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Materials analysis of the masonry of the Hagia Sophia Basilica, Istanbul

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ABSTRACT

As part of an integrated structural investigation of the 1500 year-old Hagia Sophia Basilica in Istanbul, specimens of its historic brick and mortar are being analyzed. The materials analysis uses a suite of instrumental methods including neutron and X-ray diffraction, small angle neutron scattering, thermal analysis, and automated image analysis of SEM polished sections. Data from wet chemistry analyses have also been obtained. The results show that the mortar is pozzolanic rather than a traditional carbonated lime mortar as initially assumed. The pozzolana is ground up brick dust. Large chunks of brick are also found as coarse aggregate in the mortar joints. Comparison with modern lime-pozzolana mortars suggest that the Hagia Sophia could have tensile strengths on the order of 3.5 kPa. However, unlike Portland cement concrete, such pozzolanic mortars take longer to reach full strength. This could explain some structural problems such as the significant plastic deformations of the main piers.

INTRODUCTION

The significance of the Hagia Sophia in architectural history, and the current structural investigation are described in several other papers in these proceedings. The subject of this paper is the materials analysis of the mortar. This analysis was undertaken because of anomalous results produced by the initial finite element model of the Hagia Sophia's structure.

For this model it was initially assumed that the mortar was simply a traditional carbonated lime-sand mixture. A nominal value for modulus of elasticity of $5x10^9$ Pa provided good agreement for the dynamic model in the sense that it predicted natural frequencies of vibration that were very close to measured ones. However, this value proved unsatisfactory for the static version of the model, since the calculated elastic deformations were nearly an order of magnitude lower than those actually measured. Moreover, the mortar joints would experience significant bending stresses that would at some points exceed the tensile strength assumed for the lime mortar (3.4×10^5 Pa). Therefore, this set of assumed mortar properties led to the false conclusion that the Hagia Sophia should have collapsed long ago.

At this stage, it was decided to learn more about the actual materials used in the construction of the Hagia Sophia. This research was conducted through a collaboration between the Princeton University team and researchers in the Building Materials Division of the National Institute of Standards and Technology (NIST). The main objective of the mortar investigation has been the determination of the composition of the material, from which the strength properties could be inferred. However, in the course of this study a number of other issues have come up concerning masonry technology in the early Byzantine era.

MORTAR ANALYSIS

Specimens of historic brick and mortar from the Hagia Sophia were analyzed for chemical composition and physical properties using a suite of instrumental methods including neutron and X-ray diffraction, small angle neutron scattering, thermal analysis, and automated image analysis of SEM polished sections. These analyses are described in more detail in Livingston et al. [1].

Preliminary visual inspection of the mortar in situ showed a characteristic pink color, suggesting that ground up brick dust had been added. Moreover, the joints themselves were very wide, on the order of 5-7 cm. comparable to the thickness of the brick units themselves. Within the mortar, chunks of brick up to a size of 1 cm were also found.

The conventional method for mineralogical analysis is X-ray diffraction. Figure 1 is an X-ray diffraction pattern of a typical Hagia Sophia mortar. The prominent peaks in this diffraction pattern are mainly associated with quartz and calcite, as would be expected in a traditional carbonated lime mortar. No peaks for $Ca(OH)_2$ were found, implying that the mortar was fully carbonated. Feldspar peaks also were found, presumably associated with the the brick fragments. In addition, there is a peak at 11.7° (0.756 nm), which is the single prominent peak for gypsum.

Figure 2 shows a different diffraction pattern of the same sample, made by thermal neutron diffraction rather than by X-rays. Neutron scattering cross-sections for various elements differ widely from those for X-rays. In particular, hydrogen has an extremely large scattering cross section. Hence,

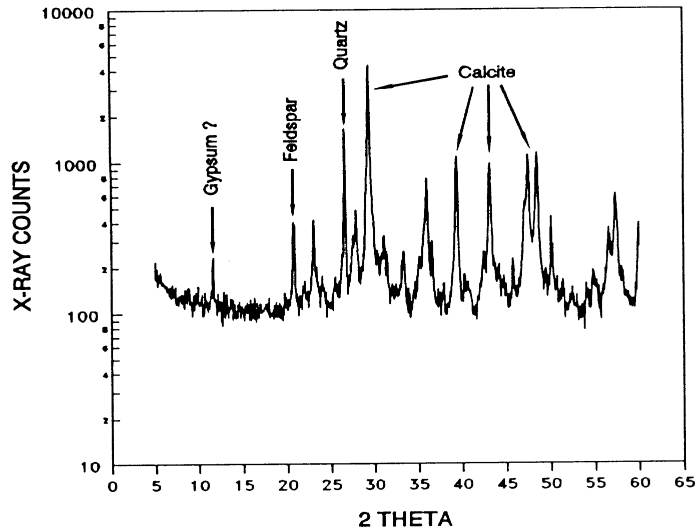

Figure 1: X-ray Diffraction Pattern of Hagia Sophia Mortar Sample

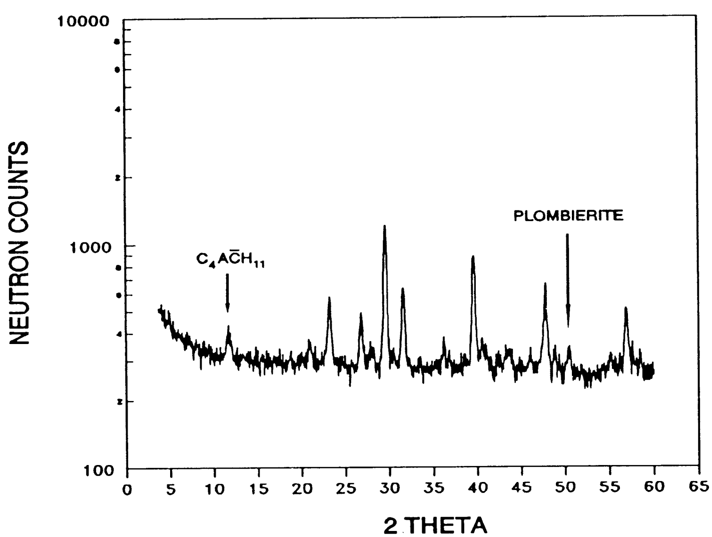

Figure 2: Neutron Diffraction Pattern of Hagia Sophia Mortar.

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neutron diffraction is useful for studying hydrous minerals like those found in cementitious materials. Moreover, the differences in scattering cross-sections means that for a given mineral, the relative intensities of peaks are often very different between the two methods. In fact, prominent peaks in the neutron pattern may not be seen in the X-ray pattern for the same mineral and vice versa. Thus combined X-ray and neutron diffraction analysis can be more effective for identification of minerals than either method alone.

In this case, the peak at 11.7° tentatively identified as gypsum in the X-ray diffraction pattern also appears in the neutron diffraction pattern. However, unlike the standard X-ray diffraction pattern for gypsum which has only this single prominent peak, the standard neutron diffraction pattern has at least three prominent peaks. These additional peaks were not seen in the Hagia Sophia diffraction pattern. On this basis, the presence of gypsum is ruled out. Instead, the peak appears to be due to tetracalcium aluminum carbonate hydrate, $Ca_4Al_2CO_3(OH)_{12}\cdot 5H_2O$, or $C_4A\overline{C}H_{11}$ in cement chemistry notation. The proportion of water of hydration is variable [2]. This mineral is typically found in cementitious materials, especially those in concretes made with limestone aggregate.

In Fig. 2, a peak for plombierite, a naturally occurring form of C-S-H gel [3], is also indicated. C-S-H is the main hydrated constituent in concrete made with Portland cement. However, the characteristic 1.4 nm peak found in C-S-H formed from Portland cement is missing in this case. Plombierite is found in nature where basaltic rock has come into contact with limestone. A similar reaction may occur in this mortar when the lime reacts with the glassy phase in the brick, which has a chemical composition very similar to some basalts [4]. The possible presence of both $C_4A\overline{C}H_{11}$ and plombierite is clear evidence of a cementitious rather than pure lime mortar.

The Hagia Sophia mortar sample was also analyzed by thermogravimetric analysis (TGA), which consists of measuring the weight loss of the sample as a function of temperature. The plot of the thermogravimetric data is presented in Fig. 3.

Three major segments of the curve can be identified. The steep initial weight loss around 200°C is associated with dehydration, presumably of the $C_4A\overline{C}H_{11}$. The more gradual weight loss between 300°C and 700°C is typical of the loss of structural water in C-S-H type minerals. Finally, the steeper weight loss above 700°C is characteristic of the loss of CO_2 from the $CaCO_3$ and $C_4A\overline{C}H_{11}$ phases. The shape of the TGA curve matches those for known mixtures of C-S-H and $CaCO_3$ [5].

Moreover, no break was observed in the curve at 500°C, which would have indicated the presence of $Ca(OH)_2$. This thermal analysis thus reinforces

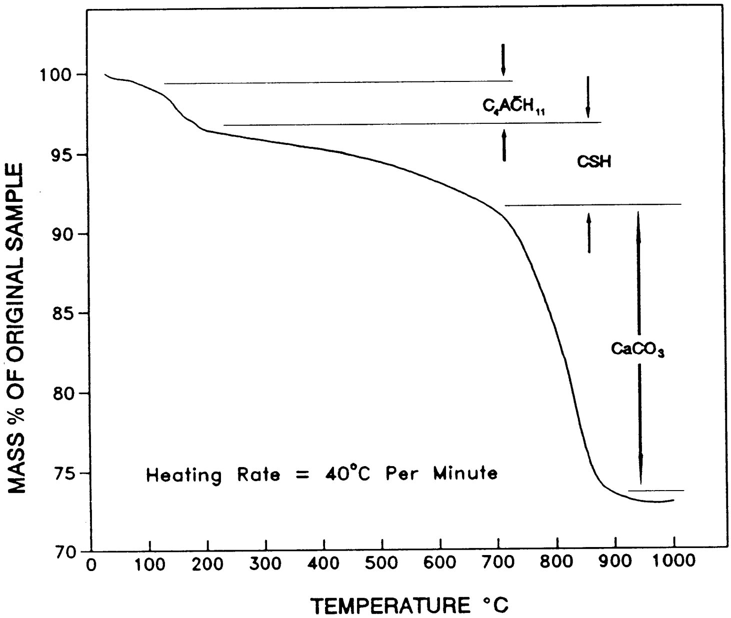

Figure 3: Thermogravimetric Analysis of Hagia Sophia Mortar.

the results of the diffraction analysis that the original lime in the mortar has fully carbonated. The amount of CO_2 evolved is approximately 20% of the original sample. This converts to a slaked lime [$Ca(OH)_2$] content of roughly 40% by weight in the original mortar.

After the completion of the TGA analysis, the residue of the sample was also analyzed by X-ray diffraction. The calcium silicate mineral phase larnite (β-C_2S) was identified. This decomposition product is characteristic of C-S-H with a low C/S ratio[6]. This result is consistent with the mineral assemblage detected in the mortar by the other analytical methods.

Polished sections of the mortar were then analyzed by scanning electron microscope (SEM) which produced both backscattered electron and X-ray elemental images. These revealed a cementitious material composed of $C_4A\overline{C}H_{11}$ and $CaCO_3$. The eroded edges and reaction rims around the brick fragments indicate a reaction between the brick and the matrix. This would be consistent with the leaching of silica and alumina from the brick, and to reaction with calcium from the lime to produce the $C_4A\overline{C}H_{11}$, C-S-H and $CaCO_3$ in the matrix.

The results overall thus indicate a pozzolanic mortar rather than a pure lime mortar, and the pozzolana is the crushed brick. The term "pozzolanic mortar", like many others concerning traditional building materials, has been used over time to mean different things. Its usage here follows that of cement chemistry, which defines a pozzolanic material as one that, unlike Portland cement, does not react by itself with water to yield cementitious phases. Pozzolanas generally contain glassy or cryptocrystalline phases that contribute soluble silica in the presence of lime. Pozzolanas can be natural such as the lava from Pozzuoli, Italy, or artificial such as fly ash, blast furnace slag, or in this case, brick. At least some of the aluminum and the soluble silica that comprise the cementitious matrix come from the glassy phases of the brick.

Mainstone [7], on the basis of chemical analyses of the Hagia Sophia mortar, has also proposed that it is pozzolanic. A typical set of data from such chemical analyses [8] is presented in Table I. The sample numbers refer to various locations around the Hagia Sophia. The free lime content represents the amount of lime or slaked lime remaining in the samples. Total lime, on the other hand, is the total amount of calcium occurring in all the phases (calcium carbonate, C-S-H, $Ca(OH)_2$, etc.). If it is assumed that the total lime in the fresh mortar mix was all in the form of slaked lime, then the ratio of free lime to total lime is an index of the degree of completion of the various chemical reactions involved in the hardening of the mortar. As can be seen from the bottom line of Table I, this ratio is in all cases less than 1%. Therefore, the original slaked lime is essentially all reacted.

The next question is how much of the slaked lime wound up in $CaCO_3$ as opposed to C-S-H. This can be estimated by the relative amounts of carbonate and soluble silica measured in each sample. Soluble silica is associated with C-S-H, while the carbonate is obviously a measure of $CaCO_3$. Both of these constituents are found in significant amounts in all the mortar samples, but the percentages vary greatly. For instance, for Sample #1, the soluble silica content is roughly ½ that of the carbonate, while the opposite is true for Sample #6. Consequently, it appears that in all cases, the binder in the mortar includes both carbonated lime and a C-S-H phase. Therefore, the mortar should not be classifed as a traditional lime-sand mixture, but rather as pozzolanic.

However, such a classification based on chemical analyses should be treated as qualitative rather than quantitative in pozzolanic mortar of this type. This is a bulk analysis which includes both the matrix and and the brick aggregate. Therefore, some soluble silica will be contributed by the brick fragments themselves, in addition to the C-S-H. Also, carbonate will be contributed by the $C_4A\overline{C}H_{11}$ as well as the $CaCO_3$. A more accurate analysis could be possible if the phases could be separated, possibly by centrifuging. Also, Biffen [5] shows that the free lime measurement by ethylene glycol extraction can be inaccurate, especially when $Ca(OH)_2$ is present along with CaO. In this case, such inaccuracy is a minor problem because it does not

TABLE I: CHEMICAL ANALYSIS OF HAGIA SOPHIA MORTAR SAMPLES

SAMPLE NO.	1	2	3	4	5	6	7	8
FREE LIME, %	0.07	0.15	0.02	0.05	0.08	0.06	0.07	0.03
INSOL.RESIDUE, %	58.9	53.9	53.4	59.3	29.8	51.6	60.9	59.5
SOLUBLE SILICA, %	5.56	6.22	8.50	6.32	10.78	14.78	7.59	5.70
TOTAL LIME, %	15.4	17.9	17.0	13.8	25.2	14.4	13.7	15.9
CARBONATE, %	11.8	13.8	12.08	9.36	17.46	7.80	10.54	11.91
SULFATE, %	0.23	0.01	0.10	0.15	1.82	0.10	0.05	0.23
TOTAL WATER, %	1.91	1.83	2.13	ND	ND	ND	ND	ND
FREE LIME / TOTAL LIME, %	0.43	0.86	0.15	0.33	0.33	0.43	0.54	0.19

change the conclusion that almost all the original slaked lime has reacted.

Finally, the data entries for sulfate in Table I are all very small. This indicates that gypsum is largely absent from the mortar. The findings from the combined X-ray and neutron diffraction analysis are thus confirmed.

Another type of analysis of the Hagia Sophia mortar, which is still underway, uses a combination of small angle X-ray and small angle neutron scattering (SAXS and SANS respectively) to characterize the microstructure. The data produced by these scattering techniques can be interpreted to provide such information as the porosity, typical particle size and fractal dimensions of stone, clays and cementitious materials [9].

Some preliminary data of the SAXS analysis of the Hagia Sophia are shown in Fig. 4 along with data from a typical Portland cement sample that is 28 days old. In this kind of plot, the slope of the curve is a direct measure of the fractal dimension. It can be seen that the shape of the curve of the Hagia Sophia differs significantly from that of the Portland cement sample. Qualitatively, the former appears much more like a natural material such as stone or clay than the latter, which is typical of a manmade material [10].

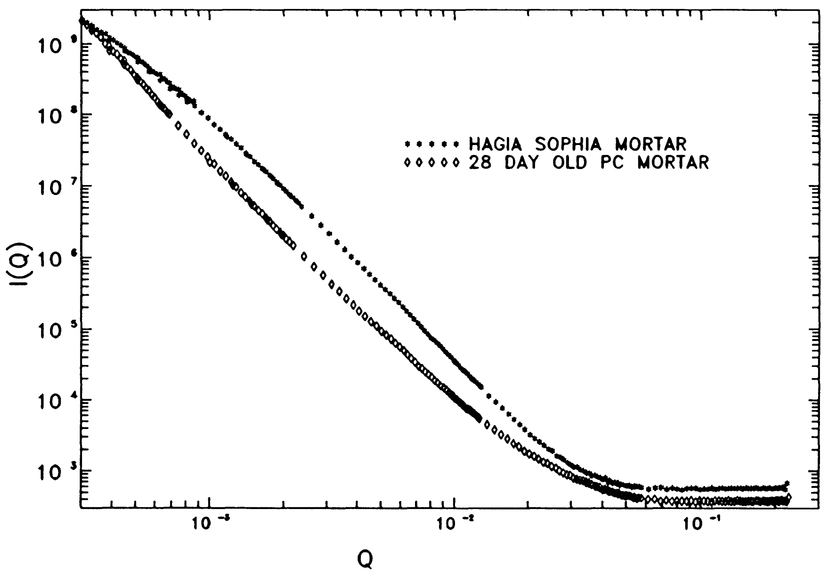

Figure 4: Plot of Small Angle X-ray Scattering Intensity as a Function of Q, the Scattering Wavevector. Q is in inverse Ångstroms (10^{-1} nm).

DISCUSSION

There is ample evidence for the use of crushed brick as a pozzolana in ancient Roman masonry. Vitruvius states that

> in using river or sea-sand, the addition of a third part composed of burnt brick, pounded up and sifted, will make your mortar of a better composition to use [11].

However, there is no documentary evidence for this during the following Byzantine period. Nevertheless, archaeological evidence shows that this proactice must have continued during this period [12]. In the subsequent Ottoman era, brick dust pozzolana was used to make what is referred to as Khorosany mortar [13]. The proportion of lime in the Hahgia Sophia mortar estimated from the TGA data, 40%, is close to the 1:2 ratio of lime to sand specified by Vitruvius.

Aside from the pozzolanic effect of the crushed brick, it is also possible that the lime itself may have included some cementitious material in the form of β-C_2S, if the original limestone contained suitable clay minerals [14]. The hydration of this might have contributed to the C-S-H phase.

The aggregate in the mortar appears to be primarily the brick fragments. The quartz identified in the diffraction patterns does not appear in the SEM images as a separate sand aggregate. Instead, it probably occurs in the brick fragments. Preliminary examination of the samples in the SEM at 1000X magnification revealed that the fine particle sizes of the calcium carbonate had a microstructure typical of carbonated lime particles rather than crushed limestone [15]. Moreover, no large grains of limestone were seen at lower magnification. Thus limestone apparently was not used as an aggregate.

STRUCTURAL IMPLICATIONS

A major advantage of pozzolanic mortars compared to pure lime ones is higher tensile strength. Table II presents some comparisons amomg the various types of mortar materials. It can be seen that pozzolanic lime mortars can reache a tensile strength of 3.5 MPa (500 psi) after a year. There is very little comparable published information on tensile strength of pure lime mortars, but values of 0.21 MPa (30 psi) for medieval mortars have been reported [16].

Using the tensile strength of 3.5 MPa from Table II in the static finite element model of the Hagia Sophia indicates that the structure as modeled would be able to resist the predicted shear and bending stresses. It would be preferable to use bond strengths (i.e. the shear strength between the mortar

TABLE II: COMPARISON OF MORTAR MATERIALS

BINDER	TIME TO SET	TIME TO FULL STRENGTH	TENSILE STRENGTH	COMPRESSIVE STRENGTH
LIME	24 hrs	100 days - yrs	0.34-0.7 MPa (50-100 psi)	9 MPa (1300 psi)
GYPSUM	½ - 1 hr	½-1 hr	4.6-5.0 MPa (670-720 psi)	46-50 MPa (6800-7800 psi)
PORTLAND CEMENT	5-8 hrs	100-150 days	2-3 MPa (300-400 psi)	21-28 MPa (3000-4000 psi)
POZZOLANIC	10-12 hrs	150 days -1 yr	3.4-3.8 MPa (500-550 psi)	14-17 MPa (2000-2500 psi)

and the brick units) to characterize the masonry. However, no data are available. Nevertheless, since bond strength is usually proportional to the tensile strength, it is evident that brick dust mortar would make stronger joints than simple lime mortar. Furthermore, in this case, tensile strength may be more relevant than bond strength given the unusual masonry design used for the Hagia Sophia, which is discussed in more detail below.

Another consideration of pozzolanic mortars is the time required to gain full strength. As shown in Table II, this can be on the order of a year for low pozzolana/lime ratios. This may explain the large deformations in the piers during construction that alarmed the architects. Under the very tight construction schedule imposed by Emperor Justinian, the critical phase when the main piers and arches were constructed could not have been much more than 18 months. This means that loads may have been applied to the piers before they had reached their full capacity. In fact, the Byzantine writer Procopius, mentions that during construction the main arches began sag because the mortar had not properly cured [17]. To fix the problem, parts of the tympanum walls had to be removed to allow the arches to "fully dry". Note that it is not necessary for a cementitious-type mortar to dry. In fact, if this type of mortar is allowed to dry out, it may not achieve full strength. The exposure to the atmosphere described by Procopius is more relevant to the carbonation process.

Moreover, the development of strength in pozzolanic mortars is more sensitive to ambient temperatures than in Portland cement-type mortars. The tight construction schedule also implies that masonry work may have continued over at least one winter, during which the temperature in Constantinople may have been low enough to affect the strength of the mortar.

Another aspect of the mortar question concerns the possibility that organic matter such as egg whites may have been added. It is known that many different organic materials were sometimes used as additives in antiquity [18]. One Byzantine documentary source, the *Diegesis*, states that the mortar of the Hagia Sophia was mixed with barley water and a decoction of elm bark [17]. However, this may not be a reliable source, partly because it was written four centuries after the event and partly because some of its statements are improbable. The function of such organic additives in ancient mortars is not well understood. In some cases, they may have improved the workability, setting time or durability rather than the tensile strength.

In any event, the architects of the Hagia Sophia seem to have been aware of the tensile strength of this mortar, in view of the unusual type of masonry construction they employed. The mortar joints are as wide (5-7 cm), if not wider, than the bricks themselves. In fact, the mortar appears to be the major constituent of the masonry with the bricks acting mostly as reinforcements. This is particularly true in the vertical joints of the main arches. In modern terminology, this material would actually be classified as a concrete instead of a mortar because of the coarse size of the brick aggregate, on the order of 1 cm. Therefore, the Hagia Sophia might be more appropriately considered as an early concrete structure rather than as one of the more familiar brick-and-mortar structures that developed later in medieval Europe. This proto-concrete technology apparently came into wide use in this period throughout the Byzantine Empire [19].

Given that concrete technology is involved, this raises the question of whether the architects of the Hagia Sophia intentionally used brick fragments rather than limestone, quartz or other stone aggregate to reduce the dead load of the structure. As shown in Table III, brick is the least dense of the materials commonly used for aggregate in concrete.

Regarding this question, the *Diegesis* suggests that the density of the brick units themselves was deliberately varied by the builders in order to lighten the dome. Here it is stated that special lightweight bricks were imported from Rhodes to build the main arches and dome [17]. Vitruvius [11]

TABLE III: COMPARISON OF SPECIFIC GRAVITIES OF VARIOUS AGGREGATES

AGGREGATE TYPE	SPECIFIC GRAVITY*
Handmolded brick	1.6 - 2.0
Sandstone	2.0 - 2.6
Limestone	2.2 - 2.6
Quartzite	2.65
Granite	2.6 - 2.7
Basalt	2.8 - 2.9

*Brick data from Baker [20]; stone data from Winkler [21].

describes bricks made of pumice that were light enought to float in water. He does not specifically mention Rhodes as a source of these bricks, but does say that they came from Pitane, a region in Asia Minor near Troy. However, as already noted, the *Diegesis* may not be a reliable source, particularly when it states that the bricks from Rhodes were one-twelfth the density of regular bricks. Nevertheless, it would be interesting to investigate this question further by measuring the densities of the Hagia Sophia bricks from various parts of the structure. Also trace element signatures of these bricks could be compared with those from Rhodes and Pitane to test the hypothesis that they were imported.

There are a number of other questions about the brick units that could be investigated. One is whether the composition of the units, whether locally-made or imported, differs from that of brick fragments used as aggregate in the mortar. This might occur if older brick buildings were being recycled to construct the Hagia Sophia. Another question is how the brick units were manufactured. The large dimensions of the units, 37.5 x 37.5 x 5 cm, and fine texture suggest that methods similar to terracotta work were employed.

CONCLUSIONS

On the basis of the analysis thus far, it appears the mortar used in the construction of the Hagia Sophia was pozzolanic rather than a simple lime mortar. The pozzolana consisted of ground brick dust, an additive commonly used during this period. While this provided a higher mortar tensile strength than lime mortar, the curing is relatively slow and temperature dependent. This slow strength development may account for the large plastic deformations of the structure. Moreover, the use of extremely wide mortar joints and coarse brick aggregate suggest that the builders made use of a concrete technology, instead of conventional brick and mortar masonry design in planning the building.

FURTHER RESEARCH

A primary objective of this study is to determine the tensile strength and elastic modulus of the mortar, which are critical parameters for the finite element structural model. The conventional civil engineering approach, which consists of loading standard size cylinders to failure, is essentially ruled out here because of the lack of large specimens, and the desire to avoid destructive tests as much as possible. Livingston et al. [1] review the problems with these standardized test methods.

The limiting strength in masonry is often the flexural strength of the interface between the brick and the mortar. Usually it is even more difficult to obtain samples of historic masonry containing both the brick and the mortar.

However, in the Van Nice collection at Dumbarton Oaks, there is at least one specimen of brick and mortar together that may be large enough to permit bond strength testing. This could be done by triaxial shear tests [22] or by applying a torque with the bond wrench method [23].

The dynamic modulus of elasticity can be determined non-destructively by measuring the speed of sound waves through the specimen [25]. Penelis et al. found that this method yielded very reliable modulus of elasticity data for Byzantine era masonry in Greece [24]. An approximate static modulus can be obtained from modulus of rupture measurements on thin disks using a method developed by Wittman and Prim [25].

The measurement of the strength properties could be made considerably simpler if the tests could be made on replica mortar samples rather than on the orginal material. However, producing a suitable replica mortar is not simply a matter of matching the proportions of lime, pozzolana and aggregate in the original. Extensive research on Portland cement materials has shown that the final strength is very sensitive to factors such as water/cement ratio, initial particle size and curing environment, that are very difficult to determine after the fact. However, the SANS/SAXS measurements of mortar microstructure may provide sufficient information to tell if a given replica mortar is a good match to the original.

Finally, another area of research concerns the dating of various parts of the structure of the Hagia Sophia, particularly the several layers of buttresses. This information could be related to known dates of earthquakes in the history of the region. This would help to identify structural weaknesses within the structure and the characteristics of its seismic response.

Several possibilities exist for dating the mortar samples. Radiocarbon dating of the carbon dioxide in the carbonate content lime should be feasible [26] In this case, it should relative straightforward, since there is apparently little or no limestone aggregate present in the mortar. Another radiocarbon method for dating mortar uses the charcoal fragments that are found in the lime as a result of the limestone calcination process [27]. An entirely difference approach would be to date the brick fragments in the mortar using thermoluminescence [28].

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