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Rupture Strength and Irregularity of Fracture Surfaces

Tomáš Ficker [1]

[1] Brno University of Technology, Faculty of Civil Engineering, Veveří 95, CZ-602 00 Brno, Czech Republic

ficker.t@fce.vutbr.cz

Abstract. Textural irregularities of fracture surfaces of cement-based materials seem to be an interesting source of information on some mechanical properties. Besides compressive strength, the flexural strength is strongly correlated with height irregularities (i.e. roughness) of fracture surfaces of hydrated cement pastes. This correlation has been a subject of experimental study. An analytical relation between flexural strength and height irregularities has been inferred. The formula contains height parameters, which represent basic descriptors of surface irregularities of fracture surfaces of cement pastes. These irregularities are governed by the capillary porosity of cement pastes with different water-to-cement ratios. The relation yields values that are in agreement with the empirical formula published in the technical literature.

1. Introduction

There is no doubt that the topology analysis of fracture surfaces is capable of providing valuable information on mechanical properties of fatigue failed materials. Besides other materials, it is the widely used cementitious materials that are the subject of fractographical research. One of the so far not resolved question concerns the possible existence of the relation between flexural strength and roughness of fracture surfaces of these materials. The research is preferably focused on cement pastes as the basic components of cement-based materials.

In the previous few years, great effort has been devoted to the research of textural irregularities of fracture surfaces [1 - 5]. It was shown that there is a close correlation between height irregularities of fracture surfaces and water-to-cement ratio r of hydrated cement pastes [3]:

$$H(r) = H_o + h_o \cdot \exp\left(\frac{r}{\alpha_o}\right) \tag{1}$$

Since the water-to-cement ratio r of cement pastes is a main controlling factor of both the height irregularities $H(r)$ and compressive strength $\sigma_c(r)$, a close correlation has also been found between compressive strength and surface height irregularities $\sigma_c(H)$ [3, 4]. Under the term 'height irregularity' (H), the root-mean-squared height of surface profile $f(x,y)$ is understood

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$$H = \sqrt{\frac{1}{L \cdot M} \iint_{(LM)} [f(x,y)]^2 \, dx \, dy} \tag{2}$$

where $L \times M$ is the area of the vertical projection of the surface profile $f(x,y)$ into the horizontal plane xy.

Surface morphology has also been characterised by means of fractal dimension D. However, it was shown that the three-dimensional height parameters H were more convenient for the description of surface irregularities, compared to fractal dimension D [5].

All the aforementioned experimental studies have verified that there is a strong correlation between compressive strength σ_c and height irregularities of fracture surfaces. The *experimental dependence* $\sigma_c(H)$ has been explored and illustrated with a large series of species, amounting to more than 100 items. A theoretical basis has also been recently published for the functional dependence $\sigma_c(H)$ [3]:

$$\sigma_c(H) = \sigma_{oc} \left(\frac{h_o}{H - H_o} \right)^{\rho_c} \tag{3}$$

That study accomplished a major part of the research that was focused on the morphology of fracture surfaces in connection with *compressive strength* but it did not solve the problem of flexural *strength* and some related complementary questions. These questions are as follows:

(i) Does the flexural (bending) strength σ_b correlate with the surface irregularities H of fracture surfaces, similar to compressive strength?
(ii) What form of the functional dependence $\sigma_b(H)$ can be expected if such a correlation exists?
(iii) If the dependence $\sigma_b(H)$ exists, does it fulfil some of the known empirical relations between compressive and flexural strengths?

The goal of this study is to find answers to these questions, support them by experimental evidence and *formulate the functional dependence* $\sigma_b(H)$.

2. Experimental arrangement

2.1. Preparation of specimens

120 specimens (3 cm × 3 cm × 10 cm) of hydrated ordinary Portland cement pastes of six water-to-cement ratios r (0.3, 0.4, 0.5, 0.6, 0.7, and 0.8) were prepared (20 samples per r-value). The specimens were rotated during hydration to achieve better homogeneity. All specimens were stored for three years (including the starting period of hydration) at $99.9 \pm 0.1\,\%$ RH, $20 \pm 2\,°C$ and at usual atmospheric pressure. The higher age of the specimens was set intentionally to test a thoroughly matured material. The specimens were fractured in three-point bending tests and one-half of the fracture surfaces was used for microscopic analyses. The other parts of the fractured specimens were processed into regular small cubes (3 cm x 3 cm x 3 cm) in order that their compressive strength might be determined by destructive tests. Bending as well as compressive tests were carried out on the precise electromechanical press WDW-200D controlled by the computer. The press was produced by the Jinan Precision Testing Equipment co., LTD [6].

2.2. Microscopic processing
The microscopic analyses were performed by the confocal microscope Olympus Lext 3100. Approximately 200 image sections were taken for each measured surface site (four sites for each sample) starting from the very bottom of the surface depressions (i.e. valleys) to the very top of the surface protrusions (i.e. peaks). The areas $L \times M = 640\,\mu m \times 640\,\mu m$ (1024 pixels x 1024 pixels, i.e. 0.625 $\mu m/\text{pixel}$) were investigated for all the 120 specimens. Each measurement was performed for the magnification 20x. Since each site measurement consisted of about 200 optical sections (digital files) with vertical steps of 0.64 μm , 96000 files were formed altogether, from which 480 three-dimensional digital reliefs $f(x, y)$ were created. The digital reliefs of the fracture surfaces were then subjected to 3D height analyses to obtain averaged *H-values* for each group of samples with particular *r-values.* At first sight it was obvious that species of different water-to-cement ratios r showed different stages of surface irregularities (surface roughness), which is illustrated in figure 1 for two groups of species, i.e. for samples with $r = 0.3$ and $r = 0.8$.

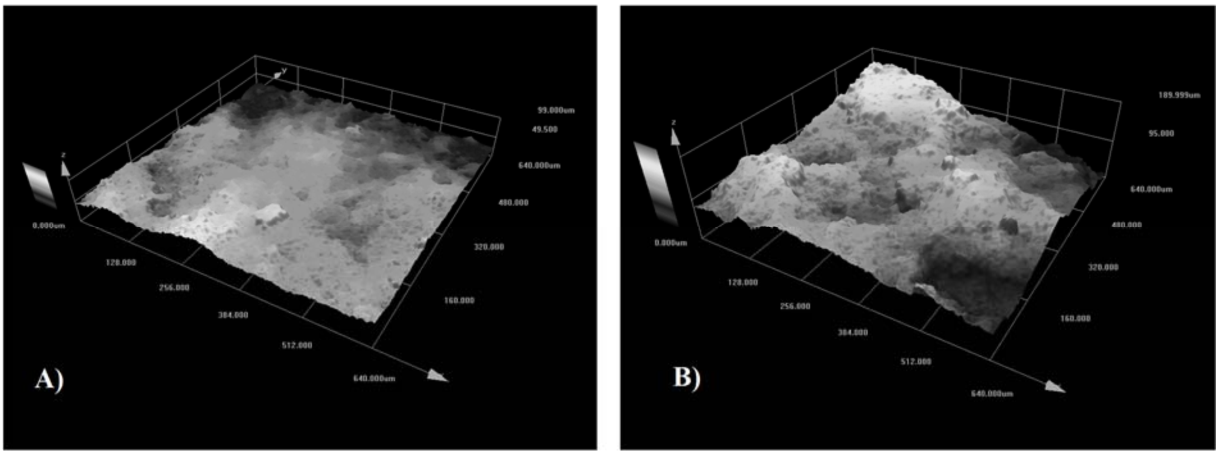

Figure 1. Confocal reconstructions of two fracture surfaces of hydrated cement pastes: A) Surface with smooth texture ($r = 0.3$); B) Surface with rough texture ($r = 0.8$)

3. Results and discussions

3.1 Functional behaviour of the tested relations.
The strong functional dependence of surface irregularities on water-to-cement ratio $H(r)$ investigated on the length scales of several tenths of micrometres demonstrates that capillary porosity is the underlying factor causing height irregularities. As is well-known, the values of water-to-cement ratio r determine the values of capillary porosity of hydrated cement pastes, whereas gel porosity is influenced only weakly. In addition, the diameters of gel pores are associated with nanometric scales, and thus optical microscopes are not capable of discerning them. For these reasons, the dependence $H(r)$ is preferably determined by the capillary structure that is projected on the fracture surfaces.

When inspecting the analytical form of the dependence $H(r)$ presented by equation (1), it is obvious that the height irregularities are described by the three parameters H_o , h_o and α_o . The first H_o determines the basic height level of the surface irregularities, whereas the second h_o represents a complementary height irregularity. The third parameter α_o is related to r and influences the 'rate' $dH(r)/dr$ with which the height irregularities $H(r)$ change their values when varying r , i.e.

$dH(r)/dr = (h_o/\alpha_o)\exp(r/\alpha_o)$. The function $H(r)$ is defined in the whole interval of reasonable values of r, i.e. starting from about $r_{\min} \approx 0.3$, when cement paste may still reach full hydration, up to the very high ratio of $r_{\max} \approx 0.8$, which is however out of common cement practice. In its domain $r \in (0.3, 0.8)$ the function $H(r)$ follows a monotonically increasing pattern (see figure 2).

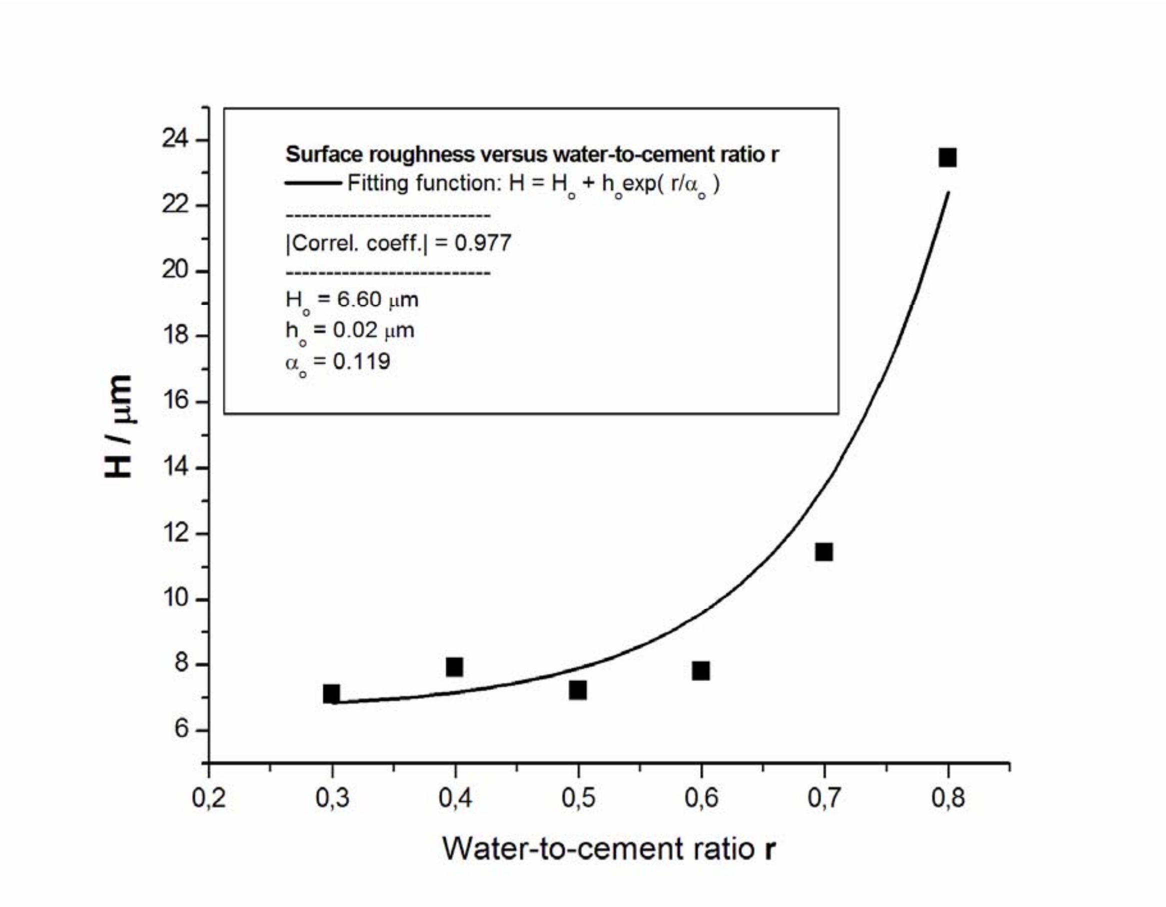

Figure 2. Surface irregularity H as a function of water-to-cement ratio r

The parameters H_o and h_o represent two *basic height descriptors* of the particular surface irregularity, which will be also explicitly included in the formula of compressive strength $\sigma_c(H)$, as can be checked with equation (3), while the parameter α_o will be included only implicitly within the exponent ρ_c. It should be expected that parameters H_o and h_o included in the formulae $H(r)$ and $\sigma_c(H)$ will have almost identical values.

3.2 Fitting procedures.
In order to verify that the *basic height descriptors* H_o and h_o in formulae (1) and (3) will assume almost identical values, the *independent fitting procedures* were carried out with both the measured dependences $H(r)$ and $\sigma_c(H)$. The nonlinear regression method based on Levenberg-Marquardt algorithm [8] has been used. As seen, the graph $H(r)$ shown in figure 2 is accompanied by the fitted values $H_o = 6.600\ \mu m$, $h_o = 0.020\ \mu m$ and the graph $\sigma_c(H)$ presented in figure 3A) with very

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similar (almost identical) values of $H_o = 6.615\ \mu m$, $h_o = 0.020\ \mu m$. This verification is crucial since it confirms the assumption that the purely *topological parameters* H_o and h_o can indeed be considered as *general descriptors of height irregularities that dominantly influence compressive strength of cement paste*.

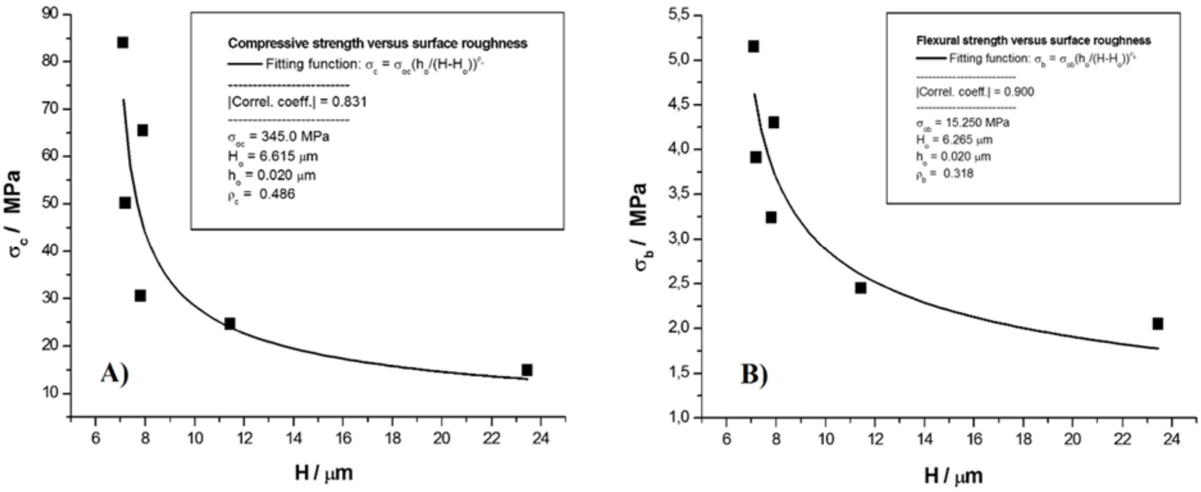

Figure 3. Mechanical strengths of hydrated Portland cement pastes as functions of surface irregularities quantified by the height parameter H: A) Compressive strength $\sigma_c(H)$; B) Flexural strength $\sigma_b(H)$.

3.3 Deriving formula for flexural strength.

Looking for a possible analytical form of the flexural (bending) strength in dependence on height irregularities $\sigma_b(H)$, some existing *empirical* relations between compressive and flexural strengths might be of assistance. These *experimental* relations usually have a power form like $\sigma_b = konst \cdot (\sigma_c)^\gamma$. For example, the Euro-Code EC-02 [7] recommends a relation between flexural strength and cube compressive strength that reads:

$$\sigma_b = 0.39(\sigma_c)^{0.67} \tag{4}$$

Such empirical relations however do not specify anything what would indicate the sought relation $\sigma_b(H)$. Nevertheless, applying equation (4) to equation (3), a formula for flexural strength emerges:

$$\sigma_b(H) = 0.3(\sigma_{oc})^{0.67}\left(\frac{h_o}{H - H_o}\right)^{0.67\rho_c} \tag{5}$$

which can be rewritten as follows:

$$\sigma_b(H) = \sigma_{ob}\left(\frac{h_o}{H - H_o}\right)^{\rho_b} \tag{6}$$

where the new parameters σ_{ob} and ρ_b have the following meanings

$$\sigma_{ob} = 0.3(\sigma_{oc})^{0.67\rho_c}\,, \qquad \rho_b = 0.67\rho_c \tag{7}$$

3.4 Fitting formula for flexural strength.

The values of the new parameters σ_{ob} and ρ_b can approximately be estimated by means of the values $\sigma_{oc} = 345$ MPa and $\rho_c = 0.486$ taken from the previously fitted graph shown in figure 3A). Although such an estimate may provide only very approximate quantities $\sigma_{ob} = 15.047$ MPa and $\rho_b = 0.326$, they may serve as a first guess for the new independent fitting procedure focused on the experimental flexural data $\sigma_b(H)$ shown in figure 3B). For this purpose the nonlinear regression method based on Levenberg-Marquardt algorithm [8] has also been used.

As can be seen in figure 3B), this new independent fitting procedure has provided optimized parameters $\sigma_{ob} = 15.250$ MPa and $\rho_b = 0.318$ almost identical to those approximately estimated by means of the compressive data.

The remaining two parameters H_o and h_o were optimized as well and their values $H_o = 6.265$ μm and $h_o = 0.020$ μm are in quite good agreement with the two parameters optimized by means of the compressive data (figure 3A), i.e. $H_o = 6.615$ μm and $h_o = 0.020$ μm. This is an extremely important fact repeatedly confirming that the purely *topological parameters* H_o and h_o can indeed be considered as *general descriptors of height irregularities dominantly influencing not only the compressive strength but also the flexural strength of cement pastes.* If these parameters were different for the compressive and flexural data, then H_o and h_o would not be capable of serving as the *general height descriptors* of surface irregularities.

3.5. *Applications of the results*

Since the mechanical strength of cement-based materials asymptotically approach certain limits as time elapses, it might be possible to measure *stable calibrated curves* $H(r)$, $\sigma_c(H)$, *and* $\sigma_b(H)$ for sufficiently matured materials.

On the basis of equations 1, 2 and 6 it is possible to derive explicit relations for the water-to-cement ratio r with which the cement pastes were originally prepared

$$r = \alpha_o \ln\left(\frac{H - H_o}{h_o}\right) \tag{8}$$

$$r = \frac{\alpha_o}{\rho_c}\ln\left(\frac{\sigma_{oc}}{\sigma_b(H)}\right)\,, \qquad r = \frac{\alpha_o}{\rho_b}\ln\left(\frac{\sigma_{ob}}{\sigma_b(H)}\right) \tag{9}$$

Let us suppose that the *stable calibration curves* $H(r)$, $\sigma_c(H)$, *and* $\sigma_b(H)$ *are formed*, i.e. the coefficients α , ρ , σ , h_o, and H_o are at our disposal. Accomplishing calculation of the original

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ratio r according to equation (8) requires microscopic measurements in order the value of surface irregularity H may be available (see equation (2)). On the other hand, equations (9) may offer a different way for determining r-value. No microscopic scanning and computing H are required. The only missing value are the actual compressive strength $\sigma_c(H)$ and/or the flexure strength $\sigma_b(H)$. These values may be estimated by means of the compressive or bending tests with samples separated from the tested matured material. The fact is that the calculations according to equations (9) are restricted to cement pastes and hardly be applicable to concrete materials.

Calculations performed on the basis of equation (8) may be applicable even to concrete. To illustrate such a situation, let us suppose that the graph $H(r)$, in figure 2 represents a *stable calibration curve*. Let us suppose that a concrete bridge made of our cement material has undergone a fatal failure and the quality of material is suspected to be the reason. Small fragments separated from the concrete bridge will serve as specimens for microscopic analysis. The microscope is capable of finding sites of pure cement matrix without aggregates. By scanning these sites and computing the corresponding surface irregularities H according to equation 2, the original water-to-cement ratio may be calculated or read directly from the calibration curve $H(r)$. Such a technique might be useful for specialists to decide whether the original mix of the concrete fulfilled the original instructions for r-value, but, of course, within some tolerance. This scenario could be realized only if the cement based materials were not affected by degradation processes (for example chemical processes). It means that such a calibration technique would be applicable to fatigue but otherwise chemically sound materials (chemically sound cement matrices).

Another type of applications may be envisaged in the field of fractography. The general *height descriptors* H_o and h_o stand for surface roughness and their values for a particular matrix indicate certain predisposition to mechanical failure.

4. Conclusions

In this paper flexural strength of cement pastes was studied. *The explicit formula for flexural strength was derived.* The formula assumes the form of a four-parametric function with one variable that is represented by the surface height irregularities of fracture surfaces. The derived formula for flexural strength agrees with the empirical relation between compressive and flexural strengths recommended by the Euro-Code EC 02 document.

The surface irregularity is introduced as the root-mean-squared height of surface profile $f(x,y)$. The graph of the flexural strength in dependence on height irregularity behaves as a monotonically increasing function whose form corresponds to power law.

The experimental study has shown that flexural strength σ_b depends on height irregularities H of fracture surfaces in the same way as compressive strength σ_c. Both the formulae $\sigma_c(H)$ and $\sigma_b(H)$ have equivalent forms and contain identical parameters H_o and h_o, which represent *basic descriptors of height irregularities of fracture surfaces* of cement pastes. Their values are governed by capillary porosity.

The height irregularities H are determined microscopically so that only small fragments of materials are needed. On the basis of the derived formulae the stable calibration curves for sufficiently matured materials may be formed to enable assessing quality of cement pastes and cement matrices in concretes. Especially the option to estimate the original value of water-to-cement ratio r of the cement matrix of old concretes seems to be very attractive.

To support validity of all results, a large ensemble of 120 specimens were analysed, 9600 digital files of microscopic photographs were registered and 480 three-dimensional digital relies $f(x, y)$ were processed.

Applicability of the obtained results might be foreseen in fractography, forensic engineering or in the field of testing the quality of cement matrices in concretes.

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