CALCINING OF OLIVINE FOUNDRY SAND IN FLUOSOLID AND TUBE-TYPE FURNACES

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Introduction

The numerous dunite occurrences in western North Carolina contain one of the State's important mineral resources - the olivine. $^{(1)}$ Olivine is a fine crystalline silicate mineral which forms an isomorphous series having magnesium-rich forsterite($\mathrm{Mg}_2\mathrm{SiO}_4$) on one end and iron-rich fayalite ($\mathrm{Fe}_2\mathrm{SiO}_4$) on the other end. The name olivine is used when less than 10 percent of the rock is fayalite, as is the case in the North Carolina deposits.

The production of olivine sand for foundry uses started in North Carolina in 1960, after some intermittent mining of olivine for the production of forsterite refractory bricks and ramming mixes (2), and has expanded to 25,000 to 30,000 tons per year in 1970. The production should be expanding rapidly because of elimination of silicosis dangers in foundries when using olivine and because of technical advantages when using olivine sand in nonferrous and ferrous foundries.

The ore as mined contains small amounts of accessory minerals such as serpentine, talc, vermiculite, chlorite and small amounts of chromite. (3, 4) With the exception of chromite, all accessory minerals should be eliminated from olivine foundry sand. The ore mined for olivine foundry sand is crushed, ground, deslimed and separated from the accessory minerals by gravity separation on spirals. The concentrated olivine sand is then dried and sized for shipment. The olivine

foundry sand shipped from North Carolina has an appealing white-graygreen color and has the following chemical analysis.

Chemical Analysis of North Carolina Olivine

MgO	50.5 %	
.si0 ₂	40.1	
Fe ₂ 0 ₃	6.7 (Fe reported as Fe ₂ 0 ₃	;)
Ca0	0.2	
Ign. Loss	0.7	

Ignition losses determined in inert atmosphere by standard procedures are used by the industry as means of quality control, together with size analysis and casting tests. Casting tests run under simulated foundry conditions reveal if there is pitting on the castings through gas evolution. Ignition loss lower than what is obtained by current mineral beneficiation methods, which is between 0.5 and 1.0 percent ignition loss, is highly desirable and will open new avenues for sales.

Heat treatment or calcining is the obvious way to obtain lower ignition losses and is used in Europe by one olivine foundry sand producer, (Magnolithe G.m.b.H., St. Lorenzen, Austria). This European producer apparently uses a rotary kiln for calcining of olivine. A disadvantage of calcining in a rotary kiln is the direct contact with combustion gases and oxidation of the iron in the fayalite component of olivine which changes the color of the foundry sand to dark brown.

Calcining of Olivine and Accessory Minerals

The thermal treatment of olivine and accompanying hydrated silicates has been studied and reported in European literature . (6,7,8)

It is of great interest to notice what happens to the accessory minerals,

especially the hydrated minerals such as serpentine and talc. As a result of thermal decomposition of the serpentine minerals, olivine-type minerals are formed. The reaction is as follows:

serpentine
$$700^{\circ}$$
 C forsterite quartz water
2 $H_4Mg_3Si_2O_9$ \longrightarrow 3 Mg_2SiO_4 + SiO_2 + 4 H_2O

forsterite quartz 1050° C forsterite enstatite 3 Mg₂SiO₄ + SiO₂ \longrightarrow 2 Mg₂SiO₄ + 2 MgSiO₃

Talc decomposes to enstatite as a result of heating at 900° C.

Calcining of olivine ore or olivine sand, therefore, can have two main functions: first, complete elimination of water of crystallization and improvement of foundry sand properties; second, increase of olivine yield by eliminating the wett gravity concentration step, which also rejects some of the olivine with the serpentine minerals. This is done through calcining by converting of unwanted water-containing magnesium silicates to desired water-free magnesium silicates. A disadvantage of calcining of olivine in conventional rotary kilns or fluid-type calciners is partial oxidation of the ferrous iron in the fayalite to ferric iron. The Fe₂0₃ formation is accompanied by the formation of the SiO2 phase, christobalte, which combines with forsterite to form the magnesium silicate, enstatite. Besides $\mathrm{Fe}_2\mathrm{O}_3$ there is formation of Fe₃0₄. The iron oxide formation is not desirable in foundry sands as binder requirements change in proportion to large surface areas formed by iron oxides. Calcination and removal of water of crystallization, without oxidation of iron, is therefore desirable

and could be accomplished by the writer in laboratory batch and continuous test work and in plant continuous work.

Laboratory Test Work

A fluidized-bed calcining furnace was built in the laboratory for studies on North Carolina and North African phosphates and for olivine calcining studies. Because of the high sintering temperature of olivine, this material was used to check the equipment out and to study fluidizing, air distribution and temperature control conditions of the constructed furnace. Whenever temperatures of about 1500° F were employed, the calcined olivine had a low ignition loss of 0.05 to 0.10 percent and a brown color. Poundage samples of calcined No. 70, No. 120, and spiral feed, unconcentrated olivine were submitted to Northwest-International olivine company for evaluation as foundry sand. The test data are presented on Tables 1, 2 and 3.

A tube-calciner principle was developed to calcine in a continuous way. In the process of testing the tube-calciner concept, it was discovered that calcining of olivine to low ignition losses could be accomplished without change of color to dark brown. The calcine produced had a white-gray color. This was noted as an added benefit. The principle involved is apparent lack of enough oxygen in between packed olivine particles and prevention of oxidation through water vapor and steam formation.

The Tube-Calciner Principle

The principle of a tube calciner is a simple one and takes advantage of the free-flowing properties of olivine sand. Figure 1 shows a schematic diagram of the laboratory tube-type calciner. A

vertical tube is filled with olivine sand. The movement of the olivine from the top to the bottom is regulated by an orifice or pulsating or vibratory device. Heat is applied to the olivine by electricity or other means, internally or externally, so that all of the olivine is at least heated to 1500° F. A thermocouple controls either the rate of heat application or the rate of calcine withdrawal. The water of crystallization and surface moisture diffuses through the olivine through the colder feed end to the outside. A short cooling zone prevents oxidation of the calcined olivine.

Laboratory Tube-Calciner Tests

Tests were run in the liminch, 1.5 KW laboratory calcining furnace on samples from Northwest-International's Burnsville operation. The laboratory tube furnace is shown in a photo in Figure 2. Results obtained when calcining No. 120 olivine sand are presented in Tables 4 and 5. In each test series the ignition loss was lowered to well below 0.1 percent, and the color stayed white-gray.

Calculated Heat Requirements to Calcine Olivine

Specific heat of olivine = 0.26 BTU/lb/° F

 1500° F X 0.26 = 390 BTU/lb olivine

 $390 \times 2000 \text{ lbs} = 780,000 \text{ BTU/ton of olivine}$

Assumed 35% heat loss, total BTU/ton of olivine = 1,200,000 BTU

If No. 2 oil is used - at 140,000 BTU/gal and cost 15¢/gal,

fuel consumption = 8.5 gal/ton of olivine; cost = 15¢ X 8.5 = \$1.28/ton of olivine.

If electric power is used - at 3412 BTU/KW and 1¢/KW Hr, power consumption = 350 KW/ton of olivine; cost = 1¢ X 350 = \$3.50/ton of olivine.

Continuous In-Plant Test Work

Northwest-International olivine company showed great interest in calcining of their olivine. Especially the possibility of calcining without color change was attractive to Northwest-International. To expedite progress and to obtain larger samples for customer evaluation, it was decided to install a tube calciner into the fire box of the rotary dryer at Northwest-International's Burnsville plant. Calculation had shown that the burner in the fire box could deliver the extra BTU's necessary to calcine two tons per hour of olivine, while still drying at rated capacity. Temperature measurements in the fire box showed up to 2600° F. After some experimentation, a $1\frac{1}{2}$ -inch I.D. Inconel tube, with feed and discharge arrangements as illustrated in Figure 3, was installed and operated as calciner. At a discharge rate of 50 pounds per hour, and discharge temperature of 1500° F, the ignition loss of No. 120 olivine could be lowered from 0.8 percent ignition loss to 0.07 percent without changing to a brown color. The calcine had a gray-white color. The results are presented in Table 6. Because of high heat concentrations, especially when the tube was installed through the center of the round fire box, even the Inconel melted. Ceramic pipes of Morganite mullite were tried but broke because of uncontrollable heat application caused by the inherent properties of the burner which could only be fired on and off.

Calcining in Container and Open Air

To test the principle of calcining of olivine without oxidation, samples of No. 120 olivine were calcined in an electric Glo-Bar furnace at 1300° F, 1500° F and 1700° F, exposed to the atmosphere and in a container with vent for water vapors. The samples were kept at the temperatures for two hours. The samples were visually inspected,

ignition losses were determined in inert atmosphere, and ferrous and ferric iron amounts were determined. The results are presented in Table 7.

The samples in the container stayed gray-white and had low ignition loss, and had less ferric iron than the samples exposed to the furnace atmosphere. The samples exposed turned brown and dark brown, had low ignition loss and contained much more ferric iron than the samples calcined in the container.

Future Work Planned

Laboratory work is planned to determine the parameters of an internally-heated tube furnace where the heating element is in the center of the tube, surrounded by olivine. It would be advisable to test parameters for an externally oil-fired furnace tube so as to be able to design a commercial furnace.

Acknowledgements

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Table l

Fluosolid Bed Calciner Data

Test No.: 8 to 13 Date: 12/15/70

Objective: Calcine No. 70 olivine in a fluid bed furnace

Material to be Calcined: No. 70 olivine sand

Ignition Loss 0.47%

Calcining Temperature: 1500° F

Calcining Time: 5 minutes

Timer Reading: A (Start Cold) - hrs.

B (At Temperature) 15 1500° F 10:00 hrs.

C (At End) 1500° F 10:05 hrs.

Calcining Time C - B: 1500° F 5 min.

Material Weight: D (At Start) 250 grams

E (After Calcining) 249 grams

Weight Loss D - E: 1 gram

 $100 - (E / D \times 100) 0.4$ %

Air Flow Rate: Room Temperature - CFH

At 1500° F Temp. 150 CFH

Remarks: Test 8-13 combined for 2 lb. sample. Ignition loss of

combined calcine 0.03%

Engineer: Immo H. Redeker Technician: John Lawrence

Table 2

Fluosolid Bed Calciner Data

Test No.: 7 and 14 Date: 12/15/70

Objective: Calcine No. 120 olivine in a fluid bed furnace

Material to be Calcined: No. 120 olivine sand

Ignition Loss 0.60%

Calcining Temperature: 1500° F

Calcining Time: 5 minutes

Timer Reading: A (Start Cold) - -

B (At Temperature) 1500° F 9:40 hrs.

C (At End) 1500° F 9:45 hrs.

Calcining Time C - B: 1500° F 5 min.

Material Weight: D (At Start) 250 grams

E (After Calcining) 241 grams

Weight Loss D - E: 9 grams

100 - (E / D x 100) 3.6%

Air Flow Rate: Room Temperature - CFH

At 1500° F Temp. 200 CFH

Remarks: Test 7 and 14 combined. Ignition loss of combined

calcine 0.02%

Engineer: Immo H. Redeker Technician: John Lawrence

Table 3

Fluosolid Bed Calciner Data

Test No.: 1 to 6 Date: 12/14/70

Objective: Calcine spiral feed in a fluid bed furnace

Material to be Calcined: Spiral feed

Ignition Loss 1.80%

Calcining Temperature: 1500° F

Calcining Time: 5 minutes

Timer Reading: A (Start Cold) - hrs.

B (At Temperature) 1500° F 9:26 hrs.

C (At End) 1500° F 9:31 hrs.

Calcining Time C - B: 1500° F 5 min.

Material Weight: D (At Start) 250 grams

E (After Calcining) - grams

Weight Loss D - E: - grams

100 - (E / D x 100) - %

Air Flow Rate: Room Temperature - CFH

At 1500° F Temp. 150 CFH

Remarks: Test 1 to 6 combined for 2 lb. sample. Ignition loss of combined calcine 0.09%

Engineer: Imm o H. Redeker Technician: John Lawrence

Table 4

Olivine Calcining in Tube Furnace

Date: 12/16/70

Cont. Run No.: 1

Material: No. 120 olivine

Tube: $1\frac{1}{2}$ -inch I.D., electrically heated to 1500° F

Discharge Hole: 3/32-inch

Objective: Calcine olivine continuously

Hrs:Min.

Time	Temp.(°F)	Gr./Min.	Sa. No.	Ign.Loss		Rema	rks		
0:55	1500	45	1-4-21	0.02	Color	stayed	white	-gray	
0:65	1500	45	1-4-22	0.02	ři.	11	Ħ	11	
0:75	1500	45	1-4-22	0.02	11	11	17	H	
Bulk Sampl	e No. 1		1-4-21	0.02	11	Ħ	n	н	
Bulk Sampl	e No. 2		1-4-22	0.02	13	11	11	\$1	
Head Feed				0.60	Grëen-	-gray	•		

Table 5
Olivine Calcining in Tube Furnace

Date: 1/14/71

Cont. Run No.: 4

Material: No. 120 olivine

Tube: l_{1}^{1} -inch I.D., electrically heated to 1500° F

Discharge Hole: 3/32-inch

Objective: Calcine olivine continuously

Hrs:Min.								
Time	Temp. (°F)	Gr./Min.	Sa. No.	Ign. Loss		Remark	s	
0:00	80	33	_	-		_		
0:15	1060	32	-	_		_		
0:30	1480	37	1-14-21	0:00	Calcined	Oliv	white	-arau
0:45	1	43			0420204	0110.	MIITCE	:-gray
1:00	meter	_	1-14-22	0.08	11	11	11	н
1:15	trouble	_	-	_	ti	11	11	11
1:30	↓	_	_	_	91	11	11	
1:45	1500	42	1-14-23	0.08	Ħ	11	• • • • • • • • • • • • • • • • • • • •	11
2:00	1500	47	-	_	tt	11	11	11
2:15	1500	45	1-14-24	0.08	11	11	11	11
2:30	-	_	_	=	u	11	11	**
2:45	1500	45	1-14-25	0.05	н	11	•1	11
3:00	1500	46	_	_	11	**	11	11
3:15	1500	47	1-14-26	0.11	11	ti	**	ti .
3:30	1500	48	. —	_				
3:45	1500	45	1-14-27	0.06	**	11	**	11
4:00	1500	45	-	_				
4:15	1500	50	1-14-28	0.12	11	Ħ	11	II
4:30	1500	52	_	_	11 -	11	11	11
4:45	1500	50	1-14-29	0.05	PI	11	11	tt
5:00	1 500	49	-	-	Ħ	11	16	п
5:15	1500	53	1-14-30	0.06	**	## .	11	11
Average		46.5		0.07	11	**	H	tt
Average		6.15 1	.b/hr.	0.07	11	11	н	If
Head Feed				0.60	Raw olivi	.ne, g	ree n-g	ıray

Table 6

Calcining at Northwest-International, Burnsville, N. C.

Test No.: 11 Date: 2/16/71

Feed: No. 120 olivine

Ignition Loss 0.8%

Pipe Size: 14-inch I.D.

Material: Inconel 600

Length of Pipe in Between Refractory: 33-inches

Burner: Iron Fireman - 11 GPH #2 Fuel Oil

Discharge: Vibrated box, 17/64-inch hole pinched

Feed Pipe: 8-inch from top

	Disc	charge		Sample	
Time	Hole	lb/hr	Temp.(OF)	No.	Ign. Loss
8:00	-	÷	-	-	-
10:14	-	47	1500-1600	112-16-1	0.08
10:54	-	47	1500-1550	1½-16-2	0.07
11:00	-	47	-	-	-
11:59	-	-	1500	-	-
1:18	-	-	-	-	-
1:24	-	43	1450	1½-16-3	0.09
2:31	-	-	-	-	-
2:37	-	41	1400	1 ¹ 2 - 16-4	0.09

Table 7

No. 120 Olivine Calcined in Open Pan and Container

		Open Pan	Container
<u>As Is</u>	Ign. Loss	0.60	0.60
	Color	Gray-Green	Gray-Green
	Fe0	6.4	
	Fe ₂ 0 ₃ -	0.5	
		•	
1300° F	Ign. Loss	0.20	0.17
	Color	Brown	Gray-White
	Fe0	5.9	6.3
	Fe ₂ 0 ₃	1.0	0.8
1500° F	Ign. Loss	0.11	0.10
	Color	Brown	Gray-White
	Fe0	4.1	6.4
•	Fe ₂ 0 ₃	3.4	0.4
1700° F	Ign. Loss	0.10	0.08
	Color	Dark Brown	Gray-White
	Fe0	5.1	6.0
	Fe ₂ 0 ₃	2.0	1.0

TUBE CALCINER FOR OLIVINE

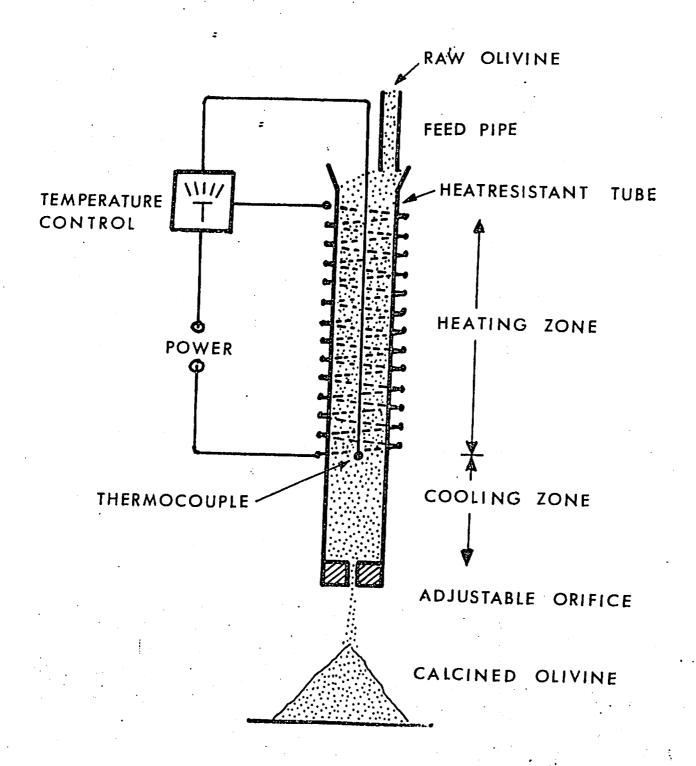
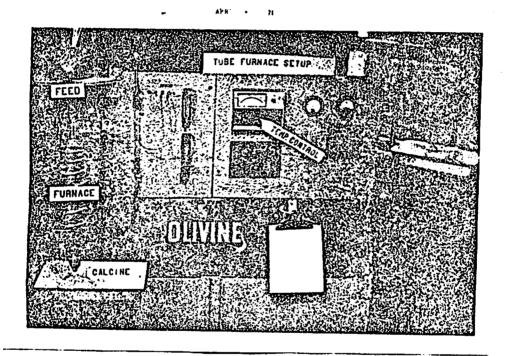
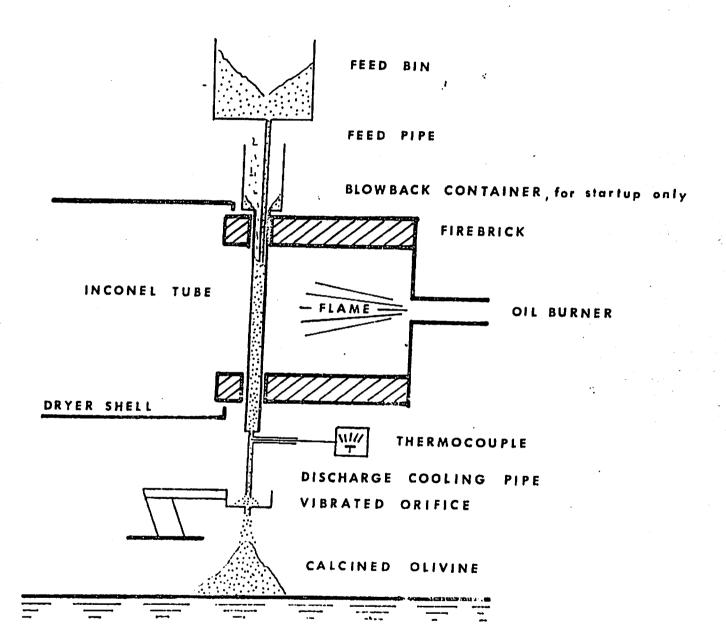


Figure 2

Tube Furnace Setup



TUBE CALCINER IN DRYER FIREBOX



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