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MICHIGAN ACADEMY OF SCIENCE ARTS AND LETTERS

VOLUME XXIV (1938)

CONTAINING PAPERS SUBMITTED AT THE ANNUAL MEETING IN 1938

VOLUME XXIV CONSISTS OF FOUR PARTS:

PART I: BOTANY AND FORESTRY

PART II: ZOÖLOGY

PART III: GEOGRAPHY

PART IV: GENERAL SECTION

Anthropology, Geology

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PAPERS OF THE MICHIGAN ACADEMY OF SCIENCE ARTS AND LETTERS

EDITORS

EUGENE S. McCARTNEY
UNIVERSITY OF MICHIGAN

ROBERT BURNETT HALL
UNIVERSITY OF MICHIGAN

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IN FOUR PARTS

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— Seneca, *Naturales Quaestiones*

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FURTHER TECHNOLOGICAL NOTES ON THE POTTERY OF THE YOUNGE SITE, LAPEER COUNTY, MICHIGAN

FREDERICK R. MATSON, JR.

I. INTRODUCTION

IN THE published technological study of the pottery found by Dr. E. F. Greenman during the excavation of the Younge site in Lapeer County, Michigan, there was only a brief discussion of the experiments in firing shrinkage (6, pp. 109–115). Sherds were refired at gradually increasing temperatures, the amount of linear shrinkage was measured, and the percentage of linear firing shrinkage (6, p. 111) was plotted against the temperature (6, Fig. 9, p. 112). A study of the average shrinkage curves and of the color changes in the clay at different temperatures indicated that the original firing temperature of the pottery might have been approximately 600° C. (6, p. 115).[1] Recently it was possible to restudy the firing data and to make a few further experiments, with the result that the conclusions concerning the firing temperature were somewhat altered. It seemed advisable, therefore, to prepare a brief report on the supplementary material.

The possibilities of error in the determination of the firing temperature by the shrinkage method have already been pointed out (6, pp. 109–110). The small size of the test pieces (2.5 × 1.5 × 1 cm.), which was necessitated by the type of dental porcelain kiln used, was perhaps an additional source of error, but the accuracy of the measurements may have largely compensated for the reduced size of the specimens. It is usually better to measure the volume firing shrinkage and then to calculate the linear shrinkage, but when one

[1] In 1907 the refiring shrinkage of sherds was used as an indicator of the temperature to which the ware was originally fired by H. Le Chatelier (5, p. 837) in the study of Greek pottery. Other investigators have also used this method, but the detailed results of their work have seldom been published.

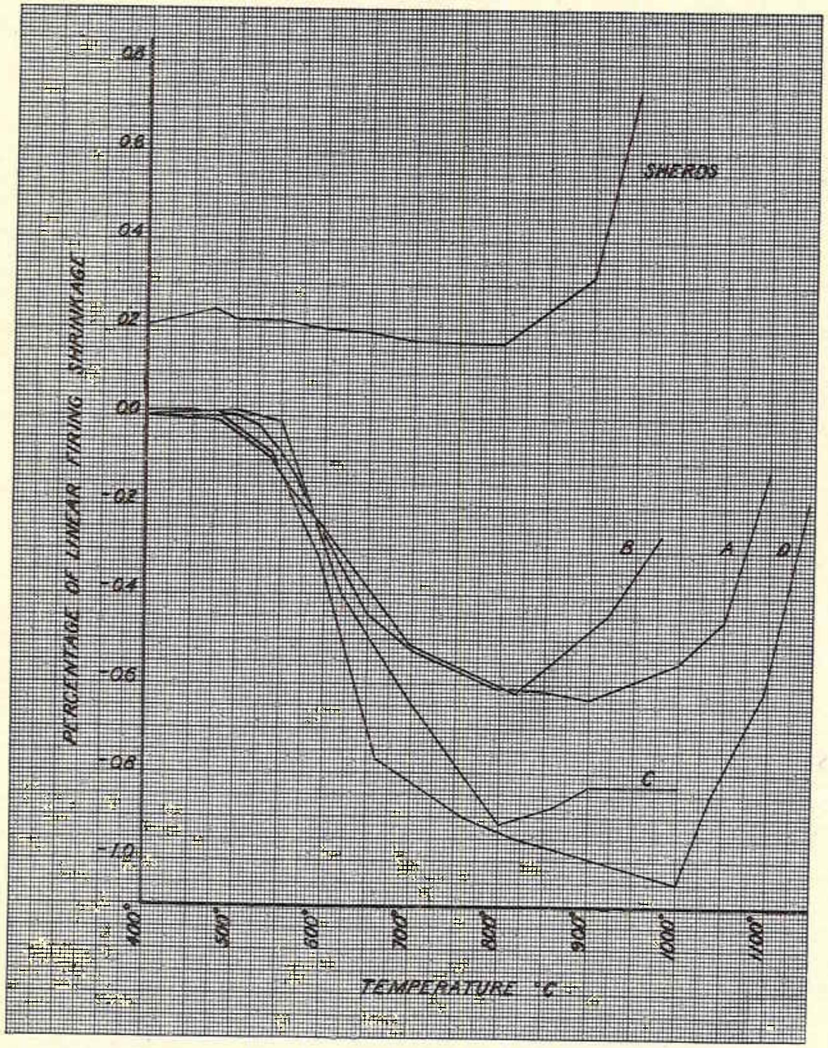

FIG. 1. Percentage of linear firing shrinkage of sherds and clay from the Younge site, Lapeer County, Michigan

Materials represented by curves *A–D*: *A*, natural clay; *B*, levigated clay; *C*, 75 per cent natural clay, 25 per cent crushed granite; *D*, 75 per cent levigated clay, 25 per cent crushed granite

is using small thin test pieces, accurate linear measurements are sufficient.

The shrinkage curves (Fig. 1) are plotted on the same scale as those previously published (6, Fig. 9, p. 112), but the present graph shows a different average shrinkage curve for the sherds and an improved plotting of the levigated clay curve (B). The sherd curve represents the average of fourteen pieces, all of which were fired to 950° C. Twelve other sherds, eight of which were used in the average curve IV of the earlier report, are not included. These twelve were fired only to 550° or 600° and were then discarded because of mechanical failures which made them unsuitable for further accurate measurements. Owing to marked individual shrinkage and expansion variations among them, they were not used for comparative studies with the briquettes. The number of briquettes averaged in each of the four groups is as follows: Six made of the natural clay (curve A) were fired to 1100° C.; four made of 75 per cent natural clay and 25 per cent crushed granite (curve C) were fired to 1000°; three of levigated clay (curve B) were fired to 980°; three of 75 per cent levigated clay and 25 per cent crushed granite (curve D) were fired to 1145°.

An examination of the curves shows that each falls into three phases: low-temperature shrinkage, expansion, and, finally, rapid shrinkage.

II. CHANGES DUE TO FIRING IN THE SIZE OF THE SHERDS AND THE CLAY BRIQUETTES

Shrinkage below 500° C.

The sherds began to shrink soon after the refiring had started and continued to do so until, on an average, 475° was reached; above this temperature expansion began (see Fig. 1). In this period there was a total shrinkage of 0.24 per cent, 0.03 per cent of which occurred between 400° and 475°. Only five of the fourteen sherds showed shrinkage above 400°.

Two of the six briquettes composed of natural clay shrank 0.01 per cent below 450°, and two of the four pieces compounded of 75 per cent natural clay and 25 per cent crushed granite (group C) maintained a slight shrinkage until 500°. The test pieces, both plain and tempered, made of levigated clay showed no initial firing shrinkage.

It is possible that if the briquettes in this temperature range had been fired more slowly, they might have had a greater shrinkage.

Three of the thirteen clays tested by Knote (4, p. 228) and three of the ten clays studied by Brown and Montgomery (2, p. 14) showed firing shrinkage below 450°. This was inferred from the increased true specific gravity reported for these six clays up to 450°; it indicated a diminution of volume because the mass remained constant. (In the C. G. S. system the specific gravity is the ratio of the mass to the volume.) Several of the British fire clays reported by Firth, Hodkin, and Turner also showed a slight shrinkage below 500° (3, p. 197).

It seems likely that the shrinkage is due to the dehydration of the clay, a process that is practically completed at 500°. Brown and Montgomery found that, when clays were heated to equilibrium conditions at increasing temperatures, the rate of loss of water was greatest between 400° and 500° (2, p. 22). Morgan concluded that under equilibrium conditions the true maximum rate of evolution of chemically combined water occurs in the range from 440° to 510°, but that the apparent temperature at which maximum evolution occurs may be raised as high as 650° by increased heating rates and greater size of specimens (7, p. 35). The small size of the test pieces probably neutralized to a large extent the effect of the rapid heating upon the dehydration temperature.

The shrinkage of the sherds may perhaps be attributed to the rehydration of the fired clay resulting from long burial in moist earth, as was suggested in an earlier report (6, p. 114). The rehydration of lightly fired clays is a slow and usually incomplete process, and may perhaps be accompanied by a slight increase in volume. The difference in the amount of shrinkage between the sherds and the briquettes (0.23 per cent) may be partly due to the fact that the briquettes were dried at a temperature of 150° before they were refired (some shrinkage may have occurred between 110° and 150°), whereas the sherds, unfortunately, were only air-dried.

Expansion between 450° and 900° C.

The refired sherds began to expand after 475° and continued to do so until about 800°, with a maximum average expansion of only 0.07 per cent and a maximum individual expansion of 0.26 per cent. All the briquettes began to expand between 450° and 500°, with the exception of those of group C, whose expansion started after 500°. The test pieces made of natural clay attained their maximum expansion at 900° and began gradually to shrink when fired above that temperature. The levigated clay reached its maximum expansion at 815°, after which it started to shrink quite rapidly. The greatest average expansion of the levigated pieces was 0.62 per cent, about the same as that for the natural clay. Curves C and D, representing the briquettes to which 25 per cent crushed granite had been added, agreed quite closely with A and B until they approached 600°. Above that, the tempered pieces began to expand more rapidly than did the briquettes of groups A and B, attaining a maximum expansion of 0.3 per cent and 0.45 per cent more than the untempered pieces.

The increased expansion of the tempered pieces is probably due to the effect upon the body of the alpha–beta quartz inversion which occurs at 573° C. At this temperature quartz experiences a sudden increase in volume. The reaction is completely reversible and there is no residual effect in the quartz itself after it has returned to the low temperature alpha state. As the speed of the inversion is high, the volume expansion, which amounts to 0.86 per cent according to Sosman (10, p. 364) (about 0.29 per cent linear expansion), causes stresses that leave their impression upon the clay matrix in the form of small cracks. Although the quartz returns to its original volume when cooled below 573°, the cracks in the clay do not disappear, but cause a permanent increase in the bulk volume of the briquettes. With each successive refiring the cracks are enlarged and there is an additional volume increase. This process continues until the clay has been fired to a temperature at which the body just begins to melt. The incipient vitrification makes the clay start to shrink toward its minimum volume, i.e. its maximum density. The higher the temperature within the vitrification range, the greater the rate of shrinkage.

The tempered briquettes made of levigated clay (group D) expanded more than did those in which the natural clay was used (group C). The fine sand occurring naturally in the clay probably offered resistance to the formation of cracks and prevented their rapid development and a corresponding increase in volume. The smaller amount of sand in the levigated pieces made it possible for the cracks to grow with less opposition and resulted in a greater maximum expansion than appeared in the natural clay. After the tempered briquettes had been fired to 900° cracks were very noticeable in those made of levigated clay but not in the ones compounded of the natural sandy clay. If volume expansion instead of linear expansion had been measured the apparent difference between the tempered and untempered groups might not have been so great, for the bulk volume effect of the cracks would have been eliminated. If the briquettes had been refired several times at temperatures between 700° and 800° further expansion would probably have occurred.

The difference in the expansion behavior of the sherds and of the briquettes can be attributed to the fact that the sherds had already been fired to a moderate temperature, probably many of them above 573°. It is likely that cracks were present in the sherds which absorbed the initial stress of the quartz inversion so that upon refiring further cracking was slight. Part of the friability of many rock-tempered sherds may be attributed to the formation of these expansion cracks.

In the discussion above only the effect of the quartz inversion has been mentioned. Since most granites such as the one used for the tempering material contain from 25 per cent to 30 per cent quartz a sufficient amount would be present to exert considerable expansive force upon the body. Several published studies show that a slight expansion of the clay when fired below the temperature of incipient vitrification is not unusual. All ten of the clays tested by Brown and Montgomery showed some expansion below 500°, as is indicated by the decrease in the specific gravity (2, p. 14). Between 500° and 550° a slight shrinkage occurred. Several of the British fire clays studied by Firth, Hodkin, and Turner began to expand by 200°. Their total expansion below 1000° varied from 0.04 per cent to 0.28 per cent (3, p. 197). These data show that transient expansion is due in part to the physical properties of the clay minerals themselves, for it often occurs below the quartz inversion temperature of 573°.

For a study of the effect of body thickness upon shrinkage, a series of briquettes that in the plastic state had thicknesses of 5, 8, 10, and 12 millimeters were prepared from the natural clay. These pieces were fired by stages to 1000°. There were no observable differences in their firing behavior which might be attributed to variations in thickness. Below 900° the maximum difference between these curves was about 0.10 per cent.

The expansion of some clays during a stage of their firing is an interesting phenomenon that has not been adequately studied as yet. Most investigations of uncalcined clays employ true specific gravity and loss of weight determinations. Shrinkage and expansion changes can be inferred from the specific gravity reports, but insufficient data are given to allow their calculation. A study of the clay expansion under better-controlled conditions than those possible in examining the Younge site material might provide a useful technological tool both in identifying clays with those used in the sherds and in determining the original firing temperatures.

Shrinkage above 800° C.

Most of the sherds and levigated clay briquettes began to shrink rapidly when fired above 800°; three sherds did not start shrinking until after 900°. The shrinkage of the natural clay began at 900°, proceeded gradually until 1050°, and then progressed more rapidly. The initial slowness was probably due to the fine sandy structure of the body, which may have been stiff enough partly to resist the early stages of the clay shrinkage. The rock-tempered natural clay shrank slightly between 800° and 900°, but then remained constant in size until 1000°. None of the samples of group C were fired above this temperature, but they doubtless would soon have begun to shrink rapidly. The rock-tempered levigated clay did not shrink until it was fired above 1000°, although the untempered levigated clay started at 815°. The reason for this discrepancy is not apparent, especially as the untempered levigated clay begins to shrink, as would be expected, before the natural clay does. It may be that the measurement of a larger number of samples would have given different results. The rate of shrinkage of the sherds and the briquettes is approximately the same, and they both begin to contract at about the same temperature; this indicates that the sherds had not been previously fired above 800° and that the clay used in the synthetic study is essentially the same as that employed by the Indians.

The shrinkage occurring above 800° or 900° indicates incipient vitrification and begins in the temperature range normal for most clays containing fluxing impurities; these cause the clays to start forming a glassy solution at moderately low temperatures. Ries and Kümmel found that all but one of the eight clays they tested showed a marked shrinkage between 900° and 1000° (8, p. 94). Several of the British fire clays tested by Firth, Hodkin, and Turner had their initial shrinkage between 750° and 900° C. (3, p. 197). Morgan has shown that the rate of heating has a very definite effect upon the temperature at which changes in the physical properties of clays may appear. He found that when a clay was heated to equilibrium a given loss might occur at a temperature as much as 180° lower than that at which the same loss was evident when the furnace temperature was increased at the rate of 260° per hour. Because of the very rapid rate at which the briquettes and the sherds were heated (to 500° in five minutes and to finishing temperature in fifteen minutes, the finishing temperature being maintained for fifteen minutes), it may be that the temperatures at which changes occurred were higher than they would have been if the clays had been fired more slowly, although the small size of the specimens partly counterbalanced the speed of the temperature rise. The rate of heating employed was similar to that of the San Ildefonso and Santa Clara II firings reported by Miss Shepard (9, pp. 455 and 457). With the exception of the Zia polychrome ware none of the modern southwestern pottery whose firing schedules have been published was kept at the maximum temperature for more than five minutes. Therefore it would seem that the maintenance of the maximum temperature for fifteen minutes in the present experiments was ample.

III. MAXIMUM FIRING TEMPERATURE

The purpose of this study of the refiring shrinkage of sherds and clays was to determine if possible the temperature at which the sherds had originally been fired. In the earlier report it was suggested that this temperature might have been about 600° C. (6, p. 115). A reexamination of the data indicates that some of the sherds may have been fired at a higher temperature. Bleininger and Brown say: "It must be realized that in burning clay products conditions of equilibrium are never reached. The contraction resulting at a certain finishing temperature is by no means constant, and upon reheating the product to the same temperature an additional shrinkage will be noted. The higher the temperature, however, the less will be the contraction upon reburning" (1, p. 42). The same statement would of course apply also to expansion during the lower stages of firing. The sherds, on an average, began to expand after 475°, that is, within the 450°–500° range in which the briquettes started. The expansion continued at a very moderate rate until 800°, after which rapid shrinkage began. Even though the sherds were originally fired to a temperature higher than 475°, they might have expanded further at that temperature, for the pottery was fired so rapidly that the shrinkage and expansion adjustments were difficult to complete in the body. This low-temperature expansion of the sherds seems to indicate that time rather than temperature may be the controlling factor. It is possible that the rehydration of the sherds is the cause of the expansion that occurs below the original firing temperature.

If it is assumed that the rate of heating in the kiln was equivalent to that used by the Indians, an estimate of the temperature to which the pottery was fired may be obtained by subtracting the percentage of maximum expansion of the averaged sherds from that of each of the four groups of briquettes and noting the temperature at the point on each curve that represents the resulting expansion figures for the briquettes. When this is done, the following values are obtained:

Natural clay..................	(A)	730°
Levigated clay................	(B)	735°
Tempered natural clay..........	(C)	775°
Tempered levigated clay........	(D)	875°

This could indicate that the sherds had a maximum firing temperature of about 750° C. Most of them were certainly fired below 800° because all except three began to shrink at that temperature and had shown a slight expansion below that point. There are so many conditioning factors affecting shrinkage and expansion that the subtraction method of temperature determination is of questionable validity.

IV. MINIMUM FIRING TEMPERATURE

In order to determine the minimum temperature to which the Younge site clay would have to be fired to lose its plasticity and its ability to slake down in water, some of the briquettes that had been used in the study of the depth of color penetration as related to time and temperature (6, pp. 118–119) were ground fine enough to pass through a 30-mesh screen, tempered with water, and reshaped. An unfired briquette was given the same treatment in order to serve as a standard for comparison.

The raw clay, after it had been pulverized in a mortar and pestle, felt like fine flour and absorbed water rather slowly. It formed a good plastic mass which, when dry, did not have a powdery surface and was not easily crushed.

The clay that had been fired between 400° and 500° for ten minutes was black, for the organic materials in the paste had been carbonized; it ground down to a fine powder that absorbed water more rapidly than did the raw clay. The amount of water that the carbonized clay could hold and still remain a plastic mass was much less than that taken up by the raw clay, and the body was of a short, mealy consistency. After the clay had dried it was quite weak and could easily be crumbled between one's fingers. Such behavior might be expected from a body that was only semiplastic.

The clay fired for fifteen minutes at 500° behaved very much like that just described.

After having been fired at 550° for fifteen minutes the clay showed a marked change in behavior. When powdered it was granular and absorbed water rapidly without attaining a plastic state. The water bound the grains together sufficiently so that a pellet could be formed. When dry the clay was very friable. The change in properties between 500° and 550° is doubtless due to the fact that the dehydration was completed in this range and the clay lost the remnants of its plastic qualities.

The clay fired for fifteen minutes at 600° was very sandy in texture when pulverized, soaked up water rapidly, and was quite difficult to shape into a pellet. When dry the surface of the briquette powdered at the slightest touch. The behavior of this piece was very similar to that of sand.

These results agree with those of Brown and Montgomery. In studying the residual plasticity of clays after they had been heated at increasingly higher temperatures they found that nine of the ten clays investigated showed a more or less pronounced loss of plasticity between 350° and 400°. "Beyond this point, the water required to produce a mass which can be moulded increases decidedly and may be equal to or greater than the weight contained in the natural plastic clay" (2, p. 20).

Other briquettes were soaked in water for several days to see how they would be affected by moisture. A pellet of unfired clay that contained 40 per cent crushed granite slaked down completely within two and one-half minutes. A raw, untempered piece rapidly developed cracks when placed in water and disintegrated in less than half an hour, but the cracked external shell remained standing throughout the four days' duration of the test. Three other specimens fired at (*a*) 500° for ten minutes, (*b*) 550° for ten minutes, and (*c*) 550° for fifteen minutes showed no signs of disintegration after having soaked in water for four days. The surfaces were possibly not quite so hard as those of the dry briquettes.

These experiments indicate that the Indians at the Younge site need not have fired their pottery at a temperature higher than 550° in order to make the clay lose its plastic properties and become resistant to the action of water. Even when burned to only 500° the body was not seriously affected by water. It is likely that little of the Indian pottery made in the eastern United States had to be fired, for structural reasons, at a temperature higher than 500°. Increased temperatures within the range attainable in an open fire would only result in the better development of the surface and body colors.

As has been previously stated (6, p. 115), colors found on the sherds could be matched by those obtained on pellets that were fired as high as 740° under oxidizing conditions. There was very little color difference noted between 600° and 740°. Pellets fired only to 500° could also be matched in color by sherds. Thus, in terms of color, there is a possible temperature range for the firing of the pottery from 500° to 740°.[2]

V. CONCLUSION

Because the degree of heat obtained in an open fire would have been far from uniform and because in successive firings the same temperature would not necessarily have been reached, it seems better to think of the firing temperature of the pottery in terms of a range rather than as any one fixed point. On the basis of the shrinkage, color, and plasticity studies, the range suggested for the Younge site pottery is 500° to 750° C.

The cultural significance of the determination of the firing temperature of pottery lies in the fact that such a determination gives

[2] The smoky open fires of the Indians would at times have had a partly reducing atmosphere, and the clear colors obtained under oxidizing conditions might not have had a chance to develop because of the incomplete oxidation of the chief coloring agent, the iron, and because of the presence of carbon. In such cases the resulting colors would tend toward the grays. Since many sherds were found that had a good buff to salmon surface color and since there is no evidence that any special attempt was made to procure a reducing atmosphere, it is permissible to base conclusions concerning the relationship of the firing temperature of the sherds and the colors that appear on them on a series of test pieces fired under oxidizing conditions.

archaeologists additional information about the technological abilities of the Indians of the region. Although the degree of heat attainable is conditioned by the type of fuel used, it also depends on the manner of firing. This may be expected to have varied with different types of pottery. Miss Shepard found that the firing temperature range of the southwestern potters whom she had studied was from 625° to 940° C. (9, p. 456). The determination of firing temperatures is a difficult and complex problem, but it is well worth investigating. It would be of interest in the study of American Indian cultures to know whether or not there has been any significant change in the firing techniques of the Southwest, for instance, from the earliest known pottery horizons to the present and whether or not the suggested 500°–750° temperature range at the Younge site can be considered representative of the technological cultures of the eastern United States. Knowledge of firing temperatures is also of interest in connection with metalworking. A metallographic study of copper implements showed that a Wisconsin spearhead was heated to about 800°, and that a Wisconsin arrow point and an Ohio axe were worked at about 500° (11, pp. 111–112). After further metallographic and ceramic studies have been made it may be possible to demonstrate regional technological differences that will be of cultural value.

University of Michigan

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