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Communication

Depth-sensing indentation method for evaluation of efficiency of secondary cementitious materials

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Abstract

An application of a depth-sensing indentation (DSI) system to characterize properties of hardened cement paste is described. The test method developed, based on the microhardness testing concept, was implemented on a universal testing frame. Preliminary experiments were dedicated to evaluate efficiency of mineral additives in cement paste from Vickers hardness data. A suitable method of specimen preparation for microhardness tests was elaborated and successfully applied. The investigation revealed a clearly linear correlation between Vickers hardness and water-to-cement (w/c) ratio. An evaluation of the efficiency of mineral additives in cement paste was possible assuming a criterion of equal Vickers hardness. The DSI test method seems to be a valuable method for studying the bulk properties of cementitious composites.

Keywords: Microstructure; Cement paste; Metakaolin; Micromechanics; Water-to-cement ratio

1. Introduction

Rational use of secondary cementitious materials in concrete requires an adequate modification of the definition of water-to-cement (w/c) ratio. This is especially important for new natural materials or by-products of new processes intended to be introduced as cement or concrete additives. The approach assumed in European Standard EN 206-1 is based on the efficiency factor k used to calculate an equivalent w/c ratio in the following formula: $w/(c+ka)$, where w, c and a denote the mass of water, cement and additive, respectively. Such an approach seems to be practical although one should be aware of its various limitations [1]. The k values given in EN 206-1 concern only silica fume and fly ash conforming to prEN 13263 and EN 450, respectively.

In order to use new mineral additives or their blends in concrete, extensive strength and durability testing is required. A novel concept to reduce such testing effort has been introduced at the Institute of Fundamental Technological Research in Warsaw by application of the depth-sensing indentation (DSI) testing method. The method is based on the microhardness testing concept, which is common for evaluation of the quality of engineering materials, particularly metals. A preliminary application of the DSI method for testing of hardened cement paste is presented in this paper.

2. Concept of microhardness testing

Microhardness testing is based on forcing an indenter into the surface of the material and determining the response in terms of size of indentation. Over 40 years ago, Lyubimova and coworkers proposed a microhardness test to investigate certain concrete properties, in particular compressive strength as well as interfacial transition zone between cement paste and aggregate [2,3]. Characterization of the microstructure and strength of cement paste by microhardness testing, as well as the application of microhardness to cement paste and concrete was discussed by Igarashi et al. [4,5]. Currently, microindentation methods are used to characterize gradients in mechanical properties of materials; by application of nanoindentation methods the elastic modulus and the hardness of pure constituents of Portland cement clinker are determined [6].

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Microhardness testing as specified, for instance, in ASTM E 384 does not refer to any specific materials, so such test methods can be applied to cement-based materials. The Vickers test is commonly used for both hardness and microhardness testing in which the indenter has a diamond shape (square-based pyramid; Fig. 1). The hardness value is defined as the ratio between the load and the contact area of the indentation in units of stress. The Vickers hardness (HV) in MPa units is calculated as [Eq. (1)]:

$$HV = \frac{2P\sin(\varphi/2)}{D^2} = 1.8544\frac{P}{D^2} \quad (1)$$

where P = load (N), D = mean diagonal of indentation (mm) and $\varphi = 136°$.

Since in the case of the Vickers indenter the angle $\varphi = 136°$, the diagonal D is

$$D \cong 7.0006h \quad (2)$$

In elastic–plastic materials the shape of the indentation is regular and quite easy to measure, contrary to the case of brittle materials, which exhibit various shapes of indentation dependent on the load level [7]. In such cases, a direct measurement of the diagonal of indentation and calculation of Vickers hardness is not possible. Therefore, to calculate an HV value for such materials the DSI method can be applied.

As is known from Ref. [8], in the range of small loads the hardness is dependent on the load level applied, and for common metals it decreases with an increase in load, as shown schematically in Fig. 2. It is argued [5] that the load variation of hardness, which is called the indentation size effect, can be a valuable tool to provide information sensitive to the microstructure of the material. Indeed, since the size of indentation is dependent on the load level, the volume affected in the test should be considered in comparison to the size of microstructural elements of the composite material.

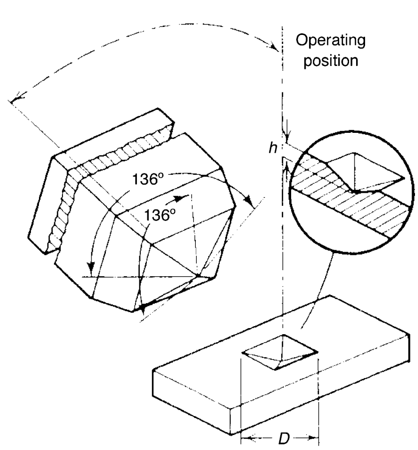

Fig. 1. Vickers indenter and the shape of indentation, D = diagonal of indentation, h = depth of indentation.

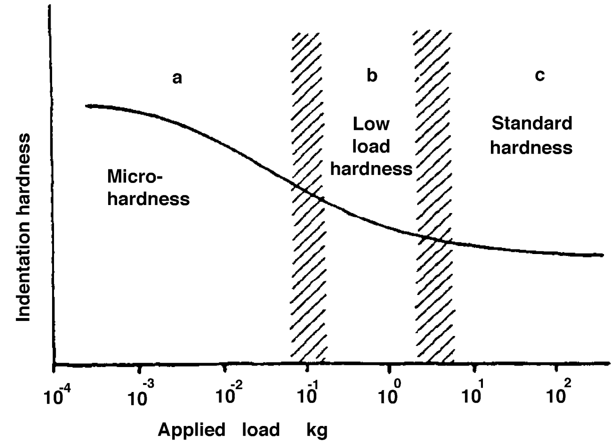

Fig. 2. Schematic representation of "indentation size effect" according to Ref. [8].

3. DSI test on cement paste

3.1. Test setup

The mechanical loading system used for DSI testing involves pushing an indenter down into the specimen while measuring force and displacement continuously. Such a testing technique was set using a Lloyd EZ 50 testing machine suitably equipped to hold the specimen and to perform proper measurements. Load measurements were taken using a high-precision load cell (the capacity of 50 N at an accuracy better then 0.5% of applied load). With a properly installed LVDT transducer it was possible to measure the displacement of the indenter (h) in tested specimens with an accuracy of 0.1 μm. The specimen-holding fixture was installed on a movable stage with a manual movement control with an accuracy of 0.001 mm. A standard Vickers indenter was applied. The measuring system and data acquisition were digitally controlled using Ondio-Nexygen software, which also enabled data postprocessing.

From measured values of load and displacement of the indenter the Vickers hardness of the tested material can be calculated, assuming a constant shape of indentation due to the known geometry of the Vickers indenter (Fig. 1). In Fig. 3, an image of indentation performed by means of the Vickers indenter in the cement paste is shown.

3.2. Materials and specimens

Materials used included hardened pure cement pastes and cement pastes with an addition of high-reactivity metakaolin used as a partial replacement of cement at equal weight. A series of cement pastes with w/c ratio in the range from 0.3

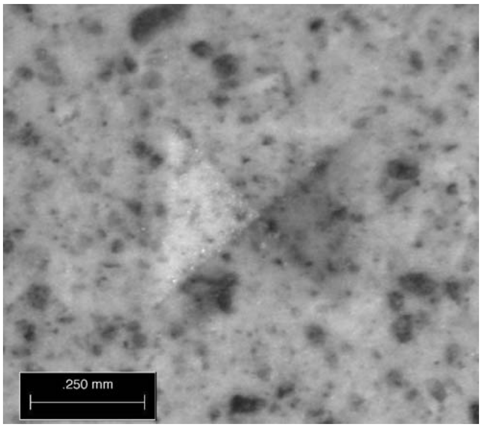

Fig. 3. An example of Vickers indentation in cement paste with 0.65 w/c ratio.

to 0.7 with steps of 0.05 was prepared. The second series of specimens was obtained using a 20% replacement of cement by high-reactivity metakaolin at variable water contents. The cement used was Portland cement CEM I 32.5 conforming to EN 197-1, and metakaolin was a commercially available product. Small beam specimens of 40×40×160 mm were cast and cured continuously at 18–20 °C in high-humidity conditions. At the age of 28 days, a slice was cut from each beam using a diamond wheel saw. The surface of the specimen was polished with silicon powders (#320, #600 and #1200) to obtain the quality adequate for microscopic investigation.

3.3. Preliminary DSI test results

The DSI test was performed at three selected levels of load: 10, 20 and 40 N. Each test result was an average of 10 measurements made on the surface of the specimen. An example of a load–displacement diagram obtained during DSI testing is presented in Fig. 4. Marked points E1 and E2 point out, respectively, the beginning and the end of indenter travel in the tested material. The mean diagonal of the indentation measured using an optical microscope corresponded well with the diagonal calculated using Eq. (2). On the basis of the indenter displacement in the tested material and at the established load the Vickers hardness value can be determined.

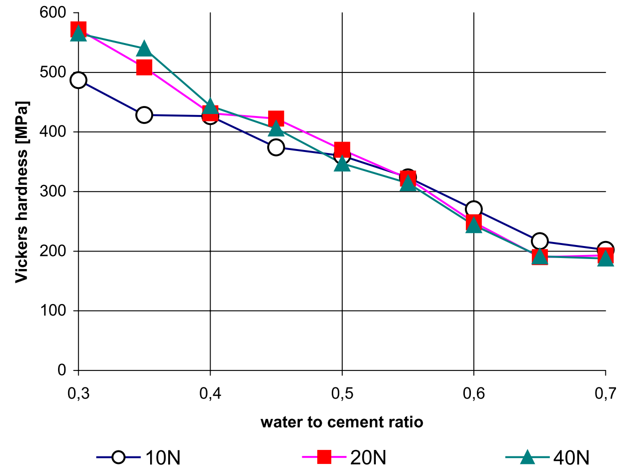

Fig. 5. Vickers hardness versus w/c ratio at different load levels (pure cement paste specimens).

As a result of this research an accurate linear relationship between w/c ratio and Vickers hardness was found. Assuming the nomenclature presented in Fig. 2, it can actually be called the low-load hardness. Results of the investigation are presented in Figs. 5 and 6. Each point represents the mean value of 10 measurements. The best linear correlation coefficient was obtained at the load of 40 N. It is evident

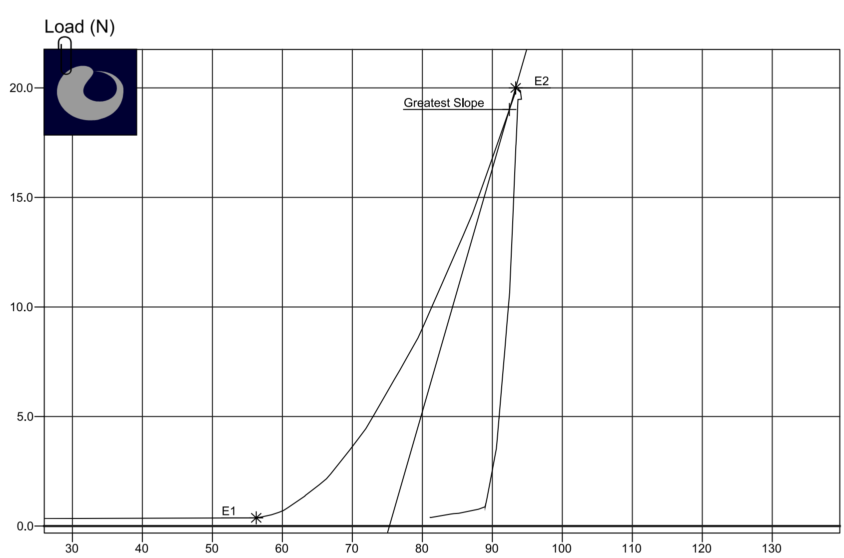

Fig. 4. Load versus indenter displacement diagram obtained during DSI testing on hardened cement paste.

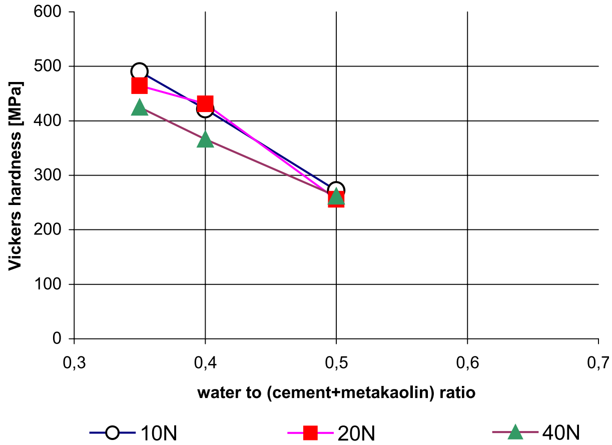

Fig. 6. Vickers hardness versus water-to-(cement + metakaolin) ratio at different load levels (metakaolin-modified cement paste specimens).

the Vickers hardness of cement paste increases with decreasing w/c ratio.

A similarly evident linear relationship between Vickers hardness and water content was found for metakaolin-modified cement pastes (Fig. 6). There was only a minor dependency of the Vickers hardness value on the applied load level.

Evaluation of the efficiency of a mineral additive in cement paste (here, high-reactivity metakaolin) is possible assuming a criterion of efficiency as, e.g., equal Vickers hardness that is shown to be related to compressive strength of cement paste (see Ref. 8 in Ref. [5]). For example, at an assumed indentation load of 20 N at an equal level of Vickers hardness, a calculated value for the efficiency factor k is obtained as follows: for HV = 480 MPa, from Fig. 5, $w_0/c_0 = 0.38$ and from Fig. 6, $w/c_0 = 0.35$.

Water content in pure cement paste and metakaolin-modified pastes are w_0 and w, respectively.

Thus, $w = 0.923w_0$ and

$$\frac{w_0}{c_0} = \frac{w}{(c + ka)} = \frac{0.923w_0}{c_0(0.8 + 0.2k)} \Rightarrow$$

$$0.8 + 0.2k = 0.923 \Rightarrow k = 0.61 \quad (3)$$

The results for this example of high-reactivity metakaolin is obtained for 20% replacement of cement—a rate of replacement far higher than commonly applied. At a constant proportion of metakaolin to cement, the estimated k coefficient is dependent on the water content in the paste. A similar calculation procedure can be established assuming a criterion of an equal indentation toughness defined by the area under the load–displacement curve. Such extensive analytical tools are only available using DSI-type testing and cannot be reached using a standard microhardness testing method. A new valuable research tool is available and it opens very interesting possibilities of assessment of local properties of cement-based materials, including efficiency evaluation of secondary cementitious materials and their blends.

4. Conclusions

1. The proposed DSI test system was mounted on a standard universal testing frame to perform the Vickers hardness test along with continuous measurements of load and displacement; it was successfully used instead of very sophisticated and expensive equipment.
2. The procedure for adequate specimen preparation for DSI tests was very similar to the surface preparation technique commonly used for preparation of specimens for microscopic determination of air-void structures.
3. The tests revealed a clearly linear correlation between Vickers hardness and w/c ratio of hardened cement pastes, both pure and modified with the addition of high-reactivity metakaolin.
4. The proposed method of determining the efficiency of secondary cementitious materials was based on the criterion of an equal Vickers hardness at a selected load level. An extension of the method using the criterion of an equal maximum indentation stiffness or an equal indentation toughness is readily available using DSI-type testing.

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