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Cement paste surface roughness analysis using coherence scanning interferometry and confocal microscopy

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ABSTRACT

Scanning electron microscopy and scanning probe microscopy have been used for several decades to better understand the microstructure of cementitious materials. Very limited work has been performed to date to study the roughness of cementitious materials by optical microscopy such as coherence scanning interferometry (CSI) and chromatic confocal sensing (CCS). The objective of this paper is to better understand how CSI can be used as a tool to analyze surface roughness and topography of cement pastes. Observations from a series of images acquired using this technique on both polished and unpolished samples are described. The results from CSI are compared with those from a STIL confocal microscopy technique (SCM). Comparison between both optical techniques demonstrates the ability of CSI to measure both polished and unpolished cement pastes.

1. Introduction

The interaction between biological cells and cement materials has become an important issue today for the development of new eco-based cements for use in construction. In the field of cement-based materials, a precise knowledge of the surface structure at different scales is necessary to help in gaining a better understanding of how biological cells of different sizes interact with them. In order to better understand processes such as microbial adhesion, it is therefore very important to conduct a multiscale description of these surfaces.

The evaluation of the surface structure of cementitious materials is also very important for many other fundamental problems such as the study of surface-to-surface interfaces, nanoindentation, mechanical properties, micro-cracking and strength analysis [1–3].

Cementitious materials are heterogeneous and porous composite materials. The structure of the porous network allows the penetration of external agents (aggressive or not) into the bulk of the material. Concerning the biodegradation of cementitious materials, these external agents may be biological elements or chemical compounds secreted by microorganisms [4,5].

Bioreceptivity is the ability of a material to be colonized by one or more groups of living organisms [6]. The main parameters of the material defining its bioreceptivity are the porosity, together with the surface condition and the chemical composition [6,7].

The porosity of cementitious materials is an extremely important parameter because it influences the surface roughness as well as the amount of water potentially present in the material. Porosity and its geometry are related to the structure of the material, which itself depends on the fabrication method. A material with significant porosity also offers a higher surface area for the reaction between the material and aggressive chemical substances with which it is in contact, leading to a potentially higher degradation rate.

Another important factor that influences the colonization of cement-based materials is the surface roughness, which is the subject of this paper.

Amongst the different techniques typically used nowadays to analyze cement paste surfaces, are scanning electron microscopy (SEM), atomic force microscopy (AFM), lateral force microscopy (LFM) and confocal microscopy.

SEM is of primary importance to detect the formation of microstructures and to detect their chemical composition [8–11]. Although the SEM technique has been used extensively to analyze cement pastes, it has been shown that quantified surface texture characteristics and precise morphologies of these materials cannot be provided by this conventional microscopic technique [12,13]. Another limitation is that SEM is most commonly used to investigate specimens under high vacuum, which can lead to alteration or damage of the microstructure's morphology [12]. Due to these limitations with SEM, there has been a growing interest amongst the cement-based material community to study the nano- and micro-structural characteristics of cement pastes using AFM techniques [13,14].

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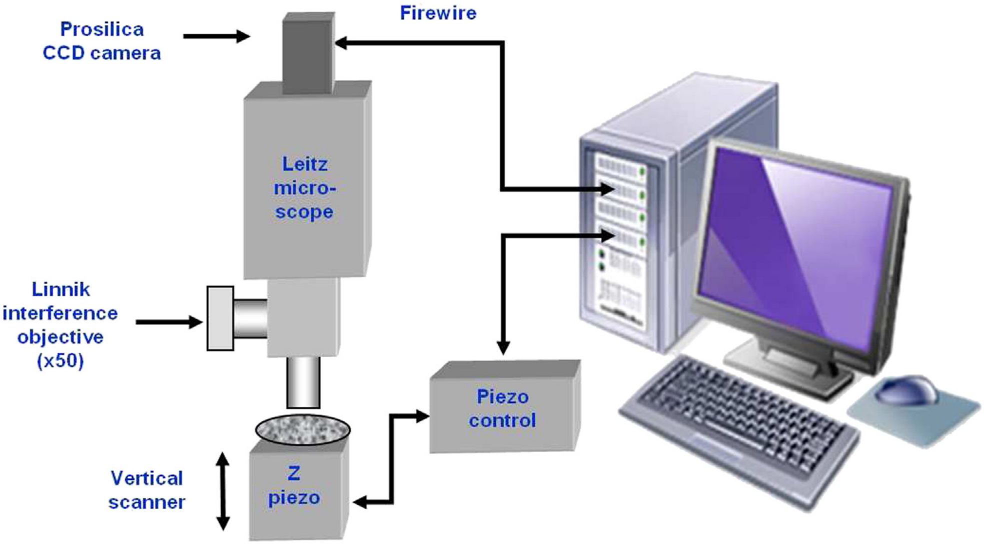

Fig. 1. Layout of CSI measurement system developed at ICube [52].

AFM can provide detailed and quantified surface texture characteristics concerning particle shape for hardened cement systems at a scale that cannot be provided by SEM [10–16]. The lateral resolution of this technique can approach 30 nm and the vertical resolution can attain 0.1 nm. In cement and concrete research, AFM has been used to study the early hydration of cement [9,17], the interaction between cement particles [18], the carbonation process [13,14], the interactions of the cement-admixture [19,20] and the morphology and surface roughness of cementitious materials [2,10–12,21–28]. However, the extensive application of AFM techniques is hampered due to the following reasons: (a) cement pastes represent porous and heterogeneous materials with complicated structures and variable chemical compositions; (b) high-resolution AFM studies require reasonably flat surfaces (<5 μm); and (c) the chemical composition of the surface cannot be directly identified by AFM. Image size is also one of the disadvantages of AFM compared with SEM. In the case of SEM, an image area of the order of millimeters × millimeters and a depth of field of the order of millimeters can be studied. AFM on the other hand can only image up to a maximum scanning area of around 150 × 150 μm and a maximum height of a few μm. Another disadvantage of AFM is the scanning speed. AFM typically takes several minutes or even up to tens of minutes for a typical scan, especially on very rough surfaces so as to avoid tip damage. Several artifacts and limitations also exist with the AFM technique [29].

Another technique that is being used more and more is that of confocal microscopy. The main advantage of this technique is its ability to obtain optical sections of the sample at different depths in a non-destructive way [30]. The lateral resolution of this technique can approach 200 nm and the vertical resolution can be up to 600 nm. Results obtained by Lange et al. [31,32], Zampini et al. [33] and Abell and Lange [34] demonstrate the utility of the confocal microscopy technique, particularly for examining the rough surfaces that are characteristic of concrete. Many other studies on cementitious materials using confocal microscopy can be found in Ref. [35–40]. One particular form of confocal microscopy for material analysis is SCM consisting of an optical probe that is moved in *X* and *Y* to build up a complete 3D image of the surface morphology. This technique has been used in this work (see Section 2.2). Recently, Refs. [3,41–46] have used confocal microscopy for the measurement of fractured surfaces of hydrated cement pastes. One of the final objectives was to find surface parameters that are sensitive to changes in the water-to-cement ratio and compressive strength of cementitious materials. While the considerable progress made with confocal microscopy shows the interest in the use of optical techniques, the technique does have the drawbacks such as is its slowness in reconstructing a 3D image from a set of tomographic image slices and the poor axial resolution that is insufficient for many applications.

CSI on the other hand allows rapid 3D measurement of surface roughness and shape in a matter of seconds or minutes and can attain nanometric axial resolution over large depths (mm) [47,48]. The technique is based on far field optical microscopy that uses white light interference fringes as a probe scanned over the depth of the sample in order to measure the surface roughness. The technique is widely used for the surface analysis of a wide range of materials, ranging from semiconductors [49], electronic and opto-electronic devices, MEMS and MOEMS [50] through diffractive optical elements [51], colloidal [29] and biomaterial layers [52]. It is also used in a tomographic mode for the characterization of complex layers [53]. Using a high speed camera and FPGA processing the 4D mode of CSI allows the measurement of 3D surfaces that evolve over time, such as soft materials, microsystems and chemical reactions [54]. Recently, this technique has been used to analyze rubber toughened polymethyl–methacrylate fracture surfaces [55]. An excellent review of the applications of interferometric techniques to cement materials up to the early 1980s was provided by Jacquot and Rastogi in 1983 [56,57]. A theoretical background on this microscopic technique can also be found in Refs. [29,52]. At present, CSI is not well known in the field of civil engineering and particularly in the measurement of cementitious materials. To our knowledge, only the work published by Erdem et al. and Chen et al. [58,59] can be cited in this area. This work concerned the measurement of the interfacial transition zone surface area roughness using a vertical scanning interferometer and a scanning electron microscopy coupled with energy dispersive X-ray technique [58]. In view of all these previous studies therefore, two things become quite clear. Firstly, the quantitative analysis of the

Table 1
The characteristics of the microscope used for surface roughness measurement.

Microscope	Objective magnification	Numerical aperture	Lateral resolution (μm)	Max image size (pixels)	Accuracy in Z (μm)	Sample pixel size (μm)	Max field of view (μm)
Leitz	×50 Linnik	0.85	0.45	1360 × 1024	0.04	0.134	184 × 138