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THEMED ISSUE ARTICLE

Quantification of surface grinding during the sample preparation of cementitious materials by optical profilometry

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Abstract

Sample preparation is of utmost importance for any microscopy and microstructural analysis. Correct preparation will allow accurate interpretation of microstructural features. A well-polished section is essential when scanning electron microscopy (SEM) is used in backscattering electron (BSE) mode and characteristic X-rays are to be quantified using an energy-dispersive spectroscopy (EDS) detector. However, obtaining a well-polished section, especially for cementitious materials containing aggregates, is considered to be challenging and requires experience. A sample preparation procedure consists of cutting, grinding and polishing. Undercutting of soft and brittle paste between harder aggregates can be overcome by vacuum epoxy impregnation offering mechanical support in the matrix. Furthermore, most of the attention during the sample preparation is given to the polishing of the sample. There is a wide range of suggestions on polishing steps, ranging from grain sizes, time and applied force; however, the final assessment of a polish surface is often subjective and qualitative. Therefore, a quantitative, reproducible guidance on the grinding steps, effect of experimental parameters and the influence of different grinding steps on the surface quality are required. In this paper, the influence of grinding was quantitatively evaluated by a digital microscope equipped with optical profilometry tools, through a step-wise procedure, including sample orientation, grinding time and the difference between cement paste and concrete. Throughout the grinding procedure, the surface profiles were determined after each grinding step. This showed the step-wise change in surface roughness and quality during the grinding procedure. Finally, the surface qualities were evaluated using optical and electron microscopy, which show the importance of the grinding/prepolishing steps during sample preparation.

KEYWORDS
grinding, optical profilometry, sample preparation, surface roughness

1 | INTRODUCTION

Understanding relationships between concrete microstructure and properties requires quantitative information about microstructure such as chemical composition (energy-dispersive X-ray spectroscopy), phase distribution, quantification of concrete microcracks and voids (image analysis) and local nano-mechanical properties such as E-modulus (nanoindentation).[1–3] For such microstructural analyses or petrographic studies, sample surface characteristics such as surface roughness, waviness and shape could be critical. Inadequate preparation techniques can lead to erroneous diagnoses of the concrete hardening or deterioration mechanisms.[4] The systematic description of the laboratory testing program and the sample preparation techniques prior imaging of a concrete microstructure or its deteriorated form are rarely reported.

Concrete sample preparation generally starts with sample extraction from the bigger samples by cutting. Subsequently, grinding steps are selected based on the available equipment in a laboratory and user's knowledge. For example, grinding is first automatically performed with a saw of a certain thickness and then with a slight downward pressure using progressively finer silicon carbide (SiC) paper and appropriate liquids on a slotted steel plate. The quality of grinding per step will have a progressive effect on the required surface preparation time or on its end quality. The final quality of surfaces after grinding is generally evaluated by an optical microscope. Then samples are polished using successively finer diamond paste for several minutes or hours.[5] Commonly, the scratch-free surface observed under light microscopy is the only criterion for evaluation of surface preparation quality. No criterion exists for the evaluation of surface roughness in relation to surface grinding operations. Scanning electron microscopy (SEM) and optical profilometry are commonly used to measure the conventional grinding wheel topography.[6] Because of the smaller tool sizes (< 1 mm), it is difficult to implement mechanical profilometry techniques for the micro grinding tools, despite their accuracy. The application of the SEM technique is time-consuming and it needs postprocessing to obtain the topography information. It is common to use optical methods in metal cutting processes for various purposes, such as process monitoring and tool condition monitoring, and for the characterisation of the finished surface.[7,8] In fact, optical profilometry allows measurement of surface irregularities with a nanometric resolution and provides a 3D reconstruction to highlight them.[9,10] Concrete surface waviness, meso-waviness and shape are influenced significantly by the type of surface preparation. At those roughness levels, it is likely that the corresponding concrete surface characteristics are influenced to a certain degree by the fine and coarse aggregates grading, but surface preparation appears to be the dominant factor.[10]

Epoxy impregnation together with the consistent use of bonded-diamond abrasive for all cutting, lapping and polishing will eliminate undercutting of soft cement paste between harder aggregates.[11] The relationship between impregnation steps and epoxy intrusion depth is studied for purpose of backscattered electron imaging.[5,12] To the best of authors' knowledge, there are not any quantitative studies in literature about the relationship between grinding steps and surface roughness measurements in cementitious materials. One reason for this might be that it is rather difficult to reproduce, analyse or evaluate the results, which formed the motivation of this study.

This paper focuses on the effects of grinding on surface roughness of cement paste and concrete samples. The system selected for preliminary study, epoxy-based sample, has been studied by monitoring the change in roughness of the resin. It has good measurement sensitivity and it is used as impregnation material for cement paste and concrete. In order to have an assessment of the grinding quality, epoxy resin and both cement paste and concrete samples have been characterised by optical profilometry. It is demonstrated that this more thorough preparation technique reduces the amount of irregularities, thus reducing the magnitude of the high-uncertainty correction, and thereby improves the accuracy of the data and imaging. It should be emphasised that the purpose of the study is not to propose an optimal surface preparation protocol but rather to explore the quantification of surface condition during grinding and polishing operations of cement based materials.

2 | EXPERIMENTAL PROGRAM

2.1 | Materials and sample preparation

The studied materials were pure hardened epoxy resin, cement paste and concrete samples. The epoxy resin was prepared using Conpox Resin BY 158 (liquid) mixed with Conpox Hardener HY 2996 (Polyamine liquid). The ratio of epoxy resin to hardener by mass was 3.33:1. The density of the epoxy resin and hardener mix was 1.16 g/cm^3. The mixture was prepared through gentle stirring for about 10 min until no tailing and air bubbles were observed.

CEM I 42.5 N (Portland cement) was used in cement paste and concrete mixtures with an identical water-to-cement (w/c) ratio of 0.50. The samples were cast and

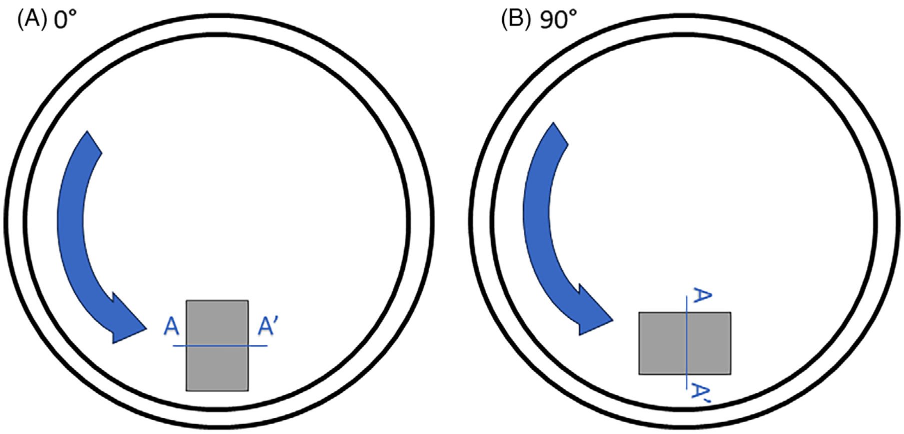

FIGURE 1 Sample placement and orientation during grinding.

moist-cured for first 28 days. Afterwards they were placed in laboratory conditions for 2 years.

2.2 | Sample cutting

The samples were sliced into several sections of 40 × 40 × 40 mm^3. Then, the sections were dried in an oven at 40°C. The epoxy, cement paste and concrete samples were used to evaluate the influence of surface hardness on grinding. Additionally, concrete samples were used to evaluate the influence of grinding on the undercutting (region between hard and soft material). The samples were glued to a working glass and by means of vacuum block cut to a 30 × 45 × 12 mm^3 sample using Pelcon Automatic Thin Section Machine for further sample preparation.

2.3 | Sample impregnation

All samples were vacuum impregnated with a low viscosity epoxy for 15 min. After hardening, the top face of the sample was ground with a FEPA P180 SiC paper in order to remove the surface layer of epoxy and to bring the surface quality to a reference state prior to further grinding.

2.4 | Sample grinding

Struers LaboPol-60 grinding and polishing machine was used for sample grinding. Struers SiC Paper with FEPA P grit numbers for wet grinding of materials were chosen. The grinding was done under a constant 300 rpm. The sample placement was chosen at the bottom of the grinding disc near the perimeter (Figure 1). The specimen position on the lapping disc was fixed during the entire experimental study, in order to control the scratching action. The samples were cleaned by rinsing with demineralised water in a ultrasonic bath after 1 min of grinding.

It was assumed that the movement of a sample without a preferred orientation during grinding would prevent orientational grinding marks. It is questionable whether such phenomenon would occur as silicon carbide grains are randomly distributed over the grinding paper (Figure 2). Therefore, the sample orientation in grinding procedure was evaluated by measuring roughness of the sample surface after being ground at fixed position (0°) and being rotated for 90° (Figure 1). First, the sample was ground for 1 min at 0° orientation, after which the surface roughness was measured. Second, the sample was ground for 1 min at 90° orientation, using a new grinding paper of the same roughness as sample at 0°. The roughness was again measured after which the grinding moves to the grid size as shown in Table 1.

2.5 | Methodology

Optical microscopy was used to examine the sample preparation in order to evaluate effectiveness of the removal of the layers and epoxy infill. For further analysis on the effect of grinding on surface roughness, a Keyence VHX-7000 digital microscope with a VHX-7100 fully integrated head was used. Samples were observed under full ring lighting with 100× magnification using a Keyence VHX-E100 lens. Based on Focus Variation microscopy,[13] a fine depth composition image was acquired by capturing 200 images in focus at different height and construct a 3D image. The main characteristics of the objectives used in the present study are presented in Table 2.

In order to ensure studying the same location during the sample preparation procedure, a placeholder was installed in the middle of the microscope x–y moving stage. For

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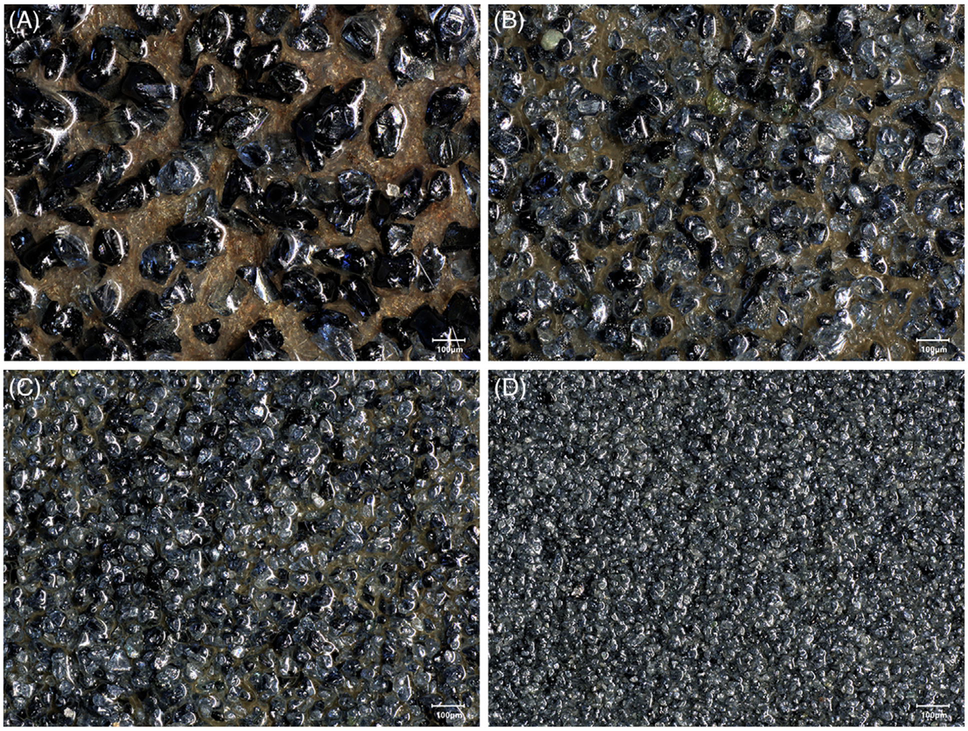

FIGURE 2 (A) P180, (B) P320, (C) P500 and (D) P1200 grinding papers as observed using a Keyence VHX-7000 digital microscope equipped with a E100-×200 variable focus lens and polarising filter. F.O.V. = 1487 μm. Reflected light imaging under an 15° angle with a partial ring illumination using HDR mode.

TABLE 1 Working steps.

Step	Grinding paper	Grain size D_{50} (μm)	Sample orientation	Methods	Duration (min)
1.1	P-grade 180	82	0	Ultrasonic bath, air gun dry	1
1.2	P-grade 180		90		1
2.1	P-grade 220	68	0	Ultrasonic bath, air gun dry	1
2.2	P-grade 220		90		1
3.1	P-grade 320	46.2	0	Ultrasonic bath, air gun dry	1
3.2	P-grade 320		90		1
4.1	P-grade 500	30.2	0	Ultrasonic bath, air gun dry	1
4.2	P-grade 500		90		1
5.1	P-grade 800	21.8	0	Ultrasonic bath, air gun dry	1
5.2	P-grade 800		90		1
6.1	P-grade 1200	15.3	0	Ultrasonic bath, air gun dry	1
6.2	P-grade 1200		90		1

TABLE 2 Specifications of optical profilometer objectives used in the present study.

Objective	Lateral resolution (x/y)	Vertical resolution (z)
×100	1.0 μm	2–4 μm

each sample, three different locations were analysed for which the coordinates were registered. Four line roughness measurements, two horizontal and two vertical, were performed for each location.

The surface profile for each sample was evaluated by using the surface roughness profile. A roughness curve is created by removing the long-wavelength component from the cross section curve. This means that for the roughness profile, only the high-frequency components of the cross section curve were recorded through a high-pass filter λc. The λc defines the intersection between the roughness

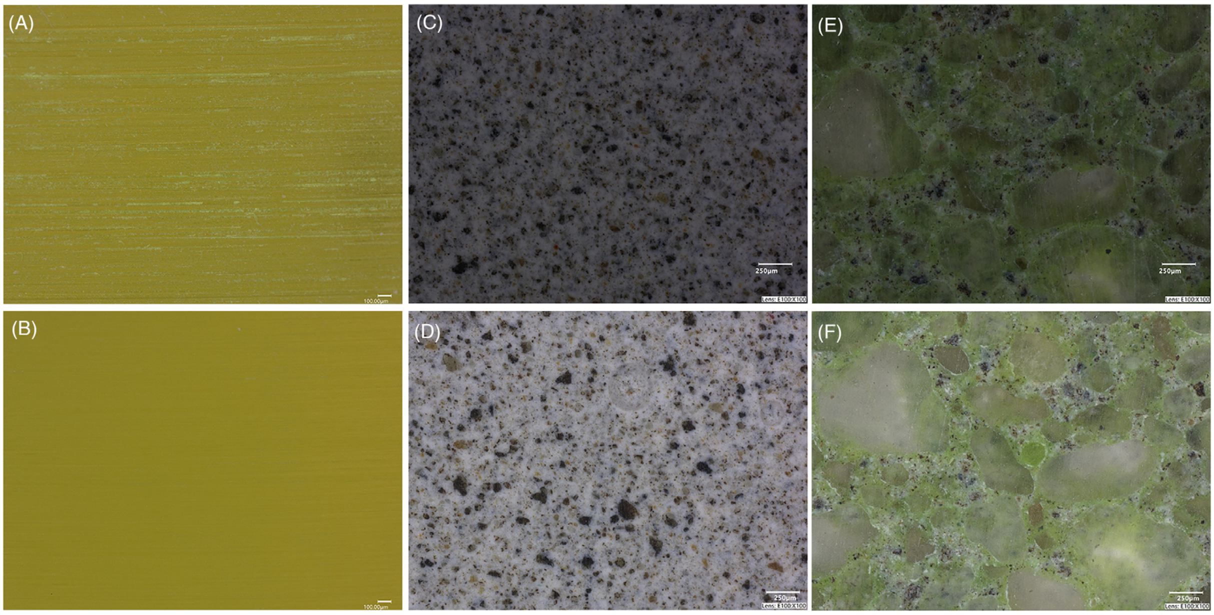

FIGURE 3 Optical micrographs of epoxy sample (A) after P180 grit and (B) after P1200 grit, cement paste sample (C) after P180 grit and (D) after P1200 grit, concrete sample (E) after P180 grit and (F) after P1200 grit. F.O.V. = 2975 μm.

and waviness components.[14] In order to exclude any possible inclination in the sample, the cutoff value for the high-pass filter λc was set at 2.5 cm, which was equal to the measurement length. By applying this filter, the roughness profile lines were corrected for any inclination into a horizontal line.

Surface texture parameters were determined using equations that comply with ISO 4287:1997 and ISO 13565:1996.[14,15] One of the most common parameter to describe roughness is the arithmetical mean (R_a) deviation of the profile.[10] Another important parameter to evaluate is the highest height of the roughness profile (R_z).[13] Additionally, the maximum profile peak height (R_p) will be defined. These parameters were determined as follows:

$$R_a = \frac{1}{l_r} \int_0^{l_r} |Z(x)|dx, \tag{1}$$

$$R_z = \max(Z(x)) + |\min(Z(x))|, \tag{2}$$

$$R_p = \max(Z(x)), \tag{3}$$

where $Z(x)$ is the height of the assessed profile at any position x and l_r is the reference length in the direction of the X-axis.

3 | RESULTS AND DISCUSSION

Figure 3 presents the micrographs of the epoxy, cement paste and concrete samples after the first grinding step with a grit paper P180 and after the last grinding step with a grit paper P1200. Since epoxy has the lowest hardness of the three samples, the scratches were easily identified after grinding with P180 paper. Furthermore, one can observe that the micrographs from the samples become brighter under fixed illumination conditions, as the grinding proceeded towards finer grades. This can be attributed to increasingly smoothening of the sample, which makes the light reflect.

By evaluating the roughness profiles of the samples after each sequential grinding step, it was found that the scratches on the sample after the first grinding step were deep, highly irregular and with a high frequency (Figure 4A). With successive grinding steps towards increasing fineness, these larger scratches were flattened into smaller defects and the profile height decreased as can be observed in Figure 4B and C. Furthermore, the smaller defects were smoothened and finally the larger defects were completely flattened.

Surface roughness profiles of the concrete samples showed the challenging nature of grinding and polishing concrete samples, due to undercutting of the cement paste near the interfacial transition zone. This undercutting was visualised by means of the total profile line in Figure 5. It can be observed, from the upper line, that around the indicated (yellow) line, there was a change in profile height. Due to the applied high-pass filter, this undercutting will not influence the further analysis of the roughness profile, bottom line. Although the sample was epoxy impregnated undercutting within a concrete sample could not be avoided with the applied equipment

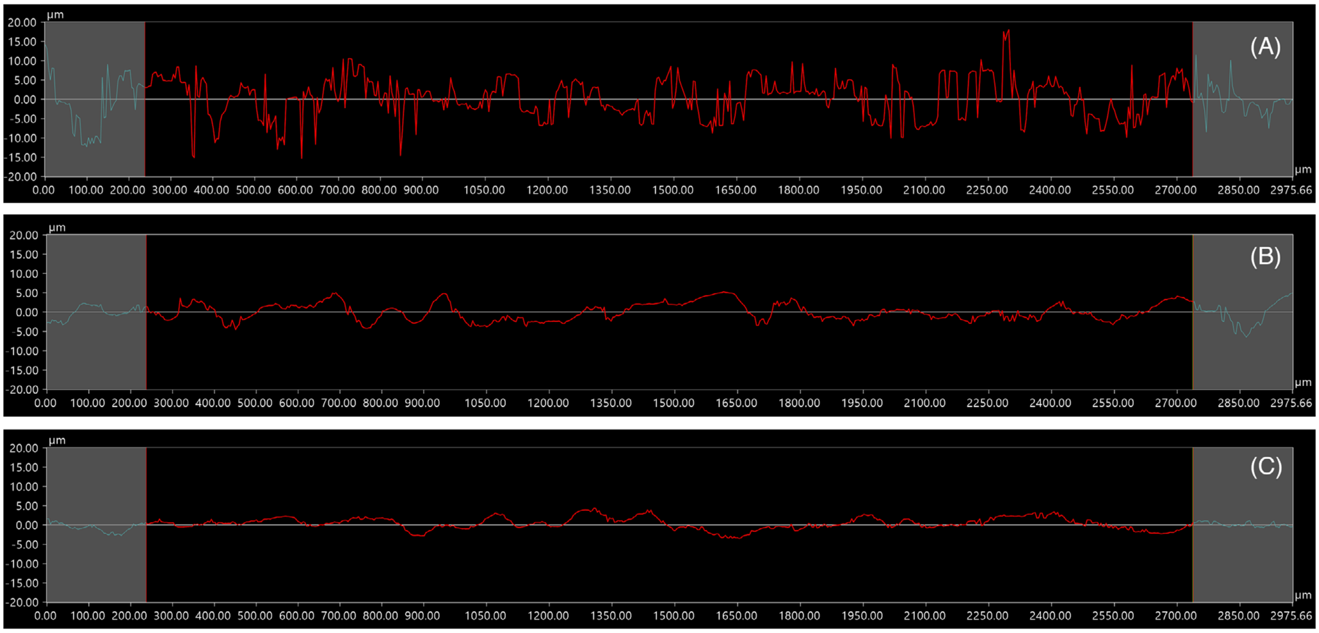

FIGURE 4 Surface roughness profile of the cement paste sample after grinding with grit paper: (A) P180, (B) P500 and (C) P1200.

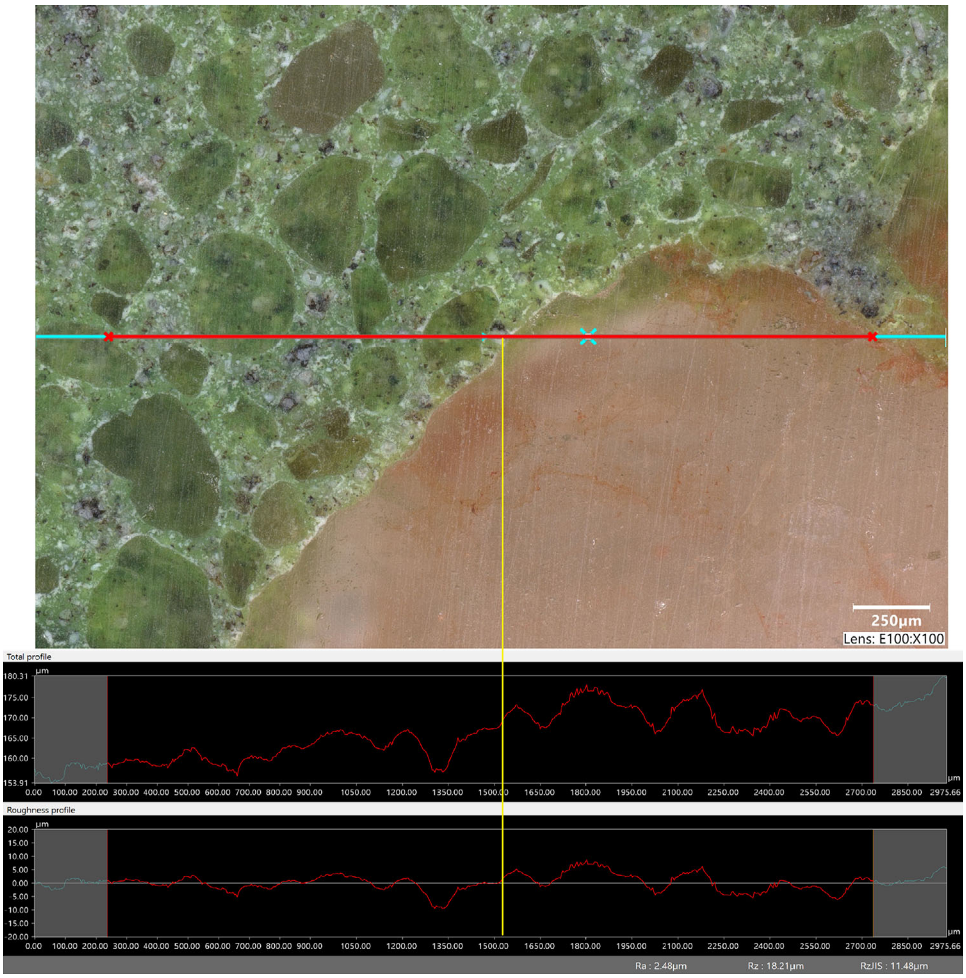

FIGURE 5 Micrograph of concrete sample with a coarse aggregate, including the profile and roughness line, F.O.V. = 2975 μm.

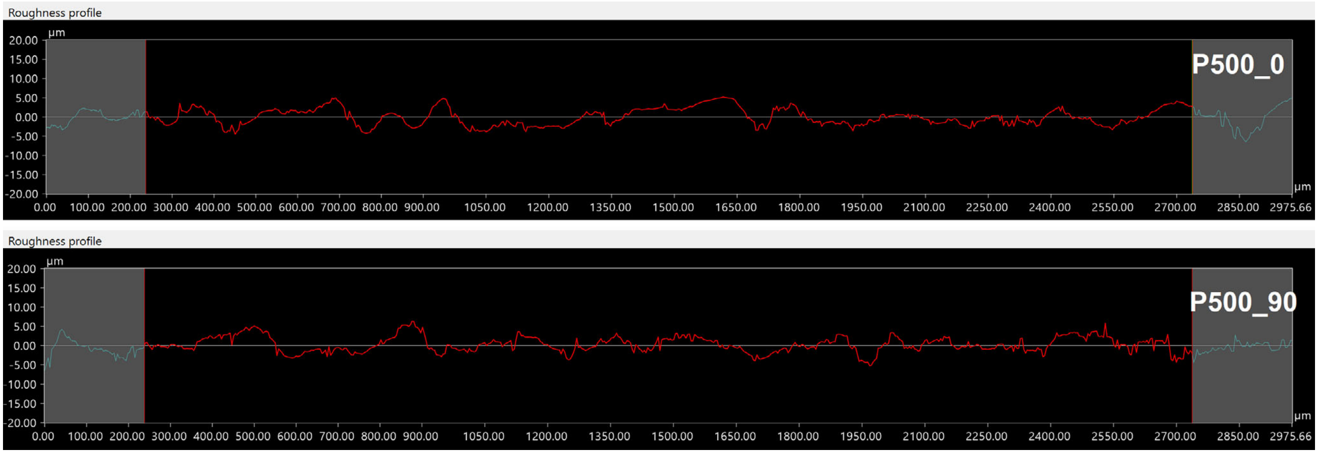

FIGURE 6 Surface roughness of the cement sample after grinding with grit paper P500 at 2 orientations.

and work procedure in this study. It should be noted that using a double polished section and bonded-diamond abrasives can minimise undercutting.

By comparing the surface roughness profiles of a ground sample after 1 min at a 0° orientation and after 1 min at 90° orientation (as shown in Figure 1), we were able to evaluate the effect of sample orientation. From Figure 6, it can be observed that although there was a small increase in frequency of defect at 90° orientation, there was no change in the overall roughness. The change in sample orientation does not decrease the arithmetical mean height (R_a) and the maximum height of the profile (R_z) and the measured values are within the standard deviation of the roughness measurements (Figure 7). Nevertheless, the results show that sample orientation becomes less influential with decreasing grit size for epoxy and cement paste samples. With a grit size of $< 30\ \mu$m, which corresponds to P500 or higher, the difference between the surface texture parameters from the two different orientations becomes smaller. This decrease in standard deviation was not observed for concrete samples. It is assumed that this can be attributed to undercutting and the surface hardness of concrete.

The influence of the grinding paper on the surface roughness was evaluated by means of the surface texture parameters and plotted against the grain size (D_{50}) of the grinding paper (Figure 7). Figure 7A shows that the arithmetical mean height (R_a) of the cement paste and concrete samples decrease with decreasing grain size. By decreasing the mean height of the sample, it can be concluded that the evaluated surface becomes smoother. Additionally, Figure 7B shows that also the maximum height of the profile (R_z) decreases for the cement paste and concrete samples. This indicates that the peaks that occur when grinding with coarse grit paper were flattened with increasing fineness. The slope of the trendline through the data points is an indication for the surface hardness.[16] The R_a and R_z values of the concrete sample decrease less significant compared to that of the cement paste. It was assumed that this is because the scratches that the grinding paper caused on a concrete sample was shallower compared to the cement paste counterpart.

The results showed that the above mentioned discussion does not hold for the pure epoxy samples, as the R_a shows a slight increase and the R_z stays unchanged with the decreasing grain size. It can be expected that the epoxy samples should behave similar to the cement paste samples as their surface hardness is less than the concrete samples. However, due to the low surface hardness of the epoxy samples, it was decided to reduce the applied manual pressure on the sample during grinding. It should be noted that when applying a lower grinding force, the depth of cut or scratch will be decreased.[17] Therefore, the R_a and R_z will not significantly change during the successive grinding steps when applying less grinding force, Nevertheless, grinding of samples will create scratches in the sample. Whether these scratches decrease in absolute height can be evaluated by means of the maximum profile peak height (R_p). Figure 7C shows that the R_p decreases with decreasing grain size of the grinding paper, which means that the epoxy samples are also flattened and well ground.

4 | CONCLUDING REMARKS

It can be concluded from this study that optical profilometry can be used to evaluate the surface roughness of samples during the sample preparation process. Although it might not be practical to use profilometry for regular laboratory work, the methodology could be very useful to establish a reproducible surface preparation protocol by targeting specific surface texture parameters: R_a, R_z and

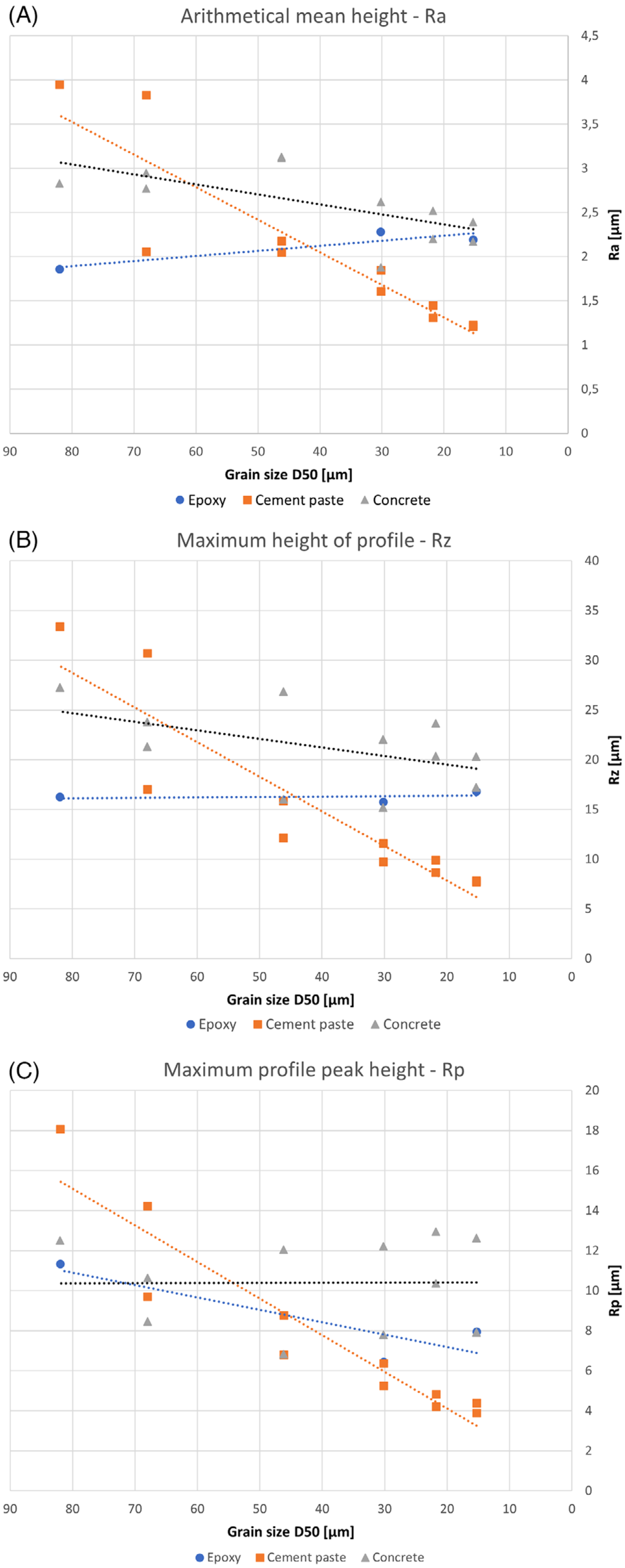

FIGURE 7 Change in surface texture parameters in relation to the grain size of applied grinding paper: (A) arithmetical mean height (R_a), (B) maximum height of profile (R_z) and (C) maximum profile peak height (R_p).

R_p. According to our study, the maximum surface roughness, R_z, that is allowed before continuing to the next grinding step, should be smaller or equal to the grain size (D_{50}) of the use grit paper. Nevertheless, this will not be measurable for the coarse grit paper, since the surface hardness of the sample influence the effect of grinding. As shown in this work, an epoxy sample with a low surface hardness was very easy to scratch, while the scratches made in the concrete sample surface were significantly less deep.

Aside from evaluating the surface texture parameters, the roughness profile of the sample should also be evaluated for smoothness. Before moving from grinding to polishing of the sample, there should not be a significant waviness in the sample and the majority of the peaks should be flattened. Ideally there should not be any waviness in the whole sample.

For future work, the same procedure of surface roughness measurements should be applied during the polishing procedure, in order to evaluate the required polishing time and impact of grinding quality on the following polishing steps. Furthermore, attention should be paid to grinding duration and the applied pressure when grinding in relation to the surface hardness, especially for softer materials like cement paste and epoxy.

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