Freeze/thaw cycling in natural stone

The stone industry is introducing new, unproven materials on a regular basis. Can the wrath of mother nature be accurately synthesized in a laboratory setting?

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The telephone in the Marble Institute of America's Technical Ofice tends to ring . . . a lot. And I'm certain that someday it will ring, and the caller is going to say, "Hey Chuck, I have this stone project. It looks great, and it's performing flawlessly. Just thought you'd like to know". Yet, after nearly a decade of answering this phone, I've not received that call. Indeed, when my phone rings, it means that there is a problem with a stone project. But when a problem exists, it typically isn't due to what I consider to be "bad stone." It usually means that a stone was simply used in an application for which it was inappropriately selected, since someone did not do their due diligence in making a carefully researched selection. One of the most difficult stone selections to properly specify is that stone that will be used in an exterior application in a freeze/thaw climate. Very little information exists to guide the specifier in making an informed decision and to give him/her, as well as his/her client, the confidence that the specified stone will perform satisfactorily in the intended role.

WHY FREEZE/THAW OCCURS

Let's start by looking at the root cause of the problem: Water. All substances have what is called a "specific gravity," which is simply a dimensionless ratio comparing the mass of that substance to the mass of an equal volume of water (technically, distilled water at 4 degrees C). For instance, various stones may have a specific gravity of anywhere from 2.0 to 3.0, meaning that their mass is 2 or 3 times that of the same volume of water. It stands to reason then, that the specific gravity of water is 1.0, because water has the same unit mass as itself. In the metric system, this comparison makes a lot more sense than it does in the units customary to us Americans, because water weighs 1 gram per ml (or cubic cm), which also means that it weighs 1 kg per liter. But what about frozen water (aka "ice")? When a gram of water freezes, it still weighs a gram. But the specific gravity of ice is not 1.0, as is liquid water. It is about 0.92, which means it is lighter than water, which explains why it floats. So this gram of ice now has a volume of 1.0 ÷ 0.92, or 1.09 ml (1.09 cubic cm), and it obviously can't fit in the space in which it was originally contained. This nearly 10% expansion is accompanied by seemingly limitless power and pressure; so much power in fact, that years ago the granite quarries in northern climates intentionally used this as a method of trimming blocks. They would drill holes in the block, fill them with water in the evening, and when they returned the next morning, they would find the block neatly split along the drill holes due to the expansion of the water freezing overnight.

Now that we've identified the culprit as water, we need to locate the accessory to the crime, which is freezing. Without the freezing, we don't have the expansion. Across the U.S., we have climates that range from virtually no freeze/thaw cycles to climates that experience a freeze/thaw cycle in over one-third of the days of a given year. Reference is made to Figure 1, which is a map of the U.S. showing approximate annual averages of freeze/thaw cycles. I suspect none of us are surprised to see that latitude influences

Certain materials have proven themselves to be a durable material in freeze/thaw environments for years, such as the Indiana limestone used for New York City's Empire State building. However, with new materials entering the marketplace at an unprecedented rate, evaluation of their performance in a freeze/thaw climate must be considered.

Photo Credit: Daniel Schwen

Degradation due to saturated freeze/ thaw cycling of a limestone cap

the number of cycles, but perhaps we are surprised to see that at some point, increasing latitude actually results in a decrease in the number of cycles. In the central part of the country, for example, we see a region from about Kansas City to Chicago where the average number is roughly 90 cycles per year. But once we get north of that region, we see that Minneapolis experiences an average of only about 70 cycles per year. This is because Minneapolis has more days that never get about freezing, so no cycle occurs on that day. Logically, if Kansas City has a mild winter, it will probably result in a decrease in cycles because there will be less nights that dip below freezing. But conversely, and perhaps somewhat illogical, if Minneapolis has a mild winter, it will likely increase the number of cycles, since there will be more afternoons when the temperature gets above freezing. Altitude is even more influential in cycling than is latitude. With just a glance at this map, one can clearly see the mountain ranges in the U.S. just by the spike in the number of freeze/thaw cycles.

DIFFERENT FREEZE/THAW CYCLES

Establishing the number of cycles is important, but we also must recognize that not all cycles are equal. A dry freeze/ thaw cycle is completely benign, and the damage caused by a cycle is increasingly more severe as the level of saturation increases. The form of the moisture plays an important part in this assessment. A brief, hard rain is actually one of the least problematic, in that most of it drains away rapidly, and the dwell time on the stone is limited, as is the likelihood of a hard freeze following quickly. A lingering mist would be more damaging, in that it keeps the stone surface wetted for a longer period and allows greater time for absorption to occur. In my experience, the worst scenario is a wet, sloppy snow, since it stays resident on the stone surface and can refreeze in place, then thaw again the next day, and so on, and so on, until the stone is undergoing freeze/thaw cycles while almost fully saturated. A second factor is the stone itself. Many stones have such low porosity that they just don't take on enough water to be significantly affected, and a few stones actually have such open porosity that they effectively evacuate the water before it has a chance to freeze. Third, and perhaps the most important factor to consider, is the orientation of the stone. Vertical work is far less vulnerable than horizontal. Certainly the face of a vertical panel is wetted, but the depth of the penetration is a fraction of what happens to a horizontal stone. I've seen numerous projects where the vertical faces are performing well, while horizontal elements of the same stone are being attacked. When the water, in any form, is allowed to dwell on a horizontal surface, the saturation level of the stone is far greater than what is achieved in a vertical orientation.

The lack of a reliable, consensus established method of testing stone to evaluate the stone's ability to resist damage from freeze/thaw cycling represents the missing tool for the one who is making the selection. Industry associations have not been particularly keen on laboratory weathering tests, showing strong preference to natural weathering as the only valid proving means. For example, the National Building Granite Quarries Association (NBGQA) publishes a statement that reads: "NBGQA recommends that whenever possible, existing installations of a particular granite be used as a measure of the material's ability to withstand the effects

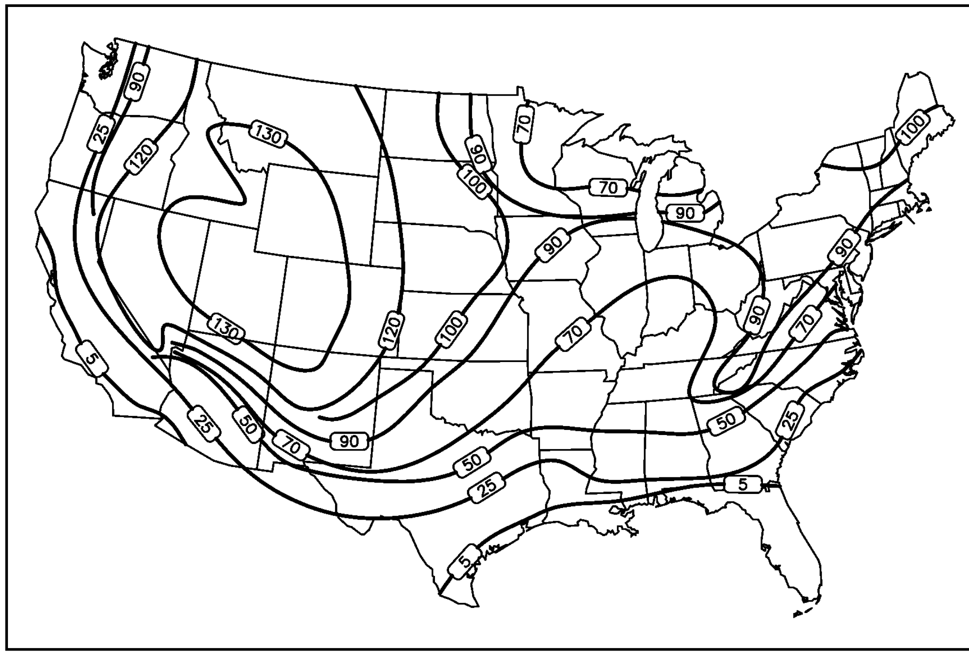


FIGURE 1: Approximate Number of Freeze/Thaw Cycles per Year

of environmental elements. The condition of the product after many years in actual service is a far more reliable indication of its ability to perform than an accelerated laboratory test."

Meanwhile, the Indiana Limestone Institute publishes a similar stance on the subject: "Poor performance by other stones has caused concern for the durability of stones in general; indeed, accelerated weathering test programs may be necessary for untried materials. Many such programs are widely promoted; they tend to be costly and time-consuming."

Both statements are essentially correct, in that there does exist consensus that the best indicator of in-service performance is actual in-service history, and there does exist consensus that accelerated weathering testing requires a substantial investment in both time and money. Yet neither statement addresses the fact that the industry and its consumers need a method of evaluating unproven materials. New deposits of stone are being tapped with a frequency that has been previously unheard of. But if in situ performance history is required to validate a stone for use, then how does one validate a new, previously unused material?

Specimen Size:	50 x 50 x 300 mm (2″ x 2″ x 12″)
Frozen:	In Air
Thawed:	Submerged
Initial Freeze Temp:	≤0°C; ≥-8°C (≤32°F; ≥18°F)
Deep Freeze Temp:	≤-8°C; ≥-12°C (≤18°F; ≥10°F)
Thaw Temp:	≥5°C; ≤20°C (≥41°F; ≤68°F)
Cycle Time:	12 hrs
Cycles:	As Specified, or until Failure, or up to 168 cycles
Degradation Tracking:	Visual & Nondestructive Sonic E Testing

FIGURE 2: Temperatures & Cycle Times for EN 12371

TEST PROCEDURES

Within the U.S. we rely mostly on test procedures authored and published by ASTM International. Within this vast library of test procedures, none exists for the purpose of evaluating a natural stone's ability to resist degradation due to freeze/thaw cycling, yet a procedure exists for numerous other construction materials. On the other side of the Atlantic, our European counterparts use procedures authored by CEN (Comité Européen de Normalisation). They are a bit ahead of us in this regard, as within the CEN documents, there exist two test procedures for this purpose.

EN 12371 is titled "Determination of Frost Resistance." It involves the freezing of a saturated specimen in air, followed by thawing it submerged. The freezing cycle is split, in that it first undergoes an "initial freeze" stage, followed by a "deep freeze" stage. There are a lot of variables and tolerances that can be applied to the test procedure, and one must really obtain a copy of the full test procedure to fully understand it, but a rough outline of the cycle is found in Figure 2.

While not testing freeze/thaw cycle resistance, there is a related European test procedure EN 14066 titled "Determination of Resistance to Aging by Thermal Shock." This procedure, outlined in Figure 3, involves heating the stone to extremely elevated temperatures in air, followed by immersing it in water at room temperature.

ASTM C666 was written for testing concrete, and is aptly titled "Standard Test

Specimen Size:	200 x 200 x 20 mm (8″x8″x ¾″)
Method:	Heated in Air; Submerged in Water
Heated Temp:	105 ±5°C (221 ±9°F)
Submerged Temp:	20 ±5°C (68 ±9°F)
Cycle Time:	24 hrs
Cycles:	20

FIGURE 3: Temperatures & Cycle Times for EN 14066

Specimen Size:	± 4″ x 11″ x 16″ (100 x 280 x 400 mm)
Frozen:	In Air or Completed Surrounded in Water
Thawed:	Submerged
Freeze Temp:	0° F (-18°C) (Tolerance = ±3°F, °1.5°C)
Thaw Temp:	40°F (4°C) (Tolerance = ±3°F, °1.5°C)
Cycle Time:	± 5 hrs
Cycles:	300
Degradation Tracking:	Visual & Nondestructive Sonic E Testing

FIGURE 4: Temperatures & Cycle Times for ASTM C666

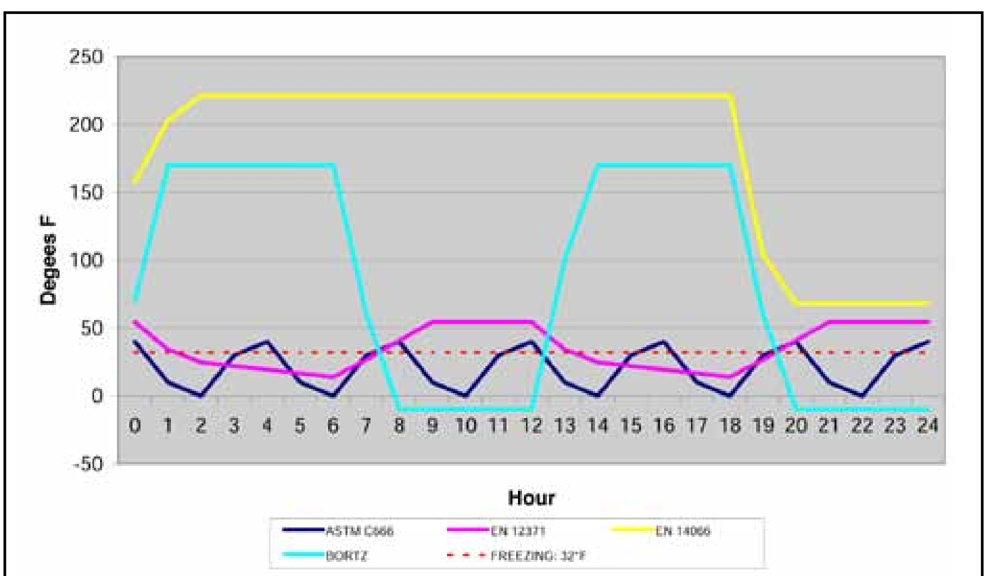
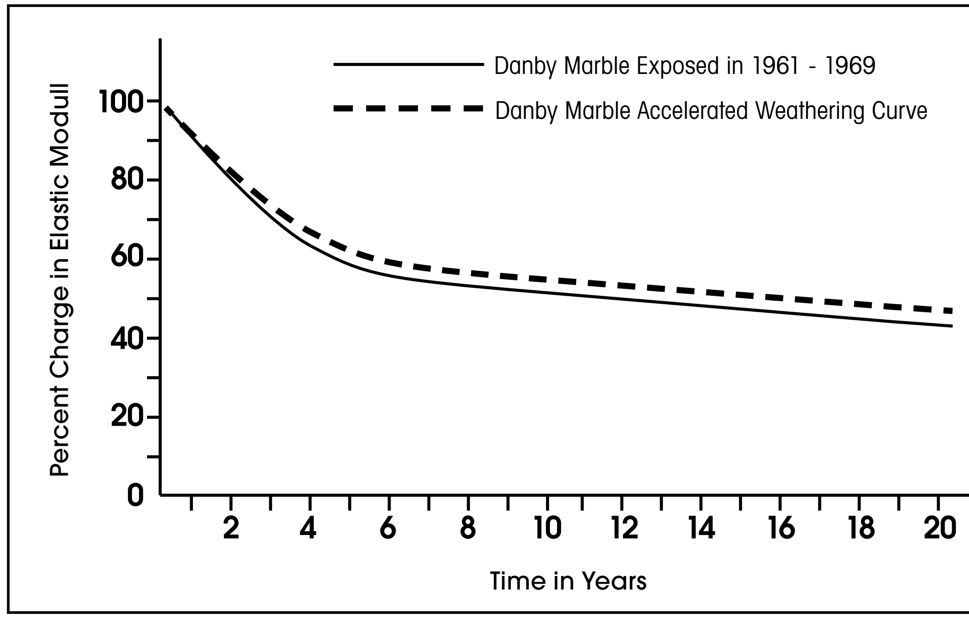


FIGURE 5: Comparison of temperature and cycle time

Procedure for Resistance of Concrete to Rapid Freezing and Thawing." Despite having been authored for use in testing concrete, this test procedure is one that has frequently been used as a platform, with modifications, to test natural stone. Again, this procedure allows the specifier of the test to toggle some options and make modifications, but the procedure will be similar to what is found in Figure 4. Perhaps the most noticeable departure from the European procedures is the extremely rapid cycling found in this procedure.

Within the U.S., I can think of no individual who has done more research on degradation of natural stone due to weathering than the late Seymour "Sy" Bortz. Mr. Bortz's research spanned decades, covering the time that he was at IIT Research Institute and later years when he was with the firm of Wiss Janey Elstner. He authored and/or co-authored many papers on this particular subject, and discussion of this topic without including Mr. Bortz's research would be woefully incomplete. His self-developed method of testing stones started with a basic platform of C666, but he made some interesting, yet very logical, modifications to it. For one, he included a slightly acidic bath using sulfurous acid (H2SO3), which was intended to provide some simulation of the effects of acid rain. And many of his tests were run with a much greater temperature span, running the samples up to 170 degrees F (77degrees C) in the thawing cycle and down as far as -10 degrees F (-23 degrees C) in the freezing cycle. These temperatures were not randomly selected, however, as they represent the extremes that he had personally measured on stone facades in the Chicago area, where he lived and worked. Of additional interest is the fact that he obtained duplicate test specimens for much of his research, and while one set was tested via accelerated laboratory methods, the other was naturally weathered on a building rooftop to serve as a control group. His research spanned enough years that the control group had experienced enough weathering to provide meaningful comparison. Having not been privy to

FIGURE 6: Correlation between sonic elasticity loss in accelerated test specimens compared to naturally weathered specimens.



the background of the development of the European or C666 procedures, I do not know if similar correlative data from natural weathering was developed when those procedures were authored.

A key element in tweaking and fine-tuning a test procedure is having ample amounts of data produced by that procedure, from which one can compare what was observed in the testing against what was observed when the tested products were put into actual service conditions. But while substantial data has been collected from the four methods mentioned here, it cannot be effectively amalgamated into one large pool of data since the methodologies of the tests differ so significantly. The range of extreme temperatures, the duration of the cycles, the level and chemistry of the bath and the specimen sizes vary so greatly so as to prohibit meaningful comparison of the results. Reference is made to Figure 5, which shows a rough comparison of a typical day in the life of a test specimen if subjected to any of these four procedures. Clearly, there is little commonality amongst the test protocols.

Just as dissimilar as the methods are their respective definitions of failure. ASTM C666 does not have any failure definition that is applicable to natural stone, since it's a concrete test. In Mr. Bortz's research, he did not actually draw a line representing the difference between "pass" and "fail," yet I've heard many producers claim that their material somehow "failed" the test, despite having no criteria that defines failure. The clearest definition of failure occurs in CEN's EN 12371, which lists five conditions that deem the stone to have failed the test:

- One or more cracks greater than 0.1 mm (0.004 inches) in width
- The detachment of any fragment greater than 30 square mm (0.05 square inches)
- Alteration of minerals within veins
- Signs of crumble or dissolution
- Loss of sonic modulus exceeding 30%

The above indicators of failure are not extreme symptoms. A crack that opens to 1/10 mm is not very wide -- about the thickness of a sheet of standard weight photocopy paper. And a fragment of 30 square mm represents an area about the diameter of a pea. Sonic modulus is a little different, in that it's not something that we can visually see. Modulus of elasticity is a measure of stiffness, and represents the ratio of stress to strain in a material. A material that can take more stress without a lot of strain (deformation) would have a high modulus of elasticity. Strength and elastic behavior are related, so if we see a reduction in one, we know that there has been a reduction in the other. The reason we measure modulus of elasticity as a surrogate for flexural strength is that the latter test would destroy the specimen. Elasticity can be measured nondestructively, so that the same specimen can be put back in the test chamber for subsequent cycles. Two methods are commonly used to measure this property. One is by bending the sample in a fixture, measuring the load from which we can calculate the stress and measuring the deflection from which we can calculate the strain, but this method carries a high risk of destroying the specimen. A safer method is sonic measurement, in which the test specimen is vibrated to determine the resonating frequency of the material, basically like a "stone tuning fork." The relationship between strength and stiffness isn't exactly linear, so one cannot say that a 30% elastic modulus loss indeed equals a 30% loss of strength, but it's probably close. From data that I've seen on this type of testing, losing 30% will occur with greater likelihood than one would expect.

In Mr. Bortz's testing, he had the advantage of comparing the sonic elasticity loss in the accelerated test specimens to his naturally weathered specimens. Figure 6 is a graph taken form a paper he published in 2007, showing nearly perfect correlation between the synthesized procedure and

Photo Credit: Cold Spring Granite Co.

A classic time-tested material, Carnelian granite from Cold Spring Granite Co., was used for the FDR Memorial in Washington, DC.

the natural exposure. Admittedly, this one of the best correlations achieved in the study, but it is nonetheless a remarkable testimonial for the potential validity of an accelerated procedure.

OBSTACLES TO TESTING

So why has the topic of testing weathering durability in stones been one where consensus is difficult to reach, when it has been achieved elsewhere? The two greatest roadblocks are: costs and fears

The costs are indeed significant, as a testing regimen of this type is easily five figures. Expensive laboratory equipment is required and occupied for a long time, significant laboratory technician time is consumed, and just the cost of energy to create the freeze/thaw cycles is considerable. And in addition to the monetary cost is the cost in time. Even if you're getting 4 cycles a day, a 150-cycle test is going to take about 1 ½ months out of your schedule, which most projects cannot absorb.

The fears that occupy the minds of many are generally in three categories: Invalidity of the test results; universal specification of the test procedure; and validation of new products. The fear of the invalidity of the test results simply requires a thorough study of the data that exists and comparing it to natural weathering. The correlation is actually far better than many are inclined to believe, and increased research and usage will allow adjustment to improve upon that correlation. Universal inclusion in specifications is definitely something that we must guard against. All too often, we see specification writers specifying every test that exists, despite the fact that some of the tests will offer no data that is influential to their design decisions. The EN procedures carry a statement reading: "It is not intended that all natural stones products should be subjected regularly to all the listed tests. Specifications in other standards should call up only relevant test methods." This is a message that we need to articulate more clearly to those authoring specifications. But why would validation of new products be a problem? If one is a quarrier of a his-

The Carnelian granite used for the FDR Memorial was specified in a range of formats and finishes.

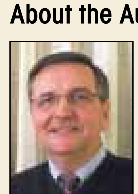
Photo Credit: Cold Spring Granite Co.

Photo Credit: Michael Kranewitter

torically proven material with demonstrated durability performance, the absence of an accelerated test procedure provides a competitive advantage, since an unproven competing material cannot be confidently used. Having a test procedure that could validate the unproven material would remove this competitive advantage.

Reaching consensus on any topic is a slow and sometimes painful process, and when the topic potentially affects the pocketbooks of those involved, the process gets even slower and more painful. Reaching consensus on this topic starts with addressing the fears. Data needs to be studied further, and procedures tweaked until the best correlation between synthesized and natural weathering can be achieved. And we must accept that this will never be a perfect replication of what actually happens to a stone, just as none of our test procedures are perfect replications of what the stone experiences in service. We do, however, need to guard against the universal specifying of any test procedure, as no project has leftover time or money to run a test unnecessarily. Would I spend my money or my client's money to run weathering tests on Dakota Mahogany granite or an Indiana limestone? Absolutely not, as I can point to hundreds of projects of either material where Mother Nature has already tested it for free. But when an unknown and unproven material is desired, the costs represent a prudent investment. ■

About the Author:



Charles J. "Chuck" Muehlbauer is the Technical Director of the Marble Institute of America, a stone industry association comprised of more than 1,600 member companies from over 50 countries. Muehlbauer is now in the fourth decade of his career in the stone industry, with experience in quarrying, fabrication, installation, anchorage design, stone selection, industrial stone applications, laboratory testing, stone forensics, estimating and both domestic and international sales. Muehlbauer is a voting member of seven national technical committees, including ASTM Committee C-18 on Dimension Stone, where he currently serves as Chairman.