

**THE PERFORMANCE OF TWO ALKOXYSILANE CONSOLIDANTS ON THREE BERIA
SANDSTONES THROUGH CONTROLLED ENVIRONMENTAL STRESS CYCLING**

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**In Partial Fulfillment
of the Requirements for the Degree
Master of Science in Historic Preservation**

By

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This thesis is dedicated to the memory of my mother, Ibis C. Swanson, and to my father, Daniel G. Swanson, Jr., whose love and support have brought me so far.

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Chapter 1: Introduction:

This thesis concerns the performance of two alkoxysilane consolidants on three Berea sandstones, and the ability of the consolidants to resist controlled environmental stress cycling. Consolidants are used to strengthen and/or solidify other materials, often porous materials such as deteriorating stone. The two consolidants tested are Conservare[®] OH100¹ (OH 100) and Funcosil 500 STE² (500 STE). Conservare[®] OH100 is a version of the industry standard for alkoxysilane consolidants, while 500 STE represents alterations to the standard formulation. It is a European product that takes the same basic alkoxysilane formula, of $\text{Si}(\text{OCH}_2\text{CH}_3)_4$, and includes the addition of pure silica nanoparticles, and elastifiers of silicic ethyl acid esters (SAE) to reduce the shrinking and cracking of the cured gel.³ In stone consolidation OH 100 deposits 30% w/v of gel, and 500 STE deposits 50% w/v of gel.

Both consolidants add strength to the stone by forming siloxane bonds (Si-O-Si), bonds between silicon and oxygen atoms within the molecules of silica (SiO_2 , silicon dioxide) in the stone and the alkoxysilane consolidant. Once the consolidants have cured from a liquid to a solid silica gel state inside the stone matrix, they are subject to the same environmental stresses as the stone itself.

¹ PROSOCO Conservare[®] OH 100 manufactured by PROSOCO, Inc., 3741 Greenway Circle Lawrence, KS 66046

² Remmers 500 STE (KSE 500 STE), manufactured by Remmers Baustofftechnik GmbH, Bernhard-Remmers-Str. 13, 49624 Lönigen

³ Wheeler, George, and Getty Conservation Institute. 2005. Alkoxysilanes and the Consolidation of Stone. Research in Conservation. Los Angeles, Calif: Getty Publications.

The gel deposition is the amount of consolidant that cures in the stone matrix, and functionally acts as the consolidant, strengthening the stone. The gel deposition rate affects the amount of VOC (volatile organic compounds) that are emitted as the consolidant cures. A higher gel deposition rate means fewer VOC released into the atmosphere, which is a statistic that is a growing focus of regulation as communities and governments seek to protect the atmosphere and environment. As regulations change, the legality of using some consolidants may change, making the testing of alternative products important.

Parliament Hill, the seat of the parliament of Canada, has three major buildings, the Centre Block with Parliamentary Library, the East Block, and the West Block. All three were constructed using a similar combination of sandstones. Parliament Hill overlooks the Ottawa River in a region with a yearly temperature which can range from 33° C in the summer to -40° C in the winter. Ottawa was for many years an industrial city, and factories were sited up and down the river. For many years a factory sat upwind of the Parliament buildings and a steam lumber mill was located below the cliff on which the buildings sat (Fig. 1 & Fig. 2); the nearby factories released pollutants into the air, and deposits of these pollutants formed on the buildings in Ottawa.⁴ The location meant that a large amount of industrial and atmospheric pollutants, as well as the environmental stresses acted on the buildings for years. Therefore, the role of environmental stresses on

⁴ "Home - Parliament Hill - PWGSC." N. p., n.d. Web. 14 Mar. 2014.

sandstone consolidants is of interest.⁵ The West Block building of the Parliament of Canada was the source of stone for this thesis.



Fig. 1. View of Parliament Hill from the Northern bank of the Ottawa River. The remains of a steam lumber mill can be seen at the base of the cliff below the Parliament buildings.⁶



Fig. 2. View of Parliament Hill from the Northern bank of the Ottawa River. The same Geological Survey photo, colored and sold as a postcard.⁷

⁵ "About Ottawa, Canada's Capital." N. p., n.d. Web. 2 Apr. 2014.

⁶ "Natural Resources Canada | Natural Resources Canada." N. p., n.d. Web. 27 Apr. 2014.

While industrial pollutants are far less of an issue today with stricter air quality regulations, the environmental forces do remain a threat. High temperatures in the summer and the cold of Ottawa winters constantly affect the stones of the Parliament buildings. Thermal expansion and contraction with the temperature changes and the freezing of water within the stone in winter can deteriorate the sandstone by weakening the bonds between the grains of stone, making it susceptible to granular disintegration and erosion by wind, and rain. With these known effects on the stone, it seems naïve to imagine that these factors would not have an effect on consolidants within the stone.

Strength testing the stone is one method to evaluate the consolidants performance after environmental stressing. This thesis looked at the strength of the stone before and after consolidation, and after controlled environmental stress cycling of the consolidated stone. Ideally, a stone consolidant makes the stone stronger than it was before consolidation, or increases the longevity or durability of the stone by increasing the ability of the stone to resist further deterioration, specifically for this study granular disintegration as defined by Price (16).⁸ This thesis investigated the ability of the consolidants to maintain the increased strength they added to the stone after the treated stone was subjected to environmental stresses designed to mimic environmental temperature and humidity fluctuations similar to those in the field. A comparative analysis of the retention of strength is important in determining the benefits of

⁷ Hessel, Peter D. K., and Canada. *From Ottawa with Love: Glimpses of Canada's Capital through Early Picture Postcards*. Ottawa: National Capital Commission, 1979. Print.

⁸ Price, C. A., and Getty Conservation Institute. *Stone Conservation: An Overview of Current Research*. Santa Monica, CA: Getty Conservation Institute, 1996. Print. Research in Conservation.

consolidation for these specific stones and buildings. Previous work by Columbia professors on consolidated marble has shown minimal benefits where accelerated weathering tests indicated a loss of almost all of the strength gained from the application of consolidant in the stone tested.⁹

For the testing in this thesis three groups of Berea sandstone were strength tested. Untreated stone demonstrated the properties of the stone itself. A test group treated with Conservare™ OH 100 served to show the properties of an industry standard alkoxy silane consolidant. The third test group, treated with Funcosil 500 STE, provided data on the performance of an elastified, high gel deposition consolidant with added silica nanoparticles. Additional test groups were subjected to controlled environmental stress cycling. The tensile strength of the consolidated materials were tested using an Instron 5569A machine, Bluehill software, and a biaxial flexure set-up, which measured the characteristics of the force applied to an individual stone sample and its deformation under the applied force. Testing the flexural bond strength of a consolidated stone can measure its change in resistance to small tensile forces similar to those that act on a stone and cause deterioration in the form of granular disintegration.¹⁰

It was expected that stone treated with 500 STE would be significantly more resistant to controlled environmental stress cycling than stone treated with OH 100 because of the added silica nanoparticles and the elastification of the consolidant intended

⁹ Wheeler, George. Director of Conservation, Graduate School of Architecture, Preservation, and Planning; Columbia University. Personal interview. Spring 2014.

¹⁰ Wheeler, Alkoxy silanes and the Consolidation of Stone., 20

to reduce cracking and shrinkage in the cured gel. Instead the performance of the two consolidants was found to be comparable.

Chapter 2: Stone Consolidants:

The goal of consolidation for architectural sandstone is generally to prevent further loss of material from granular disintegration, erosion, scaling, or other forms of disaggregation at the surface of the stone used in a building, not to increase the compressive strength of the material. This loss of grains of stone can destroy carved detail, and eventually compromise the cohesiveness of the stone itself as material is worn away from the unit.^{11 12} Consolidants have a long history of use in the field of preservation, where they have addressed the slow and inexorable deterioration due to the inevitable natural processes of disintegration. Numerous materials have been advocated for and tested, with varying results. Some potential materials have looked promising for several years of testing and limited use, only to show later that the consolidant had little to no effect in the long run. This makes the testing of consolidants particularly important.¹³ Over time it has become clear that there is no one consolidant that works for all types of stones, and that the performance of a consolidant is as dependant on the individual characteristics of the stone to be treated as it is on the consolidant itself.

¹¹ Zinsmeister, Klaus J.H., Norman R. Weiss, and Frances R. Gale. "Evaluation of Consolidation Treatment of an American Sandstone." *Bautenschutz + Bausanierung* 8.2 (1985): 79–82. Print.

¹² Wheeler, *Alkoxysilanes and the Consolidation of Stone.*, 22

¹³ Henry, Alison. *Stone Conservation: Principles and Practice*. Shaftesbury: Donhead, 2006. Print.

History

The history of stone consolidation is closely tied to the history of chemistry and the synthesis of chemicals. Often a chemical is created in a lab years before it may be tested for use in materials conservation. There are a variety of names used throughout history for the alkoxy silanes consolidants as a group and for the individual compounds used in conservation. Alkoxy silanes as a group have been called: silicon esters, silicic acid esters, ortho-silicates, alkyl silicates, as well as alkoxy silanes. Tetraethoxy silane (TEOS) has been called: ethyl silicate, tetraethyl orthosilicate, silicic acid ethyl ester and TEOS.¹⁴ The history of alkoxy silane consolidants has been well covered by George Wheeler in his 2005 book, *Alkoxy silanes and the Consolidation of Stone*. The following short summary references that seminal work.

Silicon tetrachloride was first synthesized in 1824 by J.J. Berzelius and little remarked on being simply synthesized to join the growing ranks of lab created compounds. It took approximately one hundred years before a use was found for silicon tetrachloride; it became an important material in the silicon resin industries that were developed in both the United States and the former Soviet Union for use in adhesives, silicone rubbers, coatings, and as paint additives. During the wait for a use for this new chemical, several other silicon-based compounds were synthesized in laboratories. Starting in 1846, several alkoxy silanes were synthesized: tetraethoxy silane (Ebelmen 1846), methyltriethoxy silane (Ladenberg 1874) and methyltrimethoxy silane probably in 1904.¹⁵

¹⁴ Wheeler, *Alkoxy silanes and the Consolidation of Stone*, 8.

¹⁵ Wheeler, *Alkoxy silanes and the Consolidation of Stone*, 1.

In 1861, in the search for materials to fix the failing stone of the still under construction Houses of Parliament in London, silicic ether, which is a form of ethyl silicate or tetraethoxysilane (TEOS), was proposed by A.W. von Hoffman. This was the first time that an alkoxysilane had been suggested for use on stone. In the 1920s, A.P. Laurie advocated for the use of ethyl silicate as a stone consolidant, which was the first major recommendation for its use since von Hoffman in 1861. While Laurie took out patents for several stone consolidants using ethyl silicate, its usefulness and efficacy was called into question by the early 1930s as early treatments began to deteriorate. However, this didn't prevent the use of ethyl silicate as a stone consolidant.

Through the 1940s and 1950s, ethyl silicate continued to be used in both stone conservation and other fields the paint industry. It continued to be studied, both in the lab and in the field where new uses for it were found. H. G. Emblem studied "silicon esters" in the context of paint binders and found them to be more stable than silicone based paints. He also discussed their use as concrete and stone preservatives.¹⁶ Harold Plenderlith in 1956 wrote the seminal English text on objects conservation, *The Conservation of Antiquities and Works of Art*, which mentions the use of silicon ester as an effective stone consolidant for sandstone and limestone objects that are kept indoors.¹⁷ This was the major reference to ethyl silicates in the 1950s.¹⁸

¹⁶ Wheeler, *Alkoxysilanes and the Consolidation of Stone.*, 138.

¹⁷ Wheeler, *Alkoxysilanes and the Consolidation of Stone.*, 4.

¹⁸ Wheeler, *Alkoxysilanes and the Consolidation of Stone.*, 2.

In the late 1950's and early 1960's alkoxy silanes began to be used as water repellents as well as stone consolidants. The late 1960's saw many studies on the affect of alkoxy silanes on stone. Enthusiasm and praise abounded for alkoxy silanes; great success was found for instance by Kenneth Hempel and Anne Moncrieff at the Victoria and Albert Museum in the consolidation of sugaring, or disaggregating, marble.¹⁹ But by the early 1970's the praise for alkoxy silanes had become more nuanced, with reports of the failure of alkoxy silanes to consolidate deteriorating stones. The increased number of studies involving alkoxy silanes on previously untested types of stone produced a rise in the number of conflicting reports as to the success or failure of the material. In some instances the consolidant worked as intended, giving strength to the stone, and in others the consolidant failed to prevent further deterioration to the stone as shown in field applications. Through the 1980's, 90's and 2000's, the study of alkoxy silanes used for stone consolidation rapidly expanded leading to a better understanding of their performance.

The type of stone that is being consolidated has a large effect on the success of the consolidant in laboratory testing or in the field. While alkoxy silanes can form silica gel linkages, very simply described as chains of silica molecules, between the quartz grains of siliceous sandstones, laboratory testing has shown only isolated deposits of silica gel being formed in other types of stones, such as calcitic limestones. Since these silica gel linkages are believed to be the active force in strengthening the deteriorating stone,

¹⁹ Wheeler, Alkoxy silanes and the Consolidation of Stone., 3.

limestones, lacking silica and being primarily calcium carbonate based stone, are theoretically strengthened less by an alkoxy silane consolidant. In practice however, the performance of any consolidant is dependant on the individual characteristics of the stone to which they are applied and few if any generalities or predictions can be made.

Properties

Despite their unpredictable performance, alkoxy silane consolidants are of interest to preservationists because of several properties, the most important of which are: Their low viscosity, allowing for good penetration into a stone; their ability to form strong siloxane bonds (Si-O-Si); and their resistance to discoloration when exposed to solar radiation. In order to function as something more than a surface coating, a consolidant must be able to penetrate the stone and cure within the stone matrix. To do this, a consolidant needs to have a low viscosity. Low viscosity allows the consolidant, when applied to the surface of the stone, to be drawn into the stone by capillary uptake.²⁰ Originally, solvents such as toluene, methyl ethyl ketone, acetone, ethanol, or mineral spirits, were added to alkoxy silane consolidants to decrease the viscosity of the material.²¹ This helped with the penetration of the stone, but was very toxic. Later research found that removing these additional solvents did not drastically increase the viscosity of the consolidant and their use was discontinued.²²

Siloxane bonds are relatively strong and UV-stable. The bonds are not affected by and are able to resist damage from UV light, which does not alter the bonds of the gelled

²⁰ Price, *Stone Conservation: An Overview of Current Research.*, 17.

²¹ Enjay Chemical Company. *Ketones: Acetone, Methyl Ethyl Ketone (MEK), Methyl Isobutyl Ketone (MIBK)*. [New York: N. p., 1962. 59 p.

²² Wheeler, *Alkoxy silanes and the Consolidation of Stone.*, 10

consolidant.²³ This inherent light stability makes alkoxysilanes good for exterior use, since the gel is resistant to breaking down from exposure to sunlight.

Alkoxysilane polymers can also act as water repellents, making consolidated stone hydrophobic.²⁴ This was one early purpose of the alkoxysilane treatment of stone, since at the time 'waterproofing' a stone was seen as the best method of preserving it.²⁵

Different alkoxysilanes retain their hydrophobicity for different lengths of time, but for the most part, they all are hydrophobic to a certain extent for a short time after curing.

Once the polymers are formed, their bonds cannot be chemically broken, making alkoxysilane consolidants irreversible, even with solvents or other aqueous solutions. Therefore, the use of alkoxysilanes on a stone must be a well thought out, considered decision. Because of this resistance to solvents and cleaners, pre-consolidation with an alkoxysilane before cleaning a stone can be used to strengthen friable surfaces, and the subsequent cleaning can be completed with less risk of material loss, due to either stone deterioration or the breakdown of the consolidant by the cleaning agents.

Curing

Alkoxysilane consolidants cure through hydrolysis and condensation reactions, reacting with atmospheric moisture, moisture present in the stone, or less commonly water added to the consolidant before application. The alkoxysilane monomers, molecules that can be joined to create polymers, react with water to form long molecules

²³ Wheeler, *Alkoxysilanes and the Consolidation of Stone.*, 13

²⁴ De Clercq, H., and S. De Zanche. "Silicon Based Water Repellents and Consolidants: Influence of Application Order and Timing." *Restoration of buildings and monuments: an international journal = Bauinstandsetzen und Baudenkmalpflege: eine internationale Zeitschrift* 14.5 (2008): 339–346. Print.

²⁵ Wheeler, *Alkoxysilanes and the Consolidation of Stone.*, 13

of repeating small units, polymers, inside the stone structure.²⁶ A condensation reaction is another way of creating polymers from monomers where, as the monomers join together to make a larger, more complex polymer, there is the loss of a water molecule.²⁷ In addition, an alcohol is released during the curing of the polymer, which evaporates from the stone. The curing of alkoxysilanes by hydrolysis and condensation reactions, create different forms of silicon dioxide that are chemically similar to quartz, the main material in sandstones. The polymers from the consolidant create siloxane (Si-O-Si) bonds between the grains of quartz in the sandstone making them useful for sandstone that exhibits disaggregation because they create bonds that are functional analogues to some of those originally in the stone.

The low viscosity of these consolidants allows them to penetrate the stone and deposit in the small spaces between the grains. If the spaces between undeteriorated stone substrate and the disaggregated grains of friable stone are too large however, the polymer bridges formed by alkoxysilanes are unable to secure the loose material.

Tensile stresses exerted on the pores of the gel during the curing reaction and stresses on the cured gel from environmental thermal cycling and natural weathering are factors that can lead to the formation of micro-fractures in the gel body. Micro-fractures can compromise the strength of the gel making it less effective as a consolidant. The gel at the closer to the surface of the stone matrix cures faster, as the solvent and water evaporate faster with exposure to the air. As this liquid is lost, liquid from gel deeper in the stone migrates to the exposed surface, creating tensile stresses on the already cured

²⁶ Solomons, T. W. Graham. *Organic Chemistry*. 9th ed. Hoboken, NJ: John Wiley, 2008. Print.

²⁷ Solomons, T. W. Graham. *Organic Chemistry*. 9th ed. Hoboken, NJ: John Wiley, 2008. Print.

consolidant gel deposited between the stone grains closer to the surface. Laboratory testing indicates tensile stresses in the liquid can be as high as one hundred megapascals (100 MPa), which can cause the cured gel to fracture.²⁸ Smaller spaces in the stone matrix create higher tensile stresses on the curing consolidant, increasing the likelihood of the consolidant gel cracking.²⁹ The size of the interstitial spaces in the stone also affects the amount of consolidant that ultimately penetrates the stone. As a liquid the consolidant is subject to such forces as capillary uptake, and surface tension, that when combined with the size of the interstitial spaces in the stone will affect the rate of uptake into the stone of the consolidant.³⁰ Theoretically, the introduction of certain elasticizers to the consolidant can make the silica gel deposited in the stone less likely to fracture when the consolidant cures.³¹

There have been several testing programs on the effect and efficacy of alkoxysilanes on limestones, where the alkoxysilane is less successful on a carbonate stone, compared to a silicate stone. The size of the interstitial spaces between the grains of stone and the presence of clays in the stone have the same strong effect on whether or not an alkoxysilane would be successful when applied to limestones, as when it is applied to sandstones. With carbonate stone though the presence of clay is thought to help the consolidant link together grains of limestone, and the current thought is that clay hinders

²⁸ Wheeler, *Alkoxysilanes and the Consolidation of Stone.*, 40

²⁹ Smaller interstitial spaces increase the tensile forces on the curing and cured consolidant gel, increasing the likelihood of it cracking under the pressure.

³⁰ Wheeler, *Alkoxysilanes and the Consolidation of Stone.*, 53.

³¹ *Stone Consolidation*, 211.

consolidation of a silicate stone.^{32 33} The effect of large interstitial spaces is the same as with sandstones, where the alkoxy silanes are unable to fill larger gaps. Prevailing thought is that the presence of clay in sandstones hinders consolidation.³⁴

³² Pintér, F., J. Weber, and B. Bajnóczy. "Visualisation of Solid Consolidants in Pore Space of Porous Limestone Using Microscopic Methods." *11th International Congress on Deterioration and Conservation of Stone: 15-20 September 2008, Torun, Poland: Proceedings*. Ed. Jadwiga W. Lukaszewicz and Piotr Niemcewicz. Torun: Wydawnictwo Naukowe Uniwersytetu Mikołaja Kopernika, 2008. 473–480. Print.

³³ Wheeler, G. et al. "Evaluation of Alkoxy silane Coupling Agents in the Consolidation of Limestone." *Proceedings of the 9th International Congress on Deterioration and Conservation of Stone, Venice, June 19-24, 2000*. Ed. Vasco Fassina. New York: Elsevier Inc., 2000. 541–545 (vol. 2). Print.

³⁴ George Wheeler, personal communication.

Consolidants

Type 1 – Conservare® OH 100

OH 100 is a tetraethoxysilane consolidant (TEOS) containing no added water repelling agents or additional solvents.³⁵ This consolidant is a descendent of some of the earliest alkoxy silane consolidants patented by Wacker in the early 1970's.³⁶ The consolidant gel itself is water repellent for several months after application. The curing process requires atmospheric moisture. As OH 100 cures it releases ethanol.³⁷

Type 2 – Funcosil 500 STE

Funcosil 500 STE is also a TEOS consolidant with no added solvents.³⁸ However, 500 STE contains added pure silica mineral nano-particles in suspension, and is elastified with silicic acid ethyl esters (SAE). This elastifier, in the form of hydrocarbon chains, theoretically assists in the bonding together the silane monomers.³⁹ The filled and elastified 500 STE formulation theoretically can form elastic bridges of SAE between the oligomers of TEOS by linking them together to form longer, more flexible, polymer chains, and span larger interstitial spaces than currently available alkoxy silane consolidants. The high, compared to OH 100, amount of filled and elastified gel

³⁵ Wheeler, *Alkoxy silanes and the Consolidation of Stone.*, 53.

³⁶ OH 100 deposits, through a hydrolysis curing reaction, 30% w/v of silica gel into a stone matrix creating approximately 300g of silica gel for every liter of consolidant applied, as determined by laboratory measurements of the weight of unconsolidated and consolidated stone.

³⁷ Material data safety sheets are available at the PROSOCO, Inc. website.

³⁸ The 500 STE deposits, through a hydrolysis curing reaction, 50% w/v of silica gel into the stone or approximately 500g of gel for every liter of consolidant applied, as determined by laboratory measurements.

³⁹ Remmers, KSE Modular System Technical Sheet, 2.

deposited in the stone matrix during curing is intended to decrease the potential amount of shrinkage of the consolidant. However, the addition of silica nano-particles in the consolidant may also reduce the ability of the consolidant to penetrate the stone matrix depending on the size of the interstitial spaces between the stone grains.

Comparison

These two stone consolidants are similar in many respects. Both are based on TEOS. They function the same way where, once applied to siliceous stone, the consolidants form silica gels within the stone matrices and form siloxane (Si-O-Si) bonds; these are bonds with strength similar to the bonds naturally present between the silicate minerals in the sandstone. Both have low viscosity and similarly penetrate into the stone matrix. They are irreversible applications; however it is worth noting that stone treated with both can be retreated.

The difference between the two lies in the additional components added to the 500 STE, the elastification of its silane polymers, and the amount of consolidant deposited in the stone matrix. These components are intended to decrease the shrinkage of the cured consolidant, fill interstitial spaces between the grains of the stone and lessen the brittleness of the cured consolidant. The increased amount of gel deposited is intended to increase the concentration of silica bonds formed within the stone matrix, and increase the tensile strength of the treated stone.⁴⁰

⁴⁰ Further research on VOC compliance issues is required, and is not covered as part of this study. A lower VOC emission rate may be a beneficial characteristic of 500 STE.

Chapter 3: Berea Sandstone:

Sandstone

Sandstone is sedimentary stone formed from a mixture of the disaggregated erosion products of other stones.⁴¹ While any minerals from any rock in a watershed may erode, be transported, most often by water, and eventually form sandstone, the minerals that are found in sandstone are primarily those that are physically and chemically stable such as quartz and feldspars. The environmental stresses that the mineral grains undergo in their transportation to a sedimentary basin where they deposit causes the different minerals to abrade based on their hardness or solubility. This abrasion produces different grain sizes, and shapes. The grains that make up sandstone are a size category that is classified as sand ranging from a diameter of 0.0625 mm to 2.0 mm, with volumes of about 0.00012 mm³ to 4.2 mm³.⁴² Some minerals will abrade to grains smaller than sand and form silts and clays, which can also be found in some sandstones. Quartz (SiO₂), common in the rocks of the earth's continental crust is both chemically and physically resistant to environmental stresses.⁴³ This means that it is likely to be present in large amounts in a sedimentary rock such as sandstone. In many types of sandstone there are among the pure mineral grains, stone fragments small enough to be in the sand size range that are formed of different minerals,. These rock fragments are visually distinct under magnification, and seen as an array of minerals in one grain.

⁴¹ Pettijohn, F. J. *Sand and Sandstone*. 2nd ed. New York: Springer-Verlag, 1987. Print.

⁴² Pettijohn, 1.

⁴³ The Scientific American Science Desk Reference. New York: John Wiley, 1999. Print.

The mineral composition of each different type of sandstone is the primary factor influencing its surface appearance. Sandstones that are primarily composed of quartz, known as arenites, tend to be very light gray or white in color. Some sandstones also contain mineral grains muscovite (mica) that catches the light, adding a sparkling quality to their appearance. Sandstones that are rich in clay and rock fragments can appear shades of gray, green-gray, and dark gray, such as graywacke which has very fine grains of clay that produce a very dark appearance. Iron containing minerals, often hematite or limonite give the red, brown, and tan tones to sandstone, popularly known as brownstone.⁴⁴ These minerals can also be the cause of dark inclusions in otherwise light colored stones. The appearance and color of a stone cannot be used as a method of identification, since it only provides a general sense of a stones composition.

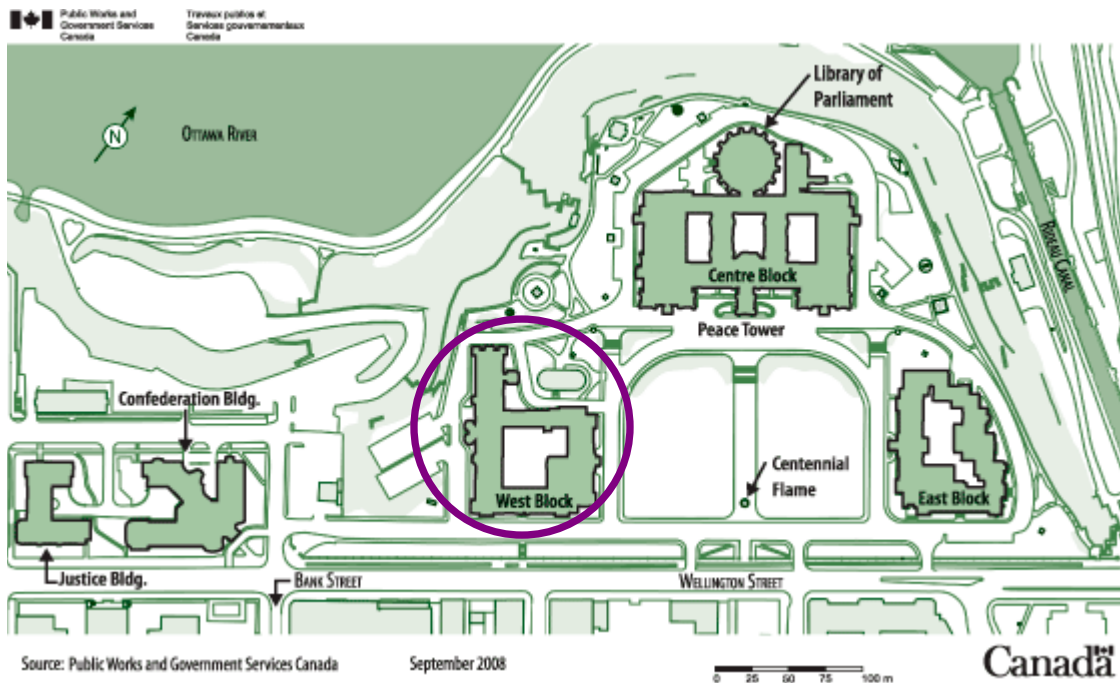
Consolidation can change the color of a treated stone, and the final appearance of consolidated stone on a building or monument should be considered before treatment is carried out.

Bedding planes, and low tensile strength, make sandstone vulnerable to erosion and deterioration. While the deterioration is apt to take place over long periods of time, with varying rates of decay, eventually sandstone can be completely compromised both aesthetically as well as structurally. This depends in large part on the mineralogical characteristics of individual sandstones, and the physical and chemical bonds that link the grains of minerals in them.⁴⁵ The surface of some sandstones will erode away a few millimeters to a certain depth revealing otherwise sound material. Other stones will

⁴⁴ Pettijohn, 29.

⁴⁵ Kourkoulis, Stavros K., ed. *Fracture and Failure of Natural Building Stones: Applications in the Restoration of Ancient Monuments*. Dordrecht: Springer, 2006. Print. (55)

deteriorate with "...blisters, crumbles, powders and flakes, leaving the remaining stone so weakened that it seems to be held together by little more than habit."⁴⁶ Often this deterioration can be linked to the dissolution of the weak bonds between the grains of stone, which is directly related to the lower tensile strength of sandstone compared to other stones such as granite. The bonds between the grains are weaker than the harder minerals of the grains themselves, and are more susceptible to the environmental stresses working on the stone.⁴⁷ Sandstones lose their component grains readily because of this low tensile strength, which can lead to erosion and other forms of deterioration from weather factors such as wind, rain and freeze/thaw cycles.



⁴⁶ Henry, 101.

⁴⁷ Castellanza, R., and R. Nova. "Oedometric Tests on Artificially Weathered Carbonatic Soft Rocks." *Journal of Geotechnical and Geoenvironmental Engineering* 130.7 (2004): 728–739. Print.

Fig. 3. Site Plan, Parliament Hill, Ottawa, Ontario. The West Block building is circled.⁴⁸

Sample Source: Parliament Hill

Parliament Hill, the seat of the Parliament of Canada, overlooking the Ottawa River in downtown Ottawa, Ontario, is the source of the substrate stone material for this thesis. Figure 3 shows a layout of the complex. Three different sandstones were used in the construction of the buildings at Parliament Hill, Nepean sandstone from near Ottawa, Berea sandstone from Ohio, and Potsdam sandstone also local to Ottawa.^{49 50} The exterior ashlar facing was constructed with buff Nepean sandstone, and the trim of windows and doors built in contrasting gray/brown Berea and reddish Potsdam stones.⁵¹ The West Block was designed by Augustus Laver and constructed from 1859 to 1909; sandstone samples for this thesis were obtained exclusively from the three construction phases of the West Block. All three stones are Berea sandstone quarried in Ohio for three main construction campaigns on the building in 1865, 1875, and 1909, and used for finished stone trim elements on the building.⁵²

⁴⁸ “Parliament Hill - PWGSC.” N. p., n.d. Web. 14 Mar. 2014.

⁴⁹ Lawrence, D. E. “Building Stones of Canada’s Federal Parliament Buildings.” *Geoscience Canada* (2001): n. pag.

⁵⁰ “Parks Canada - Federal Heritage Buildings Review - Federal Heritage Buildings Review Office.” N. p., n.d. Web. 29 Apr. 2014.

⁵¹ Lawrence, 17.

⁵² Please see Appendix A for Historic and Current photos of the West Block.

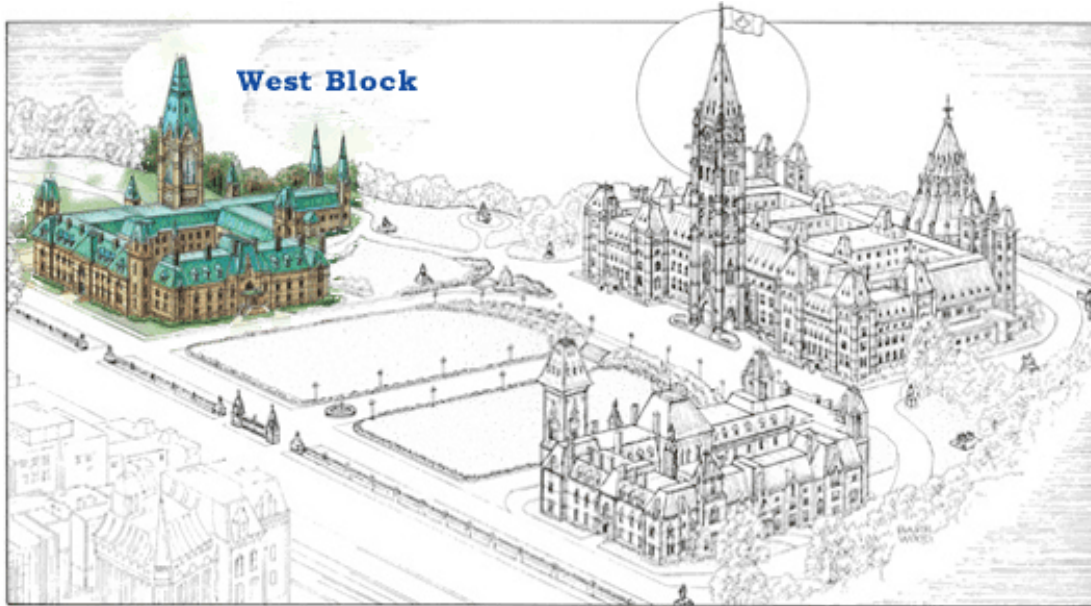


Fig. 4. Drawing of Parliament Hill, the current West Block Building is highlighted.⁵³

Designed originally as the seat of the parliament for the British Province of Canada, when the Confederation of 1867 was complete the complex was elevated to serving as the seat of the parliament for the new Dominion of Canada.⁵⁴ Figure 4 shows a drawing of the three main buildings. The building with a flag is the Centre Block, where the Parliamentary Chamber is located. The Dominion included Ontario, Quebec, New Brunswick, and Nova Scotia. Within four years it would expand to include Prince Edward Island, Manitoba, British Columbia and the Northwest Territories (today Alberta, Saskatchewan, the Yukon, NWT and Nunavut). Before they were complete, the parliamentary buildings were too small for their intended purpose. In 1876, an addition was constructed on the East elevation of the West Block building.

⁵³ “Home - Public Works and Government Services Canada (PWGSC).” N. p., n.d. Web. 14 Mar. 2014.

⁵⁴ Lawrence, 14.

The original grouping of parliamentary buildings designed in the late 1850's included the Centre Block designed by Thomas Fuller & Chilion Jones, and the East and West Blocks by Thomas Stent and Augustus Laver. The three separate designs were unified by the use of the same stones throughout. Originally, gray limestone from Quebec was specified for the buildings, but Fuller and Jones suggested instead the use of Nepean sandstone from Ontario.^{55 56} They thought the light, warm color of the Nepean would be visually more pleasing than the dark and somber gray of the limestone. The stone was also thought to be more durable than the limestone. The Director of the Geologic Survey of Canada attested to its strength and suitability as an architectural material.⁵⁷

Ground was broken on the 20th of December, 1859, and the first stones were laid in April of 1860. The East and West Blocks were completed in 1865 and the Centre Block and the Victoria Tower were completed in 1876. The group of buildings was designed in Gothic Revival style with mediaeval elements such as tracery windows, and towers.^{58 59} They were arranged with the Centre Block slightly higher than the East and West Blocks, making the 60m tall Victoria Tower the focal point of the site. The East and West Blocks were deliberately not identical, but balanced against each other.^{60 61}

⁵⁵ Lawrence, 15.

⁵⁶ Sanford, B. V. "Stratigraphic and Structural Framework of the Potsdam Group in Eastern Ontario, Western Quebec and Northern New York State." Ph.D. University of Ottawa (Canada), 2007. 304737015. (152) The Nepean sandstone was also considered an acceptable substitute stone as it was also local to the building site.

⁵⁷ Lawrence, 16.

⁵⁸ Public Works and Government Services Canada (PWGSC)- Parliament Hill.

⁵⁹ Encyclopedia Britannica, "Gothic Revival." (2014)

⁶⁰ Lawrence, 13.

⁶¹ Public Works and Government Services Canada Website- Parliament Hill

In 1897 the roof of the West Block caught fire (Fig. 5). While the damage was not extensive, the roof of the 1865 wing was destroyed, the north vent stack, and the walls of the eastern half of the 1865 wing were heat damaged. Following the fire, the roof of this wing was rebuilt and all the other roofs replaced to match. The architects chose copper sheeting rather than the original slate, as this was thought to give greater protection from fire. The worst of the damaged stone was replaced with in kind materials.

Alberta and Saskatchewan became provinces in 1905, necessitating more space on Parliament Hill, and between 1906-1914 further additions were built at the Centre, East, and West Block buildings.⁶²



Fig. 5, Historic photo of the 1897 fire, in the West Block.⁶³

⁶² Public Works and Government Services Canada (PWGSC)- Parliament Hill.

⁶³ Provided by Conservation Solutions, Inc.

The Centre Block itself caught fire on February 3rd, 1916 and was completely destroyed along with the Victoria Tower.⁶⁴ The Parliamentary library, attached to the Centre Block building, was only saved by the closing of the huge iron fire doors that a prescient librarian had insisted be installed.⁶⁵ Rebuilding work on the new Centre block started in September of 1916, designed by Jean-Omer Marchand. The Centre block and the Peace tower present today are from that rebuilding.

Historic Berea Sandstone Quarries

In the 1890's the primary quarries extracting Berea Sandstone were operated at North Amherst, Berea and Grafton, Ohio. These areas had stone beds of multiple levels and deep enough to allow for the quarrying of good quality dimension stone of two different varieties. There are two main colors of Berea sandstone, buff and a gray blue. This color difference is due to the amount, and type of iron present in the stone. Amherst and Grafton districts are the source of primarily buff stone, while Berea is the primary source of the blue-gray stone. Berea gives its name to all of the Ohio sandstone of this type, regardless of the color, or specific source of the stone. Historically, the stone was found to be resistant to deterioration and used in many public and private buildings, as well as bridges.⁶⁶

⁶⁴ "BOMB WRECKS CANADA PARLIAMENT BUILDING." *Boston Daily Globe (1872-1922)* 4 Feb. 1916 : 1. Print.

⁶⁵ Public Works and Government Services Canada Website- Parliament Hill

⁶⁶ "'BEREA' SANDSTONE." *Stone (1888-1919)* 3.1 (1890): 12. Print.

Current Restoration of Parliament Hill

In 2002, a restoration campaign began under the aegis of Public Works and Government Services Canada, and work continues today. The plan is for a complete restoration, and modernization of the buildings on Parliament Hill over the course of 25 years. The Library of Parliament was the first to be restored and this work has been completed. The three main buildings, the Centre, East and West blocks are the focus of the second major stage of the rehabilitation work.⁶⁷

Work is currently underway on the West Block, where stone for this thesis was sourced. Work on the West Block began in 2011 and is scheduled to be completed in 2017.⁶⁸ While there was damage to the West Block during the 1897 fire, the majority of the building was unscathed, and no damage sustained during the 1916 fire that destroyed the Centre block. The damage seen today is owed more to the exposure of the building stone to de-icing salts, and to the elements. The restoration will include reversal of restoration work done in the 1960's, as well as structural repairs, and new repairs to the exterior stone.^{69 70}

⁶⁷ Public Works and Government Services Canada Website- Parliament Hill

⁶⁸ Government of Canada, Public Works and Government Services Canada. "Rehabilitation of the West Block - Parliament Hill - PWGSC." N. p., 1 Nov. 2011. Web. 29 Apr. 2014.

⁶⁹ The West Block will be used for the Parliament chamber and offices during Centre Block renovations, and the courtyard is to be in-filled with a glass space to serve as the Parliamentary chamber, and the interior of the building completely redone to be made into office spaces.

⁷⁰ Site visit, and guided tour with Kelly Caldwell of Conservation Solutions, Inc., March 18, 2014. See Appendix D for renovation plans.

General Conditions of the Stone and of the Building Stone:

Typical deterioration of Berea sandstone is dependant on the physical and chemical variations resulting from the natural formation of the stone. Bownocker notes that a sandstone with thin or poorly developed bedding planes is useless for creating the large blocks of dimension stone needed for masonry construction.⁷¹ He compares Berea Sandstone to other Ohio sandstones, for instance Euclid sandstone, which is more compact and better for flagstones. All Ohio sandstones can deteriorate from granular disintegration, depending on the environmental conditions. He notes that horizontal cracking along the bedding planes is common in the quarry, and these natural fault lines can be a source of deterioration during the stones service life in a building as stresses force the stone open along these planes. In addition, Bownocker notes that it is rare for vertical cracks to form in the quarry or quarried stone. (100)

The losses and deterioration of the sandstones at Parliament Hill include the breaking of cementitious bonds between the grains making up the sandstones and the loss of cohesive strength of the stone. The compacted granular nature of the Parliament Hill sandstone suggests that deterioration present on the surface of the stone extends into the body of the stone. Since the deterioration does not only involve the surface of the stone, evaluation of the deterioration must include the sub-surface. .

The condition of the existing stone on the West Block building is dependent on its inherent differences of a natural material, as well as its location and cardinal orientation, which dictates its exposure to wind, rain, snow, and the effects of thermal expansion and

⁷¹ Bownocker, J.A., and Geological Survey of Ohio. *Building Stones of Ohio*. Geological Survey of Ohio, 1915. Bulletin (Ohio. Division of Geological Survey).

contraction. Stone on the North and Northeast facing walls of the West Block are subjected to greater environmental forces such as wind, rain, freezing/thawing, wetting and drying, and thermal expansion, than other elevations of the building.

The greatest sources of damage has been the heavy use of de-icing salts, eroding the foundation level of stone, especially at the porte-cochere entrance to the court at the center of the building. In addition, the use of Portland cement based mortar in the 1960's restoration campaign has led to the development of cracks in the stone as the cement had expanded and contracted. Areas of the building repointed, or in some cases rebuilt with Portland cement based mortar show damage from salts introduced into the wall.⁷²

Stones on the building, of all three sandstone types show damage where water, primarily from poor gutter and downspout designs, and wind have eroded their surfaces.

The fire in 1897 damaged portions of the eastern half of the 1865 wing, but the majority of the damage was repaired later that same year. Presently soot from the fire has been cleaned off in the first stages of the West Block renovation work. What remains today is stone that is a different color due to the heat of the fire.

A site visit to the West Block Building, at Parliament Hill, in Ottawa, and visual examination of sandstone at the site showed granular disintegration, erosion, and salt damage being the major sources of deterioration of the stone. Considering the condition of the deteriorated stone, a hypothesis was formed as to the cause of the deterioration. This hypothesis held that the granular disaggregation and weakening of the surface of the stone was a consequence of the destruction of the cementitious bonds between the grains

⁷² Site visit, March 18, 2014.

in the sandstone. As these bonds weaken or disappear, the surface grains are no longer held in the stone matrix, and can be lost as they are acted on by the natural forces in the environment, such as wind, rain, and snow. The bonds are broken, and the grains lost, primarily by the action of low tensile stresses on the stone.

Test Samples

Test samples were obtained from the three wings of the West Block, constructed in 1865, 1875, and 1909. Samples were labeled with the decade years, giving 1860, 1870 and 1900 groups of sample stone. The three stones sampled were all taken from areas of the building that by the original architect's specifications would be built using Berea sandstone from Ohio.⁷³ Figure 6 shows the location of stone removed for samples. The 1860 stone test samples were removed from the foundation level of the building from a stone window surround. This wall faces west, into the present courtyard. This courtyard was first formed by the construction of the 1875 wing which created a u-plan that was finally enclosed with the construction of the 1909 wing.

The 1870 and 1900 stone test samples were both removed from the colonnade level of the building. The 1870 stone test samples were removed from a stone bracket behind a gutter on a corner wall that faced east. The 1900 stone test samples were removed from the base of a column, on a North facing wall.

⁷³ Lawrence, 18

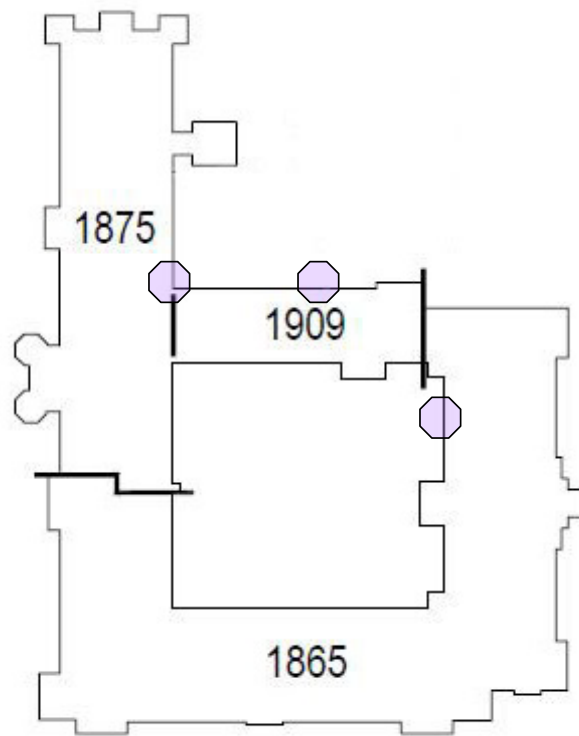


Fig. 6. Image of site plan of the West Block, with sampling locations⁷⁴

Stone Samples Description and Analysis

Stone samples from the building were evaluated using several techniques: X-ray diffraction (XRD), levigation, polarized light microscopy (PLM), scanning electron microscopy (SEM), vacuum porosimetry, and capillary uptake. The sample material used for the tests was unweathered stone from 48mm cores from the West Block building.⁷⁵ Sample stone for the petrographic analysis was selected from the stone not used for consolidation testing, crushed samples were made from unused cut sample discs, and rough sample pieces broken to make small pieces to cut thin sections from.

⁷⁴ Site plan provided by Conservation Solutions, Inc., and sampling locations marked by myself.

⁷⁵ Taken by Conservation Solutions, Inc.

In order to analyze the stone samples using PLM, 30 μ (0.03 mm) thick sections of stone were mounted on glass slides. Small samples of stones were used for scanning electron microscopy. PLM is a microscopic visual examination of the stone using transmitted polarized light, which can be used to identify the minerals present in the stone, as well as the size and shape of the mineral grains.

For both levigation and XRD, stone was crushed to a powder using an agate mortar and pestle. These tests were done on the unconsolidated stone in order to determine the mineralogical characteristics of the three Berea sandstones. XRD is a qualitative way to identify the minerals in a stone that are at or above 1-2% w/w in the stone. Levigation of finely crushed stone samples washed in water separates particles of clay from the quartz grains and other minerals. After separating heavier particles from lighter ones, the levigated sample was used for a second XRD scan of the lighter particles in order to further identify the clay in the stone.

Scanning electron microscopy, which is an electron microscope that produces images of a sample's surface topography and composition, was used on control samples in order to confirm the presence of clay in the stone, and the size of the clay plates, and in consolidated stone to try and locate silica nano-particles and cured consolidant gel. The magnification is high enough to show the smooth clay plate, in comparison to the rougher quartz grains around it. The clay particles are approximately 30 μ in size, which is in the upper range for clay particle sizes (Figure 19). SEM confirmed the presence of clay in the samples, but not the presence of nano-particles or cured gel, likely due to the particles being beyond the range of the microscope and the gel being present in other portions of the samples not examined.

Vacuum porosimetry is a technique that finds the relative pore volume and size by using a vacuum to force liquid into a porous material. The pressure needed to force the liquid into the material, when calculated against the surface tension of the liquid itself, gives information about the percent of the material which is open pore space. The resulting percentage is a volume/volume measurement of the volume of fluid, and the volume of the pore spaces of the material.

Capillary uptake is another stone characteristic that gives information about the internal structure of a porous material. Samples of stone, of the same relative size, are placed in a shallow dish of water, and the observed rate of the upwards transmission of water is tracked. The rate of water uptake in this test provides some indication of the porosity and connectivity of the pore system in the material and allows for the comparison of different materials.

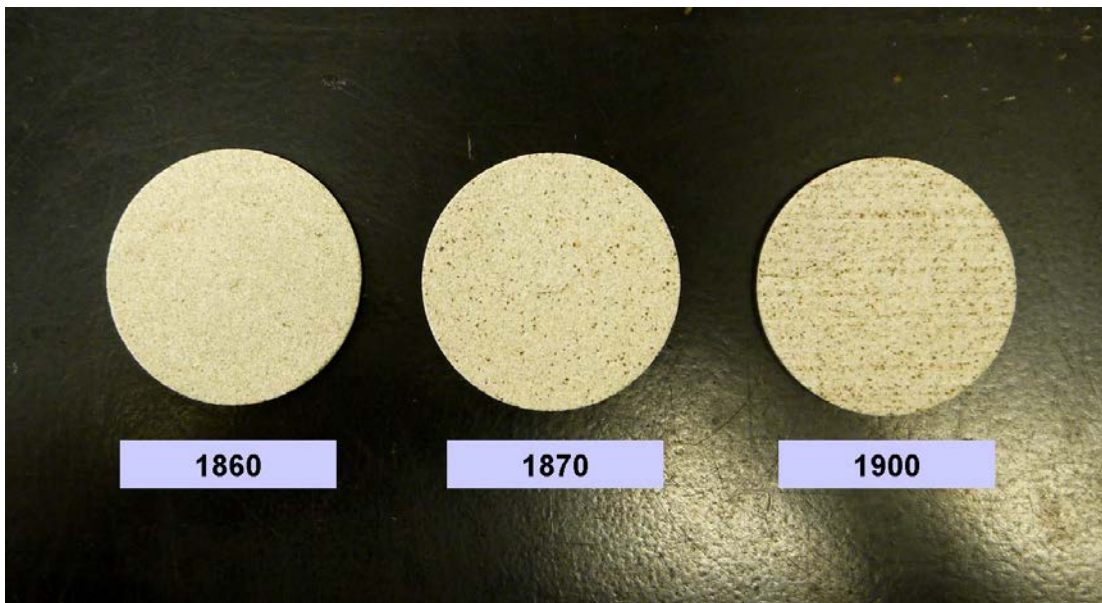


Fig. 7. Sample discs of the three sandstones, with decade of construction. ⁷⁶

⁷⁶ Photo by author.

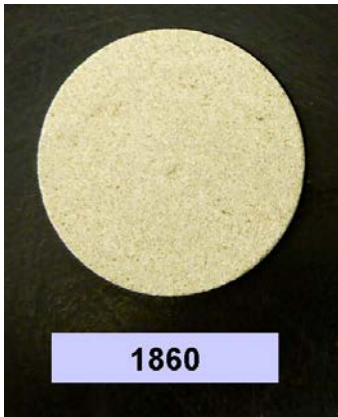


Fig. 8. 1860 Sandstone.⁷⁷

The 1860 series Berea Sandstone from the 1865 wing is a “buff” colored stone.

Visually there are very few dark inclusions and the inclusions are not visible on all samples. There are visible mica flakes throughout the stone samples. The stone is polychromatic within a range of browns with primarily yellowish gray (Munsell Color System 5Y 8/1) particles. The visible inclusions are brown and randomly distributed of moderate brown (5YR 3/4) and dusky brown (5YR 2/2) color. Visually the grains of this stone appear smaller and lighter than the 1870 and 1900 sandstones.

PLM identified the majority of the sandstone as quartz [approximately 55%] and rock fragments [approximately 45%]. The grains are sub-angular and sub-round in shape. There are very few small grains of muscovite present in the sample. (Fig. 9) All of the grains are between 0.06 mm to 0.25 mm in size, on the lower end of the scale for typical sand particles, which range from 0.06 mm to 2 mm.⁷⁸

XRD qualitatively determined the crystalline material present in the stone is primarily quartz (93%), and 1% of the sample is kaolinite. (Fig. 10) Vacuum porosimetry determined the porosity of the stone, and is 16.4% v/v. The capillary uptake rate is

⁷⁷ Photo by author.

⁷⁸ The Scientific American Science Desk Reference, 25.

approximately 0.075 in/min, taking 40 minutes to fully saturate approximately 3 inches of stone.⁷⁹

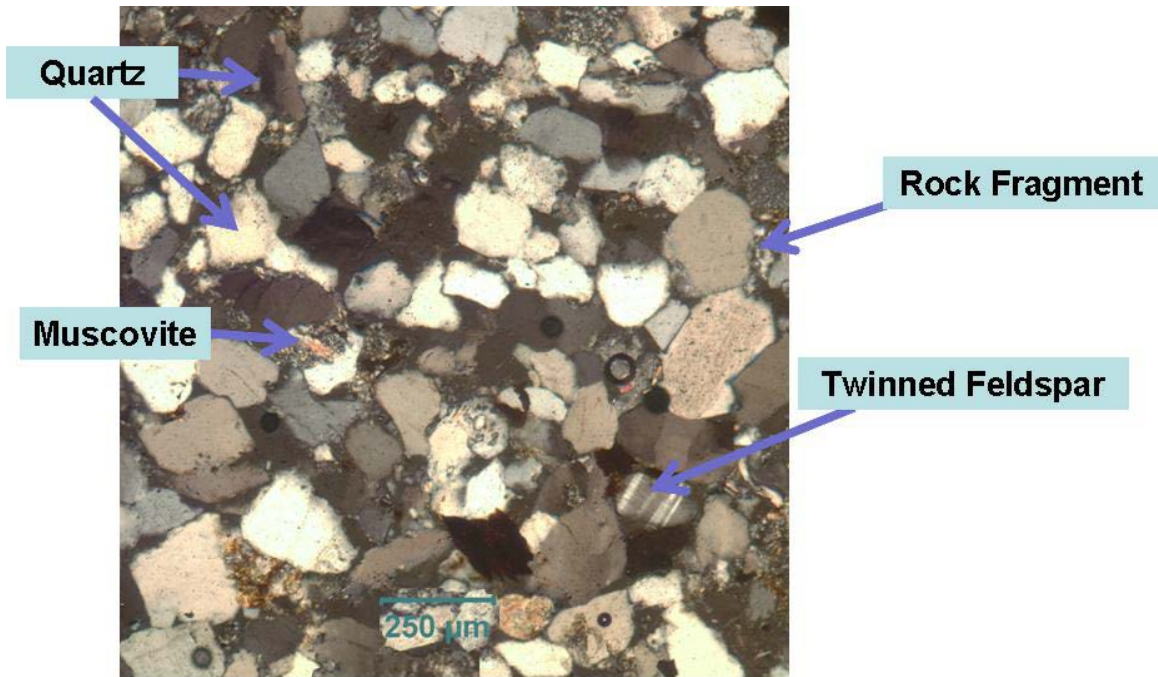


Fig. 9. 1860 Stone under PLM, 100x.⁸⁰

⁷⁹ Please see Appendix B for photos of the capillary uptake test.

⁸⁰ Photo by author.

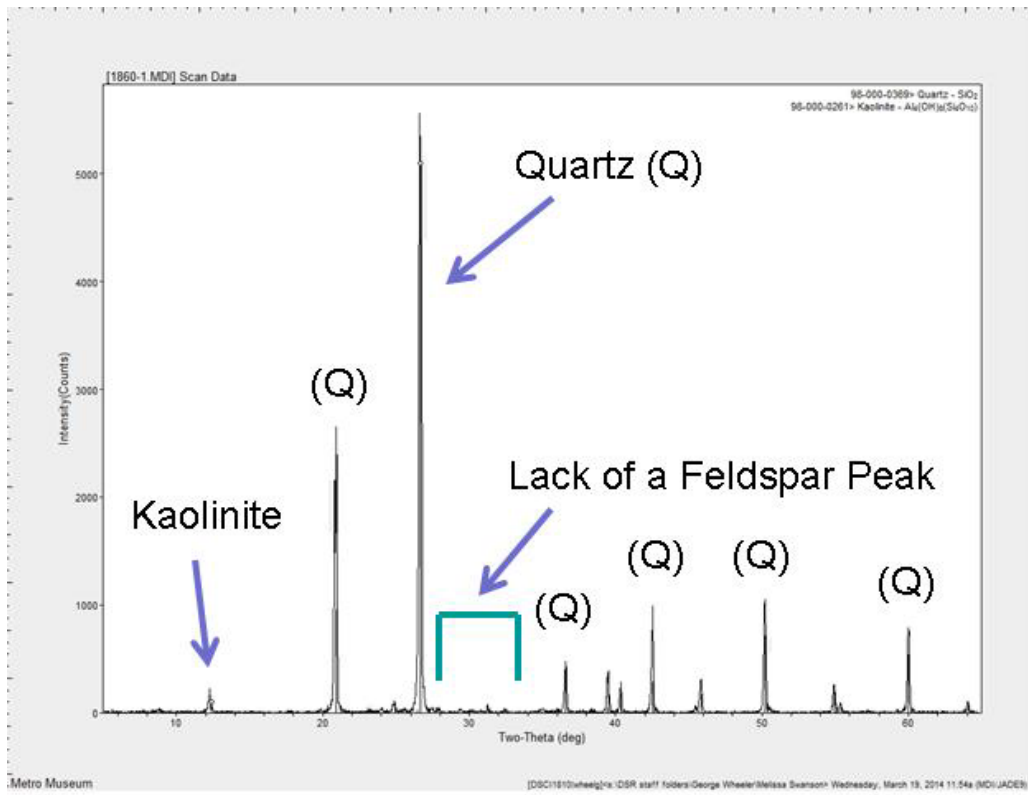


Fig. 10. XRD of 1860 Stone, the short peak to the left is Kaolinite, the other peaks are Quartz, the scale is 0-60 Two-Theta (deg).⁸¹

⁸¹ Photo by author.



Fig. 11. 1870 Sandstone. ⁸²

The 1870 series Berea Sandstone from the 1875 wing is a “buff” colored stone. Visually there are very few dark inclusions and the inclusions are visible on all samples. There are visible mica flakes throughout the stone samples. The stone is polychromatic within a range of browns with primarily grayish yellow (5Y 8/4) particles. The visible inclusions are brown and randomly distributed of moderate brown (5YR 3/4) and dusky brown (5YR 2/2) color. Visually the grains of this stone appear larger than the grains of 1860 stone and slightly lighter than the 1900 stone.

PLM identified the majority of the sandstone as quartz [approximately 57%] and rock fragments [approximately 41%]. The grains are sub-angular and sub-round in shape. There are very few small grains of muscovite present in the sample. (Fig. 12) All the grains are between 0.06 mm to 0.25 mm in size, on the lower end for typical sand particles, which range from 0.06 mm to 2 mm.⁸³

XRD qualitatively determined the crystalline material present in the stone is primarily quartz (100%). (Fig. 13) When a sample of the stone was levigated, or washed

⁸² Photo by author.

⁸³ The Scientific American Science Desk Reference, 25.

to separate the fine, particles, and a second XRD analysis found illite and kaolinite in the stone. (Fig. 14) Vacuum porosimetry determined the porosity of the stone, as 16.4% v/v. The capillary uptake rate of this stone was 0.15 in/min, higher than the 1860 stone, taking 20 minutes to fully saturate approximately 3 inches of stone.

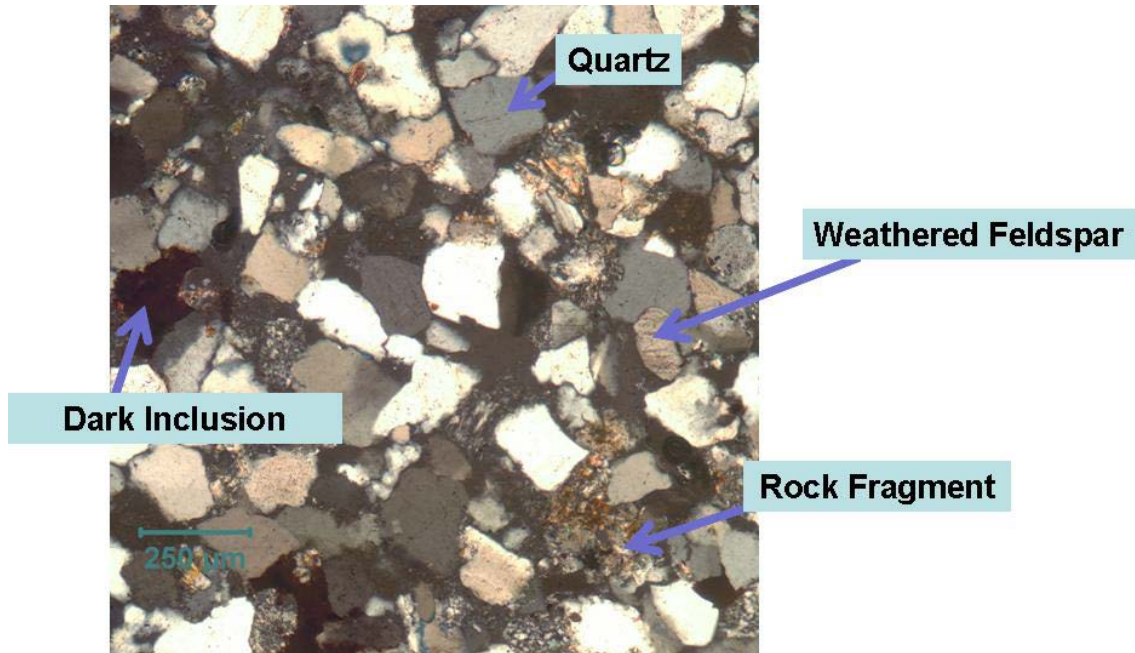


Fig. 12. 1870 Stone under PLM, 50x.⁸⁴

⁸⁴ Photo by author.

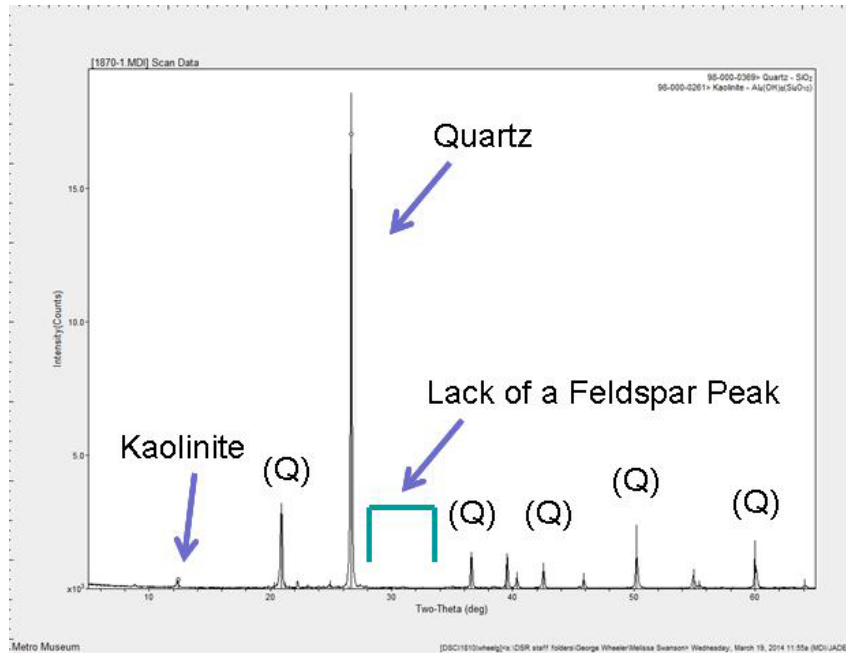


Fig. 13. XRD of 1870 Stone, the short peak to the left is Kaolinite, the other peaks are Quartz, the scale is 0-60 Two-Theta (deg).⁸⁵

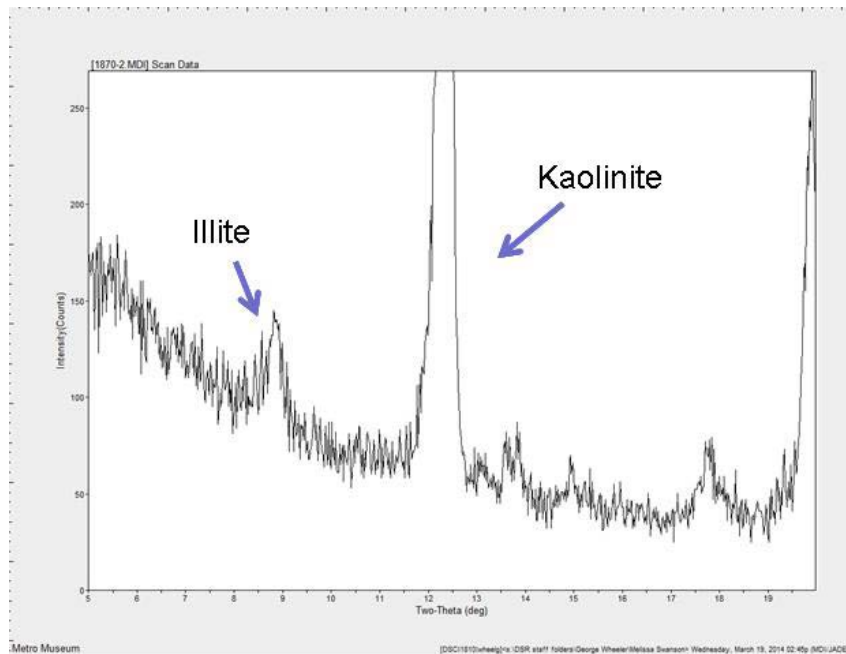


Fig. 14. XRD of 1870 Levigated Stone, the short peak to the left is Illite, another form of clay, the scale is 0-19 Two-Theta (deg).⁸⁶

⁸⁵ Photo by author.

⁸⁶ Photo by author.

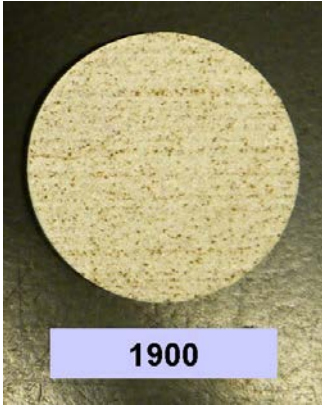


Fig. 15. 1900 Sandstone.⁸⁷

The 1900 series Berea Sandstone from the 1909 wing is a “buff” colored stone.

Visually there are many brown inclusions in the stone organized in striations with strong linear directionality. There are visible mica flakes throughout the stone samples. The stone is polychromatic within a range of browns with primarily grayish yellow (5Y 8/4) particles. The visible inclusions are brown and randomly distributed of moderate brown (5YR 3/4) and dusky brown (5YR 2/2) color. Visually, the grains of this stone appear larger than the grains of 1860 stone and darker than both the 1860 and 1870 stones. This may be due to large number of inclusions in the stone.

PLM identified the majority of the sandstone as quartz [approximately 54%] and rock fragments [approximately 41%]. The grains are sub-angular and sub-round in shape. There are very few small grains of muscovite present in the sample. (Fig. 16) All of the grains are between 0.06 mm to 0.25 mm in size, on the lower end of the scale for typical sand particles, which range from 0.06 mm to 2 mm.⁸⁸

⁸⁷ Photo by author.

⁸⁸ The Scientific American Science Desk Reference, 25.

XRD qualitatively determined that the crystalline material present in the stone is primarily quartz, (approximately 94%). No other mineral was detected. (Fig. 17) The porosity of the stone was determined using vacuum porosimetry, and was found to be 16.8% v/v. The capillary uptake rate of this stone was 0.3 in/min, the fastest of all three stones, taking 10 minutes to fully saturate approximately 3 inches of stone.

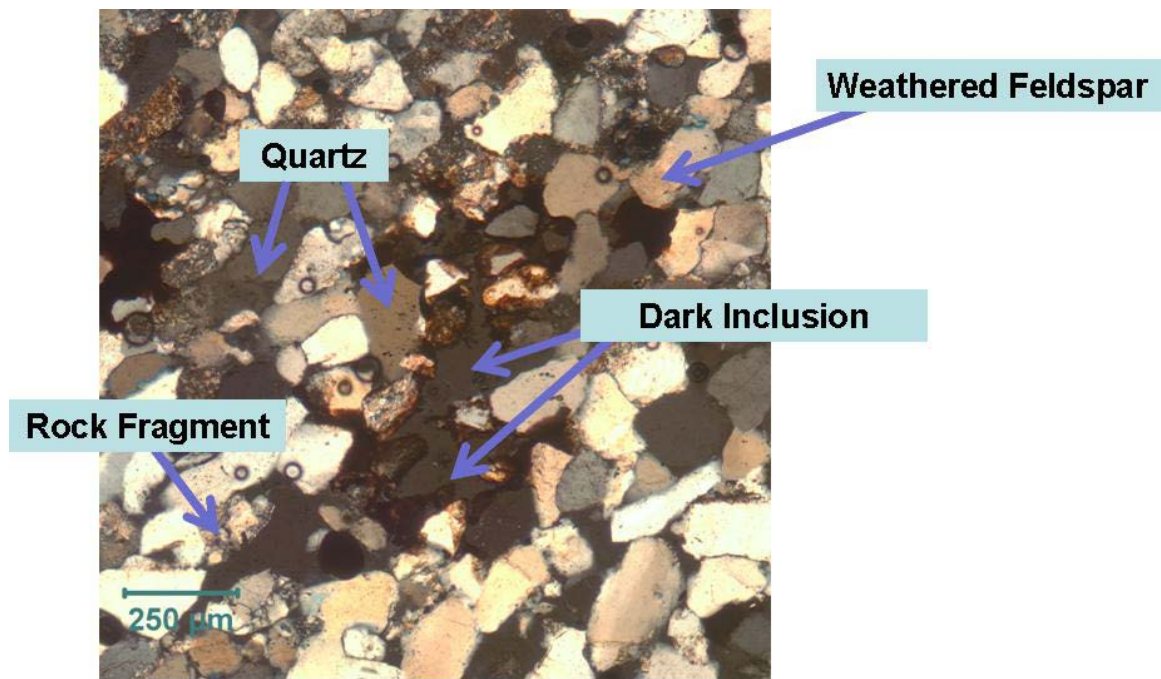


Fig. 16. 1900 Stone under PLM, 50x.⁸⁹

⁸⁹ Photo by author.

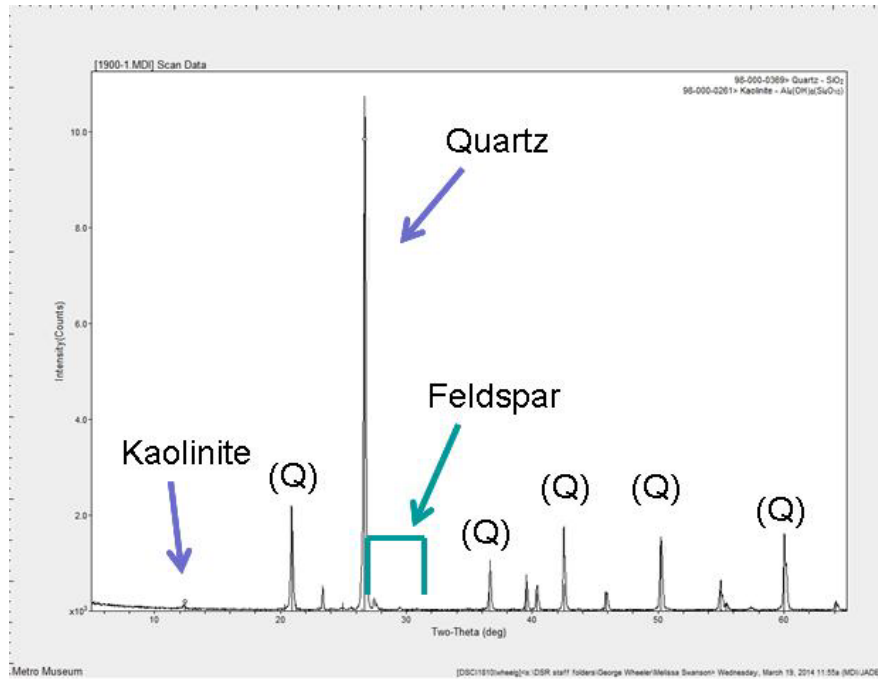


Fig. 17. XRD of 1900 Stone, the short peak to the left is Kaolinite, the other peaks are Quartz, except for the short peak directly to the right of the most prominent peak, this is a very small Feldspar peak. The scale is 0-60 Two-Theta (deg)⁹⁰

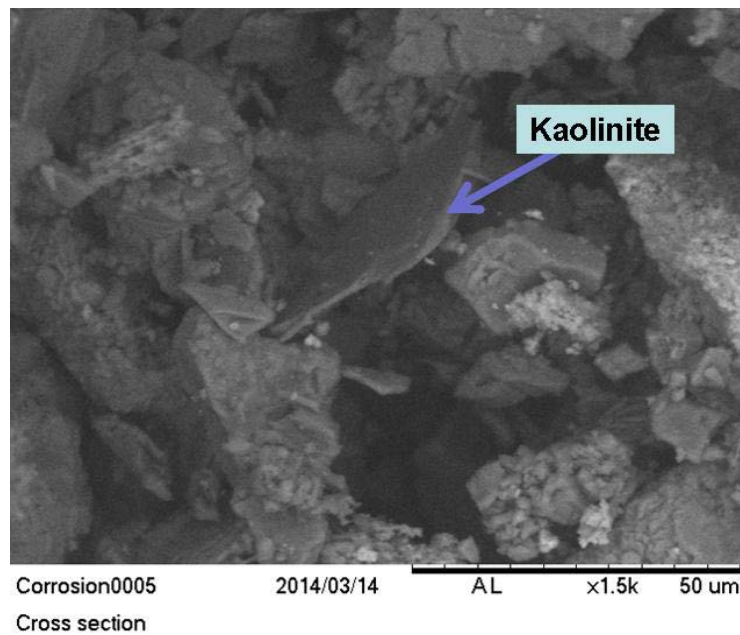


Fig. 18: SEM image of smooth, flat clay plate among quartz grains of the 1900 stone, similar clay plates are present in the other stones.

⁹⁰ Photo by author.

The results of the laboratory testing performed on the three stones from Parliament Hill confirms that they are very similar in terms of their mineralogical composition and physical properties, and supports the historic identification of these stones as Berea sandstone. Figure 19 shows the stones characteristics, organized by series year. The stones have only minor differences, the largest being the capillary uptake rates, which are higher for the 1900 stone. The differences between them can be understood as representative of the variations in a natural material which was quarried in different years, potentially from a different veins, quarries or sections of the same quarry. Variation is to be expected in natural building materials.

	1860	1870	1900
Color	Yellowish Gray 5Y 8/1	Grayish Yellow 5Y 8/4	Grayish Yellow 5Y 8/4
Color of Inclusions	Moderate Brown 5YR 3/4 Dusky Brown 5YR 2/2	Mod. Brown 5YR 3/4 Dusky Brown 5YR 2/2	Mod. Brown 5YR 3/4 Dusky Brown 5YR 2/2
Grain Shape	sub-angular sub-round	sub-angular sub-round	sub-angular sub-round
Grain Size	0.06-0.25 mm	0.06-0.25 mm	0.06-0.25 mm
Minerals	Quartz, feldspar, muscovite	Quartz, feldspar, muscovite	Quartz, feldspar, muscovite
Clay Minerals	Kaolinite	Kaolinite, illite	Kaolinite
Porosity	16.4% v/v	16.4% v/v	16.8% v/v
Capillary Uptake Rate	0.075 in/min	0.15 in/min	0.3 in/min
Other	sparse inclusions	many random inclusions	many inclusions organized in striations

Fig. 19: Chart of Three Berea sandstone characteristics.

Chapter 4: Test Method for Comparative Analysis of Treated Sandstone:

In any treatment of a stone, cleaning or consolidation, the goals of the treatment need to be established beforehand in order to evaluate the success of the treatment after it is completed. For this thesis the experimental design compared the ability of two stone consolidants to increase the flexural strength of the three individual Berea sandstones and retain that strength after undergoing controlled laboratory environmental stress cycles. An increase in the tensile strength of a stone, as measured using the ASTM standard test C1499-09 Standard Test Method for Monotonic Equibiaxial Flexural Strength of Advanced Ceramics at Ambient Temperature (Reapproved 2013), was considered a successful performance.⁹¹ Biaxial flexure testing has been used since the 1980's for the testing of consolidated stone samples.⁹² Tests methods outlined in the ATSM C1499-09 were applied to determine the comparative tensile strength of the stone samples, before and after consolidation.

For the tests, sandstone samples from Parliament Hill were consolidated with OH 100 and 500 STE, cured, and subjected to controlled environmental stress cycles to simulate natural environmental stressing. Then all the discs of consolidated sandstone were tested to their maximum biaxial flexural strength in an Instron 5569A machine with a biaxial flexure apparatus.

⁹¹ Please see Appendix A.

⁹² George Wheeler, personal communication.

ATSM C1499-09 testing provides information on the flexural strength of a material under multiple axes of tensile stresses. The test also measures the deformation of a material as force is applied. Biaxial flexure is useful in testing a stones resistance to small- or medium-scale tensile stresses such as those that affect the cohesiveness of the grains in the stone matrix, causing the granular disintegration deterioration commonly seen on these stones.⁹³

Biaxial Flexure Test Procedure

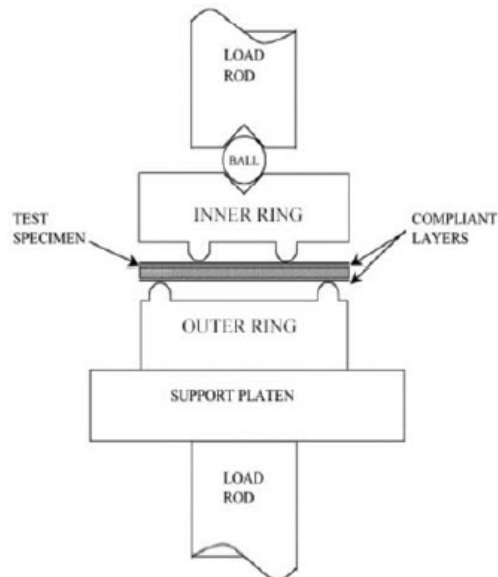


Figure 20: Equibiaxial Testing Rig
Diagram ASTM C1499-1198

An Instron 5569A machine with a biaxial flexure testing rig, using Bluehill software was used for testing the sandstone samples from Parliament Hill. Force was applied with a 5 KN load cell, at a strain rate of 0.01 mm/sec. The rig has both an upper

⁹³ Wheeler, Alkoxysilanes and the Consolidation of Stone., 34

and lower load rod, which support and provide load for the test. The lower load rod supports a platen holding an outer ring die slightly smaller in diameter than the specimen size. Through this ring die, a deflectometer is set up, which measures the deflection of the center point of the test specimen. The upper load rod presses down onto a steel ball and an inner ring die with a diameter smaller than the outer ring die. (Fig. 20)

Once the test specimen is loaded into the machine, the upper load rod lowers, applying force through the inner ring onto the test specimen. The equibiaxial flexure test machine records both the force that is applied to the disc, the deformation of the disc, and its displacement.

Sample Preparation

Core samples were obtained from each construction phase of the West Block. The cores were drilled using a 48mm diameter core drill, and prepared for testing by fabricating sample discs using an Accutom-50 precision cut-off and grinding machine used for thin-sectioning materials such as metal, ceramics and stone. The samples ranged in thickness from approximately 2 to 4 mm. The discs were labeled with a numerical designator; a series comprised all discs cut from the stone from one construction year. Sample year 1860 yielded discs numbering 1-78, 1870 were 1-91, and 1900 were 1-88.

Random selections of approximately 75 discs were taken from each sample series, and completely submerged for five minutes in a glass laboratory dish filled with one of the two consolidants (Fig. 21). The sample discs were removed from the dishes with tweezers laid on metal trays to cure, the two types of consolidated stone kept on different trays. The impregnated discs were cured for 4 weeks, at approximately 22° C and 40% relative humidity, tracked by a HOBOTM data logger.



Fig. 21. During Testing Photo: Sample discs of stone submerged in consolidant. ⁹⁴

Controlled Environmental Stressing

Each dated series was randomly divided into 3 groups: an unconsolidated control group, an OH 100 consolidant group, and a 500 STE consolidant group. The consolidant treated groups were further divided into 3 sets, a control consolidant set, a set subjected to 60 heating/cooling cycles, and a set subjected to 60 freeze/thaw cycles. The heating/cooling controlled environmental stress test subjected the samples to temperatures ranging from 30° C to 55° C. The freeze/thaw controlled environmental

⁹⁴ Photo by author.

stress test subjected the samples to temperatures ranging from -10°C to 22°C . While these ranges are not perfectly analogous to ranges found in Ottawa, the laboratory testing approximates the natural range while still maintaining a reproducible testing program. Knowing the approximate number of freeze/thaw cycles or heating/cooling cycles a year, and the number of cycles through which consolidant is able to maintain its strength, can give a sense of the service life of that consolidant.

For the heating/cooling controlled environmental stress cycles, the samples were placed in a Fisher Isotemp oven, model 106G. The oven was set to a continuous rotating eight hour cycle, four hours on, and then four hours off. The maximum temperature was set for 55°C during heating. When the oven was not actively heating the interior and samples would return to an ambient temperature of approximately 30°C .

For the freeze/thaw controlled environmental stress cycles, the samples were placed in an Excellence brand freezer, model EUF 0580. The freezer was set to a continuous rotating eight hour cycle, four hours on, and then four hours off. The maximum temperature was set for -10°C during cooling. When the freezer was not actively cooling the interior and samples would return to an ambient temperature of approximately 22°C .

Chapter 5: Testing Data and Results:

The data from the Instron machine was put through an equation to convert the results into the stress and strain data, which can be graphed in stress diagrams. For these stress strain diagrams, the x-axis is the measurement of the strain placed on the stone calculated in mm of deformation divided by the size of the sample in mm. The y-axis is the measurement of the stress applied onto the stone in MPa. The modulus of rupture of the stone, is the point at which it can no longer resist the stresses placed on it by the machine, and represents its maximum biaxial flexural strength. This point is at the peak of the graphed lines. A higher peak indicates a higher maximum biaxial flexural strength.

The testing in this thesis used a modification of ASTM standard test C1499-09 Standard Test Method for Monotonic Equibiaxial Flexural Strength of Advanced Ceramics at Ambient Temperature (Reapproved 2013). For this thesis, samples were tested to this first point of failure and slightly beyond but, not to complete fracture of the sample discs. Sandstone is not a completely rigid materials and testing slightly beyond the modulus of rupture gives information about the cohesiveness of the material after that break.⁹⁵ The stress/strain diagrams for each sample group or set in this thesis were created using an average maximum biaxial flexure strength calculated data obtained from 6 to 8 samples. Changes in the maximum biaxial flexural strength of the stone were calculated using percent change, from before and after consolidation, and before and after environmental stress cycling.

⁹⁵ The Berea sandstone tested here is more cohesive after the fracture point is reached, compared to stones such as granite or marble where there is a steep drop off in strength after the fracture point.

Results

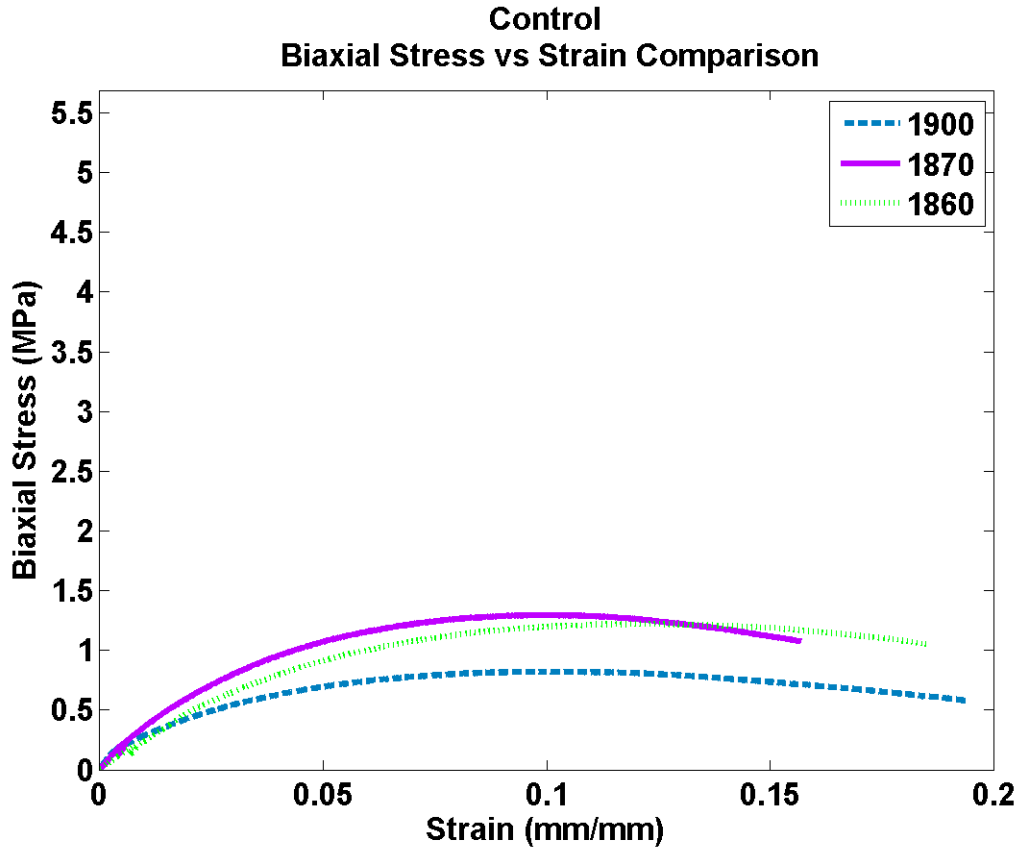


Fig. 22. Biaxial Flexure Data for Stress/Strain, Untreated and Unstressed (Control) Berea Sandstones

Figure 22 shows the biaxial flexure stress/strain curves for three untreated Berea sandstone samples one each from 1860, 1870, and 1900. The peak of these curves is the maximum biaxial flexural strength (henceforth stated as the maximum strength) of the stone or the biaxial flexure modulus of rupture, where the stone can no longer resist cracking under the force applied. The three stones tested are very similar in maximum strength. These stones are the stone samples taken from the building, where the three stones, as seen in the site visit, exhibit different degrees of deterioration. The 1900 stone is the lowest in strength of the three stones, which could be related to observed characteristics, or physical properties such as the a measurable higher capillary uptake.

1860 Control and Environmental Stress Cycled Biaxial Stress vs Strain Comparison

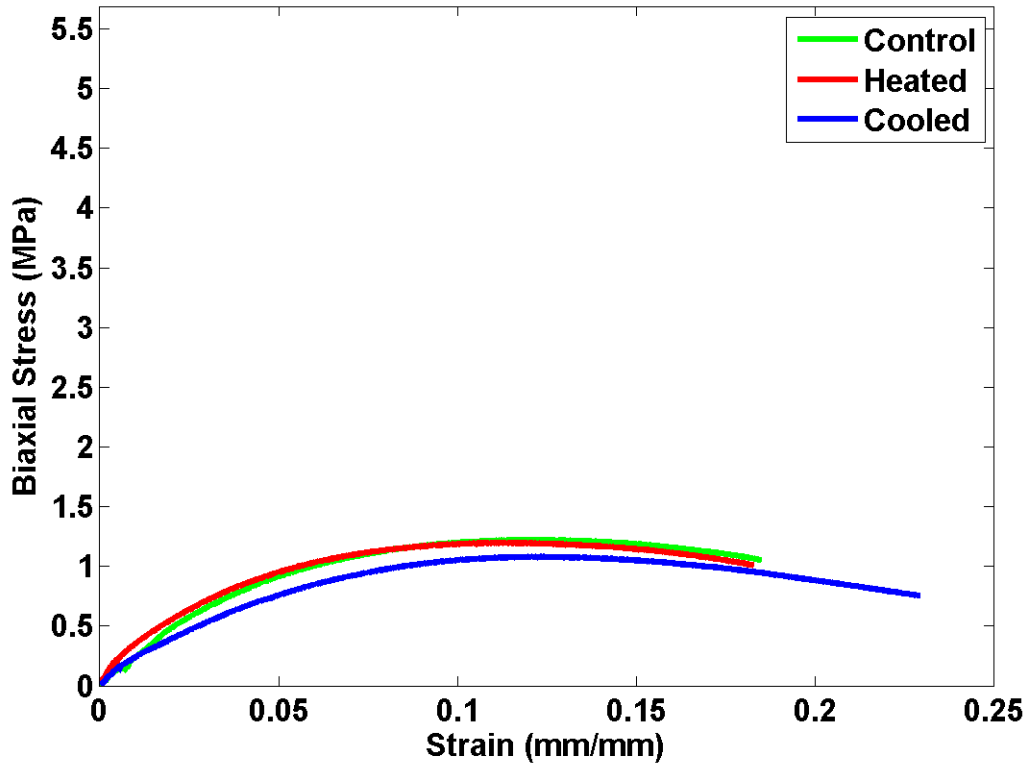


Fig. 23. Biaxial Flexure Data for Stress/Strain Graph, 1860 Control Stone, and Environmentally Stressed Control Stone

Figure 23 shows the biaxial flexure stress/strain curves for three 1860 Berea sandstone samples: An unstressed control stone shown in green; a heating/cooling stress cycled stone shown in red; and a freeze/thaw stressed cycled stone shown in blue. There was little difference in strength between the control and the hot or cold stressed stone. The maximum strength of the stone samples subjected to controlled heating/cooling environmental stress cycling were 3% less than the average maximum strength of the control samples. The average maximum strength of the stone samples subjected to controlled freeze/thaw environmental stress cycling were 18.5% less than the average maximum strength of the control.

1870 Control and Environmental Stress Cycled Biaxial Stress vs Strain Comparison

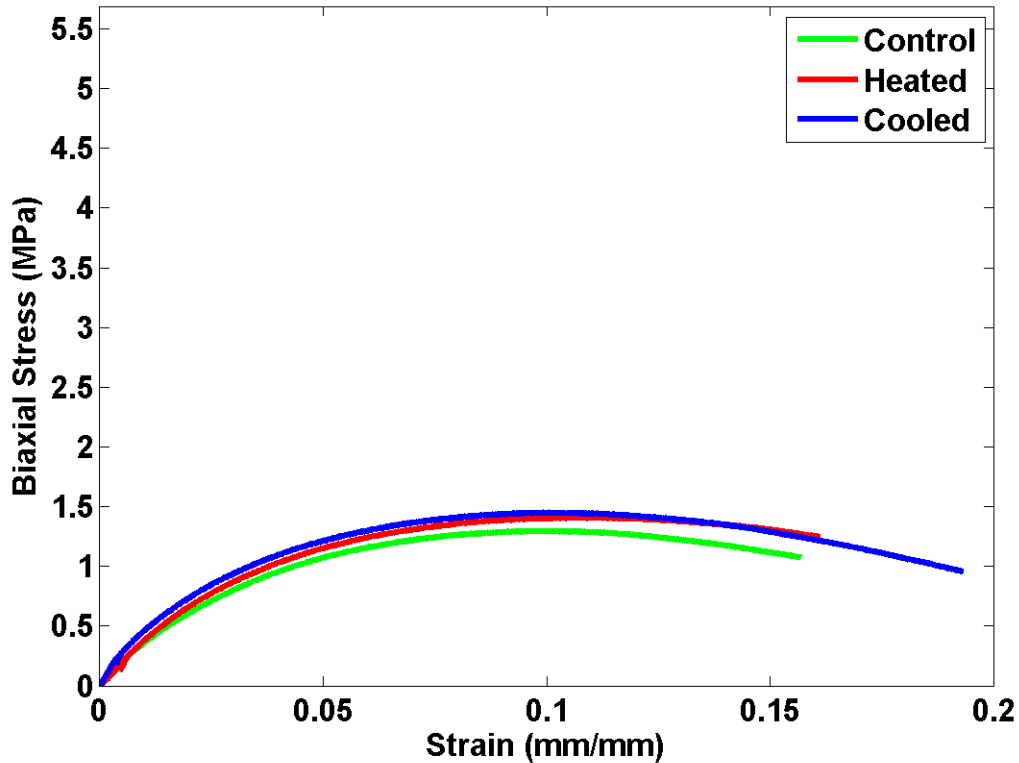


Fig. 24. Biaxial Flexure Data for Stress/Strain Graph, 1870 Control Stone, and Environmentally Stressed Control Stone

Figure 24 shows the biaxial flexure stress/strain curves for three 1870 Berea sandstone samples: An unstressed control stone shown in green; a heating/cooling stress cycled stone shown in red; and a freeze/thaw stressed cycled stone shown in blue. There was little comparable change in maximum strength between the control samples, and the environmentally stressed stone samples, either with heating/cooling or freeze/thaw. The maximum strength of the stone samples subjected to controlled heating/cooling environmental stress cycling were 3% higher than the control. The maximum strength of the stone samples subjected to controlled freeze/thaw environmental stress cycling were 6% higher than the control.

1900 Control and Environmental Stress Cycled Biaxial Stress vs Strain Comparison

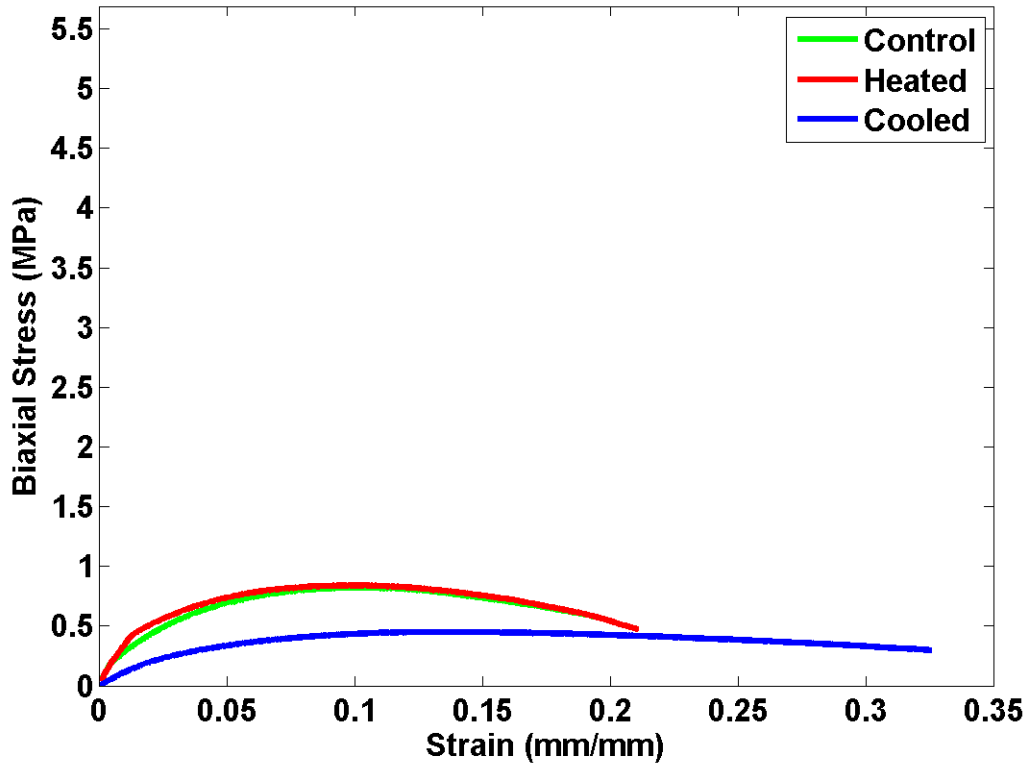


Fig. 25. Biaxial Flexure Data for Stress/Strain Graph, 1900 Control Stone, and Environmentally Stressed Control Stone

Figure 25 shows the biaxial flexure stress/strain curves for three 1900 Berea sandstone samples: An unstressed control stone shown in green; a heating/cooling stress cycled stone shown in red; and a freeze/thaw stressed cycled stone shown in blue. There was little change measured between the stone samples subjected to controlled heating/cooling stresses and the control stone samples. There was however, a significant change in the stone samples subjected to controlled freeze/thaw stress cycling. The maximum biaxial strength of the stone samples subjected to controlled heating/cooling environmental stress cycling were 6% higher than the control samples. The maximum biaxial strength of the stone samples subjected to controlled freeze/thaw environmental stress cycling were 44% less than the control, a loss of almost half of the stone's strength.

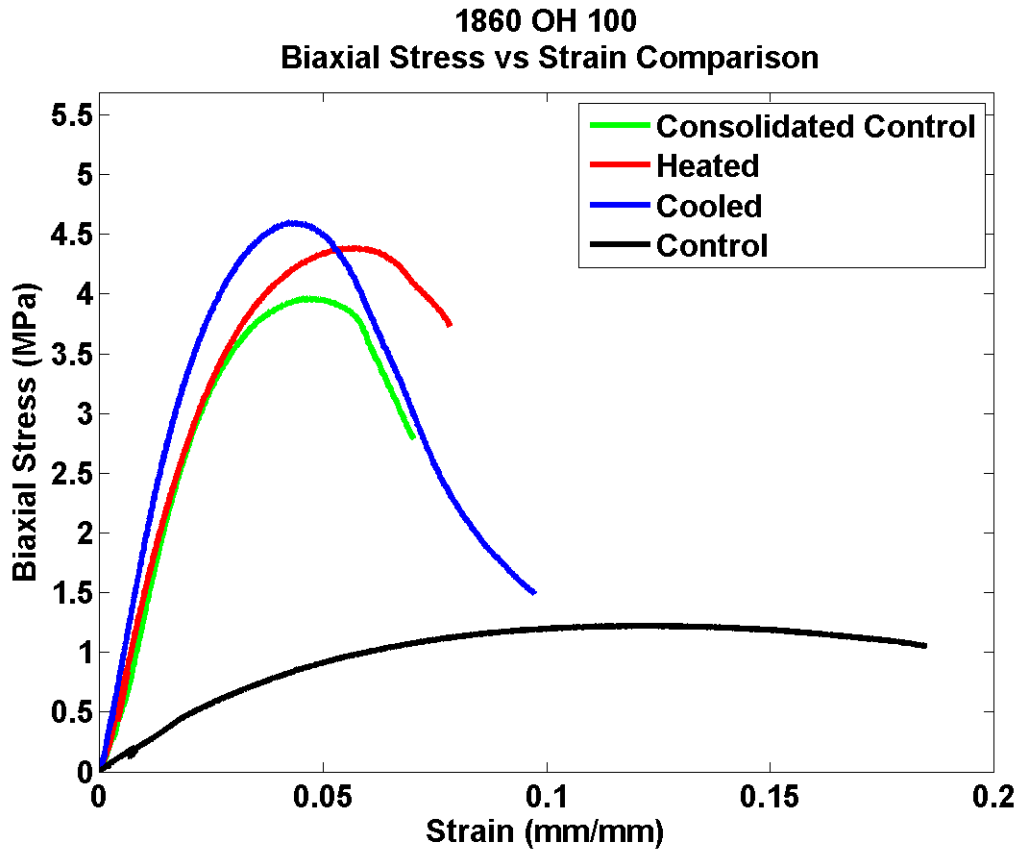


Fig. 26. Biaxial Flexure Data for Stress/Strain Graph, 1860 Control Stone, OH 100 Consolidated Stone and Environmentally Stressed Consolidated Stone

Figure 26 shows the biaxial flexure stress/strain curves for three 1860 Berea sandstone samples after consolidation with OH 100: An unstressed consolidated control stone shown in green; a heating/cooling stress cycled consolidated stone shown in red; and a freeze/thaw stressed cycled consolidated stone shown in blue. In addition, an untreated control is also shown. The consolidated control stone samples had a 204% higher maximum strength than the control stone samples. The average maximum strength of the stone samples subjected to controlled heating/cooling environmental stress cycling were 10% higher than the consolidated control stone samples. The maximum strength of the stone samples subjected to controlled freeze/thaw environmental stress cycling were 11% higher than the maximum strength of the consolidated control stone samples.

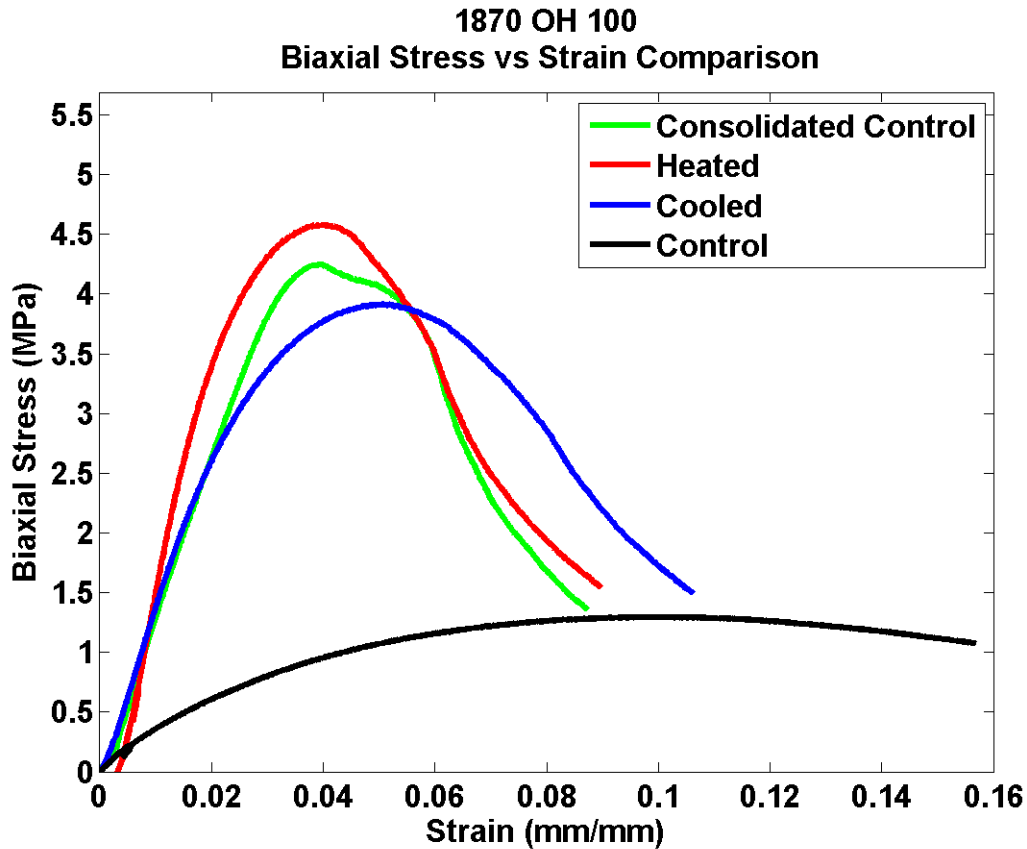


Fig. 27. Biaxial Flexure Data for Stress/Strain Graph, 1870 Control Stone, OH 100 Consolidated Stone and Environmentally Stressed Consolidated Stone

Figure 27 shows the biaxial flexure stress/strain curves for three 1870 Berea sandstone samples after consolidation with OH 100: An unstressed consolidated control stone shown in green; a heating/cooling stress cycled consolidated stone shown in red; and a freeze/thaw stressed cycled consolidated stone shown in blue. In addition, an untreated control is also shown. The consolidated control stone samples had a 173% higher maximum strength than the control stone samples. The average maximum strength of the stone samples subjected to controlled heating/cooling environmental stress cycling were 15% higher than the consolidated control stone samples. The maximum strength of the stone samples subjected to controlled freeze/thaw environmental stress cycling were 7% lower than the maximum strength of the consolidated control stone samples.

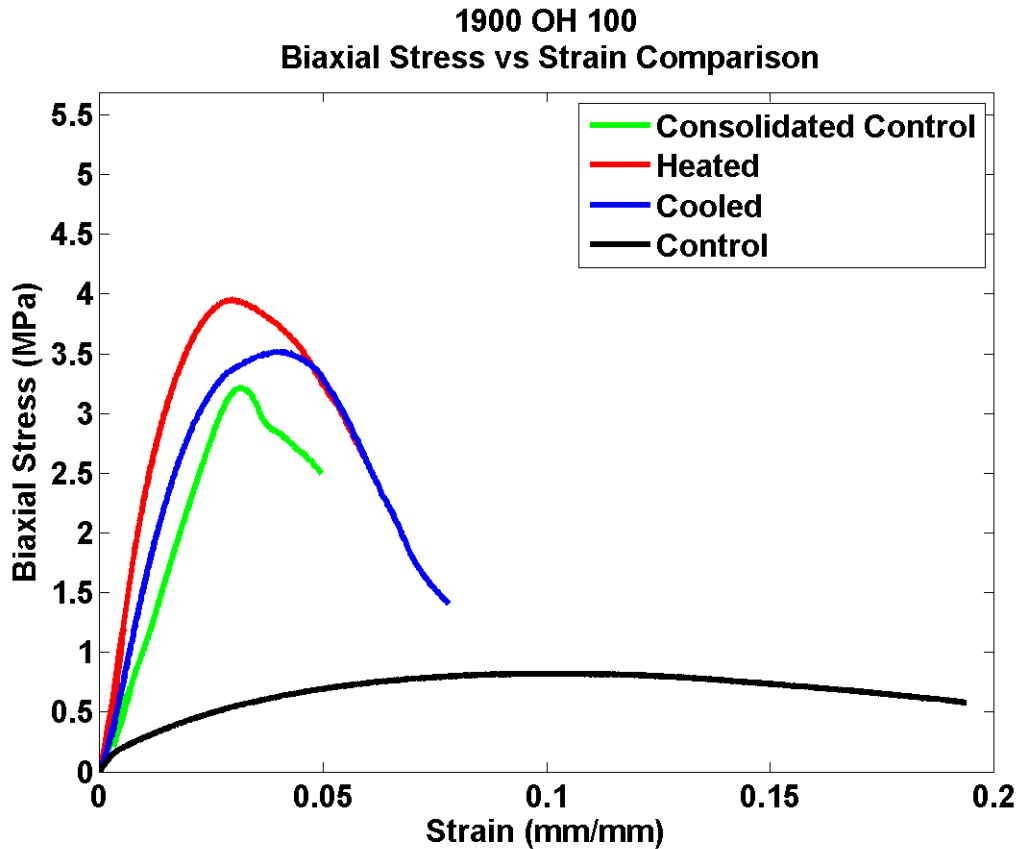


Fig. 28. Biaxial Flexure Data for Stress/Strain Graph, 1900 Control Stone, OH 100 Consolidated Stone and Environmentally Stressed Consolidated Stone

Figure 28 shows the biaxial flexure stress/strain curves for three 1900 Berea sandstone samples after consolidation with OH 100: An unstressed consolidated control stone shown in green; a heating/cooling stress cycled consolidated stone shown in red; and a freeze/thaw stressed cycled consolidated stone shown in blue. In addition, an untreated control is also shown. The consolidated control stone samples had a 305% higher maximum strength than the control stone samples. The average maximum strength of the stone samples subjected to controlled heating/cooling environmental stress cycling were 10% higher than the consolidated control stone samples. The maximum strength of the stone samples subjected to controlled freeze/thaw environmental stress cycling were 2% higher than the maximum strength of the consolidated control stone samples.

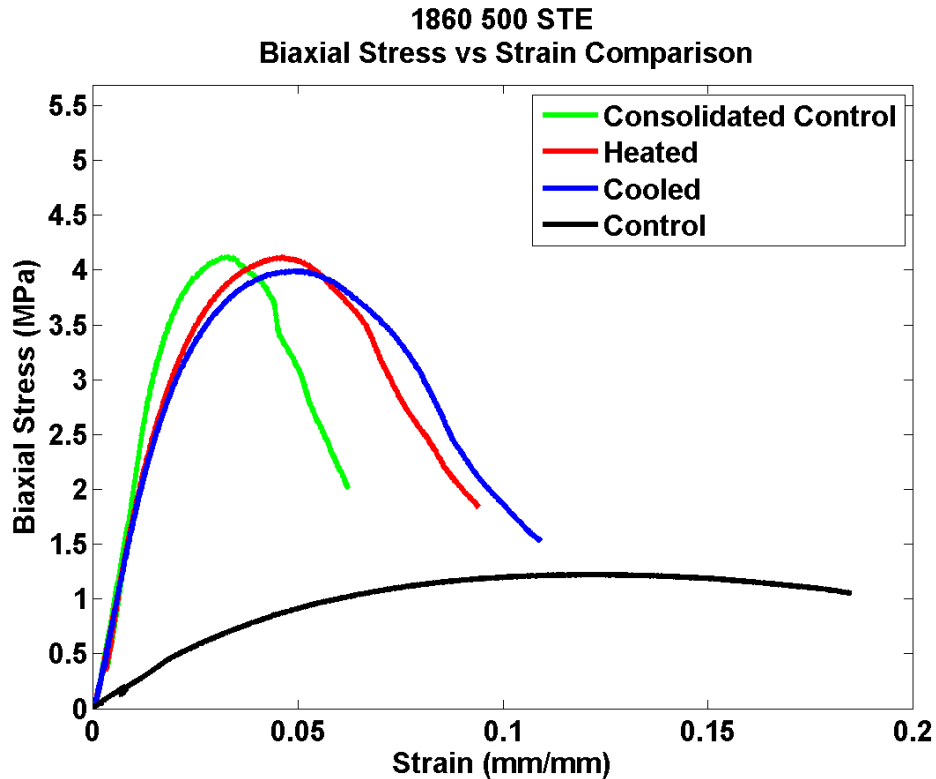


Fig. 29. Biaxial Flexure Data for Stress/Strain Graph, 1860 Control Stone, 500 STE Consolidated Stone and Environmentally Stressed Consolidated Stone

Figure 29 shows the biaxial flexure stress/strain curves for three 1860 Berea sandstone samples after consolidation with 500 STE: An unstressed consolidated control stone shown in green; a heating/cooling stress cycled consolidated stone shown in red; and a freeze/thaw stressed cycled consolidated stone shown in blue. In addition, an untreated control is also shown. The consolidated control stone samples had a 215% higher maximum strength than the control stone samples. The maximum strength of the stone samples subjected to controlled heating/cooling environmental stress cycling were 6% lower than the modulus of rupture for the consolidated control stone samples. The maximum strength of the stone samples subjected to controlled freeze/thaw environmental stress cycling were 10% lower than the consolidated control stone samples.

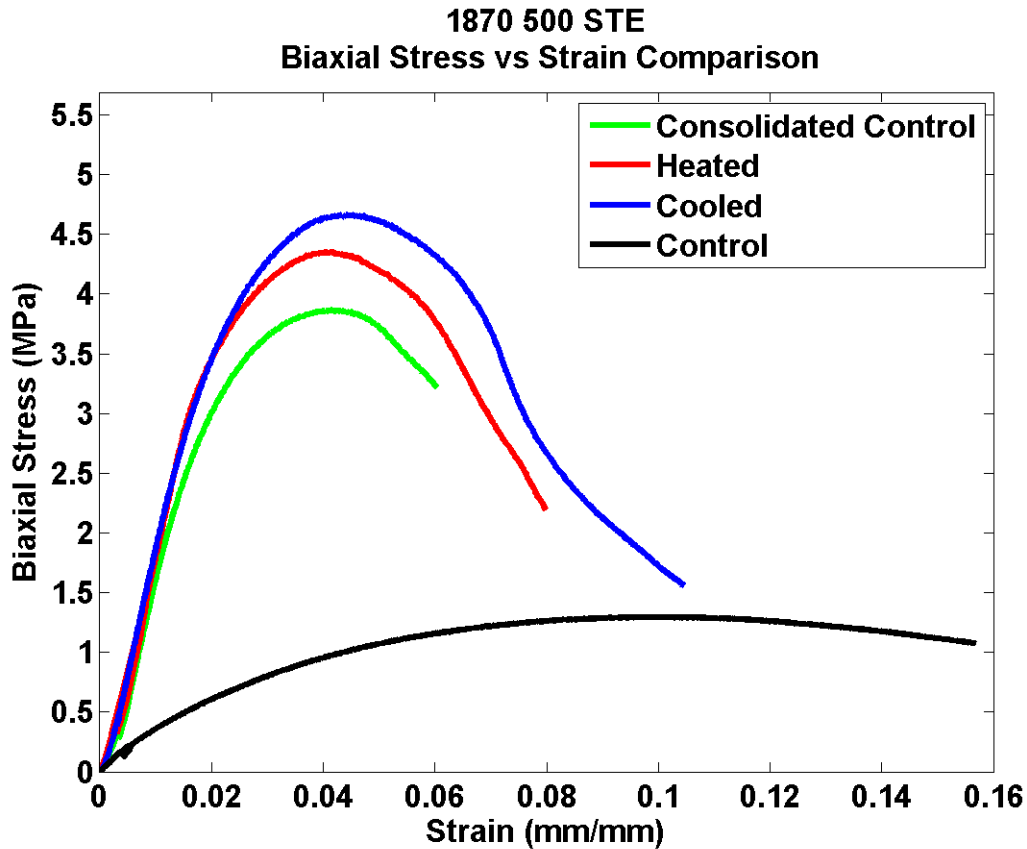


Fig. 30. Biaxial Flexure Data for Stress/Strain Graph, 1870 Control Stone, 500 STE Consolidated Stone and Environmentally Stressed Consolidated Stone

Figure 30 shows the biaxial flexure stress/strain curves for three 1870 Berea sandstone samples after consolidation with 500 STE: An unstressed consolidated control stone shown in green; a heating/cooling stress cycled consolidated stone shown in red; and a freeze/thaw stressed cycled consolidated stone shown in blue. In addition, an untreated control is also shown. The consolidated control stone samples had a 153% higher maximum strength than the control stone samples. The maximum strength of the stone samples subjected to controlled heating/cooling environmental stress cycling were 10% higher than the maximum strength of the consolidated control stone samples. The maximum strength of the stone samples subjected to controlled freeze/thaw environmental stress cycling were 18% higher than the consolidated control.

1900 500 STE
Biaxial Stress vs Strain Comparison

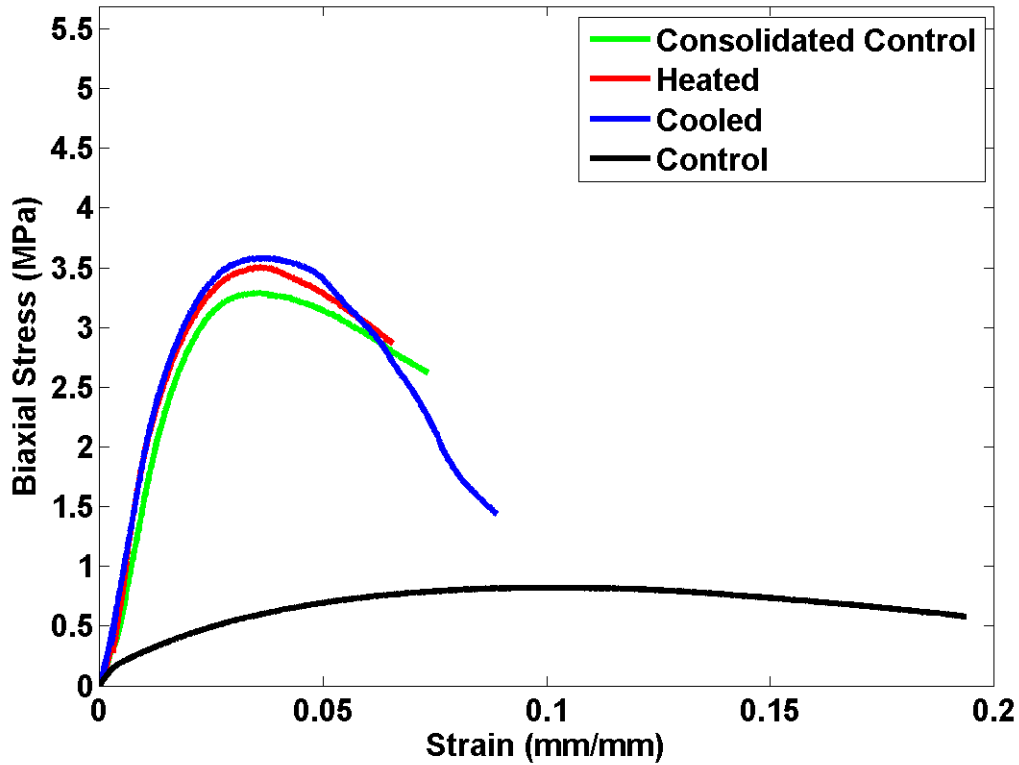


Fig. 31. Biaxial Flexure Data for Stress/Strain Graph, 1870 Control Stone, 500 STE Consolidated Stone and Environmentally Stressed Consolidated Stone

Figure 31 shows the biaxial flexure stress/strain curves for three 1900 Berea sandstone samples after consolidation with 500 STE: An unstressed consolidated control stone shown in green; a heating/cooling stress cycled consolidated stone shown in red; and a freeze/thaw stressed cycled consolidated stone shown in blue. In addition, an untreated control is also shown. The consolidated control stone samples had a 282% higher maximum strength than the control stone sample. The maximum strength of the stone samples subjected to controlled heating/cooling environmental stress cycling were 5% higher than the maximum strength for the consolidated control stone samples. The maximum strength of the stone samples subjected to controlled freeze/thaw environmental stress cycling were 11% higher than the consolidated control.

Analysis

In the 1860 sample series, controlled freeze/thaw environmental stress cycling lowered the maximum biaxial flexure strength of the control stone samples 19%, while the stone samples exposed to controlled heating/cooling environmental stress cycling measured lost an insignificant 3% of total maximum strength compared to the control stone samples.

The 1870 control stone sample series when exposed to controlled freeze/thaw environmental stress cycling did not show significant change in the maximum strength compared to the control stone samples, increasing 7%, while the stone samples exposed to controlled heating/cooling environmental stress cycling decreased 17 % in maximum strength as compared to the control stone samples.

The 1900 stone sample series had the lowest inherent strength of the three stones tested, with the average maximum flexural strength of this stone approximately 50% less than that of the 1860 or 1870 stone samples. When exposed to controlled heating/cooling environmental stress cycling the maximum strength of the 1900 control stone decreased by 6%, but when exposed to controlled freeze/thaw environmental stress cycling the 1900 control samples decreased in strength 44%.

All of the consolidated stone samples had an initial increase of strength after consolidation, and regardless of the effects of controlled environmental stress cycles on the consolidated stone, both consolidants initially worked as intended, increasing the tensile strength of the stone.

Chapter 6: Comparative Analysis:

The silica nano-particles added to 500 STE, as well as the elastification of the consolidant polymers are intended to provide strength retention in the cured consolidant allowing better retention of any gained strength through freezing weather experienced by the consolidant in the field. While freeze/thawing is not the only environmental stress that a stone experiences in its life in a building, it is understood to be the one where consolidation treatments will crack or fail.

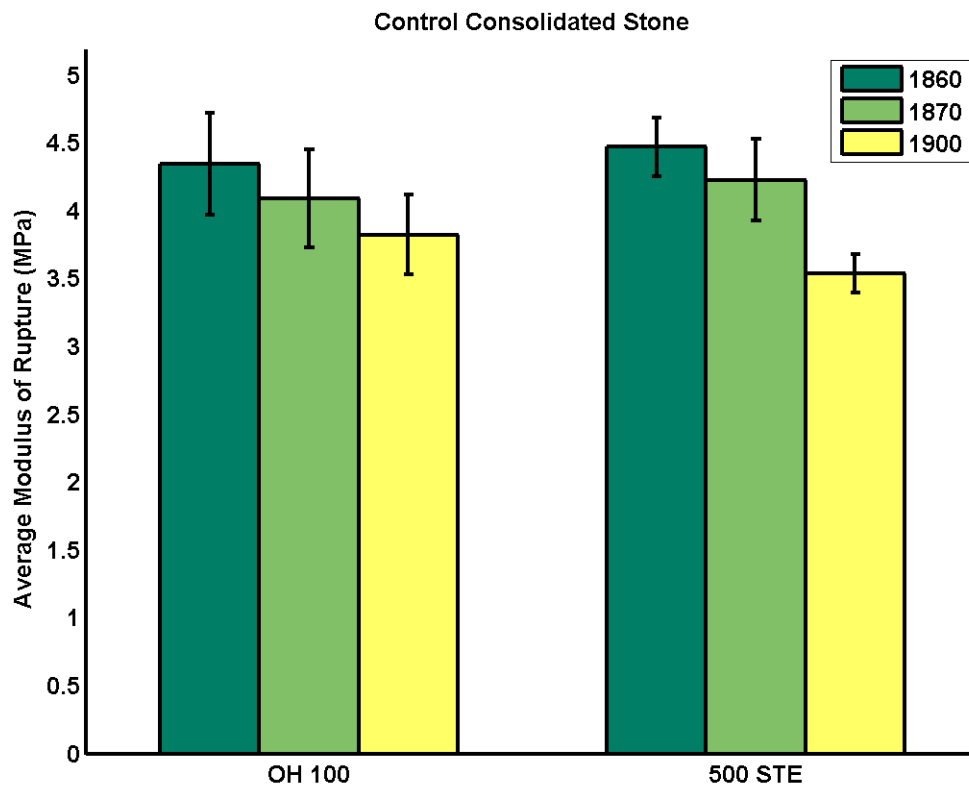


Fig. 32. Average Maximum Strength in Control Samples, and Control Consolidated Samples

Based on the results of the testing, there is a small measurable difference between the maximum strength gained using the two consolidants on these three sandstones even

after 60 cycles of controlled environmental stressing. (Fig. 32) Overall, all of the Berea sandstone samples treated with OH 100 had a (~ 4 %) higher initial strength gain compared to the Berea sandstone consolidated with 500 STE.

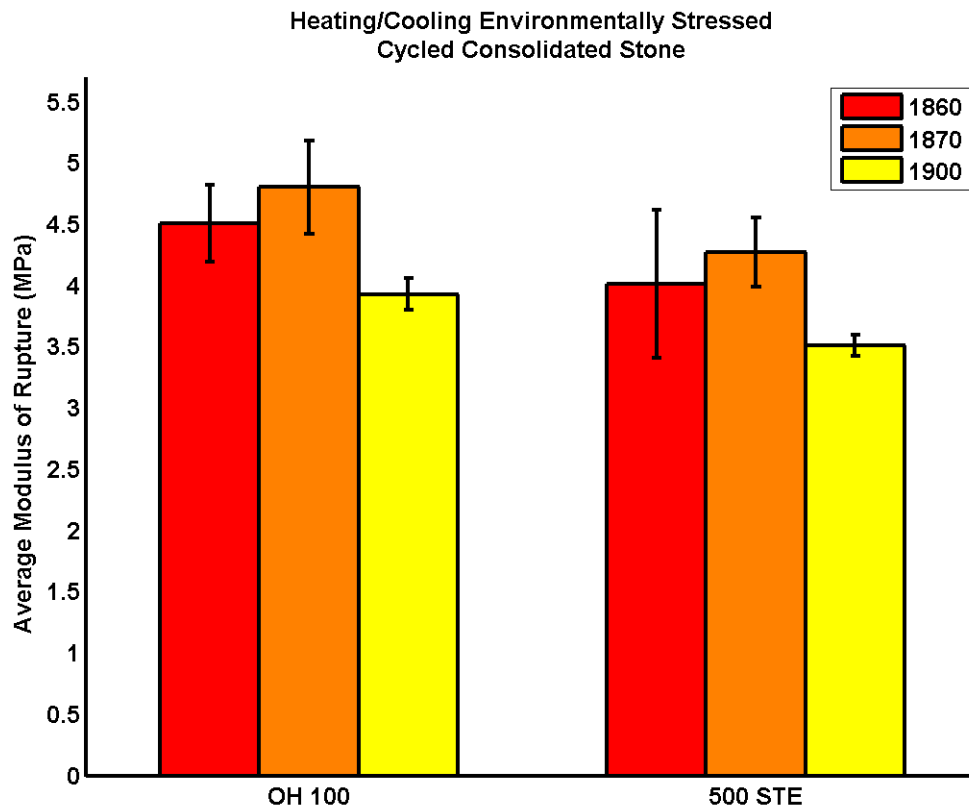


Fig. 33. Average Maximum strength in Heating/Cooling Environmentally Stress Cycled Control, and Consolidated Samples

A comparison of the performance of OH 100 and 500 STE in terms of the changes to the maximum strength after either controlled freeze/thaw or heating/cooling environmental stress cycling shows that the OH 100 consolidant provides a marginally higher retention of strength, however the margin of error in this testing is significant enough to make the difference between the two insignificant.

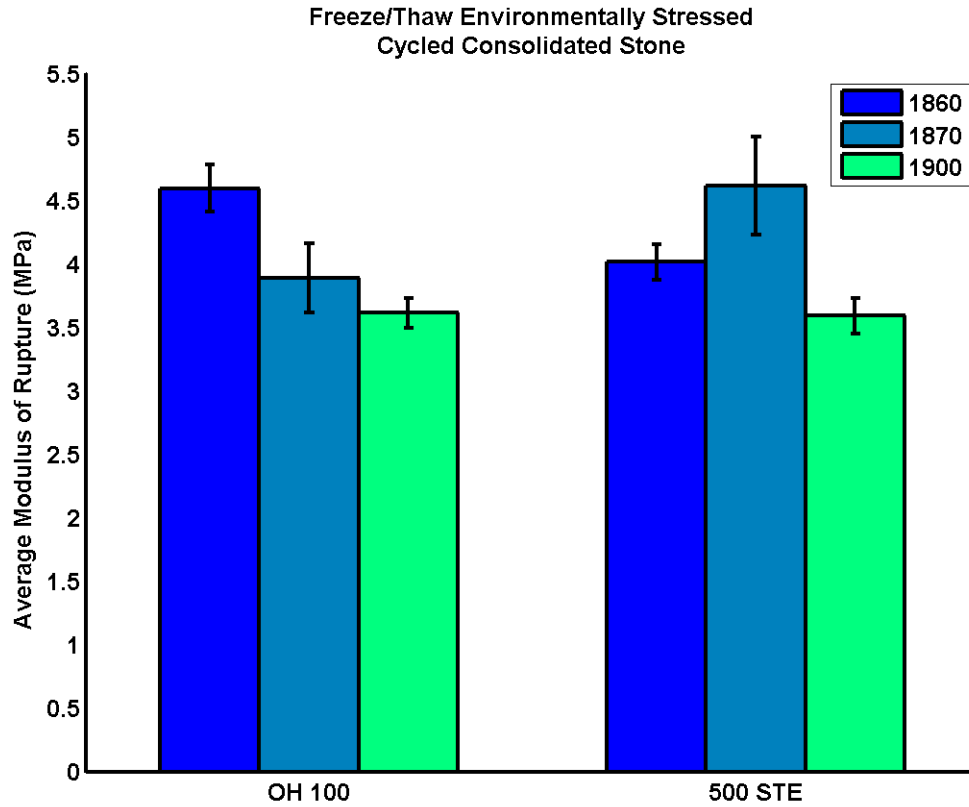


Fig. 34. Average Maximum strength in Freeze/Thaw Environmentally Stress Cycled Control, and Consolidated Samples

The controlled freeze/thaw controlled environmental testing cycles were expected to have the most affect on the consolidated stone. The results of the testing showed that the Berea sandstone from the 1870 sample series was the least affected by the freeze/thaw stresses and that the 1900 stone exhibited the lowest resultant maximum biaxial flexure strength regardless of the consolidant applied. (Fig. 34) The results of the freeze/thaw environmental stress testing showed that the Berea sandstone from the 1860 sample series stone consolidated with OH 100 retained its strength more than those consolidated with 500 STE. However with 1870 samples series, the stone consolidated with 500 STE retained more strength after testing. With the 1900 samples series, the difference between the strength of stone consolidated with the two consolidants is nominal.

Comparative Analysis of the Consolidated Sample Sets

Based on the results of the testing, there is little difference between the maximum strength gained using the two consolidants on these three sandstones even after 60 cycles of controlled environmental stressing. A comparison of the performance of OH 100 and 500 STE in terms of the changes to the maximum strength after either controlled heating/cooling or freeze/thaw environmental stress cycling shows minimal measureable differences. All of the Berea sandstone samples treated with OH 100 showed an initially higher maximum strength retention after controlled heating/cooling compared to the stone samples consolidated with 500 STE and subjected to the heating/cooling environmental stress testing. However, all consolidated stone retained their strength gain through 60 cycles of the heating/cooling environmental stressing.

The largest measurable differences using biaxial flexure testing as an indication of tensile strength in the stone samples was between the performance of the OH 100 and 500 STE in the 1860 and 1870 consolidated samples subjected to freeze/thaw environmental stress cycling. The 1860 stone sample consolidated with either OH 100 or 500 STE show similar initial strength gains for all three sandstones, increasing the stone by a factor in a range between 2.5 to 4, after consolidation. However 1860 stone samples subjected to freeze/thaw controlled environmental stress cycling, consolidated with OH 100 retained a maximum strength of the consolidant lower than the maximum biaxial strength of the 1860 sample consolidated with 500 STE and subjected to the same freeze/thaw environmental stress cycling, approximately 15% lower. The 1870 stone sample consolidated with either OH 100 or 500 STE show similar initial strength gains for all three sandstones, increasing the stone by a factor in a range between 2.5 to 4, after

consolidation. However 1870 stone samples subjected to freeze/thaw controlled environmental stress cycling, consolidated with OH 100 retained a maximum strength of the consolidant higher than that of the same stone treated with 500 STE, of approximately 19%. The 1900 consolidated stone samples performed equally well through after freeze/thaw controlled environmental stress cycling whether consolidated with OH 100 or 500 STE.

The greatest difference between the two consolidants was in the performance of the 1870 consolidated stone samples weathered in freeze/thaw controlled environmental stress cycling. The 500 STE treated stone samples increased in maximum strength after controlled environmental stress cycles, except for a minor loss of strength in the 1860 stone, under both types of environmental stressing. The OH 100 added strength in all but one of the sample categories, 1870 series stone consolidated with OH 100 showed a loss in strength from the control sample stone treated with OH 100 to the freeze/thaw environmentally stressed stone.

Both OH 100 and 500 STE performed similarly when subjected to controlled heating/cooling environmental stress cycles, where only 500 STE showed a decrease in strength in 1860 samples series environmentally stressed stone.

The data obtained from the testing shows in some instances a gain in maximum strength after weathering of the consolidated samples was an unexpected result. This gain was seen in samples from the 1860 series treated with OH 100, and subjected to heating/cooling or freeze/thaw environmental stress cycling. Samples from the 1870 series treated with OH 100 and subjected to heating/cooling gained in strength, those samples treated with 500 STE subjected to heating/cooling or freeze/thaw environmental

stress cycling also increased in strength after the stress cycling. 1900 series stone samples treated with OH 100 and samples treated with 500 STE subjected to heating/cooling or freeze/thaw environmental stress cycling gained in strength compared to the strength of the control samples. This occurrence could be due to several factors. Both OH and STE are cured through a hydrolysis reaction with water. During the freeze/thaw environmental stress cycling, water could have been driven into the stone matrix, further reacting any uncured consolidant in the stone. Additionally, during the heating/cooling weathering cycles, water is driven off, and heat increases the rate of reaction, both of which could also be increasing the extent of the reaction of the consolidant curing in the system. This could be by forcing the water further into the system, or exposing more unreacted consolidant to water to start the curing process. In the freeze/thaw environmentally stressed stone, the presence of increased moisture was observed with the formation of condensation on the samples, this could further the curing of the consolidated stone samples by introducing more water into the system to drive hydrolysis of the consolidant.

Microscopic and analytical examinations of the samples revealed that the mineralogy of the three Berea sandstones, are similar in many respects, such as mineralogical content, size and distribution of grains and interstitial spaces, and , The stones were most markedly different in their rates of capillary uptake of water, as obtained with capillary uptake testing.

Biaxial flexure results provided some conclusions regarding the overall performance of Berea sandstone as a material. This material is a slightly ductile material, having a gentle slope to the stress strain curves from flexure testing. The stone is

potentially susceptible to changes in environmental conditions, most especially freeze/thaw environmental stresses.

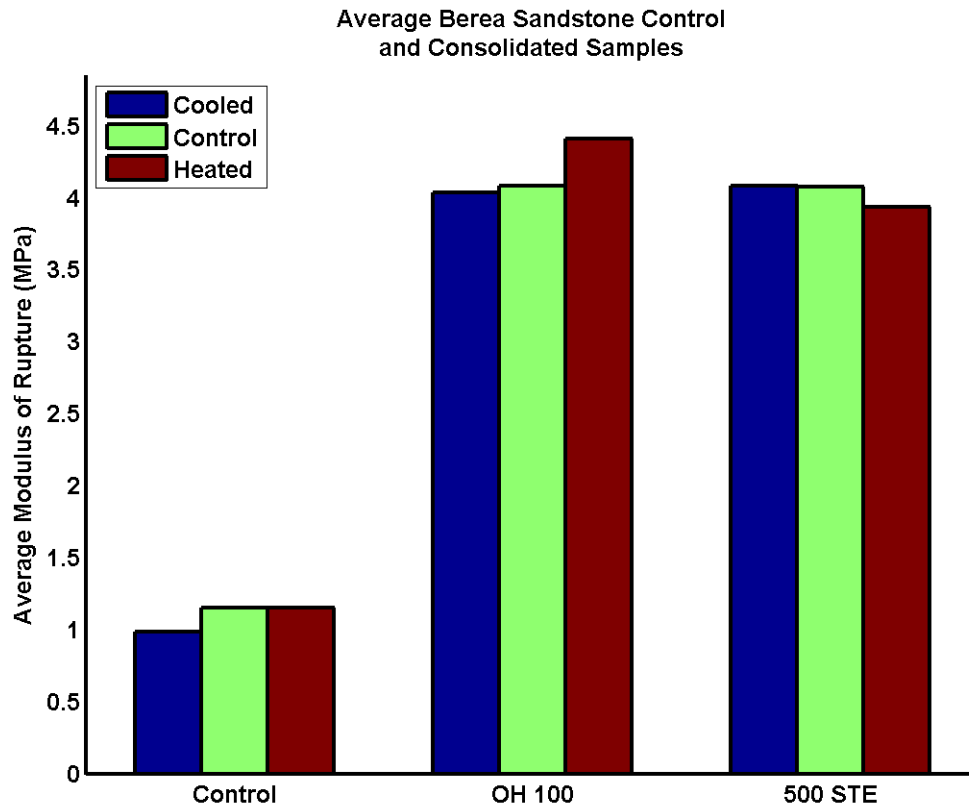


Fig. 35. Average Maximum strength in Freeze/Thaw Environmentally Stress Cycled Control, and Consolidated Samples

Figure 35, shows that the average maximum strength of the unstressed, untreated Berea sandstone was 1.23 MPa. When heated, untreated Berea sandstone lost approximately 9% of its inherent strength (down to 1.12 MPa). When subjected to freeze/thaw stress cycling, untreated Berea sandstone lost approximately 14% of its strength, from 1.23 MPa to 1.06 MPa. When treated with the OH 100 consolidant, the Berea sandstone exhibited a strength gain of 217%, with an average maximum strength of

3.9 MPa. When treated with 500 STE, it exhibited a strength gain of 209%, with an average maximum strength of 3.8 MPa.

Both OH 100 and 500 STE show similar initial strength gains for all three sandstones, increasing the stone by a factor in a range between 2.5 to 4, after consolidation. Both OH 100 and 500 STE performed similarly when subjected to controlled freeze/thaw environmental stress cycles, showing only a small loss of initial strength gain. Both OH 100 and 500 STE performed similarly when subjected to controlled heating/cooling environmental stress cycles, where only 500 STE showed a decrease in strength, which was only in the 1860 samples series.

The data obtained from the testing shows in some instances a gain in maximum strength after weathering of the consolidated samples was an unexpected result. This gain was seen in samples from the 1860 series treated with OH 100 subjected to both heating/cooling and freeze/thaw stress cycling; and samples from the 1870 series treated with both OH 100 and 500 STE and subjected to heating/cooling; and the 1900 series treated with both OH 100 and 500 STE subjected to both heating/cooling and freeze/thaw environmental stress cycling.

This strength gain after controlled environmental stress cycling could be due to several factors. Both OH and STE are cured through a hydrolysis reaction with water. During the freeze/thaw environmental stress cycling, water could have been driven into the stone matrix, further reacting any uncured consolidant in the stone. Additionally, during the heating/cooling weathering cycles, water is driven off, and heat increases the rate of reaction, both of which could also be increasing the extent of the reaction of the consolidant curing in the system. This could be by forcing the water further into the

system, or exposing more unreacted consolidant to water to start the curing process. In the freeze/thaw environmentally stressed stone, the presence of increased moisture was observed with the formation of condensation on the samples, this could further the curing of the consolidated stone samples by introducing more water into the system to drive hydrolysis of the consolidant.

There is a comparative difference in the strength increase of stone samples consolidated with OH 100 and the stone samples consolidated with 500 STE, based on the type of environmental stress cycling. The stone samples treated with OH 100 increased in strength more than stone consolidated with 500 STE by 16%, when both were subjected to heating/cooling environmental stress testing. Stone samples consolidated with 500 STE gained more strength than samples consolidated with OH 100 when subjected to freeze/thaw stress cycling, but only by 5%, an insignificant figure in comparison. A question to consider is how much percent change is significant.

In this study this question is complicated by the difference in scale between the changes caused by the consolidation of stone, on the order of 200-300% increase, compared to the smaller scale of 3-18% change between unstressed stone and environmentally stressed stone. With this difference, the sizable change in the consolidated stone strengths makes the change of the stressed stone seem less significant, since it is a much smaller percent change, and may fall well within the range of the standard deviation of error for this study. Considering that the percent change between stressed and unstressed consolidated stone observed in this study is small, this strengthens the conclusion that the performance of these consolidants through the environmental stressing is for all intents and purposes, the same.

The standard deviations in some sample sets between the average maximum biaxial flexure strength, and high or low individual samples in that set are sometimes quite high in this study, and are directly attributable to the material being tested being a natural one with a natural materials variety in characteristics. In taking averaged from multiple samples the effort was made to obtain figures which would be representative of these natural materials. The margin of error is affected though by the differences between them, where no one sample can be seen as representative of the whole material, the averaged group giving a better representation of the material.

Conclusions

The performance of all alkoxysilane consolidants has been proven to be dependant on stone characteristics such as the mineralogy, size of the grains, the size of the interstitial spaces, and the type of deterioration. For these Berea sandstones, the performance of both consolidants increased strength after consolidation. Based on the data collected in this thesis, Conservare OH 100 and Funcosil 500 STE performed equally well in consolidating the stone, and comparably well in retaining their strength through the environmental stress cycles.

The unconsolidated stone was very similar in mineralogical composition as well as in performance, as measured in the biaxial flexure test. Some samples included illite and kaolinite clays, however, not all, which may account for the different performance under environmental stress testing. The literature is inconsistent on the affect of clays on stones consolidated with alkoxysilanes, but for these sandstones, the presence of clays did not appear to have an effect on the consolidants performance.

Where there does not appear to be much, if any, strength gain attributable to the additional materials in 500 STE, they certainly did not cause loss of strength of the consolidant in the stone either. The performance of the 500 STE was similar to the OH 100 in the majority of the sample groups, and that performance was consistent with a satisfactory consolidation of the stone where the consolidant increases the maximum biaxial flexure strength of the stone at least twice as much as the pre-consolidated strength, in terms of the amount of strength gained through the addition of the consolidant.

Test groups from all three stone types saw a further increase of flexural strength in the environmental stress cycled consolidated stone. This may be due in the heat stressed stone to the thermal increase helping to further catalyze consolidant in the stone. In the cold stressed stone, the presence of increased moisture was observed with the formation of condensation on the samples, this could further curing of the stone by introducing more water into the system to drive hydrolysis of the consolidant. This is an area for potential future research.

Future Research

The results of the testing and analysis performed in this thesis lead to questions outside the scope of the current study. Further analysis to better understand the affects of consolidants on stone and that could include the following research. The analysis of the water vapor permeability of untreated, treated and re-treated stone to determine what effects on the transmission of water through the stone occur upon the filling of a certain amount of the stone matrix with consolidant and since reportedly stones consolidated with alkoxy silanes can be re-treated after previous applications the effect on the efficacy of future treatments applied to the stone should be studied. At question is whether or not the added silica nano-particles in 500 STE, as well as the elastifiers, decrease treated stones permeability significantly more than stone treated with OH 100, which would affect future treatments or cleaning of that stone.

Further investigation into the performance of alkoxy silane consolidants on particular types of stones would be of interest to see if trends do emerge in the performance of alkoxy silanes in regards to silicate and carbonate stone types. This study could include limestones containing clay to further research the effect of clay on consolidation with alkoxy silanes, as well as other stones types, like marbles or granites. Researching other stone types would provide data on whether or not the similar performance of the consolidants seen in this study is maintained with stones of different mineralogical properties. The literature is inconsistent on the affect of clays on stones consolidated with alkoxy silanes.

This thesis examined at the maximum biaxial flexural strength gain produced by the application of two specific consolidants in sandstone before and after 60 weathering

cycles of two specific types. Considering the increase in strength shown by many of the stone samples after weathering, further controlled environmental stress cycles of similar stone samples could provide information on the presumably ongoing strength gain as well as the number of cycles that result in the loss of strength, which could provide more information as to the performance of the consolidants in the field..

In areas where VOC compliance regulations prevent the use of Conservare OH 100, the Funcosil 500 STE could be a viable alternative product because the increased gel deposition rate of 50% w/v lowers the VOC emissions. Significantly higher than the 30% w/v deposited by OH 100, the increased solid deposition percentage could also be further increased with the added weight of the deposited silica nano-particles. Based on the testing completed in this thesis 500 STE is an alternative treatment from the standpoint that it is a successful alkoxy silane consolidant, increasing the tensile strength of an appropriate stone treated with it, and could therefore be a viable alternative product for OH 100 where VOC emission is a serious consideration.

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Appendix A: Historic Photos and Images of Parliament Hill, Ottawa, Ontario



“Main Buildings, Houses of Parliament”, ca. 1876 engraving by C. Schwarzburger⁹⁶



West Block of Parliament Buildings in the early stages of construction, 1861.⁹⁷

⁹⁶ De Volpi, C.P. Ottawa: A Pictorial Record, Historical Prints and Illustrations of the City of Ottawa, Province of Ontario, Canada, 1807-1882. Dev-Sco Publications, 1964. Pictorial Records Series.



The West Block of Parliament under construction, view looking West.⁹⁸



The West Block under construction, view looking at the complete North-East corner.⁹⁹

⁹⁷ <http://data2.collectionscanada.gc.ca/ap/a/a027196.jpg>

⁹⁸ <http://data2.collectionscanada.gc.ca/ap/c/c000131.jpg>

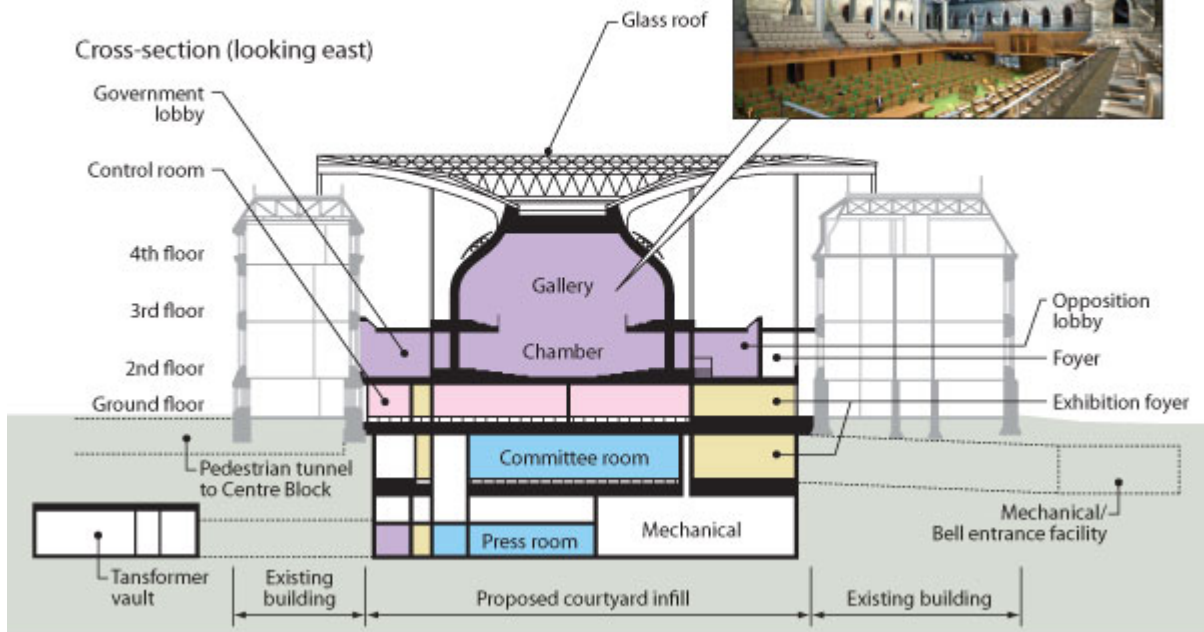


A bird's eye view of the city of Ottawa in 1876, Atlas of Canada ¹⁰⁰

⁹⁹ <http://data2.collectionscanada.gc.ca/ap/c/c009950.jpg>

¹⁰⁰ BROSIUS, H. Bird's Eye View of the City of Ottawa, Province Ontario, Canada, 1876. Drawn by Herm. Brosius. N. p., 1967.

House of Commons temporary home in West Block



West Block Proposed Construction Plan¹⁰¹

¹⁰¹ "Graphic: West Block Plan | Ottawa Citizen." N. p., n.d. Web. 28 Apr. 2014.

RENOVATING BLOCK BY BLOCK

The West Block will be vacated in January, to continue the eight-year, \$769-million restoration

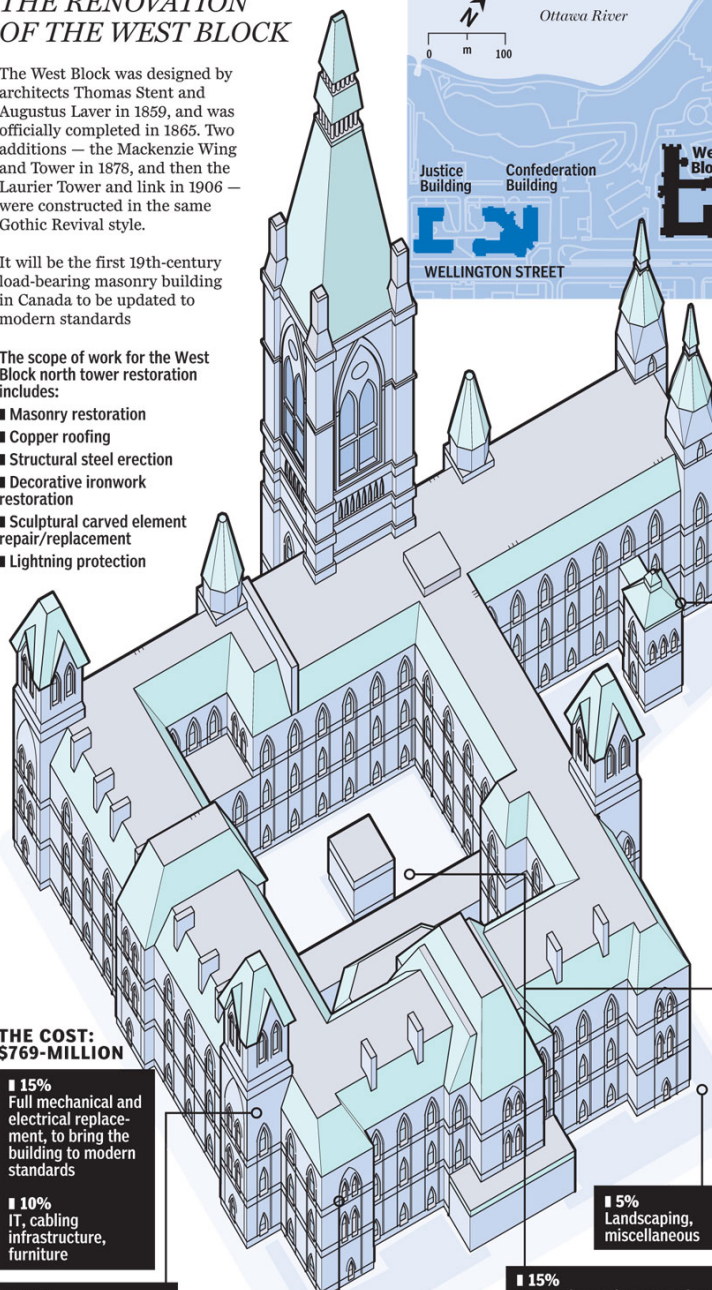
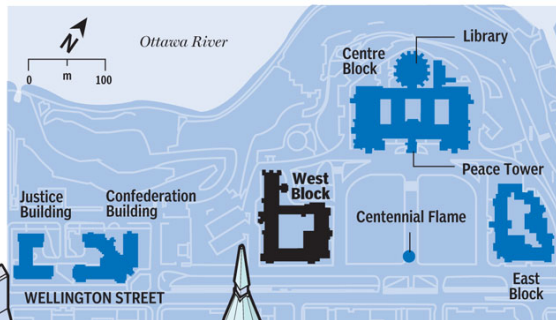
THE RENOVATION OF THE WEST BLOCK

The West Block was designed by architects Thomas Stent and Augustus Laver in 1859, and was officially completed in 1865. Two additions — the Mackenzie Wing and Tower in 1878, and then the Laurier Tower and link in 1906 — were constructed in the same Gothic Revival style.

It will be the first 19th-century load-bearing masonry building in Canada to be updated to modern standards

The scope of work for the West Block north tower restoration includes:

- Masonry restoration
- Copper roofing
- Structural steel erection
- Decorative ironwork restoration
- Sculptural carved element repair/replacement
- Lightning protection



THE WORK REQUIRED



DAMAGED CARVED ELEMENTS

■ The exterior is comprised of three different types of sandstone, which was brought in from quarries in Quebec, upstate New York and Ohio.



INTERIOR WATER DAMAGE

■ The interior is comprised of limestone. Work will be done to repair the damaged masonry and stonework inside and outside.

■ An addition of 20,000 square metres, spanning four floors (basically four football fields). Infill construction will add 25,000 square metres, doubling the size of the West Block.

MOVING OUT

50 MPs and their parliamentary suites will be relocated to La Promenade Building at 151 Sparks Street.

■ \$100-million will be spent on relocation projects for La Promenade, Rideau committee rooms and off-site food-production facility

THE COST: \$769-MILLION

■ 15% Full mechanical and electrical replacement, to bring the building to modern standards

■ 10% IT, cabling infrastructure, furniture

■ 20% Interior fit-ups, abatement, and general renovations throughout the building

■ 30% Stone masonry repairs and replacements

■ 5% Asbestos abatement, demolition

■ 5% Landscaping, miscellaneous

■ 15% Excavation and construction in the courtyard to accommodate the interim House of Commons Chamber and related functions.

THE HISTORY OF THE WEST BLOCK

■ As of 1867, the West Block was home to the Public Works Department, the Post Office Department, the Adjutant General and Militia Department, the Fisheries Department, the Bureau of Agriculture, and the model room connected with the Patent Department.

■ The government wanted to demolish the West Block in the 1950s to make room for a more modern building. But the building was saved by public works minister Alexander Mackenzie. Restoration to the interior and exterior of the building were instead completed in 1965.

■ The West Block is closed to the public, but appears on the \$5 bill.

■ Former prime minister Alexander Mackenzie, then minister of public works, oversaw the completion of the Parliament buildings. In the Mackenzie Building, he had a secret, circular staircase that led from his office outside. The staircase was used by Pierre Trudeau to avoid the press when he was slipping out to call an election.

■ In 1877, Alexander Mackenzie made the first commercial telephone call in Canada from Room 310 West Block to the Governor-General's residence.

■ The reported final cost to build the West Block, which was occupied for the first time in 1866, was \$518,352.

Appendix B: Testing Photos

Capillary Uptake Test:

The stones are arranged as follows in all photos, the 1860 stone to the left, the 1870 stone in the middle, and the 1900 stone to the right. All photos taken by Dr. Wheeler.



Fig. 1: Capillary Uptake: 1 min.



Fig. 2: Capillary Uptake: 2 min.



Fig. 3: Capillary Uptake: 4 min.



Fig. 4: Capillary Uptake: 10 min., Saturation of 1900 Stone



Fig. 5: Capillary Uptake: 15 min.



Fig. 6: Capillary Uptake: 20 min., Saturation of 1870 Stone



Fig. 7: Capillary Uptake: 25 min.

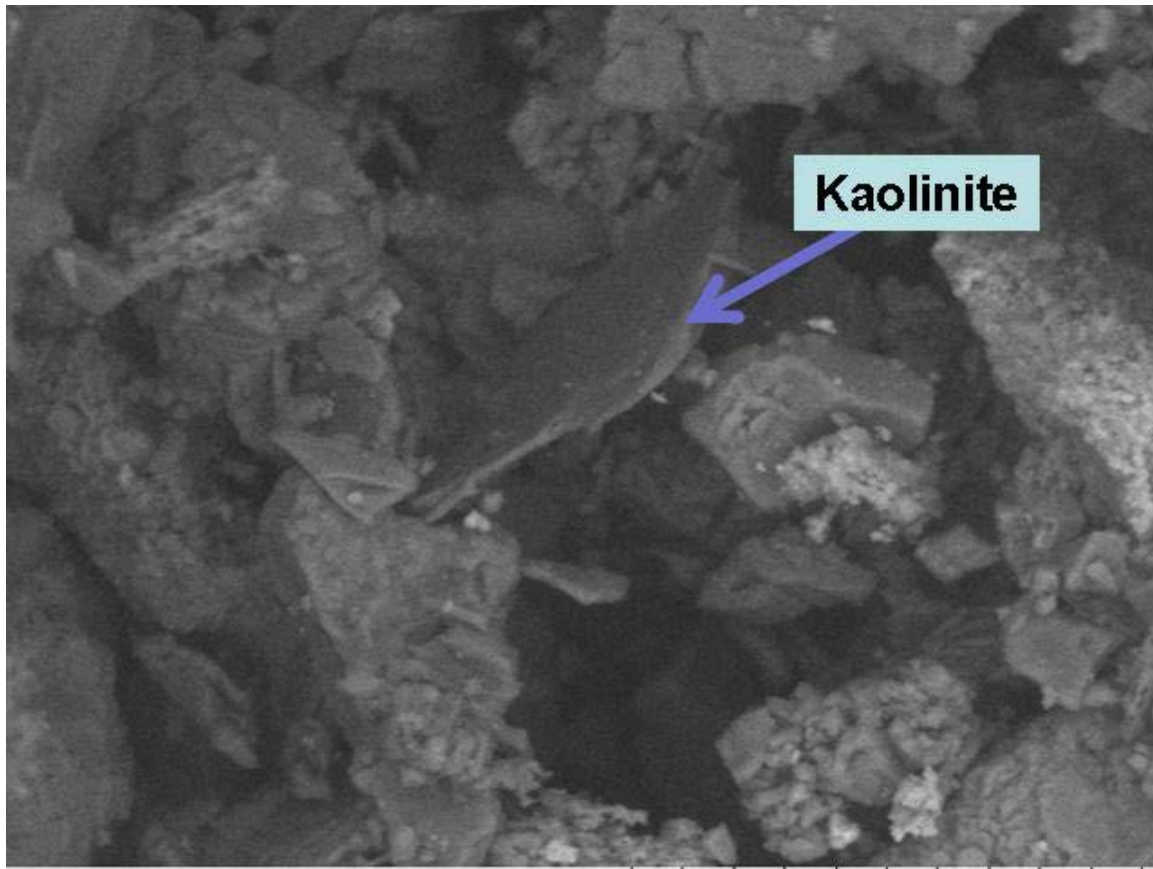


Fig. 8: Capillary Uptake: 40 min., Saturation of 1860 Stone



Fig. 18. During Testing Photo: Sample discs of stone submerged in consolidant. Photo by author.

Appendix C: SEM Image



Corrosion0005 2014/03/14 AL ×1.5k 50 um

Cross section

Fig. 1. SEM photo at 1500 magnification, showing a smooth clay plate among the primarily quartz grains of the 1900 control stone.

Appendix D: Charts and Graphs

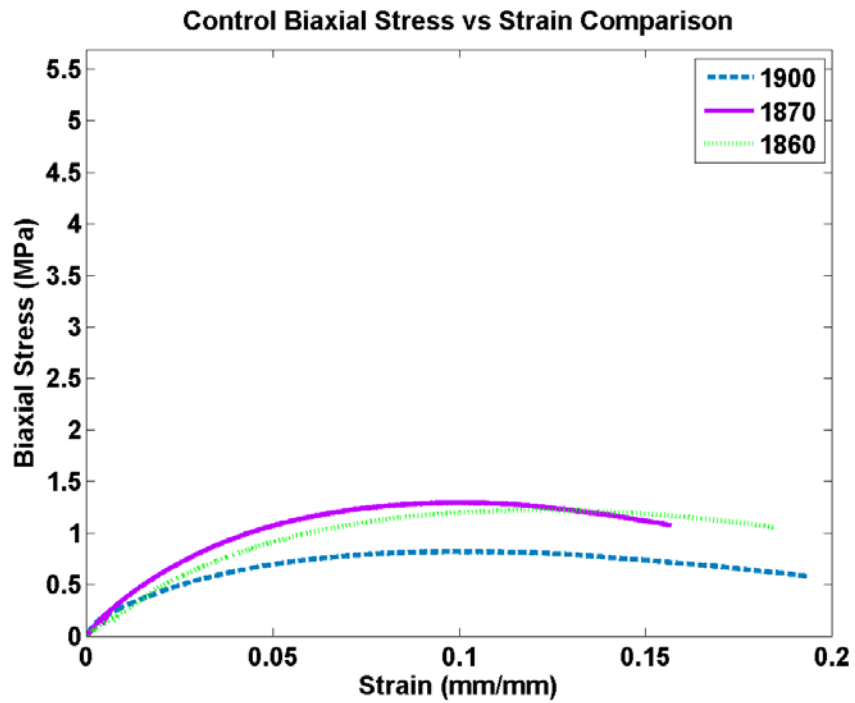


Fig. 19. Biaxial Flexure Data for Stress/Strain, Untreated and Unstressed (Control) Berea Sandstones

1860 Control and Environmental Stress Cycled Biaxial Stress vs Strain Compar

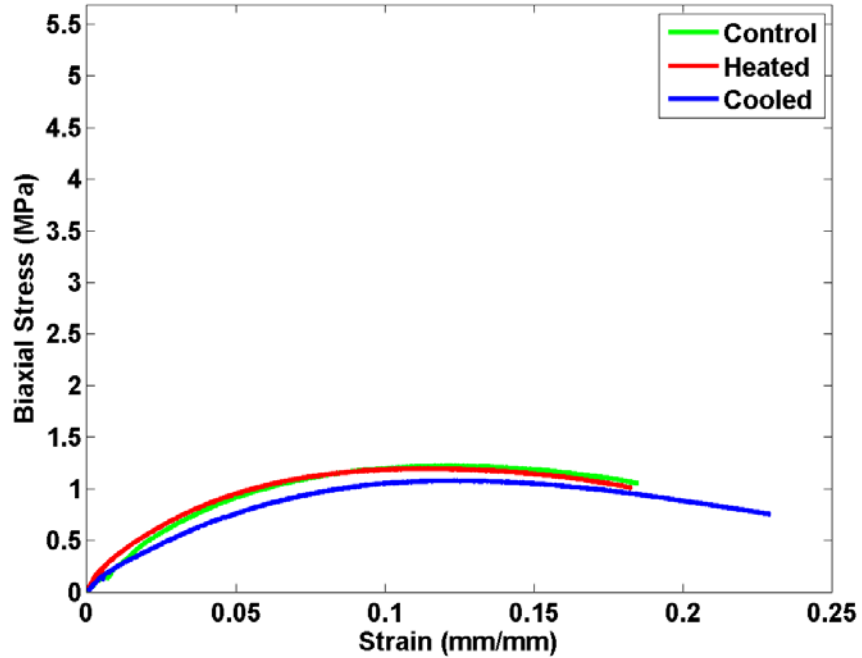


Fig. 20. Biaxial Flexure Data for Stress/Strain Graph, 1860 Control Stone, and Environmentally Stressed Control Stone

170 Control and Environmental Stress Cycled Biaxial Stress vs Strain Compar

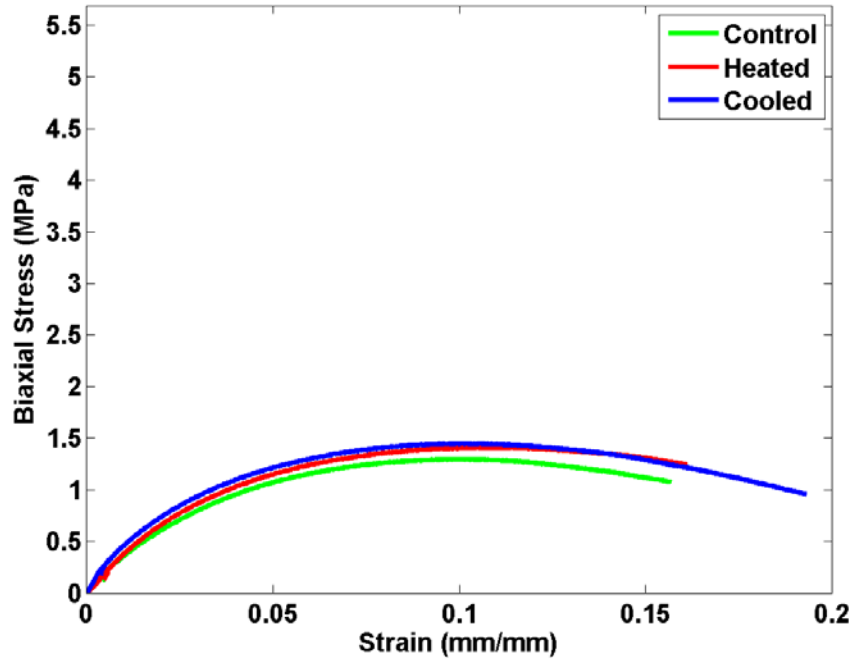


Fig. 21. Biaxial Flexure Data for Stress/Strain Graph, 1870 Control Stone, and Environmentally Stressed Control Stone

100 Control and Environmental Stress Cycled Biaxial Stress vs Strain Compar

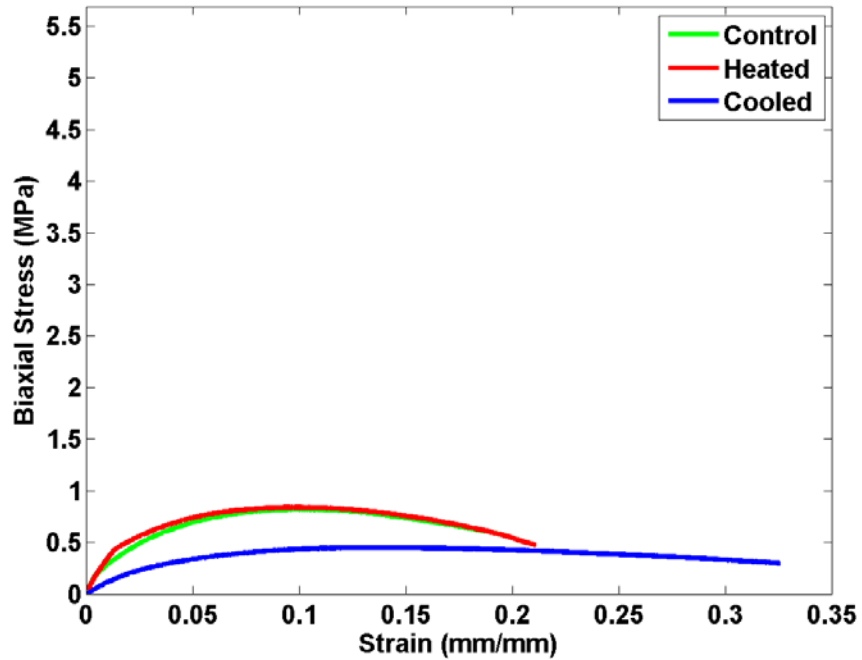


Fig. 22. Biaxial Flexure Data for Stress/Strain Graph, 1900 Control Stone, and Environmentally Stressed Control Stone

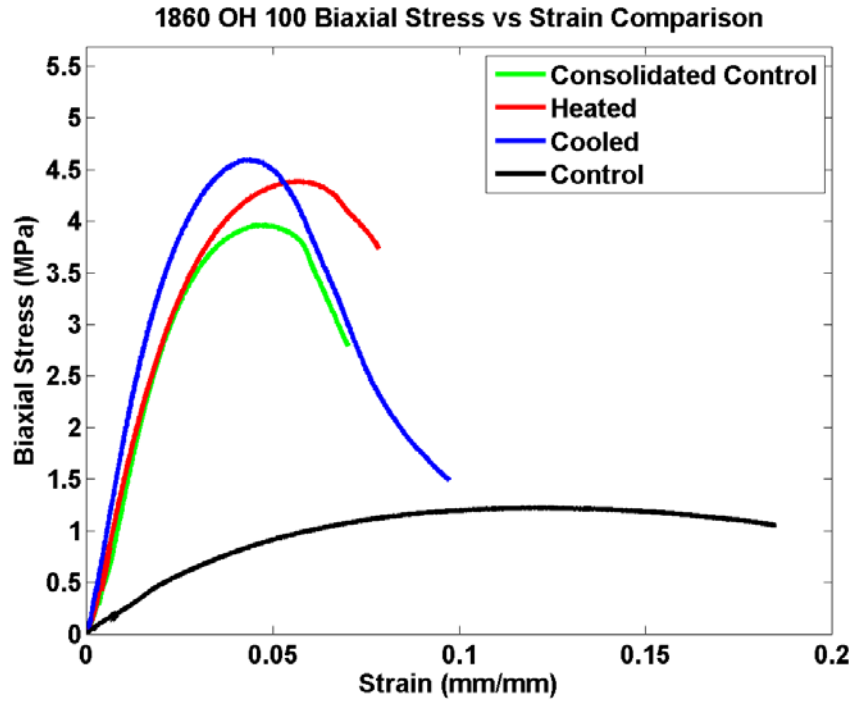


Fig. 23. Biaxial Flexure Data for Stress/Strain Graph, 1860 Control Stone, OH 100 Consolidated Stone and Environmentally Stressed Consolidated Stone

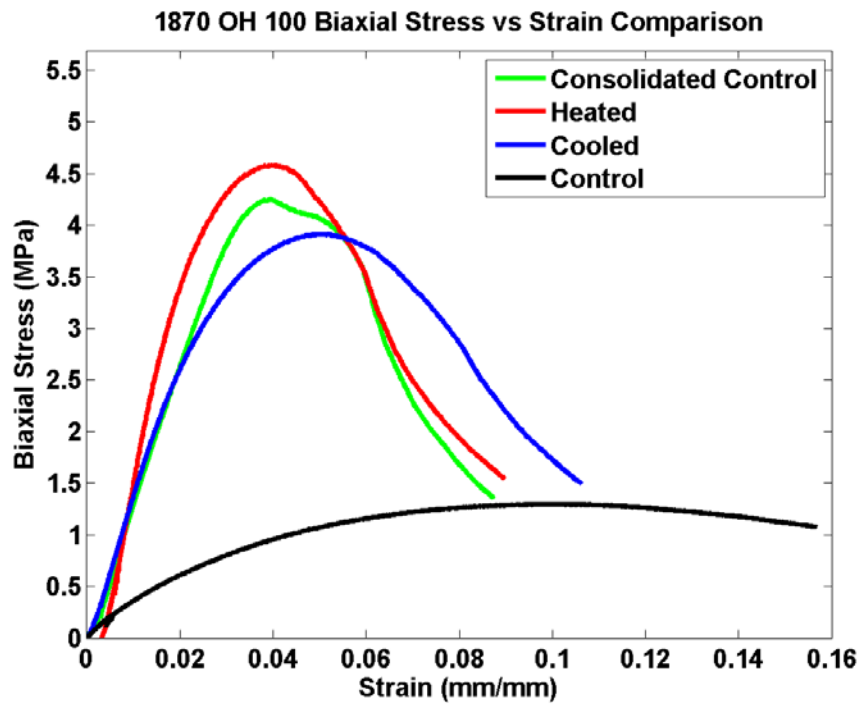


Fig. 24. Biaxial Flexure Data for Stress/Strain Graph, 1870 Control Stone, OH 100 Consolidated Stone and Environmentally Stressed Consolidated Stone

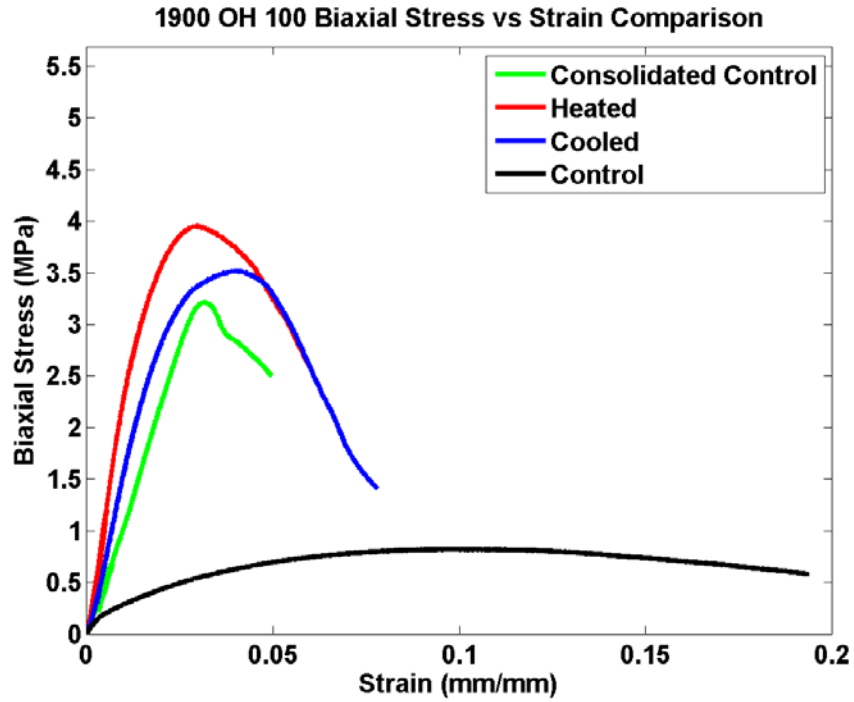


Fig. 25. Biaxial Flexure Data for Stress/Strain Graph, 1900 Control Stone, OH 100 Consolidated Stone and Environmentally Stressed Consolidated Stone

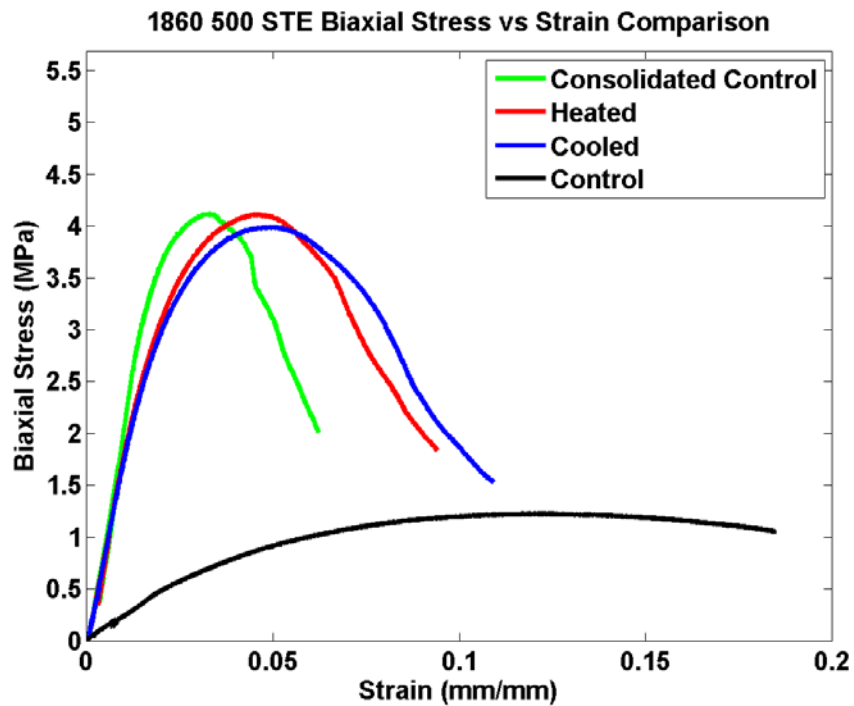


Fig. 26. Biaxial Flexure Data for Stress/Strain Graph, 1860 Control Stone, 500 STE Consolidated Stone and Environmentally Stressed Consolidated Stone

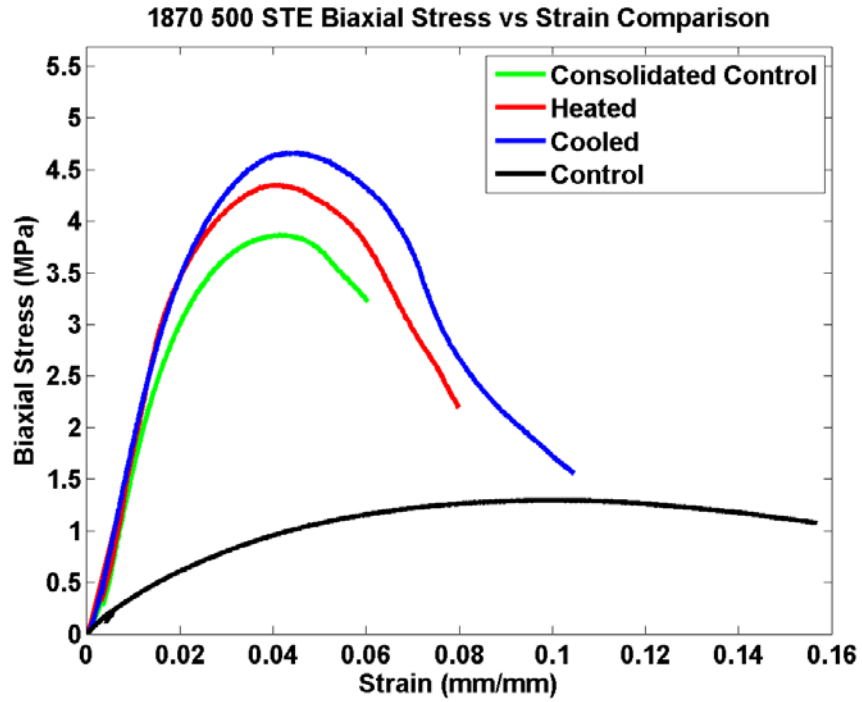


Fig. 27. Biaxial Flexure Data for Stress/Strain Graph, 1870 Control Stone, 500 STE Consolidated Stone and Environmentally Stressed Consolidated Stone

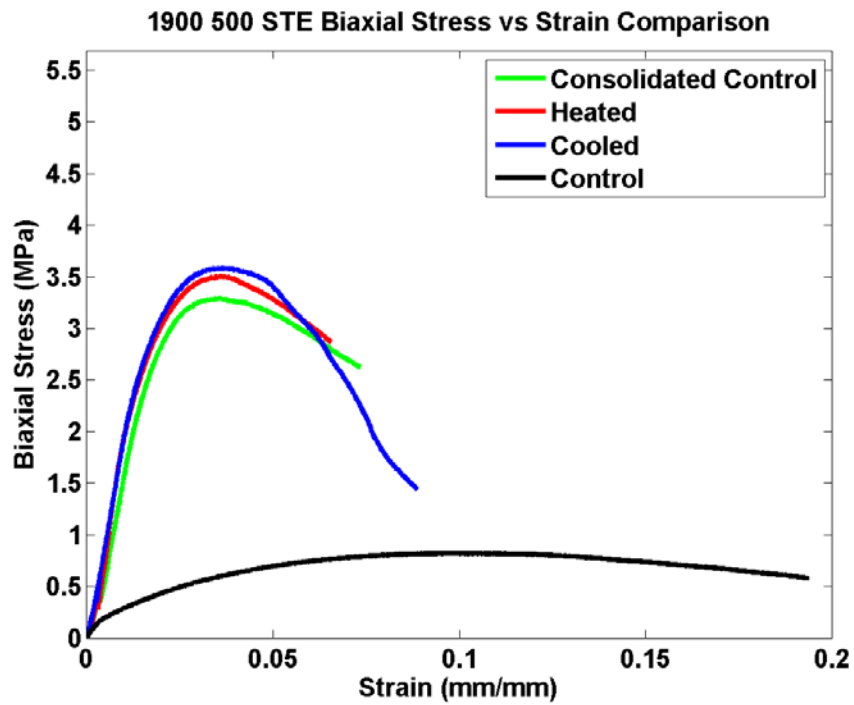


Fig. 28. Biaxial Flexure Data for Stress/Strain Graph, 1870 Control Stone, 500 STE Consolidated Stone and Environmentally Stressed Consolidated Stone

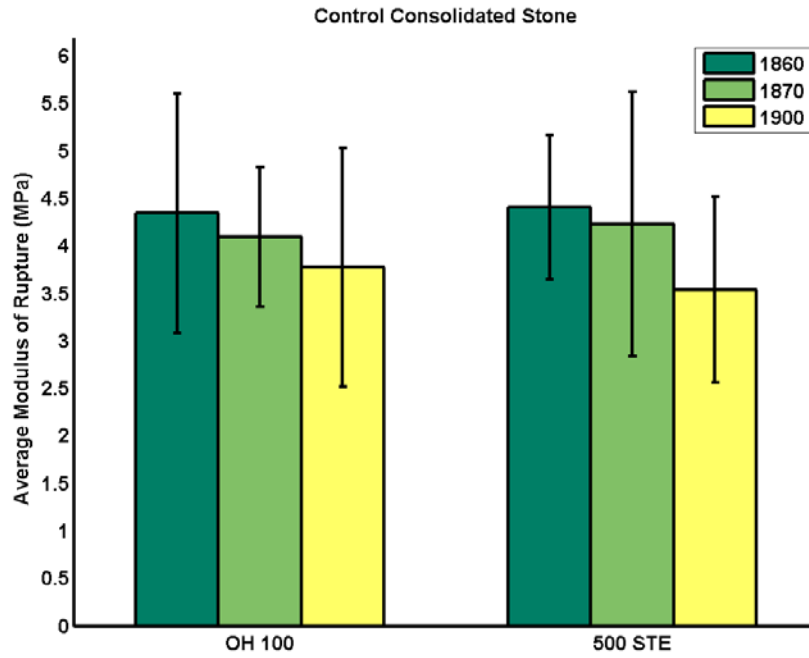


Fig. 29. Average Maximum Strength in Control Samples, and Control Consolidated Samples

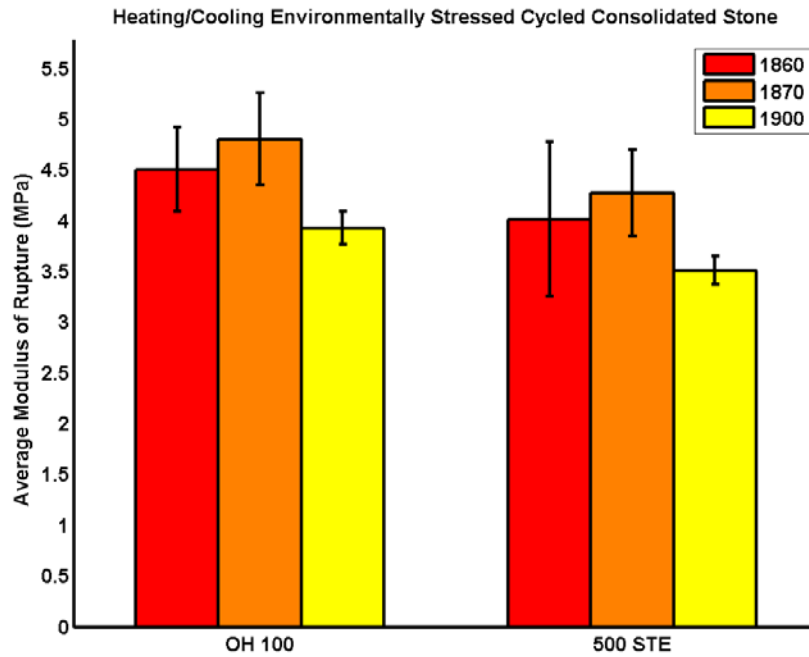


Fig. 30. Average Maximum strength in Heating/Cooling Environmentally Stress Cycled Control, and Consolidated Samples

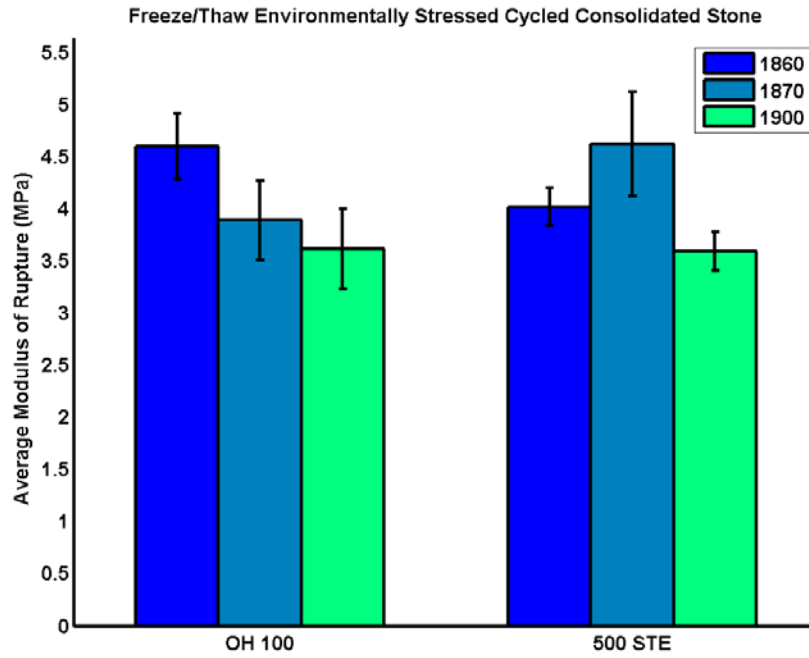


Fig. 31. Average Maximum strength in Freeze/Thaw Environmentally Stress Cycled Control, and Consolidated Samples

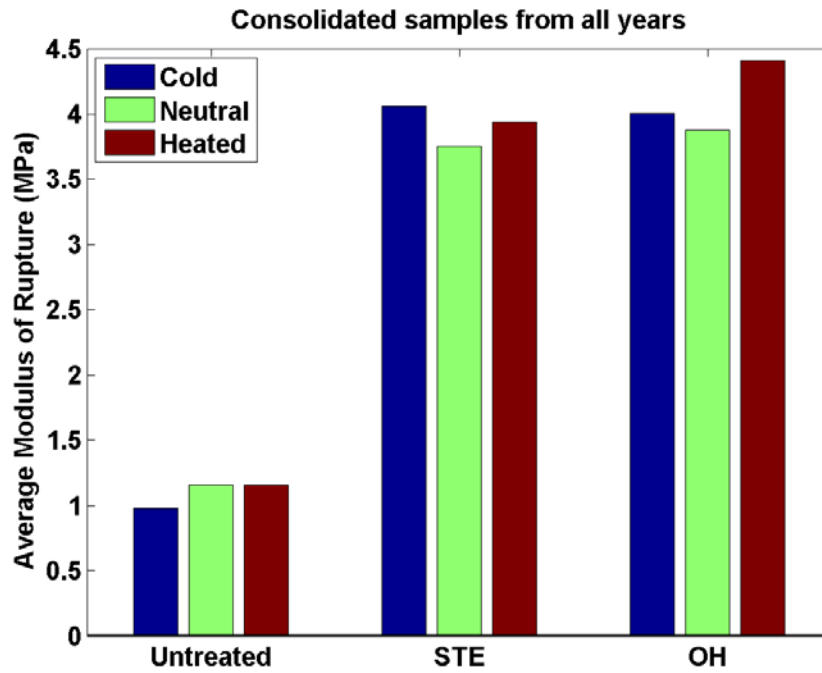


Fig. 32. Average Maximum strength in Freeze/Thaw Environmentally Stress Cycled Control, and Consolidated Samples

Appendix E: ASTM

ASTM C1499-09(2013) Standard Test Method for Monotonic Equibiaxial Flexural Strength of Advanced Ceramics at Ambient Temperature

ASTM C67-13a Standard Test Methods for Sampling and Testing Brick and Structural Clay Tile



Standard Test Method for Monotonic Equibiaxial Flexural Strength of Advanced Ceramics at Ambient Temperature¹

This standard is issued under the fixed designation C1499; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of the equibiaxial strength of advanced ceramics at ambient temperature via concentric ring configurations under monotonic uniaxial loading. In addition, test specimen fabrication methods, testing modes, testing rates, allowable deflection, and data collection and reporting procedures are addressed. Two types of test specimens are considered: machined test specimens and as-fired test specimens exhibiting a limited degree of warpage. Strength as used in this test method refers to the maximum strength obtained under monotonic application of load. Monotonic loading refers to a test conducted at a constant rate in a continuous fashion, with no reversals from test initiation to final fracture.

1.2 This test method is intended primarily for use with advanced ceramics that macroscopically exhibit isotropic, homogeneous, continuous behavior. While this test method is intended for use on monolithic advanced ceramics, certain whisker- or particle-reinforced composite ceramics as well as certain discontinuous fiber-reinforced composite ceramics may also meet these macroscopic behavior assumptions. Generally, continuous fiber ceramic composites do not macroscopically exhibit isotropic, homogeneous, continuous behavior, and the application of this test method to these materials is not recommended.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

¹ This test method is under the jurisdiction of ASTM Committee C28 on Advanced Ceramics and is the direct responsibility of Subcommittee C28.01 on Mechanical Properties and Performance.

Current edition approved Aug. 1, 2013. Published September 2013. Originally approved in 2001. Last previous edition approved in 2009 as C1499–09. DOI: 10.1520/C1499-09R13.

2. Referenced Documents

2.1 ASTM Standards:²

- C1145 Terminology of Advanced Ceramics
- C1239 Practice for Reporting Uniaxial Strength Data and Estimating Weibull Distribution Parameters for Advanced Ceramics
- C1259 Test Method for Dynamic Young's Modulus, Shear Modulus, and Poisson's Ratio for Advanced Ceramics by Impulse Excitation of Vibration
- C1322 Practice for Fractography and Characterization of Fracture Origins in Advanced Ceramics
- E4 Practices for Force Verification of Testing Machines
- E6 Terminology Relating to Methods of Mechanical Testing
- E83 Practice for Verification and Classification of Extensometer Systems
- E337 Test Method for Measuring Humidity with a Psychrometer (the Measurement of Wet- and Dry-Bulb Temperatures)
- F394 Test Method for Biaxial Flexure Strength (Modulus of Rupture) of Ceramic Substrates (Discontinued 2001) (Withdrawn 2001)³
- IEEE/ASTM SI 10 Standard for Use of the International System of Units (SI): The Modern Metric System

3. Terminology

3.1 Definitions:

3.1.1 The definitions of terms relating to biaxial testing appearing in Terminology E6 and Terminology C1145 may apply to the terms used in this test method. Pertinent definitions are listed below with the appropriate source given in parentheses. Additional terms used in conjunction with this test method are defined in the following section.

3.1.2 *advanced ceramic, n*—highly engineered, high performance predominately non-metallic, inorganic, ceramic material having specific functional attributes. **C1145**

² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

³ The last approved version of this historical standard is referenced on www.astm.org.

3.1.3 *breaking load*, $[F]$, n —load at which fracture occurs. **E6**

3.1.4 *equibiaxial flexural strength*, $[F/L_2]$, n —maximum stress that a material is capable of sustaining when subjected to flexure between two concentric rings. This mode of flexure is a cupping of the circular plate caused by loading at the inner load ring and outer support ring. The equibiaxial flexural strength is calculated from the maximum-load of a biaxial test carried to rupture, the original dimensions of the test specimen, and Poisson's ratio.

3.1.5 *homogeneous*, n —condition of a material in which the relevant properties (composition, structure, density, etc.) are uniform, so that any smaller sample taken from an original body is representative of the whole. Practically, as long as the geometrical dimensions of a sample are large with respect to the size of the individual grains, crystals, components, pores, or microcracks, the sample can be considered homogeneous.

3.1.6 *modulus of elasticity*, $[F/L_2]$, n —ratio of stress to corresponding strain below the proportional limit. **E6**

3.1.7 *Poisson's ratio*, n —negative value of the ratio of transverse strain to the corresponding axial strain resulting from uniformly distributed axial stress below the proportional limit of the material.

4. Significance and Use

4.1 This test method may be used for material development, material comparison, quality assurance, characterization and design code or model verification.

4.2 Engineering applications of ceramics frequently involve biaxial tensile stresses. Generally, the resistance to equibiaxial flexure is the measure of the least flexural strength of a monolithic advanced ceramic. The equibiaxial flexural strength distributions of ceramics are probabilistic and can be described by a weakest link failure theory, **(1, 2)**⁴. Therefore, a sufficient number of test specimens at each testing condition is required for statistical estimation or the equibiaxial strength.

4.3 Equibiaxial strength tests provide information on the strength and deformation of materials under multiple tensile stresses. Multiaxial stress states are required to effectively evaluate failure theories applicable to component design, and to efficiently sample surfaces that may exhibit anisotropic flaw distributions. Equibiaxial tests also minimize the effects of test specimen edge preparation as compared to uniaxial tests because the generated stresses are lowest at the test specimen edges.

4.4 The test results of equibiaxial test specimens fabricated to standardized dimensions from a particular material and/or selected portions of a component may not totally represent the strength properties in the entire, full-size component or its in-service behavior in different environments.

4.5 For quality control purposes, results derived from standardized equibiaxial test specimens may be considered indicative of the response of the bulk material from which they were

taken for any given primary processing conditions and post-processing heat treatments or exposures.

5. Interferences

5.1 Test environment (vacuum, inert gas, ambient air, etc.) including moisture content (for example, relative humidity) may have an influence on the measured equibiaxial strength. Testing to evaluate the maximum strength potential of a material can be conducted in inert environments and/or at sufficiently rapid testing rates so as to minimize any environmental effects. Conversely, testing can be conducted in environments, test modes and test rates representative of service conditions to evaluate material performance under use conditions.

5.2 Fabrication of test specimens can introduce dimensional variations that may have pronounced effects on the measured equibiaxial mechanical properties and behavior (for example, shape and level of the resulting stress-strain curve, equibiaxial strength, failure location, etc.). Surface preparation can also lead to the introduction of residual stresses and final machining steps might or might not negate machining damage introduced during the initial machining. Therefore, as universal or standardized methods of surface preparation do not exist, the test specimen fabrication history should be reported. In addition, the nature of fabrication used for certain advanced ceramic components may require testing of specimens with surfaces in the as-fabricated condition (that is, it may not be possible, desired or required to machine some of the test specimen surfaces directly in contact with the test fixture). For very rough or wavy as-fabricated surfaces, perturbations in the stress state due to non-symmetric cross-sections as well as variations in the cross-sectional dimensions may also interfere with the equibiaxial strength measurement. Finally, close geometric tolerances, particularly in regard to flatness of test specimen surfaces in contact with the test fixture components are critical requirements for successful equibiaxial tests. In some cases it may be appropriate to use other test methods (for example, Test Method **F394**).

5.3 Contact and frictional stresses in equibiaxial tests can introduce localized failure not representative of the equibiaxial strength under ideal loading conditions. These effects may result in either over or under estimates of the actual strength **(1, 3)**.

5.4 Fractures that consistently initiate near or just outside the load-ring may be due to factors such as friction or contact stresses introduced by the load fixtures, or via misalignment of the test specimen rings. Such fractures will normally constitute invalid tests (see **Note 14**). Splitting of the test specimen along a diameter that expresses the characteristic size may result from poor test specimen preparation (for example, severe grinding or very poor edge preparation), excessive tangential stresses at the test specimen edges, or a very weak material. Such fractures will constitute invalid tests if failure occurred from the edge.

5.5 Deflections greater than one-quarter of the test specimen thickness can result in nonlinear behavior and stresses not accounted for by simple plate theory.

⁴ The boldface numbers in parentheses refer to the list of references at the end of this standard.

5.6 Warpage of the test specimen can result in nonuniform loading and contact stresses that result in incorrect estimates of the test specimen's actual equibiaxial strength. The test specimen shall meet the flatness requirements (see 8.2 and 8.3) or be specifically noted as warped and considered as a censored test.

6. Apparatus

6.1 *Testing Machines*—Machines used for equibiaxial testing shall conform to the requirements of Practices E4. The load cells used in determining equibiaxial strength shall be accurate within $\pm 1\%$ at any load within the selected load range of the testing machine as defined in Practice E4. Check that the expected breaking load for the desired test specimen geometry and test material is within the capacity of the test machine and load cell. Advanced ceramic equibiaxial test specimens require greater loads to fracture than those usually encountered in uniaxial flexure of test specimens with similar cross sectional dimensions.

6.2 *Loading Fixtures for Concentric Ring Testing*—An assembly drawing of a fixture and a test specimen is shown in Fig. 1, and the geometries of the load and support rings are given in Fig. 2.

6.2.1 *Loading Rods and Platens*—Surfaces of the support platen shall be flat and parallel to 0.05 mm. The face of the load rod in contact with the support platen shall be flat to 0.025 mm. In addition, the two loading rods shall be parallel to 0.05 mm per 25 mm length and concentric to 0.25 mm when installed in the test machine.

6.2.2 *Loading Fixture and Ring Geometry*—Ideally, the bases of the load and support fixtures should have the same outer diameter as the test specimen for ease of alignment. Parallelism and flatness of faces as well as concentricity of the load and support rings shall be as given in Fig. 2. The ratio of the load ring diameter, D_L , to that of the support ring, D_S , shall

be $0.2 \leq D_L/D_S \leq 0.5$. For test materials exhibiting low elastic modulus ($E < 100$ GPa) and high strength ($\sigma_f > 1$ GPa) it is recommended that the ratio of the load ring diameter to that of the support ring be $D_L/D_S = 0.2$. The sizes of the load and support rings depend on the dimensions and the properties of the ceramic material to be tested. The rings are sized to the thickness, diameter, strength, and elastic modulus of the ceramic test specimens (see Section 8). For test specimens made from typical substrates ($h \approx 0.5$ mm), a support ring diameter as small as 12 mm may be required. For test specimens to be used for model verification, it is recommended that the test specimen support diameter be at least 35 mm. The tip radius, r , of the cross sections of the load and support rings should be $h/2 \leq r \leq 3h/2$.

6.2.3 *Load and Support Ring Materials*—For machined test specimens (see Section 8) the load and support fixtures shall be made of hardened steel of $HR_C > 40$. For as-fabricated test specimens, the load/support rings shall be made of steel or acetyl polymer.

6.2.4 *Compliant Layer and Friction Elimination*—The brittle nature of advanced ceramics and the sensitivity to misalignment, contact stresses and friction may require a compliant interface between the load/support rings and the test specimen, especially if the test specimen is not flat. Line or point contact stresses and frictional stresses can lead to crack initiation and fracture of the test specimen at stresses other than the actual equibiaxial strength.

6.2.4.1 *Machined Test Specimens*—For test specimens machined according to the tolerance in Fig. 3, a compliant layer is not necessary. However, friction needs to be eliminated. Place a sheet of carbon foil (~0.13 mm thick) or Teflon tape (~0.07 mm thick) between the compressive and tensile surfaces of the test specimen and the load and support rings.

NOTE 1—Thicker layers of carbon foil or Teflon tape may be used,

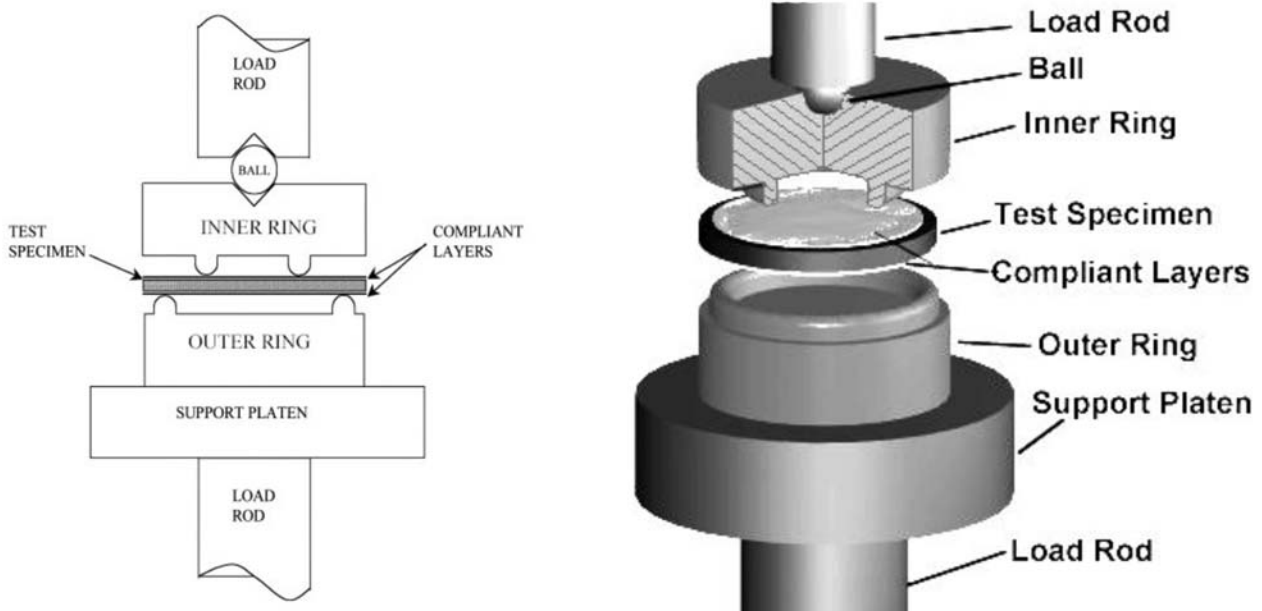
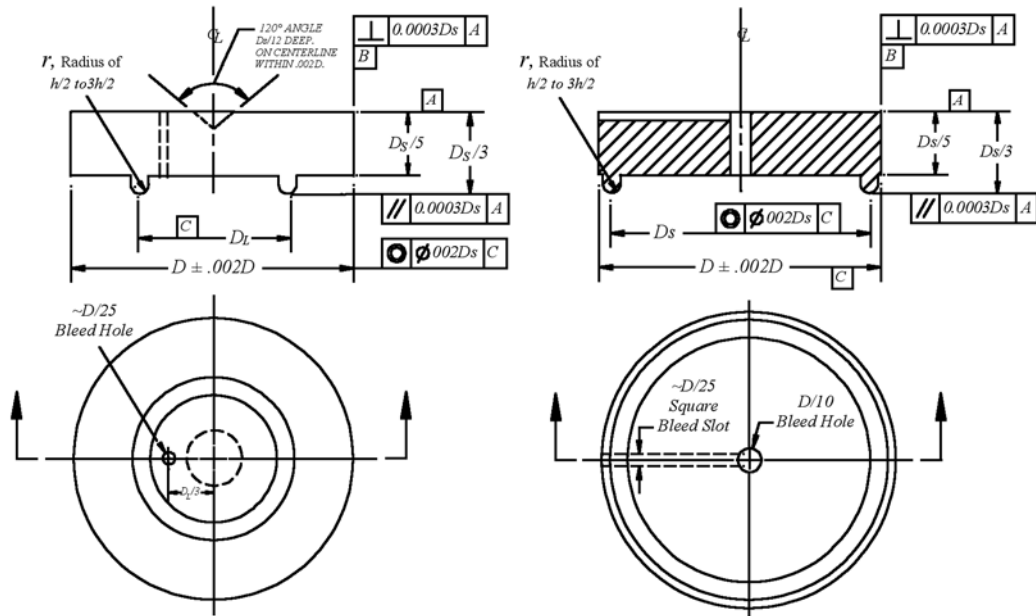
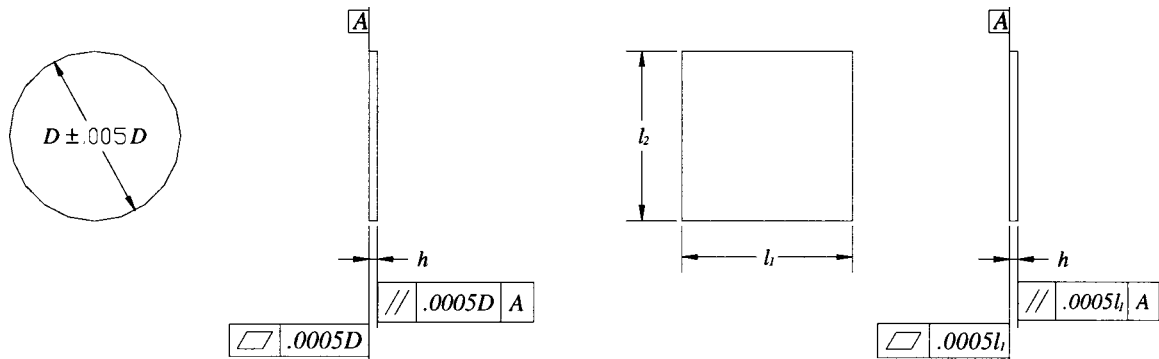


FIG. 1 Section View and Perspective View of Basic Fixturing and Test Specimen for Equibiaxial Testing



NOTE 1—0.4 to 0.8 μm surface finish. Harden to 40 Rc or greater.

FIG. 2 Load and Support Fixture Designs for Equibiaxial Testing



Round Test Specimen

Rectangular Test Specimen

FIG. 3 Recommended Equibiaxial Test Specimen Geometry (h and D or l_1 and l_2 are Determined from Eq 1-3).

particularly for very strong plates. However, excessively thick layers will redistribute the contact region and may affect results. The thicknesses listed above have been used successfully. Guidance regarding the use of thick layers cannot be given currently; some judgement may be required.

Alternatively, an appropriate lubricant (anti-seizing compound or Teflon oil) may be used to minimize friction. The lubricant should be placed only on the load and support rings so that effects of the test environment are not significantly altered. To aid fractographic examination, place a single strip of adhesive tape with a width of D_L or greater on the compressive face of the test specimen. Do not use multiple strips of tape, or a strip of tape with width less than D_L , as this may result in nonuniform loading.

6.2.4.2 *As-Fabricated Test Specimens*—If steel load and support rings are used to test as-fabricated test specimens (for example, as-fired ceramics and glass test specimens), minimize the effects of test specimen-ring misalignment by placing a

sheet of rubber or silicone (shore hardness of 60 ± 5) of approximately one-half the test specimen thickness between the test specimen and the support ring. To aid fractographic examination, place a single strip of adhesive tape with a width of D_L or greater on the compressive face of the test specimen. Do not use multiple strips of tape, or a strip of tape with width less than D_L , as this may result in nonuniform loading. To minimize the effects of friction at the load ring interface, place a sheet of carbon foil or TFE-fluorocarbon tape between the compressive surface of the test specimen and the load-ring. Alternatively, an appropriate lubricant (anti-seizing compound or TFE-fluorocarbon oil) may be used to minimize friction at the load ring. If acetyl polymer load rings are used, a compliant layer is not required. Minimize the effects of friction at the load ring interface, by placing a sheet of carbon foil or TFE-fluorocarbon tape between the compressive and tensile surfaces

of the test specimen and the load and support rings. Alternatively, an appropriate lubricant (anti-seizing compound or TFE-fluorocarbon oil) may be used to minimize friction at the load ring.

NOTE 2—As-fabricated test specimens that meet the flatness requirements in Fig. 3 may be tested as described in 6.2.4.1. A compliant layer is not necessary.

NOTE 3—The use of acetyl polymer load rings can result in sufficiently low friction (4) so that no layer is required. If the friction coefficient is less than 0.05, then the friction reduction layer may be eliminated.

6.3 *Alignment*—The load ring and support ring shall be aligned concentrically to 0.5 % of the support ring diameter. The test specimen shall be concentric with the load and support rings to 2 % of the support ring diameter.

6.4 *Allowable Deflection*—Excessive deflections can result in a calculated equibiaxial strength different than the actual equibiaxial strength. The test specimens allowed in this standard are designed to avoid excessive deflection (3, 5-7). Measurement of deflection is not required, however, center-point deflection can be measured using a deflectometer mounted in the test fixturing (Practice E83). Load-point deflection also may be measured via the test machine actuator, however, appropriate corrections for the test system compliance may need to be applied to the deflection data. Alternatively, deflection can be estimated via the elastic solutions given in section 10.1.

6.5 *Data Acquisition*—At the minimum, obtain an autographic record of applied load versus time. Either analog chart recorders or digital data acquisition systems can be used for this purpose although a digital record is recommended for ease of later data analysis. Ideally, an analog chart recorder or plotter should be used in conjunction with the digital data acquisition system to provide an immediate record of the test as a supplement to the digital record. Recording devices shall be accurate to within ± 1 % of the selected range for the testing system including readout unit, as specified in Practice E4, and shall have a minimum data acquisition rate of 10 Hz with a rate of 50 Hz preferred for the rates recommended in 9.2.2. If faster loading rates are used, then use an acquisition rate adequate to provide an error less than ± 1 % in the load reading.

6.5.1 Record crosshead displacement of the test machine or time similarly to the load or as independent variables of load.

6.6 *Dimension-Measuring Devices*—Micrometers and other devices used for measuring linear dimensions shall be accurate and precise to at least one half the smallest unit to which the individual dimension is required to be measured. For measuring the thickness, a micrometer with flat anvil faces a resolution better than or equal 0.002 mm is required. Ball-tipped or sharp anvil micrometers are not allowed because localized damage (for example, cracking) can be induced.

NOTE 4—Thickness measurement is especially critical to the calculation of the strength when the test specimens are less than 1 mm thick.

7. Precautionary Statement

7.1 Fractures of loaded advanced ceramics can occur at large loads and high strain energies. To prevent the release of uncontrolled fragments, polycarbonate shielding or equivalent

is recommended for operator safety and to capture test specimen fragments to aid fractography.

7.2 Fractures can create fine particles that may be a health hazard. Materials containing whiskers, small fibers or silica particles may also cause health hazards. For such materials, the operator is advised to consult the material safety data sheet for guidance prior to testing. Suitable ventilation or masks may be warranted.

8. Test Specimens

8.1 *Test Specimen Dimensions*—Fig. 3 illustrates test specimen geometry. The relative dimensions are chosen to ensure behavior reasonably described by simple plate theory. Choose the dimension such that the test specimen thickness, h , in units of mm, is

$$\frac{D_s}{10} \geq h \geq \sqrt{2\sigma_f D_s^2/3E} \quad (1)$$

where:

D_s = the support ring diameter in units mm,

σ_f = the expected equibiaxial fracture strength in units MPa, and

E = the modulus of elasticity in units MPa (Test Method C1259).

Choose the test specimen and support ring diameters such that the difference in diameters ($D-D_s$) is

$$2 \leq \frac{D - D_s}{h} \leq 12 \quad (2)$$

where:

D = the test specimen diameter in units of mm for circular test specimens.

NOTE 5—For test specimens machined according to 8.2.3, a non-dimensionalized overhang of $(D-D_s)/h = 2$ is generally sufficient. However, for test specimens that are scored from larger plates or for test specimens with poor edge finish, a nondimensionalized overhang of $(D-D_s)/h = 12$ may be required. For optical materials, nondimensionalized overhang larger than 12 may be required. Eq 7 is valid for overhangs as large as $(D-D_s)/h = 24$. However, such large overhang substantially alters the stress distribution, and tests performed with large overhang may result in substantially different measured strengths than tests performed with much smaller overhang. Thus, overhang of $(D-D_s)/h \leq 24$ is allowed. However, it is recommended that $(D-D_s)/h \leq 12$ be used. The edge stress for $D_s/h = 10$ varies from ~30% to ~50% of the maximum stress as $(D-D_s)/h$ varies from 12 to 2, respectively. For $D_s/h = 30$, the edge stress varies from ~12% to ~40% of the maximum stress as $(D-D_s)/h$ varies from 12 to 2, respectively [18]. The exact solution for the tangential stress at the edge of a circular plate can be calculated from:

$$\sigma_e = \frac{3F(1-\nu)(D_s^2 - D^2)}{2\pi h^2 D^2}$$

where the variables are as defined in Eq 1 and Eq 2.

It is recommended that the test specimens be circular, however, in some cases it is advantageous to fabricate rectangular test specimens. For a rectangular test specimen, the value of D for calculations with Eq 1 and Eq 2 is:

$$D = 0.54(l_1 + l_2) \quad (3)$$

where:

l_1 and l_2 = the lengths of the edges. The edge lengths should be within $0.98 \leq l_1/l_2 \leq 1.02$.

8.2 Test Specimen Preparation: Machined Test Specimens—A variety of surface preparations are acceptable. Unless the process used is proprietary, report specifics about the stages of material removal, wheel grits, wheel bonding, amount of material removed per pass, and type of coolant used. Regardless of the procedure used to machine the tensile surface of the test specimen, the flatness of the faces as well as the flatness of the edges shall be as specified in Fig. 3.

8.2.1 Application-Matched Machining—The tensile face of the equibiaxial test specimen will have the same surface/edge preparation as that given to a service component.

NOTE 6—An example of application matched machining is blanchard grinding of electronic substrates. Although damage may exist, it is acceptable as the component has such damage in its application.

8.2.2 Customary Practices—In instances where a customary machining procedure has been developed that is completely satisfactory for a class of materials (that is, it induces negligible surface/subsurface damage or residual stresses), this procedure may be used to machine the equibiaxial test specimens.

NOTE 7—Uniaxial surface grinding creates surface and subsurface microcracks, which may (or may not) be the strength-controlling flaws. Such machining cracks usually are oriented relative to the grinding direction and consequently may cause a pronounced variation in the uniaxial strength as a function of the test specimen orientation. If machining flaws dominate, equibiaxial test specimens will fail from the worst orientation and the measured equibiaxial strength will be representative of the machining damage. Further, the equibiaxial strength data may not correlate well with uniaxial data generated with standardized procedures that minimize the effects of such populations (8). Lapping or annealing can be used to minimize such effects in both equibiaxial strength tests and advanced ceramic components subjected to multiaxial stresses. Lapping needs to be sufficiently deep to remove machining damage (typically 10 to 30 μm deep). Note that surface finish is not a good indicator of the absence of machining damage.

8.2.3 Recommended Procedure—In instances where 8.2.1 or 8.2.2 are not appropriate, 8.2.3.1–8.2.3.4 shall apply.

8.2.3.1 Perform all grinding or cutting with ample supply of appropriate filtered coolant to keep the test specimen and grinding wheel constantly flooded and particles flushed. Grinding can be done in two stages, ranging from coarse to fine rates of material removal. All cutting can be done in one stage appropriate for the depth of cut.

8.2.3.2 The stock removal rate shall not exceed 0.03 mm per pass to the last 0.06 mm of material removed. Final finishing shall use diamond tools between 320 and 500 grit. No less than 0.06 mm shall be removed during the final finishing stage, and at a rate less than 0.002 mm per pass. Remove equal stock from opposite faces.

8.2.3.3 Grinding is followed by either annealing or lapping, as deemed appropriate.

NOTE 8—For alpha silicon carbide, annealing at $\sim 1200^\circ\text{C}$ in air for ~ 2 hours was sufficient to heal the grinding damage induced by the procedure in 8.2.3.2 without otherwise altering the material's strength (8). However, note that annealing can significantly alter a material's properties (9, 10), and specific procedures will need to be developed for each material.

NOTE 9—For lapping of alpha silicon carbide, the following procedure was successful in elimination of machining damage induced by uniaxial grinding: successive lapping with 15, 9 and 6 μm diamond pastes for ~ 30 , ~ 25 and ~ 15 minutes respectively (11). Approximately 10 μm of materials was removed. For tungsten carbide, successive machine lapping with 15

and 6 μm diamond pastes for ~ 60 and ~ 30 minutes, respectively, with a pressure of ~ 13.8 kPa was sufficient (12). Specific procedures will need to be developed for other materials.

8.2.3.4 To aid in post failure fractographic examination, it is recommended that the orientation of the grinding direction be marked on the test specimens. This can be accomplished with an indelible marker.

8.3 Test Specimen Preparation: As-Fabricated Test Specimens—In order to simulate the surface condition of an application in which no machining is used, limited testing of as-fabricated surfaces is allowed and precautions are recommended. The test specimen should be flat to 0.1 mm in 25 mm. For test specimens exhibiting less flatness, it is suggested that the user consider Test Method F394 or the use of fixturing designed to accommodate warped test specimens (for example, (13)). Data generated via this standard from test specimens with flatness tolerance exceeding 0.1 mm in 25 mm should be noted as warped and used only for comparison and quality control purposes.

8.4 Edge Preparation—Edge failure can be minimized by using the machining practice described in section 8.2.3. Additional beveling or edge preparation is not necessary. However, for as-fabricated test specimens exhibiting poor edge finish or for test specimens made from materials that are particularly difficult to machine without chipping of the edges, edge related failures can be minimized by using the overhang described in Eq 2 or by beveling the test specimen's tensile edge (that is, the edge of the face in contact with the support ring). If edge failures are a concern, it is recommended that the edge on the tensile face be inspected at $\sim 30\times$ magnification and any observed chips removed by beveling.

NOTE 10—For polycrystalline ceramics such as dense silicon carbides, silicon nitrides and aluminas, beveling can be accomplished by hand with 400-grit silicon carbide abrasive paper. Alternatively, a ~ 0.125 mm, 45° bevel can be ground onto the tensile edge according to the procedures in section 8.2.3. The grinding direction should be circumferential for circular test specimens and parallel to the edges for square test specimens. For softer materials or extremely strong materials, other methods may need to be developed.

8.5 Handling Precaution—Exercise care in storage and handling of test specimens to minimize the introduction of severe, extrinsic flaws. In addition, give attention to pre-test storage of test specimens in controlled environments or desiccators to avoid unquantifiable environmental degradation of test specimens prior to testing.

8.6 Number of Test Specimens—A minimum of 10 test specimens tested validly is required for the purpose of estimating a mean biaxial flexural strength. For the estimation of the Weibull parameters, a minimum of 30 test specimens validly tested is recommended. However, Practice C1239 should be consulted to determine if the resultant confidence intervals are adequate for the intended purpose. If material cost or test specimen availability limits the number of tests to be conducted, fewer tests may be conducted.

8.7 Valid Tests—A valid individual test is one that meets all the following requirements: (1) all the testing requirements of this test method, and (2) failure does not occur from the test specimen edges. Those tests failing from flaws at the edges,

while not valid, may be interpreted as interrupted tests for the purpose of censored test statistical analyses or as an indicator of edge condition.

9. Procedure

9.1 Test Specimen and Ring Dimensions—Measure the load and support ring diameters to within 0.2 % of D . Determine the test specimen diameter to 0.2 % of D by measuring at two radial positions nominally separated by 90° . For square test specimens, determine the width l_1 and length l_2 to 0.2 % at the middle of the edges. Diameter measurements can be made with a digital caliper, optical device (for example, machinists microscope) or a micrometer. In either case the resolution of the instrument shall be better than or equal to 0.01 mm. Measure the thickness to 0.5 % of h at the test specimen center and at four equally spaced positions on a diameter nominally equal to that of the support ring. If the test specimen faces are deemed to be parallel, then fewer thickness measurements may be made. To avoid damage in the critical gage section area, use a flat, anvil-type micrometer to measure the thickness. Exercise extreme caution to prevent damage to the test specimen. Alternatively, if damage is a concern even with an anvil-type micrometer, measure the thickness at the four support diameter positions prior to the test for setup purposes and measure the thickness near the test specimen center after the test. Record and report the measured dimensions. Use the average of the multiple measurements in the equibiaxial stress calculations.

9.1.1 Conduct inspection and measurements of all the test specimens and test specimen dimensions to assure compliance with the specifications of this test method.

9.1.2 Measurement of surface finish is not required; however, such information is desirable. Methods such as contacting profilometry can be used to determine surface roughness of the test specimen faces. If a contacting method is used, exercise caution to avoid causing surface damage to the test specimen. When quantified, report surface roughness and direction of the measurement with respect to the test specimen reference mark (see 8.2.3.4).

9.2 Test Modes and Rates:

9.2.1 General—Test modes and rates can have distinct influences on fracture behavior of advanced ceramics even at ambient temperatures. Test modes may involve load or displacement control. The recommended rates of testing are intended to be sufficiently rapid to nominally obtain the maximum equibiaxial strength at fracture of the material in the test environment considered. However, rates other than those recommended here may be used to evaluate rate effects. In all cases report the test mode and rate.

9.2.2 Displacement Rate—Displacement mode is defined as the control of, or free-running displacement of, the test machine actuator or crosshead. Different test specimen sizes require different displacement rates for a specified stress rate. Stress rates >30-35 MPa/s are recommended. The required displacement rate can be related to the maximum stress rate in the concentric ring test specimen as follows:

$$\dot{\delta} \equiv \left(\frac{D_s^2}{6Eh} \right) \dot{\sigma}$$

where:

- $\dot{\delta}$ = the displacement rate of the actuator or cross head in units of mm/s, and
- $\dot{\sigma}$ = the maximum value of the nominal recommended (or desired) stress rate occurring within the test specimen in units of MPa/s.

The other variables are as defined for Eq 1.

NOTE 11—The use of Eq 4 assumes that the test system compliance is small relative to that of the test specimen. If a compliant layer is used, the actual stressing rate will be lower and may be determined from the slope of a plot of load versus time. For the specific stress rate desired, the displacement rate can be increased to provide the desired stress rate.

9.2.3 Load Rate—For test systems employing closed loop controllers, a load rate can be directly applied to the test specimen. The load rate for a stress rate is calculated as follows:

$$\dot{F} = \left(\frac{2}{3} \pi h^2 \left[(1 - \nu) \frac{D_s^2 - D_L^2}{2D^2} + (1 + \nu) \ln \frac{D_s}{D_L} \right]^{-1} \right) \dot{\sigma} \quad (5)$$

where:

- \dot{F} = the required load rate in units of N/s,
- D_L = the load ring diameter, and
- ν = Poisson's ratio (Test Method C1259).

The other variables are as defined for Eq 1 and Eq 4. Alternatively, stress or load rates can be selected to minimize environmental effects when testing in ambient air by producing final fracture in 10 to 15 s:

$$t_f = \sigma_f / \dot{\sigma} \quad (6)$$

where:

- t_f = time to fracture in units of s.

9.3 Conducting the Equibiaxial Strength Test:

9.3.1 Apply cellophane tape to the compressive surface of the test specimen to retain fracture fragments. The tape should be sufficiently wide to completely cover the test specimen face. Trim excess tape as necessary to avoid interference or handling problems. Take care not to damage the tensile surface or tensile edge of the test specimen.

NOTE 12—Alternatively, fractography can be aided by drawing lines on the compressive surface of the test specimen with an indelible marker or a pencil.

9.3.2 Compliant Layer/Friction Reducing Layer—The diameter of the compliant layer and friction reduction layer should be sufficient to cover the outer diameter of the respective ring, but not be so large as to interfere with test specimen/fixture alignment. It is recommended that a hole of diameter $\sim D_s/2$ be cut in the center of the layer in contact with the support ring to allow exposure to the test environment.

9.3.3 Aligning the Test Specimen—The primary concern during testing is that the two load rings be concentric (to 0.5 % of the support ring diameter) and parallel to the test specimen faces. Prior to each test, inspect the load rings. Remove any nicks in the load and support rings (for example, polish the surfaces with emery cloth) and clean the surfaces with a suitable solvent (for example, alcohol). Assemble any compliant layer, friction reducing layer, the test specimen and load and support rings. If lubricant is use to eliminate friction, apply

it to the rings prior to assembly. Align the test specimen and fixtures. Slowly move the actuator or crosshead until a small preload is developed (for example, ~10 % of the failure load). Remove the alignment system (that is, V-blocks) and report the preload.

9.3.4 *Preparations for Testing*—Set the test mode and test rate on the test machine. Ready the autograph and data acquisition systems. Install the protective shield (see 7.1) for containment of fragments and activate the ventilation systems as required.

NOTE 13—If an extensometer is used to monitor bending, it should be zeroed without a preload applied. This will ensure that displacement due to the initial loading is observed.

9.3.5 *Conducting the Test*—Initiate the data acquisition. Initiate the test mode. After test specimen fracture, disable the action of the test machine and the data acquisition system. Report the measured breaking load to an accuracy of $\pm 1\%$ of the load range. Carefully collect any test specimen fragments from the fixturing. Place the test specimen fragments into a suitable, non-metallic container for later analysis.

9.3.6 Determine the ambient temperature and relative humidity at the end of the test in accordance with Test Method E337.

9.3.7 *Post-Test Interpretation*—For a properly conducted equibiaxial test, fracture should typically occur on the tensile surface within the diameter of the load-ring. Some fractures may also initiate from the region between the load ring and the support load ring. Frequent fracture at or near the load ring/test specimen interface implies excessive contact or friction stresses, or fixture/test specimen misalignment.

NOTE 14—Legitimate fracture may occur from outside the inner loading ring, especially in materials with a low Weibull modulus. In such cases, the disk strength reported is nonetheless based on the maximum stress that the disk sustained. In some instance, for example fracture mirror or fracture toughness calculations, the fracture stress used in the calculations is that at the failure origin.

9.4 *Post-Test Validation*—Fractographic examination of the test specimens is recommended to determine the location of test specimen fracture (Practice C1322). In particular, remnants should be examined for evidence of edge-related fractures or

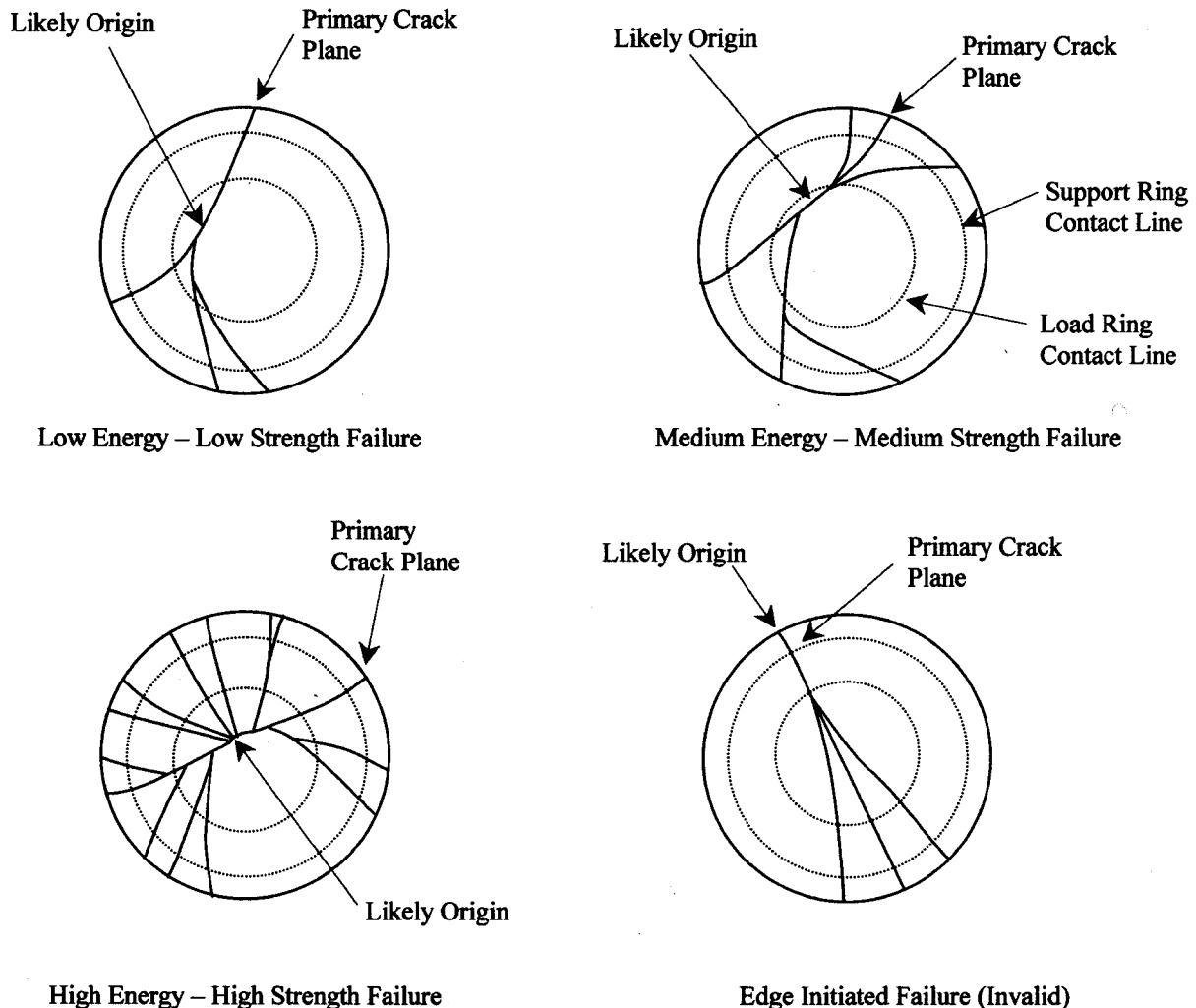


FIG. 4 Illustrations of Failure Patterns in Concentric Ring Test Specimens

repeated fractures near the load ring. Edge fracture indicates an invalid test (see Fig. 4). If the test specimens were machined by uniaxial grinding, it is recommended that the orientation of test specimens primary fracture plane relative to the grinding lay be determined. Repeated fracture parallel to the grinding lay implies the presence of significant machining damage.

10. Calculation of Results

10.1 *Equibiaxial Strength*—The formula for the equibiaxial strength, σ_f , of a circular plate in units of MPa is (6, 7):

$$\sigma_f = \frac{3F}{2\pi h^2} \left[(1 - \nu) \frac{D_s^2 - D_L^2}{2D^2} + (1 + \nu) \ln \frac{D_s}{D_L} \right] \quad (7)$$

where:

F = the breaking load in units of N. The other symbols are as defined in Eq 1 and Eq 5 in mm.

NOTE 15—The estimated strength of a plate is a weak function of Poisson's ratio, and reasonably accurate values should be used. An error of ~25 % in ν (use of $\nu = 0.21$ for a material with $\nu = 0.17$) results in an error of ~2 % in the estimated stress, and an error of ~1 % in the effective area for $m = 4$, where m is the Weibull modulus. A ~50 % error in ν (use of $\nu = 0.26$ for a material with $\nu = 0.17$) results in an error of ~4 % in the estimated stress, and an error of ~1 % in the effective area for $m = 10$.

For a rectangular test specimen, D is the diameter of a circle that expresses the characteristic size of the plate as follows:

$$D = \frac{l}{0.90961 + 0.12652 \frac{h}{D_s} + 0.00168 \ln \frac{l - D_s}{h}} \quad (8)$$

where $l = 0.5 (l_1 + l_2)$ and the other symbols are as defined Eq 1 and Eq 3.

10.2 *Plate Deflection*—The deflection for such a plate can be estimated from (6):

$$\delta = \frac{3F(1 - \nu^2) D_L^2}{8\pi E h^3} \left(\frac{D_s^2}{D_L^2} \left[1 + \frac{(1 - \nu)(D_s^2 - D_L^2)}{2(1 + \nu)D^2} \right] - \left(1 + \ln \frac{D_s}{D_L} \right) \right) \quad (9)$$

10.3 *Mean, Standard Deviation and Percent Coefficient of Variation*—For each series of tests, the mean, standard deviation, and percent coefficient of variation for each measured value can be calculated as follows:

$$\text{Mean} = \bar{x} = \frac{\sum_{i=1}^n x_i}{n} \quad (10)$$

$$\text{Standard deviation} = \text{s.d.} = \sqrt{\frac{\sum_{i=1}^n (x_i - \bar{x})^2}{n - 1}} \quad (11)$$

$$\text{Percent coefficient of variation} = \% \text{C.V.} = \frac{100(\text{s.d.})}{\bar{x}} \quad (12)$$

where:

x_i = the valid measured value and n is the number of valid tests.

11. Report

11.1 Report the following:

11.1.1 The date and location of testing.

11.1.2 All relevant material data including vintage data or billet identification data. As a minimum, report the date the

material was manufactured. For commercial materials, report the commercial designation.

11.1.3 Description of the stages of test specimen preparation including machining, heat treatments, coatings, or pre-test exposures applied either to the as-processed material or to the as-fabricated test specimens.

11.1.4 Type and configuration of the test machine (include drawing or sketch if necessary). If a commercial test machine was used, the manufacturer and model number are sufficient.

11.1.5 Material for and dimensions of the load and support rings.

11.1.6 Materials used as compliant and friction reducing layers, and the thickness of the layers, as applicable.

11.1.7 Type, configuration, and resolution of displacement measurement equipment used (include drawing or sketch if necessary). If a commercial extensometer was used, the manufacturer and model number are sufficient.

11.1.8 Test environment including relative humidity (Test Method E337), ambient temperature, and atmosphere (for example, ambient air, dry nitrogen, silicone oil, and so forth).

11.1.9 Test mode (load or displacement control) and applied test rate (load rate or displacement rate). The calculated stress rate should also be reported in units of MPa/s.

11.1.10 The values of Poisson's ratio and Young's modulus used in calculations.

11.1.11 Average diameter and thickness of each test specimen in units of mm.

11.1.12 Average surface roughness in units of μm , if measured, of the tensile face and direction of measurement relative to test specimen identification marks.

11.1.13 Preload applied to each test specimen in units of N.

11.1.14 Breaking load, F , of each test specimen in units of N.

11.1.15 Equibiaxial Strength, σ_f , of each test specimen in units of MPa.

11.1.16 Deflection at the Equibiaxial Strength, δ , of each test specimen in units of mm, if measured.

11.1.17 Location of fracture relative to the test specimen center, if applicable. Also, a summary of any fractographic analysis performed.

11.1.18 Number (n) of test specimens tested validly. In addition, report total number of test specimens tested (n_T) to provide an indication of the expected success rate of the particular test specimen geometry and test apparatus.

11.1.19 Mean, standard deviation, and coefficient of variation of the equibiaxial strength, σ_f , of the test lot in units of MPa.

12. Precision and Bias

12.1 Because of the nature of advanced ceramics and the lack of an extensive database, no definitive statement can be made at this time concerning precision and bias of this test method.

12.2 Although no definitive statement can be made regarding the precision and bias, an indication of the precision (that is, percent coefficient of variation) is shown in Table 1 for a range of advanced ceramics.

TABLE 1 Equibiaxial Test Results

Material	Equibiaxial Test Specimen Geometry (mm)				Equibiaxial Strength (MPa) σ_f	Number of Tests, n	Percent Coefficient of Variation, % C.V.
	D	D _s	D _L	h			
96 % Al ₂ O ₃ ^A (4)	23 ^B	11	5	0.4	327 ^C	42	11 ^D
99.6 % Al ₂ O ₃ ^E (4)	23 ^B	11	5	0.4	540 ^C	48	7.6 ^D
AlN ^F (4)	23 ^B	11	5	0.4	323 ^C	38	7.1 ^D
SiC ^G (8)	30	23	12	2	206	36	14
Si ₃ N ₄ ^H (14)	45	40	10	2.2	501	6	5.8
AlN ^I (15)	36 ^J	16	8	0.8	337	30	11 ^D
SiC ^G (17)	30	23	12	2	325	36	12

^A Grade ADS-96R, Coors Ceramic Company, Grand Junction, CO.

^B Test specimens were square with an edge length of 19 mm.

^C Weibull characteristic strength.

^D C.V. estimated from the approximation for Weibull modulus $m = 1.2/C.V.$ (16).

^E Grade ADS-996, Coors Ceramic Company, Golden, CO.

^F Carborundum Microelectronics, Phoenix, AZ.

^G Hexoloy SA Alpha SiC, Carborundum, Niagara Fall, NY. Now Saint Gobain Industrial Ceramics, Latrobe, PA.

^H Norton, NC 132, hot pressed silicon nitride, 1977 vintage.

^I Toshiba Corp., Japan.

^J Test specimens were square with an edge length of 30 mm.

12.3 Test results reported in Table 1 were generated by different investigators with different geometries and materials as chosen by the investigators.

13. Keywords

13.1 advanced ceramic; biaxial; concentric ring; equibiaxial; plate; strength

APPENDIXES

X1. ESTIMATION OF THE EFFECTIVE AREA AND EFFECTIVE VOLUME

X1.1 It is frequently of interest to calculate the “effective area” or “effective volume” of concentric ring test specimens. Assuming that the principal stresses control failure, the effective tensile surface area, A_e , and the effective tensile volume, V_e , can be calculated by numerical integration of the stress normalized to the maximum stress as a function of position according to the formulas:

$$A_e = \int_A \left(\frac{\sigma_1}{\sigma_{MAX}} \right)^m + \left(\frac{\sigma_2}{\sigma_{MAX}} \right)^m dA \quad (X1.1)$$

and

$$V_e = \int_V \left(\frac{\sigma_1}{\sigma_{MAX}} \right)^m + \left(\frac{\sigma_2}{\sigma_{MAX}} \right)^m + \left(\frac{\sigma_3}{\sigma_{MAX}} \right)^m dV \quad (X1.2)$$

where σ_1 and σ_2 are the tangential and radial stresses (1), and σ_{MAX} is the maximum stress as defined by Eq 7. The third principal stress, σ_3 , is generally taken as zero for thin plates.

X1.2 For general purposes, the effective tensile surface area and effective tensile volume can be estimated to better than 3 % from (19):

$$A_e \cong \frac{\pi}{2} D_L^2 \left\{ 1 + \frac{44(1+\nu)}{3(1+m)} \frac{(5+m)}{(2+m)} \left(\frac{D_s - D_L}{D_s D} \right)^2 \right\} \quad (X1.3)$$

$$\left[\frac{2D^2(1+\nu) + (D_s - D_L)^2(1-\nu)}{(3+\nu)(1+3\nu)} \right] \}$$

and

$$V_e \cong Ae \left[\frac{h}{2(m+1)} \right] \quad (X1.4)$$

for $m \geq 5$, $\nu \geq 0.17$, and $D_s/D_L = 0.5$, where m is the Weibull modulus. The other variables are as defined for Eq 1, Eq 2, and Eq 5. For $m \geq 5$ and $D_s/D_L = 0.2$, the estimates are better than 5 %. Ideally, the use of Eq X1.3 and X1.4 requires that the test data be grouped into surface failures and volume failures via fractography (Practice C1322), and the Weibull modulus be calculated with censored data analysis (Practice C1239).

X2. MULTILAYER CIRCULAR PLATES

X2.1 The strength of a circular plate made from layers with significantly different elastic constants can be determined from loading between concentric rings if the appropriate stress solution, elastic constants and assumptions are used. General formulations for thermal and mechanical loading of multilayer plates can be found in Refs 20-23 and specific formulas for normal stresses in Refs 23-26.

X2.1.1 Fig. X2.1 shows a diametrical section through the axis of symmetry of a thin, multilayer circular plate described by the cylindrical coordinates, r , θ , and z . The plate consists of n layers with individual thickness, t_i , where the subscript, i , denotes the layer number with layer 1 being at the bottom. The lower surface of layer 1 is located at $z = 0$, the interface between layers i and $i+1$ is located at h_i , and the upper surface of layer n is located at $z = h_n$ where h_n is the thickness of the plate. The relation between h_i and t_i is

$$h_i = \sum_{j=1}^i t_j \quad (i = 1 \text{ to } n) \quad (X2.1)$$

X2.1.2 The circular plate is subjected to equibiaxial flexural loading with $z = 0$ and $z = h_n$ being the support and the load surfaces, respectively. The interfaces between layers are assumed to remain bonded during loading.

X2.1.3 For a multilayer plate, the equibiaxial stress within the inner load ring for a layer i is,

$$\sigma_i = \frac{-E_i(z - z_n^*)F}{4\pi(1 - \nu_i)\Delta^*} \left[\ln\left(\frac{D_S}{D_L}\right) + \frac{(1 - \nu)(D_S^2 - D_L^2)}{2(1 + \nu)D^2} \right] \quad (X2.2)$$

(for $r = D_L/2$ and $i = 1$ to n),

where D , D_S , D_L , F , E_i and ν_i are the specimen diameter, support ring diameter, load ring diameter, applied force, elastic modulus and Poisson's ratio of the i^{th} layer, respectively. The terms z_n^* and Δ^* are

$$z_n^* = \frac{\sum_{i=1}^n \frac{E_i t_i}{1 - \nu_i^2} \left(h_{i-1} + \frac{t_i}{2} \right)}{\sum_{i=1}^n \frac{E_i t_i}{1 - \nu_i^2}} \quad (X2.3)$$

$$\Delta^* = \sum_{i=1}^n \frac{E_i t_i}{1 - \nu_i^2} \left[h_{i-1}^2 + h_i - 1 + \frac{t_i^2}{3} - \left(h_{i-1} + \frac{t_i}{2} \right) z_n^* \right], \quad (X2.4)$$

and ν has the physical meaning of an average Poisson's ratio of the layers

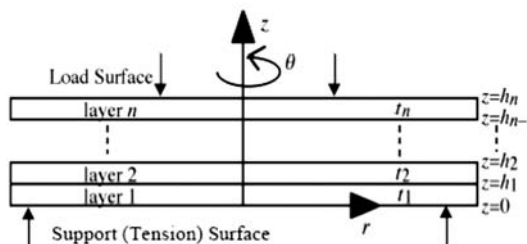
$$\nu = \frac{1}{h_n} \sum_{i=1}^n \nu_i t_i \quad (X2.5)$$

When $i = 1$, h_{i-1} (i.e., h_0) in Eq X2.3 and Eq X2.4 is defined as zero. Depending upon the strength of the individual layers relative to the stress distribution through the thickness of the multilayer during testing, failure is expected to initiate from the layer in which the tensile strength is first exceeded. This assumes good bonding between layers. If relatively poor bonding exists, delaminating could occur. Fractography to understand the failure is recommended. In addition, the material needs to behave as a continuum and thus the solution may not be applicable to systems with large porosity or channels. In such case, the materials strength may better determine by testing individual layers rather than an assembly.

X2.2 Example Calculations and Comparison with Finite Element Analysis (FEA)

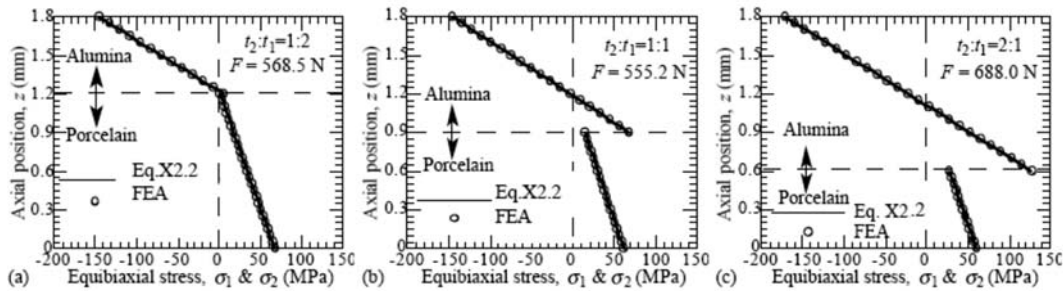
X2.2.1 For a bonded interface in multilayer systems, continuities of the displacement, the shear stress, and the stress normal to the interface are required. However, unless the material properties change continuously across the interface, the in-plane stress is not continuous at the interface. For monolayer plates, the maximum tension always occurs at the tensile surface (i.e., at $z = 0$). For multilayer plates, the equibiaxial stress is linear through the thickness in each individual layer; however, because of different elastic properties of the layers, the equibiaxial stress is discontinuous at the interface and the stress gradients are different. Fig. X2.2a-c show various stress distributions that can occur. In some cases, the maximum tension can occur at positions other than the tensile surface, as shown in Fig. X2.2c.

X2.2.2 For a $D = 16$ mm diameter bilayer plate consisting of a $t_1 = 0.6$ mm porcelain layer ($E = 64$ GPa, $\nu = 0.19$) and a $t_2 = 1.2$ mm Alumina layer ($E = 280$ GPa, $\nu = 0.23$) (total thickness



NOTE 1—Shows the coordinate system and the load and the support surfaces for an equibiaxial flexural test.

FIG. X2.1 Diametrical Section of a Thin Multilayer Circular Plate



NOTE 1—These are subjected to ring-on-ring loading for alumina to porcelain layer thickness ratios of (a) 1:2 with $F = 568.5$ N, (b) 1:1 with $F = 555.2$ N, and (c) 2:1 with $F = 688.0$ N. Both analytical and finite element analysis (FEA) results are shown.

FIG. X2.2 The Equibiaxial Stress through the Thickness of 1.8-mm thick Porcelain/Alumina Bilayer Circular Plates

of $h_n = h_2 = 1.8$ mm) subjected to loading between $D_L = 5$ mm and $D_S = 11$ mm rings, the in-plane, principal stresses in the porcelain range from 59.9 MPa at the exterior surface to 27.5 MPa at the interface ($h_1 = 0.6$ mm) for an applied load 688 N as shown in Fig. X2.2c. For the alumina layer, the in-plane stresses range from 126.6 MPa at the interface (h_1) to -171.8 MPa at the exterior surface ($h_2 = 1.8$ mm). Note that in this case, the maximum stress does not occur at the tensile surface, as is the case for monolayer plate, but at the interface between the layers. The location of the maximum tensile stress is determined by the elastic constants and layer thicknesses. If the isotropic solution (Eq 7) is used, a maximum stress of 113 MPa is predicted and the location is incorrectly assumed to be the plate tensile surface.

X2.3 Discussions

X2.3.1 It should be noted that an atomically sharp and smooth interface was assumed. This results in a discontinuity

of the in-plane equibiaxial stress at the interface. In reality, roughness might exist at the interface and the layers may interact to form an interphase at the interface. In the presence of interfacial roughness, mechanical interlocking at the interface results in stress transfer between the neighboring layers at the interface and smoothing of the stress discontinuity at the interface. In the presence of an interfacial interphase, the material properties have a continuous variation at the interface which also smooths the stress discontinuity at the interface. It should also be noted that residual thermal stresses can exist in multilayer systems because of the thermomechanical mismatch between layers. Closed-form solutions for thermal stresses in elastic multilayers have been derived elsewhere (25 and 26), and resultant stresses in a multilayer can be obtained by superposing the thermal stresses on the stresses due to equibiaxial flexural loading.

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Standard Test Methods for Sampling and Testing Brick and Structural Clay Tile¹

This standard is issued under the fixed designation C67; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the U.S. Department of Defense.

1. Scope*

1.1 These test methods cover procedures for the sampling and testing of brick and structural clay tile. Although not necessarily applicable to all types of units, tests include modulus of rupture, compressive strength, absorption, saturation coefficient, effect of freezing and thawing, efflorescence, initial rate of absorption and determination of weight, size, warpage, length change, and void area. (Additional methods of test pertinent to ceramic glazed facing tile are included in Specification C126.)

1.2 The text of this standard references notes and footnotes which provide explanatory material. These notes and footnotes (excluding those in tables and figures) shall not be considered as requirements of the standard.

NOTE 1—The testing laboratory performing this test method should be evaluated in accordance with Practice C1093.

1.3 The values stated in inch-pound units are to be regarded as standard. The values given in parentheses are mathematical conversions to SI units that are provided for information only and are not considered standard.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:²

C126 Specification for Ceramic Glazed Structural Clay Facing Tile, Facing Brick, and Solid Masonry Units

C150 Specification for Portland Cement

¹ These test methods are under the jurisdiction of Committee C15 on Manufactured Masonry Units and is the direct responsibility of Subcommittee C15.02 on Brick and Structural Clay Tile.

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² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

C1093 Practice for Accreditation of Testing Agencies for Masonry

C1232 Terminology of Masonry

E4 Practices for Force Verification of Testing Machines

E6 Terminology Relating to Methods of Mechanical Testing

3. Terminology

3.1 *Definitions*—For definitions relating to sampling and testing brick, refer to Terminology E6 and Terminology C1232.

4. Sampling

4.1 *Selection and Preparation of Test Specimens*—For the purpose of these tests, full-size brick, tile, or solid masonry units shall be selected by the purchaser or by the purchaser's authorized representative. Specimens shall be representative of the lot of units from which they are selected and shall include specimens representative of the complete range of colors, textures, and sizes. Specimens shall be free of or brushed to remove dirt, mud, mortar, or other foreign materials unassociated with the manufacturing process. Brushes used to remove foreign material shall have bristles of plastic (polymer) or horsehair. Wire brushes shall not be used for preparing specimens for testing. Specimens exhibiting foreign material that is not removed by brushing shall be discarded to ensure that damaged or contaminated specimens are not tested.

4.2 Number of Specimens:

4.2.1 *Brick*—For the modulus of rupture, compressive strength, abrasion resistance, and absorption determinations, at least ten individual brick shall be selected for lots of 1 000 000 brick or fraction thereof. For larger lots, five additional specimens shall be selected from each additional 500 000 brick or fraction thereof. Additional specimens are taken at the discretion of the purchaser.

4.2.2 *Structural Clay Tile*—For the weight determination and for compressive strength and absorption tests, at least five tile shall be selected from each lot of 250 tons (226.8 Mg) or fraction thereof. For larger lots, five additional specimens shall be tested for each 500 tons (453.6 Mg) or fraction thereof. In no case shall less than five tile be taken. Additional specimens are taken at the discretion of the purchaser.

*A Summary of Changes section appears at the end of this standard

4.3 *Identification*—Each specimen shall be marked so that it is identifiable at any time. Markings shall cover not more than 5 % of the superficial area of the specimen.

5. Specimen Preparation

5.1 *Drying and Cooling:*

5.1.1 *Drying*—Dry the test specimens in a ventilated oven at 230 to 239°F (110 to 115°C) for not less than 24 h and until two successive weighings at intervals of 2 h show an increment of loss not greater than 0.2 % of the last previously determined weight of the specimen.

5.1.2 *Cooling*—After drying, cool the specimens in a drying room maintained at a temperature of 75 ± 15°F (24 ± 8°C), with a relative humidity between 30 and 70 %. Store the units free from drafts, unstacked, with separate placement, for a period of at least 4 h and until the surface temperature is within 5°F (2.8°C) of the drying room temperature. Do not use specimens noticeably warm to the touch for any test requiring dry units. The specimens shall be stored in the drying room with the required temperature and humidity maintained until tested.

5.1.2.1 An alternative method of cooling the specimens to approximate room temperature is permitted as follows: Store units, unstacked, with separate placement, in a ventilated room maintained at a temperature of 75 ± 15°F (24 ± 8°C), with a relative humidity between 30 and 70 % for a period of 4 h and until the surface temperature is within 5°F (2.8°C) of the ventilated room temperature, with a current of air from an electric fan passing over them for a period of at least 2 hours. The specimens shall be stored in the ventilated room with the required temperature and humidity maintained until tested.

5.2 *Weight Determination:*

5.2.1 Weigh five full size specimens that have been dried and cooled (see 5.1). The scale or balance used shall have a capacity of not less than 3000 g and shall be sensitive to 0.5 g.

5.2.2 Report results separately for each specimen to the nearest 0.1 g, with the average of all specimens tested to the nearest 0.1 g.

5.3 *Removal of Silicone Coatings from Brick Units*—The silicone coatings intended to be removed by this process are any of the various polymeric organic silicone compounds used for water-resistant coatings of brick units. Heat the brick at 950 ± 50°F (510 ± 28°C) in an oxidizing atmosphere for a period of not less than 3 hours. The rate of heating and cooling shall not exceed 300°F (149°C) per hour.

NOTE 2—Additional specimen preparation requirements for specific tests are indicated in the individual test methods.

6. Modulus of Rupture (Flexure Test)

6.1 *Test Specimens*—The test specimens shall consist of whole full-size units that have been dried and cooled (see 5.1). Five such specimens shall be tested.

6.2 *Procedure:*

6.2.1 Support the test specimen flatwise unless specified and reported otherwise (that is, apply the load in the direction of the depth of the unit) on a span approximately 1 in. (25.4 mm) less than the basic unit length and loaded at midspan. Specimens

having recesses (panels or depressions) shall be placed so that such recesses are on the compression side. Apply the load to the upper surface of the specimen through a steel bearing plate ¼ in. (6.35 mm) in thickness and 1½ in. (38.10 mm) in width and of a length at least equal to the width of the specimen.

6.2.2 Make sure the supports for the test specimen are free to rotate in the longitudinal and transverse directions of the test specimen and adjust them so that they will exert no force in these directions.

6.2.3 *Speed of Testing*—The rate of loading shall not exceed 2000 lbf (8896 N)/min. This requirement is considered as being met when the speed of the moving head of the testing machine immediately prior to application of the load is not more than 0.05 in. (1.27 mm)/min.

6.3 *Calculation and Report:*

6.3.1 Calculate and report the modulus of rupture of each specimen to the nearest 1 psi (0.01 MPa) as follows:

$$S = 3W(l/2 - x)/bd^2 \quad (1)$$

where:

S = modulus of rupture of the specimen at the plane of failure, lb/in.² (Pa),

W = maximum load indicated by the testing machine, lbf (N),

l = distance between the supports, in. (mm),

b = net width, (face to face minus voids), of the specimen at the plane of failure, in. (mm),

d = depth, (bed surface to bed surface), of the specimen at the plane of failure, in. (mm), and

x = average distance from the midspan of the specimen to the plane of failure measured in the direction of the span along the centerline of the bed surface subjected to tension, in. (mm).

6.3.2 Calculate and report the average of the modulus of rupture determinations to the nearest 1 psi (0.01 MPa).

7. Compressive Strength

7.1 *Test Specimens:*

7.1.1 *Brick*—The test specimens shall consist of half brick units that have been dried and cooled (see 5.1), the full height and width of the unit, with a length equal to one half the full length of the unit ± 1 in. (25.4 mm), except as described below. When the test specimen, described above, exceeds the testing machine capacity, the test specimens shall consist of dry pieces of brick, the full height and width of the unit, with a length not less than one quarter of the full length of the unit, and with a gross cross-sectional area perpendicular to bearing not less than 14 in.² (90.3 cm²). Test specimens shall be obtained by any method that will produce, without shattering or cracking, a specimen with approximately plane and parallel ends. Five specimens shall be tested.

7.1.2 *Structural Clay Tile*—Test five tile specimens that have been dried and cooled (see 5.1) in a bearing bed length equal to the width ± 1 in. (25.4 mm); or test full-size units.

7.2 *Capping Test Specimens:*

7.2.1 All specimens shall be dry and cool within the meaning of 5.1.1 and 5.1.2 before any portion of the capping procedure is carried out.

7.2.2 Fill recessed or paneled surfaces that will become bearing surfaces during the compression test with a mortar composed of 1 part by weight of quick-hardening cement conforming to the requirements for Type III cement of Specification C150, and 2 parts by weight of sand. Age the specimens at least 48 h before capping them. Where the recess exceeds ½ in. (12.7 mm), use a brick or tile slab section or metal plate as a core fill. Cap the test specimens using one of the two procedures described in 7.2.3 and 7.2.4.

7.2.3 *Gypsum Capping*—Coat the two opposite bearing surfaces of each specimen with shellac and allow to dry thoroughly. Bed one of the dry shellacked surfaces of the specimen in a thin coat of neat paste of calcined gypsum (plaster of paris) that has been spread on an oiled nonabsorbent plate, such as glass or machined metal. The casting surface plate shall be plane within 0.003 in. (0.076 mm) in 16 in. (406.4 mm) and sufficiently rigid; and so supported that it will not be measurably deflected during the capping operation. Lightly coat it with oil or other suitable material. Repeat this procedure with the other shellacked surface. Take care that the opposite bearing surfaces so formed will be approximately parallel and perpendicular to the vertical axis of the specimen and the thickness of the caps will be approximately the same and not exceeding ⅛ in. (3.18 mm). Age the caps at least 24 h before testing the specimens.

NOTE 3—A rapid-setting industrial type gypsum is frequently used for capping.

7.2.4 *Sulfur-Filler Capping*—Use a mixture containing 40 to 60 weight % sulfur, the remainder being ground fire clay or other suitable inert material passing a No. 100 (150-µm) sieve with or without plasticizer. The casting surface plate requirements shall be as described in 7.2.3. Place four 1-in. (25.4-mm) square steel bars on the surface plate to form a rectangular mold approximately ½ in. (12.7 mm) greater in either inside dimension than the specimen. Heat the sulfur mixture in a thermostatically controlled heating pot to a temperature sufficient to maintain fluidity for a reasonable period of time after contact with the surface being capped. Take care to prevent overheating, and stir the liquid in the pot just before use. Fill the mold to a depth of ¼ in. (6.35 mm) with molten sulfur material. Place the surface of the unit to be capped quickly in the liquid, and hold the specimen so that its vertical axis is at right angles to the capping surface. The thickness of the caps shall be approximately the same. Allow the unit to remain undisturbed until solidification is complete. Allow the caps to cool for a minimum of 2 h before testing the specimens.

7.3 Procedure:

7.3.1 Test brick specimens flatwise (that is, the load shall be applied perpendicular to the bed surface of the brick with the brick in the stretcher position). Test structural clay tile specimens in a position such that the load is applied in the same direction as in service. Center the specimens under the spherical upper bearing within ⅛ in. (1.59 mm).

7.3.2 The testing machine shall conform to the requirements of Practices E4.

7.3.3 The upper bearing shall be a spherically seated, hardened metal block firmly attached at the center of the upper

head of the machine. The center of the sphere shall lie at the center of the surface of the block in contact with the specimen. The block shall be closely held in its spherical seat, but shall be free to turn in any direction, and its perimeter shall have at least ¼ in. (6.35 mm) clearance from the head to allow for specimens whose bearing surfaces are not exactly parallel. The diameter of the bearing surface shall be at least 5 in. (127.00 mm). Use a hardened metal bearing block beneath the specimen to minimize wear of the lower platen of the machine. The bearing block surfaces intended for contact with the specimen shall have a hardness not less than HRC60 (HB 620). These surfaces shall not depart from plane surfaces by more than 0.001 in. (0.03 mm). When the bearing area of the spherical bearing block is not sufficient to cover the area of the specimen, place a steel plate with surfaces machined to true planes within ± 0.001 in. (0.03 mm), and with a thickness equal to at least one third of the distance from the edge of the spherical bearing to the most distant corner between the spherical bearing block and the capped specimen.

7.3.4 *Speed of Testing*—Apply the load, up to one half of the expected maximum load, at any convenient rate, after which, adjust the controls of the machine so that the remaining load is applied at a uniform rate in not less than 1 nor more than 2 min.

7.4 Calculation and Report:

7.4.1 Calculate and report the compressive strength of each specimen to the nearest 10 psi (69 kPa) as follows:

$$\text{Compressive strength, } C = W/A \quad (2)$$

where:

C = compressive strength of the specimen, lb/in.² (or kg/cm²) (or Pa·10⁴),

W = maximum load, lbf, (or kgf) (or N), indicated by the testing machine, and

A = average of the gross areas of the upper and lower bearing surfaces of the specimen, in.² (or cm²).

NOTE 4—When compressive strength is to be based on net area (example: clay floor tile), substitute for A in the above formula the net area, in in.² (or cm²), of the fired clay in the section of minimum area perpendicular to the direction of the load.

7.4.2 Calculate and report the average of the compressive strength determinations to the nearest 10 psi (69 kPa).

8. Absorption

8.1 Accuracy of Weighings:

8.1.1 *Brick*—The scale or balance used shall have a capacity of not less than 2000 g, and shall be sensitive to 0.5 g.

8.1.2 *Tile*—The balance used shall be sensitive to within 0.2 % of the weight of the smallest specimen tested.

8.2 Test Specimens:

8.2.1 *Brick*—The test specimens shall consist of half brick conforming to the requirements of 7.1.1. Five specimens shall be tested.

8.2.2 *Tile*—The specimens for the absorption test shall consist of five tile or three representative pieces from each of these five tile. Two of the three representative pieces shall be taken from the shells and one from an interior web, the weight of each piece being not less than 227 g. The specimens shall have had their rough edges or loose particles ground off. Pieces

taken from tile that have been subjected to compressive strength tests shall be free of cracks due to failure in compression.

8.3 5-h and 24-h Submersion Tests:

8.3.1 Procedure:

8.3.1.1 Dry and cool the test specimens in accordance with 5.1 and weigh each one in accordance with 5.2.

8.3.1.2 *Saturation*—Submerge the dry, cooled specimen, without preliminary partial immersion, in clean water (soft, distilled or rain water) at 60 to 86°F (15.5 to 30°C) for the specified time. Remove the specimen, wipe off the surface water with a damp cloth and weigh the specimen. Complete weighing of each specimen within 5 min after removing the specimen from the bath.

8.3.2 Calculation and Report:

8.3.2.1 Calculate and report the cold water absorption of each specimen to the nearest 0.1 % as follows:

$$\text{Absorption, \%} = 100(W_s - W_d)/W_d \quad (3)$$

where:

W_d = dry weight of the specimen, and

W_s = saturated weight of the specimen after submersion in cold water.

8.3.2.2 Calculate and report the average cold water absorption of all specimens to the nearest 0.1 %.

8.4 1-h, 2-h, and 5-h Boiling Tests:

8.4.1 *Test Specimens*—The test specimens shall be the same five specimens used in the 5-h or 24-h cold-water submersion test where required and shall be used in the state of saturation existing at the completion of that test.

8.4.1.1 Dry and cool the test specimens in accordance with 5.1 when performing the boiling water absorption test without previously conducting the cold water absorption test.

8.4.2 Procedure:

8.4.2.1 Return the specimen that has been subjected to the cold-water submersion to the bath, and subject it to the boiling test as described in 8.4.2.2.

8.4.2.2 Submerge the specimen in clean water (soft, distilled or rain water) at 60 to 86°F (15.5 to 30°C) in such a manner that water circulates freely on all sides of the specimen. Heat the water to boiling, within 1 h, boil continuously for specified time, and then allow to cool to 60 to 86°F (15.5 to 30°C) by natural loss of heat. Remove the specimen, wipe off the surface water with a damp cloth, and weigh the specimen. Complete weighing of each specimen within 5 min after removing the specimen from the bath.

8.4.2.3 When the tank is equipped with a drain so that water at 60 to 86°F (15.5 to 30°C) passes through the tank continuously and at such a rate that a complete change of water takes place in not more than 2 min, make weighings at the end of 1 hour.

8.4.3 Calculation and Report:

8.4.3.1 Calculate and report the boiling water absorption of each specimen to the nearest 0.1 % as follows:

$$\text{Absorption, \%} = 100(W_b - W_d)/W_d \quad (4)$$

where:

W_d = dry weight of the specimen, and

W_b = saturated weight of the specimen after submersion in boiling water.

8.4.3.2 Calculate and report the average boiling water absorption of all specimens to the nearest 0.1 %.

8.5 Saturation Coefficient:

8.5.1 Calculate and report the saturation coefficient of each specimen to the nearest 0.01 as follows:

$$\text{Saturation coefficient} = (W_{c(24)} - W_d)/(W_{b(5)} - W_d) \quad (5)$$

where:

W_d = dry weight of the specimen,

$W_{c(24)}$ = saturated weight of the specimen after 24-h submersion in cold water, and

$W_{b(5)}$ = saturated weight of the specimen after 5-h submersion in boiling water.

8.5.2 Calculate and report the average saturation coefficient of all specimens to the nearest 0.01.

9. Freezing and Thawing

9.1 Apparatus:

9.1.1 *Compressor, Freezing Chamber, and Circulator* of such design and capacity that the temperature of the air in the freezing chamber will not exceed 16°F (−9°C) 1 h after introducing the maximum charge of units, initially at a temperature not exceeding 90°F (32°C).

9.1.2 *Trays and Containers*, shallow, metal, having an inside depth of 1½ ± ½ in. (38.1 ± 12.7 mm), and of suitable strength and size so that the tray with a charge of frozen units is movable by one technician.

9.1.3 *Balance*, having a capacity of not less than 2000 g and sensitive to 0.5 g.

9.1.4 *Drying Oven* that provides a free circulation of air through the oven and is capable of maintaining a temperature between 230 and 239°F (110 and 115°C).

9.1.5 *Thawing Tank* of such dimensions as to permit complete submersion of the specimens in their trays. Adequate means shall be provided so that the water in the tank is kept at a temperature of 75 ± 10°F (24 ± 5.5°C).

9.1.6 *Drying Room*, maintained at a temperature of 75 ± 15°F (24 ± 8°C), with a relative humidity between 30 and 70 %, and free from drafts.

9.2 Test Specimens:

9.2.1 *Brick*—The test specimens shall consist of half brick with approximately plane and parallel ends. When necessary, smooth any rough ends by trimming off a thin section with a masonry saw. The specimens shall be free from shattering or unsoundness, visually observed, resulting from the flexure or from the absorption tests. Additionally, prepare specimens by removing all loosely adhering particles, sand or edge shards from the surface or cores. Test five specimens.

9.2.2 *Structural Clay Tile*—The test specimens shall consist of five tile or of a cell not less than 4 in. (101.6 mm) in length sawed from each of the five tile.

9.3 Procedure:

9.3.1 Dry and cool the test specimens in accordance with 5.1. Weigh and record the dry weight of each in accordance with 5.2.

9.3.2 Carefully examine each specimen for cracks. A crack is defined as a fissure or separation visible to a person with normal vision from a distance of one foot under an illumination of not less than 50 fc. Mark each crack its full length with an indelible felt marking pen.

9.3.3 Submerge the test specimens in the water of the thawing tank for $4 \pm \frac{1}{2}$ hour.

9.3.4 Remove the specimens from the thawing tank and stand them in the freezing trays with one of their head faces down. Head face is defined as the end surfaces of a whole rectangular brick (which have the smallest area). (See Note 5.) A space of at least $\frac{1}{2}$ in. (12.7 mm) shall separate the specimens as placed in the tray. Pour sufficient water into the trays so that each specimen stands in $\frac{1}{2}$ in. depth of water and then place the trays and their contents in the freezing chamber for 20 ± 1 hour.

NOTE 5—The dimensions of some brick may prevent specimens from standing without support on one of their head faces. In such a case, any suitable rack or support that will achieve the $\frac{1}{2}$ in. (12.7 mm) separation of specimens and the specimen standing in $\frac{1}{2}$ in. (12.7 mm) depth of water will suffice.

9.3.5 Remove the trays from the freezing chamber after 20 ± 1 h and totally immerse them and their contents in the water of the thawing tank for $4 \pm \frac{1}{2}$ hour.

9.3.6 Freeze the test specimens by the procedure in 9.3.4 one cycle each day of the normal work week. Following the $4 \pm \frac{1}{2}$ h thawing after the last freeze-thaw cycle of the normal work week, remove the specimens from the trays and store them for 44 ± 1 h in the drying room. Do not stack or pile units. Provide a space of at least 1 in. (25.4 mm) between all specimens. Following this period of air drying, inspect the specimens, submerge them in the water of the thawing tank for $4 \pm \frac{1}{2}$ h, and again subject them to a normal week of freezing and thawing cycles in accordance with 9.3.4 and 9.3.5. When a normal 5-day work week is interrupted, put specimens into a drying cycle, which meets or extends past the 44 ± 1 h drying time outlined in the procedures of this section.

9.3.7 Continue the alternations of drying and submersion in water for $4 \pm \frac{1}{2}$ h, followed by 5 cycles of freezing and thawing or the number of cycles needed to complete a normal work week, until a total of 50 cycles of freezing and thawing has been completed. Stop the test when the test specimen develops a crack as defined in 9.4.3, breaks, or appears to have lost more than 3 % of its original weight by disintegration as judged by visual inspection.

9.3.8 After completion of 50 cycles, or when the test specimen has been withdrawn from test as a result of disintegration, dry and weigh the specimen as prescribed in 9.3.1.

9.4 Calculations, Examination, Rating and Report:

9.4.1 *Calculation*—Calculate the loss in weight as a percentage of the original weight of the dried specimen.

9.4.2 *Examination*—Re-examine the surface of the specimens for cracks (see 9.3.2) and record the presence of any new cracks developed during the freezing-thawing testing procedure.

Measure and record the length of the new cracks. Examine the specimens for disintegration during the freeze-thaw process.

9.4.3 *Rating*—A specimen is considered to fail the freezing and thawing test under any of the following circumstances:

9.4.3.1 *Breakage and Weight Loss*—A separation or disintegration resulting in a weight loss of greater than that permitted by the referenced unit specification for the appropriate classification.

9.4.3.2 *Cracking*—A specimen develops a crack during the freezing and thawing procedure that exceeds the length permitted by the referenced unit standard for the appropriate classification. If none of the above circumstances occur, the specimens are considered to pass the freezing and thawing test.

9.4.4 *Report*—The report shall state whether the sample passed or failed the test. Any failures shall include the rating and the reason for classification as a failure and the number of cycles causing failure in the event failure occurs prior to 50 cycles.

10. Initial Rate of Absorption (Suction) (Laboratory Test)

10.1 Apparatus:

10.1.1 *Trays or Containers*—Watertight trays or containers, having an inside depth of not less than $\frac{1}{2}$ in. (12.7 mm), and of such length and width that an area of not less than 300 in.² (1935.5 cm²) of water surface is provided. The bottom of the tray shall provide a plane, horizontal upper surface, when suitably supported, so that an area not less than 8 in. (203.2 mm) in length by 6 in. (152.4 mm) in width will be level when tested by a spirit level.

10.1.2 *Supports for Brick*—Two noncorrodible metal supports consisting of bars between 5 and 6 in. (127.00 and 152.4 mm) in length, having triangular, half-round, or rectangular cross sections such that the thickness (height) will be approximately $\frac{1}{4}$ in. (6.35 mm). The thickness of the two bars shall agree within 0.001 in. (0.03 mm) and, when the bars are rectangular in cross section, their width shall not exceed $\frac{5}{16}$ in. (7.9 mm).

10.1.3 *Means for Maintaining Constant Water Level*—Suitable means for controlling the water level above the upper surface of the supports for the brick within ± 0.01 in. (0.25 mm) (see Note 6), including means for adding water to the tray at a rate corresponding to the rate of removal by the brick undergoing test (see Note 7). For use in checking the adequacy of the method of controlling the rate of flow of the added water, a reference brick or half brick shall be provided whose displacement in $\frac{1}{8}$ in. (3.18 mm) of water corresponds to the brick or half brick to be tested within ± 2.5 %. Completely submerge the reference brick in water for not less than 3 h preceding its use.

NOTE 6—A suitable means for obtaining accuracy in control of the water level is provided by attaching to the end of one of the bars two stiff metal wires that project upward and return, terminating in points; one of which is $\frac{1}{8}$ in. (3.18 mm) and the other $\frac{1}{8}$ in. (3.18 mm) + 0.01 in. (0.25 mm) above the upper surface or edge of the bar. Such precise adjustment is obtainable by the use of depth plates or a micrometer microscope. When the water level with respect to the upper surface or edge of the bar is adjusted so that the lower point dimples the water surface when viewed by reflected light and the upper point is not in contact with the water, the water level is within the limits specified. Any

other suitable means for fixing and maintaining a constant depth of immersion shall be permitted when equivalent accuracy is obtained. An example of such other suitable means is the use of rigid supports movable with respect to the water level.

NOTE 7—A rubber tube leading from a siphon or gravity feed and closed by a spring clip will provide a suitable manual control. The so-called “chicken-feed” devices as a rule lack sensitivity and do not operate with the very small changes in water level permissible in this test.

10.1.4 *Balance*, having a capacity of not less than 3000 g, and sensitive to 0.5 g.

10.1.5 *Drying Oven*, conforming to the requirements of 9.1.4.

10.1.6 *Timing Device*—A suitable timing device, preferably a stop watch or stop clock, which shall indicate a time of 1 min to the nearest 1 s.

10.2 *Test Specimens*, consisting of whole brick. Five specimens shall be tested.

10.3 Procedure:

10.3.1 The initial rate of absorption shall be determined for the test specimen as specified, either oven-dried or ambient air-dried. When not specified, the initial rate of absorption shall be determined for the test specimens oven-dried. Dry and cool the test specimens in accordance with the applicable procedures 10.3.1.1 or 10.3.1.2. Complete the test procedure in accordance with 10.3.2, 10.3.3, and 10.3.4.

NOTE 8—There is no correlated relationship between the value of initial rate of absorption for ambient air-dried and oven-dried units. The test methods provide different information.

10.3.1.1 *Oven-dried Procedure*—Dry and cool the test specimens in accordance with 5.1.

10.3.1.2 *Ambient Air-dried Procedure*—Store units unstacked, with separate placement in a ventilated room maintained at a temperature of $75 \pm 15^\circ\text{F}$ ($24 \pm 8^\circ\text{C}$) with a relative humidity between 30 % and 70 % for a period of 4 h, with a current of air from an electric fan passing over them for a period of at least 2 hours. Continue until two successive weighings at intervals of 2 h show an increment of loss not greater than 0.2 % of the last previously determined weight of the specimen.

10.3.2 Measure to the nearest 0.05 in. (1.27 mm) the length and width of the flatwise surface of the test specimen of rectangular units or determine the area of other shapes to similar accuracy that will be in contact with the water. Weigh the specimen to the nearest 0.5 g.

10.3.3 Adjust the position of the tray for the absorption test so that the upper surface of its bottom will be level when tested by a spirit level, and set the saturated reference brick (10.1.3) in place on top of the supports. Add water until the water level is $\frac{1}{8} \pm 0.01$ in. (3.18 ± 0.25 mm) above the top of the supports. When testing tile with scored bed surfaces, the depth of water level is $\frac{1}{8} \pm 0.01$ in. plus the depth of scores.

10.3.4 After removal of the reference brick, set the test brick in place flatwise, counting zero time as the moment of contact of the brick with the water. During the period of contact (1 min \pm 1 s) keep the water level within the prescribed limits by adding water as required. At the end of 1 min \pm 1 s, lift the brick from contact with the water, wipe off the surface water with a damp cloth, and reweigh the brick to the nearest 0.5 g.

Wiping shall be completed within 10 s of removal from contact with the water, and weighing shall be completed within 2 min.

NOTE 9—Place the brick in contact with the water quickly, but without splashing. Set the brick in position with a rocking motion to avoid the entrapping of air on its under surface. Test brick with frogs or depressions in one flatwise surface with the frog or depression uppermost. Test molded brick with the struck face down.

10.4 Calculation and Report:

10.4.1 The difference in weight in grams between the initial and final weighings is the weight in grams of water absorbed by the brick during 1-min contact with the water. When the area of its flatwise surface (length times width) does not differ more than ± 0.75 in.² (4.84 cm²) (± 2.5 %) from 30 in.² (193.55 cm²), report the gain in weight of each specimen to the nearest 0.1 g, as its initial rate of absorption in 1 min.

10.4.2 When the area of its flatwise surface differs more than ± 0.75 in.² (4.84 cm²) (± 2.5 %) from 30 in.² (193.55 cm²), calculate the equivalent gain in weight from 30 in.² (193.55 cm²) of each specimen to the nearest 0.1 g as follows:

$$X = 30 W/LB \text{ (metric } X = 193.55 W/LB) \quad (6)$$

where:

X = gain in weight corrected to basis of 30 in.² (193.55 cm²) flatwise area,

W = actual gain in weight of specimen, g,

L = length of specimen, in., (cm), and

B = width of specimen, in., (cm).

10.4.3 Report the corrected gain in weight, X , of each specimen to the nearest 0.1 g, as the initial rate of absorption in 1 min.

10.4.4 When the test specimen is a cored brick, calculate the net area and substitute for LB in the equation given in 10.4.2. Report the corrected gain in weight, X , of each specimen to the nearest 0.1 g, as the initial rate of absorption in 1 min.

10.4.5 When the specimen is non-prismatic, calculate the net area by suitable geometric means and substitute for LB in the equation given in 10.4.2.

10.5 Calculate and report the average initial rate of absorption of all specimens tested to the nearest 0.1 g/min/30 in.² (193.55 cm²).

10.6 Report the method of drying as oven-dried (in accordance with 10.3.1.1) or ambient air-dried (in accordance with 10.3.1.2).

11. Efflorescence

11.1 Apparatus:

11.1.1 *Trays and Containers*—Watertight shallow pans or trays made of corrosion-resistant metal or other material that will not provide soluble salts when in contact with distilled water containing leachings from brick. The pan shall be of such dimensions that it will provide not less than a 1-in. (25.4-mm) depth of water. Unless the pan provides an area such that the total volume of water is large in comparison with the amount evaporated each day, suitable apparatus shall be provided for keeping a constant level of water in the pan.

11.1.2 *Drying Room*, conforming to the requirements of 9.1.6.

11.1.3 *Drying Oven*, conforming to the requirements of 9.1.4.

11.1.4 *Brush*, a soft-bristle brush.

11.2 *Test Specimens*:

11.2.1 The sample shall consist of ten full-size brick.

11.2.2 The ten specimens shall be sorted into five pairs so that both specimens of each pair are similar in appearance.

11.3 *Preparation of Specimens*—Remove by brushing any adhering dirt so as not to mistake it for efflorescence. Dry and cool the specimens in accordance with 5.1.

11.4 *Procedure*:

11.4.1 Set one specimen from each of the five pairs, on end, partially immersed in distilled water to a depth of approximately 1 in. (25.4 mm) for 7 days in the drying room. When several specimens are tested in the same container, separate the individual specimens by a spacing of at least 2 in. (50.8 mm).

NOTE 10—Do not test specimens from different sources simultaneously in the same container, because specimens with a considerable content of soluble salts will contaminate salt-free specimens.

NOTE 11—Empty and clean the pans or trays after each test.

11.4.2 Store the second specimen from each of the five pairs in the drying room without contact with water.

11.4.3 At the end of 7 days, inspect the first set of specimens and then place both sets in the drying oven without contact with water for 24 hours.

11.5 *Examination and Rating*—After drying, examine and compare each pair of specimens, observing the top and all four faces of each specimen from a distance of 10 ft. (3 m) under an illumination of not less than 50 footcandles (538.2 lm/m²) by an observer with normal vision. When under these conditions no difference is noted, report the rating as “not effloresced.” When a perceptible difference due to efflorescence is noted under these conditions, report the rating as “effloresced.” Report the appearance and distribution of the efflorescence.

11.6 *Precision and Bias*—No information is presented about either the precision or bias of the test method for efflorescence because the test result is nonquantitative.

12. Weight per Unit Area

12.1 *Apparatus*—A scale or balance sensitive to within 0.2 % of the weight of the smallest specimen.

12.2 *Procedure*—Weigh in accordance with 5.2 five full size structural clay tile units that have been dried and cooled (see 5.1).

12.3 *Calculation and Report*:

12.3.1 Calculate the weight per unit area of each specimen as follows:

$$W_a = \frac{nW_d}{A_{fa1} + A_{fa2}} \quad (7)$$

where:

W_a = weight per unit area of the specimen, lb/ft² (kg/m²),

n = number of faces of the specimen (1 for split tile units or 2 for all other units),

W_d = dry weight of the specimen, lb (kg),

A_{fa1} = area (height × length) of finished face of specimen, ft² (m²), and

A_{fa2} = area (height × length) of back face of specimen, ft² (m²).

12.3.2 Report the results of Eq 7 separately for each specimen to the nearest 1 g and the average to the nearest 1 g for all specimens tested.

13. Measurement of Size

13.1 *Apparatus*—Either a 1-ft (or metric) steel rule, graduated in 1/32-in. (or 1-mm) divisions, or a gage or caliper having a scale ranging from 1 to 12 in. (25 to 300 mm), and having parallel jaws, shall be used for measuring the individual units. Steel rules or calipers of corresponding accuracy and size required shall be used for measurement of larger brick, solid masonry units, and tile.

13.2 *Procedure*—Measure ten whole full-size units that have been dried and cooled (see 5.1). These units shall be representative of the lot and shall include the extremes of color range and size as determined by visual inspection. (It is permissible to use the same samples for determining efflorescence and other properties.)

13.3 *Individual Measurements of Width, Length, and Height*—Measure the width across both ends and both beds from the midpoints of the edges bounding the faces. Record these four measurements to the nearest 1/32 in. (1 mm) and record the average to the nearest 1/64 in. (0.5 mm) as the width. Measure the length along both beds and along both faces from the midpoints of the edges bounding the ends. Record these four measurements to the nearest 1/32 in. (1 mm) and record the average to the nearest 1/64 in. (0.5 mm) as the length. Measure the height across both faces and both ends from the midpoints of the edges bounding the beds. Record these four measurements to the nearest 1/32 in. (1 mm) and record the average to the nearest 1/64 in. (0.5 mm) as the height. Use the apparatus described in 13.1. Retest by the same method when required.

13.4 *Report*—Report the average width, length, and height of each specimen tested to the nearest 1/32 in. (1.0 mm).

14. Measurement of Warpage

14.1 *Apparatus*:

14.1.1 *Steel Straightedge*:

14.1.2 *Rule or Measuring Wedge*—A steel rule graduated from one end in 1/32-in. (or 1-mm) divisions, or alternatively, a steel measuring wedge 2.5 in. (60 mm) in length by 0.5 in. (12.5 mm) in width by 0.5 in. (12.5 mm) in thickness at one end and tapered, starting at a line 0.5 in. (12.5 mm) from one end, to zero thickness at the other end. The wedge shall be graduated in 1/32-in. (or 1-mm) divisions and numbered to show the thickness of the wedge between the base, AB, and the slope, AC, Fig. 1.

14.1.3 *Flat Surface*, of steel or glass, not less than 12 by 12 in. (305 by 305 mm) and plane to within 0.001 in. (0.025 mm).

14.1.4 *Brush*, a soft-bristle brush.

14.2 *Sampling*—Use the sample of ten units selected for determination of size.

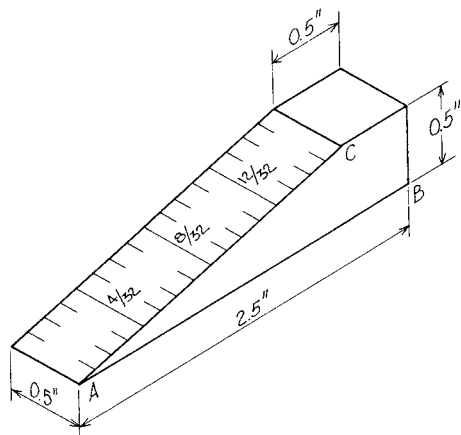


FIG. 1 Measuring Wedge

14.3 *Preparation of Samples*—Test the specimens as received, except remove any adhering dirt by brushing.

14.4 *Procedure:*

14.4.1 *Concave Surfaces*—Where the warpage to be measured is of a surface and is concave, place the straightedge lengthwise or diagonally along the surface to be measured, selecting the location that gives the greatest departure from straightness. Select the greatest distance from the unit surface to the straightedge. Using the steel rule or wedge, measure this distance to the nearest $\frac{1}{32}$ in. (1 mm), and record as the concave warpage of the surface. See Fig. 2.

14.4.2 *Concave Edges*—Where the warpage to be measured is of an edge and is concave, place the straightedge between the ends of the concave edge to be measured. Select the greatest distance from the unit edge to the straightedge. Using the steel rule or wedge, measure this distance to the nearest $\frac{1}{32}$ in. (1 mm), and record as the concave warpage of the edge.

14.4.3 *Convex Surfaces*—When the warpage to be measured is of a surface and is convex, place the unit with the convex surface in contact with a plane surface and with the corners approximately equidistant from the plane surface. Using the steel rule or wedge, measure the distance to the nearest $\frac{1}{32}$ in.

(1 mm) of each of the four corners from the plane surface. Record the average of the four measurements as the convex warpage of the unit.

14.4.4 *Convex Edges*—Where the warpage to be measured is of an edge and is convex, place the straightedge between the ends of the convex edge. Select the greatest distance from the unit edge to the straightedge. Using the steel rule or wedge, measure this distance to the nearest $\frac{1}{32}$ in. (1 mm) and record as the convex warpage of the edge.

14.5 *Report*—Report all recorded warpage measurements of each specimen tested to the nearest $\frac{1}{32}$ in. (1.0 mm).

15. Measurement of Length Change

15.1 *Apparatus*—A dial micrometer or other suitable measuring device graduated to read in 0.0001-in. (or 0.001-mm) increments, mounted on a stand suitable for holding the specimen in such a manner that reproducible results are obtained, shall be used for measuring specimen length. Provisions shall be made to permit changing the position of the dial micrometer on its mounting rod so as to accommodate large variations in specimen size. The base of the stand and the tip of the dial micrometer shall have a conical depression to accept a $\frac{1}{4}$ -in. (6.35-mm) steel ball. A suitable reference instrument shall be provided for checking the measuring device.

15.2 *Preparation of Specimen*—Remove the ends of deeply textured specimens to the depth of the texture by cutting perpendicular to the length and parallel to each other. Drill a hole in each end of the specimen with a $\frac{1}{4}$ -in. (6.35-mm) carbide drill. Drill these holes at the intersection of the two diagonals from the corners. Place $\frac{1}{4}$ -in. (6.35-mm) steel balls in these depressions by cementing in place with a calcium aluminate cement. Any equivalent method for establishing the reference length is permissible.

15.3 *Procedure*—Mark the specimen for identification and measure to the nearest 0.0001 in. (or 0.001 mm) in a controlled environment and make subsequent measurements in the same controlled environment, $\pm 2^\circ\text{F}$ ($\pm 1^\circ\text{C}$) and $\pm 5\%$ relative humidity. Record the temperature and relative humidity. Apply

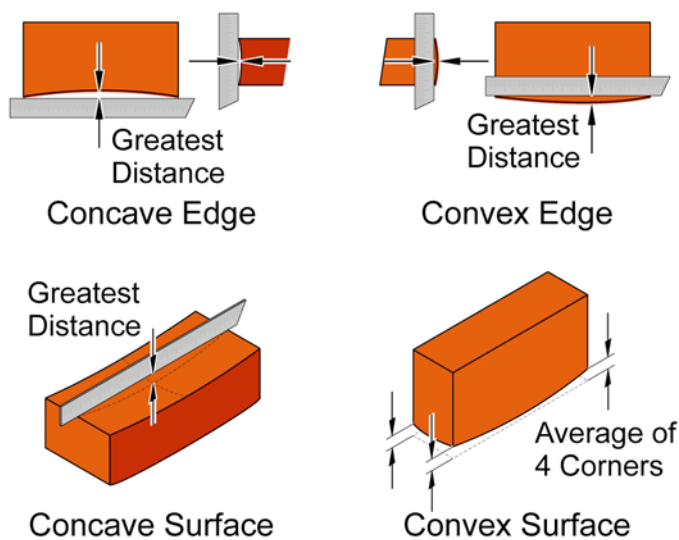


FIG. 2 Warpage Measurements

a reference mark to the specimen for orientation in the measuring device. Check the measuring device with the reference instrument before each series of measurements.

15.4 *Report*—When more than one specimen is tested, calculate and report the average length change of all specimens to the nearest 0.0001 in. (0.001 mm). The report shall include all individual recordings as well as the recorded laboratory temperature and relative humidity.

16. Initial Rate of Absorption (Suction)—Field Test

16.1 *Scope*—This test method is intended to serve as a volumetric means of determining the initial rate of absorption (IRA) of any size brick when weighing determination, described in Section 10 of these test methods, is impractical. This test method is applicable to assess the need for wetting the brick. This test method is performed on specimens taken from the field with no modification of moisture content, therefore, the IRA determined by this test method may differ from the IRA determined by the laboratory test method in Section 10, which requires drying the specimens.

16.2 *Apparatus:*

16.2.1 *Absorption Test Pan*—A watertight, rectangular pan, constructed of noncorroding material, with a flat, rigid bottom and inside depth of about 1½ in. (38.1 mm). The inside length and width of the pan shall exceed the length and width of the tested brick by a minimum of 3 in. (76.2 mm) but not more than 5 in. (127.0 mm).

16.2.2 *Brick Supports*—Two noncorroding rectangular bars, ¼ in. (6.4 mm) in height and width and 1 in. (25.4 mm) shorter than the inside width of the pan in length. The brick supports shall be placed on the bottom of the pan just before the test or shall be permanently affixed to the bottom of the pan. The space between the supports shall be approximately 4 in. (101.6 mm) shorter than the length of the tested brick. A device indicating the desired water level shall be permanently attached to the end of one of the brick supports or shall be suspended from the top of the pan (see Fig. 3 (a) and (b)). Any other device of equivalent accuracy for controlling the required water level, ⅛ in. (3.2 mm) above the brick supports, is permitted to be used in place of that depicted in Fig. 3.

16.2.3 *Timing Device*—A suitable timing device that shall indicate a time of 1 min to the nearest 1 s.

16.2.4 *Squeeze Bottle*—A plastic squeeze bottle, 100 mL capacity.

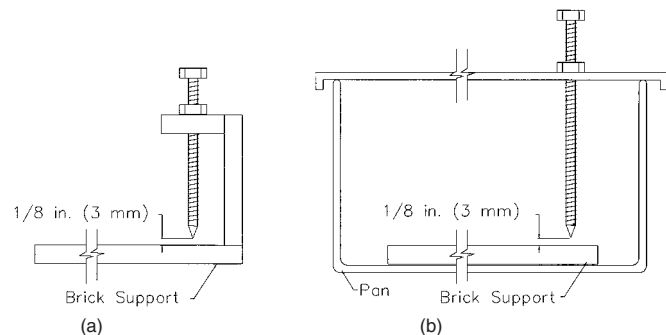


FIG. 3 Water Level Indicators

16.2.5 *Graduated Cylinder*—A plastic or glass graduated measuring cylinder, 100 mL capacity.

16.3 *Test Specimens*—Select six whole brick in accordance with the requirements of Paragraph 4.1.

16.4 *Procedure:*

16.4.1 Completely immerse one brick specimen in a container of water for 2 hours.

16.4.2 Measure to the nearest ⅛ in. (1.6 mm) the length and width of the five remaining specimens at the surface that will be in contact with water. When the test specimens are cored, determine the area of the cores at the same surface.

16.4.3 Pre-wet and drain the absorption pan and place it on a flat, level surface.

16.4.4 Remove the pre-wetted specimen from the container, shake off the surface water, and place the specimen on brick supports in the pan. Pour water into the pan until the water reaches a level ⅛ in. (3.2 mm) above the brick supports. (When using a pointed level water indicator, pour water into the pan until the water makes a minimum contact (dimpling effect).) Remove the pre-wetted brick, and tilt the brick sharply so that one corner serves as a drip point for clinging surface water to return to the pan. Gently shake the brick to make the last drop fall. Put the pre-wetted brick back into the container of water.

16.4.5 Using the graduated cylinder, fill the squeeze bottle with exactly 100 mL of water.

16.4.6 Set the first test specimen squarely on the brick supports, counting zero time as the moment the brick contacts the water. At the end of 1 min ± 1 s lift the test specimen from water and tilt the brick sharply so that one corner serves as a drip point for clinging surface water to return to the pan. Gently shake the brick to make the last drop fall.

16.4.6.1 Continue setting the remaining test specimens into the pan in the same way until all five specimens are tested. During the test add water to the pan, using the squeeze bottle, to keep the water level approximately constant at the ⅛ in. depth. Refill the squeeze bottle with 100 mL of water when empty, recording each refill.

16.4.6.2 After the last specimen is tested, place the pre-wetted brick back in the pan and restore the original level with water from the squeeze bottle.

NOTE 12—Place the brick in contact with the water quickly, but without splashing. Set the brick in position with a rocking motion to avoid the entrapping of air on its under surface. Test brick with frogs or depressions in one flatwise surface with the frog or depression uppermost. Test molded brick with the struck face down.

16.4.7 Using the graduated cylinder, measure the volume of water remaining in the squeeze bottle.

16.5 *Calculation and Report:*

16.5.1 The number of refills plus the first full bottle, times 100 mL, minus the volume of water remaining in the squeeze bottle, is the total measured volume of water in millilitres absorbed by the five specimens.

$$V_t = 100(n + 1) - V_r \tag{8}$$

where:

V_t = total measured volume of water absorbed by all tested specimens, mL,

n = the number of squeeze bottle refills, and
 V_r = the volume of water remaining in the squeeze bottle, mL.

16.5.2 When the average net surface area in contact with water of a single specimen (sum of net surface areas divided by the number of specimens) differs by $\pm 0.75 \text{ in.}^2$ (4.84 cm^2) or less from 30 in.^2 (193.5 cm^2), report the total measured absorbed volume of water divided by five, the number of tested specimens, as the IRA (Field) in $\text{g/min}/30 \text{ in.}^2$

$$\text{IRA (Field)} = \frac{V_t}{5} \quad (9)$$

16.5.3 When the average net surface area in contact with water differs by more than $\pm 0.75 \text{ in.}^2$ (4.84 cm^2) from 30 in.^2 (193.5 cm^2), calculate the equivalent volume in 1 min for 30 in.^2 (193.5 cm^2) of surface as follows:

$$V_c = \frac{30 V_t}{A_n} \left(\text{metric } V_c = \frac{193.5 V_t}{A_n} \right) \quad (10)$$

where:

V_c = average volume of absorbed water by a specimen, corrected to basis of 30 in.^2 (193.5 cm^2) of surface, mL, and

A_n = sum of net surface areas in contact with water of all tested specimens, in.^2 (cm^2).

16.5.4 *Report*—Report the corrected volume (V_c) as the IRA (Field) in $\text{g/l min}/30 \text{ in.}^2$

16.6 *Precision and Bias*—Insufficient data is currently available for a precision and bias statement.

17. Measurement of Void Area in Cored Units

17.1 Apparatus:

17.1.1 *Steel Rule or Calipers*—As described in 13.1.

17.1.2 *Graduated Cylinder*—A glass cylinder with a capacity of 500 mL.

17.1.3 *Paper*—A sheet of smooth, hard-finish paper not less than 24 by 24 in. (610 by 610 mm).

17.1.4 *Sand*—500 mL of clean, dry sand.

17.1.5 *Steel Straightedge*.

17.1.6 *Flat Surface*—A level, flat, smooth, clean dry surface.

17.1.7 *Brush*—A soft-bristle brush.

17.1.8 *Neoprene Mat*—24 by 24 in. (610 by 610 mm) open-cell neoprene sponge $\frac{1}{4}$ in. (6.4 mm) in thickness.

17.1.9 *Balance*—See 10.1.4.

17.2 *Test Specimens*—Use of a sample of ten units selected as described for the determination of size. (It is permissible to use the samples taken for the determination of size.)

17.3 *Preparation of Samples*—Test the specimens as received, except remove any adhering dirt by brushing.

17.4 Procedure:

17.4.1 Measure and record the length, width, and depth of the unit as described for the determination of size.

17.4.2 Place the unit to be tested bed down (cores vertical) on the sheet of paper that has been spread over the neoprene mat on the flat surface.

17.4.3 Fill the cores with sand, allowing the sand to fall naturally. Do not work the sand into the cores. Using the steel straightedge, bring the level of the sand in the cores down to the top of the unit. With the brush, remove all excess sand from the top of the unit and from the paper sheet.

17.4.4 Lifting the unit up, allow all of the sand in the cores to fall on the sheet of paper.

17.4.5 Transfer the sand from the sheet of paper to the balance, weighing and recording to the nearest 0.5 g.

17.4.6 With a separate portion of the sand, fill a 500 mL cylinder to the exact 500 mL graduation by allowing the sand to fall naturally and without shaking or vibrating the cylinder. Transfer this sand to the balance, weighing and recording to the nearest 0.5 g.

17.5 Calculation and Report:

17.5.1 Determine the volume of sand held in the test unit as follows:

$$V_s = \frac{500 \text{ mL}}{S_c} \times S_u \quad (11)$$

where:

V_s = volume of sand held in test unit,

S_c = weight, in grams, of 500 mL sand contained in graduated cylinder, and

S_u = weight in grams of sand held in test unit.

17.5.2 Determine the percentage of void as follows:

$$\% \text{ Void area} = \frac{V_s}{V_u} \times \frac{1}{16.4} \times 100 \quad (12)$$

where:

V_s = volume of sand determined in 17.5.1, mL, and

V_u = length \times width \times depth recorded in 17.4.1, in.^3

17.5.3 Report the results of Eq 12 in 17.5.2 for each specimen to the nearest 1 %, as the unit's percentage of void area.

18. Measurement of Void Area In Deep Frogged Units

NOTE 13—The area measured corresponds to a section located $\frac{3}{8}$ in. (9.5 mm) distant from the voided bed of the units.

18.1 Apparatus:

18.1.1 *Steel Rule or Gage or Calipers* (inside and outside)— as described in 13.1.

18.1.2 *Steel Straightedge*.

18.1.3 *Marking Pen or Scribe*.

18.1.4 *Brush*, a soft-bristle brush.

18.2 *Test Specimens*—Use a sample of 10 units selected as described for the determination of size. (It is permissible to use the samples taken for the determination of size.)

18.3 *Preparation of Sample*—Test the specimens as received except remove any adhering dirt by brushing.

18.4 Procedure:

18.4.1 Measure the length along both faces and the width along both ends at a distance of $\frac{3}{8}$ in. (9.5 mm) down from the bed containing the deep frogs. Record the measurements to the nearest $\frac{1}{32}$ in. (1 mm). Record the average of the two length measurements to the nearest $\frac{1}{32}$ in. (1 mm) as the length of the

unit and the average of the two width measurements to the nearest 1/32 in. (1 mm) as the width of the unit.

18.4.2 With the steel straightedge parallel to the length of the unit and centered over the deep frog or frogs, inscribe a mark on both faces of the frog 3/8 in. (9.5 mm) below the underside of the steel straightedge (mark 1 on Fig. 4). With the steel straightedge parallel to the width of the unit and centered over the deep frog, inscribe a mark on both faces of each frog 3/8 in. (9.5 mm) below the underside of the steel straightedge (mark 2 on Fig. 4).

18.4.3 Measure and record to the nearest 1/32 in. (1 mm) the distance between the inscribed marks on a line parallel to the length of the unit for each frog, and measure and record to the nearest 1/32 in. (1 mm) the distance between the inscribed marks on a line parallel to the width of the unit for each frog.

18.5 Calculations and Report:

18.5.1 Using the recorded length and width measurements calculate the gross area of the unit (A_u) in the plane of the unit 3/8 in. (9.5 mm) down from the frogged bed.

18.5.2 Using the distance between the inscribed marks calculate the inside area of each deep frog (A_f) in the plane of the unit 3/8 in. (9.5 mm) down from the frogged bed (see Fig. 4).

18.5.3 Determine the percentage of void as follows:

$$\% \text{ Void area} = \frac{\sum A_f \times 100}{A_u} \quad (13)$$

where:

$\sum A_f$ = sum of the inside area of the deep frogs, and
 A_u = gross area of unit.

18.5.4 Report the results of the equation in 18.5.3 for each specimen to the nearest 1 %, as the unit's percentage of void area.

19. Measurement of Out of Square

19.1 Apparatus:

19.1.1 Steel Rule or Calipers, as described in 13.1.

19.1.2 Steel Carpenter's Square.

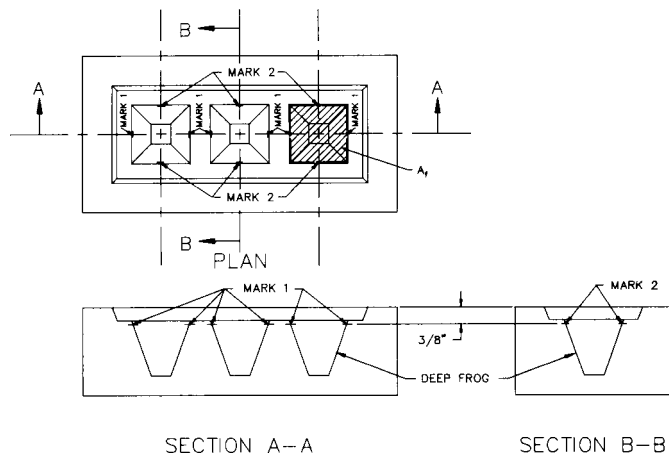


FIG. 4 Deep Frogged Units

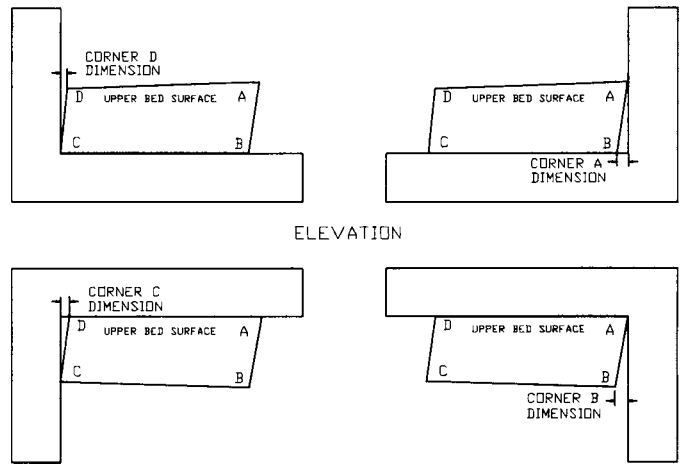


FIG. 5 Out-of-Square Measurements

19.2 Test Specimens—Use a sample of ten units selected as described for the measurement of size (see 13.2). (Samples taken for the measurement of size may be used in their as received state.)

19.3 Procedure:

19.3.1 Place one leg of a carpenter's square adjacent to the length of the unit when laid as a stretcher. Align the leg of the square parallel to the length of the unit by having the corners of the face of the unit in contact with the leg of the square. Locate the square parallel to and at or within 1/4 in. (6.4 mm) of the face to be exposed. See Fig. 6.

19.3.2 Measure the deviation due to the departure from the 90° angle at each corner of the exposed face of the unit. Record the measurement to the nearest 1/32 in. (1.0 mm) for each corner. See Fig. 5.

19.4 Report—Report the recorded measurements for each specimen tested to the nearest 1/32 in. (1.0 mm) as the unit's deviation from square.

20. Measurement of Shell and Web Thickness

20.1 Apparatus—A caliper rule graduated in not more than 1/64 in. (0.4 mm) divisions and having parallel jaws not less than 1/2 in. (12.7 mm) in length.

20.2 Test Specimens—Use a sample of five units as described for the measurement of size (see 13.2). (Samples taken for the measurement of size may be used in their as received state.)

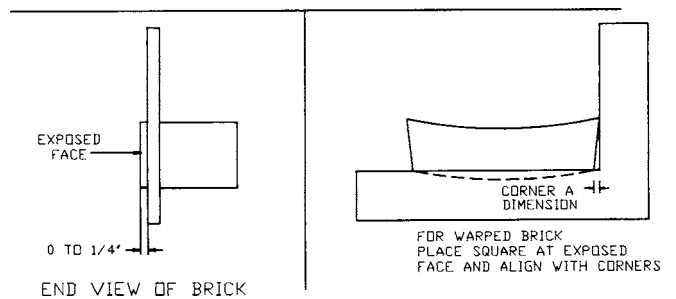


FIG. 6 Location of Carpenter's Square

20.3 *Preparation of Samples*—Remove any shards or other projections interfering with measurement of the minimum parallel distance of two surfaces.

20.4 *Procedure*—For each unit, measure the shell thicknesses and, when required, the web thicknesses at the thinnest point of each element ½ in. (12.7 mm) into the unit from either direction and record to the nearest division of the caliper.

NOTE 14—Current ASTM specifications for solid masonry units from clay or shale do not include minimum web thickness requirements.

21. Breaking Load

21.1 *Test Specimens*—The test specimens shall consist of whole full-size units that have been dried and cooled (see 5.1). Five such specimens shall be tested.

21.2 *Procedure:*

21.2.1 Unless specified and reported otherwise, support the test specimen flatwise (that is, apply the load in the direction of the height of the unit). The load shall be placed at the midspan, within ¼ in. (2 mm) of the center. If the specimens have frogs or depressions, place the specimen so that the frogs or depressions are on the underside of the specimen. The supports for the specimen shall be solid steel rods 1 ± 3/8 in. (25.4 ± 10 mm) in diameter placed ½ ± 1/16 in. (12.7 ± 2 mm) from each end. The length of each support shall be at least equal to the width of the specimen. See Fig. 7.

21.2.2 Apply the load to the upper surface of the specimen through a steel bearing plate ¼ in. (6.4 mm) in thickness and 1½ in. (38.1 mm) in width and of a length at least equal to the width of the specimen.

21.2.3 *Speed of Testing*—The rate of loading shall not exceed 2000 lbf (8896 N)/min. This requirement shall be considered as being met when the speed of the moving head of

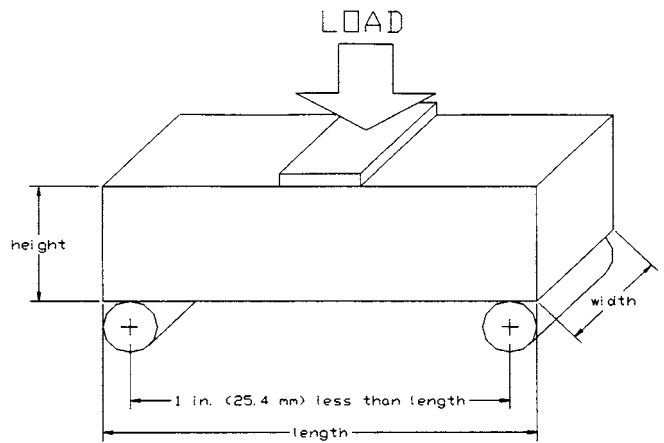


FIG. 7 Breaking Load Configuration

the testing machine immediately prior to application of the load is not more than 0.05 in. (1.27 mm)/min.

21.3 *Report:*

21.3.1 Record the unit dimensions and span length.

21.3.2 Record the transverse breaking load, *P*, of each unit to the nearest lb (N).

21.3.3 Calculate and record the breaking load per width of unit as $p = P/w$ for each unit, lb/in. (N/mm). Report the average of the breaking loads per width of all the specimens tested as the breaking load of the lot.

22. Keywords

22.1 absorption; compressive strength; efflorescence; freezing and thawing; initial rate of absorption; length change; modulus of rupture; out-of-square; sampling; size; void area; warpage

SUMMARY OF CHANGES

Committee C15 has identified the location of selected changes to this standard since the last issue (C67 – 13) that may impact the use of this standard. (December 1, 2013)

(1) Revised specimen preparation for test methods to clarify drying and cooling procedures.

Committee C15 has identified the location of selected changes to this standard since the last issue (C67 – 12) that may impact the use of this standard. (July 1, 2013)

(1) Wording in many sections was revised to conform with ASTM’s Form and Style Manual.

Committee C15 has identified the location of selected changes to this standard since the last issue (C67 – 11) that may impact the use of this standard. (June 1, 2012)

(1) New Fig. 2 on Warpage Measurement was added.

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Appendix F: MSDS

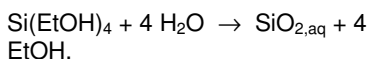
Technischer Leitfaden

KSE-Modul-System

Zur strukturellen Festigung und zur Herstellung KSE-basierter Hinterfüllmassen, Anbösch- und Kittmörtel sowie Schlämmen und Lasuren.

1-1 Steinfestiger auf Kieselsäureester-(KSE-) Basis

Neben der Minderung der Verwitterungsgeschwindigkeit durch **Quellminderung** und/ oder durch **wasserabweisende Einstellung** des Untergrundes ist im Rahmen einer zielgerichteten Natursteinkonservierung bei den meisten zu behandelnden Natursteinoberflächen eine **Konsolidierung des Gefüges mit einem Steinfestiger** notwendig. Hierfür werden seit Jahrzehnten Produkte auf der Basis von Kieselsäureester (KSE; auch: Kieselsäureethylester) eingesetzt. Die Wirkung dieser Steinfestiger beruht auf der Reaktion des Kieselsäureesters ($\text{Si}(\text{EtOH})_4$) mit Wasser (H_2O) zu festigendem Kieselgel ($\text{SiO}_{2,\text{aq}}$) nach der Formel



Als Nebenprodukt der Reaktion wird Alkohol (EtOH, „Ethanol“) freigesetzt.

1-2 „Klassische“ Steinfestiger

Der Rohstoff Kieselsäureester fällt bei seiner Herstellung in unterschiedlichen Molekülgrößen an. Daher enthalten alle auf dem Markt erhältlichen Steinfestiger sowohl monomere als auch oligomere Moleküle. Durch unterschiedliche Mischungsverhältnisse von kleinen und großen Molekülen im Fertigprodukt lassen sich die Eigenschaften des

Produktes, insbesondere die „Gel-Abscheidungsrate“ (Menge des im Porengefüge abgeschiedenen Kieselgels) deutlich variieren.

Weitere Variationsmöglichkeiten bestehen in Bezug auf

- die Reaktionsgeschwindigkeit (Art und Menge des Katalysators) und
- den (nicht zwingend notwendigen) Zusatz von Lösemittel(n).

Durch gezielte Kombination und Variation dieser veränderbaren Parameter ist ein hohes Maß an Anpassungsmöglichkeiten an den zu konsolidierenden Untergrund gegeben:

Remmers Produkt	Eigenschaften
KSE 100	<ul style="list-style-type: none"> • Gel-Abscheidungsrate: ca. 10 % • enthält zusätzliches Lösemittel
KSE 300	<ul style="list-style-type: none"> • Gel-Abscheidungsrate: ca. 30 % • frei von zusätzlichen Lösemitteln
KSE 510	<ul style="list-style-type: none"> • Gel-Abscheidungsrate: ca. 42 % • frei von zusätzlichen Lösemitteln
KSE OH	<ul style="list-style-type: none"> • Gel-Abscheidungsrate: ca. 30 % • enthält zusätzliches Lösemittel
KSE H	<ul style="list-style-type: none"> • Gel-Abscheidungsrate: ca. 30 % • mit hydrophober Wirkung • frei von zusätzlichen Lösemitteln

Remmers Produkt	Eigenschaften
KSE 300 HV	<ul style="list-style-type: none"> • Gel-Abscheidungsrate: ca. 30 % • frei von zusätzlichen Lösemitteln • mit Haftvermittlern für calcitische Untergründe

Die Endprodukte aller „klassischen“ Steinfestiger auf KSE-Basis weisen eine gemeinsame charakteristische Eigenschaft auf: Das sich im Porenraum ausbildende Kieselgel, aufgebaut aus einem ungeordneten, waserhaltigen SiO_2 -Gerüst, besitzt einen deutlich spröden Charakter. Aus diesem spröden Charakter des Kieselgels resultiert eine mittlere Größe der Kieselgel-Platten von ca. 10 μm .

Somit stoßen die „klassischen“ Steinfestiger auf Kieselsäureester-Basis immer dann an ihre Anwendungsgrenzen, wenn das zu festigende Gefüge Hohlradien größer als 10 μm aufweist. Entsprechende Hohlräume können beispielsweise

- aus der Porenradienverteilung des Natursteins (z.B. Tuff) oder
- aus der Ausbildung von Mikrorisszonen (z.B. durch Verwitterung, Trachyt, quellfähige Natursteintypen)

resultieren.

1-3 Elastifizierte Steinfestiger

Durch den Einbau sogenannter „Weichsegmente“ konnten elastifizierte Steinfestiger auf KSE-Basis entwickelt werden, die eine deutlich stärkere Tendenz zur Filmbildung als die klassischen Produkte zeigen:

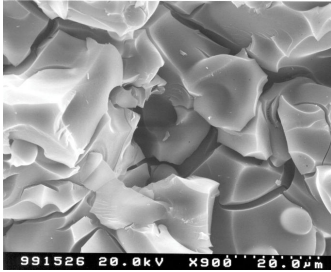


Abb. 1a: Glasfritte getränkt mit Remmers KSE OH; Größe der Gel-Platten: ca. 10 µm.

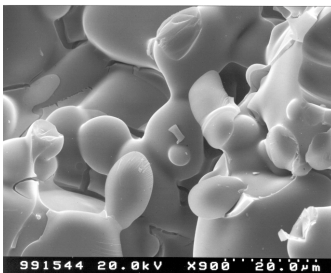


Abb. 1b: Glasfritte getränkt mit Remmers KSE 300 E; deutlich überbrückende Gelstrukturen im Porenraum.

Elastifizierte Steinfestiger eignen sich daher nicht nur zur „strukturellen Festigung“, sondern können auch als Bindemittel für Hinterfüllmassen, Anbösch- und Kittmörteln, Schlämmen und Lasuren eingesetzt werden. Modellhaft ist im Folgenden der Einsatz elastifizierter Steinfestiger / des KSE-Modul-Systems auf komplex verwitterten Untergründen gezeigt (Abb. 2):

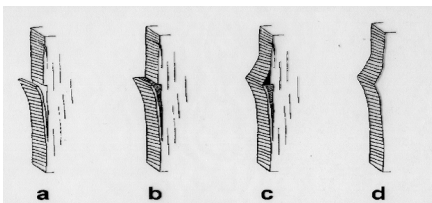


Abb. 2: Einsatz elastifizierter Steinfestiger / des KSE-Modul-Systems auf Untergründen mit komplex verwitterter Oberfläche.

- a Ausgangssituation: Verwitterte Stein- oberfläche mit intensiver Schalen- / Schuppenbildung und Mikrorissen.
- b Einbringung einer KSE-gebundenen Hinterfüllmasse zur Herstellung eines kraftschlüssigen Verbundes von der Schale zum Untergrund.

- c Antrag eines KSE-gebundenen Kitt- bzw. Anböschmörtels zum strukturellen Angleich der Steinoberfläche.
- d Strukturelle Festigung mit einem elastifizierten Steinfestiger zur Wiederherstellung der ursprünglichen, homogenen Festigkeit. Optional: Auftrag einer KSE-gebundenen Schlämme oder Lasur.

Grundsätzlich können die unter b bis d beschriebenen Arbeitsschritte jeweils mit anderen, unterschiedlichen Bindemitteln abgearbeitet werden. Allerdings birgt das Arbeiten mit nur einem Bindemittel (elastifizierter Steinfestiger) eine Vielzahl von Vorteilen.

2-1 Strukturelle Festigung

Die Elastifizierung des Kieselsäureesters bewirkt in der Regel eine - vergleichsweise - bessere Einbindung morbider Gefügebestandteile in das Gel-Netzwerk. Zudem lassen sich mit dem elastifizierten Gel höhere Rissweiten überbrücken als mit herkömmlichen Steinfestigern:

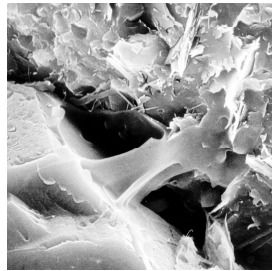


Abb. 3: Kieselgel-Brücke zwischen zwei Gefüge-Bestandteilen

Der Einsatz elastifizierter Steinfestiger ist nicht auf den Bereich Naturstein (z.B. quellfähige Varietäten wie Schilfsandstein oder bestimmte Vulkanite wie Tuff begrenzt, sondern kann auf alle mineralischen Untergründe z.B. historische Fugen und Putze, abgewitterte Ziegel übertragen werden:

Remmers Produkt	Eigenschaften
KSE 300 E	<ul style="list-style-type: none"> • Gel-Abscheidungsrate: ca. 30 % • elastifiziert
KSE 500 E	<ul style="list-style-type: none"> • Gel-Abscheidungsrate: ca. 50 % • elastifiziert

Vor allem bei hohem Schädigungsmaß unterscheiden sich die elastifizierten von den „klassischen“ Steinfestigern durch

- einen moderaten E-Modul-Anstieg (günstiges Span-

- nungs-Dehnungs-Verhalten) bei einer gleichzeitig
- hinreichend große Konsolidierung des mineralischen Gefüges.

2-2 Strukturelle Festigung: Verarbeitung

Nähere Informationen zur Vorgehensweise bei strukturellen Festigungen können den aktuellen Technischen Merkblättern der jeweiligen Steinfestiger entnommen werden.

3-1 KSE-Modul-System: Hinterfüllmassen

Verwitterte Natursteinoberflächen weisen häufig eine intensive Schalen- und/oder Schuppenbildung auf. Die damit verbundenen (Mikro-) Risse sind dabei oftmals so dimensioniert, dass sie mit nicht-gefüllten Steinfestigern nicht geschlossen werden können. Daher sind in einem der elastifizierten Steinfestiger bereits rein mineralische Schweb-/Füllstoffe eingearbeitet.

Remmers Produkt	Eigenschaften
KSE 500 STE	<ul style="list-style-type: none"> • Gel-Abscheidungsrate: ca. 50 % • enthält Schweb-/Füllstoffe • elastifiziert

Die eindispersierten Schwebstoffe lagern sich als Bodensatz ab. Dieser Vorgang ist reversibel. Daher muss Remmers KSE 500 STE vor Gebrauch jeweils gründlich aufgeschüttelt bzw. aufgerührt werden.

Remmers KSE 500 STE kann entweder direkt, ohne weiteren Zusatz feiner Zusatzstoffe appliziert werden (z.B. zum kraftschlüssigen Überbrücken von Mikrorissen bis ca. 300 µm) oder aber als Bindemittel zur Herstellung von

- Hinterfüllmassen,
- Anbösch- und/ oder Kittmörteln (s. dort),
- Schlämmen (s. dort) und
- Lasuren (s. dort) eingesetzt werden.

Hinterfüllmassen dienen der Wiederherstellung des kraftschlüssigen Verbundes von Schalen und Krusten zum Untergrund unter Berücksichtigung bauphysikalischer und physiko-mechanischer Eigenschaften.

Folgende Füllstoffe wurden speziell auf die Belange des KSE-Modul-Systems abgestimmt:

Remmers Produkt	Eigenschaft
KSE-Füllstoff A	Mineralpulver
KSE-Füllstoff B	Quarzpulver

Versuche zeigen, dass bei gleicher Ausgangszusammensetzung ein Zusatz von Vollglaskugeln (< 50 µm) die Fließfähigkeit der Masse nur unwesentlich verschlechtert, die Schrumpfrisssneigung jedoch deutlich reduziert.

3-2 Hinterfüllmassen: Systemlösung

In Bezug auf die Parameter Fließfähigkeit / Eindringverhalten, Schrumpfrisssneigung und „Bindungsfähigkeit“ haben sich folgende Richtrezepturen als günstig erwiesen:

A)

Bindemittel	Anteil
Remmers KSE 500 STE	37,5 ml
Remmers KSE 300 E	62,5 ml
Summe:	100,0 ml

Die Bindemittel-Kombination (Gel-Abscheidungsrate: ca. 37,5 %) wird vorgelegt.

B)

Alternativ können auch ausschließlich 100 ml KSE 500 STE als Bindemittel verwendet werden. Das Bindemittel (Gel-Abscheidungsrate: ca. 50 %) wird vorgelegt.

Anschließend werden auf jeweils 100 ml Bindemittel (bzw. Bindemittel-Kombination) folgende Füll- und Zusatzstoffe unter Rühren zugegeben:

Füll-/Zusatzstoffe	Anteil
KSE-Füllstoff A	64,0 g
KSE-Füllstoff B	40,0 g
Mikro-Vollglaskugel (< 50 µm) Fa. Kremer-Pigmente	26,0 g
Erdfarben-Trockenpigmente (nach Bedarf) Fa. Kremer-Pigmente	bis zu 5,0 g

Die Mischung wird homogenisiert (z.B. mit einem Stabmixer), bis die Masse keine Klumpenbildung mehr zeigt (ca. 3 – 5 Minuten Homogenisierungsdauer).

Je nach zugegebener Pigmentmenge liegt die Auslaufviskosität (DIN-4-Becher) der Hinterfüllmasse zwischen

- 31 s (ohne Pigmentzugabe) und
- 37 s (5,0 g Pigment).

3-3 Hinterfüllmassen: Verarbeitung

Zum Hinterfüllen von Schalen werden diese zunächst an ihren Rändern abgedichtet. Bewährt hat sich hier das Arbeiten mit Heißkleber, der sich ohne Rückstände wieder entfernen lässt. Anschließend wird soviel Hinterfüllmasse (in der Regel mit Hilfe einer Spritze) hinter die Schale eingebracht, bis der Hohlraum restlos gefüllt ist.

Bei saugfähigen Untergründen ist die zu hinterfüllende Schale mit einem geeigneten, wasserfreien Lösemittel (z. B. Verdünnung V 101, Remmers V KSE) oder dem Remmers KSE 100 vorzunässen.

Im Bedarfsfall können die kraftschlüssig angebondenen Bereiche nach Aushärtung des Bindemittels KSE (je nach Menge und Dicke der eingebrachten Masse 4 - 8 Wochen) nachgefestigt werden. Der verwendete Remmers Steinfestiger muss dabei den Materialeigenschaften des Untergrundes angepasst sein und ist gemäß gültigem Technischen Merkblatt zu verarbeiten.

Die Hinterfüllmasse muss während der Verarbeitung regelmäßig aufgerührt werden, um dem Absetzen der Füll-/Zuschlagstoffe entgegenzuwirken.

4-1 KSE-Modul-System: Anbösch-/Kittmörtel

Anböschmörtel dienen der Oberflächenanbindung auskragender Schalen (s. Abb. 2) etc. zur Reduzierung der Angriffsfläche für hinterwandernde Feuchtigkeit (z.B. durch Rissverschluss). Sie lassen sich farblich und strukturell an den Untergrund anpassen.

Anbösch- und/oder Kittmörtel lassen sich durch Kombination der oben genannten Füllstoffe mit geeigneten Sanden herstellen. Eine gesteinsangepasste Abtönung lässt sich

- durch Pigmentzusatz aber auch

- durch den Einsatz entsprechend gefärbter Naturstein-Brechsande erzielen.

Ursprünglich zur Kittung bzw. Ausbesserung kleinster Fehlstellen entwickelt, können mittlerweile - aufbauend auf den zwischenzeitlich gewonnenen Erfahrungen - auch größere Flächen bearbeitet werden (s. u.).

Wesentlich für die physiko-mechanischen und hygrischen Eigenschaften KSE-gebundener Mörtel ist eine optimal eingestellte Sieblinie des Zuschlags, wobei je nach Erfordernis sowohl

- Fullerverteilung wie auch
- Ausfallkörnungen

geeignet sein können. Als vorteilhaft erweist sich häufig der Einsatz von Siebfraktionen gebrochener Natursteinsande.

4-2 Anbösch-/Kittmörtel: Systemlösung

Zur Schließung auskragender Originaloberflächen kann der KSE-gebundene Anböschmörtel prinzipiell sofort nach dem Hinterfüllen der Schale angetragen werden. Bei größeren Rissen sollte jedoch die Hinterfüllmasse soweit durchreagiert sein, dass die Bestandteile des Mörtels nicht mehr in diese "hineinsacken". In diesem Fall ist der Untergrund vor dem Antragen des Mörtels mit einem geeigneten, wasserfreien Lösemittel (z. B. V 101, Remmers V KSE) oder dem Remmers KSE 100 vorzunässen.

Umfangreiche Labor- und Objektversuche haben gezeigt, dass bei größeren Schichtdicken mehrlagig mittels angepasster

- Kernmörtel (für den Unterbau) und
- Deckmörtel (ausschließlich für die obere Lage)

zu arbeiten ist.

In Bezug auf den – vergleichsweise groben - Kernmörtel ist zwischen

- **eingebundenen** Fehl- und Ausbruchstellen und
- **freistehenden** Fehl- und Ausbruchstellen, die profiliert werden müssen, zu unterscheiden.

Beim Auftrag mehrerer Lagen ist zwischen den einzelnen Anstragungsschritten eine Wartezeit von wenigstens 24 Stunden einzuhalten. Grundsätzlich ist der Untergrund vor jedem Arbeitsschritt wasserfrei (z. B. mittels Verdünnung V 101, Remmers V KSE) vorzunässen.

Die Dicke der Deckschicht darf - je nach verwendeter Rezeptur - nur zwischen 0,5 cm und 1 cm betragen. Durch Abtupfen von Bindemittelüberschüssen kann eine verbesserte Optik der Deckschicht erzielt werden.

Die Struktur des Anböschrötmels kann ca. 1 - 2 Stunden nach dem Auftragen durch Bearbeitung (z. B. Spatel, Kamm) an den Untergrund angepasst werden.

Bsp.: Deckmörtel, Schlesischer Sandstein

Zu je 100 ml Remmers KSE 500 STE werden folgende Füllstoffe gegeben

Füllstoffe	Anteil
KSE-Füllstoff A	70,0 g
KSE-Füllstoff B	40,0 g

Die Mischung wird (z.B. mittels eines Stabmixers) homogenisiert, bis die Masse keine Klumpen mehr zeigt (ca. 3 – 5 Minuten Homogenisierungsdauer).

Die hergestellte Masse wird (z. B. in einem Anrührbecher) vorgelegt. Anschließend wird

Füllstoffe	Anteil
Remmers Quarzsand F 36	334,0 g

gründlich (z. B. mit einem Spatel) eingerührt.

Die Masse nimmt dabei letztlich erdfeuchte, bröckelige Konsistenz an.

Die angegebenen Verarbeitungsparemeter sind genau einzuhalten. Ein Überschuss an Bindemittel aus Gründen der „gefälligen Verarbeitung“ ist zu vermeiden.

Die Anböschr- und Kittmörtel müssen während der Verarbeitung regelmäßig aufgerührt werden, um dem Absetzen der Zuschlagstoffe entgegenzuwirken.

Die Eignung einer speziellen Rezeptur auf einem bestimmten Untergrund ist jeweils vorab an einer ge-

eigneten, genügend großen Probe- fläche zu überprüfen.

Im Bedarfsfall können die kraftschlüssig angebondenen Bereiche nach Aushärtung des Bindemittels Remmers KSE 500 STE (je nach Menge und Dicke der eingebrachten Masse 4 - 8 Wochen) nachgefestigt werden. Der verwendete Remmers Steinfestiger muss dabei den Materialeigenschaften des Untergrundes angepasst sein und ist jeweils gemäß gültigem Technischen Merkblatt zu verarbeiten.

Naturstein-Brechsande

Mittels geeigneter fraktionierter Naturstein-Brechsande können die Anböschr-/Kittmörtel besonders gut an den Untergrund angepasst werden. Rezepturen können im Bedarfsfall erarbeitet werden. Anfragen bitte an das Remmers-Werkslabor.

5-1 KSE-Modul-System: Lasuren und Schlämmen

Verwitterte Untergründe zeigen häufig ein sehr heterogenes Oberflächenbild. Gleichzeitig stellen rückgewitterte, aufgeraute Oberflächen eine besonders große Angriffsfläche für einen weitergehenden Verwitterungsprozess (z. B. durch Feuchtigkeit- und Schadstoffeintrag) dar.

So lagern sich auf rauen Oberflächen bevorzugt Schmutzpartikel der Luft und Mikroorganismen an, wird Regenwasser von diesen entweder nur verzögert abgeleitet oder verhartet durch die entstandene Oberflächengeometrie als Wasserfilm auf der Oberfläche.

Als besonders geeignet zur Verbesserung der Wasserableitung und zur Ausbildung einer neuen Verschleißschicht haben sich KSE-gebundene Lasuren und (körperhafte) Schlämmen erwiesen.

Durch den Auftrag angepasster KSE-gebundener Beschichtungen kann die mineralische Oberfläche

- strukturell geglättet (und damit verkleinert),
- mechanisch konsolidiert und
- optisch beruhigt / verbessert werden.

5-2 Lasuren: Systemlösung

Je nach Art und Beschaffenheit des Untergrundes wurden mit folgenden Systemen gute Ergebnisse erzielt.

Bindemittel

Bewährt haben sich Gel-Abscheidungsrate zwischen 33 % und 50 %. Dementsprechend kann

- entweder ausschließlich Remmers KSE 500 STE
- oder eine Mischung aus Remmers KSE 500 STE und Remmers KSE 300 E als Bindemittel verwendet werden:

Rezeptur-Anteil	Remmers Produkt	Gel-Abscheidungsrate
mind. 16,6%	KSE 500 STE	50 %
max. 83,4 %	KSE 300 E	30 %
100,0 %	Mischung	33 - 50 %

Füllstoff

Je nach gewünschter Konsistenz und gewünschtem Lasurgrad können auf je 100 ml Bindemittel bis zu 150 g Füllstoff gegeben werden. Das Verhältnis

Remmers KSE Füllstoff A : Remmers KSE Füllstoff B sollte dabei 1,6 : 1 betragen.

Pigment

Zur farblichen Anpassung können auf 100 ml Bindemittel bis zu 6,5 g Pigment gegeben werden.

Bsp.: roter Buntsandstein

Zu je 100 ml Bindemittel bestehend aus

Bindemittel	Anteil
KSE 500 STE	16,7 ml
KSE 300 E	83,3 ml
Summe:	100,0 ml

werden folgende Füllstoffe und Zusatzmittel gegeben:

Füllstoffe / Zusatzmittel	Anteil
KSE-Füllstoff A	30,0 g
KSE-Füllstoff B	18,0 g
Erdfarben-Trockenpigmente Umbra (Nr.: 40720)	2,2 g
Mortuum (Nr.: 48220)	2,6 g
Fa. Kremer-Pigmente	

Die Mischung wird (z.B. mittels eines Stabmixers) homogenisiert, bis die Masse keine Klumpen mehr zeigt (ca. 3 – 5 Minuten Homogenisierungsdauer).

Die Eignung einer speziellen Rezeptur auf einem bestimmten Untergrund ist jeweils vorab an einer geeigneten, genügend großen Probe- fläche zu überprüfen.

Naturstein-Brechsande

Mittels geeigneter, fraktionierter Naturstein-Brechsande können die Lasuren besonders gut an den Untergrund angepasst werden. Rezepturen können im Bedarfsfall erarbeitet werden. Anfragen bitte an das Remmers-Werkslabor.

5-3 Schlämmen: Systemlösung

„Körperhafte“ Schlämmen enthalten - anders als die entsprechenden Lasuren - neben Füllstoffen auch gröbere Sandfraktionen.

Bsp.: Schilfsandstein

Zu je 100 ml Bindemittel KSE 500 STE werden folgende Zuschläge, Füllstoffe und Zusatzmittel gegeben:

Zuschlag, Füll-/ Zusatzstoffe	Anteil
Remmers Quarzsand F 36	70,0 g
Remmers KSE-Füllstoff A	50,0 g
Remmers KSE-Füllstoff B	50,0 g
Schiefermehl Fa. Kremer-Pigmente (Nr.: 40900)	4,4 g
Erdfarben-Trockenpigmente Ocker (Nr.: 40050) Eisenoxid (Nr.:4844) Fa. Kremer-Pigmente	5,0 g 1,4 g

Die Mischung wird (z.B. mittels eines Stabmixers) homogenisiert, bis die Masse keine Klumpen mehr zeigt (ca. 3 – 5 Minuten Homogenisierungsdauer).

Die Eignung einer speziellen Rezeptur auf einem bestimmten Untergrund ist jeweils vorab an einer geeigneten, genügend großen Probe- fläche zu überprüfen.

Naturstein-Brechsande

Mittels geeigneter, fraktionierter Naturstein-Brechsande können die

Schlämmen besonders gut an den Untergrund angepasst werden. Rezepturen können im Bedarfsfall erarbeitet werden. Anfragen bitte an das Remmers-Werkslabor.

5-4 Lasuren und Schlämmen: Verarbeitung

Der Untergrund muss trocken, staubfrei, tragfähig, frei von losen Teilen, Bewuchs, Staub-, Schal- und Fettrückständen sein. Der Untergrund darf nicht aufgeheizt sein (evtl. Sonnensegel setzen).

Vorbereitung

Der zu behandelnde Untergrund ist mit einem, wasserfreien Lösemittel (z. B. Verdünnung V 101, Remmers V KSE) oder einem geeigneten Remmers Steinfestiger vorzunässen.

Ist im Rahmen einer Gesamtmaßnahme eine strukturelle Festigung des Untergrundes mit einem Remmers Steinfestiger vorzunehmen, so bietet es sich in der Regel an, die Lasur bzw. die Schlämme direkt nach Abschluss der Festigungsmaßnahme nass in nass aufzutragen.

Verarbeitung

Die Flüssigkomponente wird in ein sauberes Gefäß vorgelegt und die Pulverkomponenten zugegeben. Mit einem Mischgerät werden die Komponenten intensiv zu einer homogenen Lasur bzw. Schlämme angemischt.

Die Lasur bzw. die Schlämme ist intensiv - z. B. durch Einmassieren - mit dem Untergrund in Kontakt zu bringen. Die optimalen Verarbeitungstemperaturen liegen zwischen 10 °C und 20 °C. Der behandelte Untergrund ist wenigstens 2 Wochen vor und nach dem Lasur-/Schlämmauftrag vor Beregnung zu schützen.

Die Lasuren und Schlämmen müssen während der Verarbeitung regelmäßig aufgerührt werden, um dem Absetzen der Zuschlagstoffe entgegenzuwirken.

Nachbehandlung

Je nach Beschaffenheit des Untergrundes kann eine partielle Nachfestigung der Schlämme mit einem geeigneten Remmers Steinfestiger notwendig sein.

Nach dem Auftrag der Lasur/ Schlämme ist vor weiteren Arbeits-

schritten eine Reaktionszeit von ca. 4 Wochen einzuplanen.

Der Verbrauch an Lasur bzw. an Schlämme richtet sich maßgeblich nach Art und Zustand des zu behandelnden Untergrundes sowie nach der zu lösenden Aufgabenstellung. Dementsprechend kann der Verbrauch zwischen 0,2 l/m² und 0,8 l/m² liegen. Er ist jeweils vorab an einer genügend großen, geeigneten Musterfläche zu ermitteln.

6 Allgemeine Hinweise

Untergrund

Die zur Restaurierung anstehenden Natursteinoberflächen weisen durch Verschmutzung / Patinierung unterschiedlichster Art oftmals ein vermindertes Saugvermögen auf.

Die zur Wiederherstellung des ursprünglichen Saugvermögens notwendige Reinigung der Flächen sollte möglichst schonend erfolgen, um die zu haltenden Bereiche nicht zu zerstören. Die gereinigte Fläche soll eine Woche lang vor Regen und zu starkem Aufheizen durch Sonneneinstrahlung geschützt werden.

Verarbeitungstemperaturen

Die optimalen Verarbeitungstemperaturen für das KSE-Modul-System liegen zwischen 10 °C und 20 °C (evtl. Sonnensegel setzen). Unter +5 °C muss die Verarbeitung eingestellt werden. Die behandelten Bereiche sind vor Regen und direkter Sonneneinstrahlung zu schützen.

Zusatz von Additiven

Von der Zugabe von Additiven - insbesondere von Additiven zur Beschleunigung der Reaktionszeit - wird abgeraten. Desgleichen wird von der sogenannten "Schnellhydrolyse" abgeraten, da sie eine unkontrollierte Einflussnahme auf die Gel-Bildereaktion und damit auf die resultierende Kieselgelstruktur darstellt.

Wasserabweisung

Eine gewünschte wasserabweisende Einstellung hinterfüllter und/oder angeböschter Bereiche sollte in Lasurtechnik zu erfolgen (Remmers Siliconharz-Farbsystem, z. B. Historic Lasur, Historic Schlämmlasur).

Angrenzende Flächen

Fassadenteile, die nicht mit den reaktiven Komponenten des KSE-Modul-Systems in Berührung kommen sollen, wie z.B. Fenster, la-

ckierte Flächen sowie Glas, müssen ebenso wie Pflanzen mit Baufolie (Polyethylenfolie) abgedeckt werden.

7 Arbeitsgeräte, Reinigung

Je nach Aufgabenstellung, z.B. Niederdruck-Spritzgeräte, Airlessgeräte, Spritzflasche, kleine (Holz-)Spachtel, Pinsel, Schwämme.

Reinigung im frischen Zustand mit wasserfreien Lösemitteln (z. B. Remmers V KSE), im ausreagierten Zustand ist nur noch mechanische Reinigung möglich.

8 Lieferform, Verbrauch, Lagerung

Nähere Informationen zu Lieferform, Verbrauch und Lagerung können den Technischen Merkblättern der einzelnen Produkte entnommen werden.

9 Sicherheit, Ökologie, Entsorgung

Nähere Informationen zur Sicherheit bei Transport, Lagerung und Umgang sowie zur Entsorgung und Ökologie können den aktuellen Sicherheitsdatenblättern der einzelnen Produkte entnommen werden.

Vorstehende Angaben wurden aus unserem Herstellerbereich nach dem neuesten Stand der Entwicklung und Anwendungstechnik zusammengestellt.

Da Anwendung und Verarbeitung außerhalb unseres Einflusses liegen, kann aus dem Inhalt des Merkblattes keine Haftung des Herstellers abgeleitet werden. Über den Inhalt des Merkblattes hinausgehende oder abweichende Angaben bedürfen der schriftlichen Bestätigung durch das Stammwerk.

Es gelten in jedem Fall unsere allgemeinen Geschäftsbedingungen. Mit Herausgabe dieses technischen Merkblattes verlieren vorangegangene ihre Gültigkeit.

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Conservare[®]

OH100 Consolidation Treatment

penetrating stone & masonry strengthener

Of all materials currently and historically employed in construction, masonry is one of the most durable. What has become apparent in recent years, however, is that masonry materials are not as enduring as once believed.

Placed in contemporary urban environments, these “timeless” materials decay at an alarming rate. Some deterioration may be attributed to the masonry’s natural weathering process. The majority of the deterioration, however, is the result of oversights in use and maintenance of the masonry, and of the impact that industrialization has had on our environment, i.e. “acid deposition.”

The intent of all conservation treatments is to restore the structural integrity to crumbling, decaying masonry and/or provide a means of controlling future decay. The failure of many conservation treatments lies in their inherent dissimilarity to the masonry for which they are proposed as a preservative. When selecting a conservation treatment, an important consideration is to identify those treatments with physical and chemical characteristics similar to the masonry itself.

Conservare[®] Consolidation Treatments are based on silicic ethyl esters. Their extremely small molecular structure enables them to penetrate deeply into deteriorated masonry surfaces, collecting at contact points between individual stone grains. An internal catalyst and atmospheric humidity then convert the liquid consolidant into a glass-like silicon dioxide (SiO₂) gel which binds the stone particles together. Exhibiting chemical

characteristics and thermal expansion/contraction characteristics which are virtually identical to that of natural stone, the newly deposited SiO₂ cementing matrix replaces the stone’s natural cement which has been lost due to weathering influences.

OVERVIEW

Conservare[®] OH100 is a ready-to-use consolidation treatment that stabilizes masonry by replacing the natural binding materials, lost due to weathering, with silicon dioxide. When properly applied, Conservare[®] OH100 penetrates deeply, does not form a dense surface crust, and retains the substrate’s natural vapor permeability.

In addition to the general consolidation of severely deteriorated masonries, Conservare[®] OH100 is an effective pretreatment for friable substrates that need to be strengthened before cleaning, patching or coating. Conservare[®] OH100 may be used on most types of natural stone, concrete, stucco, brick, terra-cotta, etc.

Conservare[®] OH100 is effective on unpolished marble, travertine and limestone that has been treated with Conservare[®] HCT (Hydroxylating Conversion Treatment).

SPECIFICATIONS

For all PROSOCO product specifications visit www.prosoco.com and click on “SpecBuilder” or “Solution Finder.”

ADVANTAGES

- One component – easy-to-use. Strengthens deteriorated stone.
- Low viscosity allows deep penetration. Will not form hardened surface crust.
- The new binder is mineral – similar to the original stone – no synthetic polymers.
- Rapid tack free drying – no dirt attraction.
- Forms no by-products harmful to the masonry.
- Treated surfaces “breathe” – does not trap moisture.
- New binder is acid resistant – resists acid rain.

TYPICAL TECHNICAL DATA

FORM	Colorless to slight yellow
SPECIFIC GRAVITY	0.997
pH	Not applicable
WT/GAL	8.30 lbs
ACTIVE CONTENT	100%
TOTAL SOLIDS	43% ASTM D 5095
VOC CONTENT	>400 g/L
FLASH POINT	104°F (40°C)
FREEZE POINT	<-22°F (<-30°C)
SHELF LIFE	1 year in tightly sealed, unopened container

Limitations

- Effective consolidation requires thorough laboratory and field pretesting. Contact PROSOCO for information on the recommended test programs.
- Limited shelf life – remains storage stable for approximately 12 months in sealed containers. Treated areas may bond to silicone and polyurethane molds (frequently used for casting replacement stone). Use a release agent to prevent molding compounds from adhering to the treated surface.
- Not suitable for architectural concrete block and some types of marble.
- Not suitable for use on polished marble, travertine, limestone or granite.
- Will not prevent water penetration through structural cracks, defects or open joints.
- Not recommended for below-grade application.
- May not be suitable for sale in states and districts with more restrictive AIM VOC regulations. Available in regulation-exempt small container sizes. Call Customer Care at 800-255-4255 for assistance.

REGULATORY COMPLIANCE

VOC Compliance

Conservare® OH100 Consolidation Treatment is compliant with the following national, state and district AIM VOC regulations

- US Environmental Protection Agency
- California Air Resources Board SCM Districts
- South Coast Air Quality Management District
- Maricopa County, AZ
- Northeast Ozone Transport Commission

Manufactured and marketed in compliance with USEPA AIM VOC regulations (40 CFR 59.403).
VOC Information

THE IMPORTANCE OF PRETESTING

Since building materials differ in their nature and degree of deterioration, each conservation project poses unique problems and requirements. To gain a full understanding of the ongoing deterioration and determine necessary stabilization/conservation measures, a number of laboratory and field tests are required.

Laboratory Testing

- Evaluates the physical and chemical characteristics of the substrate(s) to confirm whether consolidation is possible.
- Identifies the cause(s) of deterioration and surface preparation procedures necessary for conservation treatment.
- Determines the most appropriate conservation agent(s) and field application procedures.

For more information on the recommended testing program, read the Conservare® Stone Testing Brochure and contact your PROSOCO representative to arrange a job-site visit.

On-Site Testing

Following lab testing, a test area should be cleaned and allowed to dry. An application of Conservare® OH100 Consolidation Treatment is made following specific recommendations provided by the laboratory analysis. The job site test area should be as large as possible and representative of the condition of the entire project.

The test area is necessary to confirm application procedures under job site conditions and allow calculation of the masonry's consumption rate. The on-site tests also provide a visible sample of the effects of the treatment on actual job surfaces. Additional core samples can be taken from the test area and tested to verify depth of penetration and proper application procedures.

ALWAYS TEST

ALWAYS TEST a small area of each surface to confirm suitability and desired results before starting overall application. Test with the same equipment, recommended surface preparation and application procedures planned for general application.

PREPARATION

Following lab and on-site testing, clean the building with the appropriate Sure Klean® product. In most cases, surface contaminants such as carbon crust, salts, pigeon droppings, mildew and atmospheric stains must be completely removed to assure thorough penetration of Conservare® OH100.♦ In addition, surface sealers and repellents which may have been applied must be thoroughly removed. Contact Customer Care at (800) 255-4255 for additional cleaning recommendations.

♦In cases where even the most sympathetic cleaning program would remove an unacceptable level of surface detail, Conservare® OH100 Consolidation Treatment may be applied to the soiled surface to preconsolidate the stone. If such pre-consolidation is necessary, further evaluation will be required to ensure that no undesirable reactions take place between the consolidation treatment and the surface contaminants which may interfere with further conservation measures, i.e. subsequent cleaning, general consolidation, patching/repair, etc.

Protect people, vehicles, property, metal, glass, foliage, painted surfaces and all non masonry surfaces from contact with product, fumes or wind drift. Protect and/or divert pedestrian and auto traffic.

Ensure fresh air entry and cross ventilation during application and drying. Extinguish all flames, pilot lights and other potential sources of ignition during use and until all vapors are gone. When applying to exteriors of occupied buildings, make sure all windows, exterior intakes and air

conditioning vents are covered and air handling equipment is shut down during application and until all vapors have dissipated.

Surface and Air Temperatures

Surface and air temperatures should be between 50–90°F (10–32°C) during application. Relative humidity should be greater than 40%. Excessive surface heating can be prevented by shading with awnings.

Protect surface to be treated from direct sunlight for several hours prior to beginning application. When possible, initiate treatment when surfaces are shaded. Keep surface temperature relatively cool to prevent too rapid evaporation of Conserve[®] OH100 and to ensure proper penetration. Do not apply during rain, to wet surfaces or when there is a chance of rain. Protect from rain for two days following application.

Equipment

Apply by low-pressure spray, brush or dipping. Larger surfaces should be treated using low-pressure spray equipment, small areas with spray tanks. Mobile objects such as sculptures are best treated indoors by dipping or with the use of compresses. Contact Customer Care at 800-255-4255 or your local sales manager for more information.

Storage and Handling

Store in a cool, dry place away from potential ignition sources. Keep tightly closed when not dispensing. Published shelf life assumes upright storage of factory-sealed containers in a dry place. Maintain temperature of 45–100°F (7–38°C). Do not double stack pallets. Dispose of unused product and container in accordance with local, state and federal regulations.

APPLICATION

Before use, read "Preparation" and "Safety Information."

Dilution

Use in concentrate. Do not dilute or alter. Stir or mix well before use.

Coverage Rates

Coverage rates vary depending on the substrate and degree of deterioration. Laboratory and field testing are necessary to confirm desired results and application procedures.

Application Instructions

Ensure proper penetration and prevent crust formations by applying Conserve[®] OH100 in repeated applications referred to as "cycles." A cycle consists of three successive saturating applications at 5–15 minute intervals. Typical treatments involve two or three cycles (6–9 separate applications). Allow 20 to 60 minutes between cycles. Laboratory testing will determine the optimum delay between applications and between cycles. Additional material should be applied until excess material remains visible on the surface for 60 minutes following the last application. Once this degree of saturation is achieved over the entire surface, the first treatment is complete. Immediately flush excess surface materials using industrial grade MEK (methyl ethyl ketone) or mineral spirits. If a second treatment is necessary, allow two to three weeks curing time following first treatment.

NOTE: Laboratory testing will determine the absorption profile and conservation capacity of the substrate(s). From this information, the optimal delay between saturating coats, and dwell time between cycles will be prescribed. The work area should be limited to a size that can be treated within the prescribed time periods.

Proper timing of the application process will maximize penetration of the consolidation treatment. Deep penetration is critical to the long-term benefits of any consolidation treatment.

BEST PRACTICES

Since building materials differ in their nature and degree of deterioration, each conservation project poses unique problems and requirements. To gain a full understanding of the ongoing deterioration and determine necessary stabilization/conservation measures, a number of laboratory and field tests are required.

Protect surface to be treated from direct sunlight for several hours prior to beginning application. When possible, initiate treatment when surfaces are shaded. Keep surface temperature relatively cool to prevent too rapid evaporation and to ensure proper penetration.

Ensure proper penetration and prevent crust formations by applying Conserve[®] OH100 in repeated applications referred to as "cycles." A cycle consists of three successive saturating applications at 5–15 minute intervals.

Additional material should be applied until excess material remains visible on the surface for 60 minutes following the last application. Once this degree of saturation is achieved over the entire surface, the first treatment is complete.

Never go it alone. If you have problems or questions, contact your local PROSOCO distributor or field representative. Or call PROSOCO technical Customer Care, toll-free, at 800-255-4255.

Cleanup

Clean tools and equipment immediately with mineral spirits, denatured alcohol or an equivalent cleaning solvent. Remove over spray and spills as soon as possible.

Post-Treatment

Areas properly treated with Conservare® OH100 can receive stone repair materials, regrouting materials and PROSOCO's BMC® silicone emulsion paints after the consolidation procedures have been completed. After curing apply the appropriate Sure Klean® Weather Seal water repellent to ensure protection from further water damage.

SAFETY INFORMATION

Conservare® OH100 Consolidation Treatment is a solvent carried product and may cause symptoms typical with organic solvent exposures. This is a combustible material. Use appropriate ventilation, safety equipment and job site controls during application and handling. Read the full label for precautionary instructions before use.

First Aid

Ingestion: If swallowed, call a physician immediately. Do not induce vomiting except at the instruction of a physician. If vomiting occurs, keep head below waist to prevent entry of liquid into lungs.

Eye Contact: Rinse eyes thoroughly for 15 minutes. Get medical assistance.

Skin Contact: Rinse thoroughly. Get medical attention if irritation persists. Launder contaminated clothing before reuse.

Inhalation: Remove to fresh air. Give artificial respiration if not breathing. Get immediate medical attention.

24-Hour Emergency Information:
INFOTRAC at 800-535-5053

WARRANTY

The information and recommendations made are based on our own research and the research of others, and are believed to be accurate. However, no guarantee of their accuracy is made because we cannot cover every possible application of our products, nor anticipate every variation encountered in masonry surfaces, job conditions and methods used. The purchasers shall make their own tests to determine the suitability of such products for a particular purpose.

PROSOCO, Inc. warrants this product to be free from defects. **Where permitted by law, PROSOCO makes no other warranties with respect to this product, express or implied, including without limitation the implied warranties of merchantability or fitness for particular purpose.** The purchaser shall be responsible to make his own tests to determine the suitability of this product for his particular purpose. PROSOCO's liability shall be limited in all events to supplying sufficient product to re-treat the specific areas to which defective product has been applied. Acceptance and use of this product

absolves PROSOCO from any other liability, from whatever source, including liability for incidental, consequential or resultant damages whether due to breach of warranty, negligence or strict liability. This warranty may not be modified or extended by representatives of PROSOCO, its distributors or dealers.

CUSTOMER CARE

Factory personnel are available for product, environment and job-safety assistance with no obligation. Call 800-255-4255 and ask for Customer Care - technical support.

Factory-trained representatives are established in principal cities throughout the continental United States. Call Customer Care at 800-255-4255, or visit our web site at www.prosoco.com, to find the name of the Conservare® representative in your area.

MATERIAL SAFETY DATA SHEET

PROSOCO, Inc.



I PRODUCT IDENTIFICATION

MANUFACTURER'S NAME AND ADDRESS: PROSOCO, Inc.
3741 Greenway Circle
Lawrence, Kansas 66046

EMERGENCY TELEPHONE NUMBERS:
8:00 AM - 5:00 PM CST Monday-Friday: 785/865-4200
NON-BUSINESS HOURS (INFOTRAC): 800/535-5053

PRODUCT TRADE NAME: Conservare® OH100 Consolidation Treatment

II HAZARDOUS INGREDIENTS

CHEMICAL NAME	(COMMON NAME)	CAS NO.	NFPA CODE	ACGIH TLV/TWA	OSHA PEL/TWA
Di-n-butyltindilaurate	(-)	77-58-7	1,3,0,-	Not established	Not established
Organic Tin Compound	(-)	-	Unknown	0.1 mg/m ³	0.1 mg/m ³
Ethyl Alcohol	(Ethanol)	64-17-5	3,3,0,-	1000 ppm	1000 ppm
Tetra ethyl silicate	(Ethyl Silicate)	78-10-4	2,2,0,-	10 ppm	100 ppm

* Contains some or all of the listed ingredients.

III PHYSICAL DATA

	BOILING POINT (°F)	VAPOR PRESSURE (mm Hg)	VAPOR DENSITY (Air=1)	EVAPORATION RATE (1=Butyl Acetate)
Di-n-butyltindilaurate	N/A	N/A	N/A	N/A
Organic Tin Compound	>401	0.2 (320°F)	N/A	Very Slow
Ethyl Alcohol	180	33 (68°F)	3.0	1.9
Tetra ethyl silicate	302	1.0 (68°F)	Unknown	Unknown

	SPECIFIC GRAVITY	SOLUBILITY IN WATER	APPEARANCE AND ODOR
Conservare® OH100 Consolidation Treatment	0.997(@ 77°F)	Negligible	Clear liquid, alcohol odor

IV FIRE AND EXPLOSION HAZARD DATA

EMERGENCY OVERVIEW

Conservare® OH100 Consolidation Treatment is a clear liquid with a mild alcohol odor. It is a combustible liquid, remove all potential sources of ignition. Product may irritate skin upon contact and may cause lung damage if inhaled. Wear appropriate respiratory protection.

FLASH POINT (Method): 104°F (40°C) (closed cup)

FLAMMABLE LIMITS: Not determined.

EXTINGUISHING MEDIA: Carbon dioxide, dry chemical, alcohol-resistant foam, sand or water-mist. Do not use direct water stream. Do not use direct water stream. Avoid accumulation of water as product will float.

SPECIAL FIRE FIGHTING PROCEDURES: Do not enter confined fire space without proper protective equipment including a NIOSH/MSHA approved self-contained breathing apparatus. Cool fire exposed containers, surrounding equipment and structures with water.

UNUSUAL FIRE AND EXPLOSION HAZARDS: Vapors are heavier than air and may accumulate in low areas or areas inadequately ventilated. Vapors may also travel along the ground to be ignited at location distant from handling site; flashback of flame to handling site may occur. Never use welding or cutting torch on or near drum (even empty) because product (even just residue) can ignite explosively. As a result of hydrolysis, flammable vapors may accumulate in the container head space.

COMBUSTIBLE! Keep container tightly closed. Isolate from oxidizers, heat, and open flame. Closed containers may explode if exposed to extreme heat. Applying to hot surfaces requires special precautions.

V HEALTH HAZARD DATA

PRIMARY ROUTES OF EXPOSURE: Inhalation, skin, eyes.

CARCINOGEN INFORMATION: Not listed (OSHA, IARC, NTP).

MEDICAL CONDITIONS AGGRAVATED BY OVEREXPOSURE: This product is damaging to the liver and kidneys, and is also toxic to the lungs. Product also causes acute dermatitis and has a narcotic effect.

EFFECTS OF OVEREXPOSURE: Highly irritating to eyes. Moderately irritating to skin. High concentrations may produce anesthetic or narcotic effect. May cause kidney and liver damage and temporary disorder of sight.

EYE CONTACT: Liquid is highly irritating to the eyes. Vapors are also irritating. Possible moderate corneal injury and temporary disorder of sight.

SKIN CONTACT: Liquid is moderately irritating to the skin. Repeated, prolonged contact can result in defatting to the skin which may lead to dermatitis.

INHALATION: Breathing high vapor concentrations or prolonged breathing of lower concentrations can cause nose and throat irritation and may cause headache, dizziness and loss of consciousness.

INGESTION: Liquid ingestion may result in vomiting; aspiration of liquid into the lungs must be avoided as liquid contact with the lungs can result in chemical pneumonitis and pulmonary edema/hemorrhage.

EMERGENCY AND FIRST AID PROCEDURES:

EYE CONTACT: If in eyes, flush with large amounts of water for 15 minutes, holding eyelids apart to ensure flushing of the entire eye surface. Get medical attention immediately.

SKIN CONTACT: Remove material with a waterless skin cleaner, then wash with plenty of soap and water. Remove contaminated clothing and do not reuse until laundered. If persistent irritation occurs, get medical attention.

INHALATION: Remove victim to fresh air and provide oxygen if breathing is difficult. Give artificial respiration if not breathing. Get immediate medical attention. Designate the product.

INGESTION: DO NOT INDUCE VOMITING! Get immediate medical attention. Designate the product. If vomiting occurs spontaneously, keep victim's head below hips to prevent breathing vomitus into lungs.

VI REACTIVITY DATA

STABILITY: Stable at ambient temperatures and atmospheric pressure

CONDITIONS TO AVOID: None known.

INCOMPATIBILITY (MATERIALS TO AVOID): Oxidizing materials, acids, and alkalis, water

HAZARDOUS COMBUSTION OR DECOMPOSITION PRODUCTS: Carbon monoxide, carbon dioxide, silicon dioxide and traces of incompletely burned hydrocarbons. Ethyl alcohol from hydrolysis.

VII SPILL OR LEAK PROCEDURES

SPILL, LEAK AND WASTE DISPOSAL PROCEDURES: STEPS TO BE TAKEN IN CASE MATERIAL IS RELEASED OR SPILLED:

Eliminate potential sources of ignition. Wear appropriate respirator and other protective clothing. Shut off source of leak only if safe to do so. Dike and contain to prevent migration to sewers, soil and surface and ground water. Remove with explosion-proof equipment. Soak up residue with a noncombustible absorbent such as clay or vermiculite; place in drums for proper disposal.

WASTE DISPOSAL METHODS: Dispose of in a facility approved under RCRA regulations for hazardous waste. Containers must be leak-proof and properly labeled.

VIII SPECIAL PROTECTION INFORMATION

RESPIRATORY PROTECTION: Do not breath mists. Wear a NIOSH approved dust/mist respirator as necessary. If Threshold Limit Value (TLV) of the product or any component is exceeded, a NIOSH/MSHA jointly approved air-supplied respirator is advised in absence of proper environmental control. Engineering or administrative controls should be implemented to reduce exposure.

VENTILATION: Provide sufficient general and/or local exhaust ventilation to maintain exposure below TLV(s). Use explosion-proof ventilation as required to control vapor concentrations below the TLV(s). Ventilation may be required during product drying and curing.

PROTECTIVE CLOTHING: Wear protective clothing as required to prevent skin contact.

PROTECTIVE GLOVES: Wear solvent-resistant gloves, such as butyl rubber.

EYE PROTECTION: Chemical splash goggles in compliance with OSHA regulations are recommended. Do not wear contact lenses because they may contribute to the severity of an eye injury.

OTHER PROTECTIVE EQUIPMENT: Solvent-resistant boots and headgear as required. An eyewash should be easily accessible from the work area. Access to a safety shower is recommended.

IX SPECIAL PRECAUTIONS

WORK PRACTICES: Proper work practices and planning should be utilized to avoid contact with workers, passersby, and non-masonry surfaces. Do not atomize during application. Beware of wind drift. Over-application may contribute to fume problems. Always follow published application rates. See the Product Data sheet and label for specific precautions to be taken during use. **This product is combustible!** Always bond and ground containers during transfer. Eliminate all sources of ignition, even remote sources, as vapors may travel some distance. Smoking, eating and drinking should be prohibited during the use of this product. Wash hands before breaks and at the end of a shift.

This product will continue to evolve vapor during drying and ethyl alcohol during curing. Continue ventilation as needed during curing.

PRECAUTIONS TO BE TAKEN IN HANDLING AND STORAGE: Store away from oxidizing materials, in a cool, dry place with adequate ventilation. Keep away from heat and open flames. Keep container tightly closed when not dispensing product. Wash up with soap and water before eating, drinking, smoking or using toilet facilities. Launder contaminated clothing before reuse.

Containers of this material may be hazardous when emptied, since emptied containers retain product residues (vapor, liquid, and/or solid). All hazard precautions given in the Data sheet must be observed.

Ground equipment to prevent accumulation of static charge. Containers must be bonded and grounded when pouring or transferring materials.

OTHER PRECAUTIONS: Environmental Hazards - Keep out of surface water and watercourses or sewers entering or leading to surface waters.

X REGULATORY INFORMATION

SHIPPING: This product is not regulated when shipped domestic ground in its original, complete packaging. The product is reclassified as a hazardous material for shipping by air, ocean, or in international commerce. Consult with PROSOCO's Regulatory Department for shipping information.

National Motor Freight Classification: NMFC#33980 Class Rate: 55

SARA 313 Reportable:

Chemical name	CAS	Upperbound Concentration % by Weight
None	-	-

XI OTHER

MSDS Status: **Date of Revision:** December 20, 2010
For Product Manufactured After: N/A – no formulary change
Changes: N/A. Regulatory Review for Canadian Customer. No formulary change.
Item No: 42015
Approved By: Regulatory Department

DISCLAIMER:

The information contained on the Material Safety Data Sheet has been compiled from data considered accurate. This data is believed to be reliable, but it must be pointed out that values for certain properties are known to vary from source to source. **PROSOCO, Inc. expressly disclaims any warranty express or implied as well as any liability for any injury or loss arising from the use of this information or the materials described.** This data is not to be construed as absolutely complete since additional data may be desirable when particular conditions or circumstances exist. It is the responsibility of the user to determine the best precautions necessary for the safe handling and use of this product for his unique application. This data relates only to the specific material designated and is not to be used in combination with any other material. Many federal and state regulations pertain directly or indirectly to the product's end use and disposal of containers and unused material. It is the purchaser's responsibility to familiarize himself with all applicable regulations.

DATE OF PREPARATION: December 20, 2010