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LENGTH CHANGES OF WHITEWARE CLAYS AND BODIES DURING INITIAL HEATING, WITH SUPPLEMENTARY DATA ON MICA

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ABSTRACT

Changes in length during initial heating from room temperature to a maximum of 1,250° C were observed on nine kaolins, four ball clays, and three whiteware bodies. The effects of various forming methods, admixtures, and heating rates were observed also. Supplementary data were obtained in the form of differential heating curves for two clays, and of specific gravity and weight-loss determinations for muscovite mica. Mica exerts the greatest influence of any one factor below 1,000° C, and particle size is the predominating factor above this temperature.

CONTENTS

I. INTRODUCTION

The work of Steger [1] and others [2] on the length changes occurring in whiteware clays and in body mixtures during initial heating indicates that interesting information could be obtained by similar studies on the American clays and on body mixtures containing them.

It was thought possible that some explanation might be forthcoming also for certain effects of various heating rates on absorption and shrinkage. In 1936, Watts [3] published values for five whiteware bodies heated to "cone 10 nearly down" in 76 hr and in 108 hr. He found that the shorter schedule produced from 0.20 to 0.42 percent greater shrinkage, from 0.70 to 1.07 percent lower absorption, and from 300 to 600 lb/in.^{2} higher transverse strength. Rittgen [4] supports

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[1] W. Steger, Ber. deut. keram. Ges. **19**, 2 (1938).

[2] R. Schwarz and Wm. Klös, Z. anorg. allgem. Chem. **169**, 213 (1931). L. Tscheischwili, W. Bussem and W. Weyl, Ber. deut. keram. Ges. **20**, 249 (1939).

[3] Arthur S. Watts, J. Am. Ceram. Soc. **19**, 175 (1936).

[4] A. Rittgen, Ber. deut. keram. Ges. **19**, 113 (1938).

this finding in an article on the development of electric tunnel kilns. He found that the average porosity for the bodies he investigated was about 9 percent after a 130-hr cycle to a maximum temperature of 1,280° C in a periodic kiln, and 8 percent after a 108-hr cycle to 1,270° C and 6 percent after a 54-hr cycle to 1,280° C in tunnel kilns. Shrinkage was reported as about the same during each cycle.

II. MATERIALS

The three body mixtures tested included two talcose bodies prepared in the laboratory and one commercial "semiporcelain" body. The compositions of the laboratory bodies are given in table 1. Available chemical compositions of the clays are given in table 2.

TABLE 1.—*Batch composition of laboratory-prepared bodies*

Body number	Talc, *L*	Fla. Kaolin, *U*	N. C. kaolin, *K*	Tenn. ball clay, *CC*	Tenn. ball clay, 5	Ky. ball clay, 4	Potash feldspar, *D*	Flint, *P*
	%	%	%	%	%	%	%	%
27	25	5	20		7.5	7.5	15	20
35	20	26.7	--------	21.3	--------	--------	10.7	21.3

TABLE 2.—*Chemical composition and major mineral constituents of clays investigated* [a]

[tr=trace, nd=no determination]

Material	Laboratory designation	K_2O	Na_2O	CaO	MgO	Fe_2O_3	TiO_2	SiO_2	Al_2O_3	Ignition loss	Drying loss	Feldspar and mica	Kaolinite	Free silica
		%	%	%	%	%	%	%	%	%	%	%	%	%
German kaolin (Zettlitz) [b]	*Z*	0.84	0.44	0.29	0.27	0.65	0.18	46.9	37.4	12.9	-----	11.9	84.8	1.0
English china clay [c]	*P*	1.6	.2	.2	.2	.9	.2	47.0	37.7	12.4	-----	16.2	80.4	1.7
Vermont kaolin:														
Coarse fraction [d]	*Vc*	3.60	----	----	----	.98	1.33	63.92	22.78	6.54	0.53	30.5	27.9	37.1
Fine fraction [d]	*Vf*	1.62	----	----	----	.49	1.01	47.4	36.5	12.35	.58	13.7	79.0	4.5
North Carolina kaolin [e]	*K*	0.58	.10	.37	.42	.57	0.04	46.18	38.38	13.28	-----	7.6	90.1	0.3
Georgia "soft" kaolin [f]	*S*	.15	.10	nd	nd	.27	1.54	45.3	39.1	13.7	-----	2.1	97.2	-----
Georgia hard kaolin:														
Coarse fraction [g]	*Gc*	-----	.21	----	----	.19	1.31	45.37	38.59	13.09	1.12	1.8	96	-----
Fine fraction [g]	*Gf*	-----	.18	----	----	.21	1.76	45.06	37.84	12.75	2.09	1.5	97	-----
Florida kaolin [h]	*E*	.2	0.0	.5	tr	.8	0.5	46.3	37.7	13.7	-----	4.5	91.2	2.0
Do.[h]	*U*	.2	0.0	tr	0.0	.4	.2	46.9	38.5	13.8	-----	1.5	95.8	1.7
Alabama kaolin [i]	*T*	-----	----	(j)	.2	.49	1.44	43.5	41.2	13.7	1.26	1	93	(n)
Tennessee ball clay [k]	5	2.0	.3	.2	.3	.6	1.3	50.3	31.5	13.6	-----	20.5	61.0	11.7
Do.[l]	*J*	1.84	.22	.22	.61	1.07	1.21	53.57	29.18	11.55	-----	17.8	57.7	17.7
Do.[l]	*CC*	0.21	.08	.25	.39	0.98	1.75	57.07	27.86	11.32	-----	2.9	68.4	23.6
Kentucky ball clay [k]	4	1.2	.2	.2	.3	.6	1.5	49.9	31.4	14.7	-----	12.9	67.7	11.9
Talc [m]	*L*	nd	nd	6.9	30.5	nd	nd	55.9	1.3	5.4	-----	-----	-----	-----
Feldspar	*D*	13.4	2.0	nd	nd	nd	nd	65.6	18.4	-----	-----	-----	-----	-----

[a] Mineral compositions calculated by the method of H. S. Washington, J. Am. Ceram. Soc. **1,** 405 (1918) and assuming K_2O present as muscovite mica.

[b] For complete analysis see BS Tech. News Bul. **138,** p. 146 (1928).

[c] From J. Research NBS **19,** 65 (1937) RP1011.

[d] Composition supplied by S. C. Lyons. Alkalies reported as K_2O.

[e] Composition supplied by Kaolin Incorporated. Analysis includes also 0.08 percent of ZrO_2.

[f] From BS J. Research **11,** 327 (1933) RP594.

[g] Composition supplied by S. C. Lyons. Alkalies reported as Na_2O.

[h] From J. Am. Ceram. Soc. **18,** 163 (1935). Value for TiO_2 includes ZrO_2.

[i] Composition of clay from same deposit as sample tested, supplied by Bureau of Mines.

[j] Spectographic determinations indicated 0.17% of ZrO_2, 0.15% of CaO.

[k] From J. Am. Ceram. Soc. **21,** 371 (1938).

[l] Composition supplied by the H. C. Spinks Clay Co. Table does not include values determined for FeS_2, CO_2, P_2O_5, SO_3, and MnO.

[m] From J. Am. Ceram. Soc. **18,** 259 (1935).

[n] Excess Al_2O_3 may be present as bauxite.

The study was amplified to include several clays in addition to those used in bodies 27 and 35, as follows:

Georgia "soft" kaolin used in a previous study.[5]

Two fractions (*Gf* and *Gc*) of a Georgia "hard" kaolin supplied by S. C. Lyons.[6] Approximately 97 percent of fraction *Gf* consisted of particles 2 microns and smaller in diameter, whereas about 50 percent of fraction *Gc* was coarser than 7 microns. Diffraction patterns of each fraction showed identifiable lines of kaolinite only.

Two fractions of a Vermont kaolin were supplied by S. C. Lyons, who reports the following particle-size analyses:

Finer than	Coarse fraction (*Vc*)	Fine fraction (*Vf*)
Microns	%	%
10	28	100
4	8	98
1	6	60

X-ray diffraction patterns showed unmistakable lines for mica, quartz, and kaolinite in the coarse fraction. Mica lines were not discernible in the pattern for the fine fraction. (The patterns were made and interpreted by H. F. McMurdie.)

Florida kaolin designated *E* in table 2.[7]

Zettlitz kaolin, a portion of the international standard material described in the National Bureau of Standards Technical News Bulletin 138 (1928) and for which a heating curve has been published.[8]

English china clay, additional values for which have been published.[9]

Tennessee ball clay (designated *J* in table 2) supplied by Hewitt Wilson.

Alabama kaolin (designated *T* in table 2) supplied by Hewitt Wilson.

The bentonite used as an admixture was estimated, by petrographic examination, to be approximately 80 percent montmorillonite. (Examination by H. Insley.)

The mica used as an admixture was muscovite and had been passed dry through a No. 325 U. S. Standard Sieve.

The feldspar used as an admixture had been reground, and at least 95 percent of the particles were under 10 microns in diameter.

III. METHODS

Specimens for length-change determinations were equilateral triangular prisms, approximately 0.5 cm^2 in cross-sectional area and 5 cm long. They were obtained by grinding pieces which had been formed from plastic masses or by pressing. The pressed specimens

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[5] R. F. Geller, D. N. Evans, and A. S. Creamer, BS J. Research **11**, 327 (1933) RP594.

[6] S. C. Lyons, Bul. Am. Ceram. Soc. **18**, 321 (1939). The fine and coarse fractions are identified by the letters *D* and *E*, respectively, in his publication.

[7] This clay is designated by the letter X in the report by T. A. Klinefelter and W. W. Meyer, J. Am. Ceram. Soc. **18**, 163 (1935).

[8] Herbert Insley and Raymond H. Ewell, J. Research NBS **14**, 615 (1935) RP792.

[9] William W. Meyer and Theron A. Klinefelter, J. Research NBS **19**, 65 (1937) RP1011.

were usually made up with 15 percent of water, and the pressure was approximately 2,400 lb/in.^{2}, or 170 kg/cm.^{2} The length of each specimen was determined directly, before and after the tests, by measurements at each of the three long edges with a vernier caliper reading to 0.02 mm. By this means any appreciable deformation of a specimen could be detected.

The apparatus for measuring the length changes was adapted for use in an electrically heated furnace in which the heating chamber is a porcelain tube 63 cm long and 2 cm inside diameter. The platinum resistance winding extends over about 16 cm of the central portion of the tube. The device (fig. 1) is, in principle, the same as the fused-quartz dilatometer described by Hidnert and Sweeney,[10] the fused quartz being replaced by porcelain. A tripod of porcelain rods supports a gage reading to 0.01 mm, and a fourth porcelain rod extends from the top of the specimen to the stem of the gage. Principally because of the spring pressure in this gage, the specimens were under a total load varying from approximately 150 g at the beginning to 100 g at the end of the test. The arrangement, which will be referred to as the porcelain dilatometer, was calibrated by using specimens of fused MgO. This MgO was taken from the same shipment of material used by Heindl [11] in his study of thermal expansions to 1,800° C. For the calibration tests, appropriate specimens for the porcelain dilatometer, and also for the interferometer, were formed by using finely ground material (through a 400-mesh sieve) and a solution of gum tragacanth. The dried specimens were calcined and then heated to 1,750° C in an Arsem furnace. The rates of heating and cooling used in the calibration tests, and in the later tests of clays and bodies, are shown in figure 2.

In addition to calibrating the dilatometer, the net shrinkage indicated by the gage readings could be checked by comparison with the shrinkage obtained directly from measurements of the prisms before and after the test. It was found that the total length change of clay specimens as shown by the gage was consistently about 0.3 percent greater than that calculated from the direct measurements. This discrepancy was independent of the amount of shrinkage and is believed to have been caused by "adjustments" of the contacts at the ends of the specimen, probably during the latter portion of the test. By adjustments is meant the probable conforming of the ends of the specimens, which were comparatively uneven because of the friable nature of dry clay, to the contour of the top and bottom dilatometer contacts.

Tests with the MgO indicated that the corrections to be applied to the gage readings are the same as the thermal-expansion values for the porcelain rods, as they should be theoretically. For readings obtained at temperatures above the upper limit of the interferometer, it was necessary to extrapolate the curve for the porcelain rods. Since Heindl's values above the range of the interferometer also are based on an extrapolated correction curve, one cannot say with certainty which set of results more nearly represents the true expansion of MgO. Naturally, all of the data presented here for length changes obtained with the porcelain dilatometer involve the same extrapolated correction values.

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[10] Peter Hidnert and W. T. Sweeney, BS J. Research **1,** 771 (1928) RP29.

[11] R. A. Heindl, BS J. Research **10,** 715 (1933) RP562.

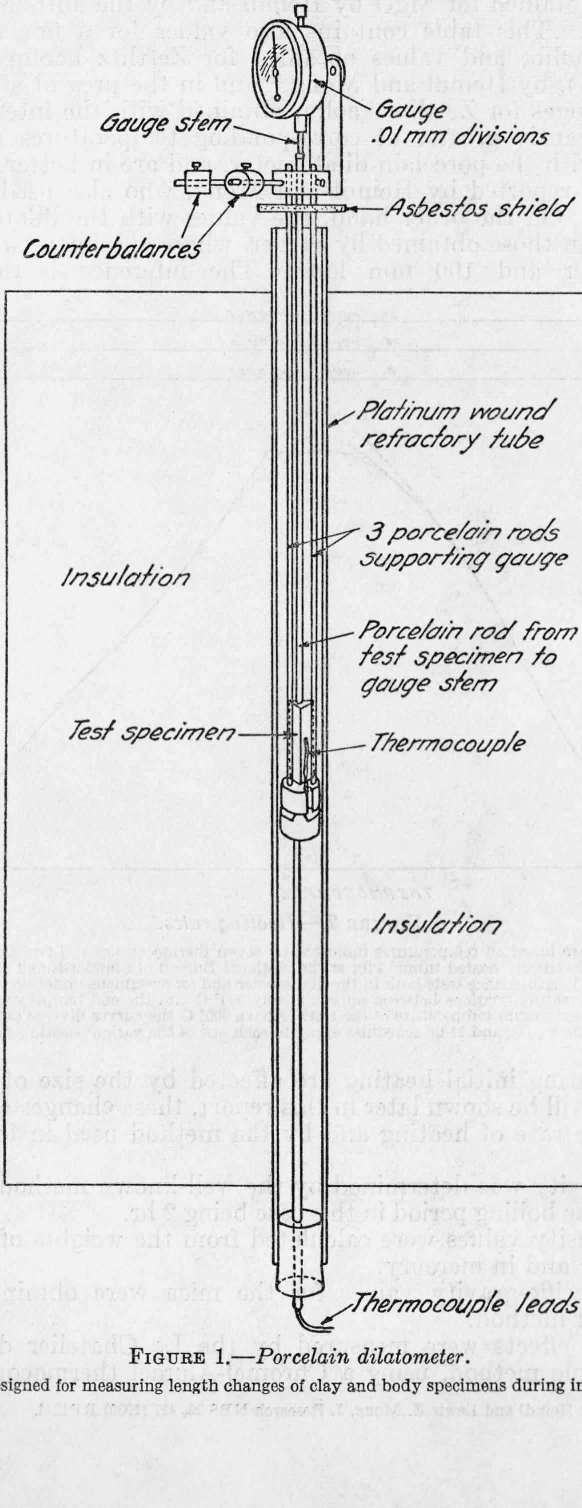

FIGURE 1.—*Porcelain dilatometer.*

Apparatus designed for measuring length changes of clay and body specimens during initial heating.

Values obtained for MgO by Heindl and by the authors are given in table 3. This table contains also values for a fine fraction of Georgia kaolin, and values obtained for Zettlitz kaolin by Steger (footnote 1), by Heindl and Mong,[12] and in the present study. The length changes for Zettlitz kaolin, obtained with the interferometer, are significantly greater at corresponding temperatures than those obtained with the porcelain dilatometer, and are in better agreement with those reported by Heindl and Mong, who also used the interferometer. On the other hand, the values with the dilatometer are greater than those obtained by Steger, whose specimens were 10 mm in diameter and 100 mm long. The inference is that length changes during initial heating are affected by the size of the specimen. As will be shown later in this report, these changes are affected also by the rate of heating and by the method used in forming the specimens.

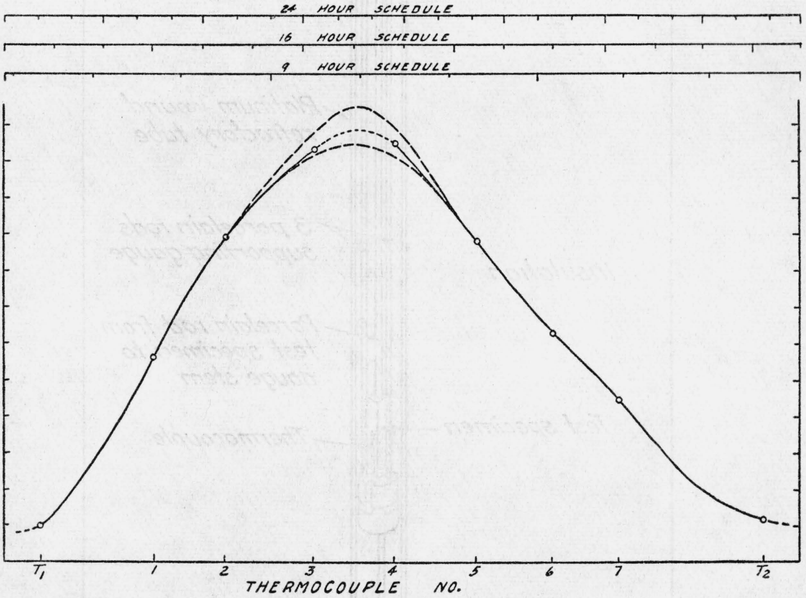

Figure 2.—*Heating rates.*

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 length-change tests both in the dilatometer and for specimens under no load.

The time-temperature relations between approximately 900° C and the end temperatures 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 one of the various heating curves.

Absorptivity was determined by the well-known method of boiling in water, the boiling period in this case being 2 hr.

Bulk-density values were calculated from the weights of the specimens in air and in mercury.

True specific-gravity values for the mica were obtained by the pycnometer method.

Thermal effects were measured by the Le Chatelier differential-thermocouple method, using a Chromel-Alumel thermocouple and a

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[12] Raymond A. Heindl and Lewis E. Mong, J. Research NBS **23,** 427 (1939) RP1243.

type K potentiometer, artificial alpha-alumina as the reference material and a heating rate of 2°C/min.

Table 3.—*Length-change values obtained with the interferometer and with the porcelain dilatometer in this investigation, compared with results obtained by other investigators*

[Values preceded by (−) indicate contraction]

From room temperature to—	Porcelain [a]	Electrically fused MgO						Kaolin *Z* (Zettlitz)					Kaolin *Gf* (fine fraction)	
		[b]	Schedule				Heindl [d]	Schedule 16 hr [b]		Heindl and Mong [b e]	Steger [f]	[e]		
			9 hr [c]		24 hr [c]									
			(1)	(2)	(1)	(2)		(1)	(2)				[b]	[e]
°*C*	%	%	%	%	%	%	%	%	%	%	%	%	%	%
100	0.020	0.070	0.08	0.06	0.07	-----	0.08	0.03	−0.10	--------	-----	0.02	−0.05	0.03
200	.055	.190	.19	.17	.19	0.19	.19	.07	−.02	0.0	0.1	.08	−.10	.04
300	.095	.317	.32	.31	.32	-----	.31	.18	.07	.05	-----	.15	.00	.10
400	.140	.446	.45	.45	.45	.45	.45	.27	.15	.14	.2	.20	.07	.17
500	.190	.582	.59	.58	.57	-----	.59	(h)	(i)	(j)	(k)	(l)	(m)	(n)
600	.245	.721	.72	.72	.72	.72	.73	−.82	−1.00	−.78	.1	−.20	−1.30	.55
700	.300	.866	.89	.86	.86	-----	.87	−1.22	−1.40	−1.10	−.4	−.67	−1.98	−1.20
800	.355	1.012	1.02	1.00	1.01	1.14	1.00	−1.52	−1.70	−1.43	−.7	−.93	−2.50	−1.58
900	.410	1.162	1.17	1.13	1.14	-----	1.15	−1.85	−2.10	−1.74	−.8	−1.10	−2.85	−1.87
1,000	(.470)	-----	1.31	1.27	1.27	1.27	1.30	-----	−3.65	−3.22	−1.8	−2.63	−4.40	−3.16
1,100	(.530)	-----	1.47	1.41	1.41	1.41	1.47	-----	-----	--------	−2.4	−3.50	-----	−3.54
1,200	(.590)	-----	1.62	1.59	1.53	1.54	1.62	-----	-----	--------	−6.5	−8.66	-----	−7.63
1,250	(.620)	-----	-----	1.62	-----	1.59	1.70							
R. T.[g]	-----	-----	.03	.02	.04	.06	--------	-----	-----	--------	-----	-----	-----	-----

[a] Values obtained with the interferometer and used to correct the gage readings. Values given parenthetically are extrapolated.
[b] Values obtained with the interferometer. Columns (1) and (2) indicate duplicate tests.
[c] Values obtained with the porcelain dilatometer.
[d] BS J. Research **10**, 715 (1933) RP562.
[e] J. Research NBS **23**, 427 (1939) RP1243.
[f] Ber. deut. keram. Ges. 19, 2 (1938).
[g] Reading upon return to room temperature.
[h] Maximum expansion 0.39% at 498°.
[i] Maximum expansion 0.26% at 496°.
[j] Maximum expansion 0.33% at 545°
[k] Maximum expansion 0.3% at 560°.
[l] Maximum expansion 0.30% at 520°.
[m] Maximum expansion 0.14% at 508°.
[n] Maximum expansion 0.25% at 505°.

IV. RESULTS

1. CLAYS

The curves in figure 3 show the characteristic differences in length changes between three typical primary kaolins (*Z*, *P*, and *K*) and four secondary kaolins (*S*, *E*, *U*, and *T*). In figure 4, which shows the length changes for three ball clays, the curves for kaolins *P* and *E* are repeated to emphasize the similarity of the ball clays to the primary kaolins in the range from room temperature to 1,000° C.

The curves in figure 5, drawn on an enlarged vertical scale, show more clearly how the behavior of kaolin *K* differs from the others shown in figures 3 and 4. These curves show also the effect of predrying at 110° versus air-drying (room temperature), and of plastic forming versus "dry-press" forming.

In figure 6, the curves shown as continuous lines were taken from reports by Steger (footnote 1) and by Rieke and Wen.[13] Curves for kaolin *K* and ball clay *CC* are given to facilitate comparison. By calculation, kaolin *K* contains 7.6 percent of feldspar and mica (table 2), of which 4.9 percent is assigned to mica. The informa-

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[13] R. Rieke and Pu-Yi Wen, Ber. deut. keram. Ges. **20**, 43 (1939).

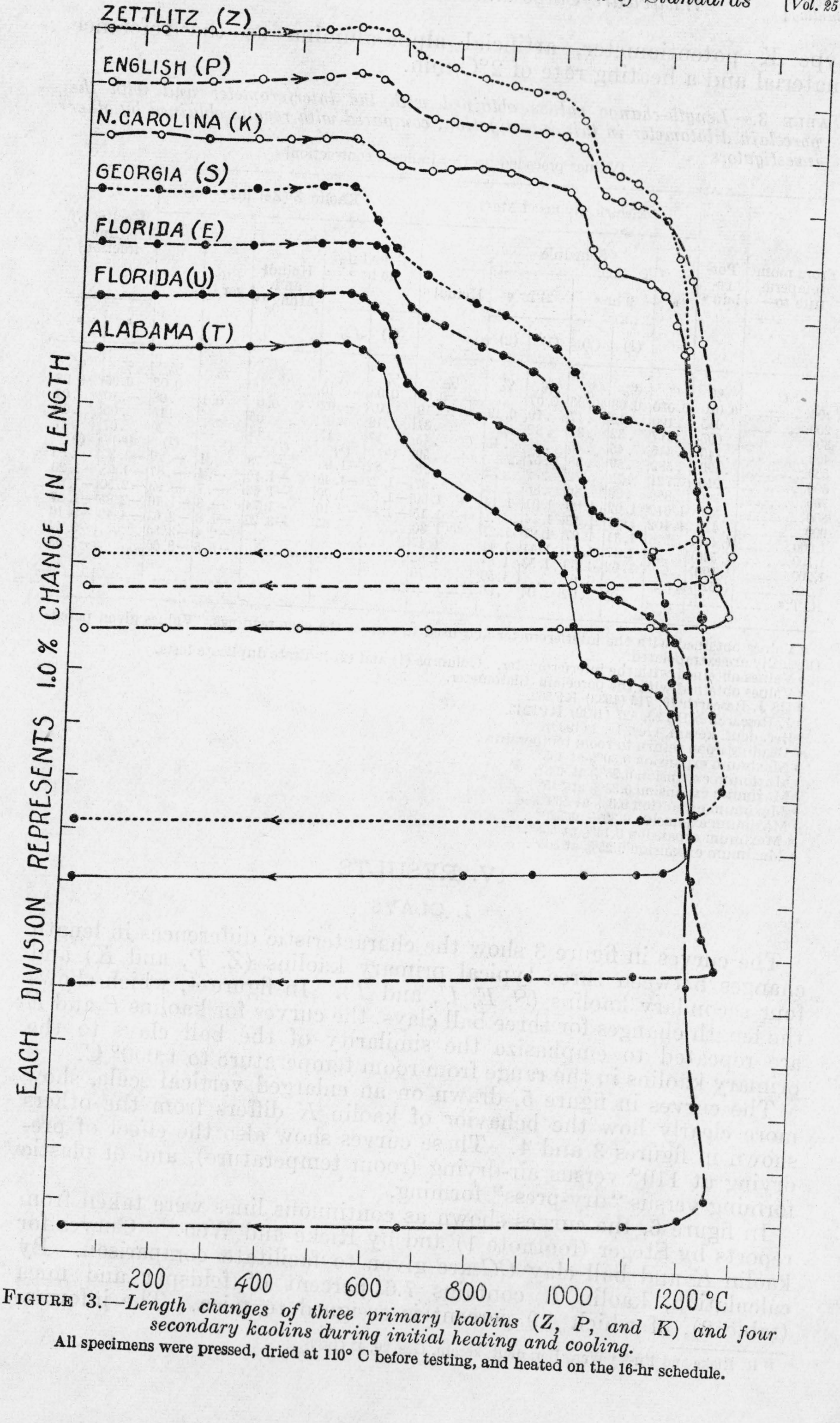

FIGURE 3.—*Length changes of three primary kaolins (Z, P, and K) and four secondary kaolins during initial heating and cooling.*

All specimens were pressed, dried at 110° C before testing, and heated on the 16-hr schedule.

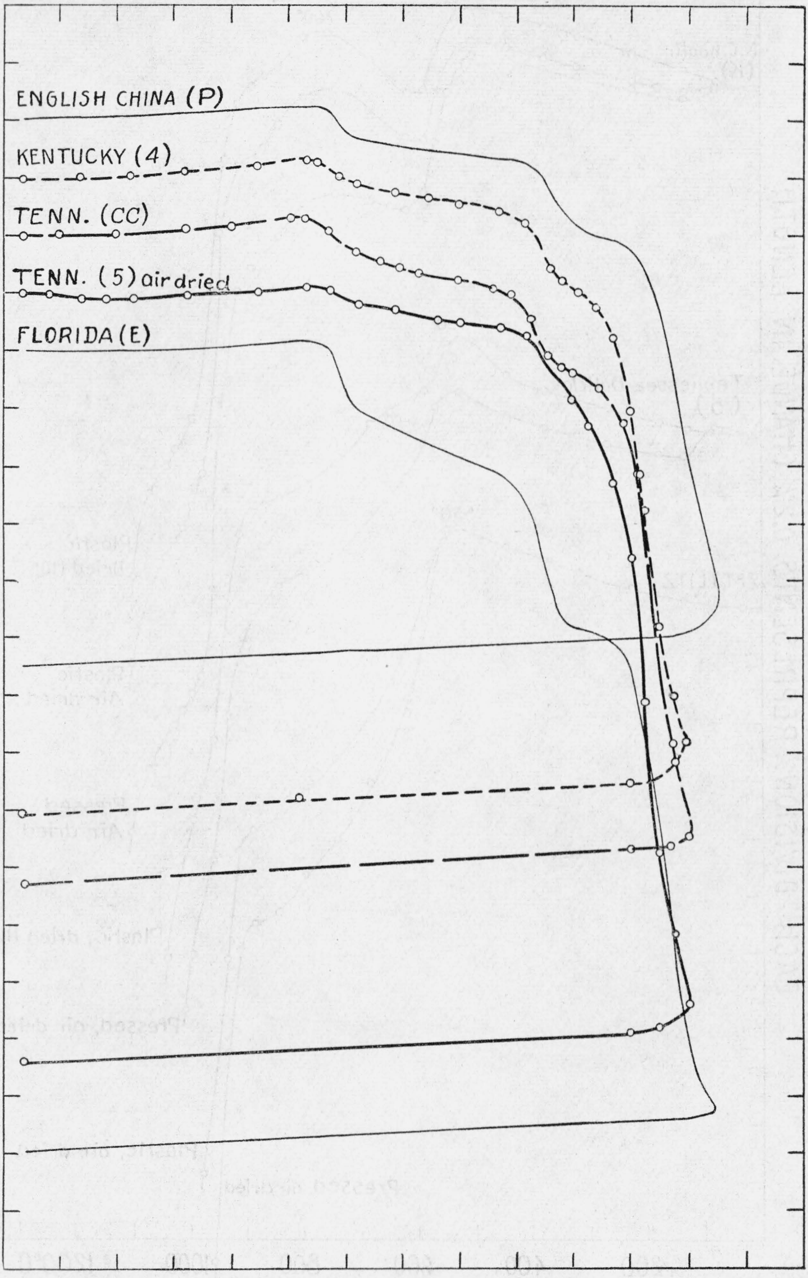

Figure 4.—*Length changes of three ball clays during initial heating and cooling.*

Curves for kaolins *P* and *E* are repeated from figure 3 to facilitate comparison. All specimens were pressed and, excepting clay 5, dried at 110° C before testing. Clays 4 and 5 were heated on the 16-hr schedule and clay *CC* on the 24-hr schedule.

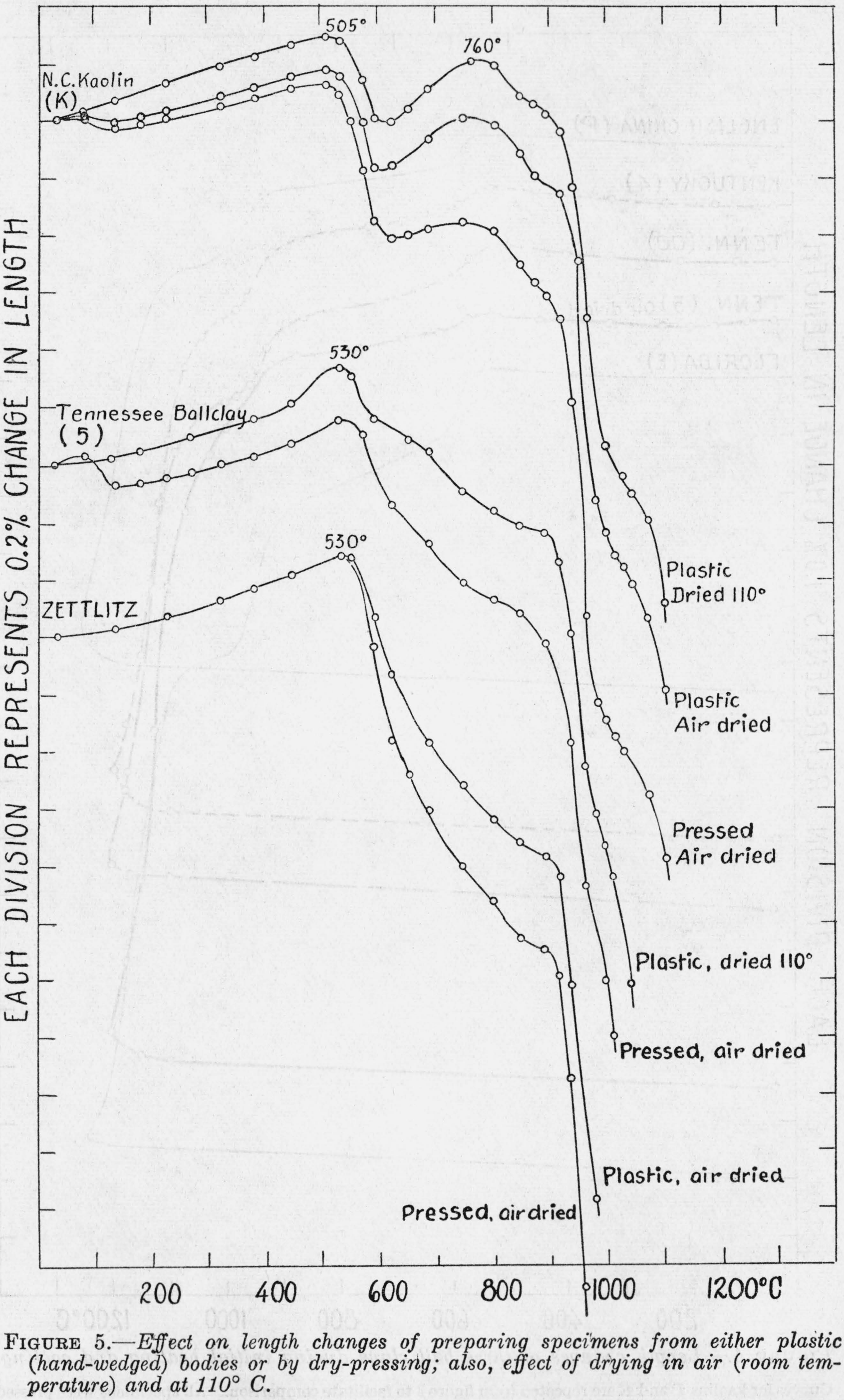

FIGURE 5.—*Effect on length changes of preparing specimens from either plastic (hand-wedged) bodies or by dry-pressing; also, effect of drying in air (room temperature) and at 110° C.*

The clays were heated on the 16-hr schedule. Note the expansion of kaolin *K* between 600° and 760° C.

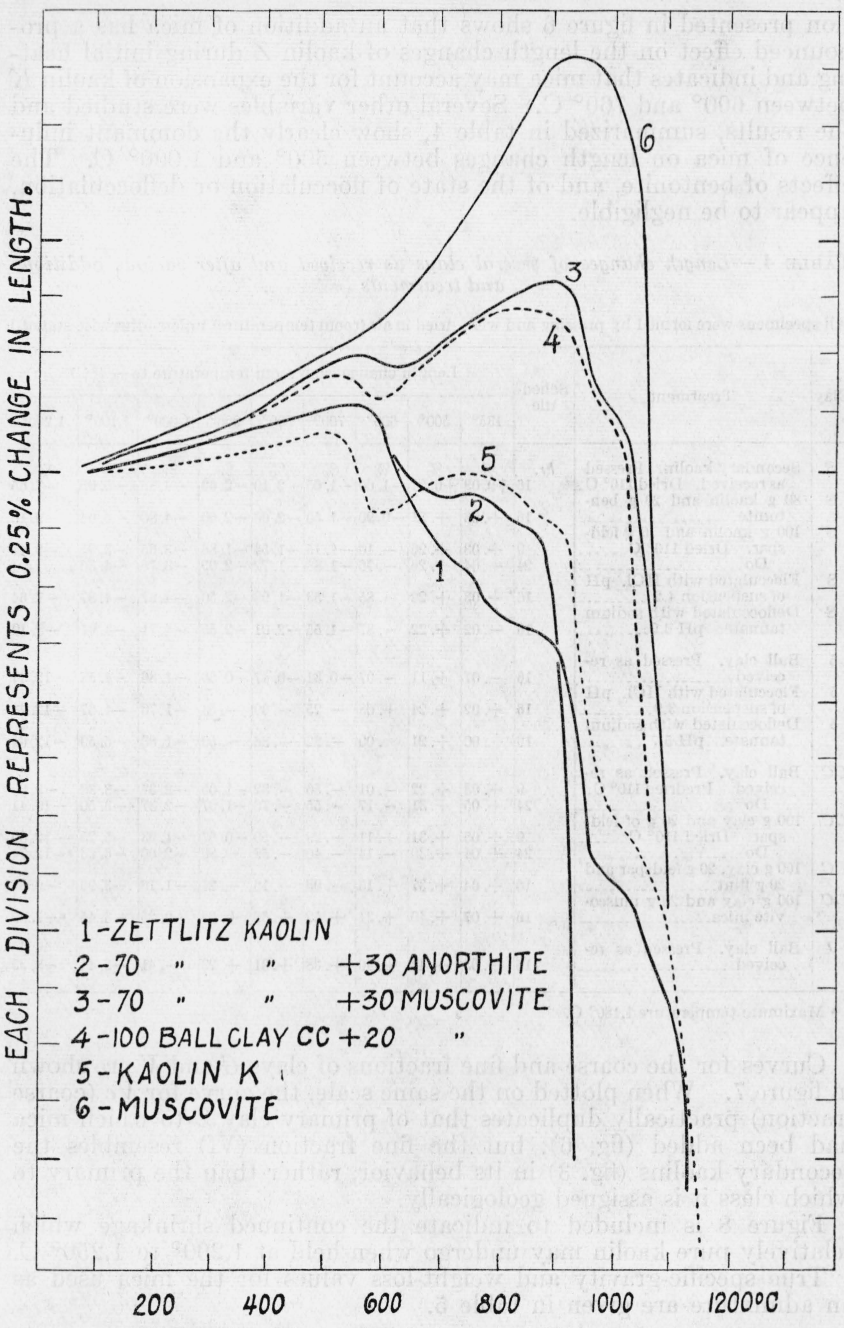

FIGURE 6.—*Effect on length change of additions of mica.*

A curve for kaolin *K* is repeated to facilitate comparison. Curves 1, 2, and 3 are taken from a report by W. Steger (Ber. deut. keram. Ges. 19, 2, 1938) and curve 6 from a report by R. Rieke and Pu-Yi Wen (Ber. deut. keram. Ges. 20, 43, 1939).

tion presented in figure 6 shows that an addition of mica has a pronounced effect on the length changes of kaolin *Z* during initial heating and indicates that mica may account for the expansion of kaolin *K* between 600° and 760° C. Several other variables were studied and the results, summarized in table 4, show clearly the dominant influence of mica on length changes between 500° and 1,000° C. The effects of bentonite, and of the state of flocculation or deflocculation, appear to be negligible.

TABLE 4.—*Length changes of several clays as received and after various additions and treatments*

[All specimens were formed by pressing and were dried in air (room temperature) unless otherwise stated.]

Clay	Treatment	Schedule	Length change from room temperature to — (°C)								
			135°	500°	600°	700°	800°	900°	1,000°	1,100°	1,200°
		hr.	%	%	%	%	%	%	%	%	%
S	Secondary kaolin. Pressed as received. Dried 110° C	16	+0.03	+0.21	−1.00	−1.65	−2.10	−2.62	−4.85	−5.05	−7.65
S	80 g kaolin and 20 g bentonite	16	+.03	+.21	−0.90	−1.50	−2.00	−2.60	−4.80	−5.04	−9.16
S	100 g kaolin and 10 g feldspar. Dried 110° C	9	+.03	+.26	−.10	−1.15	−1.54	−1.86	−3.65	−3.95	−9.41
	Do	24	+.04	+.26	−.75	−1.88	−1.72	−2.05	−3.75	−4.30	-------
S	Flocculated with HCl. pH of suspension 4.2	16	+.03	+.22	−.85	−1.53	−1.98	−2.50	−4.67	−4.87	−7.64
S	Deflocculated with sodium tannate. pH 6.9	16	−.02	+.22	−.87	−1.55	−2.01	−2.55	−4.71	−4.91	−8.10
5	Ball clay. Pressed as received	16	−.07	+.11	−.07	−0.31	−0.47	−0.66	−1.86	−4.58	−12.34
5	Flocculated with HCl. pH of suspension 3.9	16	+.02	+.24	+.03	−.25	−.39	−.53	−1.76	−4.67	−12.23
5	Deflocculated with sodium tannate. pH 5.7	16	.00	+.21	.00	−.22	−.36	−.50	−1.83	−5.59	−12.64
CC	Ball clay. Pressed as received. Predried 110° C	9	+.03	+.22	−.01	−.56	−.82	−1.05	−2.37	−3.30	−9.90
	Do	24	+.05	+.31	−.17	−.55	−.76	−1.07	−2.37	−3.50	−10.41
CC	100 g clay and 20 g of feldspar. Dried 110° C	9	+.06	+.31	+.11	−.29	−.50	−0.67	−1.93	−5.25	[a]−12.00
	Do	24	+.04	+.30	−.14	−.40	−.59	−.80	−2.06	−6.93	[a]−12.74
CC	100 g clay, 20 g feldspar and 20 g flint	16	+.04	+.37	+.13	−.06	−.16	−.27	−1.16	−3.96	−9.85
CC	100 g clay and 20 g muscovite mica	16	+.07	+.40	+.31	+.52	+.69	+.66	+0.25	−3.45	[a]−7.49
J	Ball clay. Pressed as received	16	+.05	+.43	+.40	+.38	+.31	+.23	−.41	−2.44	−8.35

[a] Maximum temperature 1,180° C.

Curves for the coarse and fine fractions of clays *G* and *V* are shown in figure 7. When plotted on the same scale, the curve for *Vc* (coarse fraction) practically duplicates that of primary clay *Z* to which mica had been added (fig. 6), but the fine fraction (Vf) resembles the secondary kaolins (fig. 3) in its behavior, rather than the primary to which class it is assigned geologically.

Figure 8 is included to indicate the continued shrinkage which relatively pure kaolin may undergo when held at 1,200° to 1,250° C.

True specific-gravity and weight-loss values for the mica used as an admixture are given in table 5.

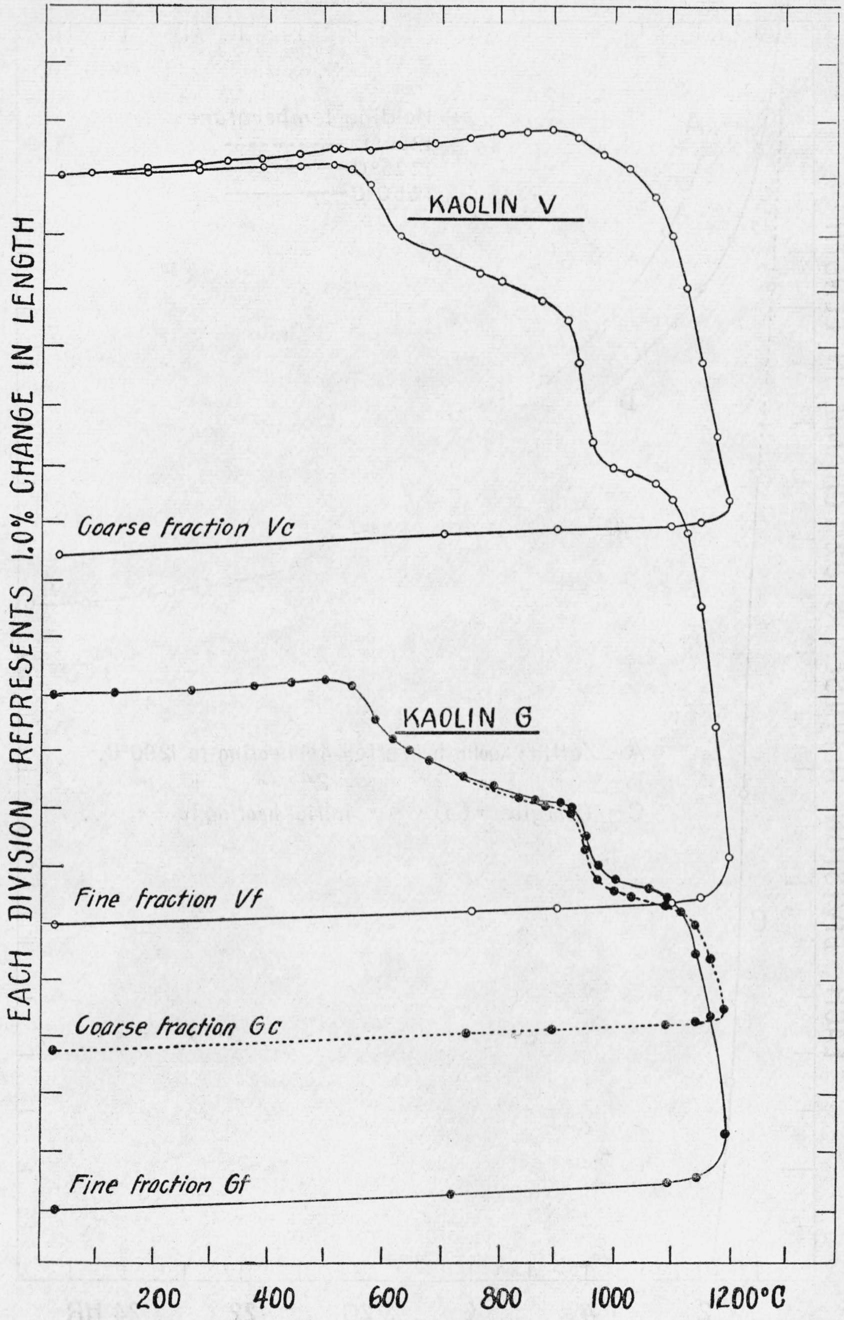

FIGURE 7.—*Length changes of coarse and fine fractions of primary Vermont kaolin V and secondary Georgia kaolin G.*

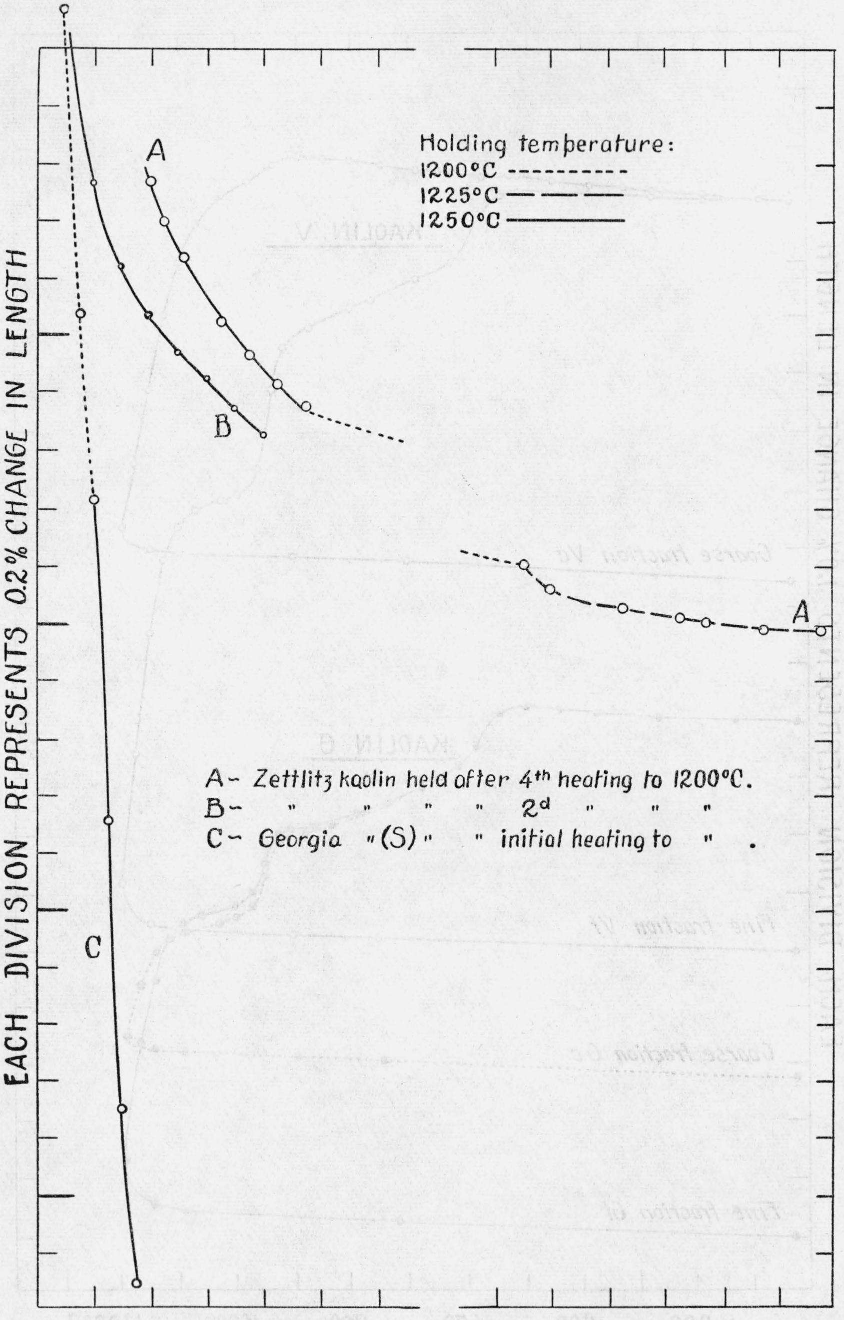

FIGURE 8.—*The continued shrinkage of two kaolins when held at 1,200°, 1,225° or 1,250° C.*

TABLE 5.—*True specific gravity of mica (25°/25°) and weight loss after various heat treatments*

Holding temperature	Specific gravity		Weight loss
° C			%
Raw	2.848	2.849	[a] 0.12
600	2.823	2.822	1.57
900	2.664	2.663	4.70
1,000	2.651	2.650	4.71
1,200	2.660	2.660	4.74

[a] Loss after holding at 110° C.

Differential heating curves, showing thermal effects, were obtained for kaolins *E* and *Z*. The endothermic effect was maximum at 578° and 574° C, respectively, and the exothermic effect was maximum at 964° and 961° C, respectively. There was no evidence of other heat effects.

2. BODIES

Results of the length-change observations on commercial body *H*, and laboratory-prepared bodies 27 and 35, are summarized in table 6 and in figures 9 and 10.

TABLE 6.—*Effects of various methods of forming, and two heating rates, on the length changes of three bodies*

[All specimens dried in air (room temperature) unless otherwise noted.]

Body	Treatment	Schedule	Length change from room temperature to—(° C)								
			200°	500°	600°	700°	800°	900°	1,000°	1,100°	1,190°
		hr	%	%	%	%	%	%	%	%	%
H	Deaired and extruded	9	+0.11	+0.57	+0.79	+0.77	+0.74	+0.71	+0.43	0.00	−2.74
H	do	24	+.09	+.56	+.77	+.76	+.72	+.70	+.42	−.38	−4.23
H	Deaired and extruded. Dried at 100° C	24	+.09	+.56	+.77	+.75	+.72	+.70	+.43	−.21	−3.81
27	Hand-wedged and dried at 110° C	9	+.12	+.42	+.48	+.51	+.54	+.49	+.07	−4.50	[a] −8.29
27	do	24	+.07	+.37	+.40	+.45	+.48	+.41	−.02	−5.27	[a] −9.02
27	do	24	+.09	+.37	+.41	+.45	+.46	+.39	−.08	−5.42	[a] −8.99
35	Hand-wedged and dried at 110° C	9	+.10	+.45	+.52	+.46	+.42	+.41	+.06	−.28	−8.80
35	Deaired and extruded	9	+.11	+.46	+.46	+.44	+.40	+.35	+.06	−2.30	−10.60
35	Hand-wedged	24	+.09	+.45	+.46	+.44	+.42	+.38	+.10	−1.85	−8.86
35	Deaired and extruded	24	+.12	+.46	+.43	+.40	+.38	+.33	.00	−3.15	−9.93
35	Pressed with 15% water. Dried 110° C	9	+.13	+.48	+.49	+.39	+.34	+.24	−.07	−2.75	−12.35
35	Pressed with 8% water. Dried 110° C	9	+.13	+.49	+.49	+.37	+.30	+.22	−.20	−2.86	−12.00
35	do	24	+.12	+.46	+.39	+.30	+.27	+.18	−.31	−5.76	−13.33
35	Pressed with 3% water	9	+.07	+.40	+.33	+.20	+.13	−.03	−.50	−3.20	−13.61
35	Pressed with 3% water. Dried 110° C	9	+.11	+.44	+.37	+.26	+.18	+.08	−.40	−3.33	−11.95

[a] Maximum temperature 1,160° C.

Body *H*, typical of the fine earthenware (or semiporcelain) type, shows the same general trend in behavior as the talcose bodies 27 and 35 which were heated to practical vitrification. There is less than 1-percent change in length until the almost abrupt shrinkage beginning above 1,100° C in body *H*, and above 1,050° C in the other two bodies.

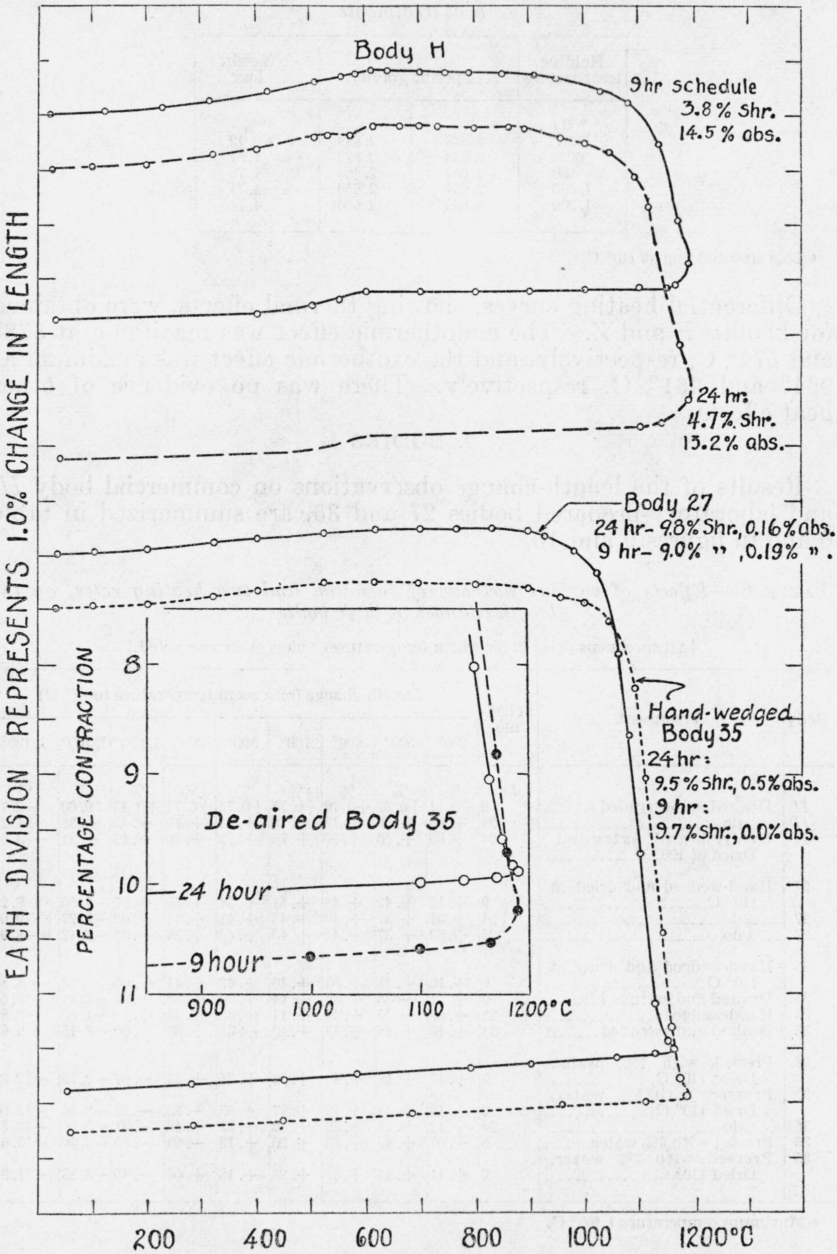

FIGURE 9.—*Length-change curves, shrinkage (shr.), and absorption (abs.) for commercial earthenware body H (deaired and extruded) and laboratory talcose bodies 27 and 35.*

Shrinkage values show total net length change during heating and cooling as indicated by the gage. The curves for hand-wedged bodies 27 and 35 were obtained on the 24-hr schedule. The curves for deaired body 35 (insert) show how the shrinkage during the 9-hr schedule exceeded that during the 24-hr schedule.

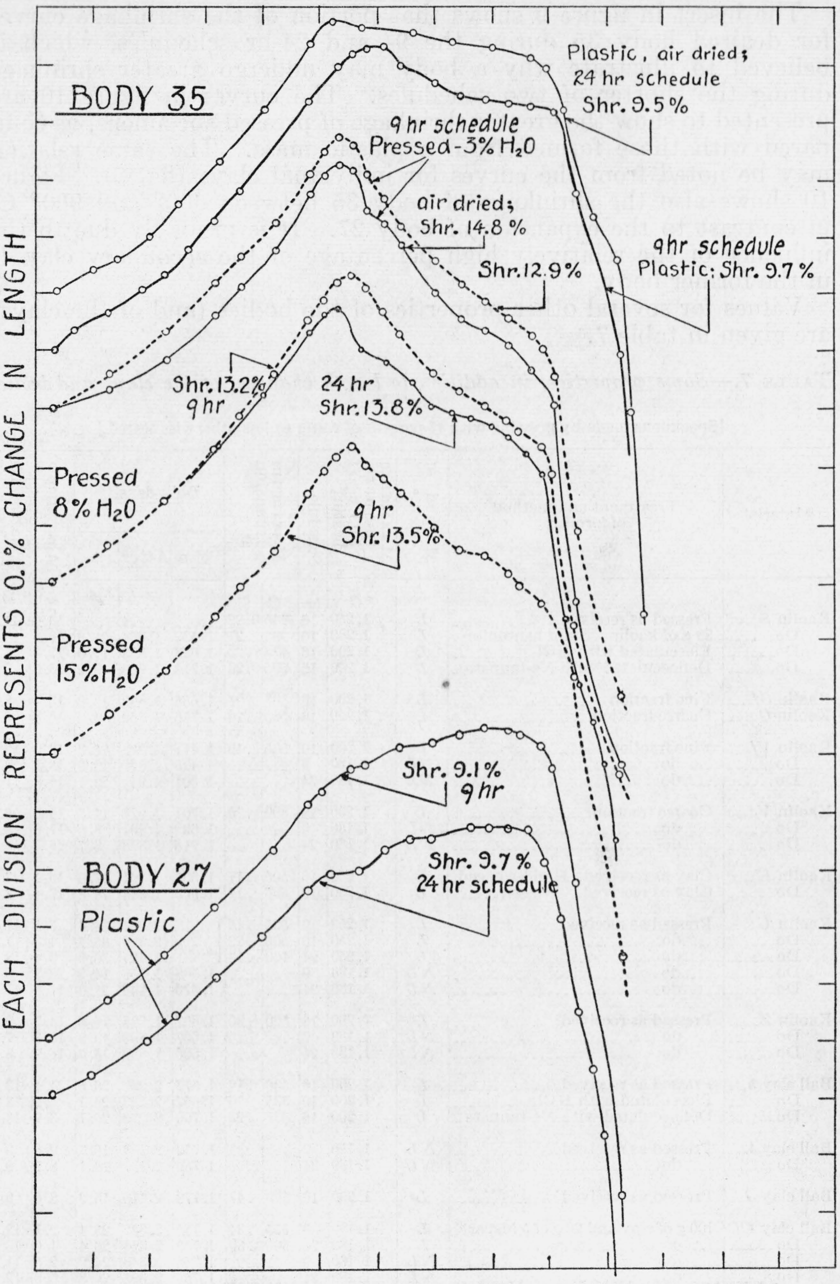

FIGURE 10.—*Length changes of bodies 27 and 35 showing effect of method of forming.*

[a] Unless otherwise noted, the specimens were dried at 110° C. Shrinkage (shr.) values are for specimens after cooling to room temperature.

The insert in figure 9 shows that portion of the shrinkage curves for deaired body 35 during the 9- and 24-hr schedules, which is believed to illustrate why a body may undergo greater shrinkage during the shorter of two schedules. The curves in figure 10 are presented to show the greater shrinkage of pressed specimens, as compared with those formed from a plastic mass. The same relation may be noted from the curves for individual clays (fig. 5). Figure 10 shows also the shrinkage of body 35 between 600° and 900° C, in contrast to the expansion of body 27. It is probably due to the influence of the relatively high percentage of the secondary clay *U* in the former body.

Values for several other properties of the bodies (and of the clays) are given in table 7.

TABLE 7.—*Some properties, in addition to length changes, of the clays and bodies tested*

[Specimens made by pressing with 15 percent of water unless otherwise stated.]

Material	Treatment and method of forming	Method of test [a]	Maximum temperature of heating	Schedule	Maximum expansion at		Bulk density			Absorption	Total shrinkage [b]
							Before	After	Δ		
			°*C*	*hr*	°*C*	%			%	%	%
Kaolin *S*	Pressed as received	*L*	1,250	16	445	0.22				13.0	11.6
Do	80 g of kaolin, 20 g of bentonite	*L*	1,200	16	505	.21	1.532	1.954	21.6	13.8	10.3
Do	Flocculated with HCl	*L*	1,200	16	475	.23	1.623	1.823	11.0	17.3	8.8
Do	Deflocculated with Na-tannate	*L*	1,200	16	505	.23	1.712	1.906	10.2	15.0	9.1
Kaolin *Gf*	Fine fraction	*L*	1,200	16	505	.27	1.635	1.899	13.9	15.7	8.9
Kaolin *Gc*	Coarse fraction	*L*	1,200	16	505	.26	1.776	1.850	4.0	16.8	5.8
Kaolin *Vf*	Fine fraction	*L*	1,200	16	505	.19	1.477	1.978	25.3	13.9	12.9
Do	do	*NL*	1,180	9			1.495	1.822	18.0	18.1	10.2
Do	do	*NL*	1,180	24			1.501	1.941	22.7	14.8	11.8
Kaolin *Vc*	Coarse fraction	*L*	1,200	16	890	.75	1.701	1.927	11.7	14.0	6.3
Do	do	*NL*	1,180	9			1.692	1.801	6.0	17.8	3.8
Do	do-	*NL*	1,180	24			1.715	1.878	8.7	15.5	4.5
Kaolin *K*	Clay as received. Hand-wedged	*L*	1,250	16	505	.17	1.505	1.761	17.0	19.1	7.9
Do	Clay as received. Pressed	*L*	1,250	16	505	.12	1.596	1.848	15.8	17.4	8.9
Kaolin *U*	Pressed as received	*L*	1,250	9	505	.15	avg. 1.530	2.334	34.4	5.5	17.4
Do	do	*L*	1,250	16	505	.22		2.379	35.7	4.3	17.6
Do	do	*L*	1,250	24	460	.13		2.424	36.9	3.4	18.1
Do	do	*NL*	1,180	9			1.508	1.758	14.2	20.2	9.5
Do	do	*NL*	1,180	24			1.526	1.933	21.0	14.8	11.8
Kaolin *Z*	Pressed as received	*L*	1,200	16	520	.30	1.607	1.928	16.6	14.7	9.5
Do	do	*NL*	1,180	9			1.607	1.803	10.9	18.3	7.3
Do	do	*NL*	1,180	24			1.606	1.890	15.0	16.2	8.3
Ball clay 5	Pressed as received	*L*	1,200	16	530	.16	1.677	2.283	26.5	4.4	13.2
Do	Flocculated with HCl	*L*	1,200	16	535	.26	1.667	2.226	25.1	5.2	13.2
Do	Deflocculated with Na-tannate	*L*	1,200	16	535	.23	1.700	2.270	25.1	3.9	13.2
Ball clay 4	Pressed as received	*NL*	1,180	9			1.752	2.103	16.7	9.6	9.3
Do	do	*NL*	1,180	24			1.750	2.137	18.1	8.7	9.9
Ball clay *J*	Pressed as received	*L*	1,200	16	550	.47	1.778	2.140	16.9	8.6	9.2
Ball clay *CC*	100 g of clay and 20 g of feldspar	*L*	1,180	9	555	.34	1.755	2.352	25.4	2.1	12.6
Do	do	*L*	1,180	24	505	.30	1.755	2.384	26.4	1.0	13.0
Do	do	*NL*	1,180	9			1.758	2.340	24.9	2.5	11.9
Do	do	*NL*	1,180	24			1.752	2.395	26.8	0.9	12.3
Do	100 g of clay, 20 g of feldspar, 20 g of flint	*NL*	1,180	9			1.798	2.244	19.9	4.2	9.8
Do	do	*NL*	1,180	24			1.796	2.270	20.8	4.1	10.2
Do	do	*L*	1,200	16	520	.38	1.798	2.285	21.3	3.5	10.6
Body *H*	Deaired and extruded	*L*	1,190	9	610	.80	avg. 1.810	1.907	5.1	14.5	3.7
Do	do	*L*	1,190	24	615	.78		1.946	7.0	13.2	4.3
Do	do	*NL*	1,190	9				1.913	5.7	14.2	3.8
Do	do	*NL*	1,190	24				1.913	5.7	14.1	3.9

See footnotes at end of table.

TABLE 7.—*Some properties, in addition to length changes, of the clays and bodies tested*—Continued

[Specimens made by pressing with 15 percent of water unless otherwise stated.]

Material	Treatment and method of forming	Method of test[a]	Maximum temperature of heating	Schedule	Maximum expansion at		Bulk density			Absorption	Total shrinkage[b]
							Before	After	Δ		
			°*C*	*hr*	°*C*	%			%	%	%
Body 27	Hand-wedged	*L*	1,160	9	790	.54	avg. 1.730	2.390	27.6	0.2	8.7
Do	do	*L*	1,160	24	765	.46		2.400	27.9	.2	9.5
Do	do	*NL*	1,190	9	----	----		2.362	26.7	.2	8.0
Do	do	*NL*	1,190	24	----	----		2.355	26.5	.3	8.5
Body 35	Pressed with 15 percent of H_2O	*L*	1,190	9	555	.53	avg. 1.846	2.447	32.5	.6	13.3
Do	do	*NL*	1,190	9	----	----		2.424	31.3	1.6	11.1
Do	do	*NL*	1,190	24	----	----		2.397	29.8	2.3	10.9
Body 35	Pressed with 8 percent of H_2O	*L*	1,190	9	555	.54	1.884	2.471	31.1	.4	12.8
Do	do	*L*	1,190	24	530	.47	1.884	2.331	23.7	.8	13.2
Do	Pressed with 3 percent of H_2O	*L*	1,190	9	530	.47	1.824	2.467	35.5	.6	12.5
Do	Deaired and extruded	*L*	1,190	9	610	.51	1.782	2.470	38.6	.4	11.3
Do	do	*L*	1,190	24	505	.47	1.783	2.448	37.3	.8	10.6
Do	Hand-wedged	*L*	1,190	9	610	.52	avg. 1.803	2.458	36.3	.0	9.4
Do	do	*L*	1.190	24	520	.47		----	----	.5	9.2
Do	do	*NL*	1,190	9	----	----		2.451	35.9	1.1	8.5
Do	do	*NL*	1,190	24	----	----		2.436	35.1	1.0	8.3

[a] *L* signifies that the specimen was tested under load in the porcelain dilatometer.
NL signifies that the specimen was under no superimposed load.
[b] Values are based on direct measurements before and after the test.

V. DISCUSSION

1. CLAYS

A reversal from expansion to contraction between 500° and 600° C, an appreciable increase in the contraction per unit temperature rise between 900° and 1,000° C and a comparatively abrupt contraction beginning at 1,050° to 1,100° C characterize the length changes for all the clays tested.[14] The reversal between 500° and 600° C, and the comparatively rapid contraction between 900° and 1,000° C, are in all probability associated with the loss of chemically combined water and with the formation of gamma-Al_2O_3. The final contraction accompanies a process known among ceramists as "vitrification," the exact physical chemistry of which is still obscure but involves increase in true density[15] and bulk density, and the formation of glass and of new or altered crystalline phases which may be submicroscopic in particle size.

a. BEGINNING OF CONTRACTION

The temperature at which a reversal from expansion to contraction takes place is affected by the rate of heating and by the nature of the clay, as the data in table 7 show, but not by the method of forming the specimens (fig. 5). It is, in general, higher for ball clays than for kaolins and higher for the more rapid heating rates.

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[14] This generalization does not include the coarse fraction of Vermont kaolin, which fraction contains, by calculation, less than 30 percent of kaolinite.

[15] T. Nakai and Y. Fukami, J. Soc. Chem. Ind. (Japan), **37,** 430 (1934). Abs. in J. Amer. Ceram. Soc. **18,** 22 (1935).

b. LENGTH CHANGES BETWEEN 500° AND 900° C

If one assumes that the length changes of the secondary kaolins approximate those of pure kaolinite, then the normal behavior of kaolinite in the range 500° to 900° C is to contract about 3 percent. This shrinkage is slightly greater for dry-pressed specimens than for those formed from plastic masses, as shown in figure 5.

Feldspar and quartz counteract the contraction, but to a much smaller degree than does mica in equivalent amounts. That mica may alter the length-change behavior considerably has been shown by Steger and by Rieke and Wen (fig. 6). For clay *CC*, an addition of 20 g of mica to 100 g of the clay completely nullified the contraction, and the net change was an expansion of 0.26 percent (table 4). This effect of mica probably involves a reaction between it and the clay, because the changes caused by it cannot be wholly accounted for by calculations based on its expansion or altered specific gravity.

The data as arranged in table 8 indicate that the characteristic group differences in length change between 500° and 900° C for the secondary kaolins, as compared with ball clay and primary kaolins, are due to the mica content rather than to their geologic history. It is suggested that the lack of a close relation between mica content and contraction, for individual clays, may be caused by the state of subdivision of the mica. For example, the length changes for ball clays *J* and 5, and the fine fraction of clay *V*, are 0.2, 0.8, and 2.6 percent, respectively. The calculated mica contents are 16, 17, and 14 percent, respectively. Petrographic examinations (by H. Insley) showed the mica particles to be coarsest in clay *J* and finest in the clay *V* fraction. The particle size of the mica may affect not only the rate and temperature range at which it decomposes, but also the extent to which the decomposition products affect other constituents in a clay-mica mixture. Zwetsch[16] found, by X-ray examinations, that mica remains unaltered below 900° C,[17] and that a break-down to alpha-alumina, gamma-alumina, and leucite begins at about 950° C.

Several investigators[18] have proposed the formation of an anhydrous alumino-silicate, or "meta-kaolin", following the expulsion of combined water from the clay molecule (about 450° to 550° C). The results of de Keyser's[19] dielectric constant determinations are interpreted by him as supporting the meta-kaolin theory. This meta-kaolin presumably breaks down between 700° and 900° C. The curves obtained in this study, for both heat effects and length changes, do not indicate a phase change in this temperature range and, to that extent, support the claim (footnotes 8 and 15) that an anhydrous alumino-silicate is not formed.

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[16] A. Zwetsch, Ber. deut. keram. Ges. **15**, 2 (1934).

[17] That diffraction patterns do not indicate an alteration in mica below 900° C, even though water of crystallization is given off at lower temperatures, may be explained by the finding of F. Rinne (Ber. Verhandl. sachs. Akad. Wiss. Leipzig, **76**, 261, 1924) that the crystal structure of muscovite was not destroyed by dehydration.

[18] R. Schwarz and G. Trageser, Chem. Erde **7**, 566 (1932). R. Schwarz and K. Nüchter, Ber. deut. keram. Ges. **15**, 514 (1934).
See also footnote 2.

[19] W. de Keyser, Ber. d. deut. keram. Ges. **21**, 29 (1940).

TABLE 8.—*Relation of mineral composition and length changes (average values)*

Material	Length change in the interval—(° C)				Mineral composition		
	500° to 900°	900° to 1,000°	1,000° to 1,100°	1,100° to 1,200°	Mica	Quartz	Kaolinite
	%	%	%	%	%	%	%
Ball clay *J*	0.2	0.6	2.1	5.9	16	18	58
Ball clay 5	.8	1.2	2.7	7.8	17	12	61
Ball clay 4	.9	1.5	1.9	6.0	10	12	68
Ball clay *CC*	1.3	1.3	.9	6.6	2	24	68
Average	0.8	1.1	1.9	6.6	11	16	64
Primary kaolin *K*	0.8	1.2	0.7	4.5	5	<1	90
Primary kaolin *P*	1.0	1.2	.3	3.4	13	2	80
Primary kaolin *Z*	1.4	1.4	1.0	5.1	7	1	85
Average	1.0	1.3	0.7	4.4	8	1	85
Secondary kaolin *E*	2.5	2.4	0.7	6.1	4	2	91
Secondary kaolin *U*	2.8	2.6	.4	6.9	1	2	96
Secondary kaolin *S*	2.8	2.1	.3	2.6	1	<1	97
Secondary kaolin *T*	3.6	2.3	.2	2.8	<1	<1	93
Average	2.7	2.3	0.4	4.6	2	1	94
Fine fraction, clay *V*	2.6	2.5	0.6	6.2	14	4	79

c. LENGTH CHANGES BETWEEN 900° AND 1,000° C

There seems to be complete unanimity of opinion in the literature that the accelerated shrinkage between 900° and 1,000° C accompanies the crystallization of amorphous alumina, derived from the kaolinite molecule, to form gamma-alumina. This has been discussed at length by Insley and Ewell (footnote 8), Heindl and Mong (footnote 12), and others (footnotes 1 and 2).

TABLE 9.—*Relation of alumina content of clays and the length change between 900° and 1,000° C*

Clay	Type	Length change	Al_2O_3 from kaolinite	Al_2O_3 from mica
		%	%	%
Vf	Primary	2.5	31	3.5
S	Secondary	2.1	38	0.3
E	do	2.4	36	1.2
U	do	2.6	38	0.4
T	do	2.3	37	.0
Average		2.4	36	1.1
Z	Primary	1.4	33	1.7
P	do	1.2	32	3.5
K	do	1.2	36	1.3
4	Ball clay	1.5	27	2.6
5	do	1.2	24	4.4
CC	do	1.3	27	0.4
Average		1.3	30	2.3
Vc	[a]	0.6	11	7.8
J	Ball clay	.6	23	4.0
Average		0.6	17	5.9

[a] Coarse fraction of primary kaolin *V*.

The data as arranged in table 9 show a fairly good direct relation for the three groups between the alumina derived from kaolinite and the contraction, if one excepts the fine fraction of Vermont kaolin (*Vf*). In this case the alumina from mica may be a contributing factor, since this alumina also crystallizes in the 900° to 1,000° C temperature range according to Zwetsch (footnote 16). In fact, to avoid placing too much emphasis on the direct relation of length change and alumina from the kaolinite, it should be noted that an equally good indirect relation is shown between the length changes and alumina from the mica.

It is interesting that a primary kaolin and four secondary kaolins constitute the group of greatest contraction, and three primary kaolins and three ball clays the intermediate group. The indication is that the particle-size distribution, which is well known[20] to be materially different in these three types of clay, does not significantly affect the length changes in this temperature range.

d. FINAL CONTRACTION

The ball clays begin their final contraction in a comparatively gradual manner at about 1,000° C, whereas the kaolins begin sharply at about 1,100° C (figs. 3 and 4). There is, however, no clearly defined difference in behavior between the ball clays and the kaolins, as shown for example by ball clay *CC* and kaolin *Z* (table 8), but the group averages show a definite trend. They indicate that chemical composition (within the limits of composition studied) is a factor, but that particle size exerts the greater influence on the final contraction. The fineness of Florida clays (*E* and *U*) was commented on by Klinefelter and Meyer (footnote 7), who refer to them as "ball kaolins." Another evidence of the influence of grain size is the greater contraction of clay *Gf* (fine fraction) compared to the coarse fraction *Gc* (fig. 7), although the chemical compositions are nearly identical.

Once under way, the final shrinkage proceeds almost abruptly but reaches a maximum in the temperature range 1,100° to 1,150° C for the ball clays (excepting clay *CC*) and in the range 1,150° to 1,200° C for the kaolins (table 10). Results are presented in this table as "contraction per 1° C temperature rise" in order to emphasize the differences in shrinkage which may exist in industrial practice within a piece of ware, or from piece to piece, because of temperature differentials. Tables 10 and 4 show also that addition of feldspar accelerated the final contraction because, at these temperatures, it acts as a flux. Below 1,000° C it had retarded contraction, probably because it acts as an inert aggregate.

It should be observed that in no case (table 7) did an individual clay, or a clay-flint-feldspar mixture, show greater shrinkage or lower absorption after heating to the same temperature on the 9-hr schedule than after the 24-hr schedule. This was noted also by Shelton and Meyer.[21]

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[20] R. H. Bray, J. Am. Ceram. Soc. **20**, 257 (1937).

[21] Geo. R. Shelton and Wm. W. Meyer, J. Am. Ceram. Soc., **21**, 371 (1938).

TABLE 10.—*Contraction of clays and bodies above 1,000° C (average values)*

Material	Schedule	Contraction per 1 degree C. rise in temperature in the range—				
		1,000° to 1,050°	1,050° to 1,100°	1,100° to 1,150°	1,150° to 1,200°	1,200° to 1,250°
	hr	%	%	%	%	%
Ball clay *J*	16	0.009	0.03	0.07	0.05	--------
Ball clay 4	16	.009	.03	.07	.04	--------
Ball clay 5	16	.015	.04	.10	.05	--------
Ball clay *CC*	9	.006	.01	.05	.08	--------
Do	24	.006	.01	.06	.07	--------
Ball clay *CC* plus feldspar [a]	9	.010	.05	.11	[b].03	--------
Do.[a]	24	.024	.07	.10	[b].03	--------
Kaolin *S*	16	.001	.003	.01	.05	0.06
Do. plus feldspar [a]	9	.002	.004	.02	.09	--------
Do.[a]	24	.002	.009	.03	.10	--------
Kaolin *E*	16	.004	.010	.05	.07	.03
Kaolin *U*	16	.003	.005	.01	.13	.08
Kaolin *T*	16	.002	.002	.01	.05	--------
Kaolin *K*	16	.004	.006	.03	.06	.03
Kaolin *P*	16	.003	.005	.02	.05	.06
Kaolin *Z*	16	.004	.013	.05	.05	--------
Body *H*, deaired, extruded	9	.002	.01	.01	[c].05	--------
Do	24	.002	.01	.03	[c].05	--------
Body 35, deaired, extruded	9	.005	.04	.08	[c].10	--------
Do	24	.008	.06	.10	[c].05	--------
Body 35, pressed	9	.006	.05	.09	[c].12	--------
Do	24	.014	.09	.13	[c].03	--------
Body 27, hand-wedged	9	.015	.07	.06	[d].01	--------
Do	24	.023	.08	.06	[d].01	--------

[a] These specimens were prepared as described in table 4.
[b] Maximum temperature 1,180° C.
[c] Maximum temperature 1,190° C.
[d] Maximum temperature 1,160° C.

2. BODIES

The effect of varying the amount of free, or mechanical, water in pressed specimens was studied briefly in connection with body 35 (tables 6 and 7). The highest density in the dry state (table 7) was obtained with 8 percent of water, which agrees with the findings of Dodd, whose work was done on much coarser-grained mixtures.[22] He obtained maximum density when using 8 to 10 percent of water and the range covered was 5 to 12 percent.

a. LENGTH CHANGES BELOW 1,000° C.

The length changes of the three bodies during the heating to 1,000° C are comparatively small (fig. 9). The presence of free silica in body *H* was shown by a small but clearly defined acceleration in the length change between 550° and 600° C during both heating and cooling. In bodies 27 and 35, the effect of the quartz inversion was overshadowed by the other body constituents (fig. 10).

It might appear that bodies such as these could be heated rapidly to 1,000° C with little danger of rupture from shrinkage differentials alone, and that the limiting factors are the internal pressures built up by volatilization of water and the combustion of organic matter. At this stage, however, bodies are very weak mechanically, and the possibility remains that microscopic lines of structural weakness could be caused by the widely divergent length changes of (for example)

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[22] E. M. Dodd, J. Am. Ceram. Soc. **17**, 465 (1938).

mica, quartz, and the secondary clays. That such internal weaknesses can develop in coarser structures, such as clay refractories, has been considered probable.[23] It seems hardly likely, however, that cracks or lines of weakness too large to be "healed" by the glass formed above 1,000° C could develop in an intimately blended mixture of particles of which practically none exceed 80 microns in diameter and of which over 90 percent (by count) are under 4 microns in diameter.

b. LENGTH CHANGES ABOVE 1,000° C.

The importance of a carefully controlled heating schedule, and uniformity in temperature distribution, during the very rapid shrinkage accompanying vitrification (fig. 9) is evident. The values in table 10 show that shrinkages of as much as 0.13 percent per 1° C rise in temperature may be expected. This rate is equivalent to a difference in shrinkage of about ⅛ in. across a 10-in. specimen for a temperature differential of only 10° C.

Figure 9 and table 10 show how the shrinkage of body 27 is retarded as it approaches vitrification during the last 60 degrees of temperature rise, and how the contraction becomes almost purely thermal as soon as the maximum temperature is passed and cooling begins. In contrast, body *H*, which is still highly absorbent after the test, shows little retardation and also continues to undergo permanent contraction as the temperature drops to 1,100° C. A similar difference may mark the shrinkage of a body heated on a long schedule as compared with the shrinkage of the same body heated on a short schedule (deaired body 35, see insert, fig. 9).

On the faster (9-hr) schedule, the deaired and extruded body 35 shows a greater contraction per degree centigrade temperature rise between 1,150° and 1,190° C (table 10) than it does between 1,100° and 1,150° C, whereas on the slower (24-hr) schedule the contraction in the higher temperature range is only half that in the lower range. Similar, but more pronounced, differences are shown by the dry-pressed specimens tested on the 9-hr and 24-hr schedules. As a result, the shrinkage on the faster schedule (which had been less than that on the slower schedule until about 1,150° C was reached) now becomes greater than that on the slower schedule. In contrast, body 27 (table 10) shows the same decrease in shrinkage as the maximum temperature is approached, regardless of heating rate, and consequently the greater shrinkage attained during the earlier stages of heating on the slower schedule is maintained to the end of the test.

The results for body 35 support Watts' findings (footnote 3). Watts suggests that bodies heated on long schedules show the lesser shrinkage because the feldspar glass has had more time to dissolve clay and silica, thereby increasing its viscosity.

According to the data obtained in this study, the greater shrinkage and lower absorption after the shorter of two heating schedules is not a fundamental and common characteristic of all ceramic bodies. It is rather the result of certain combinations of body ingredients and heating schedules favoring such a result.

c. TOTAL SHRINKAGE

Table 7 shows the effect, on total shrinkage, of the load to which specimens were subjected during test in the porcelain dilatometer.

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[23] R. F. Geller, Am. Refractories Inst., Tech. Paper No. 4 (1927).

Comparable values for specimens heated under no superimposed load were obtained for the mixture of clay *CC* and feldspar, and for bodies *H* and 35. The load apparently had no effect on body *H* during the short schedule. It increased the shrinkage 0.4 percent for body *H* during the 24-hr schedule, and the maximum increase was 2.2 percent for body 35 pressed with 15 percent of water.

Another point of interest is the effect of deairing (body 35, table 7). If the limited data are significant, they mean that deaired plastic bodies will shrink more than nondeaired ones. In any case, there is sufficient evidence to indicate that both the deaired and nondeaired plastic-formed bodies shrink appreciably less during heating than the same bodies when pressed. As pointed out previously in this paper, the greater shrinkage of pressed specimens applies also to individual clays (fig. 5).

VI. SUMMARY AND CONCLUSIONS

Length changes of 9 kaolins, 4 ball clays, and 3 whiteware bodies were observed during their initial heating to a maximum temperature of 1,250° C. The apparatus used was an especially designed porcelain dilatometer. Differential heating curves for two clays and the specific gravity and weight losses of mica after holding at several temperatures were obtained as supplementary data.

Primary kaolins and ball clays show less contraction between 500° and 900° C than do the secondary kaolins. The contraction (about 3 percent in a relatively pure clay) and the mica content show a sufficiently close relation to indicate strongly that the mica content, rather than geological history or particle-size distribution, is the predominating factor determining length change in this temperature range.

The contraction between 900° and 1,000° C is believed to be caused by the crystallization of amorphous alumina, resulting from the breakdown of the kaolinite molecule, to gamma-alumina. The data show a direct relation between the amount of this contraction and the calculated amount of alumina contributed by kaolinite.

The very rapid contraction accompanying vitrification, beginning at about 1,050° C in ball clays and 1,100° C in kaolins, varied from about 6 percent of the original length for primary kaolins to 12 percent for a secondary "ball kaolin." Particle size appears to be the principal factor determining this contraction.

Changes in length of the bodies, in the range from room temperature to 1,000° C, were gradual, and the maximum change (expansion) was 0.8 percent of the original length. Above 1,000° C the rate of change (contraction) increased rapidly. The maximum rate noted was 0.3 percent per minute for a talcose body on a "9-hr schedule" in the temperature range 1,135° to 1,170° C. Based on unit temperature rise, the maximum length change noted was 0.13 percent per 1° C for the same body during a "24-hr schedule" in the temperature range 1,100° to 1,150° C.

In every test of a body (or of an individual clay) the shrinkage per unit temperature rise during the first stages of the final rapid contraction was greater on the 24-hr schedule than on the 9-hr schedule.

In some cases, the shrinkage rate decreased more on the long schedule, during the last 25 to 50 degrees of temperature rise and the first 50 to 100 degrees of cooling, than on the shorter schedule. As a result, the shrinkage during the shorter schedule forged ahead with the effect that the total shrinkage during the shorter schedule was greater than during the longer schedule.

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