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Dimensional and ice content changes of hardened concrete at different freezing and thawing temperatures

Björn Johannesson

Department of Civil Engineering, Building 118, Technical University of Denmark, 2800 Lyngby, Denmark

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ABSTRACT

Samples of concrete at different water-to-cement ratios and air contents subjected to freeze/thaw cycles with the lowest temperature at about −80 °C are investigated. By adopting a novel technique, a scanning calorimeter is used to obtain data from which the ice contents at different freeze temperatures can be calculated. The length change caused by temperature and ice content changes during test is measured by a separate experiment using the same types of freeze–thaw cycles as in the calorimetric tests. In this way it was possible to compare the amount of formed ice at different temperatures and the corresponding measured length changes. The development of cracks in the material structure was indicated by an ultra-sonic technique by measuring on the samples before and after the freeze–thaw tests. Further the air void structure was investigated using a microscopic technique in which air 'bubble' size distributions and the so-called spacing factor, indicating the mean distance between air bubbles, were measured. By analyzing the experimental result, it is concluded that damages occur in the temperature range of about −10 °C to −55 °C, when the air content is lower than about 4% of the total volume. For a totally water-saturated concrete, damages always occur independently of the use of entrained air or low water-to-cement ratios. It is, further, concluded that the length changes of these samples correspond to the calculated ice contents at different temperatures in a linear fashion.

1. Introduction

The freeze/thaw resistance of concrete is highly dependent of the amount of entrained air besides the mechanical strength. Natural occurring amounts of air in concrete, i.e., about 1–2% of total volume, have generally been shown to produce concrete not being frost durable. For this reason, concrete for outdoor climate in countries with cold winters is almost always supplied with air contents of about 4–6%, which in most cases give a good performing product.

Here results are presented on ice contents, measured using a calorimetric technique, at different freezing/thawing temperatures. An approximate method of evaluating the ice contents from the calorimetric data is presented. Length changes are measured on separate parallel samples exposed to identical freezing/thawing conditions as the samples tested in the calorimetric device. The results from this investigation can potentially be used to identify some of the hypotheses which have been proposed for the ice growth and ice induced damage of cement based materials.

Theoretical considerations, using the concepts behind the so-called 'hydraulic pressure', state that the distances between the entrained air voids are of crucial importance for the determination of the frost resistance. The background is that formed ice in the spatial domains surrounding air voids, having much smaller pores than the entrained air spaces, pushes water not yet being frozen away from where the ice is formed and eventually reaches an air filled void. The assumption is that this flow of water builds up a mechanical pressure which, among other things, is proportional to the ice formation rate. If the entrained air voids are too far apart, the theory predicts that the developed water flow pressures exceed the tensile strength of the material. The method relies on the fact that one assumes that the domains surrounding air voids are at such a high water saturation level that pressures and hence water flows can be induced [1,2]. Since the ice formation rate is central in the above-mentioned hypothesis, investigations have been performed on the influence of freezing temperature on the mechanical behaviour of concrete. Some investigations, however, show very little influence of the rate of freezing rate, e.g. see [3,4].

If the material is totally water saturated, meaning that also the air voids are water filled, the theory behind the hydraulic pressure is not applicable since water not yet being frozen cannot escape from where the ice is formed. In this case the 'closed container model' is instead valid, which simply calculates the stress induced by the volume increase of water due to the freeze process. Further, simple thermodynamic calculations, using the Clapeyron equation, yield a pressure increase of approximately 10 MPa/K for 'free'

E-mail address: bjo@byg.dtu.dk

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water and ice existing together in equilibrium below the normal bulk melting temperature, provided that the 'container' do not yield and that no special interaction between the pore walls and the ice and water is present. Fully saturated concrete cannot withstand the high pressures predicted by Clapeyron equation, meaning that the reason for water and ice coexisting at freezing and thawing has other explanations than those obtained by constrained pressures alone. It should be carefully noted that microscopic mechanical behaviour may give other results than the acroscopic hypothesis described above.

In real situations the concrete seldom becomes totally saturated, especially the entrained air voids are of dimensions making them practically inaccessible for water. Assuming that formed ice can 'grow' in the pore structure of the material in a stress-free manner, the limit saturation degrees will be 0.917 above this value, there is no space left for growth and the ice and water will start to assert a pressure to the pore walls. The limit 0.917 is based on the volume of ice being 9% larger than water. It has been experimentally proven that damage to concrete occurs at much lower saturation degrees than predicted theoretically for stress-free growth for partly saturated situations, e.g. see the results presented in [5].

Due to implications of both the close container and hydraulic pressure theory, yet another mechanism for the action of frost damages in concrete has been proposed. This mechanism is initially based on the observations on frost-induced heaving in soils. From experiments it was observed that in the cold end of a unidirectionally cooled cylindrical soil specimen, ice lenses were able to grow by consuming water from the warmer parts of the specimen and that the resulting ice lenses were able to assert considerable pressures, [6]. Observing length changes of samples during times of a constant freeze temperature, it was suspected that also ice lenses in concrete may attract surrounding water in a way similar to that of frost-induced heaving in soils, but in this case in a microscopic perspective only. The mechanism, introduced in [7], was referred to as the osmotic micro ice body growth. According to the hypothesis, freezing is initiated in some of the larger pores when cooling proceeds, the unfrozen water in the smaller pores will have a tendency to be transported from the smaller pores into the large ones where it will freeze onto the existing ice bodies. The transient character of transport of water to the ice bodies is, according to the hypothesis, the explanation for experimentally observed expansions at constant temperature. An extended version of this hypothesis, also considering cases of salt-frost scaling, is presented in [8]. It is concluded to be difficult to verify the osmotic micro ice body growth hypothesis experimentally; this is, partly, due to that the governing processes occur at the microscopic level. Some interesting discussions on the applicability of the hypothesis are performed in [9,10].

Besides, the three main mechanisms discussed to this end it has been proposed that super saturated water and ice in a porous material, assumed water and ice having their respective bulk phase properties, should be separated by a meniscus surface being submitted to a surface energy [11]. For such cases the meniscus radius or equally the hydraulic radius can be related to the temperature of fusion. Measurements on Vycor glass have been performed, indicating the value of the surface tension in the ice–water interface [12]. In this context, relevant information is also to be found in [13].

By using the surface tension in the curved ice–water interface and adopt it as a true material parameter, it is possible to evaluate the pore size distribution by using suitable additional assumptions together with measurements on the ice contents at different temperatures, e.g. see [14]. However, the measured hysteresis in ice content between freezing and thawing situations raises a number of questions regarding the relevance of the method. It can be argued that the development of the shape of ice bodies is markedly different during freezing and thawing, which should result in different active surface areas for these cases. The interpretation of the hysteresis in freezing curves during heating and cooling is discussed in [12].

Here the relation between measured length changes during freezing and thawing is compared to ice contents at corresponding temperatures using three different concrete qualities. Experimental results of this kind can be used to partly verify different hypotheses for ice growth and its damaging effect. Performing experiments directly devoted to verifying the discussed existing ice growth hypothesis are concluded to be very difficult and fall outside the scope of this work.

Each concrete quality, in this investigation, has been produced with three nominal contents of entrained air, 2%, 4% and 6%. The samples are conditioned to two different saturation levels, totally vacuum saturated and a saturation obtained by simple storage in water at normal pressures. The samples are analyzed for their ice contents during freezing and thawing using a scanning calorimetric technique, with a novel method to evaluate the raw data from the instrument, [15, 16]. The damage after and before exposure to freeze–thaw cycles is identified by using a non-destructive sonic measurement technique. Furthermore, length changes of samples are measured during the freeze/thaw cycles adopted. Methods have been presented on separate measurements of the kind used here or measurements of the length changes of samples when placed in a calorimetric instrument for the simultaneous registration of ice growth, e.g. see [10,17–19]. Besides the above-mentioned measurements, a picture analyze method was used on thin sections of the different concrete qualities. This method gives both the total air 'bubble' content and the size distribution of the same 'bubbles' in the material.

The results from the investigation show the importance of the air bubble content in concrete and the saturation degree of the tested samples. By studying the obtained ice content curves at freezing and thawing for the different samples and saturation degrees together with the comparison with their corresponding length changes, the overall performances of the different concretes can be obtained. Further, from the results presented, important information needed for the verification of different proposed mechanisms describing frost damages is provided.

Besides the quantitative examination of the different main hypothesis for the action of freeze and thaw induced damages based on different experimental results, the present paper also demonstrates the difficulties in accurately calculating the amount of ice formed at different freezing and thawing temperatures from considering data from the calorimetric approach. With the purpose to find a more exact method, the assumption of the calorimetric approach is described in detail and suggestions on complimenting experiments and general improvements are also discussed.

Accepting some inaccuracies in the determination of the ice amounts in the samples at the freezing and thawing situations studied, the presented results are ideally suited for the verification of mechanical models aiming at constituting the relation of ice concentrations in the pores of a material to the developed strains and stresses. Such analysis is, however, outside the scope of the presented investigation.

A special calorimetric test involving measurements down to about −145 °C has been included in this investigation. Using the proposed method for calculating the ice contents at different temperatures based on the calorimetric raw data, it was concluded that no ice was formed below about −50 °C.

2. Concrete recipes and preparation of samples

Nine different recipes were prepared for the investigation. Three different, water-to-cement ratio, concrete types were