



# Analysis of the development of cluster cracks caused by elevated temperatures in cement paste



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## HIGHLIGHTS

- Modified cement paste tested after exposure to elevated temperature.
- Image analysis technique was used to evaluate the surface structure of specimens.
- Cluster cracks development process was described using the dispersal system theory.
- Density of surface cracks structures depends on cement Blaine specific surface area.

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## ABSTRACT

This article describes a study of the possibility of using innovative stereological image analysis techniques to evaluate the structure of cluster cracks on the surface of modified cement pastes. Four series of specimens that were constructed with two different Portland cements were investigated. In addition, two of the series used nanosilica as a replacement for 10% of the cement content. Cracks of the samples were created by a sudden temperature load at 200 °C. Images of the cracked surfaces were obtained via scanning at 1200 DPI; additional digital processing and measurements were performed using ImageJ v. 1.46r software. To describe the structure of the cracks, two stereological parameters were proposed: the cluster average area (CAA) and the cluster average perimeter (CAP). The goal of this study was to compare the investigated stereological parameters with the compressive strength and tensile strength of the tested specimens. In addition, while considering the structure of the cement paste as a dispersion system, the formation of the cluster structures was shown to transform under external and internal loads and affect the final properties of the composite material.

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## 1. Introduction

The adverse effects of elevated temperatures on the structures investigated in this study typically exhibit a change in the physical and mechanical properties of the heated material and stress and strain in the structure. Transformations and reactions that occur inside the multiphase structure of cement paste cause its progressive degradation as temperatures increase. In this study, an analysis is performed on cement paste, which is a two-phase mixture of cement, water and certain additives. Water begins the cement's binding process, and as time progresses, the resulting cement paste hardens and creates a stable structure capable of transferring service loads [1].

The behaviour of the cement matrix at elevated temperatures is complex due to the chemical diversity of the matrix and the presence of multiple levels of structural heterogeneity [2]. Changes in the chemical composition and microstructure of the hardened cement paste appear gradually and continuously as temperatures increase from room temperature up to 1000 °C [3–5]. At first, as temperatures increase, moisture (i.e., free water) gradually evaporates. Total removal of free water occurs at 105 °C after a sufficiently long exposure to the elevated temperature has occurred. At 105 °C, water that is strongly chemically and physically bonded, including water contained in the products of hydration, begins to be removed from the cement matrix due to dehydration and destruction of various phases. Initially, at a temperature between 150 and 180 °C, the tobermorite gel loses thermal stability [5]. In the temperature range between 200 and 400 °C, slow and then fast growth of half-bonded water (i.e., capillary and gel water) donation begins. Portlandite decomposition ( $\text{Ca}(\text{OH})_2 \rightarrow \text{CaO} + \text{H}_2\text{O}$ ) begins

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to occur most significantly between 450 and 550 °C [6,7]. At a temperature of 600–700 °C, the decay process of the CSH phase and the formation of  $\beta$ -C2S and  $\beta$ -CS occur [8,9]; concurrently, the process of decarbonization of the cement binder begins ( $\text{CaCO}_3 \rightarrow \text{CaO} + \text{CO}_2$ ). The progressive degradation of hydrated cement crystals entails a drop in the coherence of the material, which results in the disintegration of the cement paste [10].

At macro and micro scales, the result of the processes described above is the formation of cracks on the material's surface and inside the material [11,12]. Degradation of cementitious material depends significantly on the temperature to which the element is exposed; however, heating kinetics also have a significant influence. The probability of creating surface structure defects is significantly increased in the case of a sudden heat load on the sample (i.e., thermal shock) than when the temperature increases gradually up to a certain value [1,13]. For concrete, cracks are primarily caused due to different thermal deformations in the aggregate and cement matrix and stresses arising from the temperature gradient between the outer and the inner surface of material [14,15]. Additionally, an increase in the water vapour pressure in the concrete's pores results in an increase in the local tensile strength of cement paste in its nano- and microstructures, which leads to the progressive degradation of the material.

In this study, the amount of cluster cracks on the surface of modified cement pastes with and without the addition of nanosilica was analysed. As described, these cracks result from the sudden exposure of the samples to a temperature of 200 °C. Evaluation of the surface structure degradation was performed using original and innovative methods of stereological image analysis. The goal of this article is to compare the measured parameters of the quantitative description of cracks with the mechanical parameters of the tested cement composites and to explain the process of surface crack structure organization, depending on the fragmentation and chemical composition of the cement used.

The addition of nanosilica results in improved mechanical properties (e.g., compressive and tensile strength) of cement-based composites that are exposed to elevated temperatures [16,17]. Quantitative stereological studies of the structure of cement composites at different structural levels performed to date in the literature have been limited to porosity measurements and the distribution of aggregate in the cement matrix [15,18–22].

## 2. Definition of a cluster and the cluster structure development model

A cluster is a collection of molecules that form common network connections. The individual properties of each molecule can

be transformed into the integral properties of the resulting compound. The clusters have shape, dimensions and properties [2]. At the macro scale, a cluster is considered to be the area on the specimen surface that is limited by cracks or by the edge of the sample. A sample image of a tested specimen with the cluster divisions is shown in Fig. 2b.

In this study, cement paste was treated as a dispersal system where water is used as the dispersing phase, and the cement is used as the dispersed phase. The interaction of surface forces in dispersal systems is associated with physical phenomena of uncompensated molecular forces on the phase boundaries. In the presence of water, capillary forces appear between the solid-phase particles. Then, nanofilms create molecules on the surrounding fluid that are characterized by a wetting parameter ( $l$ ), a surface curvature ( $r$ ) and a surface tension ( $\sigma$ ) on the phase boundaries. As a result of environmental and fluid pressure changes (i.e.,  $\Delta P$ , which is the Laplace pressure) and the appearance of a capillary force ( $F_K$ ), there is the possibility of calculating capillary force using Eq. (1). In dispersal systems, capillary forces resulting from physical phenomena (mainly a pressure gradient) primarily determine the material's physical and mechanical properties. A dispersal system model and cluster structure creation model are shown in Fig. 1. The phenomenon is discussed further in Fic's study [2]:

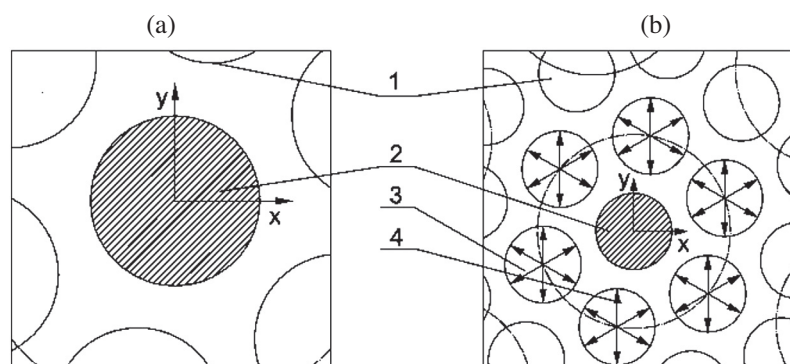
$$F_K = \Delta P S_0 \sigma l \quad (1)$$

where  $S_0$  describes the wetting surface.

Capillary forces produce mutual attractions between the molecules, which tend to form binding units, which are an  $n$ -set of molecules forming a cluster, where  $n$  is the number of particles; these binding units are typically of different sizes, where the binding unit is shown as a cluster at nano and micro scales. With an increase in the scale of observation, higher-level clusters are formed and shaped by clusters from lower levels. At the interface between clusters on the lowest possible level of structure heterogeneity, the separation surface between clusters forms cracks created in the initial period of the cement paste structure organization. As a result of an applied load, which exists in the form of elevated temperature, cement paste volume deformation occurs as a whole and in its individual phases at different levels of structural heterogeneity. This volume deformation causes the transformation of cracks into microcracks due to growth; these macrocracks then become visible on the surface of the element.

## 3. Materials and methods used

This study was conducted on four series of cement paste specimens. For each series, samples were made with three different water/binder ratios ( $w/b$ ) equal to 0.4, 0.5, and 0.6, where the binder is considered to be the total content of cement



**Fig. 1.** Two-dimensional model of a dispersion system of one (a) and many molecules forming the cluster structure (b): 1 – surrounding molecules; 2 – particles that form the structure; 3 – intermolecular interaction forces surrounding the centre; and 4 – interaction forces between the molecules that form the structure and surrounding structure [2].

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