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## SOME FACTORS AFFECTING THE PROPERTIES OF CERAMIC TALCOSE WHITEWARE

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### ABSTRACT

Talcose bodies of 12 different compositions were formed by (1) pressing, (2) hand-wedging and extrusion, and (3) hand-wedging, deairing, and extrusion. They were heated on six different schedules: to cone 4 in 16 and in 24 hours; to cone 6 in 9, 16, and 24 hours; and to cone 8 in 24 hours. Determinations were made of such properties as shrinkage, absorption, strength, and linear thermal expansion. Conclusions are expressed regarding the effects of the various forming methods and heating schedules on the properties.

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### I. INTRODUCTION

The bodies used in this study are modifications of talcose bodies previously reported on,<sup>1</sup> and are designed to be vitrified, or nearly so, after heating in the range from cone 4 to cone 6 (approximately 1,165° to 1,190° C).

The primary purpose of the work was to provide data on the effects of dry-pressing versus plastic forming, of deairing versus hand-wedging, and of various heating rates, on such properties of the bodies as absorption, shrinkage, and strength. Data were obtained also on the precision of values obtained with specimens made by the different methods, which data may be of assistance in the development of standardized methods for testing clay products.

The ultimate purpose was to assist those manufacturers of ceramic whiteware who are interested in the possibility of producing vitrified ware of good quality at temperatures sufficiently lower, and on heating schedules sufficiently shorter than those now common, to effect a real economy.

<sup>1</sup> R. F. Geller and A. S. Creamer, *J. Am. Ceramic Soc.* **20**, 137 (1937).

## II. MATERIALS USED AND METHODS OF FORMING AND TESTING THE SPECIMENS

The materials used are listed in table 1. This table shows also the combinations in which they were used to form the 12 bodies investigated.

The first talc listed in table 1 contains 6.9 percent of CaO and is from New York State, the next two contain 1.6 and 0.3 percent, respectively, and are from California, and the fourth contains a trace of CaO and is from Manchuria. The details of chemical composition have been published.<sup>2</sup>

The chemical composition of each kaolin and ball clay, and of the feldspar, has appeared in several previous reports, of which the most recent is National Bureau of Standards Research Paper 1311.<sup>3</sup>

TABLE 1.—Compositions of laboratory-prepared bodies

Material	Body number—											
	25	26	27	28	29	30	31	32	33	34	35	36
Talc (CaO, 6.9%)	% 25	% 25	% 25	%	%	%	%	%	%	%	% 20	% 15
Talc (CaO, 1.6%)				30	25	20		25	25			
Talc (CaO, 0.3%)												
Talc (CaO, trace) <sup>a</sup>												
Florida kaolin	10	10	5	10	10.7		10	5	25	20	26.7	28.3
North Carolina kaolin	25	20	20	20	21.4	22.9	25	15	20	20		
Tennessee ball clay A	5	5	7.5	5	5.4		5	5	5			
Kentucky ball clay	5	5	7.5	5	5.3		5	5	5			
Tennessee ball clay B						22.9		15	10	20	21.3	22.7
Feldspar	10	15	15	15	16.1	17.1	15	15	15	15	10.7	11.3
Flint	20	20		15	16.1	17.1	15	15			21.3	
Flint reground <sup>b</sup>			20						20			22.7
Tripoli										25		

<sup>a</sup> Practically pure talc, previously reported on in J. Research NBS 15, 55 (1935) RPS48.

<sup>b</sup> The maximum nominal diameter of particles, by microscopic examination, was 35 microns.

The "flint" used is pulverized quartz of commercial grade, for which the determined  $\text{Fe}_2\text{O}_3$ , nonvolatile residue, and ignition loss totaled 0.38 percent. The tripoli was supplied by a manufacturer of white-ware and was not analyzed.

The bodies were prepared in batches of 50 lb each. As a preliminary step, each ball clay was made into a thin slip with water, passed through a No. 200 U. S. Standard Sieve, and then dried and pulverized. The body mixtures were ground wet in ball mills for 2 hours, passed through a No. 100 sieve and a magnetic separator, and filter-pressed. The press-cakes were then placed in damp storage.

After an interval of not less than 2 weeks, a portion of each body was hand-wedged to a good workable consistency (as judged by feel) and extruded as rods  $\frac{5}{8}$  in. in diameter and cut to suitable lengths. Another portion of each body was air-dried, crushed to pass a No. 30 sieve, made up by hand with 10 percent of water, again passed through a No. 30 sieve, and pressed into bars approximately  $6\frac{1}{4}$  in. long by  $\frac{3}{4}$  in. wide by  $\frac{3}{16}$  in. thick under a pressure of about 1420 lb in.<sup>2</sup> (100 kg cm<sup>2</sup>). A hole was drilled at one end of each bar, and the bars were hung while being heated in the kiln. The bars were hung in order that they might remain free from warpage during

<sup>3</sup> See footnote 1, and also R. F. Geller and A. S. Creamer, J. Am. Ceramic Soc. 18, 259 (1935).

<sup>4</sup> R. F. Geller and E. N. Bunting, J. Research NBS 25, 15 (1940) RP1311.

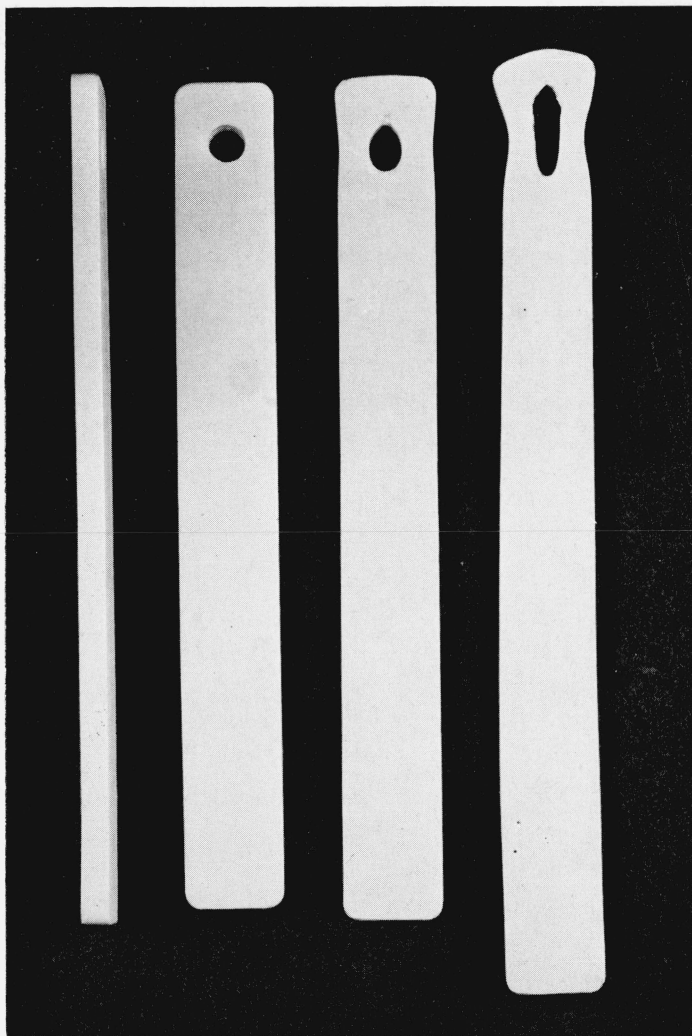


FIGURE 1.—*Specimens made by dry-pressing and hung during heating.*  
They illustrate “no deformation”, “slight deformation”, and “considerable deformation”, during heating.



FIGURE 3.—*Electrically heated tunnel kiln.*



the heating, and also to provide a relative measure of their resistance to deformation (fig. 1). These rods and bars were made in 1937, and put through the kiln in 1938, using manual control of kiln temperatures.

In 1939 the remaining portion of each body (now thoroughly dry) was crushed to pass a No. 30 sieve and again hand-wedged to a workable consistency as judged by feel. Small pieces were then placed in the extrusion cylinder and the air evacuated to a pressure of less than  $\frac{1}{10}$  atmosphere, after which the material was extruded as rods  $\frac{5}{8}$  in. in diameter and cut to appropriate lengths. No pressed bars were made for the second series of tests. The specimens were put through the kiln in 1940, after an automatic temperature control had been installed.

The "free," or mechanically held, water present in the bodies as extruded, or as pressed, was computed on the dry basis from the weights

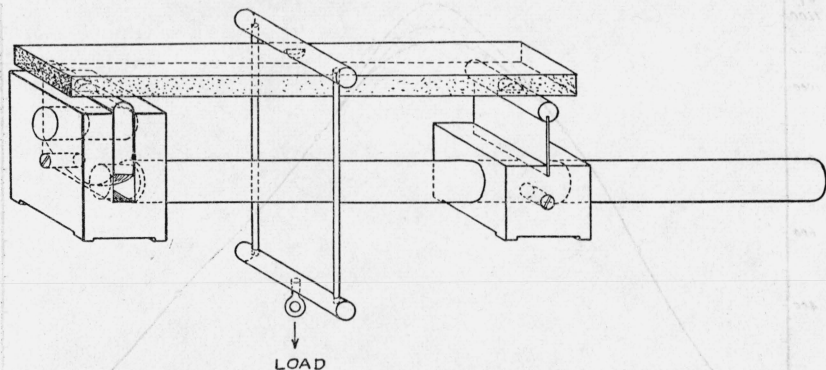


FIGURE 2.—Variable-span support used for modulus of rupture and modulus of elasticity determinations.

The apparatus can be used also for the testing of specimens in rod form.

immediately after forming and after drying to constant weight at  $110^{\circ}$  C.

The linear shrinkage of extruded rods was measured directly with a caliper, using reference marks which had been pressed into the rods, 10 cm apart, immediately after extrusion. The shrinkage of pressed bars was measured in an especially designed holder, using an Ames gage reading to 0.001 in., and observing the over-all length of the bars. Percentage values are based on the original lengths.

Absorption was determined by weighing specimens before and after autoclaving in water for 5 hours under a steam pressure of 150 lb./in.<sup>2</sup>

Moduli of elasticity (Young's) and of rupture were calculated from values obtained by center loading over a span of 5 in., unless otherwise noted. The dry specimens and the heated bars were broken on the device shown in figure 2. This device was placed on a firm base (not shown in the drawing) and the load applied to the loading saddle, which was hung from the specimen at midspan. The rocker-type and the flexural-type knife-edges are designed to prevent torsional and axial forces. The device was used also in determining the modulus of elasticity of the heated bars, for which tests a gage reading to 0.0001 in. was mounted on the base so as to indicate the deflection of the bar

under load. The heated rods were broken with the machine described by Harrison.<sup>4</sup>

Linear thermal expansions were determined by the interferometer method.<sup>5</sup>

The specimens were put through an electrically heated tunnel kiln, of the walking-beam type, designed and built at the National Bureau of Standards. The kiln proper (fig. 3) is 35 ft long. Sheet-iron vestibules, used for placing, drawing, prewarming and cooling, extend for about 5 ft from each end. The cross-sectional area available for passage of ware is 12 in. wide by 6 in. high. Heat is obtained from 36

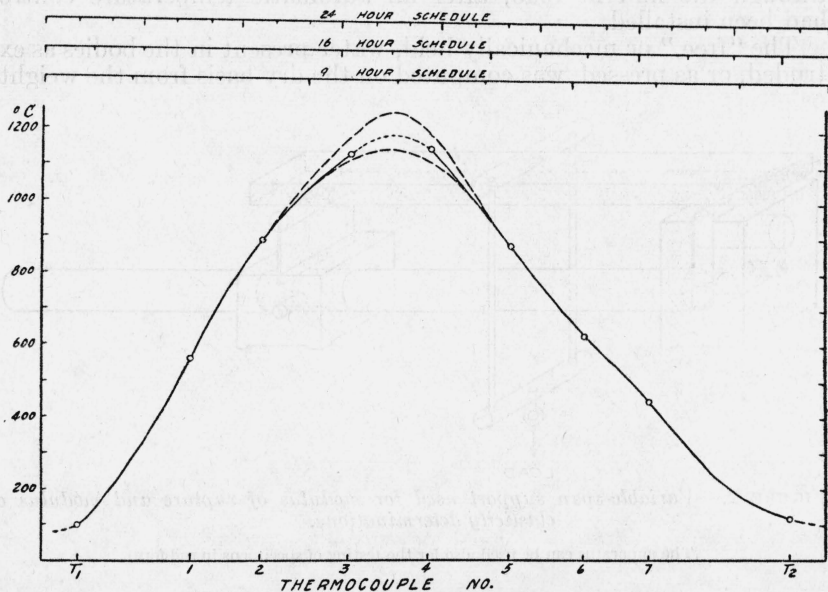


FIGURE 4.—Heating schedules.

These curves are based on temperatures indicated by seven thermocouples and two thermometers ( $T_1$  and  $T_2$ ) in the electrically heated tunnel kiln at the National Bureau of Standards and show the heating rates followed in this investigation. The relative positions of the thermocouples and thermometers, as placed along the kiln, are indicated at the bottom of the figure.

The time-temperature relations below approximately 900° C are the same, regardless of the maximum temperature of the test. Above 900° C the curves diverge, as indicated by the broken lines. The 9-, 16-, and 24-hr. schedules apply to each of the various heating curves.

Global elements mounted vertically 18 on a side and in 8 groups for control purposes.

Air was introduced in each vestibule, permitting control of vestibule temperatures and the maintenance of a slight positive pressure (0.002 to 0.005 in. of water) within the kiln.<sup>6</sup> In 1939 a change was made in the electric system. This involved the insertion of a full on-and-off automatically controlled switch in each of the two circuits supplying opposing groups of five heating elements located at the hottest section of the kiln. This arrangement maintained a desired temperature within  $\pm 3^\circ$  C with the controlling couple located about 1 in. below the crown. The total power input averaged about 80 kw when

<sup>4</sup> A. C. Harrison, *J. Am. Ceramic Soc.* 8, 774 (1925).

<sup>5</sup> George E. Merritt, *BS J. Research* 10, 59 (1933) RP515.

<sup>6</sup> This procedure was suggested by R. E. Gould, then of the Tennessee Valley Authority Laboratories, and credit is due also to McD. S. Nelson, also of these Laboratories, for personal assistance in a study of the operating efficiency of the kiln.

operating at cone 4 (1,165° C), 85 kw at cone 6 (1,190° C), and 87 kw at cone 8 (1,225° C), and the heating-and-cooling cycle of ware passing through the kiln is indicated by the curves in figure 4.

### III. RESULTS

The values for absorption, shrinkage, and modulus of rupture in tables 2 and 3 are, in most cases, the averages for 10 specimens. Values for modulus of elasticity are averages for three specimens. The plus-or-minus ( $\pm$ ) values in these tables were calculated by the method recommended by the American Society for Testing Materials,<sup>7</sup> using  $P_s=0.95$ . These values for 95 percent confidence error will hereafter be referred to simply as "the error." Since values for the same number of specimens are averaged in most cases, the values for error indicate the relative uniformity of the specimens.

TABLE 2.—Same data on bodies as formed, dried, and heated

Body No.	Free water in extruded bars		Shrinkage during drying	Modulus of rupture dry	Free water in pressed bars	Modulus of elasticity; <sup>d</sup> pressed bars				
	(a)	(b)				Cone 4 <sup>e</sup>		Cone 6 <sup>e</sup>		
			(%)	(%)	(b)	(b)	(c)	16-hr schedule	24-hr schedule	9-hr schedule
25.....	24.7	29.5	3.9 $\pm$ .1	170 $\pm$ 8	8.4	lb/in. <sup>2</sup> 8.0	lb/in. <sup>2</sup> 7.1	lb/in. <sup>2</sup> 9.3	lb/in. <sup>2</sup> 9.6	lb/in. <sup>2</sup> 10.8
26.....	28.7	28.8	4.0 $\pm$ .3	160 $\pm$ 4	8.1	10.0	8.6	9.9	10.1	9.6
27.....	30.0	28.8	3.4 $\pm$ .2	300 $\pm$ 9	8.3	7.5	7.6	9.2	8.5	8.1
28.....	22.6	28.7	4.5 $\pm$ .1	120 $\pm$ 3	8.9	8.0	7.8	9.6	9.8	9.4
29.....	32.0	27.8	3.8 $\pm$ .1	150 $\pm$ 4	10.4	9.0	8.3	9.4	9.6	10.5
30.....	31.7	24.4	4.0 $\pm$ .2	220 $\pm$ 6	8.5	7.4	7.6	9.0	9.3	9.6
31.....	30.6	28.3	4.2 $\pm$ .1	150 $\pm$ 3	9.3	7.2	6.5	10.3	9.3	9.1
32.....	31.5	26.2	4.2 $\pm$ .1	220 $\pm$ 8	9.0	8.4	7.3	7.9	9.6	9.6
33.....	32.9	30.3	4.5 $\pm$ .1	140 $\pm$ 7	7.2	7.4	6.8	9.9	9.0	9.5
34.....	32.0	30.0	4.9 $\pm$ .1	200 $\pm$ 4	7.8	6.7	5.9	9.3	9.3	8.3
35.....	30.5	27.3	4.5 $\pm$ .2	250 $\pm$ 4	-----	6.4	5.7	8.4	8.1	7.3
36.....	33.3	29.3	4.6 $\pm$ .1	260 $\pm$ 4	-----	7.0	6.1	-----	7.5	7.7

<sup>a</sup>Bars made of bodies hand-wedged and extruded, and heated in 1938.

<sup>b</sup>Bars made of bodies hand-wedged, deaired and extruded, and heated in 1940.

<sup>c</sup>Values obtained by weighing specimens when pressed and after drying at 110° C. All bodies were made up with 10 percent of water.

<sup>d</sup>Values to be multiplied by 10<sup>8</sup>.

<sup>e</sup>Heating to "cone 4" and to "cone 6" indicates approximate maximum temperatures of 1,165° C and 1,190° C, respectively. For details of heating schedules, see figure 4. Bars heated with manual kiln control.

<sup>f</sup>This body was obviously not completely air-dry before adding the water for pressing.

#### 1. WORKABILITY

Optimum workability of a plastic mixture, prepared for extrusion, was judged by feel, rather than by some mechanical means, in order to observe how closely the same operator could duplicate the proportions when working with parts of the same body but at considerably different times (in this case, 2 years). Although the hand-wedging was admittedly amateurish, it is believed to have equalled that usually done in laboratories. As shown in table 2, less water was used for 9 of the 12 bodies when the second series of specimens was prepared but, of the 4 greatest variations, 2 (bodies 25 and 28) were in one direction and the other 2 (bodies 30 and 32) were in the opposite direction. The average variation was 3.6 percent.

<sup>7</sup> Am. Soc. Testing Materials, Manual for Interpretation of Refractory Data, p. 87 (February 1935).

Am. Soc. Testing Materials, Manual on Presentation of Data, p. 40-41 (1933). Second printing, March 1937

## 2. SHRINKAGE

Comparing the values for the "a" specimens (not deaired) with those for the "b" specimens (deaired) in the columns of table 3 headed "Extruded", it is seen that, of the 45 pairs of comparable values for total shrinkage, the maximum difference for total linear shrinkage was 3.6 percent (body 27) and the average difference was 0.7 percent. In 28 of the 45 cases, the shrinkage of the deaired body was greater than that of the comparably heated nondeaired body but in only 18 of the 28 cases was the difference significant.<sup>8</sup> In one case there was no difference. In the remaining 16 cases, the shrinkage of the deaired body was less, and in 13 of these the difference was significant. These differences in shrinkage were independent of the respective amounts of water used in forming (note bodies 26, 33, and 34). As indicated by the error values, the uniformity of a deaired body was greater than that of a comparable nondeaired body in 25 cases, while it was less in only 8 cases and the same in the remaining 12. In general, the shrinkage values do not show clearly defined effects of the described differences in processing.

There was no apparent correlation between shrinkage values and the variations in composition or in heat treatments. That is, no bodies consistently showed outstandingly high or low shrinkage after all the heat treatments and, with few exceptions (for example, body 34) the shrinkage, especially of a deaired body, was not altered more than about 1 percent by the various heat treatments. In general, the pressed bodies showed greater variation in shrinkage with variation in heat treatment than the extruded bodies. For detailed data and a discussion on the shrinkage of bodies 27 and 35 during heating, see the reference given in footnote 3.

## 3. ABSORPTION

The absorption values in table 3 show that only one pressed body (No. 26) and five extruded bodies (Nos. 25, 26, 29, 30, and 32) could be classified as vitrified, or nearly so, after heating on both the 16-hour and the 24-hour schedules to cone 4.<sup>9</sup> After heating to cone 6 on the 9-hour schedule, all of the specimens (both pressed and extruded) were vitrified, or nearly so, but the pressed specimens of bodies 27, 34, 35, and 36, heated on the 16- and 24-hour schedules, could not be so classified. After heating to cone 8, all of the bodies, excepting 35 and 36, were completely vitrified.

In this connection, it is interesting to note that the pressed specimens of bodies 27, 34, 35, and 36, heated to cone 6, showed a definite increase in absorption with an increase in the length of the heating schedule. The same relation between the absorptions and the heating schedules was shown by all of the pressed bodies heated to cone 4. Some of the extruded bodies heated to cone 4 did not show this relation, and the differences in absorption were often not significant. The production of a lower relative absorption during the shorter of two heating

<sup>8</sup> The significance of differences between averages was determined by the ratio

$$\frac{\bar{X}_1 - \bar{X}_2}{\sqrt{r_1^2 + r_2^2}}$$

where  $\bar{X}$  is the average value, and  $r$  is the error ( $\pm$  value) of the average. If the ratio is 1.0 or more, the percentage confidence is 95 or more, and the difference is considered significant. If the ratio is 1.5 or more, the percentage confidence is 99 or more, and the difference is considered highly significant.

<sup>9</sup> A "nearly vitrified" body is arbitrarily defined as one having an absorption of not more than 1 percent. Heating to cone 4, 6, or 8 means heating to a nominal maximum temperature of 1,165°, 1,190°, or 1,225° C.



TABLE 3.—*Some properties of pressed, extruded, hand-wedged, and deaired bodies heated on various schedules*

TOTAL LINEAR SHRINKAGE

Body No. ( <sup>a</sup> b)	Cone 4 on 16-hour schedule		Cone 4 on 24-hour schedule		Cone 6 on 9-hour schedule		Cone 6 on 16-hour schedule		Cone 6 on 24-hour schedule		Cone 8 on 24-hour schedule	
	Pressed	Extruded	Pressed	Extruded	Pressed	Extruded	Pressed	Extruded	Pressed	Extruded	Extruded	
	%	%	% ± .4	% ± .3	% ± .9	% ± .6	% ± .2	% ± .2	% ± .1	% ± .1	% ± .1	% ± .4
25 a	12.2 ± .5	13.9 ± .4	11.5 ± .4	13.9 ± .3	11.5 ± .9	13.7 ± .6	12.8 ± .2	13.4 ± .2	13.0 ± .1	13.2 ± .1	13.2 ± .1	11.9 ± .4
25 b		12.6 ± .2		12.9 ± .2		12.9 ± .2					13.1 ± .2	
26 a	12.0 ± .4	12.1 ± .2	12.5 ± .2	11.4 ± .3	10.9 ± .1	12.0 ± .2	9.9 ± .8	11.6 ± .3	11.7 ± .4	12.1 ± .3	12.3 ± .2	10.7 ± .7
26 b		12.7 ± .1		12.5 ± .5		12.5 ± .3					12.3 ± .2	
27 a	10.4 ± .1	10.3 ± .4	10.3 ± .1	10.3 ± .2	11.3 ± .1	10.0 ± .5	10.8 ± .1	11.0 ± .2	10.7 ± .1	11.3 ± .2	11.3 ± .2	10.2 ± .2
27 b		10.5 ± .3		10.8 ± .4							14.9 ± .2	
28 a	10.7 ± .5	14.4 ± .2	9.9 ± .4	13.5 ± .4	10.7 ± .4	15.1 ± .2	11.7 ± .1	14.7 ± .2	11.8 ± .1	14.9 ± .1	15.1 ± .2	14.5 ± .2
28 b		14.0 ± .2		14.7 ± .1		14.7 ± .2					15.1 ± .2	
29 a	11.4 ± .3	14.2 ± .2	11.0 ± .2	14.3 ± .2	10.2 ± .4	15.1 ± .3	11.9 ± .1	15.0 ± .1	12.2 ± .1	15.2 ± .2	15.2 ± .2	14.7 ± .2
29 b		13.7 ± .1		14.5 ± .1							13.6 ± .2	
30 a	11.5 ± .2	13.9 ± .2	11.1 ± .2	13.5 ± .2		13.7 ± .4	11.6 ± .2	14.0 ± .2	11.4 ± .1	14.1 ± .1	14.1 ± .1	13.6 ± .2
30 b		13.4 ± .2		13.7 ± .2		13.5 ± .1					14.7 ± .2	
31 a	10.6 ± .6	13.7 ± .4	9.8 ± .2	13.8 ± .2	12.8 ± .1	14.9 ± .1	12.2 ± .1	14.8 ± .2	11.7 ± .3	15.0 ± .2	15.0 ± .2	14.6 ± .3
31 b		13.5 ± .3		14.1 ± .2							13.8 ± .5	
32 a	11.4 ± .5	14.5 ± .3	10.5 ± .2	14.3 ± .1	11.3 ± .5	14.3 ± .1	12.0 ± .1	15.0 ± .1	12.1 ± .1	15.1 ± .1	15.1 ± .1	13.8 ± .5
32 b		13.8 ± .1		13.8 ± .1		13.5 ± .1					13.8 ± .5	
33 a	11.0 ± .5	13.3 ± .3	10.1 ± .2	13.5 ± .2	11.7 ± .4	13.9 ± .6	11.8 ± .1	14.3 ± .1	11.9 ± .1	13.9 ± .2	13.9 ± .2	13.5 ± .5
33 b		14.0 ± .2		14.6 ± .2		14.7 ± .2					14.9 ± .1	
34 a	9.7 ± .9	12.6 ± .6	8.7 ± .2	12.0 ± .4	11.8 ± .3	13.9 ± .3	11.7 ± .2	13.8 ± .3	11.5 ± .2	14.9 ± .2	14.5 ± .3	14.0 ± .4
34 b		13.3 ± .2		13.5 ± .1		14.9 ± .2					15.1 ± .1	
35 a	10.4 ± .6	12.1 ± .5	9.4 ± .2	11.8 ± .2	11.6 ± .3	12.3 ± .3	11.3 ± .1	12.0 ± .4	10.5 ± .2	11.3 ± .4	11.3 ± .4	11.5 ± .5
35 b		12.2 ± .1		12.0 ± .2		12.7 ± .1					12.4 ± .3	
36 a	11.6 ± .4	12.5 ± .4	10.8 ± .1	13.4 ± .2		13.0 ± .4	12.2 ± .1	12.8 ± .3	11.7 ± .1	13.0 ± .4	13.0 ± .4	11.9 ± .4
36 b		13.2 ± .1		13.4 ± .2		14.0 ± .2					13.7 ± .2	

See footnotes at end of table.



TABLE 3.—Some properties of pressed, extruded, hand-wedged, and deaired bodies heated on various schedules—Continued

## ABSORPTION °

Body No. (a b)	Cone 4 on 16-hour schedule		Cone 4 on 24-hour schedule		Cone 6 on 9-hour schedule		Cone 6 on 16-hour schedule		Cone 6 on 24-hour schedule		Cone 8 on 24-hour schedule.
	Pressed	Extruded	Pressed	Extruded	Pressed	Extruded	Pressed	Extruded	Pressed	Extruded	Extruded
25 {a	2.4 ± 1.1	0.2 ± 0.1	4.0 ± 0.6	0.5 ± 0.4	0.0	0.0	0.0	0.0	0.0	0.0	0.0
25 {b		0.4 ± 1		0		0		0		0	
26 {a	0.2 ± .2	0	0.5 ± .3	0	0	0	0	0	0	0	0
26 {b		0		0		0		0		0	
27 {a	4.1 ± .6	.9 ± .5	4.6 ± .1	1.3 ± .3	.4 ± 0.0	0	1.9 ± 0.5	0	2.9 ± 0.5	0	0
27 {b		.5 ± .0		0.4 ± .1		0		0		0	
28 {a	2.6 ± 1.2	.8 ± .8	4.3 ± .6	1.1 ± .6	0	0	0.0	0	0.1 ± .1	0	0
28 {b		2.2 ± .4		0.4 ± .2		0		0		0	
29 {a	0.3 ± .2	0.5 ± .4	2.2 ± .5	.4 ± .3	0	0	0	0	0	0	0
29 {b		.8 ± .1		0		0		0		0	
30 {a	1.1 ± .5	.3 ± .3	1.9 ± .5	.2 ± .2	.4 <sup>d</sup>	0	0	0	0	0	0
30 {b		.2 ± .1		0		0		0		0	
31 {a	4.2 ± 1.2	1.3 ± .7	5.0 ± .2	2.3 ± .7	0	0	.6 ± .1	0	.7 ± .6	0	0
31 {b		2.3 ± .6		1.6 ± .2		0		0		0	
32 {a	1.7 ± .9	0.6 ± .4	3.5 ± .4	0.5 ± .2	.7 ± .6	0	0	0	.1 ± .1	0	0
32 {b		.3 ± .1		.1 ± .0		0		0		0	
33 {a	2.2 ± .9	.8 ± .5	3.8 ± .4	1.6 ± .6	0	0	.1	0	.6 ± .2	0	0
33 {b		1.3 ± .1		0.5 ± .1		0		0		0	
34 {a	4.7 ± 1.8	3.5 ± 1.5	6.2 ± .3	4.8 ± 1.2	.2 ± .2	.1 ± 0.1	.5 ± .2	0	1.8 ± .5	.5 ± 0.4	0
34 {b		4.8 ± .2		3.7 ± .3		0		0		0	
35 {a	3.7 ± 1.4	2.3 ± .8	5.7 ± .1	3.3 ± .6	.3 ± .0	.3 ± .1	1.6 ± .2	.9 ± 0.2	3.1 ± .5	.9 ± .3	.4 ± .1
35 {b		2.1 ± .1		1.9 ± .1		.3 ± 0		0		.7 ± 0	
36 {a	2.7 ± .8	1.4 ± .6	4.2 ± .2	1.8 ± .3	.0 <sup>d</sup>	0	1.0 ± .1	.5 ± .2	2.4 ± .3	.5 ± .2	.2 ± .1
36 {b		1.1 ± .1		0.9 ± .1		0		0		.1 ± 0	

MODULUS OF RUPTURE

	$lb/in.^2$	$lb/in.^2$	$lb/in.^2$	$lb/in.^2$	$lb/in.^2$	$lb/in.^2$	$lb/in.^2$	$lb/in.^2$	$lb/in.^2$	$lb/in.^2$	$lb/in.^2$	$lb/in.^2$
25	7,400 ±500	11,000 ±700	6,900 ±500	9,300 ±850	9,700 ±350	9,300 ±600	9,900 ±250	10,600 ±750	9,300 ±450	12,100 ±1,000	7,400 ±300	
		10,500 ±600		12,600 ±350		12,300 ±250				13,500 ±350		
26	9,300 ±250	10,300 ±750	9,300 ±550	9,100 ±800	9,600 ±400	7,900 ±300	9,200 ±250	9,300 ±350	9,200 ±350	9,800 ±150	5,200 ±300	
		12,500 ±250		11,700 ±200		11,000 ±250				11,800 ±600		
27	7,000 ±300	6,400 ±850	7,300 ±250	7,000 ±450	8,700 ±450	7,100 ±600	7,600 ±250	7,500 ±900	7,000 ±300	9,500 ±600	7,900 ±500	
		9,500 ±500		10,300 ±450								
28	8,000 ±800	9,200 ±600	7,000 ±150	7,400 ±650	9,800 ±300	8,300 ±350	10,000 ±300	8,800 ±1,000	9,900 ±450	8,700 ±600	5,500 ±550	
		9,100 ±550		11,500 ±300		12,200 ±350				12,700 ±250		
29	9,400 ±600	6,700 ±400	7,900 ±350	7,500 ±650	9,800 ±450	8,200 ±800	10,000 ±350	9,700 ±550	9,800 ±450	8,900 ±650	7,100 ±1,050	
		8,700 ±600		12,100 ±650						11,000 ±700		
30	7,600 ±450	9,000 ±900	7,400 ±300	9,000 ±800		10,200 ±250	9,000 ±250	7,100 ±600	9,500 ±350	9,600 ±700	7,900 ±950	
				12,100 ±250		11,300 ±400				11,200 ±300		
31	6,900 ±450	7,600 ±1,000	6,300 ±300	7,500 ±900	10,000 ±250	10,300 ±600	8,900 ±350	8,800 ±600	7,600 ±450	8,100 ±420	9,400 ±700	
		9,200 ±500		10,600 ±200						11,500 ±300		
32	8,100 ±200	9,700 ±900	7,400 ±100	9,000 ±750	8,400 ±600	9,200 ±500	8,100 ±300	8,900 ±750	8,700 ±350	10,100 ±600	7,800 ±750	
		11,100 ±450		11,600 ±700		10,700 ±200				12,600 ±400		
33	7,000 ±250	8,800 ±500	6,800 ±100	8,600 ±500	9,600 ±350	10,000 ±450	9,700 ±250	9,100 ±1,050	8,600 ±650	9,900 ±1,000	6,500 ±700	
		10,400 ±400		11,200 ±300		11,200 ±700				11,300 ±550		
34	7,400 ±400	7,400 ±750	7,000 ±200	8,000 ±700	9,200 ±400	10,100 ±350	9,000 ±200	9,200 ±950	8,400 ±550	8,700 ±650	8,900 ±700	
		9,700 ±500		9,000 ±350		12,400 ±250				10,500 ±300		
35	5,700 ±450	8,500 ±350	5,400 ±300	8,500 ±350	7,400 ±250	8,600 ±200	6,900 ±150	8,500 ±200	6,000 ±450	9,200 ±250	6,600 ±250	
		9,800 ±200		9,700 ±200		10,200 ±300				8,700 ±150		
36	6,000 ±100	8,600 ±450	5,900 ±250	9,000 ±550		10,300 ±300	6,800 ±300	9,100 ±450	6,800 ±150	8,600 ±450	8,800 ±400	
		10,400 ±200		10,000 ±400		11,200 ±200				11,500 ±150		

<sup>a</sup> Bodies "a" were either pressed, using 100 kg/cm<sup>2</sup> (and amounts of water given in table 2), or hand-wedged and extruded, and heated in 1938 with manual control of kiln temperatures.

<sup>b</sup> Bodies "b" were hand-wedged deaired and extruded, and heated in 1940 with automatic control of the kiln temperatures.

<sup>c</sup> In all cases where the average value is 0. 0., the computed error was ±0.0.

<sup>d</sup> Only 1 specimen.

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schedules was reported by Watts<sup>10</sup> and also observed for bodies 27 and 35 in a previous study (see footnote 3).

The maximum temperature attained during the shorter of two schedules "to the same cone" is somewhat higher than during the longer schedule.<sup>11</sup> One might, therefore, at first thought ascribe the lower absorption, after the shorter schedule, to the higher maximum temperature, but this explanation does not apply to results from the previous study (footnote 3), in which the specimens were taken to the same maximum temperature.

In 19 of the 46 comparable pairs of values in table 3, the deaired bodies had the same absorption (zero) as the nondeaired. Of the remaining 27 cases, the absorptions for the deaired bodies were less in 21, and greater in 5 cases. These five all occurred with bodies heated to cone 4 in 16 hours.

The uniformity of the deaired bodies, as indicated by the absorption-value errors, was usually considerably greater, and in no case less, than for the hand-wedged bodies. The average of the errors for the percentage absorption values is 0.5 for the hand-wedged, 0.3 for the pressed, and 0.1 for the deaired specimens.

#### 4. STRENGTH

The modulus of rupture values show some well-defined differences resulting from the various methods of forming the specimens. The most pronounced are the higher values for deaired and extruded specimens as compared with the values for pressed specimens heated on comparable schedules. Of the 41 pairs of these values in table 3, 40 show a higher strength for the deaired bodies, and the difference is highly significant in 38 cases. The average difference in strength for specimens heated to cone 4 was 3,400 lb/in.<sup>2</sup>, for specimens heated to cone 6 it was 2,600 lb/in.<sup>2</sup>, and the grand average was 3,100 lb/in.<sup>2</sup>.

The higher strength of deaired and extruded specimens compared with those hand-wedged and extruded is also clearly defined. Although the average increase in strength is only 2,300 lb/in.<sup>2</sup>, the average values for individual deaired bodies were higher in 41 out of 43 cases (table 3). Of these 41, the differences were significant in 39 cases, of which 34 were highly significant.

The difference between pressed specimens and those made of hand-wedged bodies is not so striking but, in the majority of cases, the latter had the higher strength. Of 58 pairs of comparable values in table 3, 39 differences showed the hand-wedged and extruded rods to have higher strength (average 1,400 lb/in.<sup>2</sup>) and 18 to have lower strength (average 1,000 lb/in.<sup>2</sup>). Of the 39 higher strengths, 26 differences were significant and 20 of these were highly significant. Of the 18 lower strengths, only 6 were significant and 5 of these were highly significant.

The variations in strength with increase in severity of heat treatment (from cone 4 on the 16-hour schedule to cone 8 on the 24-hour schedule) showed no common trend but, with three exceptions (bodies 31, 34 and 36), the strength, after the cone 8 treatment, was comparatively

<sup>10</sup> Arthur S. Watts, *J. Am. Ceramic Soc.* **19**, 175 (1936).

<sup>11</sup> During the operation of the kiln in 1940, the controlling thermocouple was set at the following temperatures for the various schedules:

Cone 4 in 16 hr.....	1,143° C.
Cone 4 in 24 hr.....	1,138° C.
Cone 6 in 9 hr.....	1,175° C.
Cone 6 in 24 hr.....	1,160° C.

low and the bodies were probably overfired. The following tabulation summarizes results of the cone 4 and cone 6 heat treatments for the 12 bodies. Columns *A* give the number of bodies which were significantly stronger, and columns *B* the number which were significantly weaker, after heating on the shortest schedule, compared with those heated on the longest schedule.

	Cone 4		Cone 6	
	A	B	A	B
Pressed.....	4	0	6	0
Hand-wedged.....	4	1	5	4
Deaired.....	2	6	2	5

Watts (footnote 10) reported from 300 to 600 lb/in.<sup>2</sup> higher transverse strength for bodies heated on the shorter of two schedules. Fritz<sup>12</sup> found that specimens cooled relatively rapidly showed an increase in strength of more than 35 percent over those cooled in the usual manner, and he contends that the more rapid cooling is responsible for the higher strength. To investigate further the effect of rapid cooling, the halves of pressed bars of bodies 27, 31, and 35, heated to cone 6 on the 24-hour schedule and broken across a 5-in. span, were subjected to the following test: One-half of each specimen was broken on a 2½-in. span without further treatment. The other half of each specimen was heated to 1,100° C and cooled to room temperature in 4 hours. The untreated half-specimens of these three bodies had average moduli of rupture of 7,000 ± 200, 7,300 ± 300, and 6,200 ± 300 lb/in.<sup>2</sup>, respectively. For the rapidly cooled halves, the values were 7,400 ± 300, 7,800 ± 300, and 6,600 ± 200 lb/in.<sup>2</sup>, respectively. The difference was significant in each instance and supports Fritz' contention.

The averages of the errors for the modulus of rupture determinations, grouped by method of processing and heat treatment, are as follows:

	Cone 4, 16 hr.	Cone 4, 24 hr.	Cone 6, 9 hr.	Cone 6, 24 hr.
	<i>lb/in.<sup>2</sup></i>	<i>lb/in.<sup>2</sup></i>	<i>lb/in.<sup>2</sup></i>	<i>lb/in.<sup>2</sup></i>
Hand-wedged.....	680	660	440	590
Pressed.....	400	280	380	410
Deaired.....	420	360	320	360

According to these averages, the uniformity of the pressed specimens is approximately the same as that of the deaired specimens.

It cannot be said with certainty that the greater strength of the deaired bodies was caused entirely by the processing, since these bodies were heated after the automatic temperature control had been installed. It seems reasonable to assume, however, that the deairing

<sup>12</sup> E. H. Fritz, *J. Am. Ceramic Soc.* **20**, 26 (1937).



is largely responsible, because it would have required irregularities in manual control considerably greater than those actually observed ( $\pm \frac{1}{2}$  cone) to account for the differences.

#### 5. ELASTICITY

Elasticity values were determined for pressed bars only and are given in table 2. They vary from  $5.7 \times 10^6$  to  $10.8 \times 10^6$ , and the average for bodies heated to cone 6 was somewhat higher than for those heated to cone 4.

#### 6. EXTENSIBILITY

The extensibility, or the amount a body can stretch before rupture, is the ratio of the modulus of rupture to the modulus of elasticity. The extensibility could, therefore, be calculated for pressed specimens by using the data in tables 2 and 3. The individual values ranged from  $8.2 \times 10^{-4}$  to  $11.9 \times 10^{-4}$  and averaged  $9.3 \times 10^{-4}$ . There was a small but not consistent variation in extensibility with variation in heat treatment.

#### 7. THERMAL EXPANSION

Results of linear thermal-expansion determinations are given in table 4. Because of the many variables in composition, it would be extremely difficult to explain all of the variations in expansion, but there appear to be reasons for some. For example, one body made with reground flint (No. 27) has a relatively low thermal expansion, while two others (bodies 33 and 36) do not. In the case of body 33, the total amounts of feldspar and of free silica, introduced either as such or in the clays, were the same within 2 percent as the amounts in body 27, and the use of lime-free talc is probably the reason for its higher expansion (see reference in footnote 2). The materials for body 36, however, contained in comparison 10 percent less of talc, about 5 percent less of feldspar, and about 6 percent more of free silica.

There is also no consistent relation between the thermal expansion and the method of forming, or the heat treatment, except that the expansions for bodies heated to cone 8 are markedly lower and indicate an appreciable solution of quartz in bodies heated above cone 6 ( $1,190^\circ \text{C}$ ).

#### 8. DEFORMATION

The dry-pressed specimens of bodies 25 to 29, inclusive, showed slight deformation (fig. 1) after the heating to cone 6 on the 9-hour schedule, and specimens of body 26 showed considerable deformation after heating to cone 6 on the 16- and 24-hour schedules. All others showed no deformation.

#### 9. COLOR

Specimens of bodies 25 to 27, inclusive, heated to cone 4, and all specimens heated to cone 6, were not quite white. Their color was a very light gray. All other specimens were light buff or ivory in color.



TABLE 4.—Linear thermal expansion of heated bodies

Body No.*	Heat treatment <sup>b</sup>		Coefficient of linear thermal expansion (multiply values by 10 <sup>-6</sup> )							Total expansion to 700° C
	Maximum temp.	Schedule	Room temp to 100° C	100° to 200° C	200° to 300° C	300° to 400° C	400° to 500° C	500° to 600° C	600° to 700° C	
	<i>Cone</i>	<i>hr</i>								%
26 ex	4	16	5.2	6.5	7.4	7.9	8.1	11.3	5.9	0.511
pr	4	16	4.7	6.7	7.0	7.1	8.2	10.4	6.3	.493
ex	4	24	5.2	6.4	7.2	7.4	8.3	10.9	5.8	.500
pr	4	24	5.1	6.3	7.0	7.0	8.2	10.7	5.9	.490
ex	6	9	4.7	5.5	6.2	6.6	7.6	9.1	5.4	.440
pr	6	9	4.6	6.5	6.6	6.9	8.2	10.9	6.5	.491
ex	6	16	4.8	6.2	6.7	6.9	7.9	10.1	5.6	.472
pr	6	16	4.3	6.4	6.5	7.3	7.5	8.7	5.4	.452
ex	6	24	4.2	6.9	6.7	7.2	8.2	10.3	5.2	.478
pr	6	24	4.6	5.8	5.9	6.2	6.9	8.9	5.3	.425
ex	8	24	3.1	4.0	4.6	5.0	6.1	7.6	3.3	.330
27 ex	6	9	3.3	4.3	4.9	4.9	5.8	7.2	4.1	.338
pr	6	9	3.1	4.8	4.8	5.1	6.6	7.3	4.4	.353
ex	6	16	3.2	3.8	3.9	5.2	5.9	7.3	4.7	.333
pr	6	16	4.0	4.1	4.9	5.5	6.2	7.4	4.1	.351
ex	6	24	3.7	4.6	5.0	5.5	6.0	7.7	4.5	.362
pr	6	24	3.3	4.4	4.8	5.2	6.0	7.2	4.0	.341
ex	8	24	2.8	3.4	3.7	4.3	4.7	5.4	3.9	.276
32 ex	6	9	5.0	5.9	6.6	7.0	7.8	9.8	5.4	.464
pr	6	9	5.5	6.7	6.8	7.9	8.0	11.4	5.5	.504
ex	6	16	5.0	5.9	5.9	7.0	8.1	10.3	5.3	.464
pr	6	16	5.2	7.5	7.1	6.9	8.0	9.8	5.7	.491
ex	6	24	4.6	6.0	5.5	7.9	8.1	8.8	5.8	.457
pr	6	24	5.0	6.9	7.6	7.5	8.3	10.7	5.8	.506
33 ex	6	9	4.9	7.4	7.2	7.1	8.2	9.5	5.4	.486
ex	6	16	5.3	7.2	6.4	7.5	8.9	11.2	5.8	.512
ex	6	24	5.1	8.8	7.7	7.4	8.3	9.9	5.9	.520
34 ex	6	9	5.2	7.6	7.4	7.0	8.2	9.2	5.2	.487
ex	6	16	5.8	7.4	7.6	7.6	8.3	12.0	5.5	.539
ex	6	24	6.0	8.5	7.8	7.6	8.5	10.7	5.1	.529
35 ex	4	16	5.2	7.5	7.5	7.3	8.8	12.0	5.7	.528
pr	4	16	5.6	8.4	7.5	7.5	8.6	11.9	5.0	.532
ex	4	24	6.4	8.6	7.9	7.4	8.4	12.5	5.9	.556
pr	4	24	6.0	8.0	7.6	7.2	8.0	10.6	5.8	.517
ex	6	9	5.0	7.8	6.5	6.1	7.1	9.1	5.1	.457
pr	6	9	6.0	8.0	7.5	6.9	8.1	10.4	5.5	.509
ex	6	16	5.6	8.8	8.0	7.0	7.9	10.3	5.4	.518
pr	6	16	6.0	10.0	8.8	7.0	8.3	10.7	5.3	.547
ex	6	24	5.9	9.8	9.4	6.9	7.1	10.2	6.1	.541
pr	6	24	6.4	9.8	7.7	6.9	8.1	12.0	4.6	.541
36 ex	6	9	5.2	8.1	7.9	7.7	9.1	10.2	4.9	.520
ex	6	16	6.1	11.0	7.7	6.9	7.8	10.5	5.4	.540
ex	6	24	6.0	11.8	8.0	6.8	7.7	9.8	5.4	.542

\* The "ex" indicates bodies that were hand-wedged and extruded, and "pr" indicates so-called dry-pressed bodies. For the amounts of water used, see table 2.

<sup>b</sup> The maximum temperatures of cone 4, cone 6, and cone 8 are approximately equivalent to 1,165°, 1,190°, and 1,225° C, respectively. For details regarding the 9-, 16-, and 24-hour schedules, see figure 4. These specimens were heated on the manually controlled kiln schedules.

#### IV. DISCUSSION AND CONCLUSIONS

To depend on the judgment of the operator for the determination of optimum workability of a plastic ceramic body in the preparation of laboratory specimens is open to criticism. In this study, the same operator, working with parts of the same compositions, varied the amount of "water of plasticity" used in one body over 7 percent, and the average variation for the 12 bodies investigated was 3.5 percent. Yet the most recently published compilation of methods for testing

clays states<sup>13</sup> simply that "The plastic clay body is prepared \* \* \* by mixing it with the required water of plasticity, \* \* \*." As yet there is no generally accepted mechanical means for standardizing the workability of ceramic bodies.

Unfortunately, the data do not distinguish the individual effects of variations in water of plasticity, of hand-wedging, and of deairing. They do show unmistakably that the uniformity of specimens, as indicated by the values for absorption and for transverse strength, is improved by deairing.

Pressed specimens are easily prepared, even with bodies having relatively poor workability, and the precision of the results compares favorably with that for deaired and extruded bodies.

The progress of vitrification and the final strength of pressed and extruded specimens may differ in important degree. Pressed laboratory specimens should not, therefore, be used to represent compositions which are formed commercially from plastic masses, and vice versa.

Transverse strength values showed the most pronounced differences of any of the properties determined, resulting from the three forming methods (pressing; hand-wedging and extrusion; deairing and extrusion). Deaired specimens averaged 3,100 lb/in.<sup>2</sup> higher in modulus of rupture than the pressed, and 2,300 lb/in.<sup>2</sup> higher than the hand-wedged.

Specimens were heated to cone 4 on 16- and 24-hour schedules, and to cone 6 on 9-, 16-, and 24-hour schedules. About one-third of the specimens showed the higher transverse strength after the shortest schedule both to cone 4 and to cone 6. Additional data were presented to support the theory that this effect was the result of the comparatively rapid cooling on the short schedule.

The average strength of the pressed bodies ranged from 5,400 to 9,400 lb/in.<sup>2</sup> for those heated to cone 4 and from 6,000 to 10,000 lb/in.<sup>2</sup> for those heated to cone 6. Corresponding values for the hand-wedged bodies are 6,400 to 11,000 lb/in.<sup>2</sup> and 7,100 to 12,100 lb/in.<sup>2</sup>. For the deaired bodies, they are 8,700 to 12,600 lb/in.<sup>2</sup> and 9,700 to 13,500 lb/in.<sup>2</sup>.

The shrinkage was not greatly affected by the various heat treatments (usually less than 1 percent total range).

The variations in heat treatment had little effect on the extensibility. The averages for the 12 bodies ranged from  $9.2$  to  $9.9 \times 10^{-4}$ .

After heating to cone 6 on the 9-hour schedule, all of the specimens were vitrified, but, after heating to cone 4, only one pressed body and five extruded bodies were vitrified, or nearly so (less than 1-percent absorption). The change in absorption "from cone 4 to cone 6" is appreciably greater for the pressed bodies than for the extruded bodies. Several bodies showed increase in absorption with increase in the length of the heating schedule when heated to the same cone. There is no reason to believe that this is caused by so-called overfiring.

WASHINGTON, October 14, 1940.

<sup>13</sup> J. Am. Ceramic Soc. 11 445 (1928).