, 30-b-1-D was selected as the batch for the graphite crucible because 30-b-1-A has the fast oxidation rate at high temperature.

The No. 4 crucible, with favorable batch 30-b-1-D, could use 105 times, when in operation of Brass melting.

## 8. Conclusion

The following conclusion was attained in view point of the manufacture techniques of the carbon bonded graphite crucible:

A) Pyongtaek graphite flake is poor grade for the manufacture of the graphite crucible because of its low carbon content of 84.7% and its particle size is so fine that the constant rate of the oxidation shows comparative higher value of 0.101.

B) Relations between the raw materials for the

carbon bonded graphite crucible represent the increasing oxidation loss and porosity with increasing addition of graphite and the increase in porosity is proportional to the oxidation percentage of graphite. Increasing addition of silicon carbide brings the increase in oxidation percentage and porosity, and increasing amount of ferro-silicon has little effect on porosity and oxidation percentage. Increasing addition of glassy material decreases the oxidation percentage and porosity while excess addition provides high porosity caused by bloating.

C) Mixing ratio for the carbon bonded graphite crucible can be fixed as follows:

### Pyongtaek

Graphite flake	40 parts
Silicon carbide	15 parts
Ferro-silicon	25 parts
Inorganic bonding agent	15 parts
Tar pitch	11 parts
Cryolite	3 parts
Ferro-Manganese	2 parts

D) Physical properties of the prepared crucible are as follows:

Bulk density	2.31
Apparent density	2.58
Porosity	15.2%
Water absorption	6.01%
Compressive strength	438kg./cm <sup>2</sup>
Tensile strength	256kg/cm. <sup>2</sup>
Oxidation loss	3.77%

E) The most favorable inorganic bonding agent has the following composition:

SiO <sub>2</sub>	71.30%
Al <sub>2</sub> O <sub>3</sub>	2.85%
B <sub>2</sub> O <sub>3</sub>	13.57%
Na <sub>2</sub> O+K <sub>2</sub> O	8.11%
CaO+MgO	0.93%
ZrO <sub>2</sub>	2.24%

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