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# THERMAL DECOMPOSITION OF TALC

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### ABSTRACT

A nearly pure talc was investigated both unheated and after heating at numerous temperatures ranging up to  $1,435^{\circ}$  C. The studies included the measurement of heat effects, weight losses, and changes in true specific gravity occurring on heating talc. X-ray and microscopical examinations were made of the heated samples.

There was no change in the crystal structure of the talc up to 800° C. At 800 to 840° C the talc decomposed to enstatite, amorphous silica, and water vapor. The enstatite gradually changed to clinoenstatite around 1,200° C, and the amorphous silica changed to cristobalite around 1,300° C, giving clinoenstatite and cristobalite as final products.

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# I. INTRODUCTION

Talc, both pure and combined with fluxing and with bonding agents, has been used for many years by ceramists in the manufacture of specialties. More recently, interest in talc as an ingredient of whiteware and of clay refractories, especially saggers, has resulted in technical studies of its potentialities.

Because of the promising results reported in recent literature and the probability of the increasing use of talc in the ceramic industry, it seemed desirable to have fundamental data on the process and products of its thermal decomposition. This report is the result of an investigation in which such data were obtained.

### II. MATERIALS

The talc, from Manchuria, has the following chemical composition: <sup>1</sup> MgO, 32.32 percent; SiO<sub>2</sub>, 61.34 percent; CaO, trace;  $R_2O_3$  (mostly  $Al_2O_3$ ), 0.71 percent; ignition loss, 5.81 percent. Petrographic examination shows at least 95 percent to have the correct indices of refrac-

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<sup>1</sup> Analyzed by A. S. Creamer and reported in the J. Am. Ceram. Soc. 18, 259 (1935). 22381-35-8

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tion for talc, as given by Larsen and Berman.<sup>2</sup> Whether the small amount of  $R_2O_3$  be ignored, or whether it be assumed to be present as  $Al_2O_3$  in kaolinite, the molecular ratio of MgO, SiO<sub>2</sub> and H<sub>2</sub>O is very close to 4:5:1.54. This molecular ratio is of interest because it should throw some light on the controversial question of the true formula of talc,<sup>3</sup> since this talc exceeds in purity any heretofore reported in the literature, as far as known to the authors.

Reference materials used in preparing comparison X-ray patterns included the magnesium silicates and several forms of silica. Of these, only the clinoenstatite (monoclinic MgO·SiO<sub>2</sub>), enstatite (orthorhombic  $MgO \cdot SiO_2$ ), and cristobalite (the high temperature form of  $SiO_2$ ) will be described. The clinoenstatite was a pure artificial product.<sup>4</sup> The enstatite was from Espedalen, Norway, and according to chemical analysis<sup>5</sup> contained 6.43 percent of FeO replacing MgO. Despite the impurity, the X-ray pattern is probably identical with that of pure enstatite since there is very little change in lattice dimensions in the enstatite-hypersthene solid-solution series. The cristobalite was an artificial product prepared by heating pure silica gel at 1.400° C.

### **III. METHODS AND RESULTS**

### 1. HEAT EFFECTS

Thermal effects were observed by the differential thermocouple method with  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> as the reference material. Readings of emf, generated by a couple composed of Pt and Pt-10-percent Rh wires, were taken at 1-minute intervals by means of a K-type potentiometer. The furnace temperature was varied at the practically constant rate of 6° C per minute.

One set of data is plotted in figure 1, the ordinates being the galvanometer deflections representing the temperature difference between the neutral body and the sample, and the abscissas being the temperatures of the sample. In a total of six such heating curves, the minima of the heat effects were reproduced within  $\pm 3^{\circ}$  C. The plot <sup>6</sup> shows two endothermic effects, a small one from about 530 to 572° C and a larger one from about 860 to 953° C. The reactions causing both heat effects were irreversible.

### 2. WEIGHT LOSSES

Weight losses were determined on 1- to 10-g samples heated in platinum containers at the temperatures, and for the intervals of time, given in table 1. The losses, calculated in terms of percentage of the original weight of the sample, are given in table 1 and plotted in Each value represents a single determination. figure 1.

 <sup>&</sup>lt;sup>2</sup> The Microscopic Determination of the Non-Opaque Minerals, U. S. Geological Survey Bul. 848 (1934).
 <sup>3</sup> N. H. and A. N. Winchell, Elements of Optical Mineralogy, part II (John Wiley & Sons, Inc., New

TABLE	1.—Relation	of	temperature	and	time	of	heat	treatment	to	weight	loss,	true
		spe	cific gravity	and .	crysta	l st	ructi	ire of talc				

Temperature of heating	Time of heating	Weight loss	True specific grav- ity $\frac{25^{\circ}}{25^{\circ}}$	Crystalline phases indicated by X-ray pattern		
°C	hr	Percent				
Unheated		1 01 00 000	2.78			
340 + 3	18	0.10	2.78			
$380 \pm 3$	22	. 18				
420 + 3	22	. 50				
$450 \pm 3$	22	. 84				
$480 \pm 3$	23	.94				
$520 \pm 10$	20	1.07	2.80			
772 + 3	16	1.34	2.81	Tale.		
782 + 3	20	1.40				
$795 \pm 2$	18	1.74	2.83	Do.		
$806 \pm 2$	17	2,45				
$816 \pm 2$	24	3.77				
$825 \pm 2$	18	4.58				
$840 \pm 2$	20	5.42	2,91	Enstatite.		
$900 \pm 2$	24	5.49				
$1.000 \pm 10$	17		2,94	Do.		
$1,200 \pm 10$	2	5.80	2.96	Enstatite, +clinoenstatite.		
$1.300 \pm 10$	4	5.80	2,96	Clinoenstatite, +cristobalite.		
1,435 (cone 17)	5	5.86	3.01	Do.		



 $\begin{array}{c} \textbf{Figure 1.} \\ -- Typical \ heating \ curve, \ and \ curves \ showing \ weight \ loss-temperature \ and \\ true \ specific \ gravity-temperature \ relations \ for \ a \ nearly \ pure \ talc \end{array}$ 

### 3. VOLUME CHANGES

The true specific gravity of the unheated and of heated samples was determined in general according to the American Ceramic Society standard method.<sup>7</sup> A 25-ml picnometer and 5-g sample of the talc were used, and the determinations were made at  $25 \pm 0.01^{\circ}$  C.

Since talc which has not been strongly ignited is very slowly wet by water, care must be taken to see that all of it is under the water after the boiling treatment. The results are given in table 1 and plotted in figure 1. Each value is the average of two determinations, which agreed within 0.002.

# 4. X-RAY AND MICROSCOPICAL EXAMINATIONS

Determinations of the crystalline compounds present in the heated samples were made by comparison of X-ray powder diffraction patterns. Copper K-radiation from a demountable ion-type tube and a camera of 4.1-cm radius were used. The camera was divided so that comparison patterns of the test and reference materials could be photographed on the same film. Supplemental examinations of the talc samples were made with the petrographic microscope.

The results are summarized in table 1. The presence of enstatite in the samples heated at 840 and  $1,000^{\circ}$  C was not considered certain because the patterns at these two temperatures were quite diffuse, and because the differences between the patterns of enstatite and clinoenstatite are few and unpronounced. The patterns of the material heated at these two temperatures were identical and differed definitely from the pattern of the material heated at  $1,300^{\circ}$  C. The evidence indicated that these differences correspond with the more pronounced differences between patterns of enstatite and clinoenstatite. Both the pattern of talc heated at 840 and  $1,000^{\circ}$  C, and the pattern of talc heated at  $1,300^{\circ}$  C, were definitely present in the pattern of the sample heated at  $1,200^{\circ}$  C.

The unheated talc and all samples heated below 800° were identical microscopically and had the following optical properties:

 $\alpha = 1.539 \pm 0.001; \beta = \gamma = 1.589 \pm 0.001; \gamma - \alpha = 0.050; (-) 2V = \text{practically zero; parallel extinction; positive elongation.}$ Heated at 840° C.—Plates and needles with parallel extinction the

Heated at  $840^{\circ}$  C.—Plates and needles with parallel extinction the same as the original tale, but the birefringence had decreased to about 0.010. Grains were no longer homogeneous, but the individual components were too finely divided to be distinguished. Gross indices of grains varied from 1.585 to 1.595.

Heated at 1,000° C.—Same as at 840°, but birefringence was reduced still further, and gross indices of grains varied from 1.590 to 1.600. Heated at 1,200° C.—Same as at 1,000°, but birefringence was re-

Heated at 1,200° C.—Same as at 1,000°, but birefringence was reduced still further, and gross indices of grains vary from 1.595 to 1.610.

Heated at  $1,300^{\circ}$  C.—Still some vestiges of the needle and plate structures of the original talc. Mosaic particles composed of weakly birefringent matrix material (clinoenstatite) of index about 1.65 and isotropic inclusions (amorphous silica) of index much lower than the matrix.

Heated to  $1,435^{\circ}$  C (cone 17).—Same as at  $1,300^{\circ}$  C, except that the crystals were a little larger and all trace of the talc structure had disappeared.

7 J. Am. Ceram. Soc. 11, 453 (1928).

### IV. DISCUSSION

The large endothermic heat effect at 860 to 953° C is obviously associated with the weight loss and increase in specific gravity between 800 and 840° C.<sup>8</sup> The X-ray patterns show that, below the temperature range of rapid loss of water, the material retains the same crystal structure as the unheated talc. They also show that the sample heated at 840° C, which has undergone the larger and comparatively rapid loss in weight, has changed to enstatite and (probably) amorphous silica according to the reaction: 4MgO.5SiO<sub>2</sub>·H<sub>2</sub>O  $\rightarrow 4(MgO \cdot SiO_2) + SiO_2 + H_2O.$ 

Enstatite appears to remain the only crystalline phase up to about 1,200° C, at which temperature a gradual inversion to clinoenstatite is evidently in progress. This is in agreement with the results of Bowen and Schairer,<sup>9 10</sup> who place the inversion point between enstatite and clinoenstatite at about 1,145° C, and state that the inversion attains a measurable rate at temperatures only a few degrees above 1.145° C.

Since cristobalite first appeared at 1,300° C, it is evident that the silica present in excess of the 1:1 ratio remained in the amorphous condition up to some point between 1,200 and 1,300° C. This is the general temperature range in which cristobalite first appears in heated kaolin and in heated silica gel and glass.<sup>11</sup>

When heated to high enough temperatures the final products of the decomposition of talc are therefore clinoenstatite and cristobalite.

The relatively small endothermic heat effect at 530 to 572° C is probably associated with the smaller loss in weight at 380 to  $450^{\circ}$  C. The cause of this heat effect is not readily apparent. The X-ray patterns show that no change in crystal structure has taken place. Therefore, the effect cannot be due to loss of water in the crystal lattice and adsorbed water would be given off more gradually and probably at a lower temperature.

The most plausible explanation is that advanced by Foshag and Wherry.<sup>12</sup> They observed that a certain talc containing 2 molecules of water lost about half of this water when heated "to a dull red heat" (probably about 600° C) without losing its optical homogeneity. The remaining water was lost at a much higher temperature and with loss of homogeneity. Reasoning from the low degree of hardness and platy character of talc, they assumed the water in excess of 1 molecule to be held electrostatically between basal cleavage planes. The temperature at which water held in this manner is lost should be intermediate between that of water bound in the crystal lattice and that of adsorbed water. Loss of such water should cause only a small change in volume and no change in crystal structure, and hence should not produce any marked changes in optical properties or density. Their hypothesis is strongly supported by the data in this paper which show particularly that (1) the change is accompanied by a small, but reproducible heat effect, and (2) that it is not accompanied by any change in crystal structure.

<sup>Heat effects associated with loss of chemically combined water usually appear in heating curves at temperatures about 100° C higher than the temperatures at which this water is lost under static conditions.
Am. J. Sci. 29, 151 (1935).
Krause states that clinoenstatite forms at about 900° C. Ber. deut. keram. Ges. 10, 94 (1929).
R. B. Sosman, The Properties of Silica, p. 97-9, 473-5 (The Chemical Catalog Co., New York, N. Y., 1927).
R. A. Heindl, W. L. Pendergast, and L. E. Mong, B. S. J. Research 8, 199 (1932) RP410. H. Insley and R. H. Ewell, J. Research NBS 14, 626 (1935) RP792.
Am. Mineral. 7, 167 (1922).</sup> 

According to Gruner's <sup>13</sup> recent work on the crystal structure of talc, the unit cell of talc contains 12 Mg, 16 Si and 48 O atoms, corresponding to a simplest formula 3 MgO·4SiO<sub>2</sub>·H<sub>2</sub>O. This would indicate that water in excess of one molecule has a random arrangement and does not diffract X-rays. While Gruner's result conflicts with the MgO·SiO<sub>2</sub> ratio of 4:5 found for the Manchurian talc, it does support the evidence in this paper that one molecule of water is bound in the lattice and behaves differently from water in excess of one molecule. However, support from Gruner's work loses some weight since he does not give an analysis of the talc he used.

# V. SUMMARY

A sample of a nearly pure talc was investigated both unheated and after heating at 18 temperatures from 340 to  $1,435^{\circ}$  C. The studies made included the determination of heat effects, weight losses, and changes in true specific gravity occurring on heating talc. X-ray and microscopical examinations were made of the heated samples.

Water in excess of 1 molecule was mostly driven off between 380 and 500° C. This water loss was accompanied by a small endothermic heat effect, but not by any change in crystal structure or optical properties.

The molecule of combined water was driven off between 800 and  $840^{\circ}$  C. This water loss was accompanied by a large endothermic heat effect and an increase in true specific gravity from 2.83 to 2.91, and by breakdown of the talc into enstatite and amorphous silica.

Inversion of the enstatite to clinoenstatite took place gradually, both phases being observed in material heated at  $1,200^{\circ}$  C, and only clinoenstatite in material heated at  $1,300^{\circ}$  C. The material heated at  $1,300^{\circ}$  C also showed conversion of the amorphous silica to cristobalite. Thus, the final products of the thermal decomposition of talc are clinoenstatite and cristobalite. There was a further gradual increase in the true specific gravity from 2.91 to 3.01 on heating from 840° up to the highest temperature,  $1,435^{\circ}$  C.

The data support the hypothesis of Foshag and Wherry that water in talc in excess of 1 molecule is not constitutional and may be held electrostatically between basal cleavage planes.

13 Z. Krist. 88, 412-9 (1934).

WASHINGTON, September 11, 1935.