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SOME PHYSICAL PROPERTIES OF MICA

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ABSTRACT

This paper gives data on the linear thermal expansion, changes in structure, power factors, and effects of heat treatments on the thickness, opacity, and color of micas (muscovite, phlogopite, biotite, ripidolite, and zinnwaldite) from different domestic and foreign sources. Tremendous linear thermal expansion was noted for some samples of phlogopite and biotite micas in a direction perpendicular to the cleavage plane. The transitions shown in the expansion curves of these samples at elevated temperatures appear to be related to the structural changes indicated in the X-ray diffraction patterns. Heat treatment of two phlogopite micas and two muscovite micas to 600° C, with or without a load, caused considerable increases in the power factors of the former and only slight changes in the latter. Nearly all the muscovite samples showed the greatest increases in thickness (up to 155 percent) after heat treatment at 800° C. The large increases in thickness of nearly all the muscovite samples accompanied changes from clear or translucent to opaque, or from polychrome to metallic color. None of the species of mica can be considered as a substance or material of fixed and reproducible properties. The physical properties of mica depend largely upon the chemical composition, the nature of the crystals, their magnitude and orientation, the presence of impurities, the way in which these enter the structure, the heat treatment, etc. Some of the phlogopite and biotite micas that possess tremendously high thermal expansion in a direction perpendicular to the cleavage plane may be used for high-expanding elements in temperature-responsive devices but may be unsatisfactory for use in devices in which large changes in dimensions with changes in temperature are not desirable.

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I. INTRODUCTION

As an outgrowth of tests made in connection with a military problem, an investigation of the linear thermal expansion of various kinds of micas (muscovite, phlogopite, biotite, ripidolite, and zinnwaldite) was undertaken. The determinations were made in a direction perpendicular to the cleavage plane of the mica. The work was extended to include an investigation of structural changes indicated by Laue X-ray diffraction patterns, power factors, and effects of heat treatments on thickness, opacity, and color.

Mica [1] [1] [2] is one of the most important strategic materials in time of war [3], and is indispensable in some modern applications in

TABLE 1.—*Sources and varieties (or qualities) of mica*

Sample No.	Source	Variety or quality [a]	Submitted by—
		A. MUSCOVITE MICA	
1682	Brazil	White 1441 punch	BG Corporation.
1684	Canada	Amber	Simmonds Aerocessories, Inc.
1685	Brazil	No. 3	Do.
1686	do	No. 5	Do.
1687		Plate mica (processed sheet supplied by New England Mica Co).	Do.
1689	Los Volcansillos Mine, Guatemala	Verde claro	Office of Economic Warfare.[b]
1690	do	Verde oscuro	Do.
1691	Durania Section, Colombia	Black stained [c]	Do.
1692	do	Stained [d] or good stained [e]	Do.
1693	El Mico Prospect, San José de la Montaña Mica District, Colombia.		Do.
1694	Near Camana, Peru		Do.
1695	Near Camana, Ferreyros, Peru		Do.
1696	Amber Mine, Concepcion Area, Depto. Santa Cruz, Bolivia.		Do.
1697	La Negra Mine, Concepcion Area, Depto. Santa Cruz, Bolivia.		Do.
1698	San Juan Mine, Concepcion Area, Depto. Santa Cruz, Bolivia.		Do.
1699	New Hampshire	First quality	Colonial Mica Corporation.
1700	do	Second quality	Do.
1701	Old Mike Mine, 4½ miles north of Custer, S. Dak.	First quality	Do.
1702	do	Second quality	Do.
1703	Mill Race Mine, Avery County, N. C.	Green, first quality	Do.
1704	do	Green, second quality	Do.
1705	Sink Hole Mine, near Bandana, Mitchell County, N. C.	Ruby, first quality	Do.
1706	Hawkins Mine, Stokes County, N. C.	Ruby, second quality	Do.
1707	Petaca Area, N. Mex	First quality	Do.
1708	do	Second quality	Do.
1729	Purdy Mine, Mattawan Twp., Ontario, Canada.	Clear,[f] No. 1 quality	Bureau of Mines, Canada.
1731	Simard Mine, Bergeronnes Twp., Saguenay Co., Quebec, Canada.	Ruby	Do.
1741	San Jorge Mine, Concepcion Area, Depto. Santa Cruz, Bolivia.		Office of Economic Warfare.[b]
1742	Cordoba, Argentina	Semi clear	Do.
1743	do	Semi stained	Do.
1744	do	Stained [d]	Do.
1745	do	Light green	Do.
1746	Catamarca, Argentina	Clear [f]	Do.
1747	do	Semi clear	Do.
1748	San Luis, Argentina	do	Do.
1749	Hibbs Mine, Hebron, Maine		H. W. Bearce, National Bureau of Standards.

[a] For classification of quality of mica, see ASTM designation: D351-38, ASTM Standards, Part III, Nonmetallic Materials, p. 391 (1942) (Am. Soc. Testing Materials, Philadelphia, Pa.).

[b] Now Foreign Economic Administration.

[c] *Black stained:* Apt to contain some mineral inclusions consisting of magnetite (black).

[d] *Stained:* Free of mineral inclusions and cracks, but may contain considerable clay and vegetable stains and may be more wavy and softer than the better qualities.

[e] *Good stained:* Free of mineral inclusions and cracks but contains air inclusions, some vegetable inclusions, and may be somewhat wavy.

[f] *Clear:* Free of all mineral and vegetable inclusions, stains, air inclusions, waves, or buckles. Hard transparent sheets.

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[1] The designation "mica" has been traced back to a publication by Agricola in 1546.

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

[3] It has been stated in the public press that mica is probably the most important single war material for without it "there would be no radar, no radio, no electric gun controls, and no detector devices."

TABLE 1.—*Sources and varieties (or qualities) of mica*—Continued

Sample No.	Source	Variety or quality [a]	Submitted by—
		B. PHLOGOPITE MICA	
1681	Madagascar	Black, No. 995	BG Corporation.
1683	do		Simmonds Aerocessories, Inc.
1688	Mozambique, Africa		Office of Economic Warfare.[b]
1709	do		Do.
1710	do	Silver	Do.
1711	Burgess, Canada	National Museum specimen No. 78218.	National Museum.
1712	St. Lawrence County, N. Y.	National Museum specimen No. C3681.	Do.
1732	St. Lawrence Mica Co. Mine, Petit Pré., Quebec, Canada.	Spark plug grade	Bureau of Mines, Canada.
		C. BIOTITE MICA	
1713	E. Blake Mine, Newdale, N. C.	National Museum specimen No. 90467.	National Museum.
1730	Faraday Twp., Hastings Co., Ontario, Canada.		Bureau of Mines, Canada.
1733	Ledford Cove Mica Mine, Macon County, N. C.		Geological Survey.
1734	Saunders Quarry, Oxford County, Maine.		Do.
		D. RIPIDOLITE MICA	
1714	Three miles south of Westchester, Pa.	National Museum specimen No. 79023.	National Museum.
		E. ZINNWALDITE MICA	
1735	Morefield Mine, Amelia, Va		Geological Survey.

time of peace. It is used in many products, such as condensers, electric generating and motive equipment, radio tubes, airplane spark plugs, electric-light bulbs and sockets, X-ray apparatus, heating elements, and so forth.

Samples of mica were obtained from the corporations and Government agencies indicated in table 1.

II. MATERIAL INVESTIGATED

Fifty samples consisting of five types of mica were investigated for three or more of the following physical properties: Thermal expansion; changes in structure; power factors; and effects of heat treatments on thickness, opacity, and color. Available information about the sources and the varieties (or qualities) of these samples is indicated in table 1.

No chemical analyses were made on the samples of mica used in this investigation. The chemical composition of muscovite[4] mica may be represented approximately by $[Al_2]$ $(AlSi_3)O_{10}(OH, F)_2$ K. Aluminum, $[Al_2]$, is replaced by magnesium in phlogopite[5] mica,

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[4] The name *muscovite* for potash mica was reported by De Boot [2] to be derived from the locality Muskovia in Russia, where this mica was found.

[5] Phlogopite mica has a pearly to submetallic luster on cleavage surfaces, with a coppery reflection, which gave rise to its name, derived from the Greek word "phlogopos," firelike [1].

$[Mg_3](AlSi_3)O_{10}(OH, F)_2 K$, and by magnesium, lithium, titanium, ferric and ferrous iron in other micas. Additional information about the chemical composition of different micas may be obtained from publications by Horton [3], Hendricks and Jefferson [4], Pauling [5], Mohr [6], and Mellor [7]. Mica contains chemically combined water. The most common impurities in mica are iron oxides, silica, and clay. The spotting or staining of mica has been ascribed to the inclusion of thin scales or streaks of dark minerals, most frequently iron oxides.

Hendricks and Jefferson [4] stated that moscovite mica has a unique structure because of its distorted layer, whereas the biotite micas have variable structures because their layers are symmetrical. The distortion of the layer in muscovite mica is a result of incomplete filling of the octahedral positions, so that some octahedral edges will be differently surrounded than others. All octahedral positions in the biotite micas are filled and the octahedral edges are equivalent.

Hendricks and Jefferson [4] assumed that many elements of a mosaic in a mica crystal showing a well-developed structure are perfectly regular, and any diffuse scattering is due to random shifts in a limited number of the mosaic elements.

III. PHYSICAL PROPERTIES

1. THERMAL EXPANSION

The fused-quartz-tube expansion apparatus described by Hidnert and Sweeney [8] was modified so that short samples of mica could be used. With two exceptions,[6] each sample consisted of 20 to 105 disks of mica, approximately 16 mm in diameter. The disks of various thicknesses were stacked to form a cylindrical sample 16 to 23 mm in height (see footnote 6), in order to determine the linear thermal expansion in a direction[7] perpendicular to the cleavage plane. The sample rested on a flat disk of fused quartz, which was placed at the bottom of the fused-quartz tube in order to avoid bending the disks of mica by the curvature of the bottom surface of the tube. Similarly, a flat disk of fused quartz was placed on top of the sample. A 45-cm movable fused-quartz rod rested on the upper flat disk of fused quartz and extended above the open end of the fused-quartz tube. This rod is hollow but closed at the ends. The bottom of the rod is concave and the top is flat. A bar of steel (about 38 cm long) with a weight at each end was placed horizontally at the top of the 45-cm movable fused-quartz rod, so that the center of gravity of the steel bar and weights was at a point on the vertical axis of the fused-quartz rod. The foot of a dial indicator graduated to 0.01 mm rested on top of the steel bar. One junction of a Chromel-Alumel thermocouple was attached to the side of the upper flat disk of fused quartz above the sample of mica. An electric furnace surrounding the tube containing the sample was used to control temperature during heating and cooling.

Each sample of mica was subjected to a load represented by the sum of the weights of the upper fused-quartz disk, the movable

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[6] Sample 1681A consisted of 1 disk 0.3 mm in height (thickness). Sample 1709 consisted of 10 disks and was 3 mm in height.

[7] The axis of the cylindrical sample represented the direction perpendicular to the cleavage plane of the mica.

fused-quartz rod, the steel bar and weights, and the force exerted by the foot of the dial indicator. In most of the expansion determinations, the load on the sample was 30 lb/in.^{2} (2.11 kg/cm ^{2}). In nine determinations the load [8] was 1 lb/in.^{2} (0.07 kg/cm ^{2}) and in one determination the load was 10 lb/in.^{2} (0.70 kg/cm ^{2}).

The dial indicator fastened near the top of the fused-quartz tube indicated the differential expansion between the sample and an equivalent length of fused quartz. A correction for the small expansion [9] of fused quartz was applied. When enormous expansion beyond the range (13 mm) of the dial indicator was about to occur, the dial indicator was removed from the fused-quartz tube, and measurements of the displacements of the upper surface of the movable fused-quartz rod above the upper end of the fused-quartz tube were made with calipers and a steel scale graduated in millimeters. When the sample had contracted sufficiently during cooling so that it was again possible to make observations within the range of the dial indicator, the indicator was replaced on the fused-quartz tube.

Figures 1 and 2 of the publication by Hidnert and Sweeney [8] show photographs of the fused-quartz-tube apparatus and auxiliary equipment before the modifications indicated in the present paper were made. Prints showing details of the original apparatus are available for persons interested in constructing this equipment.

Figures 1 to 21, inclusive, indicate the results obtained on the linear thermal expansion of 30 samples of mica (muscovite, phlogopite, biotite, ripidolite, and zinnwaldite) in a direction perpendicular to the cleavage plane when subjected to a load. In most cases, the load was 30 lb/in.^{2}

The results obtained on the linear thermal expansion of 14 samples of moscovite mica in a direction perpendicular to the cleavage plane when subjected to a load of 30 lb/in.^{2} are shown in figures 1 to 5, inclusive. The samples of muscovite mica were maintained at room temperature in the thermal-expansion apparatus under the load indicated for 17 to 23 hours before they were heated. During this interval of time the shrinkage of the samples varied from 0.1 to 0.3 percent. Most of the shrinkage at the initial temperature occurred during the first few (1 to 7) hours. During the first heating, a slight shrinkage was noted for most of the samples when they were maintained for approximately 1 hour at 100°, 150°, 200°, and 250° C, respectively. Slight additional shrinkage was observed when the samples were held overnight at approximately 250° C. Most of the samples contracted in an irregular manner during the first heating from room temperature to 600° C. This contraction for the entire range was less than 2 percent for all except three samples. In one sample (1687, fig. 2), the contraction during heating between room temperature and 600° C was 4.1 percent. In two samples (1682 and 1696, figs. 1 and 4), an expansion of 0.4 percent occurred on the first heating from room temperature to 600° C. Smooth contraction curves were obtained for all samples except 1684 (fig. 1) and 1687 (fig. 2), when they were cooled from 600° C to room temperature. Most of the curves obtained on heating and cooling in the second test are close to the corresponding curves obtained on cooling in the first test. The authors are of the opinion that the shrinkages that occurred in the samples of muscovite micas at room temperature prior

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[8] When the load was 1 lb/in.^{2}, the steel rod and weights were not used.

to heating and at higher temperatures on the first heating resulted principally from the load on the samples which forced the laminas, or layers, of the micas closer together. It is probable that evolution of moisture was also a factor in the shrinkages of these micas on the first heating.

The results obtained on the linear thermal expansion of 10 samples of phlogopite mica in a direction perpendicular to the cleavage plane when subjected to a load are shown in figures 6 to 15, inclusive. From the curves it can be seen that the linear thermal expansion of some of the samples is tremendous. In fact, this expansion is many times that of any other known solid material.

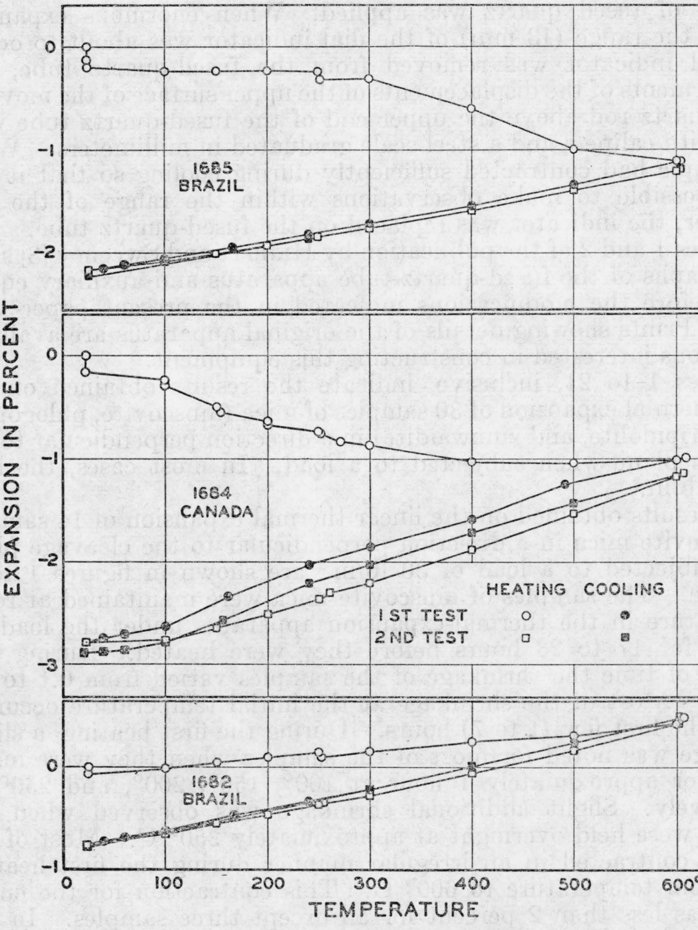

FIGURE 1.—*Linear thermal expansion of three samples of muscovite mica from Brazil and Canada in a direction perpendicular to the cleavage plane when subjected to a load of 30 lb/in.*^{2}

Five of the samples of phlogopite mica were maintained at room temperature in the thermal-expansion apparatus under a load of 30 lb/in.^{2} from 16 to 21 hours before heating. During this interval the shrinkage varied from 0.0 to 0.2 percent, slightly less than for the muscovite samples tested. Samples 1681A (fig. 7) and 1709 (fig. 10), on which no shrinkage was detected at room temperature, were made up of 1 and 10 disks, respectively, whereas the other three samples of phlogopite mica contained 55 or 80 disks.

Four samples of phlogopite mica (1681, 1681A, 1688, and 1711, figs. 6, 7, 9, and 12) expanded from 50 to 166 percent when heated from room temperature to 600° C under a load of 30 lb/in^2. The expansion curves of these four samples show that the rate of expansion is comparatively low between room temperature and 100° C but very high for a short interval[9] between 100° and 250° C. Above 250° C the rate of expansion is lower, although it remains high up to 600° C. The expansion curves of these samples of phlogopite are irreversible.

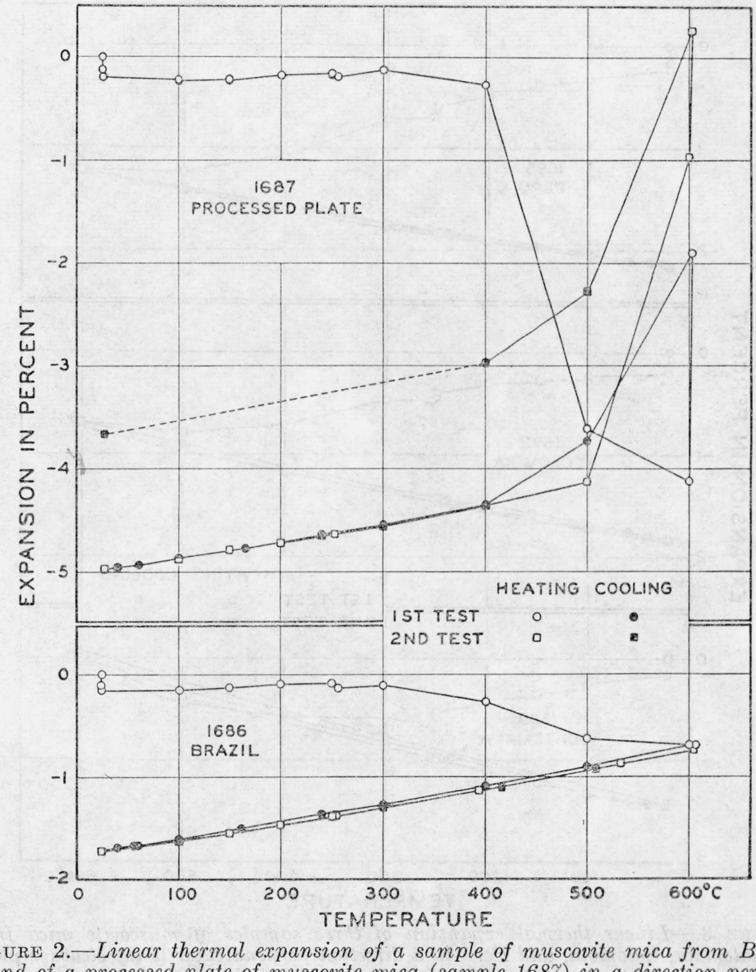

FIGURE 2.—*Linear thermal expansion of a sample of muscovite mica from Brazil and of a processed plate of muscovite mica (sample 1687) in a direction perpendicular to the cleavage plane when subjected to a load of 30 lb/in.*^{2}

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[9] During the first heating of samples 1681 and 1688, evolution of moisture was noted between 150° and 200° C.

In general, the cooling curve lies above the heating curve, and on cooling, the rapid contraction takes place at a lower temperature than does the rapid expansion on heating. When the heating and cooling cycle was repeated the curves on heating and cooling were closer together, and the total expansion was less on each successive cycle. The first run on sample 1681A is unusual in that the curve obtained on cooling from 600° to about 200° C lies below the curve obtained on heating from about 200° to 600° C. The curves on heating and cooling between 100° and 600° C for the fourth test of sample 1711 are nearly reversible. All four of these samples of phlogopite mica returned to within 2 percent of their original lengths after they were heated to 600° C and cooled to room temperature under a load of 30 lb/in^2.

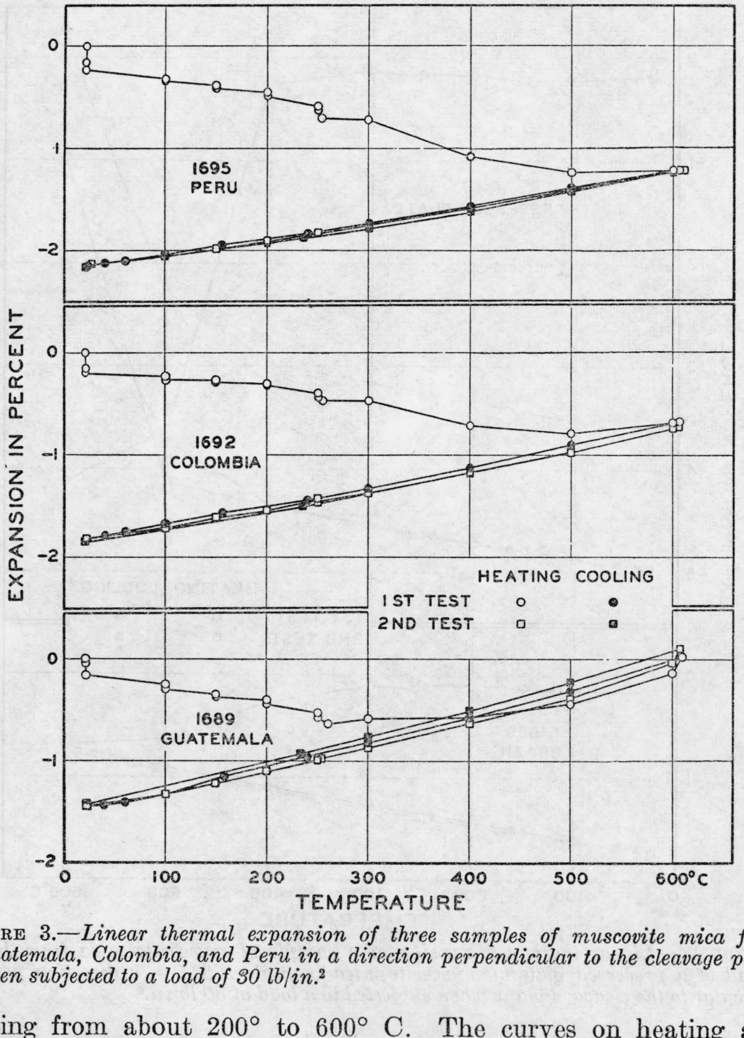

FIGURE 3.—*Linear thermal expansion of three samples of muscovite mica from Guatemala, Colombia, and Peru in a direction perpendicular to the cleavage plane when subjected to a load of 30 lb/in.*^{2}

Phlogopite samples 1681 (fig. 6) and 1681A (fig. 7) were from the same source and supposedly were of identical material, the only apparent difference in the samples being that 1681 was made up of 80 disks to form a stack 17.2 mm high, whereas 1681A consisted of 1 disk 0.28 mm thick. The great difference in the expansion seems to indicate that this phlogopite mica is not uniform.

Phlogopite sample 1711 (fig. 12) was heated three times under a load of 1 lb/in². Under this load the expansion from room temperature to 600° C was the highest of any mica measured, varying from about 300 percent for the first heating to about 240 percent for the third heating. The fourth run on this sample was made with a 30 lb/in². load, which reduced the total expansion to 50 percent.

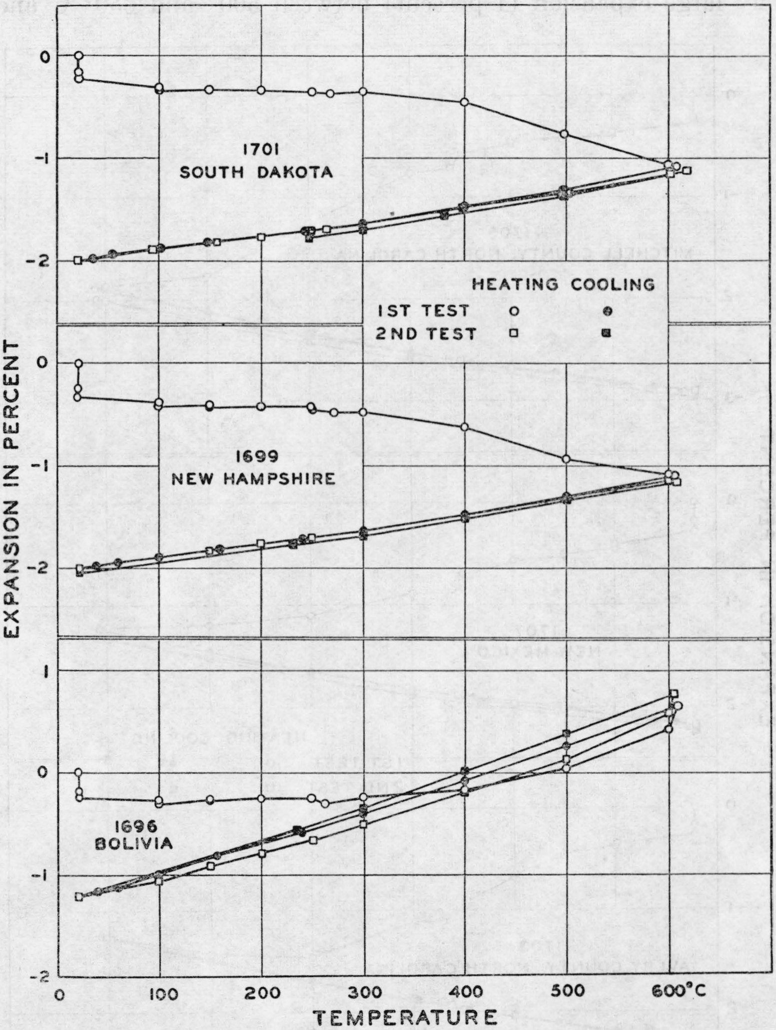

FIGURE 4.—*Linear thermal expansion of three samples of muscovite mica from Bolivia, New Hampshire, and South Dakota in a direction perpendicular to the cleavage plane when subjected to a load of 30 lb/in.²*

Phlogopite samples 1683, 1712, and 1732 (figs. 8, 13, and 15) when subjected to a load of 30 lb/in^2. expanded much less (all below 15 percent) than the four samples of phlogopite mica discussed above, but there is a similarity in the shapes of the expansion curves. The similarity is more apparent after the first heating. During the first heating, samples 1712 and 1732 indicated shrinkage up to 200° C and 150° C, respectively. The former sample also indicated comparatively large expansion (4 percent) between 500° and 650° C and a

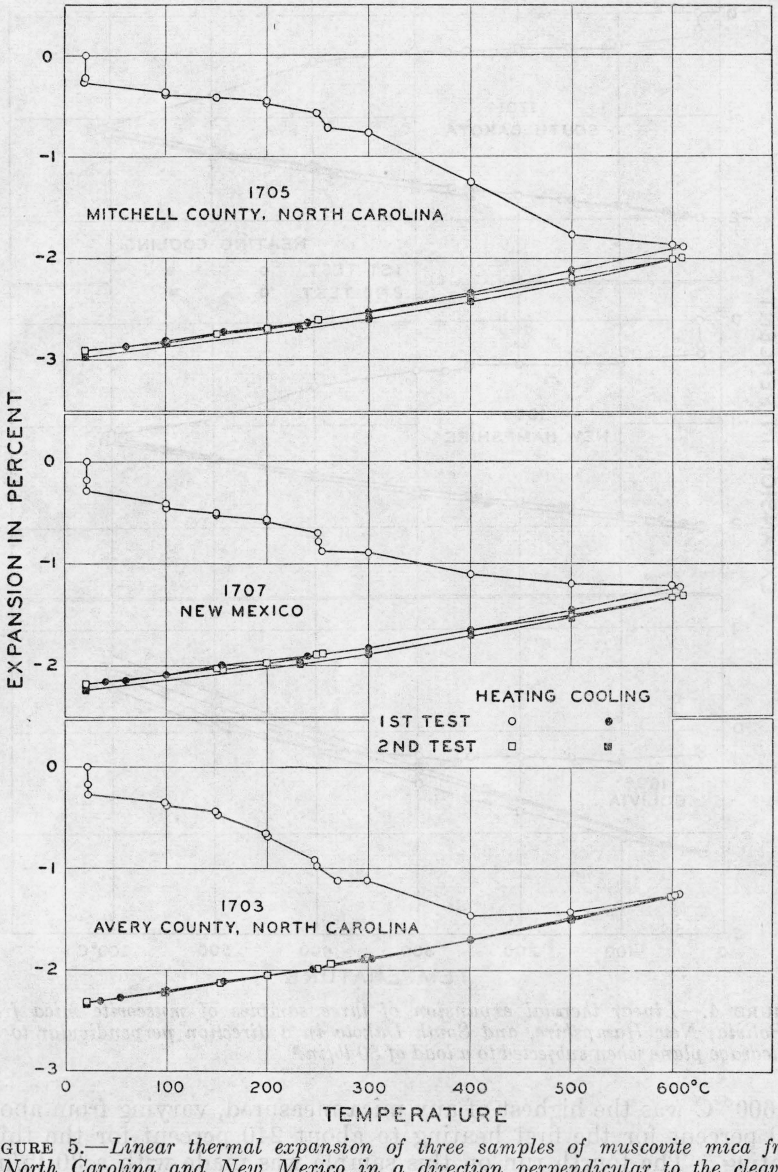

FIGURE 5.—*Linear thermal expansion of three samples of muscovite mica from North Carolina and New Mexico in a direction perpendicular to the cleavage plane when subjected to a load of 30 lb/in.^{2}*

shrinkage of 3 percent during further heating from 650° to 700° C. A duplicate of sample 1712 (fig. 14) under a load of 1 lb/in.^{2} showed practically no shrinkage when heated from room temperature to 200° C and a much larger expansion (17 percent) between 500° and 600° C. Repeated heating under a 10 lb/in^2. or 1-lb/in^2. load eliminated the high expansion or shrinkage between 500° and 700° C on samples 1712 and 1712A (figs. 13 and 14).

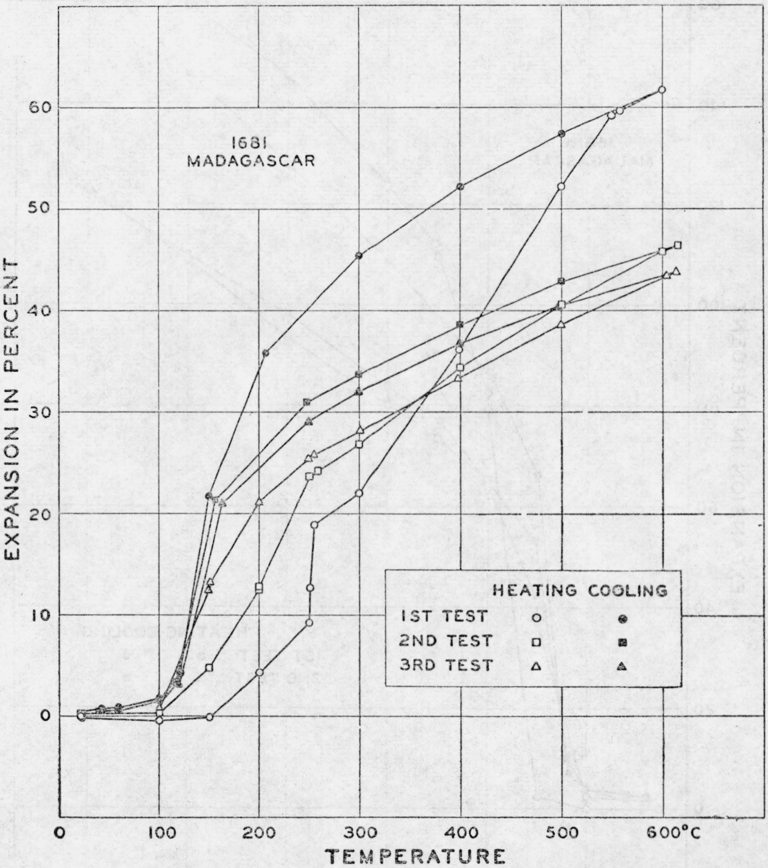

Figure 6.—*Linear thermal expansion of phlogopite mica from Madagascar (80 disks, sample 1681) in a direction perpendicular to the cleavage plane when subjected to a load of 30 lb/in.*^{2}

The expansion curves of phlogopite samples 1709 (fig. 10) and 1710 (fig. 11) do not resemble the curves for any of the other phlogopite micas. Both samples exhibited large shrinkage when heated above 500° or 600° C during the first heating under a load of 30 lb/in^2.

The expansion curves of four samples of biotite micas are shown in figures 16 to 19, inclusive. These curves are irreversible on the heating and cooling cycles.

A shrinkage of 0.4 percent occurred when the biotite sample 1713 from Newdale, N. C. (fig. 16) was maintained in the thermal-expansion

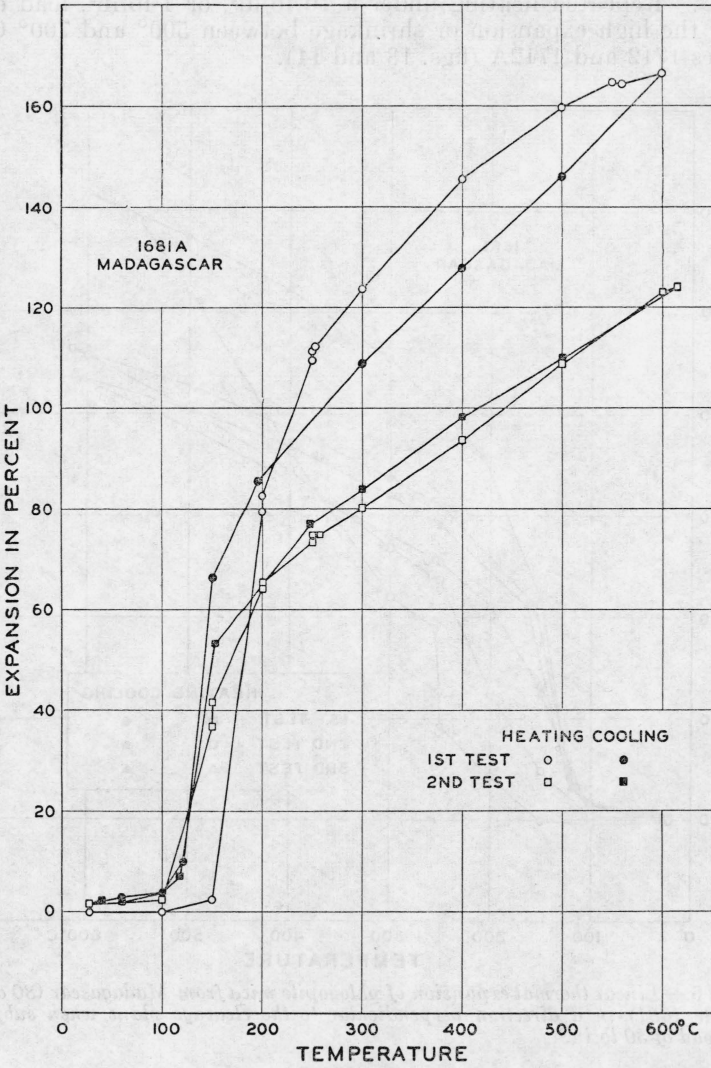

FIGURE 7.—*Linear thermal expansion of phlogopite mica from Madagascar (one disk, sample 1681A from same lot as sample 1681, fig. 6) in a direction perpendicular to the cleavage plane when subjected to a load of 30 lb/in.*^{2}

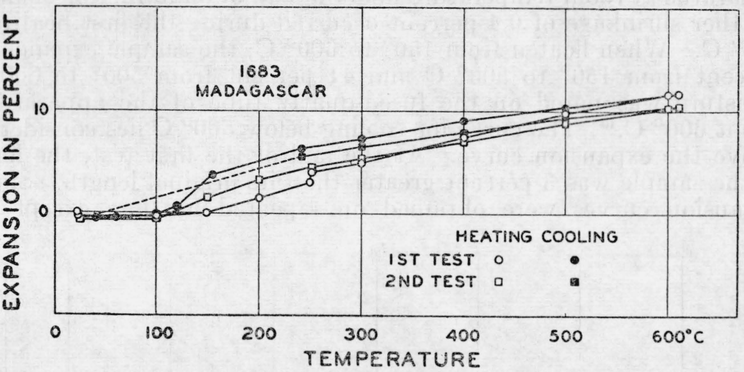

FIGURE 8.—*Linear thermal expansion of a sample of phlogopite mica from Madagascar in a direction perpendicular to the cleavage plane when subjected to a load of 30 lb/in.*^{2}

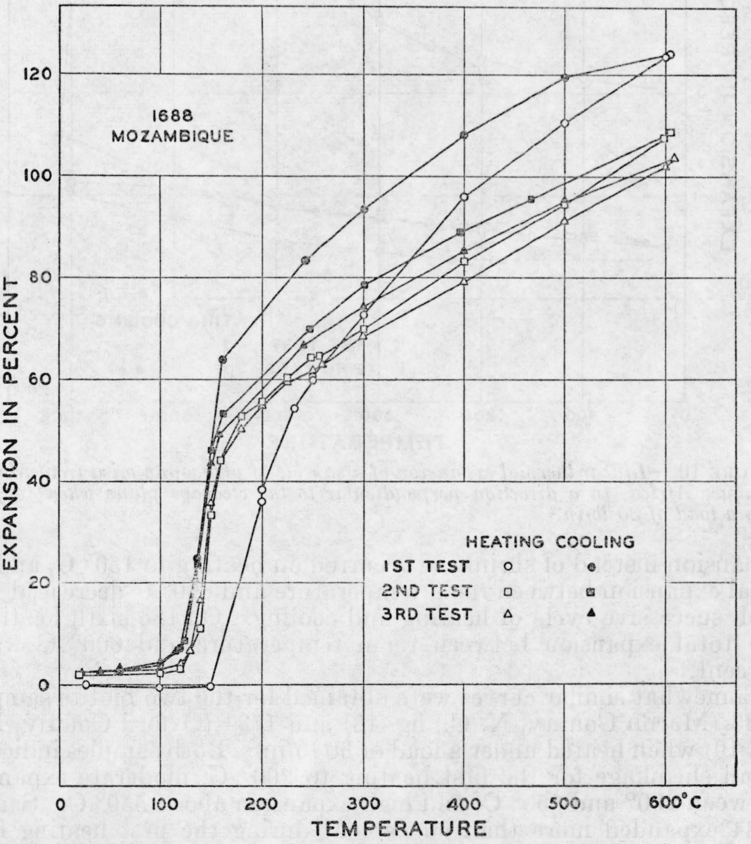

FIGURE 9.—*Linear thermal expansion of a sample of phlogopite mica from Mozambique, Africa, in a direction perpendicular to the cleavage plane when subjected to a load of 30 lb/in.*^{2}

apparatus at room temperature under a load of 30 lb/in.^{2} for 22 hours. Further shrinkage of 0.4 percent occurred during the first heating to 150° C. When heated from 150° to 600° C, the sample expanded 11 percent from 150° to 500° C and 41 percent from 500° to 600° C. Moisture was noted on the fused-quartz tube of the apparatus at about 600° C.[10] The curve for cooling below 500° C lies considerably above the expansion curve. At the end of the first test, the length of the sample was 3 percent greater than its original length. Similar expansion curves were obtained on repeated heating, except that expansion instead of shrinkage occurred on heating to 150° C, and the total expansion between room temperature and 600° C decreased with each successive cycle of heating and cooling. On the sixth heating,[11] the total expansion between room temperature and 600° C was 6 percent.

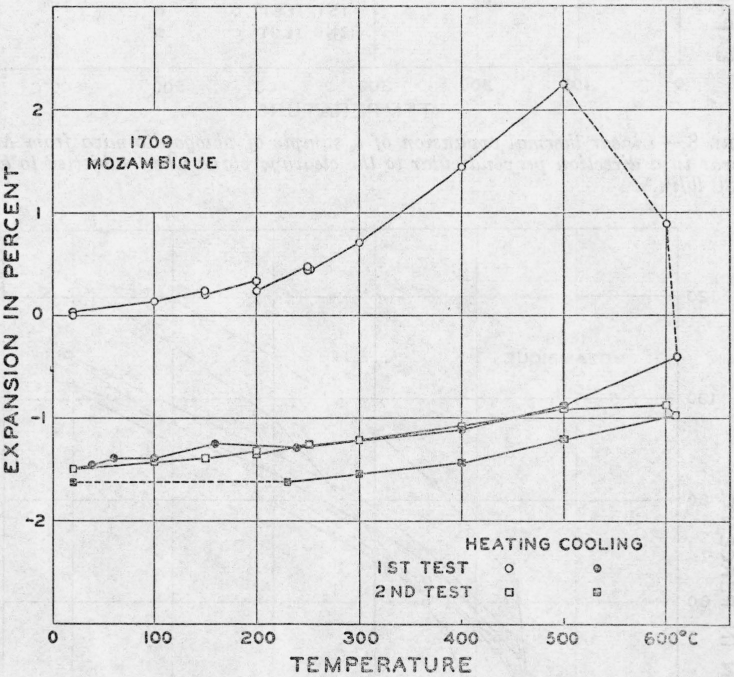

FIGURE 10.—*Linear thermal expansion of a sample of phlogopite mica from Mozambique, Africa, in a direction perpendicular to the cleavage plane when subjected to a load of 30 lb/in.^{2}*

Somewhat similar curves were obtained for the two biotite samples, 1733 (Macon County, N. C., fig. 18) and 1734 (Oxford County, Me., fig. 19) when heated under a load of 30 lb/in.^{2} Both samples indicated small shrinkage for the first heating to 200° C, moderate expansion between 200° and 550° C, and high expansion above 550° C. Sample 1734 expanded more than 60 percent during the first heating from

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[10] Moisture was also noted at higher temperatures during the sixth heating.

[11] After the sixth test, the color of some of the biotite mica disks of sample 1713 was found to have changed from black to a golden copper color.

550° to about 665° C. A shrinkage amounting to more than 20 percent occurred during further heating of this sample from 665° to 700° C. The length of sample 1734 after the first heating and cooling cycle was 6 percent greater than the original length. The maximum expansion of each sample during the second heating was less than that observed in the first heating.

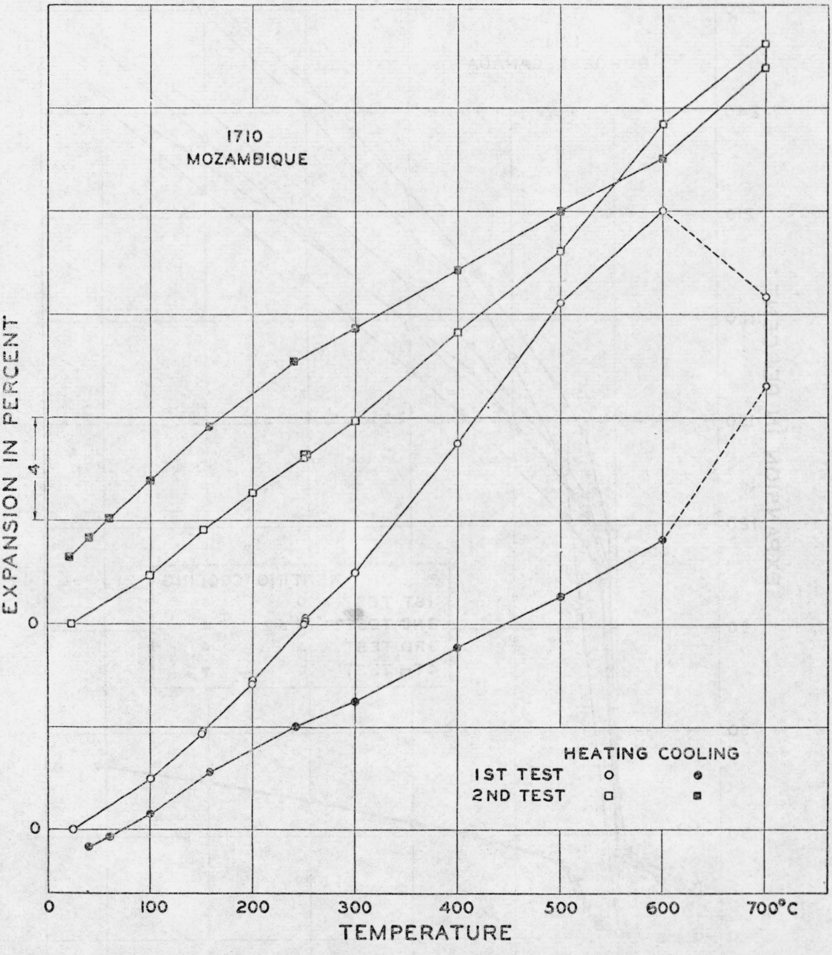

FIGURE 11.—*Linear thermal expansion of a sample of phlogopite mica from Mozambique, Africa, in a direction perpendicular to the cleavage plane when subjected to a load of 30 lb/in.² during the first test and 1 lb/in.² during the second test.*

The tests were plotted from different origins.

The expansion curve of biotite sample 1730 from Ontario, Canada (fig. 17) is different from the curves for the three biotite samples of the United States. A shrinkage of 0.4 percent occurred during the first heating to 250° C with a load of 30 lb/in.², and then an expansion of only 0.8 percent when heated from 250° to 700° C. When cooled from 700° C to room temperature the sample contracted 1.7 percent.

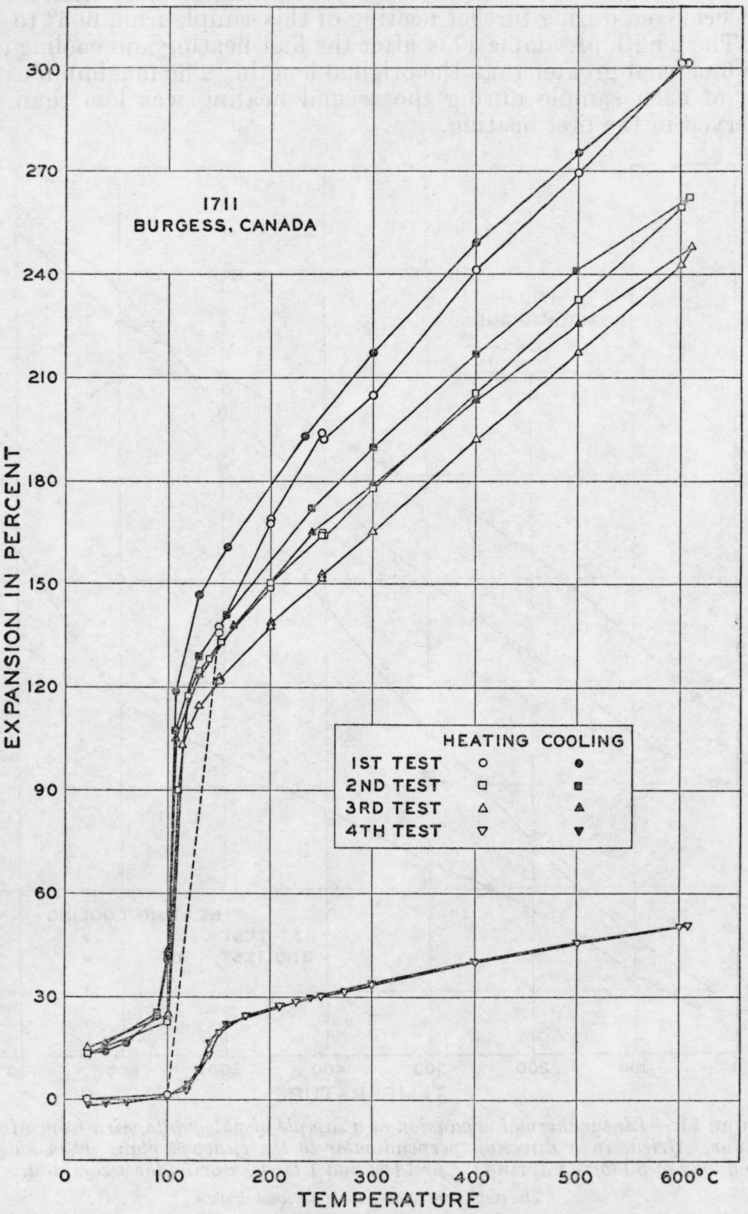

FIGURE 12.—*Linear thermal expansion of a sample of phlogopite mica from Burgess, Canada, in a direction perpendicular to the cleavage plane when subjected to a load of 1 lb/in.² during the first, second, and third tests and 30 lb/in.² during the fourth test.*

The fourth test was plotted from the same origin as the first test.

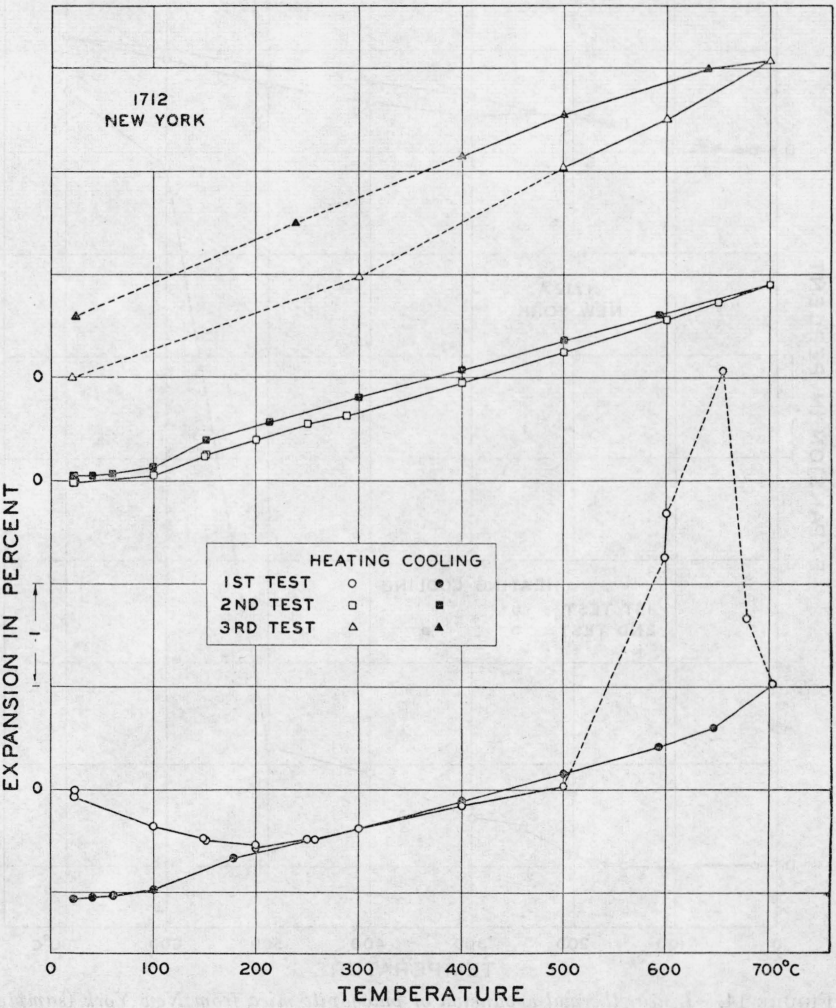

FIGURE 13.—*Linear thermal expansion of phlogopite mica from New York (sample 1712) in a direction perpendicular to the cleavage plane when subjected to loads of 30, 10, and 1 lb/in². during the first, second, and third tests, respectively.*

The tests were plotted from different origins.

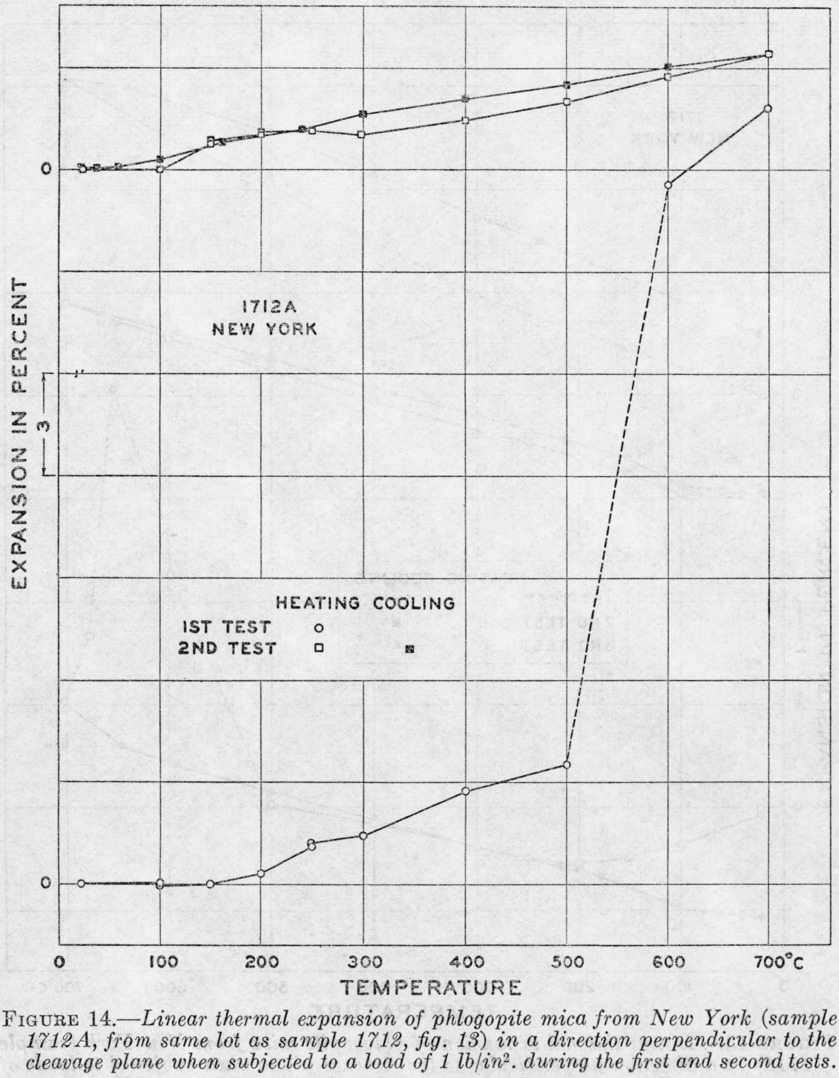

FIGURE 14.—*Linear thermal expansion of phlogopite mica from New York (sample 1712A, from same lot as sample 1712, fig. 13) in a direction perpendicular to the cleavage plane when subjected to a load of 1 lb/in². during the first and second tests.*

The tests were plotted from different origins.

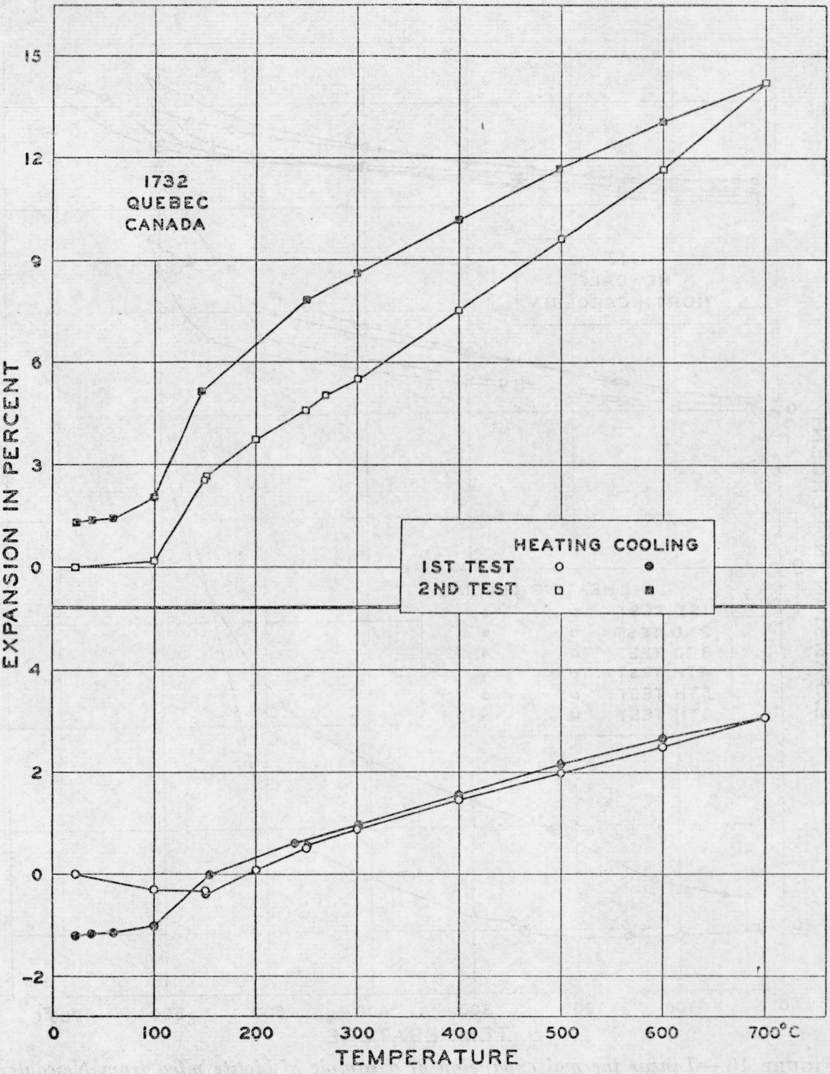

FIGURE 15.—*Linear thermal expansion of phlogopite mica from Quebec, Canada, in a direction perpendicular to the cleavage plane when subjected to loads of 30 and 1 lb/in². during the first and second tests, respectively.*

The tests were plotted from different origins.

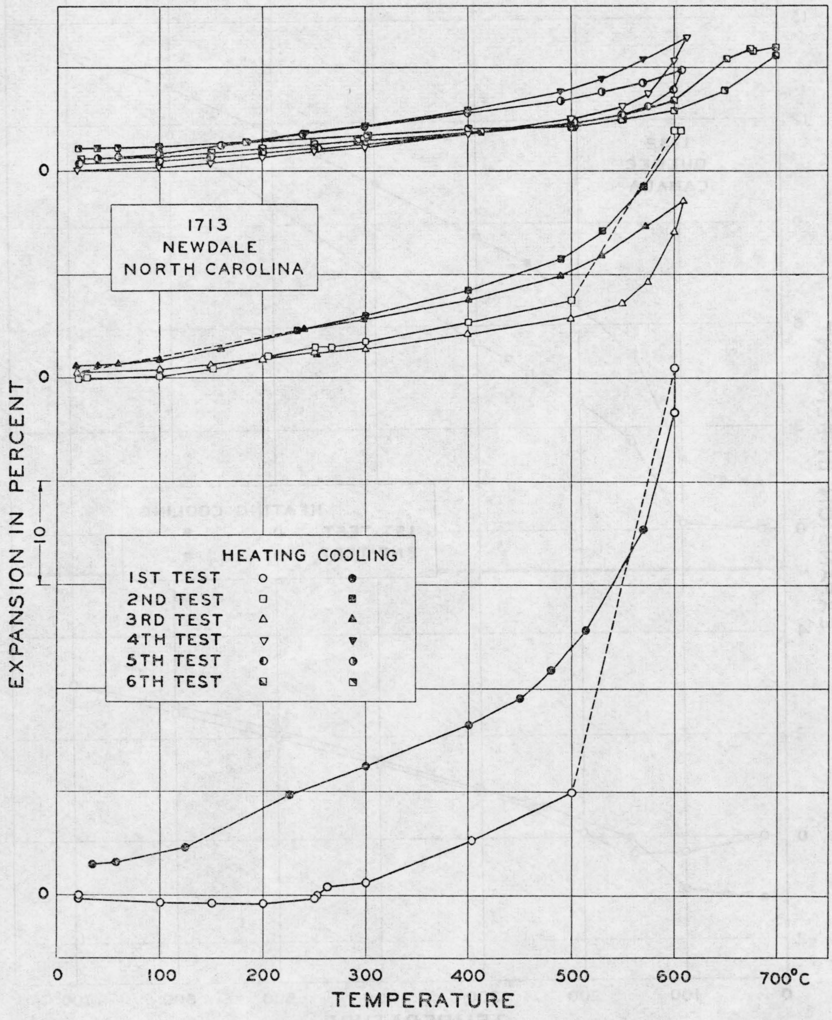

FIGURE 16.—*Linear thermal expansion of a sample of biotite mica from Newdale, North Carolina, in a direction perpendicular to the cleavage plane when subjected to a load of 30 lb/in².*

Tests 1, 2, and 4 were plotted from different origins.

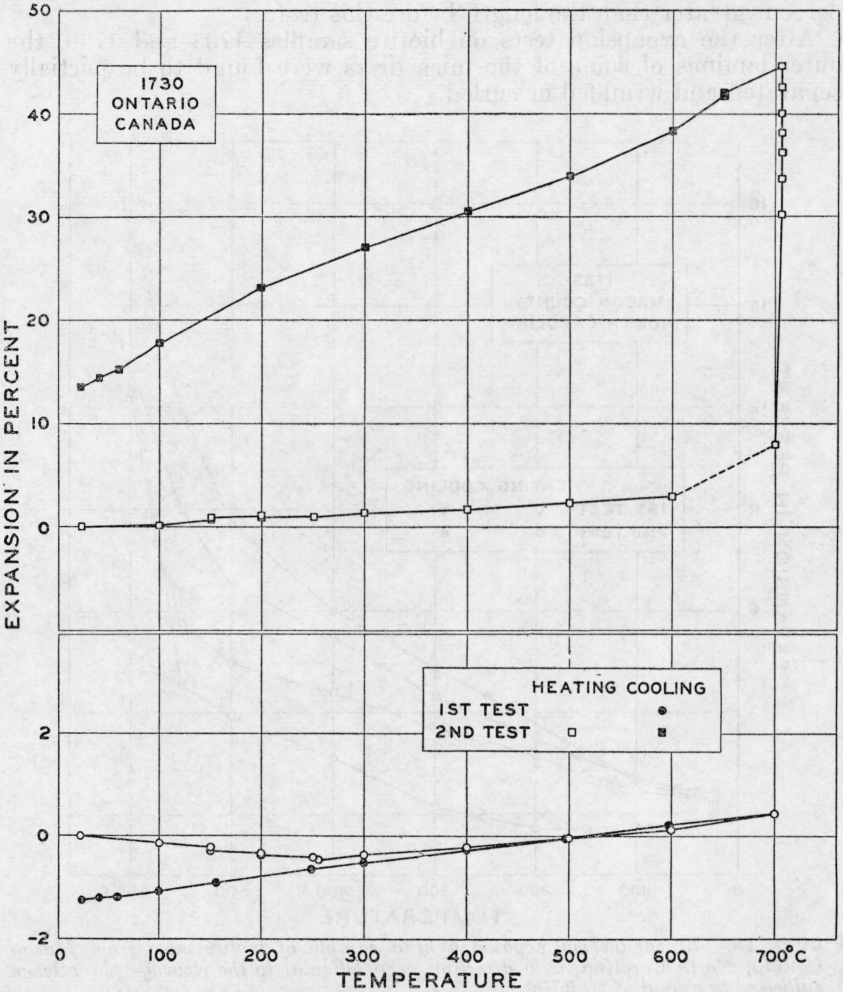

FIGURE 17.—*Linear thermal expansion of a sample of biotite mica from Ontario, Canada, in a direction perpendicular to the cleavage plane when subjected to loads of 30 and 1 lb/in². during the first and second tests, respectively.*

The tests were plotted from different origins.

During the second heating and cooling cycle, the sample was subjected to a load of only 1 lb/in.^{2} The expansion was 3 percent from 20° to 600° C. At about 700° C, the sample expanded considerably (about 36 percent). The curve obtained during cooling lies considerably above the expansion curve. The length after the second test was 14 percent greater than the length before this test.

After the expansion tests on biotite samples 1733 and 1730, the outer laminas of some of the mica disks were found to be partially separated and wrinkled or curled.

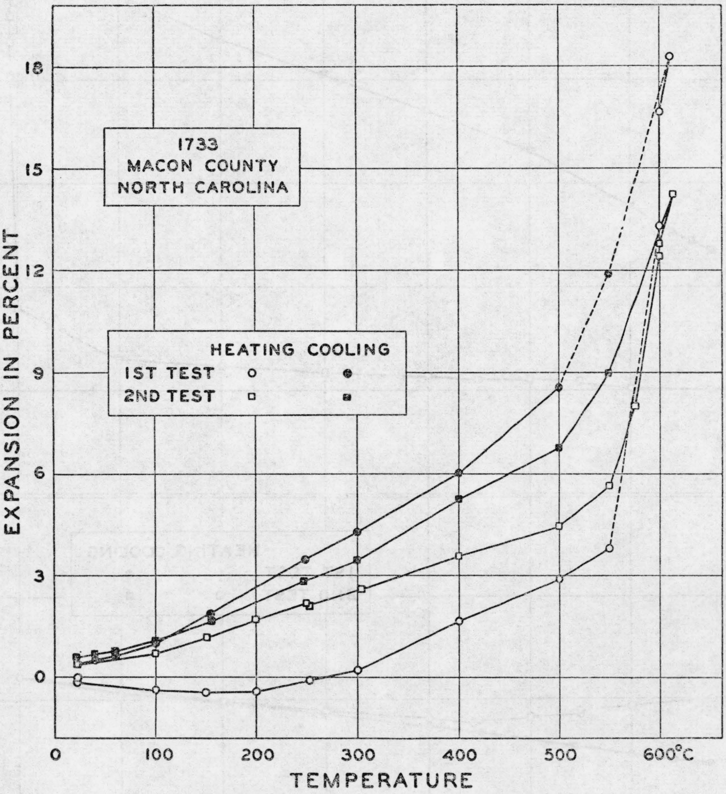

FIGURE 18.—*Linear thermal expansion of a sample of biotite mica from Macon County, North Carolina, in a direction perpendicular to the cleavage plane when subjected to a load of 30 lb/in^2.*

Figure 20 shows the results obtained on a sample of ripidolite mica from Pennsylvania that was subjected to a load of 30 lb/in.^{2} during two cycles of heating and cooling. The sample indicated shrinkage during the first heating from 20° to 700° C. At 100°, 150°, 200°, and 250° C, shrinkage was noted when the sample was held at constant temperature for one-half hour or longer. The large shrinkage indicated on the curve at about 250° C occurred during a period of 17 hours. The shrinkage of the sample when heated from 20° to 700° C was 5 percent. When cooled from 700° to 20° C the sample contracted regularly. The total contraction for this temperature range was 0.8 percent. During the second heating and cooling cycle, the sample expanded and contracted, respectively, in a regular manner. The expansion for the second heating from 20° to 700° C was only 0.5 percent.

The results obtained on a sample of zinnwaldite mica subjected to a load of 30 lb/in.^{2} during two cycles of heating and cooling are shown in figure 21. Shrinkage was noted when the sample was heated to 250° C. A slight shrinkage occurred at 250° C when the sample was kept at constant temperature for 17 hours. When heated from 250° to 700° C, the sample expanded 1.1 percent. An increase in the rate of expansion was noted between 600° and 700° C. The contraction during cooling from 700° to 20° C was 1.8 percent. Expansion occurred throughout the range during the second heating from 20° to 700° C. An increase in the rate of expansion also occurred between 600° and 700° C. An expansion of 0.1 percent was noted when the sample was maintained at 700° C for 10 minutes. The latter observation, and the fact that the rate of expansion increased between 600° and 700° C, indicate the probable existence of a transition in the neighborhood of 700° C[12].

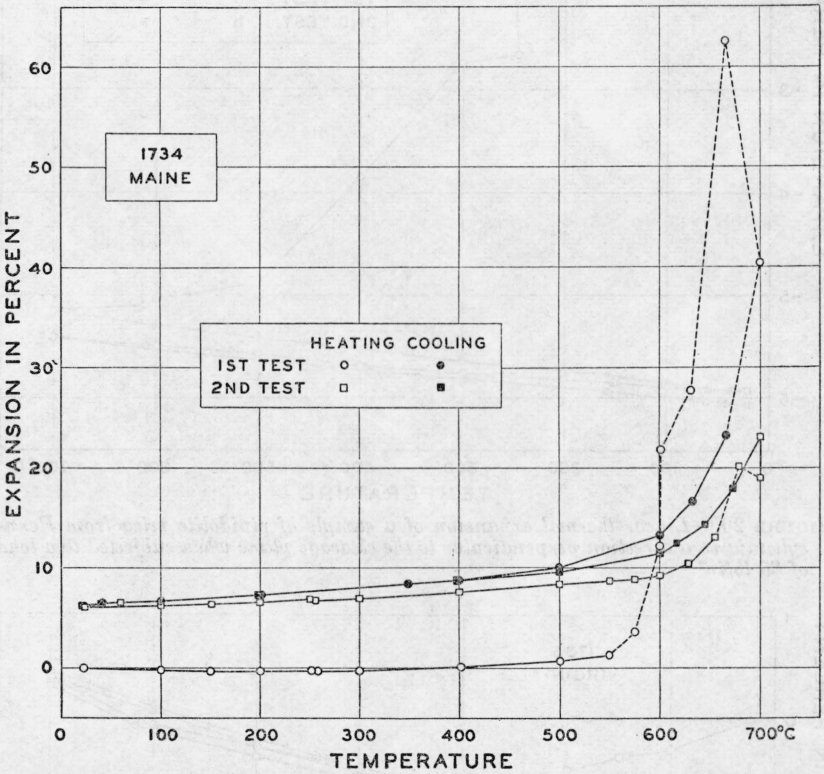

FIGURE 19.—*Linear thermal expansion of a sample of biotite mica from Maine in a direction perpendicular to the cleavage plane when subjected to a load of 30 lb/in^2.*

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[12] Expansion measurements were terminated at 700° C in order to avoid the danger of the mica disks fusing to the fused-quartz parts of the apparatus.

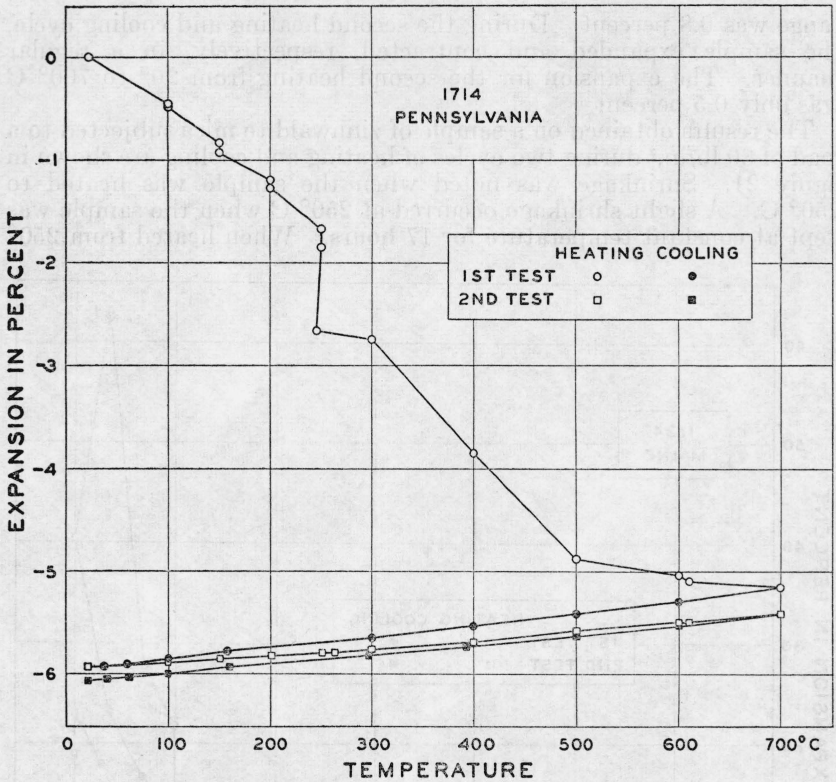

FIGURE 20.—*Linear thermal expansion of a sample of ripidolite mica from Pennsylvania in a direction perpendicular to the cleavage plane when subjected to a load of 30 lb/in².*

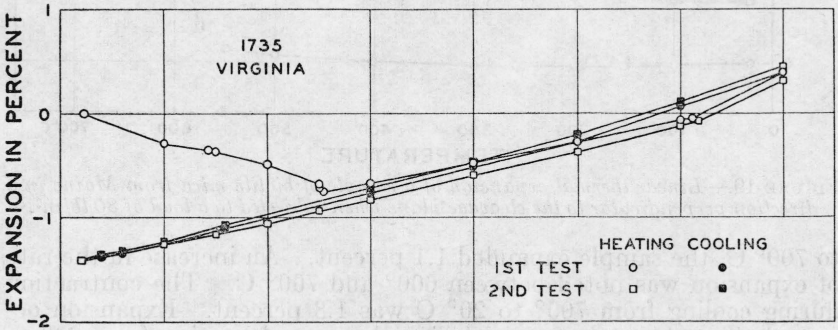

FIGURE 21.—*Linear thermal expansion of a sample of zinnwaldite mica from Virginia in a direction perpendicular to the cleavage plane when subjected to a load of 30 lb/in².*

Table 2 gives average coefficients of expansion and coefficients of contraction of the samples of mica that were computed from the data plotted in figures 1 to 21, inclusive. The table also shows the changes in length of the samples after they were heated and cooled. Most of the coefficients of the samples of phlogopite and biotite micas are extraordinarily high. The maximum coefficients of expansion of these two types of mica are 0.0552 and 0.00411 per degree centigrade, respectively. The coefficients of expansion of the samples of muscovite, ripidolite, and zinnwaldite micas are comparatively low. The maximum coefficient of expansion of these three types of mica is 0.000036 per degree centigrade. In most cases when the load perpendicular to the cleavage plane of samples of phlogopite and biotite micas was decreased, the coefficients of expansion and contraction increased. Conversely, by applying heavier loads to these micas, it is possible to reduce the expansion.

TABLE 2.—*Coefficients of expansion*[a] *of mica in a direction perpendicular to cleavage plane*

Sample No.	Source	Test	Load perpendicular to cleavage plane of sample	Average coefficients of expansion per degree centigrade		Average coefficients of contraction per degree centigrade		Change in length after heating and cooling[b]
				20° to 300° C	300° to 600° C	600° to 300° C	300° to 20° C	
	A. MUSCOVITE MICA							
			lb/in.²	$\times 10^{-6}$	$\times 10^{-6}$	$\times 10^{-6}$	$\times 10^{-6}$	*Percent*
1682	Brazil	1	30	8	9	23	20	−0.8
		2	30	17	24	23	20	−.0
1684	Canada	1	30	(c)	(c)	28	33	−2.8
		2	30	21	34	29	31	−.1
1685	Brazil	1	30	(c)	(c)	20	16	−2.2
		2	30	15	18	19	16	−.0
1686	do	1	30	(c)	(c)	19	16	−1.7
		2	30	15	18	(d)	(d)	−.0
1687	Plate mica (processed sheet supplied by New England Mica Co.).	1	30	(c)	(c)	(e)	16	−5.0
		2	30	15	(f)	(g)	------	+1.3
1689	Los Volcansillos Mine, Guatemala	1	30	(c)	15	27	23	−1.4
		2	30	21	28	28	24	+.0
1692	Durania Section, Colombia	1	30	(c)	(h)	21	18	−1.8
		2	30	16	21	22	17	−.0
1695	Near Camana, Ferreyros, Peru	1	30	(c)	(c)	17	15	−2.1
		2	30	14	17	18	13	−.0
1696	Amber Mine, Concepcion Area, Depto. Santa Cruz, Bolivia.	1	30	(c)	22	34	29	−1.2
		2	30	25	36	37	31	+.0
1699	New Hampshire	1	30	(c)	(c)	18	13	−2.0
		2	30	13	16	17	13	−.0
1701	Old Mike Mine, 4½ miles north of Custer, S. Dak.	1	30	(c)	(c)	18	13	−2.0
		2	30	13	16	18	------	------
1703	Mill Race Mine, Avery County, N. C.	1	30	(c)	(i)	21	15	−2.3
		2	30	16	20	21	17	−.0
1705	Sink Hole Mine, near Bandana, Mitchell County, N. C.	1	30	(c)	(c)	21	13	−2.9
		2	30	13	18	20	13	−.1
1707	Petaca Area, N. Mex	1	30	(c)	(c)	19	13	−2.2
		2	30	13	17	18	13	−.1

See footnotes at end of table.

TABLE 2.—*Coefficients of expansion* [a] *of mica in a direction perpendicular to cleavage plane*—Continued

Sample No.	Source	Test	Load perpendicular to cleavage plane of sample	Temperature range	Average coefficient of expansion per degree centigrade	Average coefficient of contraction per degree centigrade	Change in length after heating and cooling [b]
			B. PHLOGOPITE MICA				
			lb/in.²	° *C*			*Percent*
1681	Madagascar	1	30	150 to 255	0.00182		+0.5
				300 to 550	.00149		
				600 to 300		0.000546	
				207 to 122		.00372	
				100 to 24		.000164	
		2	30	22 to 100	.000026		+.2
				100 to 250	.00153		
				300 to 600	.000632		
				615 to 300		.000404	
				300 to 157		.000864	
				157 to 117		.00462	
				100 to 22		.000138	
		3	30	22 to 100	.000051		−.0
				100 to 200	.00202		
				200 to 302	.000688		
				302 to 604	.000504		
				614 to 299		.000375	
				299 to 163		.000810	
				163 to 120		.00414	
				100 to 22		.000121	
1681A	Madagascar	1	30	27 to 100	.000001		+1.8
				150 to 200	.0157		
				200 to 300	.00426		
				300 to 500	.00180		
				600 to 300		.00192	
				300 to 195		.00223	
				150 to 121		.0198	
				100 to 26		.000289	
		2	30	26 to 100	.000097		.0
				100 to 150	.00729		
				200 to 300	.00152		
				300 to 600	.00143		
				614 to 300		.00128	
				300 to 153		.00207	
				153 to 117		.0130	
				100 to 29		.000299	
1683	Madagascar	1	30	100 to 200	.000178		−.7
				300 to 600	.000223		
				611 to 300		.000141	
				300 to 155		.000248	
				155 to 120		.000870	
				100 to 25		.000067	
		2	30	25 to 100	.000020		−.0
				100 to 300	.000293		
				300 to 600	.000166		
				611 to 300		.000127	
1688	Mozambique, Africa	1	30	150 to 200	.00752		+1.9
				200 to 300	.00343		
				300 to 600	.00170		
				605 to 300		.00100	
				162 to 120		.0136	
				100 to 25		.000261	
		2	30	22 to 100	.000059		+.5
				120 to 180	.00834		
				200 to 300	.00143		
				300 to 598	.00126		
				606 to 301		.000995	
				301 to 162		.00182	
				162 to 125		.0120	
				100 to 23		.000222	
		3	30	23 to 100	.000092		+.3
				120 to 160	.00995		
				200 to 300	.00135		
				300 to 600	.00112		
				609 to 300		.000941	
				300 to 159		.00178	
				159 to 125		.0119	
				100 to 21		.000224	

See footnotes at end of table.

TABLE 2.—*Coefficients of expansion* [a] *of mica in a direction perpendicular to cleavage plane*—Continued

Sample No.	Source	Test	Load perpendicular to cleavage plane of sample	Temperature range	Average coefficient of expansion per degree centigrade	Average coefficient of contraction per degree centigrade	Change in length after heating and cooling [b]
B. PHLOGOPITE MICA—Continued							
			lb/in.²	° *C*			*Percent*
1709	Mozambique, Africa	1	30	19 to 99	0.000013	----------	−1.5
				300 to 500	.000077	----------	
				611 to 300	----------	.000026	
		2	30	20 to 300	.000010	----------	−.1
				300 to 600	.000012	----------	
				609 to 300	----------	.000019	
				300 to 20	----------	.000003	
1710	Mozambique, Africa	1	30	24 to 300	.000362	----------	−1.1
				300 to 600	.000468	----------	
				600 to 300	----------	.000210	
				300 to 40	----------	.000217	
		2	1	22 to 300	.000283	----------	+2.6
				300 to 600	.000384	----------	
				600 to 300	----------	.000220	
				300 to 21	----------	.000318	
1711	Burgess, Canada	1	1	22 to 100	.000167	----------	+13.3
				100 to 150	.0272	----------	
				300 to 600	.00322	----------	
				607 to 300	----------	.00275	
				300 to 158	----------	.00397	
				107 to 90	----------	.0548	
				90 to 22	----------	.00165	
		2	1	22 to 100	.00121	----------	+1.3
				100 to 120	.0478	----------	
				200 to 300	.00276	----------	
				300 to 600	.00273	----------	
				608 to 300	----------	.00237	
				300 to 157	----------	.00341	
				107 to 90	----------	.0476	
				90 to 22	----------	.00154	
		3	1	22 to 100	.00127	----------	+.2
				100 to 114	.0552	----------	
				300 to 600	.00259	----------	
				610 to 300	----------	.00222	
				300 to 164	----------	.00300	
				108 to 90	----------	.0457	
				90 to 22	----------	.00157	
		4	30	22 to 100	.000179	----------	−1.6
				119 to 151	.00522	----------	
				210 to 300	.000666	----------	
				300 to 600	.000570	----------	
				608 to 300	----------	.000543	
				300 to 157	----------	.000830	
				140 to 120	----------	.00614	
				100 to 22	----------	.000337	
1712	St. Lawrence County, N. Y	1	30	200 to 300	.000018	----------	−1.1
				300 to 500	.000020	----------	
				593 to 300	----------	.000027	
				100 to 22	----------	.000012	
		2	10	21 to 288	.000024	----------	+.1
				288 to 600	.000030	----------	
				593 to 300	----------	.000027	
				300 to 21	----------	.000027	
		3	1	21 to 300	.000035	----------	+.6
				300 to 600	.000051	----------	
				500 to 23	----------	.000041	
1712A	St. Lawrence County, N. Y	1	1	300 to 500	.000104	----------	----------
		2	1	23 to 100	−.000002	----------	+.1
				298 to 600	+.000057	----------	
				600 to 300	----------	.000046	
				300 to 23	----------	.000057	
1732	St. Lawrence Mica Co. Mine, Petit Pré., Quebec, Canada.	1	30	150 to 300	.000083	----------	−1.2
				300 to 600	.000054	----------	
				600 to 301	----------	.000057	
				100 to 22	----------	.000025	
		2	1	22 to 100	.000023	----------	+1.3
				200 to 301	.000176	----------	
				301 to 601	.000206	----------	
				600 to 300	----------	.000149	
				100 to 23	----------	.000098	

See footnotes at end of table.

TABLE 2.—*Coefficients of expansion* [a] *of mica in a direction perpendicular to cleavage plane*—Continued

Sample No.	Source	Test	Load perpendicular to cleavage plane of sample	Temperature range	Average coefficient of expansion per degree centigrade	Average coefficient of contraction per degree centigrade	Change in length after heating and cooling [b]
C. BIOTITE MICA							
			lb/in.²	*°C*			*Percent*
1713	E. Blake Mine, Newdale, N. C.	1	30	100 to 200	0.000000	----------	+3.0
				300 to 500	.000434	----------	
				500 to 600	.00411	----------	
				600 to 450	----------	0.00184	
				450 to 300	----------	.000441	
				300 to 34	----------	.000357	
		2	30	21 to 300	.000130	----------	+.6
				300 to 500	.000198	----------	
				500 to 600	.00164	----------	
				608 to 490	----------	.00105	
				490 to 300	----------	.000293	
				300 to 20	----------	.000195	
		3	30	20 to 300	.000081	----------	+.6
				300 to 500	.000153	----------	
				500 to 600	.000830	----------	
				609 to 490	----------	.000609	
				490 to 299	----------	.000225	
				299 to 19	----------	.000161	
		4	30	19 to 300	.000079	----------	+.7
				300 to 500	.000136	----------	
				500 to 600	.000567	----------	
				613 to 490	----------	.000427	
				490 to 300	----------	.000171	
				300 to 22	----------	.000131	
		5	30	22 to 300	.000064	----------	+.5
				300 to 504	.000103	----------	
				504 to 600	.000337	----------	
				608 to 490	----------	.000251	
				490 to 300	----------	.000131	
				300 to 21	----------	.000111	
		6	30	22 to 293	.000063	----------	+1.0
				293 to 500	.000086	----------	
				500 to 600	.000220	----------	
				600 to 700	.000511	----------	
				700 to 600	----------	.000534	
				600 to 500	----------	.000135	
				500 to 300	----------	.000055	
				300 to 21	----------	.000044	
1730	Faraday Twp., Hastings Co., Ontario, Canada.	1	30	300 to 600	.000016	----------	−1.3
				600 to 700	.000032	----------	
				700 to 300	----------	.000024	
				300 to 23	----------	.000027	
		2	1	22 to 300	.000049	----------	+13.5
				300 to 600	.000055	----------	
				600 to 700	.000496	----------	
				706 to 301	----------	.000435	
				301 to 22	----------	.000485	
1733	Ledford Cove Mica Mine, Macon County, N. C.	1	30	200 to 300	.000063	----------	+.4
				300 to 550	.000144	----------	
				550 to 610	.00244	----------	
				610 to 550	----------	.00107	
				550 to 500	----------	.000669	
				500 to 300	----------	.000215	
				300 to 22	----------	.000141	
		2	30	22 to 304	.000079	----------	+.2
				304 to 500	.000095	----------	
				500 to 550	.000238	----------	
				550 to 613	.00136	----------	
				613 to 550	----------	.000830	
				550 to 500	----------	.000446	
				500 to 300	----------	.000166	
				300 to 22	----------	.000102	
1734	Saunders Quarry, Oxford County, Maine.	1	30	22 to 300	−.000008	----------	+6.1
				300 to 550	+.000061	----------	
				550 to 601	.00403	----------	
				600 to 348	----------	.000190	
				348 to 23	----------	.000071	
		2	30	23 to 300	.000030	----------	+.2
				300 to 600	.000078	----------	
				600 to 399	----------	.000133	
				399 to 198	----------	.000065	
				198 to 20	----------	.000062	

See footnotes at end of table.

TABLE 2.—*Coefficients of expansion*[a] *of mica in a direction perpendicular to cleavage plane*—Continued

Sample No.	Source	Test	Load perpendicular to cleavage plane of sample	Temperature range	Average coefficient of expansion per degree centigrade	Average coefficient of contraction per degree centigrade	Change in length after heating and cooling [b]
	D. RIPIDOLITE MICA						
			lb/in.²	*° C*	$\times10^{-6}$	$\times10^{-6}$	*Percent*
1714	Three miles south of Westchester, Pa.	1	30	700 to 300		12	−5.9
				300 to 21		11	
		2	30	21 to 300	6		−.1
				300 to 700	9		
				700 to 296		10	
				296 to 22		9	
	E. ZINNWALDITE MICA						
					$\times10^{-6}$	$\times10^{-6}$	*Percent*
1735	Morefield Mine, Amelia, Va.	1	30	300 to 600	20		−1.4
				700 to 300		29	
				300 to 21		23	
		2	30	21 to 300	21		+.0
				300 to 600	23		
				700 to 300		29	
				300 to 22		24	

[a] In parts A, D, and E of the table, each coefficient of expansion is expressed in the usual way as a whole number multiplied by 10^{-6}. However, in parts B and C, where most of the coefficients are extremely large, they are expressed as decimal fractions. The first coefficient in part B is 0.00182. If written as in part A, it is 1820×10^{-6}.

[b] Determined from the heating and cooling curves in figures 1 to 21. The plus sign indicates an increase in length and the minus sign a decrease in length.

[c] Sample indicated shrinkage when heated during this temperature range.

[d] Average coefficient of contraction from 600° to 20° C, 18×10^{-6} per degree centigrade.

[e] Average coefficients of contraction from 600° to 500° C and from 500° to 300° C, 177×10^{-6} and 41×10^{-6} per degree centigrade, respectively.

[f] Average coefficients of expansion from 300° to 500° C and from 500° to 600° C, 22×10^{-6} and 316×10^{-6} per degree centigrade, respectively.

[g] Average coefficient of contraction from 600° to 500° C, 248×10^{-6} per degree centigrade.

[h] Sample indicated shrinkage when heated to 500° C; coefficient of expansion from 500° to 600° C, 10×10^{-6} per degree centigrade.

[i] Sample indicated shrinkage when heated to 400° C; coefficient of expansion from 400° to 600° C, $10\times10^{-}$ per degree centigrade.

Megaw [10] and Wood [11] reported data on the linear thermal expansion of mica in a direction perpendicular to the cleavage plane. Their results and those given by other observers on the linear thermal expansion of mica in a direction parallel to the cleavage plane are given in table 3. This table also includes data on the linear thermal expansion of pulverized mica, rods prepared from pulverized mica, and of micanite.

From his X-ray examination of the phlogopite mica specimens used in his determinations of their thermal expansion, Wood [11] found that, in contrast with the usual effect of annealing, the heat treatment of mica may bring about a pronounced mosaic formation without apparent change in external appearance; that this structure is not necessarily stable but may revert appreciably to the initial state during cooling; and that these particular thermal and structural characteristics are interdependent. He concluded that measurements of thermal expansion of the lattice constant perpendicular to the cleavage plane indicate that the transition to the mosaic structure does not,

TABLE 3.—*Coefficients of linear thermal expansion of mica by previous observers*

Observer	Date	Material	Direction in which linear expansion was determined	Temperature range	Average coefficient of expansion per degree centigrade	Remarks
				° *C*	×10⁻⁶	
Braesco [12, 13]	1913 & 1920	Rod of pulverized mica agglomerated with sodium silicate and water.	Along axis of rod			Observed regular expansion up to about 900°C. No quantitative data were reported.
Souder and Sweeney [14]	1925	Micanite (layers of mica stuck together with shellac. About 5 percent of the weight of the material was shellac.)	Parallel to cleavage plane	18 to 80	8.9	
Ebert [15]	1927	Mica	do	−253 to −190	2.4	Coefficients of expansion computed by Hidnert and Dickson from data by Ebert.
				−190 to 0	6.3	
				0 to +100	9.0	
				+100 to 250	9.0	
				250 to 500	11.0	
Ebert [16]	1928	Mica, ruby	do	−253 to −190	2.4	Coefficients of expansion computed by Hidnert and Dickson from data by Ebert who stated that his values are accurate to about 5 percent.
				−190 to 0	6.3	
				0 to +100	9.0	
				+100 to 250	9.5	
				250 to 500	11.3	
Heindl [17]	1932	Pulverized muscovite mica heated to 1,225° C.	No definite direction	20 to 100	7.0	Three small tetrahedra were used for the sample of material for determination of linear expansion by interference method. The coefficient of expansion from 20° to 100° C was obtained from unpublished data.
				20 to 900	8.3	
Megaw [10]	1933	Flake of phlogopite mica from Ceylon.	Perpendicular to cleavage plane.	20 to 100	14.9	Used X-ray method. Probable error of coefficient of expansion, $\pm 1.5 \times 10^{-6}$.
Ebert [18]	1935	Phlogopite mica	Parallel to cleavage plane	0 to 100	13.5	Coefficients of expansion computed by Hidnert and Dickson from data by Ebert who stated that the accuracy of his values is 1 to 2 percent.
				100 to 200	13.5	
				200 to 300	14.5	
				300 to 400	14.5	
				400 to 500	13.0	
		Muscovite mica	do	0 to 100	8.5	
				100 to 200	8.0	
				200 to 300	10.0	
				300 to 400	12.0	
				400 to 500	12.0	
Wood [11]	1937	Four specimens of Canadian phlogopite mica (dark, medium, and light).	Perpendicular to cleavage plane.	20 to 400	16.2 to 18.3	Used X-ray method. Expansion curves between room temperature and 400°C are smooth, and the points obtained in the rising and falling halves of the cycle lie on the same curve.
Rieke and Pu-Yi Wen [19]	1939	Rod prepared from pulverized muscovite mica.	Along axis of rod	100 to 600	12.	Derived from expansion curve published by Rieke and Pu-Yi Wen. They reported a contraction from about 925° to 1050° C.
				600 to 800	35.	

however, affect the perfection of atomic arrangement in the individual crystallites and that no chemical changes are involved.

The term "thermal expansion" as used by the present authors for the micas they investigated represents the thermal expansion of the laminas of one or more disks of mica in a direction perpendicular to the cleavage plane plus the displacements or separations between the laminas during heating or cooling.

2. CHANGES IN STRUCTURE

By H. C. Vacher [13]

The expansion and contraction curves of some phlogopite micas (figs. 6, 7, 9, and 12) suggested that changes in structure occurred between 100° and 250° C. No such change in this temperature range was indicated for the muscovite micas or for the biotite micas. The curves for the first heating-cooling cycle for biotite micas from North Carolina and Maine (figs. 16, 18, and 19) are very steep between 500° and 600° C (or 700° C), but for successive cycles the steep portion is less pronounced. This indicated that a change in the structure of these biotite micas was initiated between 500° and 600° C, which became more complete after each cycle.

In determining the coefficient of thermal expansion for the crystal lattice of Canadian phlogopite with an X-ray spectrometer, Wood [11] found that the lattice expansion and contraction perpendicular to the cleavage plane was uniform and followed the same curve between 18° and 400° C. He showed, however, that the diffraction lines obtained from some samples were considerably more diffuse and broken in the range from 200° to 400° C than in the range from 18° to 200° C. It was concluded that some phlogopite micas form a macromosaic structure when heated above 200° C that incompletely reverts to the initial condition when cooled below 200° C. It is very probable that a change in structure of this nature would be registered on thermal-expansion and contraction curves. In order to determine if this is true for some micas used in the present investigation, Laue diffraction patterns were obtained on some samples (see table 4) at room temperature and at elevated temperatures.

A sketch of the furnace used in obtaining the Laue patterns is shown in figure 22. The furnace was constructed by winding Nichrome wire, 0.020-in. diameter, around asbestos board (⅛ by 1⅜ by 2¾ in.) containing a channel 1/16 in. deep by 11/16 in. wide. The mica-disk specimen (⅝-in. diameter) was sandwiched between two aluminum strips (0.045-in. and 0.010-in. thickness) and slid along the channel until it was opposite the pinhole collimator. The primary and diffracted beams passed through holes (⅛-in. and ¼-in. diameter, respectively) in the specimen holder. Aluminum foil (0.002-in. thickness) covered the ¼-in. diameter hole to reduce the temperature gradient. Diffracted beams from crystals in the aluminum foil were registered on the film, but they were easily distinguished by their small size from those diffracted from the mica specimen. The furnace was fastened to the pinhole collimator by means of a flange and aluminum strip. In this position the collimated beam was perpendicular to the cleavage plane of the specimen. The temperature was

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[13] Division of Metallurgy, National Bureau of Standards.

determined with a Chromel-Alumel thermocouple that had been previously calibrated to correspond to a thermocouple located adjacent to the specimen and in the path of the collimated beam. Unfiltered radiation from a molybdenum target was used in obtaining the Laue patterns. A pattern was obtained at room temperature, and then the temperature was increased to 100° C and maintained constant to within 15° C. After exposing the film for 4 hours, the temperature was increased to 150° C and another film was exposed for 4 hours. The furnace was cooled after each 4-hour exposure period unless there was sufficient time to obtain a 4-hour exposure at a different temperature during the same day. Table 4 shows the heating-cooling cycles and the temperatures at which patterns were obtained for each specimen of mica.

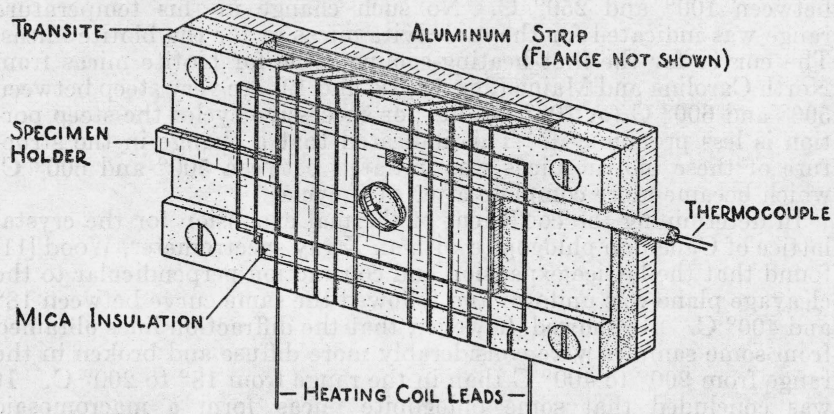

FIGURE 22.—*Furnace used in obtaining Laue X-ray diffraction patterns of mica at elevated temperatures.*

Different micas differed characteristically within the range of temperatures investigated. Figure 23 illustrates the change that occurred in muscovite mica (sample 1682) between 100° and 145° C. The type of pattern (fig. 23, A) obtained at room temperature before and after the heating-cooling cycles, consisted of single diffraction spots, whereas at 145°, 190°, 240°, and 285° C some of the spots were doubled (fig. 23, *B*). The doubling of spots at elevated temperatures cannot always be considered as reversible, for patterns of the type shown in figure 23, *B*, were obtained from other muscovite mica samples in the "as received" condition. Double spots indicate that diffractions were registered from two crystals differing slightly in orientation. It is noteworthy that the double spots are symmetrical to the line of symmetry (*Y*, fig. 23). The thermal-expansion and contraction curves (sample 1682, fig. 1) show no evidence of a transition to correspond to the change in structure that occurred between 100° and 145° C.

Patterns typical of the changes that occurred in some phlogopite micas (samples 1681, 1681A, and 1688) when heated to elevated temperatures are shown in figure 24. Inspection shows that spots in three zone lines (marked *X*, *Y*, and *Z* in fig. 24) intersecting at approx-

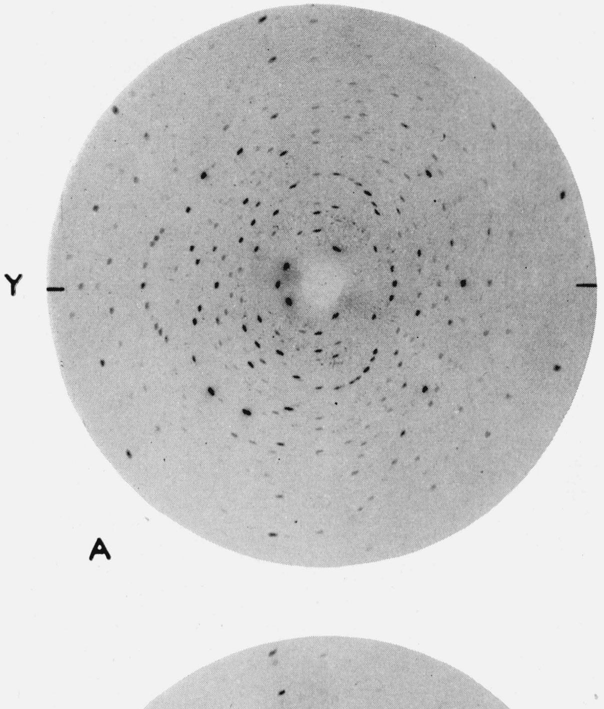

FIGURE 23.—*Laue X-ray diffraction patterns of muscovite mica from Brazil (sample 1682).*

A, Typical of patterns obtained at and below 100° C; *B*, typical of patterns obtained at 145°, 190°, 240°, and 285° C. Double spots are the result of diffractions by two crystals of slightly different orientations. Molybdenum target, distance from specimen to film, 4 cm. Reduced ¼.

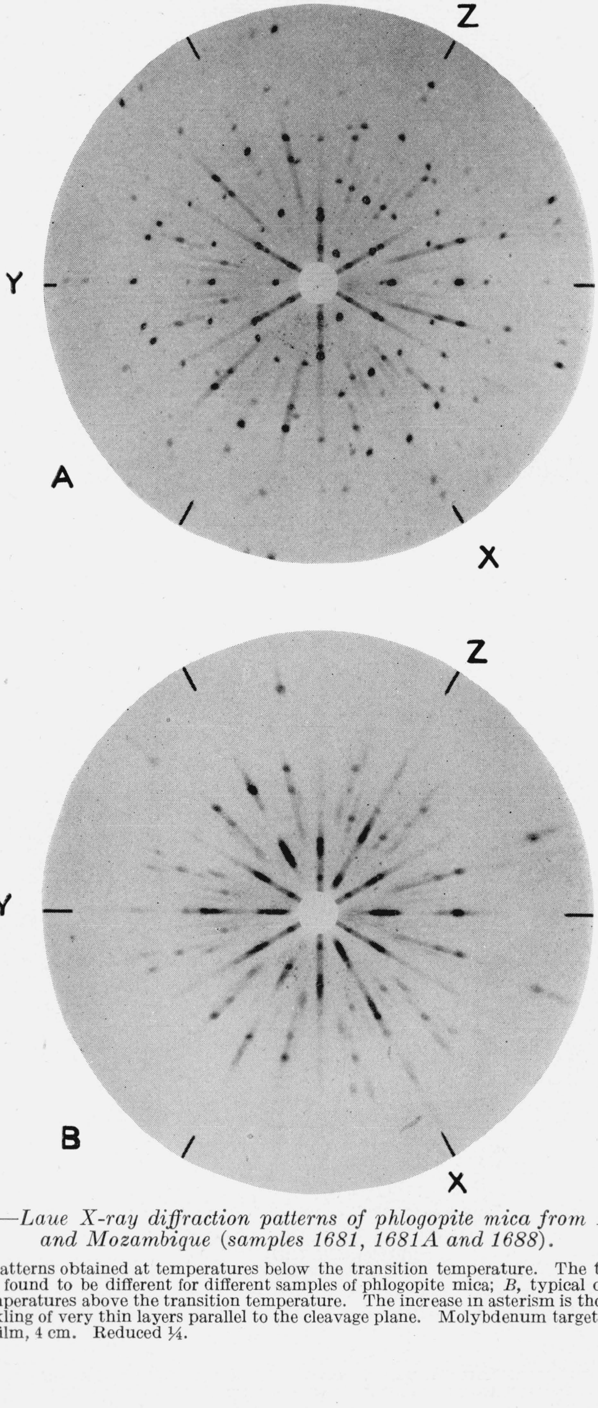

FIGURE 24.—*Laue X-ray diffraction patterns of phlogopite mica from Madagascar and Mozambique (samples 1681, 1681A and 1688).*

A, Typical of patterns obtained at temperatures below the transition temperature. The transition temperature was found to be different for different samples of phlogopite mica; *B*, typical of patterns obtained at temperatures above the transition temperature. The increase in asterism is thought to be the result of buckling of very thin layers parallel to the cleavage plane. Molybdenum target, distance from specimen to film, 4 cm. Reduced ¼.

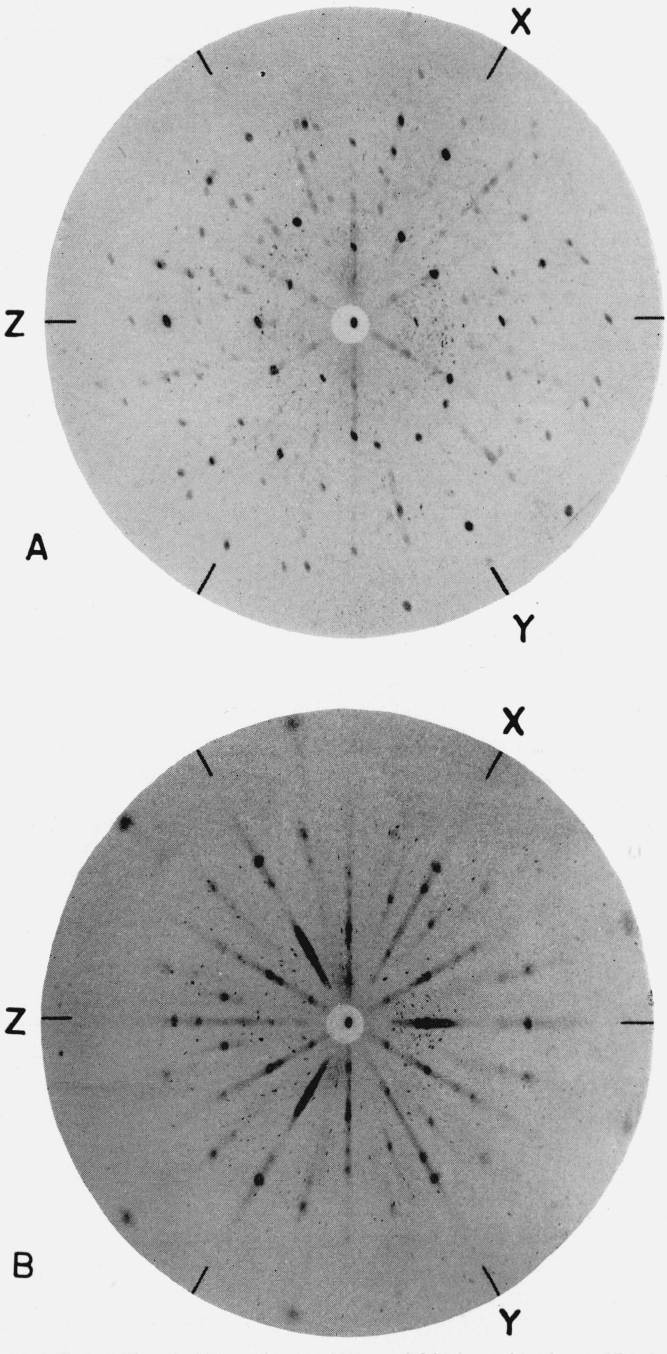

FIGURE 25.—*Laue X-ray diffraction patterns of biotite mica from North Carolina (sample 1713).*

A, Obtained before heating; *B*, typical of patterns obtained at 475°, 590° C, and after cooling to room temperature. The patterns indicate that heating caused a change in crystal symmetry and buckling of thin layers parallel to the principal cleavage plane. Molybdenum target, distance from specimen to film, 5 cm. Reduced ¼.

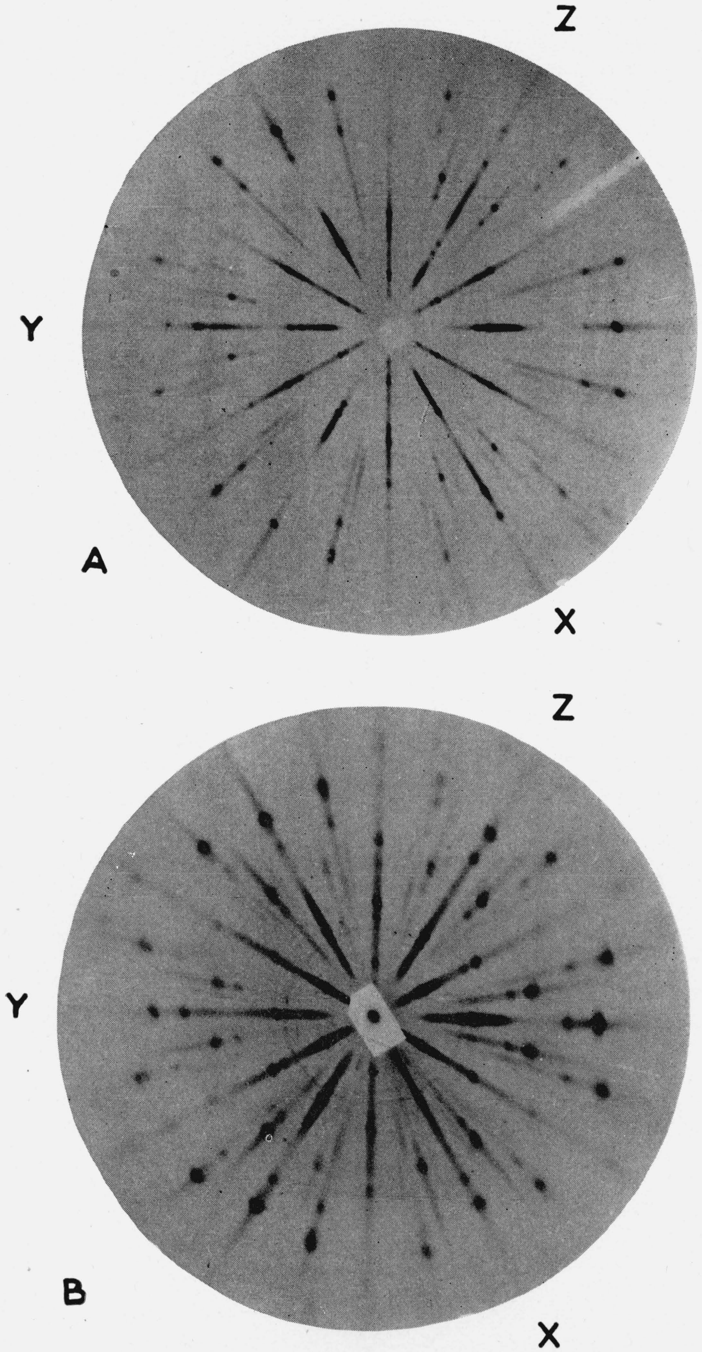

Figure 26.—*Laue X-ray diffraction patterns of phlogopite and muscovite mica after heating to 800° C. and cooling to room temperature.*

A, Phlogopite mica from Mozambique (sample 1688); *B*, muscovite mica from Brazil (sample 1682). Heating has transformed the structure (fig. 23) of muscovite mica to a structure similar to that of phlogopite mica. Molybdenum target, distance from specimen to film, 6 cm. Reduced ¼.

imately 120°, developed considerable asterism (radial streaks) at elevated temperatures. In the patterns obtained from Madagascar phlogopite, asterism appeared on these zones between 190° and 245° C and became more pronounced at temperatures between 275° and 385° C. However, after Madagascar phlogopite was cooled from 385° C to room temperature a pattern showed that the structure reverted to its initial condition. The behavior of the Mozambique phlogopite was slightly different from that of the Madagascar phlogopite. Asterism on the *X*, *Y*, and *Z* zone lines appeared between 145° and 185° C and did not increase during heating to 385° C. However, after the Mozambique phlogopite was cooled to room temperature a slight amount of asterism was present. Patterns obtained from specimens of phlogopite micas that had been heated and cooled incident to the thermal-expansion determinations showed slightly more asterism than the patterns obtained from specimens that had been heated and cooled in the furnace shown in figure 22. This increase in retained asterism was probably caused by the higher temperature to which the specimens were heated during the thermal-expansion determinations (see page 315). The temperature ranges in which Madagascar and Mozambique phlogopite micas indicated breaks in the expansion and contraction curves (figs. 6, 7, and 9) are in good agreement with the temperature ranges in which a change in structure was indicated.

TABLE 4.—*Heating cycles and temperatures at which Laue patterns* [a] *of mica were obtained*

Sample No.	Mica	Heating cycle	Temperatures
			°C
1682 [b]	Muscovite, from Brazil, as received	1	20, 100, 20
		2	20, 145, 20
		3	20, 190, 240, 20
		4	20, 285, 20
1681 [b]	Phlogopite, from Madagascar, as received	1	20, 100, 145, 20
		2	20, 200, 20
		3	20, 245, 275, 20
		4	20, 340, 385, 20
1681A [c]	Phlogopite, from Madagascar, as received	1	20, 100, 135, 20
		2	20, 135, 190, 20
		3	20, 240, 290, 20
		4	20, 340, 385, 20
1688 [b]	Phlogopite from Mozambique, Africa, as received	1	20, 105, 20
		2	20, 145, 185, 20
		3	20, 245, 20
		4	20, 275, 340, 20
		5	20, 385, 20
1713	Biotite, from North Carolina, as received	1	20, 475, 20
		2	20, 590, 20
1713 [d]	Biotite, from North Carolina	1	20, 475, 20
		2	20, 590, 20

[a] See figs. 23 to 26, inclusive.
[b] Laue pattern was also obtained after specimen was heated to 800° C and cooled to room temperature.
[c] From same lot of mica as sample 1681.
[d] After heating incident to thermal expansion determinations. (See fig. 16.)

The structure developed in biotite mica (sample 1713) at elevated temperatures did not revert to its initial condition as did that developed in the muscovite and phlogopite micas. The structure of the biotite mica before and after heating is indicated by the patterns of figure 25. The initial pattern of the biotite mica is similar to the initial patterns of the phlogopite micas. At 475° and 590° C and after cooling to room temperature, the patterns of the biotite mica were alike and had a degree of asterism similar to that shown in patterns obtained from the phlogopite micas in the range from 185° to 385° C. The permanent change in structure brought about by heating to 475° and 590° C is in agreement with the anomalous thermal expansion and contraction curves obtained for biotite mica (fig. 16).

The fact that samples of muscovite mica indicated very large increases in thickness after heating to 800° C (see page 344) suggested the probable existence of a transition in muscovite mica at elevated temperatures.[14] The stable structure produced in biotite mica at elevated temperatures also suggested that the initial structures of muscovite and phlogopite micas might be altered permanently if heated to a sufficiently high temperature. Accordingly, patterns were obtained from muscovite mica (sample 1682) and phlogopite micas (samples 1681 and 1688) after heating to 800° C and cooling to room temperature. The pattern obtained from Madagascar phlogopite (sample 1681) showed some asterism but in general was similar to pattern *A* in figure 24. However, the pattern obtained from Mozambique phlogopite (sample 1688) shows considerable asterism (pattern *A*, fig. 26) and is similar to the patterns obtained at temperatures between 185° and 385° C (pattern *B*, fig. 24). The pattern obtained from the muscovite mica after heating to 800° C and cooling to room temperature also shows considerable asterism (pattern *B*, fig. 26). Inspection of the specimens showed that heating to 800° C caused little change in the Madagascar phlogopite but had caused the Mozambique phlogopite and the muscovite mica to swell and to separate partly into numerous laminas.

The structures of the micas investigated appeared to be fragmented by heating, as indicated by the asterism of all reflections. The asterism of the type shown in the initial patterns (before heating) was confined to certain crystallographic zones, thereby indicating a destruction in the lattice periodicity perpendicular to the principal cleavage plane [21]. The fragmentation produced in mica as a result of heating appears to be caused by the independent buckling of very thin layers parallel to the principal cleavage plane. The asterism resulting from buckling is superimposed on the asterism resulting from a destruction in the lattice periodicity.

3. POWER FACTORS

By E. L. Hall[15]

The power factors[16] of some samples of muscovite and phlogopite micas were determined at 100 and 1,000 kc/s in terms of a type 722–N General Radio precision condenser. The samples were in the form of 16-mm (⅝-in.)-diameter disks that had been punched from larger pieces of mica. A tinfoil disk approximately 14 mm (⁹⁄₁₆ in.) in diameter was cemented to one side of each mica specimen, and a tinfoil disk about 10 mm (⅜ in.) in diameter was cemented to the other side of the specimen with small amounts of clear petroleum jelly, thus forming a small electric condenser. Four test specimens were prepared from each sample. The capacitance of the test specimens ranged between 70 and 200 micromicrofarads, depending upon thickness. Further details of the test method and equipment were published in a recent paper [23]. Table 5 gives the results obtained.

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[14] Zwetsch [20] found a change in the X-ray spectra of muscovite mica above 600° C.

[15] Electrical Division, National Bureau of Standards.

[16] A tentative method of test for power factor of mica has been published [22].

TABLE 5.—*Power factors of some muscovite and phlogopite micas before and after treatment*

Sample number	Description	Treatment	Power factors	
			100 kc/s	1,000 kc/s
			Percent	*Percent*
1682 [a]	Muscovite mica from Brazil.	As received	0.16 .08 .04 .04	0.10 .06 .03 .03
			Avg 0.08	0.06
1682 [a]	do	After heating to 600° C and cooling to room temperature with load of 30 lb/in.[2] incident to thermal expansion tests. (See fig. 1.)	0.13 .04 .15 .09	0.10 .04 .10 .06
			Avg 0.10	0.08
1689 [a]	Muscovite mica from Guatemala.	As received	0.06 .10 .05 .08	0.06 .07 .06 .06
			Avg 0.07	0.06
1689 [a]	do	After heating to 600° C and cooling to room temperature with load of 30 lb/in.[2] incident to thermal expansion tests. (See fig. 3.)	0.08 .08 .06 .06	0.06 .06 .04 .05
			Avg 0.07	0.05
1681 [a]	Phlogopite mica from Madagascar.	As received	0.18 .34 .12 .28	0.07 .16 .05 .11
			Avg 0.23	0.10
1681 [a]	do	After heating to 600° C and cooling to room temperature with load of 30 lb/in.[2] incident to thermal expansion tests. (See fig. 6.)	1.00 .93 .96 1.25	0.39 .37 .42 .62
			Avg 1.04	0.45
1681 [b]	do	After heating to 600° C and cooling to room temperature without load.	0.90 .42 .37 .39	0.42 .18 .15 .17
			Avg 0.52	0.23
1688 [a]	Phlogopite mica from Mozambique, Africa.	As received	0.95 .94 1.03 .87	0.67 .63 .65 .52
			Avg 0.95	0.62
1688 [a]	do	After heating to 600° C and cooling to room temperature with load of 30 lb/in.[2] incident to thermal expansion tests. (See fig. 9.)	1.93 1.77 2.1 2.2	0.94 .88 1.06 --------
			Avg 2.0	0.96
1688 [b]	do	After heating to 600° C and cooling to room temperature without load.	>3 >3 >3 >3	1.55 1.53 1.63 1.93
			Avg >3	1.66

[a] Power-factor measurements were made at 25° C and relative humidity of 50 percent.
[b] Power-factor measurements were made at 26° C and relative humidity of 34 percent.

The power factors of nearly all specimens of muscovite mica from Brazil and Guatemala were slightly greater at 100 kc/s than at 1,000 kc/s. The power factor of each specimen of phlogopite mica was considerably greater at 100 kc/s than at 1,000 kc/s. The power factors of the samples of phlogopite mica from Madagascar and Mozambique are considerably larger than the power factors of the samples of muscovite mica from Brazil and Guatemala. Heating the samples of phlogopite and muscovite micas to 600° C, with or without a load on each sample, caused considerable increases in the power factors of the phlogopite micas and only slight changes in the power factors of the muscovite micas. The power factors of the phlogopite mica from Madagascar which was heat treated without a load, are less than the power factors of this mica heat treated with a load. However, the power factors of the phlogopite mica from Mozambique heat treated without a load are considerably greater than the power factors of this mica heat treated with a load.

An examination of table 5 indicates divergent values for the power factors of different specimens of a given mica. These differences were not due to errors of observation but to variations in the specimens. A discussion of differences of the power factors of mica is given in a previous paper [23].

4. EFFECTS OF HEAT TREATMENTS ON THICKNESS, OPACITY, AND COLOR[17]

Five disks (about ⅝-in. diameter) were cut from each of the 50 micas, and their thicknesses were measured to the nearest 0.0001 in. with a Zeiss micrometer that applied a load of 36 oz (1.02 kg) between parallel contacts having 0.315-in. (8.00 mm) diameter. The thicknesses of the disks of mica ranged from 0.0036 to 0.0594 in. (0.092 to 1.51 mm).

One disk from each kind of mica was placed on a slab of flint clay and heated without a load in an electric furnace to 600° C maintained at this temperature for 1 hour, and then allowed to cool in the furnace. A second, third, fourth, and fifth disk of each kind of mica was heated to 700°, 800°, 900°, and 1,000° C, respectively, in a similar manner. After the disks of mica were cooled to room temperature, their thicknesses were measured with the Zeiss micrometer that applied the load indicated above. Table 6 shows the changes in thickness (in percent) of the disks of mica.

All of the muscovite micas except the processed plate mica (sample 1687) indicated changes in thickness of 2 percent or less after heating to 600° C. Nearly all of the muscovite samples indicated the greatest increases (up to 155 percent) after heating to 800° C. These results suggest the probable existence of a transition in muscovite micas between 600° and 800° C or a marked increase in the rate of cleavage in this temperature range. For nearly all of the muscovite samples, the increase in each sample after heating to 900° C is approximately equal to the increase after heating to 1,000° C and these changes are considerably less than those after heating to 800° C. Horton [3] stated that at 400° C, muscovite mica begins to give up minute quantities of its water of crystallization, at 700° C normal muscovites

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[17] Designations of opacity and color and their interpretations have been taken from a report on test 43C-97/44 from the Photometry and Colorimetry Section of the National Bureau of Standards.

lose about one-tenth percent of their weight, and as dehydration progresses the mica swells and loses its transparency. The data on muscovite micas (under load) in table 6 are not in agreement with Horton's statement relating to increase in swelling, if applied to temperatures between 800° and 1,000° C.

The changes in thickness of the phlogopite samples ranged from a decrease of 25 percent to an increase of 39 percent. From these results it appears that phlogopite mica resists elevated temperatures better than muscovite mica. Horton [3] indicated that phlogopite micas, particularly light-colored varieties, are more resistant to heat than muscovite micas and will stand a temperature of 800° C without appreciable dehydration, probably because they contain less water of crystallization.

The outstanding feature of three of the four biotite samples is the fact that they show large increases in thickness (17 to 134 percent) after heating to 600° C. The changes of the four biotite samples after heating to higher temperatures indicated increases from 1 to 45 percent of the original thickness.

The maximum change in thickness (increase of 45 percent) of the ripidolite sample was observed after heating to 800° C. For the zinnwaldite sample, the maximum change (increase of 120 percent) was noted after heating to 1,000° C.

Some of the heat-treated specimens of mica when measured without a load were found to have increased 8 to 20 times the original thickness. When measured with the Zeiss micrometer which applied a load of 36 oz, the thickness of these specimens was reduced to less than two times the original thickness (see table 6).

In order to indicate the changes in appearance which each mica had undergone as a result of the heat treatments at elevated temperatures, the color designations of the disks of mica after the heat treatments and of disks that had not been heated are given in table 6. The appearance of each mica disk is indicated by an opacity and a color designation, and if necessary, by a one- or two-word description of texture.

The opacity of each specimen of mica is indicated as clear, translucent, or opaque. If, by looking through the specimen, objects (chimney and trees silhouetted against the sky) could be seen distinctly, the specimen was noted as "clear." If the specimen transmitted a noticeable amount of light but so scattered as to yield indistinct images or no image, it was noted as "translucent." If no noticeable light was transmitted, the specimen was noted as "opaque."

The colors of all specimens not exhibiting metallic luster were designated in accord with the ISCC–NBS method of designating colors described by Judd and Kelly [24] and by Kelly [25]. These designations accord with those used in the National Formulary (7th edition, 1942).

Most of the opaque specimens of mica have a metallic luster and were given a color designation by comparison with polished samples of silver, gold, and copper. These color designations and their meanings are as follows:

Silver.—Substantially the same body color and highlight as polished silver.

Gold.—Substantially the same body color and highlight as polished gold.

TABLE 6.—*Color designations and changes in thickness* [a] *of 50 samples of mica after heating to elevated temperatures and cooling to room temperature*

The following abbreviations of color terms are those proposed by Nickerson and Newhall [26]. To these have been added abbreviations for metallic colors and terms descriptive of opacity and texture:

B=blue.
b=bluish
Bk=black
Br=brown
br=brownish.
cl=clear.
Cp=copper.
d=dusky.
dk=dark.
G=green.
g=greenish.
Go=gold.
go=golden.
Gr=gray.
lt=light.
mod=moderate.
O=orange.
o=orange.
Ol=olive.
ol=olive.
op=opaque.
p=pale.
Po=polychrome.
R=red.
r=reddish.
Sl=silver.
sl=silvery.
ta=tarnished.
tr=translucent.
v=very.
wk=weak.
Y=yellow.
y=yellowish.

Sample No.	Source	Description of appearance before heating	After heating to 600° and cooling to 20° C.		After heating to 700° and cooling to 20° C.		After heating to 800° and cooling to 20° C.		After heating to 900° and cooling to 20° C.		After heating to 1,000° and cooling to 20° C.	
			Description of appearance	Change in thickness	Description of appearance	Change in thickness	Description of appearance	Change in thickness	Description of appearance	Change in thickness	Description of appearance	Change in thickness
A. MUSCOVITE MICA												
				Percent		*Percent*		*Percent*		*Percent*		*Percent*
1682	Brazil	cl, v p Br	cl, y Gr, Br spots	1	tr, Sl, y Gr	1	op, Sl	118	op, y Sl	41	op, Sl	44
1684	Canada	cl, v p O	cl, v p O	1	cl, y Gr	0	cl, y Gr	0	tr, Sl, lt br Gr	1	op, y Sl	47
1685	Brazil	cl, y Gr	cl, y Gr	1	tr, p Br, Br spots.	8	op, Sl	115	op, Sl	51	op, y Sl	45
1686	do	cl, v p Br	cl, wk Y	0	tr, Sl, p Br, Po	2	op, Sl	140	op, y Sl	54	op, Sl, p Br	52
1687	Plate mica (processed sheet).	tr, dk Sl	op, dk Sl	22	op, dk Sl	62	op, dk Sl	90	op, dk Sl	68	op, Sl, wk O	66
1689	Guatemala (Verde claro)	cl, p Ol	cl, d YG	1	tr, mod Ol, Po	1	op, sl Go	66	op, r Go	14	op, sl r Go	22
1690	Guatemala (Verde oscuro)	cl, ol Bk	cl, mod Ol	−0	op, sl g Go, Po	9	op, Go	71	op, go Cp	33	op, go Cp	24
1691	Colombia (Black stained)	cl, y Gr	cl, p Ol, Br spots	0	op, y Sl, Po	31	op, sl Go	81	op, r Go	[b] 64	op, r Go	51
1692	Colombia (Stained or good stained).	cl, p Ol	cl, p Ol	0	op, y Sl, Po	22	op, sl Go	124	op, r Go	46	op, r Go	52
1693	Colombia	cl, p Ol, Bk specks.	cl, wk Ol, Bk specks.	−1	op, y Sl	39	op, Go	126	op, r Go	56	op, sl r Go	44
1694	Peru	cl, y Gr	cl, y Gr	−1	op, Sl	46	op, sl Go	113	op, Go	61	op, sl Go	52
1695	do	cl, y Gr	cl, p Ol	−1	tr, y Sl	23	op, sl Go	65	op, sl Go	46	op, r Go	42
1696	Bolivia	cl, lt y Br	cl, p Br	−1	cl, wk Br, Po stripes.	0	op, dk Sl, Po	66	op, dk r Gr	27	op, r Go	22
1697	do	cl, lt Gr	cl, y Gr	+1	cl, p Ol, Po	−1	op, ta Sl	59	op, dk go Cp	23	op, dk go Cp	29
1698	do	tr, Sl	op, Sl	1	op, Sl	+24	op, y Sl	100	op, Go	44	op, sl r Go	40
1699	New Hampshire (first quality).	cl, p Br	cl, p Ol	0	cl, p Ol, Po, Br spots.	−1	op, y Sl	110	op, sl Go	50	op, sl Go	45

1700	New Hampshire (second quality).	cl, p Br	cl, wk YG, Y spots.	0	tr, y Sl, Po	+3	op, y Sl	96	op, Go	49	op, sl Go, wk y O.	40
1701	South Dakota (first quality).	cl, p Br	cl, y Gr	0	tr, b Sl	6	op, Sl	106	op, Sl, lt Gr	52	op, Sl lt Gr	58
1702	South Dakota (second quality).	cl, v p Br	cl, p Br	0	op, b Sl	22	op, Sl	104	op, Sl, lt Gr	58	op, Sl, lt Gr	60
1703	North Carolina (first quality).	cl, mod Ol	cl, wk Ol G	1	op, ta Sl, Po	15	op, dk Go	125	op, go Cp	29	op, go Cp	34
1704	North Carolina (second quality).	cl, mod Ol	cl, mod Ol	0	op, ta Sl, Po	28	op, r Go	101	op, go Cp	34	op, go Cp	34
1705	North Carolina (first quality).	cl, p Br	cl, v p Br	1	op, Sl	13	op, y Sl	101	op, sl Go	41	op, sl Go	44
1706	North Carolina (second quality).	cl, v p Br	cl, p Br	1	op, Sl	20	op, y Sl	104	op, sl Go	48	op, sl Go, mod y Br.	44
1707	New Mexico (first quality).	cl, wk YG	cl, wk YG	1	cl, wk YG, Po	−1	op, ta Sl, Po	83	op, dk Go	35	op, sl Go	30
1708	New Mexico (second quality).	cl, wk YG	cl, wk ol G	0	tr, g Sl, Po	+2	op, dk Go, Po	74	op, dk go Cp	31	op, dk go Cp	32
1729	Ontario, Canada	cl, br Bk	cl, br Bk	1	op, ta Sl, Po	9	op, sl Go	[b] 155	op, go Cp	51	op, go Cp	[b] 160
1731	Quebec, Canada	cl, lt y Br	cl, v p Br	0	op, y Sl	101	op, Sl	20	op, y Sl wk y O.	69	op, y Sl, wk y O.	63
1741	Bolivia	cl, y Gr	cl, wk Y	1	cl, wk Y, Po	−2	op, ta Sl	85	op, ta Sl	38	op, ta Sl	38
1742	Argentina (semiclear)	cl, p Br	cl, p Ol	−1	op, Sl	+29	op, sl Go	88	op, Go	51	op, Go	48
1743	Argentina (semistained)	cl, p Ol	cl, p Ol	−1	op, Sl	25	op, Go	91	op, r Go	45	op, r Go	47
1744	Argentina (stained)	cl, p Ol	cl, p Ol	0	op, Sl	40	op, sl Go	72	op, sl Go	47	op, sl Go	49
1745	Argentina	cl, y Gr	cl, y Gr	−1	cl, p Ol, Po	2	op, y Sl	83	op, y Sl	48	op, y Sl	49
1746	Argentina (clear)	cl, y Gr	cl, y Gr	0	tr, Sl, p Br	17	op, Sl	79	op, Sl	56	op, Sl	57
1747	Argentina (semiclear)	cl, v p Br	cl, v p Br	−2	tr, Sl, p Br, Po	16	op, Sl	90	op, y Sl	62	op, y Sl, lt y Br.	50
1748	do	cl, y Gr	cl, y Gr, Br spots	0	tr, Sl, p Br	7	op, Sl	53	op, y Sl, mod y Br.	44	op, y Sl, lt y Br.	41
1749	Maine	cl, y Gr									op, Sl, p Br	52

B. PHLOGOPITE MICA

1681	Madagascar	cl. d Br	op, ta Sl	5	tr, ta Sl, d Br	0	op, sl Go	34	op, r Go	10	op, r Go	12
1683	do	op, br Bk	op, br Bk	−1	op, br Bk	−1	op, v dk Go	−1	op, dk Go	2	op, r Go	6
1688	Mozambique, Africa	op, br Bk	op, v dk Go	+1	op, dk Go	+24	op, dk r Go	+39	op, dk r Go	17	op, dk r Go, Sl spots.	9
1709	do	op, br Bk	op, br Bk	−7	op, br Bk	−2	op, br Bk	−4	op, br Bk, Po	4	op, dk go Cp	7
1710	do	tr, y Sl	op, y Sl	+7	op, y Sl	−6	op, sl Go	−6	op, Go	−11	op, sl r Go	−25
1711	Burgess, Canada	op, sl Cp	op, y Sl	5	op, y Sl	+6	op, y Sl	+5	op, y Sl	+3	op, y Sl	+8
1712	New York	cl, v p Br	tr, Sl	5	tr, Sl	0	tr, y Gr	−3	tr, Sl	2	op, Sl, lt Gr	8
1732	Quebec, Canada	op, br Bk	op, br Bk, speckled.	−0	op, br Bk	−1	op, br Bk, Po	0	op, dk r Go	−1	op, dk r Go	2

See footnotes at end of table.

TABLE 6.—*Color designations and changes in thickness*[a] *of 50 samples of mica after heating to elevated temperatures and cooling to room temperature*—Continued

Sample No.	Source	Description of appearance before heating	After heating to 600° and cooling to 20° C.		After heating to 700° and cooling to 20° C.		After heating to 800° and cooling to 20° C.		After heating to 900° and cooling to 20° C.		After heating to 1,000° and cooling to 20° C.	
			Description of appearance	Change in thickness	Description of appearance	Change in thickness	Description of appearance	Change in thickness	Description of appearance	Change in thickness	Description of appearance	Change in thickness
				C. BIOTITE MICA								
				Percent		*Percent*		*Percent*		*Percent*		*Percent*
1713	Newdale, N. C	op, Bk	op, dk go Cp, Po	[b]75	op, Cp	29	op, go Cp	37	op, Cp	45	op, Cp	11
1730	Ontario, Canada	op, Bk	op, Bk	—0	op, Bk	1	op, Bk, Po	20	op, Bk	23	op, Bk	9
1733	Macon County, N. C	op, br Bk	op, d y Br, flakes	[b]17	op, dk O flakes, Po.	18	op, Cp	33	op, dk Br	30	op, Cp, Po	7
1734	Oxford County, Maine	op, br Bk	op, dk y Br, flakes.	[b]134	op, Cp	28	op, Cp	[b]37	op, Cp	[b]25	op, mod y Br	34
				D. RIPIDOLITE MICA								
1714	Pennsylvania	op, g Bk	op, ol Bk	1	op, Bk, Po	2	op, ta Sl	45	op, y Sl	14	op, y Sl	9
				E. ZINNWALDITE MICA								
1735	Virginia	tr, dk y Br	tr, mod Br	0	op, wk Br	6	op, wk Br	25	op, br Gr, mat	45	op, dk Gr, mat	120

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[a] During the measurement of the thickness of each specimen of mica, the micrometer applied a load of 36 oz between parallel contacts having 0.315-in. diameter. Each negative value in the table indicates a decrease in thickness.

[b] With no load on specimen, the increase in thickness was 8 to 20 times the original thickness.

Copper.—Substantially the same body color and highlight as polished copper.

Yellowish silver.—More like silver than gold.

Silvery gold.—More like gold than silver.

Reddish gold.—More like gold than copper.

Golden copper.—More like copper than gold.

Silvery copper.—More like copper than silver.

Tarnished silver.—Highlight darker and yellower than that of polished silver.

Polychrome.—Many colored, as by interference colors.

Many of the specimens of mica have an appearance partially like polished metal and partially dissimilar. The luster is metallic and the highlight can be described fairly well by the above terms. The body color, however, is lighter than that of the metal on account of light being diffused from the body of the specimen so that the metallic term is only partially applicable. These specimens have accordingly been given a double designation, a designation of the metallic luster by the above terms combined with a body-color designation in accord with the method of designating colors by Judd and Kelly [24].

The specimens of mica were examined by light from the north sky. The designations of body color were found by comparisons with the charts of the Munsell Book of Color, complete 40-hue-chart edition, specimen and charts being illuminated along angles centering on 45 degrees and viewed approximately along the perpendicular. The designations of metallic luster were obtained chiefly by observations in which the specimen and the metallic standard were held in the same plane, illuminated at about 20 degrees from the perpendicular, and viewed at the angle of mirror reflection.

All but two of the specimens of mica exhibited luster, either metallic or vitreous. The two exceptions were designated "mat" in table 6 (sample 1735, heat treated at 900° and 1,000° C).

The appearance of the mica specimens gives a fairly reliable indication of the degree to which heat treatment caused cleavage along crystal planes. The first indication of cleavage is the change from vitreous to a polychrome luster, and for clear specimens the change to translucency. This first indication seems to be quickly followed by a change from polychrome luster to metallic luster, and for clear specimens the change from translucent to opaque. Both of these changes are to be expected from the development of many cleavages. A third stage of cleavage is indicated by the development of a body color lighter than that of the corresponding polished metal. The final stage of disintegration, indicated by a loss of luster, is the breaking up of the crystals into minute elementary crystals. Only the sample of zinnwaldite mica reached this stage; the heat treatment to 1,000° C left it an ashlike, mat dark gray.

The color differences among the original untreated micas seem to be ascribable to inclusions of varying amounts. The spots that develop from the heat treatment suggest segregation of these materials.

Large increases in thickness of nearly all of the muscovite samples accompanied changes from clear or translucent to opaque, or from polychrome to metallic color.

Horton [3] reported data by F. R. Caldwell, of the National Bureau of Standards, on some physical properties (color, condition of surface, opacity, separation of laminas, texture, and size) of 19 samples of mica (muscovite, phlogopite, and biotite) after heat treatments (without a load) to various temperatures between 600° and 1,200° C. There was no perceptible change in these micas after heating at 600° C except that a biotite mica from South Dakota swelled to 10 times its original thickness and a phlogopite mica from Quebec, Canada, swelled to about 8 times its original thickness. At 800° C all muscovite samples showed radical changes, most of them swelling considerably. Two of the Canadian and all of the Madagascar phlogopites were not notably affected at 800° C. At 1,200° C all the micas blistered and were so changed as to be unsuitable for use.

IV. DISCUSSION

It has been stated by Mohr [6], Lewis, Hall, and Caldwell [27], Horton [3], Houk [28] and Gwinn [29] that muscovite and phlogopite micas withstand heating between 400° and 600° C without appreciable change. The results obtained in the present investigation indicate that there was little change in the samples of muscovite mica when heated to 600° C, but there were appreciable changes in some of the samples of phlogopite mica when heated to 600° C. Some of the samples of phlogopite mica had an enormous thermal expansion when heated to this temperature. Heating some samples of phlogopite mica to 600° C and cooling to room temperature caused appreciable changes in power factors, opacity, and thickness.

There is some evidence [11] indicating that the thermal expansion of the elementary crystals of phlogopite mica is comparatively small. The pronounced increases in the thermal expansion of some phlogopite micas (figs. 6, 7, 9, 12, and 14) and biotite micas (figs. 16 to 19, inclusive) may be ascribed to the displacement or tilting of the elementary crystals of the mica laminas on heating.

Powell and Griffiths [30] investigated the thermal conductivity (under pressure) of some muscovite and phlogopite micas up to a temperature of 600° C, in a direction perpendicular to the cleavage plane. Five specimens of Indian muscovite mica showed slight variations of the thermal conductivity with temperature over the range 100° to 600° C. However, Canadian and Madagascan phlogopite micas indicated a pronounced decrease in thermal conductivity between 150° and 250° C, which was only partially reversible on cooling. Subsequent examination of these micas by Wood [11] by means of X-ray crystal analysis revealed the fact that in these samples that suffered appreciable change in conductivity, the arrangement of the elementary crystals composing the mica laminas became displaced from their ordered setting at approximately the same temperature as the thermal conductivity change. It was considered that the surface crystals became stepped as a result of the heat treatment, even though the surface still appeared to be approximately plane.

Wood [11] concluded that these structural changes alone appeared to be responsible for the anomalies in the thermal conductivity of phlogopite mica. He stated that "The fall in thermal conductivity of these micas at elevated temperatures is to be explained by the physical disturbances in the macrostructure in which an initially perfect crystal disintegrates into innumerable elements, themselves intrinsically perfect but differing very widely in orientation from the initial uniform formation. It is reasonable to suppose that the extra boundaries thus produced increase the resistance to the heat flow. It is a new and interesting point that such a mosaic formation may be brought about by a rise in temperature and may actually exhibit a partial recovery on cooling, and, as found subsequently, on aging. The tendency to recovery and the critical temperature required suggest that the fragmentation is not due to escape of gas, known to be evolved by heated mica, but rather associated more intimately with the atomic structure."

In view of the fact that certain micas possess tremendously high thermal expansion in a direction perpendicular to the cleavage plane, it is possible to use them in temperature responsive devices.[18] For example, one or more disks of a high-expanding mica in combination with a metal, alloy, or nonmetallic material may be employed so that when this combination is heated or cooled, a large differential expansion or contraction occurs. The large differential expansion and contraction offer a basis for controlling and indicating temperatures over a wide, useful range. The action caused by the differential expansion or contraction can be converted into a linear or angular movement. The enormous thermal expansion, many times that of any other known solid material, makes it possible to use a considerably smaller expanding element in a temperature responsive device than is possible with a so-called "high-expanding" element of any other solid material. When high-expanding mica is used to operate thermal responsive devices it may not be necessary to depend on mechanical magnification which is often required with other materials.

The high-expanding micas, which can be used to operate temperature responsive devices, may present serious difficulties when used for some other purposes. For example, the use of such micas as insulating parts in electric devices where large dimensional changes are not desired, would affect the usefulness of the devices when subjected to large temperature changes. Furthermore, the use of high-expanding micas in locked or rigidly bound parts of a device subject to large temperature changes would undoubtedly result in permanent injury to the device.

V. SUMMARY AND CONCLUSION

This paper gives data on the linear thermal expansion, changes in structure, power factors, and effects of heat treatments on thickness, opacity, and color of micas (muscovite, phlogopite, biotite, ripidolite, and zinnwaldite) from different domestic and foreign sources.

The results obtained on the thermal expansion of 30 samples of micas in a direction perpendicular to the cleavage plane when subjected to a load over the range 20° to 600° C (or 700° C) are indicated in figures 1 to 21, inclusive. Tremendous expansion was noted for some of the samples of phlogopite and biotite micas. The maximum coefficients of expansion of the samples of phlogopite and biotite micas are 0.0552 (between 100° and 114° C) and 0.00411 (between 500° and

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[18] During the preparation of the present paper, the attention of the authors was called to U. S. Patent 2,246,536, which was granted to Josef Reinthaler on June 24, 1941. His invention relates to thermostat and other thermomotive means by the use of a pile of plates of muscovite mica to 600° C and of phlogopite mica to 1,200° C. The present authors tested the 36 muscovite micas indicated in table 1 between 20° and 600° C, and did not find any that are suitable in this temperature range for temperature responsive devices.

600° C) per degree centigrade, respectively. The coefficients of expansion of the samples of muscovite, ripidolite, and zinnwaldite micas are comparatively low. The maximum coefficient of expansion of the samples of these three types of mica is 0.000036 per degree centigrade.

Laue X-ray diffraction patterns of a few samples of micas (muscovite, phlogopite, and biotite), at room temperature and at elevated temperatures, are shown in figures 23 to 26. The transitions shown in the expansion curves of two phlogopite samples and a biotite sample at elevated temperatures appear to be related to the structural changes indicated in the X-ray diffraction patterns in figures 24 and 25. These changes may be ascribed to the buckling of very thin layers during heating, resulting in the displacement or tilting of the elementary crystals of the mica laminas.

The power factors of two samples of phlogopite mica from Madagascar and Mozambique were found to be considerably greater at 100 kc/s than at 1,000 kc/s. The power factors of these samples are considerably larger than the power factors of two samples of muscovite mica from Brazil and Guatemala. Heat treatment of the samples of phlogopite and muscovite micas to 600° C with or without a load, caused considerable increases in the power factors of the former and only slight changes in the latter.

The effects of heat treatments on the thickness, opacity, and color of 50 samples of various micas are indicated in table 6. Nearly all of the muscovite samples showed the greatest increases in thickness (up to 155 percent) after heat treatment at 800° C. The results on the changes in thickness of the phlogopite and muscovite samples indicate that the former resist elevated temperatures better than the latter. The large increases in thickness of nearly all of the muscovite samples accompanied changes from clear or translucent to opaque, or from polychrome to metallic color.

None of the species of mica can be considered as a substance or material of fixed and reproducible properties. There are wide variations in such properties as thermal expansion, power factor, and color. These properties of mica depend largely upon the chemical composition, the nature of the crystals, their magnitude and their orientation, the presence of impurities, the way in which these enter the structure, the heat treatment, etc.

Some of the phlogopite and biotite micas, which possess tremendously high thermal expansion in a direction perpendicular to the cleavage plane, may have decided advantages when used for high-expanding elements in temperature responsive devices.

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