Evaluation of Dependencies between Physico-Mechanical Properties and the Thermal Cracks' Geometry of Cement Pastes Modified with Metakaolinite Using the LSM Method

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Abstract. The paper presents the dependencies assessment that occur between physico-mechanical properties, and the parameters describing the geometry of thermal cracks of modified cement pastes. The subject of the research is cement paste modified with metakaolinite, which has been subjected to the influence of an elevated temperature. The pozzolanic additive was used as a substitute for 10% of the cement's mass. Four series of cement pastes were analyzed, which differed from each other with the class of Portland cement used and the metakaolinite content. Within each series, samples were made with 3 water/binder ratios equal to 0.4, 0.5, and 0.6, respectively. As part of the earlier research, the basic physical and mechanical features of the cement matrix were determined, such as: compressive strength, tensile strength at bending, and apparent density. The tests were carried out in accordance with EN standards on reference and thermally loaded samples. The elevated temperature load caused cracks on the surface of the cement matrix, which created a characteristic network of cracks referred to as the cluster cracks – the thermal cracks – the map cracking. The computer image analysis tools were used to quantify the cracks' structure. The samples' surfaces were scanned and the following parameters were determined: the cluster average area, the cluster average perimeter, and the crack average width. The aim of the study conducted was to determine whether on the basis of the measurement of the geometrical characteristics of thermal cracks it is possible to estimate with a good accuracy selected physico-mechanical properties of modified cement pastes. The measurement of geometrical features of material's cracks is a non-destructive and non-invasive method, in contrast to tests aimed at determining, in particular, the mechanical properties. The statistical analysis tool, i.e., the least squares method (LSM) was used to define dependencies that occur between material properties and the geometry of thermal cracks. The quality of matching the calculated functional dependencies to the empirical data was evaluated using three diagnostic statistics: the determination coefficient, the standard error of estimation, and the coefficient of random variation. The results obtained indicated the existence of very strong correlations between the compressive strength, apparent density, and the cracks' geometrical parameters. This allows an accurate estimation of these two material characteristics based on the analysis of the cement paste surface cracks, which has been subjected to elevated temperatures. The analyzes carried out are also of great practical importance because the results obtained can be used to assess the degree of degradation of a cementitious material that has been damaged due to a thermal interaction.
1. Introduction

Volumetric deformation of a cementitious material is one of the effects of an elevated temperature impact on its structure. As a result, the composite may crack and cracks combine or intersect through propagation creating a specific cracks' structure defined in the literature as the thermal cracks – the cluster cracks – the mapcracking [1-3]. The analysis of this cracks' structure seems to be important from a technological point of view, because the cracking of a cement matrix weakens its structure and increases the risk of penetration of harmful substances into the composite. This causes progressive degradation of the material, and in the case of a reinforced concrete, reinforcement corrosion may occur. On the other hand, the analysis of thermal cracks poses methodological challenges. Image analysis tools come in handy, which are used in concrete technology to the cracks evaluation. The study described so far in the literature show that only local parameters of cracks are analyzed, e.g., an opening width, crack length, etc. [4-7]. There is a shortage of works in which the structure of thermal cracks is analyzed in a global way, and the results obtained are related to the material properties. The areas formed on the sample's surface, which are limited on each side with a crack, or a crack and the sample's edge, are called the clusters [1, 8-10]. The previous research [11-15] has proved that image analysis is a very valuable tool in assessing the cluster cracks structure in cement matrix exposed to the elevated temperatures. An additional advantage of image analysis is the fact that it is a non-destructive test, the potential of which is still not fully discovered.

The aim of the study was to determine and evaluate dependencies that occur between the stereological parameters, which are describing the cluster cracks, and the physico-mechanical properties of a cement matrix. The classic cement matrix as well as modified one by the addition of a metakaolinite was evaluated. For this purpose, the statistical analysis tool in the form of the least squares method (LSM) was used. The quality of matching the calculated functional dependencies to the empirical data was verified using the diagnostic statistics, i.e., the coefficient of determination, the standard error of estimation, and the coefficient of random variation. In addition, the correlation strength between variables was determined by calculating the Pearson's correlation coefficient, which statistical significance was verified by the significance test of the correlation coefficient. It was determined which material properties of the cement matrix can be estimated with a high accuracy, based on the results obtained from non-destructive tests related to the measurement of geometrical properties of thermal cracks.

2. Materials and methods

2.1. Cement paste and its properties

The tests were carried out on 4 series of cement paste samples. Within each series, samples with three w/b ratios were tested – 0.4, 0.5, and 0.6. The use of the water/binder ratio was due to the fact that metakaolinite was used as a substitute for 10% of a cement's mass. The following series of cement pastes were evaluated:

- C42 – 100% CEM I 42.5R, water,
- C42MT – 90% CEM I 42.5R, 10% metakaolinite, water,
- C52 – 100% CEM I 52.5R, water,
- C52MT – 90% CEM I 52.5R, 10% metakaolinite, water.

Two Portland cements were used – the CEM I 42.5R and CEM I 52.5R, which have almost identical chemical composition, differing only in terms of the specific surface area. The detailed chemical composition of the cements was given in [11, 14]. Thus, the grinding degree of cement grains determines the class of the cement in this case. All samples were made as bars with dimensions of 40x40x160 mm. The specimens were made in accordance with the procedure described in EN 196-1 [16] and then matured in air-dry conditions (average temperature – 22 °C, average relative humidity – 50 %) for 28 days. After this period, the samples were loaded with an elevated temperature of 250 °C for 4 hours. The use of such a method of loading samples with the elevated temperature was aimed
at extracting the structure's defects in the form of cracks, without excessive deterioration of material properties. The cement matrix is thermally and chemically stable at 250 °C. The main factor causing the degradation of the material is the saturated water vapor pressure associated with the intensive evaporation of free water. This mechanism has been described in detail in [11, 14].

Then a set of basic physico-mechanical properties of the cement matrix was determined:

- the tensile strength \( f_{ct} \) using three-point bending, in accordance with EN 12390-5 [17], \( f_{ct(R)} \) – the reference samples, \( f_{ct(T)} \) – the samples after thermal load,
- the compressive strength \( f_{c} \), in accordance with EN 12390-3 [18], \( f_{c(R)} \) – the reference samples, \( f_{c(T)} \) – the samples after thermal load,
- the bulk density \( D \), in accordance with EN 12390-7 [19], \( D(R) \) – the reference samples, \( D(T) \) – the samples after thermal load.

In the case of \( f_{ct} \) and \( D \), the results obtained are the arithmetic mean of 6 samples, and in the case of \( f_{c} \) – 12.

In order to identify the surface structure of the cracked cement matrix (example image of the cracked surface is shown in figure 1), the samples' surfaces were scanned and subjected to the image analysis using the original procedure described in [11], the effect of which was the measurement of three stereological parameters describing quantitatively the thermal cracks:

- \( \bar{A} \) – the cluster average area – the parameter of \( R^{(2)} \) space,
- \( \bar{L} \) – the cluster average perimeter – the parameter of \( R^{(2)} \) space,
- \( \bar{I} \) – the crack average width – the parameter of \( R^{(1)} \) space.

The obtained results of mean values were summarized in table 1. They were discussed in detail and interpreted in [11], where the analysis of the results was carried out in the context of the description of the self-assembly processes of the cement matrix structure at the initial stage of the cement binding.

Table 1. The physico-mechanical properties of the modified cement pastes and the geometric characteristics of thermal cracks.

| Series | C42 | C42MT | C52 | C52MT |
|--------|-----|-------|-----|-------|
| w/b    | 0.4 | 0.5   | 0.6 | 0.4   | 0.5   | 0.6 | 0.4   | 0.5   | 0.6 |
| \( f_{c(R)} \) [MPa] | 61.4 | 43.4 | 34.2 | 67.2 | 48.3 | 36.9 | 69.5 | 49.7 | 39.1 |
| \( f_{c(T)} \) [MPa] | 5.80 | 4.35 | 3.16 | 6.51 | 4.76 | 3.37 | 5.74 | 4.39 | 3.86 |
| \( D(R) \) [g/cm³] | 1.692 | 1.528 | 1.416 | 1.682 | 1.484 | 1.372 | 1.750 | 1.574 | 1.432 |
| Reference samples – before thermal loading |
| \( f_{c(T)} \) [MPa] | 39.3 | 23.1 | 15.9 | 40.1 | 23.3 | 14.8 | 49.3 | 30.4 | 21.1 |
| \( f_{c(T)} \) [MPa] | 2.97 | 2.59 | 1.32 | 2.88 | 2.31 | 1.35 | 1.35 | 1.35 | 1.16 |
| \( D(T) \) [g/cm³] | 1.513 | 1.376 | 1.277 | 1.514 | 1.325 | 1.238 | 1.549 | 1.388 | 1.266 |
| Samples after thermal loading |
| \( \bar{A} \) [mm²] | 97.64 | 61.55 | 85.32 | 118.2 | 84.12 | 95.53 | 119.5 | 96.35 | 107.15 |
| \( \bar{L} \) [mm] | 43.09 | 51.15 | 68.32 | 46.12 | 57.04 | 81.93 | 34.87 | 41.24 | 58.32 |
| \( \bar{I} \) [mm] | 0.039 | 0.045 | 0.056 | 0.038 | 0.050 | 0.060 | 0.041 | 0.048 | 0.079 |
2.2. The Least Squares Method (LSM)

As a part of the study the equations of curves were calculated, which describe the relationships that occur between the stereological parameters and the physico-mechanical properties of the material. The dependencies developed relate to the condition of the material after the thermal loading. In addition, previous research has proved that between $\bar{A}$ and $L$ there is a very strong correlation [11, 14, 15], which causes that the geometric dependence of the resulting clusters is unchanged regardless of the technological variables in the process of manufacturing of modified cement pastes. Thus, the considerations being the subject of this work were limited to the description of the relationships between one of the parameters of the $R^2$ space ($\bar{A}$ in this case) and the physico-mechanical properties.

The following relationships were developed: $f_{c_{c(T)}}(\bar{A})$, $f_{c_{f(T)}}(\bar{I})$, $f_{c_{f(T)}}(\bar{A})$, $f_{c_{f(T)}}(\bar{I})$, $D_{(T)}(\bar{A})$, and $D_{(T)}(\bar{I})$. For this purpose, the LSM was used [20, 21]. The method is based on selection of a curve, for which the sum of distances’ squares of all empirical points from the respective points on the curve is minimal. It formally can be written as:

$$\sum_{i=1}^{n} (y_i - \hat{y}_i)^2 = \sum_{i=1}^{n} e_i^2 = \text{min}$$  \hspace{1cm} (1)

where:

$y_i$ – the actual value of the dependent variable (the explained),
$\hat{y}_i$ – the predicted value of the dependent variable (the explained),
$e_i = y_i - \hat{y}_i$ – the rest of the model.

Then to evaluate the curve fit to the empirical data a number of diagnostic statistics were used, including:

- the coefficient of determination – $R^2$:

$$R^2 = 1 - \frac{\sum_{i=1}^{n} (y_i - \hat{y}_i)^2}{\sum_{i=1}^{n} (y_i - \bar{y})^2}$$  \hspace{1cm} (2)

where: $\bar{y}$ – the average value of the dependent variable (the explained),

- the standard error of estimation – $S_e$:

$$S_e = \sqrt{\frac{\sum_{i=1}^{n} e_i^2}{n - k - 1}}$$  \hspace{1cm} (3)

where: $n$ – the number of observations,
$k$ – the number of explanatory variables,

- the coefficient of random variation – $W$:

$$W = \frac{S_e}{\bar{y}} \times 100\%$$  \hspace{1cm} (4)
In addition, the values of the Pearson’s correlation coefficients ρ were calculated to assess the correlations. In addition, the significance test of correlation coefficient was performed. The zero hypothesis \( H_0: \rho = 0 \) is tested against the alternative \( H_1 \) hypothesis: \( \rho \neq 0 \). On the basis of the test statistic, the \( p\)-value was determined and compared with the significance level \( \alpha = 0.05 \). An important test result (in the case when \( p\)-value \( \leq \alpha \)) indicates the rejection of the \( H_0 \) hypothesis and the adoption of the \( H_1 \), i.e., the correlation tested is statistically significant. All calculations were carried out in the Microsoft Excel software.

3. Results and discussions

In figure 2 – figure 4 the following relations are shown: \( f_{cT}(\bar{A}) \), \( f_{cT}(\bar{I}) \), \( f_{cfT}(\bar{A}) \), \( f_{cfT}(\bar{I}) \), \( D_{T}(\bar{A}) \), and \( D_{T}(\bar{I}) \). It was noted that the nature of the occurrences is best reflected by the power function. The highest matching degree of the curves calculated to the empirical data was obtained in a situation, in which the results were divided into the series (correlations for individual series were stronger, compared to \( \rho \) in global terms). The equations calculated of the model curves, along with the values of the diagnostic statistics and the correlation coefficients are shown in table 2. Negative values of the correlation coefficients indicate that the physico-mechanical parameters examined decrease in value with the increase of \( \bar{A} \) and \( \bar{I} \). Thus, the cement matrix is able to carry a larger load, the more clusters are formed on its surface, but it should be remembered that this is accompanied by a drop in the crack width.

Table 2. The equations of curves that describe the dependencies between the physico-mechanical properties and the stereological parameters with the values of diagnostic statistics (\( R^2 \), \( S_c \), \( W \)), correlation coefficients (\( \rho \)), and the results of its significance test.

| Relationship | Series | Equation | \( R^2 \) | \( S_c \) [MPa] | \( W \) [%] | \( \rho \) [-] | \( p\)-value (the \( \rho \) significance test) |
|--------------|--------|----------|----------|----------------|-----------|----------|----------------------------------|
| \( f_{cT}(\bar{A}) \) | C42    | \( y=2332x^{-0.915} \) | 0.82     | 4.52          | 17.42       | -0.80    | 0.002                             |
|               | C42MT  | \( y=2157x^{-0.852} \) | 0.90     | 3.60          | 13.78       | -0.88    | <0.001                            |
|               | C52    | \( y=1362x^{-0.815} \) | 0.84     | 5.30          | 15.69       | -0.78    | 0.003                             |
|               | C52MT  | \( y=2537x^{-0.882} \) | 0.91     | 4.20          | 11.65       | -0.89    | 0.001                             |
| \( f_{cT}(\bar{I}) \) | C42    | \( y=0.253x^{-1.485} \) | 0.60     | 6.63          | 25.56       | -0.72    | 0.008                             |
|               | C42MT  | \( y=0.411x^{-1.347} \) | 0.67     | 6.65          | 25.46       | -0.67    | <0.001                            |
|               | C52    | \( y=0.865x^{-1.32} \)  | 0.79     | 6.05          | 17.89       | -0.83    | 0.001                             |
|               | C52MT  | \( y=0.558x^{-1.335} \) | 0.76     | 6.42          | 19.06       | -0.86    | <0.001                            |
| \( f_{cfT}(\bar{A}) \) | C42    | \( y=90.78x^{-0.744} \) | 0.85     | 0.31          | 13.58       | -0.91    | <0.001                            |
|               | C42MT  | \( y=56.95x^{-0.628} \) | 0.98     | 0.10          | 4.81        | -0.98    | <0.001                            |
|               | C52    | \( y=5.45x^{-0.308} \)  | 0.81     | 0.09          | 7.08        | -0.81    | 0.001                             |
|               | C52MT  | \( y=14.58x^{-0.470} \) | 0.78     | 0.16          | 10.57       | -0.78    | 0.003                             |
| \( f_{cfT}(\bar{I}) \) | C42    | \( y=0.047x^{-1.252} \) | 0.63     | 0.49          | 21.18       | -0.77    | 0.003                             |
|               | C42MT  | \( y=0.101x^{-0.999} \) | 0.69     | 0.40          | 18.44       | -0.82    | <0.001                            |
|               | C52    | \( y=0.318x^{-0.485} \)  | 0.80     | 0.10          | 7.40        | -0.84    | 0.001                             |
|               | C52MT  | \( y=0.130x^{-0.783} \) | 0.75     | 0.17          | 11.28       | -0.81    | 0.001                             |
| \( D_{T}(\bar{A}) \) | C42    | \( y=2.983x^{-0.134} \) | 0.80     | 0.048         | 3.46        | -0.83    | 0.001                             |
|               | C42MT  | \( y=3.172x^{-0.161} \) | 0.85     | 0.049         | 3.58        | -0.86    | <0.001                            |
|               | C52    | \( y=3.008x^{-0.166} \)  | 0.82     | 0.053         | 3.80        | -0.83    | 0.001                             |
|               | C52MT  | \( y=3.687x^{-0.201} \) | 0.90     | 0.043         | 3.10        | -0.90    | <0.001                            |
| \( D_{T}(\bar{I}) \) | C42    | \( y=0.635x^{-0.253} \) | 0.55     | 0.071         | 5.10        | -0.72    | 0.009                             |
|               | C42MT  | \( y=0.584x^{-0.277} \) | 0.64     | 0.076         | 5.58        | -0.75    | <0.001                            |
|               | C52    | \( y=0.650x^{-0.262} \)  | 0.81     | 0.056         | 3.97        | -0.74    | <0.001                            |
|               | C52MT  | \( y=0.517x^{-0.318} \) | 0.75     | 0.067         | 4.82        | -0.86    | <0.001                            |
In global terms, the strongest correlation occurs for the dependence $f_{c(T)}(\bar{A})$ and $D_{c(T)}(\bar{A})$; $\rho$ is equal to -0.78 and -0.79. The weakest global correlation was observed for $f_{c(T)}(\bar{A})$ and $f_{c(T)}(\bar{I})$, but in case of the analysis of relationships divided into series, the individual correlation coefficients for $f_{c(T)}(\bar{A})$ assume high values (from -0.81 to -0.98). The -0.98 result achieved for the C42MT shows an almost complete correlation between $f_{c(T)}$ and $\bar{A}$. It is worth noting that in each case the significance test of the correlation coefficient reached $p$-value below the adopted significance level $\alpha = 0.05$, which means that each correlation calculated is statistically significant. In addition, it was noticed that after exchanging 10% of the cement's mass with metakaolinite, the relationships obtained are characterized by stronger correlation. For the C42MT and C52MT, the $\rho$ values oscillate between -0.75 and -0.98 and are in most cases higher than for the C42 and C52 (from -0.72 to -0.91).

Figure 2. Relationship between $f_{c(T)}$ and: a) $\bar{A}$; b) $\bar{I}$

Figure 3. Relationship between $f_{c(T)}$, and: a) $\bar{A}$; b) $\bar{I}$
The functional curves are best reflected by the empirical data when the material feature depends on \( \bar{A} \). For the dependencies \( f_{c(T)}(\bar{A}) \), \( f_{ef(T)}(\bar{A}) \), and \( D(T) (\bar{A}) \) the \( R^2 \) parameter reached values in the range from 0.78 to 0.98, which indicates very good model matching. In the case when the \( \bar{I} \) is a variable a slightly worse degree of curve fit to the empirical data was observed – \( R^2 \) from 0.55 to 0.81. In addition, the lowest \( W \) values (<6%) obtained the dependencies \( D(T) (\bar{A}) \) and \( D(T) (\bar{I}) \), which allows to estimate with a great accuracy the apparent density of the cement pastes based on both \( \bar{A} \) and \( \bar{I} \).

In the case of \( f_{ef(T)}(\bar{I}) \) a clear influence of the cement's class on the results of diagnostic statistics was observed. Samples made of CEM I 52.5R obtained slightly higher values of \( R^2 \) than samples made of CEM I 42.5R. The \( S_e \) values for the C52 and C52MT series were about 3-5 times smaller compared to C42 and C42MT, and the \( W \) values were about 2-3 times smaller. This means that the estimation of the tensile strength on the basis of \( \bar{I} \) for samples made of cement with a higher grain size distribution will be more accurate. For the remaining relationships, the impact of the cement's class is small. For the majority of cases, it was noticed that the total effect of assessing the quality of the curve fitting to the measurement data gives slightly better results for the metakaolinite modified samples (C42MT and C52MT) – higher \( R^2 \), lower \( S_e \) and \( W \), compared to C42 and C52.

4. Conclusions
The paper presents the results of the analysis, the aim of which was to assess the dependencies that occur between the geometrical properties of the cement pastes thermal cracks and selected physico-mechanical parameters. On the basis of analyzed and interpreted research results, final conclusions were formulated:

- As the values of the geometric characteristics of the clusters increases, the values of \( f_{c(T)}(\bar{A}) \), \( f_{ef(T)}(\bar{A}) \), and \( D(T) (\bar{A}) \) decrease.
- The curves calculated are characterized by the best degree of matching to the empirical data in the case when dependencies are considered in the criterion of the results division into the series.
- The strongest correlation is characterized by the relations \( f_{c(T)}(\bar{A}) \) and \( D(T) (\bar{A}) \). For the relationships, high values of the determination coefficient were obtained (from -0.78 to -0.90). On the other hand, very low values of \( S_e \) and \( W \) (<4%) obtained for \( D(T) (\bar{A}) \) indicate that the estimation of the bulk density based on the average cluster area will be characterized by the smallest error of all dependencies examined.

![Figure 4. Relationship between \( D(T) \) and: a) \( \bar{A} \); b) \( \bar{I} \)]
• The average cluster area is a parameter for which the relationships obtained are characterized by a stronger correlations and more favourable values of the diagnostic statistics in comparison to the dependencies obtained with the crack average width.

• It was not noticed that the cement's class influenced the quality of the dependencies obtained, with the one exception – for $f_{\sigma(T)}(I)$ the matching quality of the curves calculated to the empirical data differed significantly depending on the cement's class.

• Replacing 10% of the cement's mass with metakaolinite causes a slight increase in the correlation coefficient value, and the functional curves slightly better reflect the empirical data. In particular, for $f_{\sigma(T)}(A)$ and C42MT series, the correlation coefficient reached the value equal to -0.98, which indicates the almost complete inverse correlation.

• The big advantage of the image analysis is the fact that it is a non-destructive examination. The conducted research has shown that knowing the geometrical characteristics of surface cracks and a material's composition, it is possible to estimate the selected physico-mechanical properties with great accuracy, which is of great practical importance. This may improve the process of assessing the degradation degree of cementitious material that has been damaged due to excessive thermal load.

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