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**EVALUATION OF REFRACTORY MATERIALS FOR A
NUCLEAR WASTE INCINERATOR**

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PMRD-78-002

**MATERIALS TECHNOLOGY R&D
PHYSICAL METALLURGY**

Rockwell International

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Rocky Flats Plant
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**U. S. DEPARTMENT OF ENERGY
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MATERIALS TECHNOLOGY R&D
PHYSICAL METALLURGY

SUBJECT DESCRIPTORS

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ROCKWELL INTERNATIONAL
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EVALUATION OF REFRACTORY MATERIALS FOR A NUCLEAR WASTE INCINERATOR

V. K. Grotzky, P. A. Kneale, and A. R. Teter

ABSTRACT

An experiment to find a suitable refractory lining for a nuclear waste incinerator has been completed. Eleven brick and six castable products were analyzed by optical and scanning microscopy. All the materials were fashioned into cup shapes and subjected to temperatures ranging from 800 °C to 1200 °C for as long as six weeks. Some of the cups were charged weekly with pellets made from ash materials that would contact an incinerator liner.

Refractory products containing a high percentage of aluminum oxide had the greatest resistance to cracking and slag buildup.

INTRODUCTION

Seventeen materials were subjected to temperatures ranging between 800 and 1200 °C for various periods of time to determine their suitability as potential incinerator liner material. One hundred seventy small cups (approximately 2.5-cm square) were fabricated from the materials selected for the test. A three gram pellet, made from representative ash materials that an incinerator liner would come in contact with, was used to charge the cups (see Table 1). The ash for the refractory slag test was obtained by burning 270 kilograms of simulated non-radioactive waste. The resultant ash was 70% carbon and 30% oxides. Chemical analysis of the oxide ash composition is shown in Table 2.

The purpose of the evaluation was to determine the resistance of the refractories to slag penetration and buildup and to see if the slag was reacting with the cups and changing the structure. Also, the effect of thermal shock was to be observed on air cooled cups. Some thermal shock tests were made using 5-cm X 2.5-cm X 2.5-cm bars

TABLE 1. Incinerator Waste Feed Composition

Material	Weight Percent
Paper	39.99
Polyvinyl Chloride	7.91
Polyethylene	22.76
Rubber (neoprene)	18.81
Kerosene	9.47
Tributyl Phosphate	1.06
	<u>100.00</u>

TABLE 2. Chemical Analysis of Simulated Slag Composition (ASH)

Oxide	Percent
Al ₂ O ₃	25.19
SiO ₂	32.30
Na ₂ O	1.81
P ₂ O ₅	7.80
TiO ₂	11.50
PbO	0.52
SnO ₂	0.04
Fe ₂ O ₃	3.70
MgO	5.17
CaO	2.51
Cr ₂ O ₃	0.86
CdO	2.32
ZnO	<u>6.42</u>
	<u>100.00</u>

of the refractory brick which were rapidly quenched in water. The results of those tests are reported separately.¹

TABLE 3. Sample Designation and Composition

Sample Designation	Description or Trade Name	Bricks
		Primary Composition
7A	Mullite XD	71% Al_2O_3 , 24% SiO_2
8A	Corulite	87% Al_2O_3 , 9% SiO_2
9A	Korundal XD	88% Al_2O_3 , 11% SiO_2
10A	HW Corundum	99% Al_2O_3
M	HW Magnesite	93% MgO , 4% SiO_2
C	Chromex B	33% MgO , 28% Cr_2O_3 , 19% Al_2O_3 , 14% Fe_2O_3
Z	HW Zircon	66% ZrO_2 , 32% SiO_2
SH	Harbide	89% SiC , 7% SiO_2
SN	Norton	90% SiC , 8% SiO_2
SON	Silicon Oxynitride	90% Si_3ON_2 , 7% SiO_2
SD	Super Duty Fire Clay	48% Al_2O_3 , 48% SiO_2
		Castables
C6A	High Alumina Castable	62% Al_2O_3 , 30% SiO_2
C7A	Alusa Castable	73% Al_2O_3 , 21% SiO_2
C8A	HW Korundal Plastic	86% Al_2O_3 , 9% SiO_2
C9A	Castalast G	94% Al_2O_3 , 5% CaO
CM	HW Perimix	90% MgO , 6% SiO_2
CC	HW Chrome	28% Cr_2O_3 , 30% Al_2O_3 , 16% MgO , 13% Fe_2O_3

PROCEDURES AND RESULTS

The cups were divided into two types, bricks and castables as listed in Table 3. One group of specimens was exposed to a temperature of 1200 °C for six weeks (1000 hours) with a new charge of slag added to the cups each week. Samples were withdrawn at the end of each week, cut in half and observed optically for penetration and deterioration. Along with the above run, one set of cups was charged only once and left in the furnace for the entire six weeks. Color photographs of the results from the first group are shown in Figures 1 through 17.* The small group pictures at the top of each figure sheet shows at a glance the progressive deterioration of the cups as a function of

time and additional charges. The enlarged individual pictures below show the reaction zones in greater detail. The colors are not true but do bear some similarity to the actual colors of the material used in this experiment.

The remaining three groups of specimens were treated as follows:

17 cups charged once and left in the furnace for 96 hours at 800 °C.

17 cups charged once and left in the furnace for 96 hours at 1000 °C.

17 cups charged once and left in the furnace for 96 hours at 1200 °C.

The results of those tests are shown in Figures 18 through 34.

* All original photographs in this RFP are in color and on file in the library at Rocky Flats. Reproductions in this RFP are in black and white.

DISCUSSION

As each week was completed, during the 1200 °C six week test, a set of cups was withdrawn, sectioned, and carefully examined to determine the amount of slag buildup and/or the amount of penetration that had occurred. They were inspected for cracks and any indication of crumbling as well. Observations from that examination are shown in Table 4.

It was felt that those specimens that were left in the furnace for the full six weeks at 1200 °C had been subjected to the most severe conditions possible for incinerator liner material for a given time. Consequently, they were analyzed by Scanning Electron Microscopy (SEM) utilizing energy dispersive X-ray as well as by optical metallography. A summary of the metallographic observations is shown as Table 5.

While it was possible, by SEM, to determine the elements present in the reaction zone between the slag and the cup, it was not possible to tell what elements from the slag were penetrating the cups. Many of the same constituents of the cups were also present in the slag. There was definite evidence, however, of grain boundary penetration as well as grain replacement in many of the specimens along the slag to cup interface. This would indicate that some elements in the slag are reacting with the cups and forming a detrimental compound. A typical example of slag constituents penetrating into the matrix, as observed on the metallograph, is shown in Figure 35.

Those specimens that were charged once and placed in the furnace at 800, 1000, and 1200 °C, respectively, and held for 96 hours, showed very little deterioration. Actually there was no melting of the pellets at 800 °C and very little at 1000 °C which accounts for the little to no reaction with the cups.

CONCLUSIONS

Although all the materials showed a deterioration in some areas during the 1200 °C test, it should

be kept in mind that the severity of the test may be greater than that required for normal incinerator operation. It is possible that use of a lower operating temperature will accomplish the decomposition of the waste and prolong the life of the liner. The results of this experiment indicate that satisfactory operating temperatures may need to be no greater than 1000 °C.

It appears, from this analysis, that those cups containing a high percentage of aluminum oxide proved to be the most suitable as potential incinerator liner material. Consequently, recommendations are as follows:

First, #9A (Korundal XD; 88% Al_2O_3 ; Brick). Slow penetration of waste into the slag cup to about 1/8". Slow buildup of slag to approximately 1/8". Excellent resistance to cracking.

Second, #C9A (Castalast G; 94% Al_2O_3 ; Castable). Progressive penetration into the cup to approximately 1/8". Slag buildup to about 1/4" for the entire six-week run. Excellent resistance to cracking.

Third, #10A (HW Corundum; 99% Al_2O_3 ; Brick). Slow penetration until the sixth week. Minimum slag buildup. Good resistance to cracking.

The SH, SN, and SON, which are high silicon carbide and silicon oxynitride, appear to be suitable materials, also. A glaze tends to form on these materials instead of penetrating which may seal the pores in the material and could possibly increase the effective lifetime of the incinerator. Some experiments should be made to see if the glaze of slag could be removed satisfactorily after reaching an undesirable thickness, without damaging the liner.

Several of the specimens that were charged only once showed very little deterioration after being held at 1200 °C for the full six weeks. Consequently, it appears that the thermal shock from

TABLE 4. Weekly Analysis of the Test Cups

Spec Number	Slag Penetration (inches)						Slag Buildup*						Evidence of Cracking						Remarks
	1st Week	2nd Week	3rd Week	4th Week	5th Week	6th Week	1st Week	2nd Week	3rd Week	4th Week	5th Week	6th Week	1st Week	2nd Week	3rd Week	4th Week	5th Week	6th Week	
7A	3/16	1/4	1/4	Full Wall	Full Wall	Full Wall	+	++	+++	+++	+++	++++	None						May be starting to crumble during 4th week. Still good at 6th week.
8A	1/16	1/8	1/8	3/16	Full Wall	Full Wall	+	+	+++	+++	+++	++++	None			→ Crack	Crack	Crack	
9A	1/16	1/16	1/8	3/16	3/16	3/16	+	++	++	+++	+++	++++	None						
10A	3/16	1/4	1/4	Full Wall	Full Top Wall	Full Wall	+	++	++	++	+++	+++	None			→ Crack	Crack	Grain Bdry Cracks	
M	1/32	1/32	1/32	1/32	1/32	1/2 Wall	+	++	+++	+++	++++	+++	Crack	Crack	Crack	Crack	Crack	Crack	Penetration through bottom 4th; sidewalls bad 6th.
C	?	?	?	?	?	?	+	++	+++	++++	++++	+++							The color of this material prevented observation of slag penetration.
	(see remarks)																		
Z	Full Wall	Full Wall	Full Wall	Full Wall	Full Wall	Full Wall	+	++	+++	+++	+++	+++	None						Very rapid penetration.
SH	?	?	?	?	?	?	++	+++	No Samp	++++	+++	+++	None	OK	No Samp	OK			Wets entire outside of cup 1st, 2nd, 4th, and 5th weeks.
	Not possible to see penetration										++	++							
SN	0	0	0	0	0	0	++	+++	+++	++++	++	+++	None						Wets back 1/2 way 1st week Full 2nd week.
SON	0	0	0	0	0	0	++	+++	+++	++++	++++	+++	None						Wets 1/2 way 1st week, 2/3 2nd week, full 3rd week. Slag buildup decreased due to wetting increase.
SD	Full Wall	Full Wall	Full Wall	Full Wall	Full Wall	Full Wall	+	++	+++	+++	++++	++++	None			→ Crack	Crack	Crack	Broke on 3rd week.
C6A	1/8	1/8	3/16	1/4	Full Wall	No Samp	+	+	++	++	++	No Samp	None	OK	Crack	Crack	Crack	No Samp	Cracked in 4th week, also 5th week.
C7A	1/8	Full Wall	Full Wall	Full Wall	Full Wall	Full Wall	+	++	++	+++	+++	++++	Craze Crack	Craze Crack	Craze Crack	Crack	Crack	Crack	Crumbles 2nd week, cracked 4th, 5th, and 6th weeks.
C8A	1/16	1/16	1/8	1/4	No Samp	Full Wall	+	++	+++	++++	No Samp	++	Crack				→ No Samp	Small Crack	Cracks 1st week, 2nd, 3rd, 4th, and 6th weeks.
C9A	1/8	1/8	1/8	1/8	3/16	3/16	++	++	+++	+++	+++	++++	None						Progressive buildup but no cracks. Penetration not severe.
CM	0	1/16	1/16	1/16	1/16	3/16	+	+	+++	+++	+++	++++	Cracks or Crumbles						Crumbles from start.
CC	?	?	?	?	?	?	?	?	?	+++	No Samp	++++	Porous						Porous at start.
	Not possible to see penetration																		

*An increase in pluses indicates an increase in slag buildup.

TABLE 5. Optical Observations at the End of the Six Week Test (1200 °C for 1000 hours)

<u>BRICKS</u>	
Sample	Remarks
7A	Glassy needle structure along the cup to slag interface. An unidentified black phase has penetrated into the grain boundaries. Fairly large grains. The zone of greatest reactivity was at the bottom of the cup. Reaction with the cup at the end of six weeks is severe.
8A	Glassy phase has formed at the interface of the slag and the bottom of the cup (needle-like structure). Black phase at the grain boundaries. Large grains. Reaction with the cup at the end of the six week period is severe.
9A	Area of greatest reaction is at the bottom of the cup. Overall, the reaction with the cup is slight. Mostly a fine grain structure. Some small grains appear to have diffused into the slag.
10A	The slag reacts mildly with the cup. Grains are fairly large and some are separated by a black and a brownish phase. Bottom and side walls were attacked equally.
M	Sidewall areas were examined. Penetration was very slight. A black and rust colored phase appear to be the most damaging.
C	There are mixed large and small grains. The matrix has a webby rust colored binder around black grains. The rust colored structure breaks down at the interface of the slag and the inside bottom of the cup. The degree of reaction is difficult to determine because of the color of the matrix.
Z	Small grain material. No fractures. Mostly a yellowish gold color where the reaction has occurred. Some black speckled areas. The speckles surround the grains but the grains still appear to be tight. The reaction between the slag and cup is severe. The area examined was at the intersection of the side walls and the bottom of the cup.
SH	The cup is made up of medium sized grains. A very slight reaction occurred between the slag and the cup. The reaction is confined to the mottled structure. The dark gray grains are untouched. There are no fractures. The area examined was at the bottom of the cup.
SN	The matrix contains two distinct grains, mottled and dark gray. There was a slight reaction at the interface of the slag and cup. Very similar to SH. No fractures were observed. The bottom of the cup was examined metallographically.
SON	A very small grain material prevails in the matrix. No apparent reaction between the slag and cup. Two small cracks were observed in the bottom of the cup but may have been caused during fabrication. The bottom area of the cup was examined.
SD	Fairly large grains prevail in the matrix. There appears to be a small grain that has dissolved and migrated into the slag.
<u>CASTABLES</u>	
C6A	No sample for the sixth week.
C7A	Large grains are intermixed with a powdery structure. Significant amount of intergranular cracking that has the appearance of shrinkage cracks. Slag reacts with the grains and grain boundaries. The side walls were examined. The reaction is severe.
C8A	Mixed grain structure and some are very large. Penetration of a black phase through approximately half of the wall thickness. The black phase primarily attacks the grain boundaries. The reaction is severe. The side walls were the area examined.
C9A	Fine grains or fragments of grains make up large grains. Some of the grains have been pulled into the slag. The bottom of the cup was examined. The reaction was mild.
CM	No analysis; crumbled specimen.
CC	The matrix is made up of a cobweb-like structure that tends to disappear near the interface of the slag and the cup. Only large dark globs remain which may be clumps of small black grains. Material is very porous. The degree of reaction was not determined because of the color of the matrix.

recharging or the continued addition of fresh charges is the cause for the ultimate breakdown of most of the materials used in the experiment. A gradual addition of wastes to be incinerated may reduce the thermal shock and prolong the life of some of the materials.

REFERENCES

1. "Incinerator Development," Quarterly Report, Chemistry R&D, October 1977. C. Plock, A. Teter, H. Robinson, P. Faccini. Pilot Plant Development, Rocky Flats.

ILLUSTRATIONS

(Figures 1 through 35)

(7A) Mullite XD (Brick)

The First Slag Cup, in the Composite Photograph on the Left, had One Charge and was Left in the Furnace for the Full 6 Weeks. The remaining cups show the effect of adding a new charge each week for 6 weeks.

1st Week

2nd Week

3rd Week

4th Week

5th Week

6th Week

One Charge and Remained in the Furnace for Six Weeks

FIGURE 1. Mullite XD (Brick)

(8A) Corulite (Brick)

1st Week

2nd Week

3rd Week

4th Week

5th Week

6th Week

One Charge and Remained in the Furnace for Six Weeks

FIGURE 2. Corulite (Brick)

(9A) Korundal XD (Brick)

1st Week

2nd Week

3rd Week

4th Week

5th Week

6th Week

One Charge and Remained in the Furnace for Six Weeks

FIGURE 3. Korundal XD (Brick)

(10A) HW Corundum (Brick)

1st Week

2nd Week

3rd Week

4th Week

5th Week

6th Week

One Charge and Remained in the Furnace for Six Weeks

FIGURE 4. Corundum (Brick)

(M) HW Magnesite (Brick)

1st Week

2nd Week

3rd Week

4th Week

5th Week

6th Week

One Charge and Remained in the Furnace for Six Weeks

FIGURE 5. Magnesite (Brick)

(C) Chromex B (Brick)

1st Week

2nd Week

3rd Week

4th Week

5th Week

6th Week

One Charge and Remained in the Furnace for Six Weeks

FIGURE 6. Chromex B (Brick)

(Z) HW Zircon (Brick)

1st Week

2nd Week

3rd Week

4th Week

5th Week

6th Week

One Charge and Remained in the Furnace for Six Weeks

FIGURE 7. Zircon (Brick)

(SH) Harbide (Brick)

Back Side of Cups Showing the Glaze
That Forms on the Entire Surface

1st Week

2nd Week

No Specimen
3rd Week

4th Week

5th Week

6th Week

One Charge and Remained in the Furnace for Six Weeks

FIGURE 8. Harbide (Brick)

(SN) Norton (Brick)

Backside of the Cups Showing the Progress of the Glaze Each Week.

1st Week

2nd Week

3rd Week

4th Week

5th Week

One Charge and Remained in the Furnace for Six Weeks

6th Week

FIGURE 9. Norton (Brick)

(SON) Silicon Oxynitride (Brick)

By the end of the 3rd Week, a Glaze had Covered the Entire Backside of the cup.

1st Week

2nd Week

3rd Week

4th Week

5th Week

One Charge and Remained in the Furnace for Six Weeks

6th Week

FIGURE 10. Silicon Oxynitride (Brick)

(SD) Super Duty Fire Clay (Brick)
There Were 2 Specimens in the 5th Week

1st Week

2nd Week

3rd Week

4th Week

5th Week

6th Week

One Charge and Remained in the Furnace for Six Weeks

FIGURE 11. Super Duty Fire Clay (Brick)

(C6A) High Alumina (Castable)

1st Week

2nd Week

3rd Week

4th Week

5th Week

No Specimen
6th Week

One Charge and Remained in the Furnace for Six Weeks

FIGURE 12. High Alumina (Castable)

(C7A) Alusa (Castable)

1st Week

2nd Week

3rd Week

4th Week

5th Week

6th Week

One Charge and Remained in the Furnace for Six Weeks

FIGURE 13. Alusa (Castable)

(C8A) HW Korundal Plastic (Castable)

1st Week

2nd Week

3rd Week

4th Week

No Specimen
5th Week

6th Week

One Charge and Remained in the Furnace for Six Weeks

FIGURE 14. Korundal Plastic (Castable)

(C9A) Castalast G (Castable)

1st Week

2nd Week

3rd Week

4th Week

5th Week

6th Week

One Charge and Remained in the Furnace for Six Weeks

FIGURE 15. Castalast G (Castable)

(CM) HW Perimix (Castable)

1st Week

2nd Week

3rd Week

4th Week

5th Week

6th Week

One Charge and Remained in the Furnace for Six Weeks

FIGURE 16. Perimix (Castable)

There Were Two Specimens for the 3rd Week and None for the 5th Week

1st Week

2nd Week

3rd Week

4th Week

No Specimen
5th Week

6th Week

One Charge and Remained in the Furnace for Six Weeks

FIGURE 17. Chrome (Castable)

FIGURE 18. AD

7A - Mullite XD (Brick)

7A - 800 °C

7A - 1000 °C

7A - 1200 °C

FIGURE 19. Mullite XD (Brick)

8A - Corulite (Brick)

8A - 800 °C

9A - 1000 °C

8A - 1200 °C

FIGURE 20. Corulite (Brick)

FIGURE 21. Korundal (Brick)

FIGURE 22. Corundum (Brick)

M - HW Magnesite (Brick)

M - 800 °C

M - 1000 °C

M - 1200 °C

FIGURE 23. Magnesite (Brick)

C - Chromex B (Brick)

C - 800 °C

C - 1000 °C

C - 1200 °C

FIGURE 24. Chromex B (Brick)

Z - HW Zircon (Brick)

Z - 800 °C

Z - 1000 °C

Z - 1200 °C

FIGURE 25. Zircon (Brick)

SH - Harbide (Brick)

SH - 800 °C

SH - 1000 °C

SH - 1200 °C

FIGURE 26. Harbide (Brick)

SN - Norton (Brick)

SN - 800 °C

SN - 1000 °C

SN - 1200 °C

FIGURE 27. Norton (Brick)

SON - Silicon Oxynitride (Brick)

SON - 800 °C

SON - 1000 °C

SON - 1200 °C

FIGURE 28. Silicon Oxynitride (Brick)

SD - Super Duty Fire Clay (Brick)

SD - 800 °C

SD - 1000 °C

SD - 1200 °C

FIGURE 29. Super Duty Fire Clay (Brick)

C6A - High Alumina (Castable)

C6A - 800 °C

C6A - 1000 °C

C6A - 1200 °C

FIGURE 30. High Alumina (Castable)

C7A - Alusa (Castable)

C7A - 800 °C

C7A - 1000 °C

C7A - 1200 °C

FIGURE 31. Alusa (Castable)

C8A - HW Korundal Plastic (Castable)

C8A - 800 °C

C8A - 1000 °C

C8A - 1200 °C

FIGURE 32. Korundal Plastic (Castable)

C9A - Castalast G (Castable)

C9A - 800 °C

C9A - 1000 °C

C9A - 1200 °C

FIGURE 33. Castalast G (Castable)

CC - HW Chrome (Castable)

CC - 800 °C

CC - 1000 °C

CC - 1200 °C

FIGURE 34. Chrome (Castable)

50X

FIGURE 35. Specimen Z (HW Zircon - 66% ZrO_2 ; 32% SiO_2) Polarized light optical micrograph showing a dark and speckled phase of slag penetration in and around the normal matrix grains.