

ARCHAEOLOGISTS STUDY THERMAL SHOCK

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ABSTRACT

Archaeologists have studied ancient ceramics since the beginning of the discipline. A recent awakening of interest in ceramic technology has led some archaeologists to the study of vessel function and durability. A key approach in these studies has been thermal shock analysis. This paper reviews archaeological studies of thermal shock and considers the pitfalls and promises of a variety of techniques in the study of ancient ceramics.

INTRODUCTION

Pots, pots everywhere—their broken remains are one of the most widespread traces of human occupation. Archaeologists have long been aware of the utility of ceramic studies in the reconstruction of past human behavior. However, most ceramic studies have concentrated on the construction of descriptive typologies in order to establish temporal frameworks and cultural boundaries within a cultural–historical framework. For such purposes, stylistic attributes have often been assumed most sensitive to temporal and social variability and the most easily analyzed (but see Plog [1] for a critique of the assumptions inherent in this approach). As a result, stylistic studies have become an essential part of archaeological research.

However, archaeologists have also been interested in the functions of the ceramic vessels they excavate and the technology that produced them, an approach Braun [2] has called the study of pots as tools. The most common approach to the study of ceramic function and technology has been through ethnographic reviews of vessel form and production in order to derive functional correlations for further archaeological testing (e.g., refs. 3 and 4). At best, these measures are abstractions one step removed from the actual production and use of a pot. Dissatisfaction with this approach has led to a growing interest in materials science analytic approaches in the archaeological study of ceramic manufacture and use. This paper will present a number of basic materials science concepts and analytic approaches in the study of thermal shock and review their use in archaeological studies of ceramics.

THERMAL SHOCK RESISTANCE AND TESTING PROCEDURES

Thermal shock failure is a function of thermal stress, defined as stress arising from a temperature gradient; thermal stress resistance is resistance to weakening or fracture from thermal shock (ref. 5, p. 3). Thermal shock in turn refers to a sudden transient thermal change; thermal shock resistance is that property of a body that enables it to withstand such change without fracturing (ref. 5, p. 3; ref. 6, p. 44). Thermal shock failure is a primary failure mode in ceramics. As a result there is a considerable body of literature devoted to thermal shock and fracture (e.g., refs. 7–11). Non-industrial potters are also aware of the problems of thermal shock, particularly where repeated heating and cooling cycles occur in the cooking of starchy stews and gruels (see reviews in refs. 2, 12, 13 for archaeological examples, and ref. 3 for a recent ethnographic review).

In contrast to a relative lack of analytic basis for mechanical impact testing, thermal shock testing of ceramics is based on better analytical treatments [13,14]. Briefly, resistance to thermal shock is governed by the differential thermal expansion of materials. When a vessel is heated rapidly, the exterior bottom surface expands more rapidly than the interior. As a result, the exterior is subject to compressive stresses while the interior is under tension stress; on cooling, the reverse occurs. When these stresses exceed the strength of the material, thermal shock failure occurs (ref. 15; ref. 10, p. 417). The usual form of failure is spalling, the breaking away of pieces of a shape or structure (ref. 5, p. 3). Spalling is an excellent example of crack propagation in the real world (ref. 5, p. 8).

Thermal shock resistance is conditioned by many factors. Size and shape have been the most obvious to archaeologists as well as to craft potters and industrial ceramists. The greater the size of the vessel, the greater the nondimensional (nonlinear) heat-transfer properties (ref. 5, pp. 10–12; ref. 16). This can be minimized by thinner vessel walls [17,18] and more spherical shapes (ref. 19, p. 448; ref. 6, p. 44), a phenomenon which has been recognized by ethnographically studied potters and by archaeologists interested in ceramic function as well (e.g., refs. 2–4, 20). However, most archaeological studies of the relationship between vessel shape and wall thickness, on the one hand, and thermal shock resistance properties, on the other, have generally assumed that the relationship holds for the ceramic under study, rather than tested it in any empirical fashion. Such untested assumptions underly much of the debate about proposed functions of archaeological vessels. For example, Raab's study [21,22] of Hohokam ceramic function was based on the assumption that porous schist-tempered ceramics served for storage and cooking. Doyel's rebuttal [23] reviewed other archaeological studies, noting that a variety of vessel size and shape classes were characterized by such temper, suggesting a lack of functional specificity for temper materials (ref. 23, p. 43). Neither attempted to independently

assess the thermal properties of the ceramics or link them with other observations of function, such as the presence or absence of sooting. As a result, the literature abounds with calls for more studies to resolve the confusion [24].

Other factors which affect thermal shock resistance can be grouped under the heading of heat flow properties. These include specific heat, and the coefficient of thermal expansion (ref. 8, p. 156). Specific heat is the amount of heat energy that must be absorbed by a unit weight of material to raise its temperature one degree [25]. The two standard heat quantities in use are the British Thermal Unit (BTU) and the calorie; both are measured in relation to the specific heat of water. The BTU is the amount of heat required to raise one pound of water 1°F; the calorie is the amount of heat needed to raise one gram of water 1°C. The units are described in BTU lb-°F or cal g⁻¹°C⁻¹ [25]. In turn, heat capacity is the total amount of heat needed to raise the specimen temperature one degree. The relationship between the two is shown by [25].

Heat capacity of specimen = (specific heat) (weight of specimen)

Specific heat is usually measured with a calorimeter which uses a known amount of water to absorb heat from a heated specimen placed in it [25]. Heat capacity is measured by noting the temperature rise in a specimen of known weight. This temperature rise is then compared with the temperature rise of a sample of known specific heat and weight (e.g., water) heated at an identical thermal energy input (ref. 8, p. 157).

Thermal conductivity refers to the flow of heat across a material by conduction. It is measured by determining the rate of heat flow and temperature drop across a material heated from one side. It is calculated according to the equation

$$k = \frac{Qx}{A(T_2 - T_1)}$$

where k = thermal conductivity (in BTU = in/°F-ft²-h), Q = heat flow through specimen in BTU/h, x = thickness of specimen in inches, A = area of one side of specimen in ft², T_2 = temperature of hot surface in °F and T_1 = temperature of cold surface in °F [8]. Generally, materials with high heat capacities have low diffusivity values, that is, they heat at a relatively slow rate; the reverse holds true as well [25]. Such measurements have considerable archaeological potential in the study of ceramic function but have not been used to date, perhaps due to the unfamiliarity of most archaeologists with the physics of materials.

Ceramic materials expand upon heating and contract when cooled. The amount of expansion depends on the original length of the specimen, the change in temperature and the coefficient of thermal expansion (ref. 8, p.

158). This coefficient is a mean measure of change in length which occurs between room temperature and some higher temperature; as a result the coefficient depends heavily on the upper temperature value [26]. In general, the volume thermal expansion coefficient of a material is approximately three times that of the linear thermal expansion coefficient [26]. Both kinds of thermal expansion coefficients are calculated by measuring changes in length and volume as a function of temperature. The standard methods of making these measurements include interferometry (American Society for Testing and Materials Standard Method of Test C539-66 and C327-56) and dilatometry (American Society for Testing and Materials Standard Methods of Test C 372-56).

Thermal shock resistance of materials is not assigned a numerical value; instead different materials are compared in their resistance to shock (ref. 8, p. 159). In general, the strength of any solid material is an inverse function of crack length; the longer the crack, the weaker the material (ref. 27, p. 95). For two different materials, the initially stronger one will require a greater temperature change to initiate fracture. However, the mechanics of crack propagation are such that after fracture, the originally weaker material will be stronger; because more energy is required to initiate cracking in the stronger material, the cracks that do occur are longer and more severe, much like the "marvellous one-horse shay" of Oliver Wendell Holmes poem (ref. 27, p. 95; ref. 28, p. 65). The nature of strength degradation is dependent on the initial crack length; when the initial crack is short, cracks are propagated kinetically and strength decreases discontinuously on quenching (ref. 29, p. 250). When the initial cracks are long, there is a gradual decrease in strength with quench temperature. Gupta's review indicated that strength after thermal shock and the extent of crack propagation varied inversely and directly, respectively, with the initial strength of the material [29].

In commercial ceramics, selection of materials for minimum changes due to thermal shock is based on minimizing the extent of crack propagation, rather than avoiding the initiation of fracture from thermal stress (ref. 27, p. 90). In fact, materials with high densities of micro-cracks are generally highly resistant to thermal stress (ref. 30, p. 28). Crack propagation can be minimized by selecting materials with high values of Young's modulus and low values of tensile strength; in contrast, initial cracking from thermal strength can be minimized by selection of materials with high tensile strength, high thermal conductivity values and low values of Young's modulus (ref. 31, p. 1036). Since a material cannot be simultaneously high and low in tensile strength, these two approaches conflict and some sort of compromise is required in the production process. The compromises faced by the materials scientist are similar to the compromises in materials and techniques made by the potter. For example, as discussed earlier, the use of larger temper particles makes clay more adhesive so that molded clays keep their shapes more easily; at the same time, the very increases in adhesiveness hinder the

use of the clay with a wheel (ref. 32, p. 75). In another example, the more sophisticated the technique of the potter the larger the output but the more selective the requirements become for the raw materials (ref. 33, p. 393).

Both mechanical and non-destructive methods have been used to study thermal shock. These methods have been presented elsewhere (e.g., refs. 6, 7, 14, 34). However, these methods are at best one step away from the actual study of durability due to the indirectness necessitated by their non-destructive nature. The most direct tests, as indicated earlier, are simulated service tests (ref. 5, p. 14; ref. 35, p. 16). In archaeological research, simulated service tests have the advantage that testing measures can be tailored to examine specific presumed functions of materials and vessels.

The experimental method most frequently used to determine the thermal shock resistance of ceramics is the use of the quenching technique to determine the critical temperature differential (Δt_c) at which fracture occurs (ref. 13, p. 255). The usual method is subjection of test specimens to a series of controlled temperature changes such as from boiling water to ice water, until failure occurs; the number of cycles endured before failure is a measure of the material's thermal shock resistance (ref. 36, p. 162; see also review in ref. 10). Of especial importance in the determination of thermal shock values are the thermal conductivity of the material(s) and the temperature differential between the solid body and the cooling medium (ref. 35, p. 16). The drastic temperature changes employed in commercial testing are generally much greater, particularly in terms of the heating temperatures, than conditions of aboriginal use (e.g., the Δt_c of 1000°C used to test single crystal, "perfect" surface materials [37] versus boiling and cooling cornmeal mush). However, the temperature differential of the water quench affects only the rate of failure rather than the thermal shock values themselves; as the differential increases, materials fail sooner but as long as all materials are tested with a constant differential, thermal shock comparability is maintained (see review in ref. 38). The cyclical testing process involved further accentuates the results of the testing procedure (ref. 5, p. 14).

ARCHAEOLOGICAL STUDIES OF THERMAL SHOCK RESISTANCE

Thermal shock resistance has attracted the most archaeological attention, and several different approaches have been employed. Braun [2] linked changes toward thinner vessel walls and finer temper particles in Woodland ceramics from the lower Illinois River valley with shifts to maize-based subsistence to postulate a technological change in which increased thermal shock resistance of ceramic vessels was required by the processing demands of maize-based gruels and stews. The inferred technological shift is supported by evidence of vessel shape and a detailed time-series chronometric model. However, no attempt was made to assess the actual thermal shock resistance of the ceramics studied.

In contrast, Steponaitis [39] used Hasselman's [14] theory of thermal fracture in ceramics to arrive at two measures of thermal shock resistance of relevance to the prehistoric ceramics of Moundville, Alabama. The first was the severity of shock in terms of differential temperature gradient required to initiate cracking; the second was the amount of strength degradation that occurs when this is reached (ref. 39, p. 38). In his analysis, high thermal shock resistance was indicated by high resistance to initial cracking or minimal strength degradation upon cracking [39]. Although he realized the ideal approach would be to determine these values empirically from a number of ceramic specimens, he was constrained by limited sample and specimen size. As a consequence, he attempted to measure a number of physical properties affecting thermal shock resistance. These were then used to calculate a series of thermal shock resistance parameters in order to compare the paste compositions of the two ceramic-ware classes under study (ref. 39, p. 39). The relevant properties chosen for study were porosity, thermal diffusivity, elasticity and tensile strength [39].

His measurement of thermal diffusivity employed an innovative custom-built device designed by Alan Franklin and C.K. Chaing of the National Bureau of Standards. It is fully described in ref. 39, p. 299 and hence will only be briefly described herein. Basically it consisted of a soldering iron to provide a heat source on one side of the sample and thermocouple to measure changes on the unheated side of the sample. An x - y recorder produced a graph of temperature change versus time from which diffusivity was calculated according to the formula

$$D = \frac{d^2}{t}$$

where D = diffusivity, d = distance between the heat source and thermocouple (i.e., sherd thickness in mm), and t = delay time, the interval between the contact of the soldering iron at the upper surface and the abrupt increase in temperature at the lower surface [39]. However, in practice, the increase in temperature was often gradual, making it difficult to determine the precise point at which the temperature increase began. This was overcome by application of a square root transformation to the x -axis (time, replotting of the curve, and extrapolation of the linear portion of the resulting curve to the point of junction with the x -axis). This was used as the terminus of the delay time [39].

The thermal diffusivity values were plotted against percentage of visible shell temper but no clear relationship was detected (ref. 39, p. 40). However, the three properties, thermal diffusivity, elasticity and tensile strength, were used to estimate thermal shock resistance in the following formula (ref. 39, p. 41; derived from ref. 40, pp. 6, 7)

$$R = \frac{S(1 - \nu)D}{\alpha E}$$

where R = thermal shock resistance, S = tensile strength, ν = Poisson's ratio, α = thermal expansion coefficient and E = elasticity (Young's modulus). Poisson's ratio refers to the ratio of thickness decrease to the length increase of a material when a tensile load or stress is applied [4]. Steponaitis did not measure Poisson's ratio or the thermal expansion coefficient for the ceramics he analyzed, assuming instead that both were constant for all the specimens, given their mineralogical similarity and the fact that the calcite temper has approximately the same thermal expansion characteristics as low-fired clay (ref. 39, p. 41; after ref. 42, p. 117). As a result the thermal shock parameter reduced to

$$R = \frac{SD}{E}$$

and Steponaitis (ref. 39, p. 42) reasoned that the greater the value of this parameter, the greater the temperature differential that could be endured before the onset of strength degradation or fracture [31].

In contrast, once cracking has initiated, specimen resistance to strength loss was held to be proportional to

$$R' = \frac{GE}{S^2(1 - \nu)}$$

where G was a measure of the surface fracture energy. Surface energy refers to the energy associated with the formation of new surfaces (ref. 43, p. 473). Surface fracture energy represents the energy needed to break the atomic bonds of atoms lying in a fracture plane of a material. The surface fracture energy most familiar to non-physicists is the surface tension of liquids (ref. 44, p. 122). An insect, such as a water-strider, is able to walk on the surface of the water because its kinetic energy is less than the surface energy of the water. Measurement of surface fracture energy is a complex process, generally conducted on very thin specimens (fibers and whiskers) carefully prepared and handled before testing (ref. 37, pp. 45–48; ref. 44, pp. 122, 123; ref. 43; ref. 45). In any case, specimen size limitations precluded measurement of this parameter in the Moundville specimens and it was treated as a constant, reducing the thermal shock parameter to

$$R' = \frac{E}{S^2}$$

In general, the greater the R' value, the less strength lost when Δt is reached (ref. 39, p. 42; after Hasselman [31]). The values of the R and R' parameters were plotted against the volume percentage of shell for the Moundville ceramics analyzed (ref. 39, p. 42, Fig. 12, Fig. 13). This indicated there was no significant difference between coarse and fine tempered wares in the level of thermal shock needed to cause initial failure (ref. 39, p. 42). In contrast, the R' parameter was much more strongly correlated with the percentage of

shell, indicating that coarse wares would tend to lose proportionally less strength once cracking had been initiated [39]. Further analysis of the R and R' parameters indicated that the drop in strength, or amount of strength degradation, was positively related to initial strength (ref. 39, p. 43, and Fig. 14).

Steponaitis concluded this segment of analysis with a brief discussion of the compromises made by the potter, a discussion similar to that already reviewed herein by van der Leeuw [33] and Kalsbeek [32]. In particular, Steponaitis (ref. 39, pp. 43, 44) noted that the use of finely ground temper would produce a vessel with high initial strength but one which would lose a great deal of this strength upon subjection to thermal shock. Coarsely tempered vessels, on the other hand, would have less initial strength but would retain more of it after thermal shocking.

Similar conclusions were reached in Bronitsky's analysis [46,47] of the relationship between temper type, grade and amount and thermal shock resistance. Here he constructed a set of briquettes identical to those discussed earlier in the impact resistance analysis. Five briquettes from each temper/grade/amount set were immersed in boiling water for 5 min and then immediately placed in ice water. Each immersion in hot water and subsequent ice water quench was considered one cycle and the number of cycles required to cause failure was recorded. Those briquettes that survived 40 cycles of quench testing were then impact tested to examine the strength degradation due to thermal shock (ref. 47, pp. 5, 6).

Some sets of briquettes had to be omitted from the impact testing portion of the thermal shock study due to failure of all briquettes within those sets during the quench testing cycles. Sets omitted for this reason were coarse sand (both 40 and 80% temper by weight), coarse unburned shell (both 40 and 80% by weight) and the untempered control briquettes fired at 600 and 1000°C (ref. 47, p. 9). These failures immediately showed that fine temper was more resistant to thermal shock than coarse temper in all cases since four of the six coarse temper groups did not survive to provide data for analysis.

The remainder of the briquettes were then impact tested. Both the amount of energy needed to induce initial cracking and final failure were observed in order to provide a data set comparable to the initial impact testing set so that degradation of strength could be assessed. Strength degradation refers to the amount of strength lost during the course of thermal shocking and is reflected in lower mean initial cracking and final failure values (see Braun's concept of performance, ref. 2, p. 110). The data showed that briquettes with burned shell temper suffered considerable strength degradation for both fine and coarse temper grades, much more so than either sand or unburned shell temper (ref. 47, Table 10). The initial cracking values for fine burned shell temper were about the same as the other temper types, due to its greater strength degradation, in contrast to its much higher values in the non-ther-

mal shocked impact-resisting portion of the study discussed earlier. Similarly, final failure values for briquettes with fine burned shell temper were about the same as fine sand temper, and lower than fine unburned shell temper. Briquettes with coarse burned shell temper also underwent considerable strength loss but their much higher strength values before thermal shocking, as shown in the previous impact resistance study, resulted in briquette survival during the immersion cycles, in contrast to the other coarse temper types (ref. 47, p. 11).

Bronitsky concluded his analysis with a discussion of the physical and chemical factors responsible for the differential performance of the briquette sets during the impact resistance and thermal shock resistance testing program (ref. 47, pp. 11–14). In part, the performance followed a general ceramic principle that specimen strength increases with decreasing particle size, although the reasons for this are unclear at present [48]. In addition, the performance may be due to greater irregularity in the shell temper as compared to the riverine sand, creating a greater number of volume crack sources which would distribute stresses more evenly [49] and creating a better bone with the clay [50]. In addition, the superior performance of the burned shell tempered briquettes may be due to the increased irregularity resulting from the friability induced by burning, as well as reducing the likelihood of spalling from calcite decomposition and hydration of calcium carbonate which occurs at temperatures above 800°C (ref. 47, p. 12; see also ref. 39, pp. 4–6; ref. 51). At the same time, burning the shell before use may render its rate of thermal expansion similar to that of the clay, reducing risk from inhomogeneous expansion during firing. Unfortunately, the study did not use any techniques to measure the regularity of the particles such as thin-section studies of briquettes, rendering the discussion informed speculation at best.

Nonetheless, the similarity of results between the Steponaitis and Bronitsky analyses suggests that temper characteristics can play a significant role in ceramic durability. The similarity also indicates a measure of validity for Steponaitis' indirect estimation of parameters (ref. 52, p. 450). Braun's study further points out the role of temper and wall thickness, in particular, in performance. Unfortunately, what is lacking is a serious attempt to integrate results of laboratory analysis with analysis of a statistically meaningful sample of archaeological ceramics. This has largely been due to the destructive nature of the tests. One approach which has been suggested to remedy this has been the construction of a large series of briquettes approximating the range of temper and clay characteristics of the archaeological ceramics to be studied [53]. This would permit the testing of as many briquettes as needed for whatever physical parameters are required. As a result, the performance of a small archaeological sample of ceramics would be more accurately related to the total range of variability in the briquettes, permitting a clearer understanding of the test results from archaeological ceramics.

However, this approach has only been suggested and has not yet been empirically tested.

The ultimate result of this research will be a clearer understanding of the problems faced by the ancient potter. As we begin to be aware of the compromises required by functional needs and material availability, we can approach a true evaluation of ceramic technological change, firmly grounded in the ceramic ecology.

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