A comparative study of the American Society for Testing Materials spall test and the Federal Specifications Spall Test

Justus Jordan Beinlich

A COMPARATIVE STUDY OF THE AMERICAN SOCIETY FOR TESTING MATERIALS SPALL TEST AND THE FEDERAL SPECIFICATIONS SPALL TEST.

BY

JUSTUS JORDAN BEINLICH.

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THESIS

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Approved by.......................

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The author also wishes to thank the following manufacturers who so kindly donated the firebrick used in this study.

A.P. Green Fire Brick Company

Laclede-Christie Clay Products Company

General Refractories Company

Harbison-Walker Refractories Company

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ILLUSTRATIONS

A COMPARATIVE STUDY OF THE AMERICAN SOCIETY FOR TESTING MATERIALS SPALL TEST[1] AND THE FEDERAL SPECIFICATIONS SPALL TEST[2].

INTRODUCTION

Spalling is one of the two main causes of failure of fire clay refractories in industrial furnaces, while erosion by slags is the other main cause of failure.

Spalling, in the case of refractories, is defined[3] as the breaking or crushing of the unit due to thermal, mechanical, or structural causes, presenting newly exposedsurfaces of the residual mass.

A number of spall tests have been developed by various investigators, but most of these tests have not been universally accepted because they do not simulate service conditions and do not give uniform results.

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1. Present American Society for Testing Materials Tenative Standard; Serial Designation C 38 - 31 T.
2. Present Quenching Test as given in Federal Specifications for Fire-Clay Brick; HH-B-671a; Oct. 18, 1932.
3. Standard Definition of Spalling, American Society for Testing Materials Designation: C 71 - 31.

Although mechanical and structural spalling undoubtedly play an important part in the life of a refractory, most of the tests attempt to classify the specimen only in respect to resistance to thermal shocks.

The first spalling tests were made by alternately heating the specimen in a furnace and cooling them in air; later, water was used as a cooling medium, and in addition, water vapor in an air blast was also tried at one time[1].

Undoubtedly, each of the various types of tests has its merits, but at the present time the water dip spall tests seem to be preferred by both the Federal Specifications Board and the American Society for Testing Materials. This type of spall test may not simulate actual service conditions as closely as the Mellon Institute Spall Test[1], never-the-less, it is more adaptable to control since it is more rapid.

Since the Federal Specifications Board and the American Society for Testing Materials Tests are so widely used it was thought that it might be well to

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1. Stuart M. Phelps, S.M. Swain, and R.F. Ferguson, "A Service Spalling Test for Refractories", Jour. Amer. Ceram. Soc., 14 (5), 389-402 (1931)

make a comparative study of the two tests in order, if possible, to determine the advantages and eccentricities of each.

A difference of opinion exists between several investigators[1] on the mathematical concepts and physics of crack formation dealing with spalling. F.H. Norton contends that spalling in fire brick is due to shear and tension stresses set up in the brick during heating and cooling. In addition, he has developed a mathematical equation for the calculation of the stresses. F.W. Preston contends that all spall fractures are tensile in origin and that Norton's theory is in error.

PURPOSE OF INVESTIGATION.

Commercial brands of fireclay brick will be tested with both the American Society for Testing Materials and the Federal Specifications Spall Tests in order to determine whether the two tests are consistent in the indication of resistance to spalling.

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1. F.W. Preston, "A Theory of Spalling", Jour. Amer. Ceram. Soc., 16 (3), 131-133 (1933).

It is suggested[1] that the properties of a refractory affecting thermal spalling are reversible thermal expansion, elasticity, heat transfer, plastic flow, and strength.

It is hoped that it will be possible to explain the discrepancies that may arise in the results of this work in terms of the above physical properties or else find a reasonable agreement between the two tests.

OUTLINE OF PROCEDURE

In order to make a comparison of the two tests it was attempted to keep all factors as nearly as possible alike and still be in accordance with the procedures set down in the tests.

The Federal Specifications quenching test calls for a twenty-five hour reheat on the test specimen before the regular quenching test is conducted. No reheat is called for in the American Society for Testing Materials Test. Five bricks of each brand to be tested will be subjected to the reheat test as specified and

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1. Stuart M. Phelps, S.M. Swain, and R.F. Ferguson, "A Service Spalling Test for Refractories," Jour. Amer. Ceram. Soc., 14 (5), 389-402 (1931).

the reheated ends of the specimen will be examined and measured both before and after the reheating in order to determine whether any visible structural changes have taken place. The brick will then be subjected to the regular procedure as outlined in HH-B-671a under article F-2c (1).

Five standard brick of each brand as used for the above procedure will be subjected to the spall test as given in the American Society for Testing Materials Tenative Standard C38-31T.

The results of the brands on the two tests will be compared and, if possible, the differences will be explained.

MATERIALS

Ten standard 9 inch straights of each of four commercial brands of fire brick were used in the tests. The companies furnishing these brick and the brand supplied by each company are set forth in Table I. The designations given each brand in this study are set forth in Table II.

TABLE I.

Manufacturer	Brand
A.P. Green Fire Brick Company	Empire (Dry Press)
Laclede-Christie Clay Products Company	King
General Refractories Company	Acme Dry Press
Harbison-Walker Refractories Company	Walsh XX

TABLE II.

Brand	Serial Designation and Specimen Number			
	Fed. Specif. Test		A.S.T.M. Test.	
Empire Dry Press	A	1 thru 5	B	21 thru 25
King	C	6 thru 10	D	26 thru 30
Acme Dry Press	E	11 thru 15	F	31 thru 35
Walsh XX	G	16 thru 20	H	36 thru 40

METHOD OF PROCEDURE

Five of the ten brick of each brand which were sent for the test were chosen at random and the dimensions of the end to be reheated accurately measured and recorded. The twenty brick were then distributed in the door of an oil fired furnace in such a manner that the average conditions of the five brick of any one brand were the same as those of any other brand.

The reheating was then carried on as specified in the procedure given in the Federal Specification for Fire-Clay Brick, which is as follows:

F-2c. Quenching test.- The quenching test shall be conducted on standard 9-inch brick. These brick shall be placed without bond in the door of a furnace so that one end is exposed to atmospheric temperature and the other is flush with the inner wall of the furnace. The furnace is then brought to 1400^{o} C. (2552^{o} F.) at a uniform rate in approximately 20 hours and maintained for five hours at that temperature, which is measured by thermocouples flush with the inner face of the test brick. The brick should not be removed until they have cooled to atmospheric temperature without induced draft.

After the reheated brick were removed from the door of the furnace they were again examined and measured. It was found that the measurements were not changed and the only difference visible was the development of hair line cracks on the reheated surfaces of the brick.

Two sets of five brick each were subjected to the spall test during the same run.

The procedure followed was that as given in Federal Specifications for Fire-Clay Brick, which is as follows:

F-2c(1). The quenching is conducted in the following manner: The reheated end of the brick is placed in the door of a suitable furnace which is being held at a temperature of 850° C. (1562° F.) as indicated by a thermocouple (s) placed close to and flush with the inner end face of the test brick. The one end of the brick should be flush with the inner face of the furnace and the outer end should be exposed to the free circulation of the air.

F-2c(2). At hourly intervals the heated end of the brick is immersed to a depth of 2 inches in running cold water for three minutes. The brick is then removed, allowed to steam in the air for five minutes, and returned to the furnace door. This cycle is repeated until the specimen has failed or has complied with the requirements.

F-2c(3). The brick is considered to have failed when the entire plain surface of the heated endhas completely spalled away. Pieces adhering to the test end shall be removed when the structure has become so weakened that it will not support the weight of the brick when held either vertically or horizontally by the weakened portion.

F-2c(4). The results of any one brand shall be reported as the average of five specimens.

The brick which were reserved for the American Society for Testing Materials Test were also run in tests composed of five brick of each of two brands and procedure as outlined in the present American Society for Testing Materials Tenative Standard: C 38 - 31 T, which is as follows:

1. Object. This method of control test is intended for determining the comparative resistance of high-duty fire-clay brick to spalling action by subjecting them to repeated rapid temperature changes. It is not to be used as a suitability test, as experience has shown that results obtained in this control test are

not comparable with results obtained in service on the same brand of brick. This test is of value, however, as a control test in maintaining the uniformity of fire-clay brick in so far as thermal spalling is concerned.

2. (a) Preparation of Sample. The sample shall consist of at least five standard 9-inch brick or of five shapes, which may be cut to standard size with a grinding wheel.

(b) Test specimens of shapes such as boiler arch brick shall be cut to the standard 9-inch size. For this purpose a "cut-off" wheel is recommended.

3. (a) Procedure. The specimen shall be weighed and placed in the door of a furnace operated at 1350° C. (2462° F.) care being taken to prevent the direct heating of more than the $2\frac{1}{2}$ by $4\frac{1}{2}$-inch end. After one hour the specimen shall be withdrawn from the furnace and stood on end in a tank of flowing cold water from 10 to 21° C. (50 to 70° F.) so that the hot end of the brick will be immersed to a depth of 2-inches. After three minutes the sample shall be withdrawn from the water, allowed to steam five minutes and then returned to the furnace. The door of the furnace shall be closed while the bricks are out to prevent the lowering of the

temperature. The alternate heating and cooling shall be continued in hourly cycles until a loss of 20 per cent by weight has occured in each brick.

(b) When pieces begin to fall off, the sample shall shall be laid on a 4.5 by 9.0-inch asbestos board, divided into 50 equal squares, and the approximate percentage of loss estimated after each removal from the water. After the completion of the test, the brick shall again be weighed and the percentage of loss determined.

4. The report shall show for each brick the number of dips before loss started, the percentage of loss after each cooling and the total number of dips until a loss of 20 per cent by weight has occurred.

The loss in weight after each dip was determined by weighing on a torsion balance and recording as weight remaining in grams instead of estimating the loss by means of an asbestos board marked off as described in the procedure.

The brick were photographed in order to show the degree to which they were spalled. (Figure 1 thru figure 8).

PRESENTATION OF DATA

The measurements taken on the brick before and after reheating are given in table III.

No measurable change in size was found on the reheated end of the brick, and the only visible effects of the reheat was the development of hair line cracks on the reheated surface of each specimen.

The apparent stability of the specimen which had been reheated is attributed to hard burning in the original firing and to a dense structure.

Tables IV thru XI give the per cent loss after each dip for the quenching test.

Table XII is a summary of Tables IV thru XI.

TABLE III

Series	Specimen Number	Dimensions before Reheating	Dimensions after Reheating
A	1	2 7/16 x 4 7/16	2 7/16 x 4 7/16
	2	2 7/16 x 4 7/16	2 7/16 x 4 7/16
	3	2 7/16 x 4 7/16	2 7/16 x 4 7/16
	4	2 7/16 x 4 7/16	2 7/16 x 4 7/16
	5	2 7/16 x 4 3/8	2 7/16 x 4 3/8
C	6	2 7/16 x 4 7/16	2 7/16 x 4 7/16
	7	2 7/16 x 4 3/8	2 7/16 x 4 3/8
	8	2 7/16 x 4 3/8	2 7/16 x 4 3/8
	9	2 7/16 x 4 3/8	2 7/16 x 4 3/8
	10	2 1/2 x 4 7/16	2 1/2 x 4 7/16
E	11	2 7/16 x 4 1/2	2 7/16 x 4 1/2
	12	2 1/2 x 4 1/2	2 1/2 x 4 1/2
	13	2 1/2 x 4 1/2	2 1/2 x 4 1/2
	14	2 1/2 x 4 1/2	2 1/2 x 4 1/2
	15	2 7/16 x 4 1/2	2 7/16 x 4 1/2
G	16	2 7/16 x 4 7/16	2 7/16 x 4 7/16
	17	2 1/2 x 4 1/2	2 1/2 x 4 1/2
	18	2 1/2 x 4 1/2	2 1/2 x 4 1/2
	19	2 1/2 x 4 1/2	2 1/2 x 4 1/2
	20	2 7/16 x 4 1/2	2 7/16 x 4 1/2

TABLE IV SERIES A

Number of Dips	Specimen Number 1	2	3	4	5
37	0.1 %	0.2 %	0.2 %	0 %	0 %
45	0.2	0.2	0.2	0	0
70	0.4	0.8	0.6	0.4	0.5

TABLE V SERIES B

Number of Dips	Specimen Number 21	22	23	24	25
2	1.0 %				
4	4.0				
10	9.0			0.3 %	0.15 %
14	9.5	2.4 %	0.5 %	0.5	0.5
28	9.5	2.4	0.5	0.7	0.7
30	16.4 end came off		0.7	0.9	
31	16.8				
35			2.9		
42	17.1	5.7			
46		18.3 end came off			
62			7.0	1.2	1.2
64				4.5	
70	18.6	21.0	7.4	4.7	1.4

TABLE VI SERIES C

Number of Dips	Specimen Number 6	7	8	9	10
58			17.0 % end came off no previous loss		16.8 % end came off no previous loss
67				25.1 % end came off no previous loss	
70	0.8 %	1.1.%			

TABLE VII SERIES D

Number of Dips	Specimen Number 26	27	28	29	30
11	0.9 %			0.2 %	
14		4.4 %	0.7 %		
20		6.1			
28		6.8	1.4	1.0	
30		6.9	2.4	1.3	1.0%
39		7.5	3.3	1.9	1.6
41	26.9 end came off		4.7		

TABLE VII (continued)

Number of Dips	Specimen Number 26	27	28	29	30
42		8.7 %	13.5 %	1.9 %	1.6 %
46		11.3			
48			22.0 end off		2.3
50		11.9		2.5	2.3

TABLE VIII SERIES E

Number of Dips	Specimen Number 11	12	13	14	15
33					3.5 %
36			27.8 % no previous loss		
37		14.3 % end partly off		22.0 % no previous loss	
50	1.2 %	19.0			3.8

TABLE IX SERIES F

Number of Dips	Specimen Number 31	32	33	34	35
11	1.9 %	0.7 %	0.5 %	0.5 %	0.5 %
16	3.0	0.8	0.6	0.5	0.6
20	3.4	0.8	3.9	0.6	0.7
22			6.6	1.6	6.2
24			9.7		
28	5.1	2.4	10.1	1.8	
31	5.1	3.0	11.0	11.4	9.4
34				28.0	
36	6.3	4.9	14.5		9.9
39	9.4	5.5	14.6		9.9
46	11.0	8.5	20.9		16.5
50	11.6	8.9			20.2

TABLE X SERIES G

Number of Dips	Specimen Number 16	17	18	19	20
39				29.8 %	dropped at
40	17.2 %				beginning
41		20.3 %			of test
	no	previous	losses		
50			2.4 %		

TABLE XI SERIES H

Number of Dips	Specimen Number 36	37	38	39	40
16	1.2 %	0.4 %	0.5 %	0.4 %	9.4 %
21	1.2	0.5	4.7	0.6	12.0
22	4.2		14.3		27.8
28	8.4	0.6	15.1		
31	8.5		15.1	0.6	
33			23.8		
39	14.0	0.6		0.8	
40	23.9				
42				8.9	
46		1.0		8.9	
50		2.4		11.9	

TABLE XII

Series	Test	Brand	Specimen Number	Number of Dips	Percent Loss
A	Fed. Spec.	Empire D.P.	1	70	0.4 %
			2	70	0.8
			3	70	0.6
			4	70	0.4
			5	70	0.5
	Average			70	0.54
B	A.S.T.M.	Empire	21	70	18.6
			22	70	21.0
			23	70	7.4
			24	70	4.7
			25	70	1.4
	Average			70	10.6 %
C	Fed. Spec.	King	6	70	0.8
			7	70	1.1
			8	58	17.0
			9	67	25.1
			10	58	16.8
	Average			64.6	12.16

TABLE XII (continued)

Ser-ies	Test	Brand	Specimen Number	Number of Dips	Percent Loss
D	A.S.T.M.	King	26	41	26.9 %
			27	65	24.6
			28	48	22.0
			29	70	13.9
			30	70	12.3
	Average			58.8	19.94
E	Fed.	Acme D.P.	11	50	1.2 %
			12	49	19.0
			13	36	27.8
			14	41	22.0
			15	50	3.8
	Average			45.2	14.76 %
F	A.S.T.M.	Acme D.P.	31	50	11.6
			32	50	8.9
			33	46	20.9
			34	34	28.0
			35	50	20.2
	Average			46	17.92

TABLE XII (continued)

Series	Test	Brand	Specimen Number	Number of Dips	Percent Loss
G	Fed. Spec.	Walsh XX	16	40	17.2 %
			17	41	20.3
			18	50	2.4
			19	39	29.8
			20	Dropped at start of test	
	Average			42.5	17.43 %
H	A.S.T.M.	Walsh XX	36	40	23.9
			37	50	2.4
			38	33	23.8
			39	50	11.9
			40	22	27.8
	Average			39	17.96

Figure 1

Empire D.P. Brick after subjection to the Federal Specificatioms Spall Test.

Figure 2

Empire D.P. Brick after subjection to the A.S.T.M. Spall Test.

Figure 3

Laclede King Brick after subjection to the Federal Specifications Spall Test.

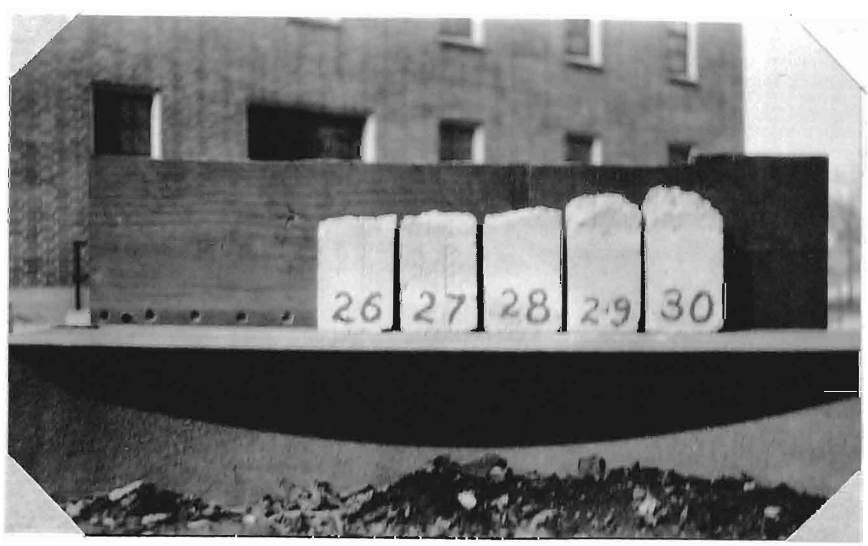

Figure 4

Laclede King Brick after subjection to the A.S.T.M. Spall Test.

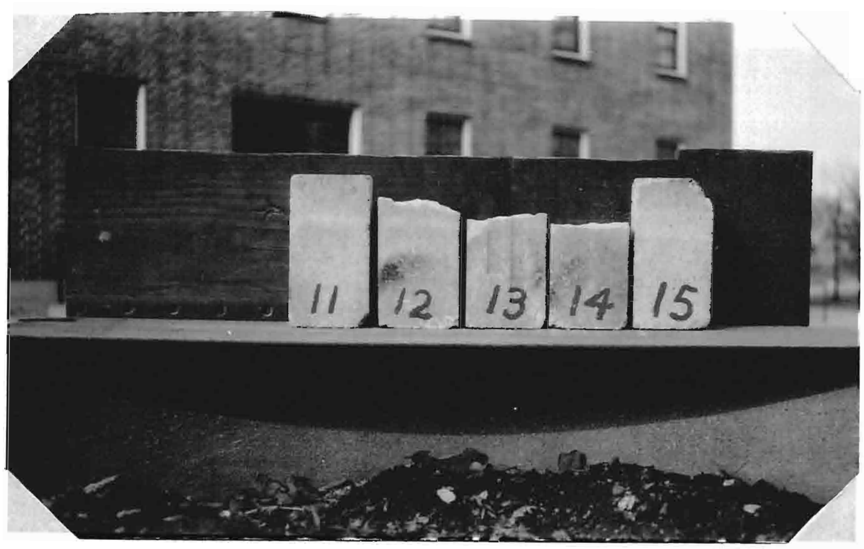

Figure 5

Acme D.P. Brick after subjection to the Federal Specifications Spall Test

Figure 6

Acme D.P. Brick after subjection to the A.S.T.M. Spall Test.

Figure 7

Walsh XX Brick after subjection to the Federal Specifications Spall Test.

Figure 8

Walsh XX Brick after subjection to the A.S.T.M. Spall Test.

DISCUSSION OF RESULTS

At first thought it might be expected that the prolonged reheating of the specimen would reduce the resistance to spalling by developing a denser structure with a more glassy bond.

The above statement, however, does not hold for any of the brands tested, although, it is conceded that one test on five specimen is not sufficient to draw any positive conclusions.

All the brands showed less resistance to spalling when subjected to the American Society for Testing Materials spall test which is conducted at 1350° C. than when subjected to the Federal Specifications Spall Test which is conducted at 850° C. with a previous reheat at 1400° C., as is shown by the averages given in Table XII.

Little can be said as to the uniformity of results on individual samples in any one test. The brick fail over a rather large range of dips, specimen 40 in series H having failed in less than half the number of dips that specimen number 37 withstood with the loss of only 2.4 %. Several of the other series show almost equally poor conformity between specimen, although this fact is more evident in the series subjected to the American Society for Testing Materials Test. (Table XII)

More gradual spalling was noted during the American Society for Testing Materials Tests than during the other, The gradual spalling was a crumbling of the heated end of the brick so that rather small portions were lost at a time and when a large portion became detached it was only after the immersed portion of the brick was crumbling. On the other hand, the brick subjected to the Federal Specifications Tests usually suffered little loss before the entire end became detached due to the development of large cracks. These cracks usually developed near the water line and in a line almost coincident to, or parallel with, the water line. By water line is meant the line separating the immersed portion of the brick from the exposed portion when the brick is dipped into water.

This difference in the type of spalling is probably due to the fact that the brick are less elastic at 850° C. than at 1350° C. so that strains are set up between the cooled portion of the brick and the area immediately above the cooled portion. Accepting the fact that elasticity is greater at 1350° C., it is due to a film of matrix around the unmelted grains. This assumption may then be used to explain the crumbling of the end of the brick by attributing the crumbling to the fact that the matrix forms a glass on cooling. This glass may be shattered by the difference in temperature between the inner portion of the brick and the surface, or, more probably, to the thermal shock which it is subjected to on its return to the furnace.

CONCLUSIONS

No absolute conclusions will be drawn at this time as the study has not included enough brands to verify the probable results.

The indicated conclusions of this study are:

1. The Federal Specifications Test gives more uniform results than the American Society for Testing Materials Test.

2. The reheat, as conducted on a single face of the specimen, develops a condition in the specimen which is similar to that produced in the heating up of refractories in service, and is therefore desirable.

3. The average percent loss is less for an equal number of dips in the Federal Specifications test than

it is in the American Society for Testing Materials Test.

4. The difference in the types of spalling shown by the two tests is concluded to be due to the presence of higher elasticity at 1350° C. than at 850° C.

APPLICATION TO INDUSTRY

It will be advantageous to the manufacturer to use the Federal Specifications Test as a suitability test as the results obtained are more comparable with results obtained in service than are results obtained from the other test.

When a comparison of the resistance to thermal spalling of several brands of firebrick is desired, the American Society for Testing Materials Test is recommended because the reheat test is omitted and the time required to run the test is less than that required for the Federal Specifications Test. This is because the American Society for Testing Materials Test is more severe on the refractories and shows more spalling for a smaller number of dips.

RECOMMENDATIONS FOR FURTHER RESEARCH

For further study of the two tests which were used in this work, it is suggested that the tests include more than the minimum number of specimen called for in the procedures and that the tests be extended over a large number of brands of fire brick.

ABSTRACTS

A Service Spalling Test for Refractories, S.M. Phelps, S.M.Swain, and R.F. Ferguson, Jour. Amer. Ceram. Soc. 14, (5), 389 - 402 (1931)

A spall test furnace which the authors originally used is described along with the spall test procedure. An improved design of the furnace is shown and explained along with a revised procedure. The procedure for a recommended service spalling test is outlined in the procedure.

A Study of the Spalling Test for Fire Clay Brick, C.M. Booze, Proc. A.S.T.M., 26 (1), 277-280 (1926)

A short history of spall tests is given. A discussion of air and water cooling is given. Reheating the specimen from the standpoint of the old method which subjects the entire specimen to the reheat is discussed. Data obtained on brick tested by several modifications of the A.S.T.M. method is given. The most satisfactory results were obtained by a procedure conforming to the present test. There is greater conformity between samples of one lot, more gradual spalling, greater difference between good and poor brick, and better agreement with practical results.