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Geometrical roughness analysis of cement paste surfaces using coherence scanning interferometry and confocal microscopy

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In this paper, geometrical roughness numbers named RN_x, RN_y and RN_S are formulated as a possible analytical tool for cement-based material surface characterization using coherence scanning interferometry (CSI) and STIL confocal microscopy technique (SCM). Recently, cement paste surface maps have been used to establish a link between surface statistical roughness parameters and the measuring scale (Apedo et al., 2015). The objectives of the present paper are to study how geometrical roughness numbers can be used as a tool to analyze the colonization of cement-based material surfaces by microorganisms as well as to perform other subsequent studies. Observations from a series of images acquired using both techniques (CSI and SCM) on both polished and unpolished samples are described. Using a new method named "window resizing", the results from CSI are compared with those from SCM. It appears that the surface available for colonization (convolution) is smaller than the surface developed by the measuring tool (sampling). The new method also allows the identification of the fractal regions and the associated fractal dimensions of both polished and unpolished cement pastes.

1. Introduction

Cement-based materials are heterogeneous, porous and rough composite materials with very complicated microstructures. During their service life, these materials are exposed to environments containing biological agents (microorganisms) and chemical compounds which may or may not be aggressive. The deterioration of cement-based material structures usually starts at the surface and progresses into the material [2]. The main factors that allow the penetration of aggressive agents into cement-based materials are their porosity [3–5] and their roughness [3,4,6,7], both of which influence their bioreceptivity which is the ability of the material to be colonized by one or more groups of living organisms [3,8]. Among the microorganisms able to colonize surfaces, bacteria are known to participate in the first step of biofilm formation.

The size of a bacterium ranges from 0.1 μm to 10 μm and its shape is variable, ranging from a sphere (for cocci) to rod-shaped (for bacilli) and spiral (for vibrios). Bacterial colonies form clusters which have a size of several tens of micrometers. The multiscale characterization of material surfaces appears to be an important area of the investigation to help provide a better understanding of how these external agents can form biofilms and interact with these surfaces.

The factor that is studied in this paper is the surface roughness. To characterize cementitious material surfaces, standard roughness parameters are often used with topographic reconstruction techniques such as confocal microscopy [1,9–25] and atomic force microscopy (AFM) [26–41].

Using scanning electron microscopy (SEM) or SEM coupled with energy-dispersive X-ray (EDX), some studies allow the detection of the formation of the microstructures and their chemical composition or provide qualitative analysis of hydration processes and products of cement-based materials [27–32,41–45].

More recently, Apedo et al. [1] have introduced a multiscale analysis of cement paste surface roughness. Two new optical profilometry techniques, coherence scanning interferometry (CSI) and scanning confocal microscopy (SCM) have been used in the surface reconstruction. A new method named "window resizing" has been introduced in the calculation of the standard roughness parameters. The information about the characteristics of the techniques already used in cement-based surface analysis has also been reported in this paper.

The knowledge of the standard roughness parameters provides the necessary information to understand the process of surfaces colonization by the microorganisms. But this information becomes very limited if it is necessary to go deeper into the knowledge concerning the

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relation linking the size of the microorganisms and the roughness geometry.

At this point, it then becomes interesting to determine the geometric roughness parameters. This consists of knowing the developed lengths and surfaces.

It should be noted, however that these geometrical parameters may depend on the measurement technique (in terms of its resolution and accuracy). It is also important to consider that the developed surface is a parameter that has no meaning in itself. Only the surface viewed by a probe of a given size has a meaning. Indeed, when the accuracy and the resolution increase (the probe size decreases for example), the surface viewed by the probe increases until it reaches a certain limit. The notion of surface appears subjected to different interpretations with no precise signification and only its projection is quantifiable.

Today, although many studies of cement-based materials have been performed using statistical roughness parameters, to our knowledge, very few investigations have been addressed using geometrical roughness parameters. These parameters, known as roughness *RN* numbers make it possible to better quantify the developed surface area available for colonization or reaction.

The studies already performed with roughness *RN* numbers deal with fracture surfaces of hydrated cement-based materials using confocal microscopy [9–12,16,25,46,47]. Using a magnification of ×90, (which controls the lateral resolution) and a z slice of 10 μm (which controls the z resolution), Lange et al. [9] compared the roughness numbers of several specimens of hydrated cement pastes and mortars. A great deal of their study was devoted to the implementation of the confocal technique and software processing of confocal optical sections in digital surface topographic maps. It has also been found that the fracture surface areas of cement paste are 1.8 times greater than the nominal projected surface areas and that of mortars range from 2.4 to 2.8 times greater than the nominal surface areas.

Lange et al. [10] have extended the analysis of Lange et al. [9] to the study of the correlation between the roughness numbers of fracture surfaces and the mechanical parameters such as critical stress intensity factor K_{Ic}, critical effective crack length a_c, compressive strength σ_c, total porosity and effective pore diameter. These analyses pointed to a strong correlation between roughness numbers and stress intensity factor K_{Ic} as well as crack length a_c whereas only a weak correlation has been observed with the compressive strength σ_c. Almost no correlation has been found for all the other material properties (total porosity and effective pore diameter). The paper also addresses the fractal dimension calculation as a function of the roughness numbers. Testing a notched concrete beam using three-point bending, Zampini et al. [11] used confocal microscopy to analyze the region near the interface between the cement paste and the aggregate. The roughness numbers in the proximity of the paste-aggregate interface has been found to be higher than that of the paste outside the interface. A correlation between the critical stress intensity factor K_{Ic}, the critical crack extension δa_c and the roughness of the fracture surfaces of cement-based materials has been found. Lange et al. [46] analyzed using confocal microscopy, the relationship between a cement based material's strength and its roughness *RN* number. Cement based matrices reinforced by randomly dispersed microfibers have been tested using both uniaxial tensile and three-point bending. Abell and Lange [12] obtained for cement pastes, the relation $\frac{K_{Ic}}{K_{Im}} = \sqrt{RN}$ initially established by Xin et al. [47] for S_i single fractured crystals. Abell and Lange [12] also established the relation between the roughness number and the fracture toughness values for mortars as $\frac{K_{Ic}}{K_{Im}} = RN^{0.45}$. Kurtis et al. [16] demonstrated the potential applications of confocal microscopy through surface roughness measurements using *RN* numbers. Ficker et al. [25] introduced a new roughness parameter known as fractal roughness number Rn_o which is scale-dependent only within the region of fractality of fractured cement pastes. This new parameter has been shown to be lower than the ordinary roughness number *RN*. The relationship between the water-to-cement ratio w/c and both *RN* and Rn_o has been provided. The correlation between fractured cement paste compressive strength and the roughness numbers *RN* and Rn_o has also been investigated.

All these studies lead us to conclude that the roughness *RN* numbers are very useful for the characterization of cementitious materials. Knowing that these roughness parameters depend on the measuring scale, a multiscale analysis seems to be required.

Although CSI showed its performance in the characterization of various kinds of materials [48–58] its use remains relatively unexplored in the field of cement-based materials [1,59,60].

In view of these previous studies, therefore, two things become quite clear. Firstly, the quantitative analysis of the surface roughness of cementitious materials using *RN* numbers is very important. Secondly, CSI has a great potential in this area but requires careful study to explore the performance, limitations and protocols for successful measurement in view of the high roughness and inhomogeneous nature of cementitious materials.

The present paper extends the work presented in [1]. Both polished and unpolished cement paste surfaces already measured with CSI and SCM and presented in [1] are used to quantify multiscale roughness *RN* numbers introduced by "window resizing". The paper describes the roughness *RN* numbers calculation method using "window resizing". The identification of fractal region and its fractal dimension of both polished and unpolished cement pastes is also investigated.

2. Method

2.1. CSI and SCM

The microscopy techniques (CSI and SCM) used in this paper have already been described in [1]. CSI and SCM are two optical profiling techniques for measuring a material surface's topographic map. These two optical techniques are different due to their accuracy in Z (0.04 μm for CSI and 0.1 μm for SCM), their lateral resolutions (0.45 μm for CSI and 2 μm for SCM), their Z-resolutions (1 nm for CSI and 10 nm for SCM) and the extent of the surfaces that they allow to explore (184 μm × 138 μm for CSI and 4.5 mm × 4.5 mm for SCM). Thus, the two techniques make it possible to measure the topographic map of surfaces at two different scales. Other characteristics of these techniques can be found in [1].

Areas selected in the middle of the samples were scanned by both techniques.

Using CSI with a camera pixel size of 0.13 μm × 0.13 μm, the scanned areas consisted of 183 μm × 138 μm for the polished samples and 178 μm × 99 μm and 69 μm × 55 μm in the case of the unpolished samples.

In the case of SCM, the scanned areas consisted of 2 mm × 2 mm squares and the pixels were recorded every 4 μm × 4 μm.

2.2. Geometrical parameters measurement by window resizing

Among the methods that allow the quantification of the surface roughness of cement-based materials, one of the most widely used is the statistical analysis based on the determination of standard roughness parameters such as the amplitude parameters, the spacing parameters and the hybrid parameters [1,19–27,29,33,35,39,41,61]. Among these standard roughness parameters may be mentioned: the altitude difference between the highest and lowest measured points H_{mm} [1], the average of the absolute irregularities R_a [1,19–25,29,35,41,61], the root mean square (rms) of the irregularities R_q [1,19,23–27,33,35,39,61], the skewness R_{ks} and the kurtosis R_{ku} [23,25]. Some of these standard roughness parameters have already been used in conjunction with the window resizing method [1].

A parameter that is also commonly used to quantify the roughness of cementitious materials is the roughness number *RN*. This parameter is generally used to quantify the roughness of fractured surfaces [12,16,