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Properties of Barium-Strontium Titanate Dielectrics

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The results on barium-strontium titanates are given for heat treatments, absorption, shrinkage, thermal expansion, dielectric constant (*K*) at −60° to 85° C, and also for *Q*, the reciprocal of the power factor, at 25° C for frequencies of 50 to 20,000 *kc/s*. A few measurements of *K* and *Q* were made at 3,000 *mc/s*. Specimens matured (absorption less than 0.1 percent) at 1,250° to 1,430° C. *K* values ranged from 34, for $BaO{:}4TiO_2$, to several thousand for specimens with compositions on the $BaTiO_3$-$SrTiO_3$ join. Specimens having the composition $BaO{:}5TiO_2$ were unique in that their temperature coefficient of *K* was practically zero. *Q* values ranged from 50 to 100 for test pieces containing over 40 percent of BaO, and from 400 to 10,000 for those with less than 40 percent of BaO. *K* values did not change appreciably with frequency. Linear thermal expansions between 25° and 700° C ranged from 0.6 to 0.84 percent. Many of these specimens could be used in radio equipment.

I. Introduction

The properties of titanates containing barium and strontium have been investigated only recently [1, 2, 3],[1] although dielectrics containing titanium have been used in radio equipment since 1925 [4], and the high values of the dielectric constant of some of the oxides of titanium have been known since 1903 [5]. Many patents have been issued covering the preparation of dielectrics from titania in combination with such oxides as MgO, ThO_2, ZrO_2, SiO_2 and as titania-filled organic plastics. Some of the titanates were conspicuous because they exhibited outstandingly high values of the dielectric constant [1]. Although many data have been published [1, 2, 3] on specific titanates, a knowledge is desirable of the properties of such materials over a wide range of compositions. In 1944 a systematic study of ceramic dielectrics, of which this paper constitutes the first report, was undertaken at the request of the Army Signal Corps.

II. Preparation of Specimens

A stock of titania, designated TMO, together with chemically pure quality barium and strontium carbonates, were used in the preparation of all specimens with compositions shown in figure 1. Three other grades of titania were tested also.

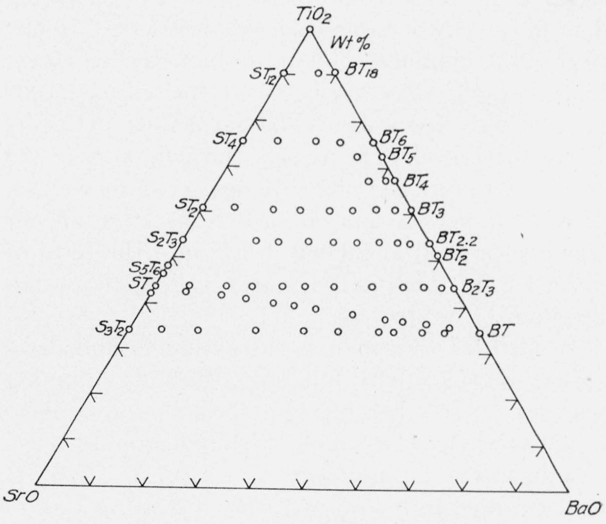

FIGURE 1.—*Ternary diagram for the system* BaO-SrO-TiO_2, *showing compositions studied.*

B×BaO; S=SrO; T=TiO_2.

The chemical analysis, furnished by the maker, and some properties of each of the four grades are given in table 1.

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[1] Figures in brackets indicate the literature references at the end of this paper.

TABLE 1.—*Composition, specific surface, and crystal form of four brands of commercial titanium dioxide, and also the heat-treatments and properies of specimens prepared from these materials*

Grade designation	Composition [a]			Specific surface [b]	Crystal form [c]	Heat treatments for 1 hr at—		Shrinkage [d]	Absorption [d]	Dielectric constant (K) at 25°C and 1,000 kc/s	Reciprocal (Q) of power factor at 25°C and 1,000 kc/s
	TiO_2	SiO_2	Al_2O_3								
	Wt %	*Wt %*	*Wt %*	*cm²/g*		°*C*	°*C*	*Percent*	*Percent*		
TMO	98.7	0.25	0.2	39,000	Rutile	1,100	1,305	14.1	3.33	76	182
						1,100	1,350	16.6	0.72	87	1,000
						1,100	1,400	17.6	.02	91	5,200
WD	98.7	.4	.03	36,000	do	1,100	1,305	7.5	10.50	42	7
						1,100	1,350	9.5	9.62	49	6
						1,100	1,425		7.02	([e])	([e])
R	98.2	.5	.1	27,000	do	1,100	1,305	18.3	0.00	97	7,000
						1,100	1,350	19.6	.01	98	7,700
						1,100	1,400	18.9	.01	98	8,800
A	98.8	.2	[f] .2	8,000	Anatase	1,400	None	15.8	.00	97	4,700

[a] Compositions furnished by maker.

[b] Determined by R. L. Blaine with the Lea-Nurse apparatus, air-permeability method.

[c] Determined by X-ray diffraction by H. F. McMurdie.

[d] Values obtained after final heat treatment.

[e] Conductance too high for measurement.

[f] Also contains 0.45% alkali.

Weighed batch constituents were added to distilled water containing a wetting agent (2 drops of aerosol per 250 ml of water), and the suspension stirred for 1 hr with a high-speed stirrer. After drying the caked mixture it was passed through a No. 50 sieve and moistened with 10 percent, by weight, of a 2 1/2-percent starch suspension. The moist powder was then dry-pressed at 10,000 lb/in.² into cylinders (5/8 in. in diameter by 3/4 in. long), which, after drying, were heated at 1,100° to 1,245° C for 1 hr. These calcined cylinders were pulverized and passed through a No. 325 sieve. After moistening with 5 percent, by weight, of a 2 1/2-percent starch suspension, the powder was dry-pressed at 20,000 lb/in.² into the form of disks 1/8 in. thick and of various diameters ranging from 1/4 in. to 1 in.

In all heat treatments, the cylinders and disks rested on platinum foil supported by alumina plaques. After heating the specimens overnight to about 1,000° C in an electrically heated furnace, the maturing temperature of 1,250° to 1,430° C was attained in 3 to 5 hr, using a heating rate of about 3 deg C a min, and was held for either 1 or 2 hr within ±5 deg C of the desired temperature. The furnace heating chamber was heated on all sides and was uniform in temperature within ±5 deg C. All temperatures were measured with calibrated Pt to Pt-Rh thermocouples.

III. Test Methods

Absorption was determined by immersing the weighed specimen in chemically pure carbon tetrachloride, where it remained while the liquid was boiled for 5 hr and allowed to cool. After quickly drying the surface of the specimen, it was weighed in a stoppered bottle. Carbon tetrachloride was used instead of water to avoid any hydration of the specimens. Any gain in weight was divided by the density of the CCl_4 in order to find the equivalent gain, had water been used. Percentage absorption equals equivalent gain in weight, divided by dry weight, multiplied by 100. The percentage of shrinkage was obtained from the diameter of the mold used, minus the average diameter of the matured specimen, divided by the diameter of the mold, multiplied by 100.

The dielectric constant (K), and the Q-value,[2] were obtained from measurements on a Boonton "Q" meter, type 160–A, whose condenser had been calibrated against a calibrated precision condenser. This meter was operated in a cabinet controlled at 25° ± 0.5° C and a relative humidity of 40 percent or less. The titanate specimens were heavily coated on both sides with a silver

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[2] This value constitutes a "figure of merit" in radio-circuit design, and when greater than 10, is nearly equal to the reciprocal of the cosine of the phase angle (θ). High Q-values for inductors and condensers are necessary for good selectivity in tuned circuits.

paste and heated to 750° C in an electric muffle furnace to give silver electrodes about 0.01 in. thick. Any extraneous silver on the side of a disk was removed with fine sandpaper. Specimens were kept at a relative humidity of 40 percent or less and at room temperature for several hours and then at 25° ± 0.5° C for ½ hr before the measurements were made. Disks with a capacity greater than the capacity used in the variable condenser in the instrument were measured in series with a suitable condenser.

The capacitance from which the dielectric constant was computed was determined at 1,000 kc/s, and temperatures ranging from −60° to +85° C, by the resonant circuit[3] capacity substitution method. The specimens were held between brass electrodes in an insulated chamber 1 ft^3 in volume. This chamber could be cooled with dry ice or heated by a heating coil, and was controlled to ±0.25° C. Measurements were made at +40°, +60°, +80°, +85°, +70°, +50°, +30°, +20°, 0°, −20°, −40°, −60°, −50°, −30°, −10°, and +10° C. Prior to each measurement, the temperature of the specimen was maintained constant for 15 or more minutes.

Measurements at a frequency of 3,000 mc/s were made on specially shaped specimens in the Massachusetts Institute of Technology coaxial wave guide instrument [6].

Thermal-expansion determinations were made by the interferometer method at a heating rate of 2½ to 3 deg C a minute.

IV. Results and Discussion

One of the difficulties encountered in the preparation of titanate dielectrics is the lack of uniformity in commercial grades of titanium dioxide. The four grades investigated varied not only in composition but also in grain size and crystal structure. Consequently, matured specimens of impure TiO_2 did not exhibit identical properties, as illustrated by the data in table 1. Such results, showing grade *WD* to be the least satisfactory for preparing dielectrics of this type, may not warrant the rejection of a given grade for the preparation of dielectrics that contain other materials, such as BaO or SrO. In the experiments on three grades (table 2), the choice of grade was immaterial so far as the properties of matured specimens of $SrTiO_3$ was concerned. The maturing temperature and other properties of specimens may vary considerably for some specimens when different grades of TiO_2 are used.

In table 3 data are given on the composition, heat treatment; absorption; shrinkage; dielectric constant (*K*); and *Q*, the reciprocal of the power factor, for matured specimens of the titanates studied.

TABLE 2.—*Properties of barium and strontium titanate specimens prepared from three grades of commercial titanium dioxide*

Grade of TiO_2	Heat treatments [a]		Shrinkage	Absorption	Dielectric constant (*K*) at 25° C and 1,000 kc/s	Reciprocal (*Q*) of power factor at 25° C and 1,000 kc/s
	Temperature	Time				
Specimens of $BaO{:}TiO_2$						
	° C	*hr*	*Percent*	*Percent*		
TMO	1, 385	2	10. 6	0. 05	1, 500	100
WD	1, 400	1	10. 9	. 03	1, 650	74
R	1, 400	1	6. 9	1. 07	1, 300	144
Specimens of $SrO{:}TiO_2$						
TMO	1, 375	2	15. 2	0. 01	264	8, 000
WD	1, 400	1	14. 8	. 01	270	7, 600
R	1, 400	1	16. 3	. 01	270	8, 000
Specimens of 57.4% $SrO{:}TiO_2$+42.6% of $BaO{:}TiO_2$						
TMO	1, 420	2	13. 7	0. 00	680	10, 000
WD	1, 430	1	10. 7	2. 44	655	31
R	1, 430	1	9. 7	2. 22	605	4, 800

[a] All preparations were given a preliminary calcination at 1,245° C for 1 hr.

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[3] A Hartley oscillator containing a No. 71A tube was used. This was powered on the filament and plate by storage batteries. Resonance was determined on a loosely coupled pick-up circuit, tuned by a precision condenser, and current was indicated by a vacuum thermocouple, using a galvanometer, lamp, and scale.

TABLE 3.—*Composition, heat treatment, absorption, shrinkage, dielectric constant, K, and Q, of bodies in the system* $BaO-SrO-TiO_2$

Specimen designation	Proportion of end members of join		Composition weight			Heat treatment			Absorption	Shrinkage	Dielectric constant, *K*, at 25° C and—			Reciprocal, *Q*, of power-factor at 25° C and—		
						No. 1	No. 2									
			BaO	SrO	TiO_2	1 hr at—	Temperature	Time			50 kc/s	1,000 kc/s	20,000 kc/s	50 kc/s	1,000 kc/s	20,000 kc/s
$SrO:12TiO_2$ $BaO:18TiO_2$																
	Weight percent	*Weight percent*	*Percent*	*Percent*	*Percent*	*° C*	*° C*	*hr*	*Percent*	*Percent*						
ST12	100	0	--------	9.8	90.2	1,100	1,310	1	0.00	16.2	113	113	114	2,500	3,500	2,000
18BS6	36	64	6.2	3.5	90.3	1,100	1,275	1	.00	16.6	83	82	82	1,200	5,400	6,700
BT18	0	100	9.6	--------	90.4	1,100	1,275	1	.00	15.0	75	74	74	260	1,000	7,000
$SrO:4TiO_2$ $BaO:6TiO_2$																
ST4	100	0	--------	24.5	75.5	1,100	1,350	1	0.00	18.0	148	147	152	800	4,000	1,300
6BS2	75	25	6.2	18.3	75.5	1,100	1,310	1	.01	17.9	120	119	121	5,400	4,000	1,400
6BS5	45	55	13.2	11.3	75.5	1,100	1,275	1	.00	17.9	78	77	76	3,400	3,400	3,900
6BS7	25	75	18.1	6.3	75.6	1,100	1,275	1	.06	16.3	53	53	51	>500	1,550	3,700
BT6	0	100	24.3	--------	75.7	1,100	1,275	1	.01	14.0	46	45	44	85	400	2,200
$SrO:3.4TiO_2$ $BaO:5TiO_2$																
5BS8	18	82	22.7	5.0	72.3	1,100	1,285	1	0.06	16.2	44	43	42	265	1,600	4,000
BT5	0	100	27.7	--------	72.3	1,100	1,325	1	.04	12.0	39	37	37	1,500	2,300	1,300
$SrO:2.7TiO_2$ $BaO:4TiO_2$																
4BS8	15	85	27.4	5.0	67.6	1,100	1,300	1	0.01	16.4	43	43	42	>2,000	10,000	5,000
4BS9	8	92	30.4	2.0	67.6	1,100	1,300	1	.01	15.4	37	37	36	>1,600	2,400	4,000
BT4	0	100	32.4	--------	67.6	1,100	1,330	1	.03	16.9	34	34	35	>1,600	2,000	3,700
$SrO:2TiO_2$ $BaO:3TiO_2$																
ST2	100.0	0.0	--------	39.3	60.7	1,100	1,330	1	0.09	16.3	186	185	185	1,600	4,500	2,600
BS1	84.0	16.0	6.2	33.1	60.7	1,100	1,300	1	.00	19.1	163	162	161	1,700	2,200	1,000
BS3	66.0	34.0	13.2	26.0	60.8	1,100	1,315	1	.00	19.9	121	120	119	5,100	2,100	1,200
BS4	51.5	48.5	18.1	21.1	60.8	1,100	1,315	1	.00	19.2	90	90	90	3,900	2,500	1,900
BS6	39.2	60.8	23.7	15.4	60.9	1,100	1,290	1	.00	19.0	75	75	74	3,300	1,600	1,600
BS7	28.2	71.8	28.0	11.1	60.9	1,100	1,260	1	.01	18.1	64	64	63	2,900	2,100	2,600
BS8	15.9	84.1	32.8	6.3	60.9	1,100	1,260	1	.00	17.4	51	50	49	2,300	1,200	2,000
BS9	8.0	92.0	36.2	2.8	61.0	1,100	1,260	1	.00	17.5	49	49	48	>2,000	1,200	2,600
BT3	0.0	100.0	39.0	--------	61.0	1,100	1,260	1	.03	17.4	44	44	43	720	650	800
$2SrO:3TiO_2$ $BaO:2.2TiO_2$																
S2T3	100.0	0.0	--------	46.3	53.7	1,100	1,310	1	0.02	16.2	200	200	196	450	2,600	3,500
BS22	71.5	28.5	13.2	33.1	53.7	1,100	1,350	1	.02	17.9	153	151	149	270	650	1,500
BS23	61.0	39.0	18.1	28.2	53.7	1,100	1,360	1	.09	17.1	153	150	149	560	680	1,600
BS25	49.0	51.0	23.7	22.6	53.7	1,100	1,310	1	.02	17.3	148	147	145	1,700	1,500	1,600
BS26	39.5	60.5	28.0	18.3	53.7	1,100	1,290	1	.06	17.6	134	133	138	600	550	700
BS271	29.0	71.0	32.8	13.5	53.7	1,150	1,295	1	.00	17.6	154	153	153	1,200	850	500
BS279	20.7	79.3	36.7	9.6	53.7	1,150	1,295	1	.00	17.5	210	205	200	500	480	250
BS28	12.5	87.5	40.5	5.8	53.7	1,150	1,295	1	.00	16.9	255	255	255	385	170	90
BS29	6.0	94.0	43.5	2.8	53.7	1,150	1,295	1	.00	15.4	215	215	210	130	100	50
BT2.2	0.0	100.0	46.3	--------	53.7	1,150	1,250	1	.00	12.9	97	95	92	140	75	40

TABLE 3.—*Composition, heat treatment, absorption, shrinkage, dielectric constant, K, and Q, of bodies in the system* BaO-SrO-TiO$_2$—Continued

Specimen designation	Proportion of end members of join		Composition weight			Heat treatment			Absorption	Shrinkage	Dielectric constant, K, at 25° C and—			Reciprocal, Q, of power-factor at 25° C and—		
						No. 1	No. 2									
			BaO	SrO	TiO$_2$	1 hr at—	Temperature	Time			50 kc/s	1,000 kc/s	20,000 kc/s	50 kc/s	1,000 kc/s	20,000 kc/s
BaO:2TiO$_2$																
	Weight percent	*Weight percent*	*Percent*	*Percent*	*Percent*	*° C*	*° C*	*hr*	*Percent*	*Percent*						
BT2	--------	100	49.0	--------	51.0	1,100	1,290	1	.02	16.4	204	200	197	100	70	35
SrO:TiO$_2$ 2BaO:3TiO$_2$																
ST	100.0	0.0	--------	56.4	43.6	1,200	1,375	2	0.01	15.2	260	260	260	5,500	8,000	10,000
B2S1	89.0	11.0	6.2	50.2	43.6	1,100	1,350	1	.01	16.2	260	255	246	370	1,300	7,000
B2S2	76.4	23.6	13.2	43.2	43.6	1,100	1,350	1	.09	16.2	290	285	280	500	850	2,400
B2S3	67.7	32.3	18.1	38.3	43.6	1,100	1,310	1	.07	17.6	315	310	300	2,100	1,900	2,400
B2S4	57.7	42.3	23.7	32.7	43.6	1,100	1,310	1	.06	17.9	380	375	385	1,300	1,000	900
B2S5	50.0	50.0	28.1	28.2	43.7	1,100	1,290	1	.02	17.1	460	450	465	1,000	800	500
B2S6	40.0	60.0	33.7	22.6	43.7	1,100	1,290	1	.03	18.0	670	660	645	650	520	325
B2S72	27.8	72.2	40.5	15.7	43.8	1,100	1,290	1	.03	17.8	970	950	950	210	190	95
B2S83	17.0	83.0	46.6	9.6	43.9	1,100	1,290	1	.04	17.5	1,090	1,070	1,070	110	100	45
B2S9	10.0	90.0	50.5	5.6	43.9	1,200	1,285	2	.02	15.1	910	895	885	80	80	35
B2S95	5.0	95.0	53.3	2.8	43.9	1,200	1,285	2	.00	14.7	845	850	850	80	60	30
B2T3	0.0	100.0	56.1	--------	43.9	1,250	1,300	1	.01	10.8	910	900	890	70	50	25
SrO:TiO$_2$ BaO:TiO$_2$																
SB9	90.2	9.8	6.2	50.8	43.0	1,245	1,385	2	0.03	14.3	300	300	295	5,000	4,700	10,000
SB79	79.9	20.1	13.2	44.5	42.3	1,245	1,400	1	.03	15.0	355	355	345	>2,300	10,000	10,000
SB72	72.5	27.5	18.1	40.0	41.9	1,245	1,400	1	.03	14.6	415	415	405	>2,700	8,000	10,000
SB64	64.0	36.0	23.7	35.0	41.3	1,245	1,400	1	.10	13.3	510	515	505	3,000	7,200	10,000
SB57	57.4	42.6	28.0	32.4	39.6	1,245	1,420	2	.00	13.7	660	680	670	>5,000	10,000	9,000
SB50	50.0	50.0	32.8	28.2	39.0	1,245	1,420	2	.01	14.3	870	900	900	5,700	8,500	1,700
SB39	39.0	61.0	40.1	22.0	37.9	1,245	1,420	2	.00	13.2	1,600	1,600	1,600	2,000	4,500	1,200
SB34	34.8	65.2	42.8	19.6	37.6	1,245	1,400	1	.02	12.5	2,600	2,600	2,600	1,100	1,700	1,800
SB32	32.4	67.6	44.4	18.3	37.3	1,245	1,400	1	.01	12.5	3,300	3,300	3,300	700	1,000	900
SB29	29.0	71.0	46.7	16.3	37.0	1,245	1,420	2	.00	12.8	6,000	6,000	6,000	250	550	200
SB26	26.8	73.2	48.1	15.1	36.8	1,245	1,400	1	.00	12.3	7,000	7,000	7,000	110	380	165
SB23	23.8	76.2	50.1	13.4	36.5	1,245	1,420	2	.00	12.4	7,500	7,500	7,500	30	110	50
SB21	21.4	78.5	51.7	12.0	36.3	1,245	1,400	1	.00	11.7	3,300	3,300	3,300	27	110	45
SB18	18.5	81.5	53.5	10.4	36.1	1,245	1,400	1	.01	11.5	2,300	2,300	2,300	30	105	45
SB16	16.2	83.8	55.1	9.1	35.8	1,245	1,420	2	.00	11.4	1,900	1,900	1,900	35	120	60
SB1	10.0	90.0	59.1	5.6	35.3	1,245	1,385	2	.08	13.0	1,500	1,500	1,500	40	110	60
BT	0.0	100.0	65.7	--------	34.3	1,245	1,385	2	.05	10.6	1,500	1,500	1,500	80	100	50
3SrO:2TiO$_2$ BaO:TiO$_2$																
BS36	40.0	60.0	39.4	26.4	34.2	1,245	1,425	2	0.02	13.2	300	300	300	15	50	165
BS37	29.0	71.0	46.6	19.2	34.2	1,245	1,425	2	.06	14.6	900	900	900	400	850	700
BS375	24.7	75.3	49.5	16.3	34.2	1,245	1,425	2	.02	15.9	1,600	1,600	1,600	350	700	400
BS38	16.3	83.7	55.0	10.7	34.3	1,245	1,430	2	.01	13.5	4,400	4,400	4,400	35	155	80
BS39	10.0	90.0	59.1	6.6	34.3	1,245	1,425	2	.00	15.6	2,400	2,400	2,400	50	150	75
S$_5$T$_6$	--------	--------	-----	52.0	48.0	1,200	1,400	1	0.02	17.5	238	237	238	2,100	5,000	2,900
7ST	--------	--------	-----	54.0	46.0	1,200	1,400	1	.00	18.2	257	256	256	4,400	3,900	2,900
3ST	--------	--------	-----	58.0	42.0	1,200	1,400	1	.00	15.6	196	196	195	3,600	1,900	1,100

The range in composition of the specimens is illustrated by the diagram in figure 1.[4] Some of the preparations with compositions indicated by points on the join $3SrO:2TiO_2$–$BaO.TiO_2$(fig. 1), even though mature, disintegrated after several months storage in porous paper envelopes. As stability of capacitors after prolonged exposure to moisture in the air is important, several specimens of $SrO.TiO_2$ and those with compositions represented by points on the $BaO.TiO_2$–$SrO.TiO_2$ join were remeasured for K and Q after storage under room conditions for 6 months or more. The values of K and Q had not changed, which indicated that no disintegration had occurred.

Two objects of the heat treatments were (1) to expel the CO_2 from the carbonates, and (2) to induce reactions among the remaining oxides so that mature specimens (less than 0.10 percent of absorption) resulted. Although a single heat treatment sufficed for a few preparations, a preliminary heat treatment for calcining most of the batches was necessary. One advantage of calcination was the elimination of difficulties experienced in dry-pressing disks from the extremely fine-grained original materials.

In calcining the batches, a relatively high temperature was unsatisfactory. When such temperatures approached those for maturing the disks, the resulting calcines were more difficult to pulverize and to form into compact dry-pressed disks than were the calcines produced at temperatures 100°C or more below those of the final heat treatments. Possibly an objection to calcining at relatively low temperatures is the high shrinkage (10 to 20 percent) that occurs on maturing. Such an objection is more than offset by the ability to produce practically nonabsorbent specimens from these calcines, whereas the "high calcined" material was much more difficult to mature. In this study, over 500 disks were made because of the difficulty in finding the narrow range in temperature at which many of the specimens matured.

In the second heat treatment for preparation of mature specimens, the maximum temperature varied from 1,250°C, for $BaO:2.2\ TiO_2$, to 14,30°C for specimens with compositions in the join $BaO.TiO_2$–$3SrO:2TiO_2$ (fig. 1). Although the time at the maximum temperature of the heat treatments was varied widely, 1 to 30 hr, a duration of 1 or 2 hr was sufficient for calcining the batches and maturing the specimens. Approximate maturing temperatures are indicated in figure 2.

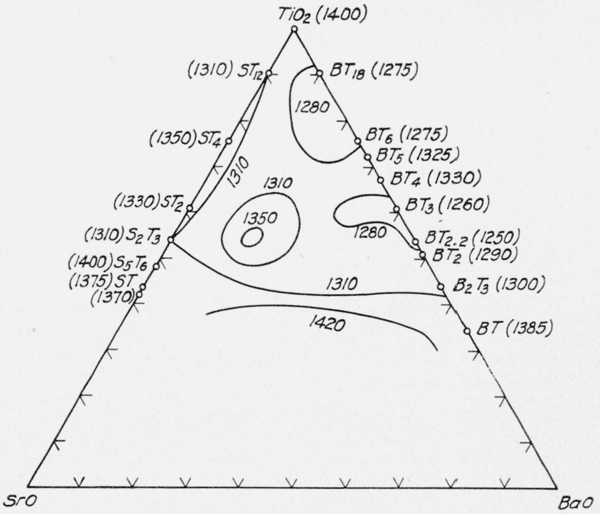

FIGURE 2.—*Maturing temperature after sintering treatment.*
B=BaO; S=SrO; T=TiO_2.

In the determination of the dielectric constant and Q, the advantage of specimens with low values of absorption (0.1 percent or less) is apparent from the data on specimens *TMO* (table 1), which are typical for all specimens studied. Although the values of K and Q decrease as the absorption increases, the amount of change in K is much less pronounced than that in Q. The values of K were duplicated usually within 1 or 2 percent, but variations of several percent in the values of Q were not uncommon.

The data in table 3 show the effect of variation in composition on the dielectric constant (K). Values of 91 to 98 for mature specimens of titania prepared from grades *TMO*, *A*, and *R*, table 1, are within the range of values obtained by other investigators [1, 2], for the dielectric constant of such material. Additions of BaO to TiO_2 resulted in (1) gradually decreasing values of K (at 25° C) from 98 to a minimum of 34 ($BaO:4TiO_2$), followed by (2) increasing values of K to a maximum of 1,500 ($BaO:TiO_2$). The values of K increased rapidly when the content of BaO was increased from 46.3 to 65.7 percent.

By contrast, additions of SrO to TiO_2 caused the values of K to increase gradually from 98 to 260

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[4] The abbreviated designation of the composition of specimens is evident from a comparison of column 1 with 2 and 3 in table 3. Thus, ST_{12} is an abbreviation for $SrO:12TiO_2$.

($SrO:TiO_2$). Similarly, increasing values of K were observed in some specimens containing both BaO and TiO_2 when the SrO content was increased and the concentration of TiO_2 maintained constant. This is illustrated by compositions on the joins $BaO:18TiO_2$-$SrO:12TiO_2$, $BaO:6TiO_2$-$SrO:4TiO_2$, and $BaO:3TiO_2$-$SrO:2TiO_2$, as shown in table 3. However, increasing the content of SrO with the concentrations of TiO_2 constant, but less than 61 weight percent, caused the values of K to increase rapidly to a peak and then to decrease. This is illustrated by compositions on the joins $BaO:2.2TiO_2$-$2SrO:3TiO_2$, $2BaO:3TiO_2$-$SrO:TiO_2$, and $BaO:TiO_2$-$SrO:TiO_2$. At 25° C, changes in the dielectric constant ranging from 1,000 to 3,000 resulted from small alterations in the compositions in the join $BaO:TiO_2$-$SrO:TiO_2$, as shown in table 3.

The dielectric constant (K), measured at 1 *mc/s*, was affected also by the temperature of the specimens, as indicated by the data in table 4. The values for 30°, 50°, and 80° C, can be found by interpolation for most of the specimens. The smallest variations in K were observed for specimens with binary compositions in the range 61.0 to 75.6 weight percent of TiO_2 and 24.4 to 39.0 weight percent of BaO, whereas the largest variations in K were found for specimens that had compositions represented by points in the join $BaO:TiO_2$-$SrO:TiO_2$.

TABLE 4.—*Dielectric constant, at 1 mc/s, from −60° to +85° C, and temperature coefficient of dielectric constant at 0° C*

Specimen [a] designation	Values of K at (° C)													Temperature coefficient of K at 0° C
	−60°	−50°	−40°	−30°	−20°	−10°	0°	+10°	+20°	+40°	+60°	+70°	+85°	
														ppm
TiO_2(A)[b]	105.7	104.5	103.3	102.3	101.2	100.3	99.5	98.7	97.9	96.4	95.1	94.3	93.3	−850
TiO_2(R)[b]	106.2	104.9	103.6	102.5	101.5	100.4	99.3	98.6	98.0	96.8	95.5	94.0	93.6	---------
ST12	131	128	126	123.5	121.5	120	118	116.5	115	112.5	110	109	107	−1350
18BS6	90.7	89.5	88.4	87.5	86.6	85.8	85.0	84.4	83.7	82.7	81.6	81.2	80.4	−830
BT18	80.5	79.7	78.9	78.3	77.6	77.0	76.5	76.0	75.4	74.3	73.4	73.0	72.5	−750
ST4	186	180	174	168	163	159	155	152	149	143	138	135	131	−2500
6BS2	152	147	142	138	135	132	128	126	123	118	114	112	110	−2200
6BS5	88.5	86.4	84.7	83.4	82.3	81.2	80.2	79.2	78.0	76.2	74.8	74.2	73.5	−1300
6BS7	56.3	55.8	55.4	55.0	54.6	54.3	54.0	53.7	53.3	52.5	52.0	51.7	51.3	−650
BT6	46.5	46.3	46.2	46.0	45.8	45.6	45.4	45.3	45.1	44.9	44.7	44.6	44.5	−370
5BS8	43.1	43.0	42.9	42.8	42.8	42.7	42.7	42.6	42.5	42.3	42.3	42.2	42.2	−200
BT5	36.7	36.7	36.7	36.7	36.7	36.7	36.7	36.7	36.7	36.7	36.7	36.7	36.7	0
4BS8	44.0	43.7	43.5	43.3	43.1	42.9	42.7	42.6	42.5	42.4	42.3	42.3	42.2	−380
4BS9	36.8	36.8	36.8	36.8	36.9	36.9	36.9	37.0	37.0	37.2	37.3	37.3	37.4	+100
BT4	33.0	33.1	33.2	33.3	33.4	33.5	33.6	33.7	33.7	33.9	34.0	34.0	34.0	+260
ST2	255	244	233	224	214	206	200	195	189	180	171	167	162	−3000
BS1	220	207	197	190	183	178	173	168	164	156	149	146	141	−2300
BS3	160	151	145	140	135	132	128	126	123	118	114	112	109	−2400
BS4	112	108	105	103	100	98	96	94	92	89	86	84	82	−2200
BS6	92.8	89.5	86.7	84.5	82.0	80.0	78.5	77.0	76.0	73.5	71.2	70.3	69.0	−1900
BS7	78.9	76.3	74.0	72.0	70.2	68.6	67.4	66.2	65.2	63.4	61.8	61.2	60.3	−1800
BS8	55.9	54.8	53.9	53.2	52.6	52.1	51.8	51.4	51.1	50.4	50.0	49.8	49.4	−730
BS9	52.4	52.0	51.5	51.1	50.8	50.4	50.1	49.8	49.5	49.0	48.6	48.4	48.1	−640
BT3	44.6	44.5	44.5	44.4	44.3	44.3	44.2	44.1	44.1	43.9	43.8	43.8	43.7	−160
S2T3	274	262	250	240	230	221	213	207	202	193	184	179	173	−3200
BS22	210	201	191	183	175	168	162	158	154	146	139	136	132	−3100
BS23	230	216	203	192	183	175	167	161	155	144	134	131	126	−4100
BS25	241	220	202	190	179	170	162	156	150	140	131	128	122	−4400
BS26	216	198	182	170	159	151	145	140	135	127	118	115	111	−4100
BS271	303	277	253	230	208	194	181	170	160	145	132	127	123	−5900
BS279	288	292	296	297	293	284	269	246	226	187	162	152	141	---------
BS28	227	234	239	243	249	252	254	256	256	247	226	214	190	---------
BS29	174	181	185	192	196	198	201	204	207	216	225	231	230	---------
BT2.2	85.2	86.6	88.0	89.3	90.8	92.0	93.2	94.1	95.1	96.8	98.5	99.5	101	+1200
BT2	163	168	173	177	182	187	192	196	200	204	210	214	221	+2600

See footnotes at end of table.

TABLE 4.—*Dielectric constant, at 1 mc/s, from −60° to +85° C, and temperature coefficient of dielectric constant at 0° C*—Con.

Specimen [a] designation	Values of *K* at (° C)													Temperature coefficient of *K* at 0° C
	−60°	−50°	−40°	−30°	−20°	−10°	0°	+10°	+20°	+40°	+60°	+70°	+85°	
														ppm
S5T6	340	320	305	290	275	265	257	250	243	227	215	208	200	−3400
3ST	231	222	214	206	200	194	189	184	181	173	166	163	158	−2600
7ST	360	342	325	312	300	289	279	269	262	244	231	224	215	−3500
ST	367	345	330	315	300	290	280	270	261	246	232	226	219	---------
B2S1	382	359	340	324	309	295	282	271	260	241	226	221	213	−4400
B2S2	479	442	412	386	362	341	320	304	289	270	250	242	229	−5700
B2S3	560	505	460	425	395	370	350	330	317	290	267	258	245	−5600
B2S4	825	710	630	570	520	475	435	405	385	345	310	295	280	−6200
B2S5	1290	1090	940	820	720	640	570	520	470	420	370	350	320	−10700
B2S6	1760	1650	1480	1270	1090	970	860	780	700	575	490	450	410	---------
B2S72	1160	1190	1200	1210	1200	1180	1130	1080	1015	825	675	610	530	---------
B2S83	920	950	980	1010	1025	1045	1055	1060	1085	1090	980	910	800	---------
B2S9	735	775	810	845	880	910	940	970	995	1055	1120	1155	1125	---------
B2S95	610	640	670	700	730	755	780	805	825	870	920	945	980	---------
B2T3	600	630	660	690	720	745	780	825	870	900	920	935	980	---------
SB9	466	424	397	381	348	343	325	313	303	283	264	256	247	−3800
SB79	622	565	520	485	450	422	397	380	362	332	306	297	279	−5000
SB72	827	755	690	635	580	535	500	460	430	385	345	330	311	−7600
SB64	1415	1210	1020	870	770	690	630	580	535	470	420	395	367	−9200
SB57	3000	2150	1500	1210	1020	890	780	710	645	550	480	450	410	---------
SB50	13400	6300	3700	2400	1850	1450	1200	1020	925	750	625	580	520	---------
SB39	3200	3450	4000	5800	11500	7200	4000	2500	1900	1270	950	850	740	---------
SB34	3720	3640	3800	4200	5400	12000	10000	5000	3600	2000	1350	1170	970	---------
SB32	3340	3300	3300	3400	3800	5700	15000	11000	6000	2450	1550	1320	1070	---------
SB29	2180	2370	2400	2400	2450	2800	3600	10000	8500	3200	1820	1370	1190	---------
SB26	1970	2230	2750	2750	2750	2750	3200	5000	13800	4700	2360	1850	1420	---------
SB23	1240	1400	2180	2060	1980	1980	2020	2300	3000	7500	3100	2400	1720	---------
SB21	1450	1700	2570	2450	2350	2350	2350	2350	2600	11000	4800	3500	2370	---------
SB18	1250	1400	1600	1850	2150	2150	2050	2100	2200	3100	8500	5200	3200	---------
SB16	940	1050	1250	2000	1850	1710	1650	1650	1700	2050	7300	6500	3740	---------
SB1	780	830	900	950	1100	1700	1600	1500	1450	1410	1600	2050	8200	---------
BS37	1670	1800	2270	2000	1600	1350	1100	950	820	690	560	500	435	---------
BS375	2330	2400	2600	2900	3650	4000	3080	2300	1850	1250	920	820	690	---------
BS38	1290	1650	1950	2050	2100	2150	2300	2600	3300	4320	2380	1950	1420	---------
BS39	1250	1350	1550	1800	2450	2400	2300	2250	2200	3000	7500	6000	2100	---------
BT	805	815	860	910	980	1060	1160	1310	1570	1360	1300	1330	1440	---------

[a] For compositions, see tables 1 and 3.

[b] Grade designation (table 1).

The effect of temperature, over the range −60° to +85° C, upon the dielectric constant of specimens in the system BaO-TiO_2 is illustrated by figure 3. In diagram *A* the negative temperature coefficient of *K* for titanium dioxide is indicated by the curve TiO_2. It is seen that increasing the content of BaO in the specimens (to approximately 28 weight percent), as in BaO:5TiO_2, resulted in a reduction of the negative temperature coefficient. Attention is directed to the specimen containing 27.7 percent of BaO, because the dielectric constant remained unaltered by changes in temperature within the range −60° to +85° C, as shown by the curve BaO:5TiO_2. Certain irregularities in the dielectric constant appeared when the concentration of BaO was more than 28 weight percent. Specimen BaO:4TiO_2 (32.4 percent of BaO) had a slightly positive temperature coefficient of *K* as well as the lowest observed value for *K*, as shown by the curve BaO:4TiO_2 in diagram *A*, figure 3. A further increase of 6.6 percent of BaO resulted not only in a higher value of *K* but also in a slightly negative temperature coefficient, as illustrated by curve BaO:3TiO_2 in diagram *B*. As the concentration of BaO increased within the range 39 to 65.7 weight percent (BaO:3TiO_2 to BaO.TiO_2), the dielectric constant of the specimens exhibited more pronounced changes with temperature as well as increased values of *K*, as illustrated by

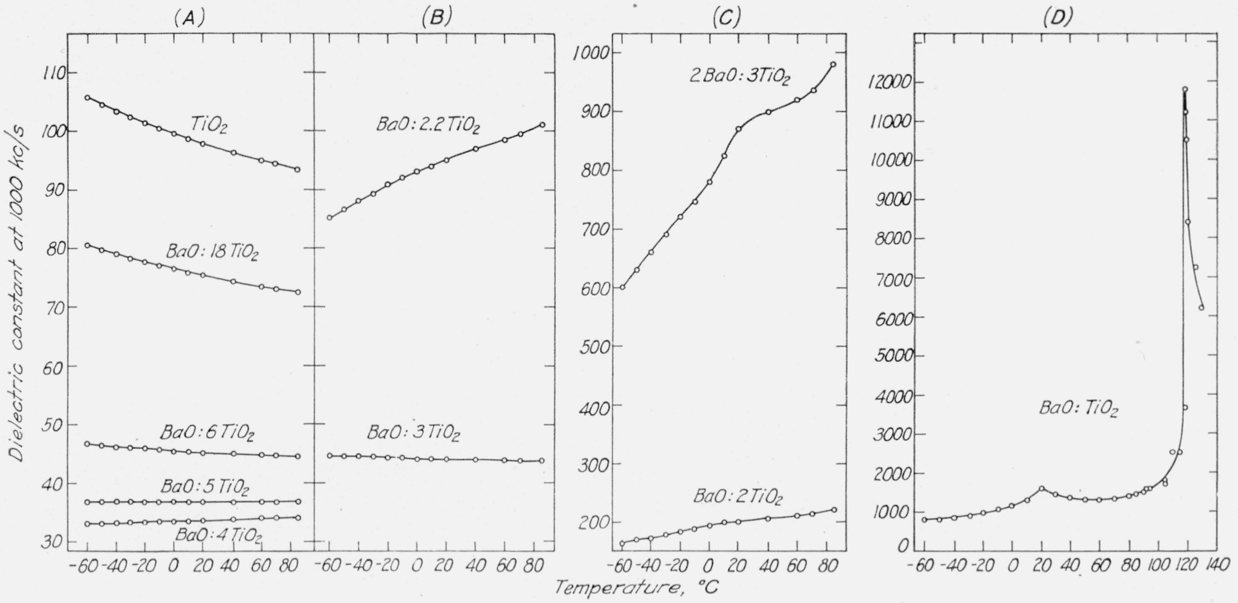

FIGURE 3.—*Variation of dielectric constant with temperature, at 1,000 kc/s, for specimens with compositions in the binary system,* TiO_2–$BaTiO_3$.

diagrams *B*, *C*, and *D* (fig. 3). It should be noted that the scales were altered in the ordinate of diagrams *C* and *D*, because of the high values of *K*.

Other investigators [1, 2] have found considerable variation in dielectric constant with changes in temperature of specimens prepared from $BaO.TiO_2$. In the present study, diagram *D*, figure 3, shows the character of the dielectric-constant–temperature curve for these specimens. Although a peak value of 11,800 *K* was observed at 118° C, variations of as much as 1,000 or more in the values of *K* were caused by small changes in temperature within the range of 110° to 125° C. The rapid variation of *K* with temperature occurs at a reversible change in crystal structure from pseudocubic to the cubic perovskite structure [2], indicated by X-ray studies.

Additions of SrO to specimens containing BaO and TiO_2, denoted by the joins in the diagram of figure 1, resulted in negative temperature coefficient of *K* for most of the specimens, as shown in the last column of table 4. Coefficients were not computed for all the specimens because of the irregularities in the curves of dielectric constant versus temperature.

The portion of the ternary diagram in which peaks were observed in the dielectric-constant—temperature curves is indicated in figure 4. These peaks were a maximum for specimens with compositions indicated by points in the join $BaO.TiO_2$-$SrO.TiO_2$. The curve with a peak at 41° C (fig. 5) for one of these specimens, *SB21*, resembles the curve for $BaO.TiO_2$ (fig. 3, *D*). It was found that increasing the content of SrO in this series of compositions on the $SrO.TiO_2$-$BaO.TiO_2$ join resulted in decreasing the temperature at which the peak in dielectric constant occurred (fig. 6). Extrapolation of the curve indicates a peak for

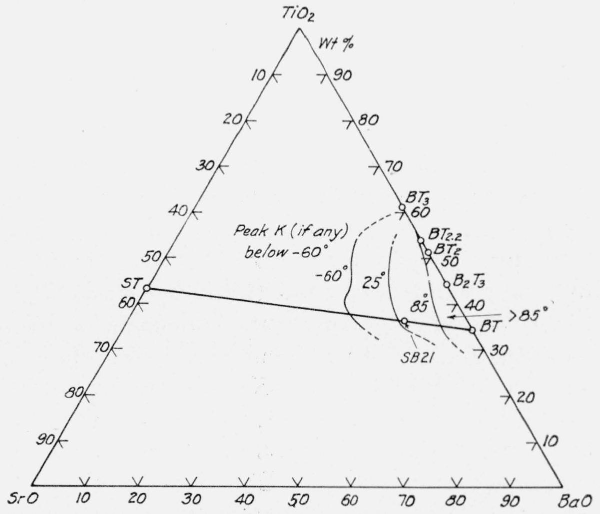

FIGURE 4.—*Relation between the temperature at which the peak dielectric constant occurs and compositions in the* BaO-SrO-TiO_2 *system.*

B=BaO; S=SrO; T=TiO_2.

$SrTiO_3$ at a temperature of approximately −240° C. Similar data by other investigators indicate that this peak is near −260° C [7].

Figures 7, 8, and 9 show the lines of constant values of K at −60°, 0°, and +60° C with variations in composition over a portion of the ternary system BaO-SrO-TiO_2. These diagrams indicate the changes in the values of K that result from simultaneous alterations of both temperature and composition.

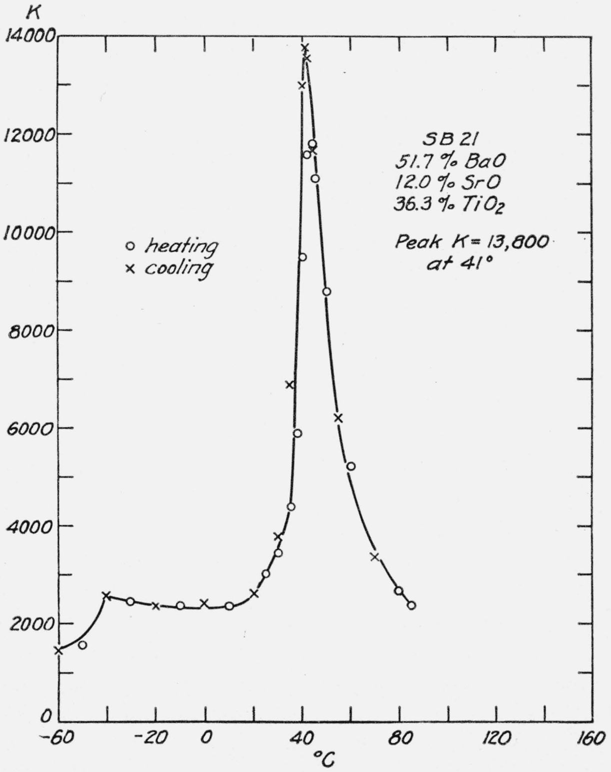

FIGURE 5.—*Variation of dielectric constant with temperature, at 1,000 kc/s, for specimen SB21 with composition on the* $SrTiO_3$-$BaTiO_3$ *join.*

Despite the relatively low dielectric constant (37) of specimens having the composition $BaO{:}5TiO_2$, the zero temperature coefficient of K is noteworthy. It was found, however, that an addition of 5 percent of SrO to this composition led to a negative coefficient, as in specimens 5BS8 (table 4). Specimens 4BS8 also had a low negative coefficient, whereas 4BS9 had a positive coefficient. Thus, specimens having zero temperature coefficient of K are limited in composition to a small region in the vicinity of the composition indicated by the point BT5, figure 1. For some uses, such as delay lines, the dielectric with zero temperature coefficient is desirable.

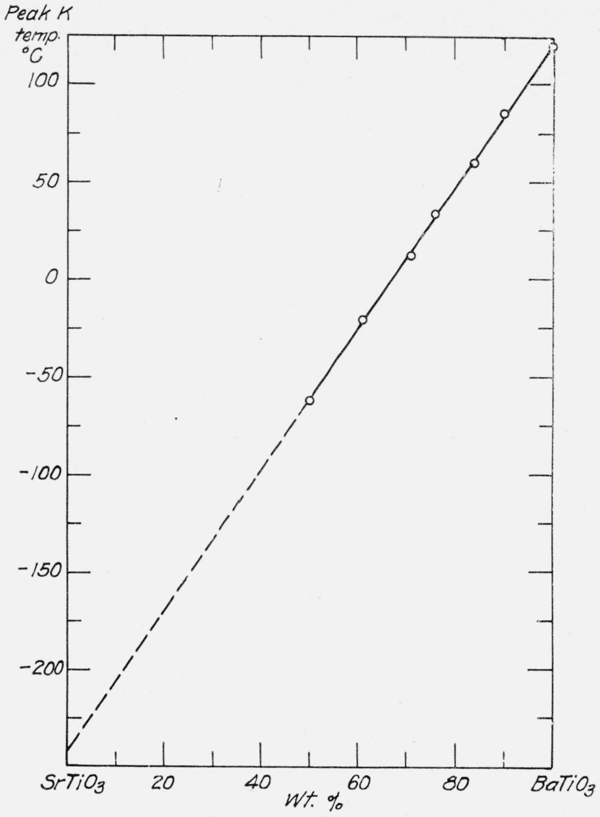

FIGURE 6.—*Variation of the temperature at which the peak dielectric constant occurs with composition on the* $SrTiO_3$-$BaTiO_3$ *join.*

The dielectric constant was not affected by variations in frequency within the range 50 to 20,000 kc/s. It is believed that the slight differences in observed values of K are within experimental error.

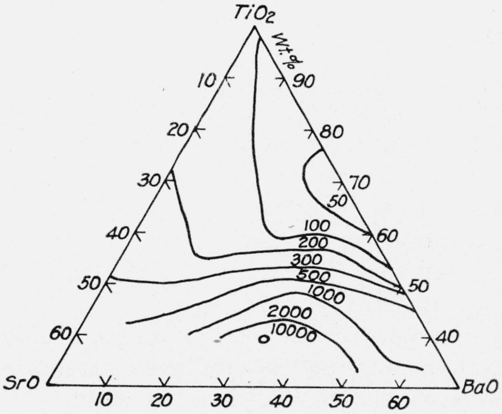

FIGURE 7.—*Constant K with varying compositions at −60° C.*

The variation in values of Q caused by alterations in composition and frequency is shown by the data in table 3. Although most of the specimens had relatively high values of Q (400 to 10,000), those containing more than 46.0 percent of BaO exhibited low values of from 50 to 100. With specimens having a constant and relatively low content of TiO_2, an increase in the concentration of SrO was accompanied by increased values of Q, as illustrated in table 3. As an absorption of over 0.01 percent was attended by a lower value of Q, some of the specimens with absorptions over 0.01 percent probably exhibited values of Q lower than the best obtainable.

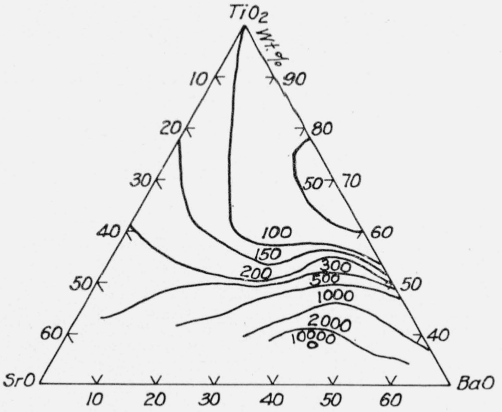

FIGURE 8.—*Constant K with varying compositions at 0° C.*

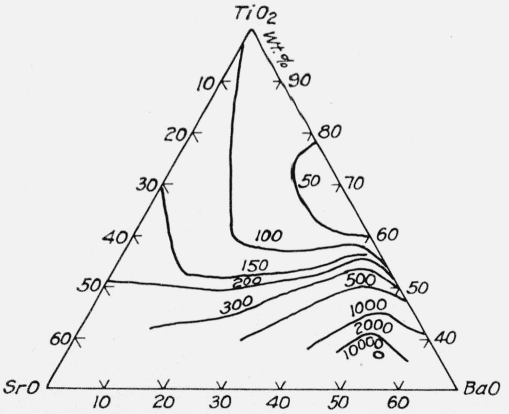

FIGURE 9.—*Constant K with varying compositions at +60° C.*

TABLE 5.—*Dielectric constant K, and Q, the reciprocal of the power factor, at 25° C and 1 mc/s and 3,000 mc/s*

Specimen designation	K at 1 mc/s	Q at 1 mc/s	K at 3,000 mc/s	Q at 3,000 mc/s
BaO: $3TiO_2$	43	3, 600	42	460
BS6	75	2, 400	76	520
BS7	71	1, 000	74	310

The data in table 3 show little correlation of Q with frequency. In some instances, a specimen had low Q at 50 kc/s and fairly high Q at 20,000 kc/s, for example, *BT*6. Often a duplicate specimen exhibited a high Q at all frequencies. As an example of the variation in Q values of specimens receiving the same preparation and heat treatments, the data in table 6 are given. Although the variation in the dielectric constant is not large, the Q values show a very large variation. However, the specimen with the highest absorption has the lower Q value. The reason for these differences is not apparent, but it may be connected with a number of factors, such as the size of crystals and pores, as well as the state of oxidation of the titanium compounds.

Variations in the properties of specimens of the same composition, *SB*50, which received different heat-treatments, are given in table 7. These specimens have a fairly large maturing range with small variation in dielectric constant. Similar data could be given for most of the other compositions of low BaO content.

TABLE 6.—*Properties of specimens of* $SrTiO_3$, *sintered for 1 hr at 1,245° C and heated at 1,350° C for 1 hr*

Absorption	Shrinkage	Dielectric constant (K), 25° C			Reciprocal (Q) of power factor, 25° C		
		50 kc/s	1,000 kc/s	20,000 kc/s	50 kc/s	1,000 kc/s	20,000 kc/s
Percent	*Percent*						
0.11	14.8	268	264	260	450	1, 350	3, 500
0.06	15.0	249	246	243	2, 100	3, 200	4, 800
0.05	15.0	258	254	249	900	4, 000	8, 000
0.05	14.8	253	250	245	1, 000	1, 350	4, 500
0.01	15.2	260	260	260	5, 500	8, 000	10, 000
0.00	15.0	252	249	245	900	4, 000	8, 000

TABLE 7.—*Properties of specimens of SB50, sintered at 1,245° C for 1 hr, and heated at 1,350° C, 1,385° C, and 1,420° C for 2 hr*

Heated 2 hr	Absorption	Shrinkage	Dielectric constant (*K*), 25° C			Reciprocal (*Q*) of power factor, 25° C		
			50 kc/s	1,000 kc/s	20,000 kc/s	50 kc/s	1,000 kc/s	20,000 kc/s
° *C*	*Percent*	*Percent*						
1,350	0.02	14.3	870	900	890	2,900	6,100	1,600
1,350	.05	14.3	870	890	900	3,400	5,800	1,300
1,385	.05	14.2	870	870	890	5,400	7,000	1,600
1,385	.07	14.3	870	870	890	5,700	7,500	1,400
1,420	.01	14.3	860	860	870	2,800	8,500	1,700
1,420	.02	14.3	870	860	870	2,300	7,500	1,200

In the specimens having a high BaO content, and low *Q* values, the variations in the dielectric constant and the *Q* values are more nearly equal, as shown by the data in table 8. In general, for specimens having a low BaO content, the variations in the dielectric constant, for well vitrified samples, are much less than the variations in the *Q* values, although for specimens of high BaO content, and low *Q* values, these variations are more nearly equal.

TABLE 8.—*Variations in properties of some specimens containing over 40 percent of BaO*

Specimen designation	Heat treatments				Absorption	Dielectric constant (*K*), 25° C			Reciprocal (*Q*) of power factor		
	Temperature	Time	Temperature	Time		50 kc/s	1,000 kc/s	20,000 kc/s	50 kc/s	1,000 kc/s	20,000 kc/s
	° *C*	*hr*	° *C*	*hr*	*Percent*						
SB16	1,245	1	1,375	2	0.00	1,930	1,850	1,810	33	74	60
	1,245		1,385	2	.05	1,890	1,840	1,820	36	112	62
	1,245		1,420	2	.00	1,920	1,850	1,790	36	119	62
B_2S95	1,200	1	1,310	2	.05	860	830	820	83	61	28
	1,200		1,285	2	.00	840	820	810	74	52	27
BT	1,245	1	1,385	2	.05	1,460	1,410	1,360	79	93	50
	1,245		1,425	2	.08	1,480	1,480	1,410	82	84	44
Bt [a]	1,245		1,400	1	.03	1,690	1,650	1,670	134	74	28
Bt [b]	1,245				1.07	1,330	1,300	1,330	98	144	78

[a] $BaTiO_3$ made with TiO_2, grade *WD*.
[b] $BaTiO_3$ made with TiO_2, grade *R*.

Coating the specimens, particularly those having relatively high absorption, with wax or paraffin increased the values of *Q*. The improvement, however, was not permanent, for on standing a gradual decrease in *Q* was noted.

At a frequency of 3,000 mc/s, a greater difference was observed in the values of *Q* than of *K* compared with their respective values found at a frequency of 1 mc/s, as indicated by the data in table 5. As most specimens had a dielectric constant that was too high for determination by this method, only these three specimens were measured at 3,000 mc/s.

The thermal expansion of the specimens was high, ranging from 0.59 to 0.84 percent between room temperature and 700° C, despite the wide variation in composition (table 9). Figure 10 illustrates a peculiar feature of the expansion curve for specimens with compositions in the join $BaO:TiO_2$-$SrO:TiO_2$. The flat section of these curves occurred in the range of temperature characterized by a peak in the dielectric constant.

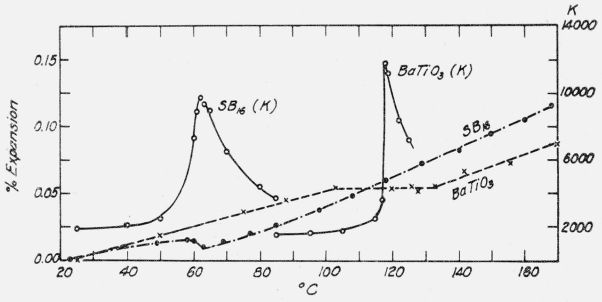

FIGURE 10.—*Thermal expansion for a specimen of* $BaTiO_3$ *and for SB16 (table 3) in the region of the peak dielectric constant, over the range 25° to 170° C.*

Solid lines for dielectric constant and broken lines for thermal expansion.

TABLE 9.—*Linear thermal expansion*

Specimen designation	Temperature range from 25° C to—								
	100° C	200° C	300° C	400° C	500° C	600° C	700° C	800° C	900° C
	Percent	*Percent*	*Percent*	*Percent*	*Percent*	*Percent*	*Percent*	*Percent*	*Percent*
TiO_2	0.06	0.14	0.23	0.32	0.41	0.50	0.60	0.69	0.79
BT18	.05	.14	.23	.31	.41	.50	.59		
BT6	.05	.14	.24	.33	.43	.54	.64		
BT5	.06	.15	.24	.34	.45	.57	.68		
BT4	.06	.14	.23	.32	.41	.51	.60	.70	
BT3	.06	.16	.26	.37	.48	.59	.71		
BT2.2	.06	.16	.26	.37	.49	.61	.72		
BT2	.07	.16	.28	.40	.52	.65	.78		
BT	.05	.13	.25	.38	.52	.66	.80		
SB64	.07	.18	.30	.42	.55	.67	.80		
SB29	.07	.19	.32	.44	.58	.71	.84		
SB29F [a]	.07	.19	.29	.41	.54	.68	.79		
SB16	.04	.16	.28	.41	.55	.68	.81		
ST	.07	.17	.29	.40	.51	.63	.74		
ST2	.06	.16	.26	.36	.46	.55	.65	.75	
ST4	.06	.15	.25	.35	.44	.55	.65		

[a] 3% MgF_2 added.

Although the construction of a phase diagram for this system was outside the scope of this investigation, there were indications, based on maturing temperatures, of the existence of compounds in various regions of the diagram, figure 2. For example, a binary compound in the region of BaO:4TiO_2 is suggested by the maturing temperature, dielectric constant, and X-ray spectrogram. In fact, a comparison of X-ray patterns for all the specimens in the system BaO-TiO_2 indicated the formation of other compounds in addition to BaO:4TiO_2 and BaO:TiO_2.[5] The maturing temperature of 1,350° C for specimens in the region 18 percent BaO, 28 percent SrO, 54 percent TiO_2 is suggestive of a ternary compound, but the suggestion is not supported by data on the dielectric constant or by X-ray patterns.

V. Conclusions

Mature specimens (absorption less than 0.10 percent) can be made from mixtures of titanium dioxide with barium and strontium carbonates, which have compositions represented by points in the system $BaTiO_3$-$SrTiO_3$-TiO_2. The dielectric constant of these specimens varies from 34 to several thousand, whereas the Q values vary from about 50 to several thousand. Most of these specimens exhibit a large negative temperature coefficient of dielectric constant, which is not too great in some to prevent their use as bypass and coupling condensers. A few have positive temperature coefficients, although that of one specimen (BaO:5TiO_2) is practically zero. When properly fabricated, some of these titanate bodies could be used also as filter or storage condensers, and as delay lines for microwave signals.

VI. References

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[5] The authors are indebted to H. F. McMurdie for the X-ray spectrograms and their interpretation.