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Applications and possibilities of forecasting methods for building ceramics: A review of Literature

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--ABSTRACT--

The structure of ceramics is non-uniform, and many scientists have studied the size and distribution of pores and capillaries in ceramic products. The mechanism of disintegration of ceramic products during freezing is reviewed in detail. Rapid forecasting methods based on one parameter are not promising. Forecasting methods, when based on a higher number of parameters, are much more reliable and better show the frost resistance of ceramic products. In the current period, two directions of ceramic product forecasts are distinguished, one area basis on structural and strength parameters, the other on deformation parameters. Rapid forecasting methods allow for the evaluation of the frost resistance of ceramic products quickly enough, and by applying them, it is possible to stop the entry of low-quality products into construction.

KEYWORDS; *frost resistance, forecasting method, porous reserve ,dilatometry*

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I. INTRODUCTION

Construction ceramics are used in different areas. In colder areas, they are exposed to the effects of frost caused by frozen water penetrating the pores of the material. It is known that freezing of water-saturated porous products increases the volume of the water by 9 % as it passes through the solid state, resulting in tension and deformation. The ability of a material to resist tension and deformation depends not only on its structure but also on its phase composition. It is now known that the crystallisation pressure is responsible for most of the tension in the pores of the material. Damage occurs when these tensions exceed the local tensile strength. In this case, the problem arises, reducing the mechanical strength of ceramic products and requiring costly repairs [1-5]. The frost resistance of products has been interpreted in different ways and is mainly related to the ceramic product properties, the total volume of the pore and capillary space [6, 7] and the size distribution of the pores [8-10]. In ceramic products, decomposition is caused by moisture and temperature. Not only the freezing of water, but also the redistribution of water in the products before and during freezing has a significant effect on the breakdown of the porous structure. The frost resistance of ceramic products depends on the saturation and the water-ice and ice-water phase transformations. Since most of the methods used to derive and validate tension generation theories use indirect parameters related to phase transformation. It is difficult to reach a consensus on the exact explanation of tension evolution [11]. Attempts have been made to better understand the origin of the tensions that develop in tiles during the freezing process. The analysis of the interaction between liquid, ice and solids allows us to divide this average pressure into two parts. One may be due to the pore size distribution and the experimental variation in the volume fraction of the ice produced by freezing. The second part expresses the additional pressure due to the liquid phase. Obviously, the strict pore size distribution plays a secondary role compared to the pressure generated by the liquid phase. The importance of the liquid phase is due to the ability of water to move within the pore volume. The dominant parameters seem to be the distribution of the ice crystals formed and the permeability of the porous material to liquids [12]. The tensions that occur in a ceramic product upon uniform saturation and freezing are dependent on the size of the pores and the geometry of the pores and capillaries. Initially, micro cracks are formed in the material. These micro cracks will grow and merge with each other until the final crack occurs. The main parameters affecting the durability of ceramic materials, according to the nature of the material, are porosity, modulus of elasticity, permeability and boundary conditions [10, 13]. Through a series of experiments, researchers have found that it is not only porosity and pore size that are important in assessing the frost resistance, but also the mechanical properties of the material that enable higher strength ceramics to withstand the internal tensions that arise from the soaking of water [14, 15].

The tests showed that, in particular, water in the smaller pores is instantly unfrozen, while in the larger pores it is surrounded by an ice phase due to a decrease in freezing temperature inversely proportional to the radius of the pore. It has also been found that the wider the pore size range and the slower the rate of cooling, the greater the proportion of the volume of water that is not instantaneously frozen that causes frost damage [16].

The probability of dangerous tensions and deformations is lower with a more favourable structure containing reserve pores, which provide a free space for the entry of excess moisture. It has long been known that increasing the firing temperature of a ceramic product makes the pores and capillaries larger and increases their durability [17, 18]. Increasing the firing temperature reduces the total volume of the pores and capillaries and increases the glass phase [17,18].

Some researchers assess the frost resistance by structural characteristic (volume of reserve pores). If this structural characteristic exceeds 9 %, they consider the material to be frost resistant. Such a material is assessed by the mercury porometry method. Due to the uneven structure of the samples, the data obtained can be very unreliable [18]. Fagerlund [19, 20] showed many years ago that below a certain degree of saturation in Scrit (saturation degree S, defined as the ratio of the moisture content to the moisture content when all accessible pores are filled with water), brick can be exposed to hundreds and thousands of times the freezing temperature above and below the freezing point with no measurable damage. He defined the degree of saturation above which fracture can occur as Scrit.

Many scientists have long tried to find different characteristics of ceramic products that can be predicted. One of the parameters related to frost resistance is scrit. The water saturation coefficient K_n has been used for this. Practice has shown that rapid methods based on a single parameter are unreliable. Many scientists consider that it is more reliable to use a complex set of parameters to judge frost resistance [4, 5]. Such methods are based on complex parameters. A correlation is established between frost resistance and structural and strength characteristics [4,5].

II. FORECASTING METHODS ANALYSIS AND DISCUSSION

PREDICTING FROST RESISTANCE FROM STRUCTURAL AND STRENGTH CHARACTERISTICS

The size of the pores in the brick material influences the resistance of the brick to freezing and thawing cycles [4, 5, 21]. According to the literature, pores larger than 1 µm (large pores) can be easily filled with and removed by water, increasing the durability of bricks [21, 22]. According to Kung [23], small pores (less than 0.1 µm) have little effect on the resistance of bricks to freeze-thaw cycles, as the water in them only freezes at very low temperatures, while medium sized pores are harmful. The Maage coefficient/factor (Fc) to predict the resistance to freeze-thaw cycles is based on experimental results and a statistical model with two main variables, the total pore volume (PV) and the number of pores of a specific diameter, i.e. pores larger than 3 µm (P3). DF > 70 - high probability of frost resistance in severe climates - 55 < DF < 70 - undefined frost resistance zone and - DF < 55 - low probability of frost resistance [24 – 27], speaks of a positive effect of large pores on the resistance of bricks to freeze/thaw cycles. In addition to Maage, the assessment of the resistance of bricks to freeze-thaw cycles based on the characteristics of the material's pore system has been proposed by some other authors, such as Koroth, Vincenzini, Franke and Bentrup, Litvan and Nakamura [28]. Most of these authors consider that larger pairs have a positive effect on resilience. Franke and Bentrup additionally introduced the average pore radius as a parameter to assess the resistance of bricks to thaw cycles [29]. Koroth et al. [30] also developed a durability index based on the assumption that a pore fraction greater than 3 µm plays a decisive role in the frost resistance of clay bricks. Franke and Bentrup [31,32] have shown on 40 different types of bricks (new and old) that the most important factor is the average pore radius. Bricks with at least 40% pores with a diameter greater than 0.25 µm or with an approximate number of pores. 40% of pores with a diameter greater than 1.4 µm have good frost resistance [33]. If the predominant pores in the material are between 0.5 and 1.4 µm, the material has low frost resistance. Robinson [34] in turn observed that materials with a predominance of pores less than 1 µm in diameter have low cold resistance, in contrast to materials with a predominance of larger pores greater than 2 µm in diameter. Pores greater than 3 µm in diameter have been shown to have a significant effect on the improvement [35,36].

The frost response of ceramic products depends on parameters such as the raw material and its composition, the firing process, and the properties of the manufactured products, such as the pore size distribution, the shape of the pores and the strength of its structure, or the prediction of the cold resistance. However, indirect methods are also used, which provide a prediction of frost resistance based on knowledge of freezing phenomena, which are influenced by porosity distribution, degree of saturation, freezing rate, etc. [37].

The authors concluded that a combination of properties would better predict durability in service. The proposed combinations would be specific saturation factor maxima (or initial absorption rate ranges) where the boiling water absorption is above or below a certain value. The indirect method of ASTM C 1167-03 [37] has received more attention in the past and is quick and simple to implement, predicting the frost resistance by the saturation factor S. This factor is the ratio between the water absorption after 24 h soaking in cold water and the water absorption after 5 h boiling. The classification of freeze-thaw resistance is as follows: - S < 0,74 - high probability of frost resistance in harsh climates, - conditions 0,74 < S < 0,84 - indeterminate frost zone resistance, and - S > 0,84 - low probability of freeze resistance.

For the prediction of frost resistance, the following equation has been proposed using several characteristic physico-mechanical parameters [5] :

$$S \approx \frac{r^{0,17}\ \sigma_b}{T^3 P^2 tg\alpha\sqrt{E}} \cdot \frac{B}{D} \quad (1)$$

where FS - Frost resistance number; r - average pore radius; T - penetration coefficient, the strength coefficient based on tensile and flexural strength; P - total porosity of the specimen; tg - angle of curvature of the integral curve for the size distribution of the pores and capillaries; E - modulus of elasticity of the material; B/D - the ratio between the tile's surface area and the tile's width. In this case, it is assumed that at FS11 the specimens are considered to be non-resistant, 11-35 are transient and 35 are frost resistant. Applied only to the frost resistance of tiles, but not widely used.

The prediction methods were combined with studies carried out by direct methods. Sadūnas, Šiaučiulis, Kaminskas concluded that frost resistance can be predicted by capillary rise and flexural strength. Subsequently, a further empirical prediction formula was developed to better analyze structural and strength characteristics [5]:

$$M = \frac{e^{-2,63} R_l^{1,06} A^{0,61}}{\left({}^{q}/_{H^2}\right)^{0,939}} \quad (2)$$

M - frost resistance in cycles; e - base of natural logarithm; R_l - bending strength; A – destruction work of the volumetric unit of material; q – relative quantity of displaced ice; H - capillary rise height in 1 hour;

Evaluates frost resistance, but would only allow cycles to be evaluated by considering charring fractures (rather than those produced by one-way freezing).

Subsequently, another rapid prediction method has been developed by establishing a correlation between its strength, structural characteristics and frost resistance. The formula presented was used to predict the in-service frost resistance from its structural and strength characteristics [4,5]:

$$M= \frac{R_l^{0,727}\ A^{0,903}}{(q/H^2)^{0,553}(\Delta H/H)^{0,839} K_v^{10,561}} \quad (3)$$

M - frost resistance in cycles of unidirectional freezing; e - natural logarithm basis; R_l - bending strength; A – destruction work of the volumetric unit of material, q/H^2 - porous structure irregularity characteristics, Kv - water saturation coefficient.

On the positive side, this method provides a reasonably rapid determination of indicators and also shows the impact of technological factors. The qualitative firing of the ceramic product can be judged by A and R_l, cracks by A, irregularities in the structure of pores and capillaries by H/H_2 , and the frost resistance of the structure by q/H^2. The methods developed can be used to predict frost resistance within 3-7 days. This equation satisfactorily expresses the relationship between frost resistance and is suitable for predicting the frost performance of ceramic products in terms of structural and stiffness characteristics.

Further prediction equations have been derived for comparison with one-way freezing methods. The deformation of water-saturated ceramic products during freezing is known to be tension dependent. The magnitude of the tension depends on the degree of water saturation and the rate of freezing. The amount of moisture migrating to the surface to be cooled depends on the pore and capillary structure of the material, partly on the size of the sample (with which the percentage of moisture is related etc.). In the case of unidirectional freezing, the destructive tensions are concentrated at the surface of the specimen. Therefore, this prediction method was compared with the one-sided freezing and thawing.

Another rapid prediction method is the assessment of the 3-day water absorption (one of the main indicators in the calculation of frost resistance). Water absorption is also determined by vacuuming the sample. This opens up the possibility to calculate the pore space reserve (R) as well as the ratio of pore to capillary thickness. The water content gain in 10 min in two different directions under vacuum and in one direction under normal conditions is determined. In this case, anisotropy can be assessed. The full set of basic structural properties as well as the maximum and minimum height of rise.

It is known from the theoretical basis that the reserve, water-free pore and capillary space is filled with water and undergoes surface decomposition by cyclical unidirectional cooling and thawing and by simulated rain due to migration and phase transformations. The reserve R and the relative wall thickness D are directly proportional to the cold resistance index. The ratio G1/g2 or G1/G2 will compensate for the anisotropy of the structure depending on the effective porosity of the product being < 26 %, in which case the main index of pore and capillary reserve filling will be G2, and in the case of > 26 % the index is g2. For the inhomogeneity of the structure in the direction of the frost resistance, the characterisation of the h/h_{min}. parameter is unambiguous, and the index itself is inversely proportional to the frost resistance parameter [4,5, 38, 39].

$$F_{R1}=0,231\,\frac{R^{1.068}\ D^{1.345}\ G_1^{0.275}\ G_2^{0.663}}{N_h^{0.285}\ g_2^{0.830}},\ W_e \leq 26\%\ (4)$$

$$F_{R1}=0{,}051\,\frac{R^{1.642}\,D^{2.332}\,G_1^{0.695}\,g_2^{0.779}}{N_h^{0.334}\,G_2^{1.145}},\ W_e>26\%.\ (5)$$

Similar equations are derived for the end of specimen disintegration because the ceramic products are destructing in two stages. The first phase starts with the appearance of visible lesions, and the second stage lasts from the first signs to the removal of the frozen surface.

$$F_{R2}=\ 0.223\,\frac{R^{\ 1.456}D^{0.759}G_1^{0.384}G_2^{0.852}}{N_h^{0.334}\,g_2^{1.034}},\ W_e\le 26\%\ (6)$$

$$F_{R2}=0.063\,\frac{R^{1.813}\,D^{2.135}G_1^{0.178}\,g_2^{1.335}}{N_h^{0.395}\,G_2^{0.517}}\ W_e>26\%.\ (7)$$

The in-service frost resistance using these equations can be carried out within 10-12 days. These equations are relatively universal since they have been developed for ceramic products manufactured by different techniques and in different plants. These formulas are currently used by scientists [14, 39-41].

PREDICTING FROST RESISTANCE USING A DILATOMETER

Another rapid method of assessing frost resistance is the dilatometric method, which is very sensitive to internal changes and correlates with those occurring during cyclical freezing and thawing. As early as 1951, a rapid prediction method using a dilatometer was proposed. The authors considered that if the deformation is negative and reaches 1 to 2,5 μm, the material is resistant to frost. Angenitskaya measured the linear deformation of ceramic materials during freezing using strain gauges. Other researchers have used a quartz dilatometer for volumetric cooling to rapidly assess the frost resistance of ceramic tiles. They found that the deformation of the quality-fired samples was lower compared to the deformation of the poorly fired tiles. The researchers split into two parts. Some thought it was not appropriate, while others did not doubt the validity of the method [42]. Dilatometry can be used as a method for assessing the weathering behaviour of rocks and the role of frost action [43] in water migration during this process. As anisotropic behaviour can only be observed by measuring the sample in different directions, we believe that dilatometric studies will become an important method for the analysis of decomposition processes due to frost action.

Other studies have shown that cylinders of calcareous rocks undergoing wetting/drying cycles (not subject to freezing) vary in length. These changes in length are most significant when water enters or leaves the smallest pores in the rock. Most authors observing the shrinkage of frozen rock samples have had great difficulty in explaining their observations [44,45]. The primary role of unfrozen water migration has been emphasised by several authors, both in freeze dilatometry [46] and as a mechanism to explain the formation of ice lenses perpendicular to the direction of freezing [46]. Indeed, water migration in one particular direction, i.e. towards the freeze line, can be considered responsible for the formation of segregation ice and rock wedging [47]. Frost dilatometry can be used to determine the critical degree of saturation [48].

The surface layers of ceramic products are the most dangerous for damage, where maximum tensions and deformations occur and develop. The dilatometric device DUM-01 has been developed [4,5,49,50] . The method of deformation variation by cooling the ceramic product from one side is convenient in that, even without knowing the structural or compressive properties of the material, the direct results of the effect of the frost can be seen in the form of stress-strain pronation. After the research, the decomposition process was analyzed in three stages. During the first phase, defective pores and capillaries are filled with water. In the second stage, ceramic products break bonds, and internal cracks appear. In the third stage, visible cracks appear [51]. The first decay of ceramic products may occur in the weakest areas, where the highest values of the general linear deformations were observed.

The result of the breakdown is recorded by the variation of the residual deformation. Studies have shown that strain rates correlate strongly with frost resistance. One way of expressing the strain in terms of relative area units on the automatic recording device S1, S2,S3, S4, where S1 and S3 are the area of defomation obtained during the 1st and 3rd cycles, and S2, S4, is to derive the equation [5]:

$$M=\ e^{k_1+k_2S_1+k_3S_2+k_4S_3+k_5S_4}\ (8)$$

In a more detailed analysis, the equations for the beginning and end of the decay were derived.
The measurement time per sample is 5 hours, excluding 48 hours of water saturation. Subsequent studies have shown that often the optimum performance in terms of frost resistance is obtained from the equations [5]:

$$M=e^{4{,}78\,-\,k_1\Delta h_1-k_2K_v+k_3N}\ (9)$$

Δh_1 - the largest common linear deformation in the 1st freezing cycle, K_v-water saturation coefficient, N-degree of structural inhomogeneity.
The results of this equation are reliable.
It is also possible to predict the performance of the frost resistance from the strain indices, but it is better to use the dilatometric tests to explain the processes involved in the cooling and thawing of ceramic products [49-51].

III. CONCLUSION

Scientists have explained in detail the mechanism of destruction of ceramic products. The complete filling of pores and capillaries with water is necessary for the formation of the anti-collapse situation. The ice volume in large pores and defective areas gradually fills up due to the migration of water from small pores and capillaries, where the freezing of water in this cyclical freezing and thawing process is limited. Further cyclic freezing and thawing, due to the additional gradual filling of the pores and capillaries with water, generates high tensions, which gradually build up to high tensions, which initially cause internal local indentations, which gradually merge and complete delamination occurs. It was found that if large pore capillaries prevail in the system, the water is easily pushed from the frost zone to the warmer zones and in the finely distributed - in the form of water films - to the freezing ice. The destruction process takes place over a period of time, from the first signs of disintegration to the complete disintegration of the surface and the decomposition of deeper layers. For ceramic products used for objective finishing, it is necessary to record not only the beginning but also the end of disintegration. The nature and speed of surface disintegration and its further development depend on the location of the defect zones in the ceramic product.

Prediction methods based on a single parameter are not objective and should be discarded. It was found that the structure of the products, especially the texture, is different. Prediction of frost resistance when sown in different ways is not accurate enough. Rapid predicting methods should be used for rapid assessment. They can be used to stop the introduction of low-quality products into construction.

In order to predict the frost resistance of ceramic cladding products, it is necessary to use methods based on unidirectional freezing for prediction. Methods for predicting the serviceability of products are particularly useful for manufacturers of such products, as they allow a relationship to be established between the main technological factors and parameters of production and the serviceability of the products.

Dilatometric methods are best suited for the analysis of the degradation processes of ceramic products.They can also be used effectively to predict the frost resistance of ceramic products.

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