A Collection of Papers Presented at the
86th Annual Meeting, and the
1984 Fall Meeting
of the Materials & Equipment and
Whitewares Divisions

Cullen L. Hackler
Chairman, Proceedings Committee

April 29-May 3, 1984
David Lawrence Convention
Center, Pittsburgh, PA

September 26-29, 1984
Bedford Springs Hotel, Bedford, PA

ISSN 0196-6219
Published by
The American Ceramic Society
65 Ceramic Drive
Columbus, OH 43214

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Included in this fifth volume of *Ceramic Engineering and Science Proceedings* are papers from the 1984 Annual Meeting in Pittsburgh and the 1984 Joint Fall Meeting in Bedford Springs. The percentage of papers presented at the meetings, relative to the number published, is improving as many authors are understanding how rapidly these papers are published. It is our sincere hope that many more of our members will present and publish papers so that the exchange of information can be used to benefit our industry as a whole.

Direct contact with the individual authors is encouraged in case of any doubts, misunderstandings, or questions, as there may be some inadvertent inaccuracies or misprints due to our efforts to get this published quickly.

As Chairman of the Proceedings Committee for the Whitewares and Materials & Equipment Divisions, I want to welcome John C. Melman to the committee and thank him for his work in preparing this volume. Additionally, thanks goes to the authors, program chairmen, session chairmen, and others who helped in this publication.

Cullen L. Hackler  
Chairman  
Proceedings Committee  
Whitewares and Materials & Equipment Divisions
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Factors Affecting the Modulus of Rupture of Clay-Based Bodies

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A study was made of the effects of processing, forming, and test parameters on the measured green strength of clay-bonded systems.

Introduction
Since every ceramic forming operation requires some measure of dry strength, one of the most widely used tests is the dry modulus of rupture test (MOR). For this reason, the work was undertaken in order to summarize and review some of the variables that can affect the results of this test. In addition, the practical application of MOR test results to a production situation was kept in mind.

Everyone normally thinks of adding fine-grained clay at the expense of coarse-grained clay to increase MOR. This paper will address some of the other factors that will affect MOR that may be ignored when making body changes or during testing MOR.

Most of this work was done on a typical sanitaryware slip casting body using 30% ball clay, 20% kaolin, 30% feldspar, and 20% flint. A strength testing unit* at the technical service laboratory in Sandersville, GA was used for the majority of this work using a travel rate of 0.5 cm/min (0.2 in./min).

In many cases, the figures presented here were compiled from several different trials and reflect averages rather than actual single data points. Also, most of this work was done within the normal working range of the bodies tested. Extreme cases, such as very high, or very low, levels of sulfate, deflocculant or solids were avoided because they become irrelevant in practical plant applications.

The factors affecting MOR of clay-based bodies were broken into two basic categories: test procedure-related factors, and body-related factors.

Test Related Factors
Throughout this discussion, the term “50% relative humidity strength” is used. This is defined as the strength of bars that were first dried, then held in a desiccator at 50% relative humidity for at least 24 h after they were removed from the oven. It is felt that the results from this test more closely approach the actual strength of ware in a production situation. As will be seen, the results from this test are usually between 50–60% of the actual dry strength.

The 50% relative humidity (RH) test is important for two other reasons. First, some factors which have a significant effect on dry strength may have very little effect on the 50% RH strength of a body. Second, this reduction
in strength from exposure to the atmosphere occurs quite rapidly. Figure 1 shows that a 15% reduction in strength was seen after only one h at 50% RH, which represents 1/3 of the total change that occurred. Over 80% of the total change that occurred was seen after only 4 h, and as can be seen in Fig. 2, no further change was observed after 8 h of exposure to the 50% RH atmosphere. This means that when ware is being handled during finishing, spraying, and kiln loading, it will be significantly weaker than dry ware directly out of the ware dryers. The less time that elapses between the dryer and the last handling operation, before the kiln, the less strength will be lost. Also, this emphasizes the importance of testing the 50% RH strength since it relates to the actual strength of ware in the plant.

From an MOR testing standpoint, this could lead to the conclusion that bars should be broken as soon as they are removed from the dryer. However, another factor that affects MOR is the temperature of the bars. As can be seen in Fig. 3, a reduction in strength of over 10% was seen when breaking hot bars as opposed to bars cooled to room temperature in a dry atmosphere. For this reason, our standard test procedure for MOR calls for cooling the bars in a desiccator maintained at near 0% relative humidity until they have reached room temperature before testing. This procedure eliminates problems from the relationship between test results and strength reduction from temperature and exposure to the atmosphere.

The next test procedure-related variable that was examined was the breaking span which was varied from 2.5-10 cm (1-4 in.), using 1.9 cm (0.75 in.) round bars. Figure 4 shows the resulting drastic increase in MOR in the shorter spans. This difference may be attributed to the fact that as the span approaches the bar diameter, crushing and shearing forces come into play rather than the simple flexural strength measure in the longer spans. Our standard test procedure uses 1.9 cm (0.75 in.) round bars with a 5 cm (2 in.) span.

Other test procedure-related factors were also examined which bear mentioning, although nothing contradicted previous work reported in the literature. The MOR of round bars was found to be about 25% greater than that of rectangular bars.

Also, handling of the bars was found to be quite important. The MOR of 1.9 cm (0.75 in.)-round bars varied over 689 MPa (100 psi) depending on whether the surface was scratched, dry-sponged or left alone.

With respect to casting technique, cast bars that were quick-dried in the mold lost just under 5% of their strength. This was also true of hot slip cast in a cold mold and cold slip cast in a hot mold. However, cold slip in a cold mold and hot slip in a hot mold performed equally well.

Body Related Factors

Of the body related factors, the amount of electrolyte, or position on the deflocculation curve had one of the most significant effects on strength. Figure 5 shows that throughout the entire range tested for this body, the dry strength increased nearly 25% as the amount of sodium silicate was increased. Even within the normal working range for the body, the increase was still 15% or about 550 MPa (80 psi). In Fig. 6, the effect of deflocculation on both dry strength and 50% RH strength within that working range can be seen. The reduction in strength between the 50% RH bars and the dry bars was about the same throughout the entire range, varying between 58-61% of the dry strength. The important point here is that the slope of these two lines is
about the same in this figure. This was not true for all the variables that were measured.

Another body-related factor is the soluble sulfate ion concentration in the slip. Figure 8 shows that a change of over 250 ppm sulfate (dwb) produced a reduction in dry strength of over 10%. This relates to about a 170 MPa (25 psi) reduction for every 100 ppm increase in sulfate. However, the change in strength is much less noticeable when looking at the strength of bars equilibrated at 50% RH. Here, the same increase in sulfate ion concentration produced a reduction in strength of less that 5% or only about 40 MPa (6 psi) for a 100 ppm sulfate change. The fact that the slopes of the lines are different means that the ware out in the plant will not lose much strength because of a sulfate increase, but the dry test would lead you to think that it does.

An interesting point can be made about the effect of organic content on strength and how this relates to the sulfate effect (Fig. 7). In this series of tests, varying levels of predigested organic were added to the test slips when they were prepared. An increase in dry strength of 50% was seen with an increase of 0.3% organic. The same effect was also seen on 50% RH strength. This relationship becomes important in light of the fact that it did not exist with regard to the effect of sulfate on strength. In reviewing Fig. 8, it was seen that although a decrease in dry strength occurred with increasing sulfate levels, the 50% RH strength remained essentially unchanged. Since the casting properties of a sanitaryware body with low organic and low sulfate can be similar to those of a body with higher organic and higher sulfate, the indications here are that at 50% RH, where the strength of a body relates closely to strength in the plant, a body with higher sulfate and higher organic level will likely be stronger than one with lower levels of each. This trend is less obvious when looking at dry strength because the negative effect of increased sulfate tends to cancel the positive effect from increased organic. By not checking 50% RH strength, this real strength increase might be missed.

The last body-related factor examined was specific gravity. Figure 9 shows that an increase in specific gravity from 1.80–1.85 produced an increase in dry strength of 25% and an increase in 50% RH strength of 20%. However, this increase in specific gravity also decreases casting rate. Since most plants try to maintain a constant casting rate, the dotted lines show the effect of specific gravity on strength at constant casting rate. The effect is diminished because of the effect of increased electrolyte in the lower gravity samples necessary to maintain a constant casting rate. However, if a plant is able to tolerate a decrease in casting rate, of up to 15%, a specific gravity increase is a good way to increase MOR. Note that this is the same type of effect that is seen when adding fine clays for coarse clays to increase strength. Also, it should be noted that even when adjusted for casting rate, the increased strength due to increased specific gravity is still noticeable in both the dry and 50% RH tests. In addition, the effect of specific gravity on 50% RH strength is somewhat diminished as compared to its effect on dry strength. Notice that the slopes of the lines show the same result as was seen in the sulfate effect, and this again stresses the importance of measuring MOR using both dry and 50% RH tests.

Summary

The MOR test is important because it is the best indication available of what the strength of ceramic products will be during critical dry-ware han-
dling operations. However, test procedure-related factors can significantly vary the results of the test. Important test-related factors are time exposed to the atmosphere, the temperature of the bars, the span used for testing, the shape of the bars, handling of the bars, and the casting technique used.

In addition, although most of us normally think about the effect of fine-vs coarse-grained clays on strength, it is really these other body-related factors that play a more significant role in increasing or decreasing MOR. The body-related factors studied here include amount of electrolyte, sulfate level, organic content and specific gravity. Of course there are others, however, it is the combined effect of all of these variables that will determine the effect of changes made to a body on MOR.

*Instron Corp., Canton, MA.

Fig. 1. MOR vs time at 50% RH.
Fig. 2. MOR vs time at 50% RH.

Fig. 3. MOR vs bar temperature.
Fig. 4. MOR vs breaking span.

Fig. 5. MOR vs amount of sodium silicate.
Fig. 6. MOR vs amount of sodium silicate.

Fig. 7. MOR vs effective organic.
Fig. 8. MOR vs sulfate.

Fig. 9. MOR vs specific gravity.
Application of Texas Bentonites in Structural Clay Brick Formulations

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Texas bentonite additions were made to Oklahoma clay brick formulations lacking natural clay colloid. Significant improvements in extrudate quality (reduction in surface lamination, ease in extrusion) and green strengths were realized with as little as 0.5 to 1.0% bentonite additions.

In the ceramics industry, there are essentially two approaches to manufacturing, materials and equipment. When a sustained problem develops in an established plant, typically a materials solution is sought if the alternative is major capital investment. In recent history, however, equipment modernization and improvements such as spray drying, pelletization, fast firing, etc., has enabled manufacturers to more fully utilize less-processed materials in the greenfield development of a plant. But even in this case, “additives” to materials systems are sometimes required to develop needed performance. Bentonites are such “additives” and provide the “working” properties needed in materials processing.

Oklahoma Brick Co. used a mixture of indigenous clays as their primary clay body. This body was appreciably coarse-grained as compared to typical porcelain bodies (see Fig. 1). The methylene blue index (MBI), a measure of clay surface, has been suggested* as an indicator of extrudability, and varies from 1.5–3.0 meq/100 g for the Oklahoma brick body. MBIs for porcelain bodies can range from 2.2 for sanitaryware bodies, to 3.5 for dinnerware bodies, and to 4.5 for electrical porcelain bodies. However, the relationship of MBI to porcelain bodies assumes that the bodies are fully dispersed in water.

The extrudability of a ceramic body is not only a function of the total colloid matter present, but also the extent of the clay-water interaction. In the case of Oklahoma brick, the clay body was milled, dry mixed, and wetted just prior to pugging and extrusion. The resultant extrudate block was found to be discontinuous due to poor bonding qualities. Brick cut from the block tended to have rough surfaces, weak corners, and chipping along dieholes. Often, the load-bearing capacity, or strength of the brick, was not sufficient to prevent cracking in the bottom-tiered areas and, on occasion, an entire load would fail.
Attempts were made to improve the natural plasticity of the primary clay body by adding more water but strengths were not improved in this manner. Die-lube pressure, indicative of the compaction of a clay mass through the Oklahoma brick process for a given moisture content was reduced when increasing water addition and the resultant extrudate exhibited less resistance to extrusion and greater flow characteristics. These are characteristics of a "short" clay body or body lacking natural colloid to develop plasticity.

During one y of testing, Oklahoma brick found that 0.5 to 1.0% Texas bentonite weight addition made the following changes:

1. Increased die-lube pressures on average of 15% at their normal moisture level (14%) which resulted in more continuous and compact extrudate blocks.
2. Extrudate surfaces were smoother and easier to cut. Cosmetically, a better-appearance brick was made possible.
3. The load-bearing capacity of the brick was improved. Results are shown without the addition (Fig. 2A) and with 0.5% Texas bentonite addition (Fig. 2B).

Over this same time period, Oklahoma brick tested a Wyoming bentonite product, appreciably sodium bentonite-form, and observed:

1. The resultant brick "swelled" excessively at only a 0.5% weight addition. Brick were found to expand from 5–7.5 vol% from die dimensions.
2. Fired brick had decreased fired bulk density and increased size relative to that of normal production. These changes were outside Oklahoma brick specifications.
3. Surface and bulk cracking in fired brick.
4. Increased tendency for "leaners" on kiln cars. "Leaners" is a term used to describe an unsteady brickload where the top brick in a stack shrink faster than the bottom brick, causing a leaning effect.

These problems were noted with the Texas bentonite addition. Figure 3 shows a kiln car of brick made without the addition and Fig. 4 shows a car of brick made with the addition. However, it was noted that there were many variables in the Oklahoma brick process which could account for failed brick not directly related to the clay body. It is emphasized that in order to fully appreciate the benefit of Texas bentonite, quality control inspection was required for:

1. Grinding and pugmill areas—moisture checks on materials before and after extrusion. Monitoring die-lube pressures and amperage (powder consumed in extrusion) readings.
2. Drying—Note drying conditions very carefully when running tests. Rapid drying could cause ware to crack.
3. Loading/Unloading patterns—note the condition before and after firing in order to determine which setting pattern works best for a given process.

**Summary and Conclusion**

1. Texas bentonite additions from only 0.5 to 1.0 wt% enhanced the extrudability of the Oklahoma brick body while increasing strength and bulk density.
2. Texas bentonite additions did not cause excessive swelling as did Wyoming (sodium) bentonites. Wyoming bentonites were also found to de-
crease bulk density and increase the size of brick relative to Texas bentonites while increasing the tendency for unstable, uneven brickload conditions.

*G. W. Phelps; private communication.

Fig. 1. Particle size distribution of the Oklahoma Brick Co. (OBC) body compared to that of a typical porcelain body.