Use of ASTM Standards for Testing Freeze-Thaw Resistance of Fly Ash Bricks

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ABSTRACT

Recently, a new type of brick made from 100% fly ash without firing the bricks in kilns was developed by Freight Pipeline Company. Since the technology is new and the product (fly ash bricks) has not yet been mass-produced, there is no existing standard for this type of brick. However, because the brick is intended for use to replace clay bricks for buildings, it is reasonable to have the brick meet the same existing ASTM standards for clay bricks used for buildings, namely C62 , C67 and C216. While C62 and C216 specify the minimum requirements for clay bricks used for buildings, C67 specifies how clay bricks must be tested to satisfy the requirements.

Problems occur in testing the freeze-thaw resistance of fly ash bricks according to C67 not only because C67 was designed for testing fired clay bricks instead of non-fired fly ash bricks but also due to some arbitrariness in the standard subject to misinterpretation. In this research, the C67 method was modified somewhat to make it more meaningful and less arbitrary for testing fly ash bricks. Experiments were conducted to evaluate the difference in freeze-thaw cycles for fly ash bricks tested according to modified and unmodified methods. Test results showed that the modified test method yields somewhat more meaningful and conservative results than the unmodified method. Due to this, and due to the fact that the modified method is much easier and less time-consuming to use than the unmodified C67 method, the modified method is currently used in research and development for evaluating the freeze-thaw resistance of fly ash bricks. It may also form the foundation or blueprint of a future ASTM Standard for testing fly ash bricks.

1. INTRODUCTION

In the last 4 years (since 2005), a new type of brick, made from Class C fly ash without using any cement or another binder or aggregate, has been developed by the Freight Pipeline Company (FPC) under the sponsorship of the National Science Foundation (NSF) [1, 2][1]. Since the technology is new and has not yet been used commercially, there is no existing standard for this type of brick to meet. However, because the fly ash

[1] Numerals in [ ] represent corresponding items in REFERENCES.

brick is intended for use to replace clay brick for buildings, it is reasonable to expect that the fly ash brick should meet the same existing standards for clay bricks used for buildings, which are ASTM standards C62 [3], C67 [4] and C216 [5].

Note that both ASTM C62 and C216 specify the minimum requirements for clay bricks used for buildings. While C62 is for bricks where their appearance is not a requirement, C216 is for facing bricks of which both appearance and structural properties are specified. Otherwise, both are identical in terms of the requirements on the structural properties of bricks. For instance, both require that for bricks used in severe weather regions (i.e., in places where freezing occurs in winter), the average compressive strength of 5 brick samples must be at least 3,000 psi, and the average freeze-thaw resistance of the bricks must be at least 50 freeze-thaw cycles. Both C62 and C216 only specify the requirements for bricks without showing how the bricks must be tested for various properties to satisfy the requirements. The test methods, on the other hand, are specified in detail in C67. Therefore, C67 deals with test methods used to determine whether bricks meet the requirements of C62 and C216 or not.

2. PROBLEMS WITH ASTM C67

In following ASTM C67 methods to test fly ash bricks, several problems exist. ***The first problem stems from the fact that C67 was designed for testing fired clay bricks instead of non-fired fly ash bricks, which have different characteristics.*** For instance, C67 requires that test samples (half bricks) be first dried in a ventilated oven for 24 hours or more at 230 to 239 ^{o}F before they are to be tested for compressive strength and freeze-thaw resistance. Note that oven drying does not change the properties of fired clay bricks because the bricks were pre-heated to over 2,000 ^{o}F in kilns during manufacturing. Such high temperature kiln firing has vitrified the clay brick into a glass-like material which is inert to heating in the oven. In contrast, fly ash bricks being manufactured either at room temperature or at slightly above room temperature are affected by high temperature heating in oven. For instance, tests conducted in this NSF project showed that the compressive strength of fly ash bricks that were dried in oven according to ASTM C67 is approximately 20% higher than the compressive strength of room-dried fly ash bricks. The freeze-thaw resistance of fly ash bricks, on the other hand, is adversely affected by such oven drying. Therefore, ***it is inappropriate to use the compressive strength and freeze-thaw resistance of oven-dried fly ash bricks because in building construction bricks will never be heated to much above room temperature (except when a building is on fire), and hence will have a significantly different compressive strength and freeze-thaw resistance than given by the test results based on oven-dried fly ash bricks.*** This shows that fly ash bricks should not be heated in oven before tested for compressive strength and freeze-thaw resistance.

A second problem with ASTM C67 is that the freeze-thaw test conducted according to C67 is not only slow and cumbersome but also imprecisely specified in the Standard. As a result of the ambiguity of the Standard, different individuals or laboratories using the same standard to test the same bricks will often yield very different results and outcomes. As stated by Grimm [6], ***"The current standard freeze-thaw test (as specified in C67) is expensive, time-consuming, unrealistic, and incapable of certain identification of nondurable bricks."***

Problems with the freeze-thaw test conducted according to ASTM C67 have prompted ASTM to allow the use of water absorption test results to determine the durability (freeze-thaw resistance) of clay bricks. According to C62 and C216, if bricks in severe weather regions have 5-hour hot water absorption value of less than 17% and have saturation coefficient less than 0.78 (78%) -- based on the average from 5 brick samples -- the bricks are deemed to be freeze-thaw resistant and need not be subjected to the freeze-thaw test. This compromise of ASTM has brought great relief to clay brick manufacturers, distributors and users. As a result, today few if any contractor or manufacturer test each batch of bricks brought to any construction site for freeze thaw. Instead, they run the water absorption tests which are much easier and faster to do. However, research conducted in both United Kingdom and United States has shown poor correlation between the absorption requirements of C62/C216 and the freeze-thaw cycle requirements under the same standards [7]. For instance, Robinson, Holman and Edwards [8] showed that using the water absorption standard of C62/C216 to determine durability would accept as many as 22.8% of bricks that could not pass the required freeze-thaw cycles, whereas 31.5% of the bricks that have passed the required freeze-thaw cycles would not pass the absorption criteria. This shows the low reliability in using the water absorption criteria of C62/C216 to determine the durability (freeze-thaw resistance) of clay bricks.

3. FPC STANDARD METHOD FOR TESTING FLY ASH BRICKS

Due to the lack of any existing ASTM or other standards for testing non-fired fly ash bricks, Freight Pipeline Company (FPC), the company that has developed the new fly ash brick technology under a National Science Foundation (NSF) grant, has developed its own test method for assessing the adequacy of the compressive strength and freeze-thaw resistance of fly ash bricks, to be referred to hereafter as "Modified ASTM C67 Method for Testing Fly Ash Bricks," or simply as the "Modified Method."

3.1. Compressive Strength Test

The Modified Method for testing the compressive strength of fly ash bricks is identical to that of C67 for clay bricks, except that we use room dried bricks instead of oven-dried bricks for the test, for reasons explained before. By room drying, we require that the test samples (half bricks) be left inside a temperature-controlled and well-ventilated room at a temperature between 60 ^{o}F and 90 ^{o}F for at least a week (7 days). Then the samples are tested for compressive strength in the same manner as specified in C67. If the samples have compressive strength higher than 3,000 psi, it is deemed to be adequate for use in severe weather (SW) regions. So, the procedure and criteria are identical to that of ASTM C67 for testing clay bricks, except that drying of the bricks is done differently.

3.2. Freeze-Thaw Resistance Test

The freeze-thaw tests conducted by FPC are different from that of ASTM C67 in that the tests are conducted automatically instead of manually. This not only reduces the arbitrariness in testing and inconsistency of test results done by different persons, but also speeds up the tests. Instead of being able to conduct only one freeze-thaw cycle a day, the automated tests enable 3 to 6 cycles a day depending on the freezer used and the number of bricks being tested in the freezer. What it takes two months to complete a set of freeze-thaw tests conducted according to ASTM C67 can now be completed in two weeks by using the Modified Method. This has greatly streamlined and improved the freeze-thaw tests. However, an important question remains: Is 50 cycles of freeze-thaw conducted using the Modified Method the same as 50 cycles of freeze-thaw conducted using the C67 manual method? The following experiment was conducted in this project to answer this question.

The purpose of this experiment is to compare the **fast** (automatic) freeze-thaw test method normally used by FPC for determining the freeze-thaw resistance of fly ash bricks with the **slow** (manual) freeze-thaw test method described in ASTM C67, to see their differences and relationship. The test was designated as Set 62, which used the high-grade fly ash from Unit-3 boiler of the Thomas Hill Power Plant in Missouri. Note that Unit 3 is a pulverized-coal burner, and the ash produced is very pure – having less than 0.5% of unburned carbon or LOI (Loss-on-Ignition). The fly ash is class C.

A total of fifteen (15) batches of half-bricks were made for the test. ASTM C67 requires half-bricks to be used in both compressive strength and freeze-thaw resistance tests. Each half-brick has dimensions of 4” x 4” x 2.2”. Hereafter, half-bricks will be referred to simply as “bricks”. With 4 bricks in each batch, a total of sixty (60) bricks were made for the test. However, due to some error in making the bricks of batches 7 and 8, the eight bricks made in these two batches were not used for the test. Thus, the total number of bricks tested was fifty-two (52). All the bricks in this set were compacted at 1800 psi pressure, followed by 24-hour steam cure at 160 ^oF, and followed by 18 days of bag cure. No air cure followed before the bricks were tested for freeze-thaw resistance. The test data are summarized in Table 1.

ASTM C67 requires that freezing of bricks be done by standing half-bricks in a tray containing 0.5 inch depth of water. The water and bricks in the tray are allowed to freeze in the tray for 20 hours. Then the tray is manually removed from the freezer and immersed in a bath of water in a thawing tank for 4 hours, with the water temperature being kept between 65 ^oF and 85 ^oF. The Standard requires that ***“…the temperature of the air in the freezing chamber will not exceed 16 ^oF (-9 ^oC) one hour after introducing the maximum charge of units, initially at a temperature not exceeding 90 ^oF (32 ^oC).”*** The phrase ***“not exceed 16 ^oF”*** is troublesome because it allows any temperature below 16 ^oF to be used in the test. As will be shown in the test results to be presented next, whether one uses 15 ^{o}F or – 4 ^{o}F makes a great difference in the test results.

For the automatic fast freeze-thaw test developed and used by FPC, the bricks also stand in a tray of 0.5 inch of water, and the tray is allowed to freeze in the freezer (freeze-thaw chamber) until a thermocouple imbedded inside a sensor brick reads 15 ^{o}F. Then the freezer is automatically turned off by a PLC (programmable logic controller), and an electric heater wrapped around the tray is turned on, heating the tray and melting the ice in the tray until the brick temperature rises to 65 ^{o}F, when the next freezing cycle begins. In doing so, one can complete 3 to 6 cycles of freeze-thaw a day, which is much faster than the manual test of one cycle a day. This in a nutshell explains how the manual C67 slow test and the automatic fast test were performed in this study. The manual test was conducted under two different conditions – (a) severe, and (b) moderate. The "***severe***" condition was conducted at the freezer air temperature of - 4 ^{o}F (minus 4 degree Fahrenheit). On the other hand, the "***moderate***" condition was conducted with the freezer temperature being +15 ^{o}F. While both satisfy ASTM C67, the former (severe condition) freezes the bricks faster and hence generated greater thermal shocks to the bricks than did the latter. It was of interest to see if bricks tested under "severe" condition would fail in fewer freeze-thaw cycles than under "moderate" condition. It was also of interest to see whether the fast automatic test would be more or less severe than the two manual tests -- ***severe*** and ***moderate***.

The test results are given in Table 1; they are explained and analyzed as follows:

(a) Comparison of fast (automatic) test with slow (manual) test under "Severe Condition" (old Thomas Hill Ash – i.e., ash received prior to February 2008):

It can be seen from Table 1 that the 7 bricks (1-1 to 2-3) in the fast automatic test failed in 48 to 74 cycles, with an average of 59.4 cycles. In contrast, the 8 bricks in the slow manual test under "***severe***" condition (bricks 3-1 through 4-4) failed in 24 to 41 cycles, with an average of 32.4 cycles. So, the ratio of failure cycles between the automatic test and the manual test under "***severe***" condition is 59.4 divided by 32.4, which is 1.833. This means that the manual test conducted at 1 cycle per day under "***severe***" condition is significantly more destructive to fly ash bricks than the automatic test, resulting in a failure cycles 1.833 times less than that of the fast automatic test. This is caused mainly by the low air temperature (- 4 ^{o}F) used in the test under "***severe***" condition.

The above test result holds only for fly ash bricks made with air-entrainment agent (AEA). Another set of similar tests was conducted using 8 bricks made the same way as before except without AEA. The bricks are 5-1 through 6-4. As can be seen from Table 1, the 8 bricks in the automatic test failed in 21 to 34 cycles, with an average of 24.6 cycles. In contrast, the 8 bricks in the manual test (9-1 through 10-4) failed in 6 to 11 cycles, average being 8.63 cycles. So, the ratio of failure cycles between the two test methods is 24.6 divided by 8.63, equal to 2.85. This means that for fly ash bricks made without AEA, the bricks tested manually under "***severe"*** condition failed in about three times fewer freeze-thaw cycles than that of bricks tested in the fast automatic test.

Table 1. Freeze-thaw test result for Set No.62

Test Method	Brick No.	AEA	Failure Cycles	Failure Mode	Remark
Batch 1 through 11 used old Thomas Hill Ash Brick 1-1 through 4-4 (i.e., the first 4 batches of bricks) were made with air-entrainment agent.					
Fast Test (Automatic)	1-1	yes	52	Bottom layer lost	
	1-2	yes	52	Bottom front lost	
	1-3	yes	52	“	
	1-4	yes	48	“	
	2-1	yes	74	Bottom back lost	
	2-2	yes	69	Bottom corner lost	
	2-3	yes	69	Bottom back lost	
	2-4	yes			Brick damaged when made
Slow Test (Manual under Severe Cond.)	3-1	yes	27		
	3-2	yes	24		
	3-3	yes	31		
	3-4	yes	30		
	4-1	yes	35		
	4-2	yes	39		
	4-3	yes	41		
	4-4	yes	32		
No AEA was used in making the bricks below (5-1 through 10-4):					
Fast Test (Automatic)	5-1	no	21		
	5-2	no	21		
	5-3	no	22		
	5-4	no	21		
	6-1	no	34		
	6-2	no	26		
	6-3	no	26		
	6-4	no	26		
	7-1 to 7-4	no			Bricks not used for the test
	8-1 to 8-4	no			Bricks not used for the test
Slow test (manual under Severe Cond.)	9-1	no	10		
	9-2	no	11		
	9-3	no	9		

	9-4	no	6		
	10-1	no	8		
	10-2	no	9		
	10-3	no	8		
	10-4	no	8		
AEA was used in making the bricks below (11-1 through 15-4)					
Slow Test (Manual under Moderate Condition)	11-1	yes	45	Cumulative weight loss is 0.7%	
	11-2	yes	49	Cum. wt. loss is 0.52%	
	11-3	yes	55		
	11-4	yes	51	Cum. wt. loss is 0.8%	
Batch 12 through 15 used new Thomas Hill ash (ash received in February 2008); compaction and curing conditions remained the same as those of Batches 1 through 11.					
Fast Test (Automatic)	12-1	yes	70	Mass loss	
	12-2	yes	46	" "	
	13-1	yes	65	" "	
	13-2	yes	41	" "	
	14-1	yes	151		
	14-2	yes	74	Mass loss	
	15-1	yes	65	" "	
	15-2	yes	41	" "	
Slow Test (Manual under Moderate Condition	12-3	yes	75		
	12-4	yes	>100		
	13-3	yes	61	Mass loss	
	13-4	yes	67	" "	
	14-3	yes	57	" "	
	14-4	yes	94		
	15-3	yes	>100		
	15-4	yes	>100		

Note: *"**Severe condition**" of manual (slow test) was conducted with the freezer set at lowest temperature (air temperature approximately -4 ^{o}F). "**Moderate condition**" of the manual (slow test) was conducted with the freezer set at a higher temperature (air temperature approximately +15 ^{o}F). Both conditions are within the purview of ASTM Standard C67.*

(b) Comparison of fast automatic test with slow manual test under "Moderate Condition" (old Thomas Hill Ash – i.e., ash received prior to February 2008):

For the slow manual test under **"*moderate*"** condition, we made 4 bricks (11-1 to 11-4) from the old Thomas Hill ash (ash received prior to February 2008), and another 8 bricks (12-3 to 15-4) from the new Thomas Hill ash (ash received in February 2008). They are separately identified due to the possibility that the fly ash properties might have changed for the ashes received from the same power plant at two different times. All the 12 bricks made completed curing on June 10, 2008 and then were tested for freeze-thaw resistance. As can be shown from Table 1, these 12 bricks passed freeze-thaw cycles ranging from 45 to over 100, with an average of 71.2 cycles. Note that when a brick has passed 100 cycles and still not failed, the test was discontinued and the test result was reported as 100 cycles (>100 cycles). Therefore, the true average cycles of the 12 bricks, had the ones that passed 100 cycles be allowed to continue the test, will certainly be above 71.2 cycles, which is significantly above the minimum cycles required by ASTM C62 and C216. It is of interest to note that the two bricks that failed the 50-cycle F/T test were made of the old Thomas Hill ash. All the 8 bricks that were made of the new Thomas Hill ash passed a minimum of 61 cycles. This shows that the quality of the new Thomas Hill fly ash was better than that of the old ash received earlier.

Separating the old ash data from the new ash data, one sees that for the bricks made from the old ash in the slow manual freeze-thaw test conducted under "***moderate"*** condition (i.e., bricks 11-1 through 11-4), the average freeze-thaw cycles passed by the bricks was 50. In contrast, as reported before when tested under "***severe***" conditions, the bricks made from the same fly ash passed only an average of 32.4 cycles. This means that the freeze-thaw cycles passed by the same bricks tested under "***moderate"*** condition is 1.54 times larger than tested under "***severe"*** condition. This shows that the ***severe*** condition is much more destructive to bricks than the ***moderate*** condition is. Since both "***moderate"*** condition and "**severe**" conditions are allowed under ASTM C67, this shows that testing fly ash bricks under "***severe"*** condition is unnecessary for satisfying the requirements of C67.

Note that what differentiates "***severe"*** condition from "***moderate***" condition is the temperature of the air in the freezer, being -4 ^oF (minus 4 degree F) rather than 15 ^oF . It has been observed during tests that when bricks are frozen to -4 ^oF and then suddenly taken out from the freezer and dipped into 65 ^oF to 85 ^oF water in the thawing tank as required by C67, the ultra-low-temperature ice around the bricks in the tray will suddenly crush into pieces, generating loud crushing noise and tremendous impact force on the bricks surrounded by the ice. This force, due to thermal shocks and ice crushing, has nothing to do with the force that causes freeze-thaw damage to bricks when bricks are tested at any temperature without thermal shocks. Because such large thermal shocks (change of temperature of 80 degrees F within one or two seconds during the thawing test) never happens under natural conditions when bricks are used on buildings, it is neither necessary nor the intent of C67 for bricks to be able to withstand 50 times of such thermal shocks. This shows that when one conducts freeze-thaw tests of any bricks (not just the fly ash bricks) using the slow manual method described in C67, one should not subject the test to such extreme thermal shocks. This study showed that such thermal shocks can be minimized to an insignificant level by testing under the "moderate" condition with freezer air being set at 15 ^{o}F, or by using the fast automatic freeze-thaw test method developed by FPC .

Going back to Table 1, sixteen bricks in batches 12, 13, 14 and 15 were made of new Thomas Hill fly ash -- we ran out of the old Thomas Hill ash in May 2008). Eight (8) of the bricks made (12-1 to 15-2) were subjected to the fast (automatic) test, and the other 8 for the slow manual test for comparison. As can be seen from Table 1, the 8 bricks for the fast automatic test lasted from 41 to 151 cycles, with an average of 69.1 cycles. This appears better than the average of their counterparts made from the old Thomas Hill ash (bricks 1-1 to 2-4), which lasted an average of 59.4 freeze-thaw cycles. This shows that the new Thomas Hill ash received in February 2008 appears somewhat better than the old Thomas Hill ashes used before.

Table 1 shows that for bricks made from the new Thomas Hill ash tested according to C67 under "***moderate"*** conditions (i.e., for the last 8 bricks listed in Table 1), the freeze-thaw cycles passed by the bricks range from 61 to over 100 cycles, with an average of 81.8. As discussed in the last paragraph, when the same kind of bricks made from the new ash was tested in the fast automatic test, they lasted an average of 69.1 cycles. The ratio of the cycles passed in the manual test under moderate condition and that in the automatic test is 81.8 divide by 69.1, which is 1.18. This shows that the fast automatic test of freeze-thaw developed by FPC is slightly more conservative than the slow manual test of C67 under "***moderate***" conditions. Due to this, one should feel safe to evaluate the freeze-thaw resistance of fly ash bricks by using the fast automatic test method rather than the time-consuming, cumbersome and arbitrary manual method given in the current C67 Standard. ASTM needs to consider this both in its future renewal or revision of the existing standard C67 for clay bricks, and in developing a new standard for the fly ash brick to be used in the future.

4. CONCLUSION

Based on the foregoing test results, the following can be concluded:

(1). ASTM C67, which is the accepted national standard for testing bricks and tiles made from clay and shale, can be adapted for testing the freeze-thaw resistance of non-fired fly ash bricks. One major change that must be made to C67 before it is suitable for testing fly ash bricks is that the test samples (half-bricks) should not be oven dried at a temperature higher than the boiling point of water. Otherwise, it would cause significant change of brick properties, which distorts test results. For the freeze-thaw resistance test, no drying of test samples is necessary at all because C67 requires a 4-hour soaking of bricks in water prior to freeze-thaw tests. For the compaction test, drying of the fly ash brick samples can be done either by low-temperature heating in oven (at a temperature significantly below the boiling point of water), or drying at room temperature for at least a week if dried in still air, and for at least 24 hours if fan dried.

(2). The manual freeze-thaw test specified in ASTM C67 is cumbersome and time-consuming to perform. It is also somewhat arbitrary and depends on the experience, care and interpretation of the experimenter. Consequently, oftentimes different experimenters report significantly different test results for the same products. Most of these problems can be solved by using the automatic fast test used by FPC.

(3). The automatic fast test yields slightly more conservative values of freeze-thaw cycles than does the slow manual method of C67. Therefore, if bricks tested based on the automatic test pass the minimum 50 cycles required by ASTM C62 or C216, the same bricks should be able to pass more than 50 cycles in the manual slow test conducted according to C67, provided that care has been taken in adjusting the freezer setting to avoid excessive thermal shocks, and to achieve the "***moderate***" condition described in this paper.

(4). Future ASTM standard should consider changing the manual freeze-thaw test to automatic freeze-thaw test in a manner similar to that developed by FPC and reported herein. All those who manufacture, sell and use bricks, be it clay bricks or fly ash bricks, will benefit from such a change. The change will reduce test duration from two months to two weeks, and will prevent many human errors and arbitrariness associated with the manual method specified in the current C67.

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