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MICROSCOPIC OBSERVATION OF CRACKS IN CONCRETE — A NEW SAMPLE PREPARATION TECHNIQUE USING DYE IMPREGNATION

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ABSTRACT

A new technique to prepare concrete sections for microscopic examinations is briefly described. The procedure involves the impregnation of a concrete section with a colored dye diluted in an alcohol. After a short impregnation period during which the concrete is constantly kept humid, the section is polished under water. Contrary to most dye impregnation techniques, this method does not involve any drying of the sample. Observations of cracks using optical microscopy can therefore be made without concern about drying shrinkage-induced cracking. In addition to a detailed description of the preparation technique, various examples of the application of the method are given. The possibilities of using this dye-impregnation technique in combination with an image-analysis system are also discussed.

RÉSUMÉ

Une nouvelle technique de préparation d'éprouvettes pour l'observation de fissures dans le béton au microscope optique est brièvement décrite. La méthode consiste essentiellement à imprégner une éprouvette de béton avec un colorant rouge préalablement dilué dans un alcool. Après une courte période d'imprégnation durant laquelle le béton est constamment conservé humide, l'éprouvette est légèrement polie sous eau. Contrairement à la plupart des techniques d'imprégnation au colorant, cette procédure ne nécessite aucun séchage de l'éprouvette de béton et n'induit donc aucune fissuration par retrait. La méthode d'imprégnation est détaillée et des exemples d'application de la technique sont donnés. Les possibilités offertes par le couplage de cette technique d'imprégnation avec l'analyse d'images sont brièvement discutées.

Introduction

The initiation and the propagation of cracks in cement-based composites have been studied extensively over the past decades. The subject is of considerable importance for engineers since crack formation has a direct bearing on the overall performance of cementitious materials. For instance, the presence of preexisting cracks (in the bulk matrix or at the paste-aggregate interfaces) has been found to significantly influence the mechanical behavior of mortars and concretes [1-3]. Numerous studies have also clearly indicated that the development of a connected crack network contributes to increase the permeability and the diffusivity of concrete [4-6]. Such an increase of the material transport properties is generally accompanied by a substantial reduction of its durability [7, 8].

Although the process of crack formation in cement-based composites is nowadays fairly well understood from a macroscopic point of view, the microstructural aspects of this phenomenon are much less documented. The limited information on the subject might be attributed, to a great extent, to the fact that there exists actually very few techniques specifically developed to observe cracks in cementitious materials. As all high surface area porous solids, cement-based materials are extremely moisture sensitive and tend to shrink and crack upon drying. Since they require to dry the sample to certain degree, most classical microscopic observation techniques are not suited to the study of cracks in cementitious materials. If they can be used for qualitative studies, these techniques cannot provide any reliable quantitative information on the crack formation process.

Over the past decade, a great deal of effort has been spent towards developing experimental techniques better suited to the study of cracks in cement-based materials. After a brief critical overview of the recent developments in that field, a new technique to prepare sections for microscopic examination of cracks in cement-based materials is presented. The procedure involves the impregnation of polished samples with a colored dye diluted in an alcohol. In addition to a detailed description of the preparation technique, various examples of the application of the method are given. The advantages and the drawbacks of using this dye-impregnation technique in combination with other methods or with an image-analysis system are also discussed.

Background

Before reviewing the various techniques developed to study cracks in cement-based materials, the characteristics of a good technique deserve to be defined. The first quality of such a technique lies in its ability not to induce any cracks during the preparation of the sample. In that respect, any method that requires even the slightest drying should be set aside. The ideal technique should also be simple, economic, rapid and characterized by a high resolution, i.e. that it should be able to detect very fine cracks. As emphasized by RINGOT et al. [9], a good technique should permit the characterization of the initial state of the material with respect to cracking. According to this requirement, acoustic methods and laser speckles methods, that can monitor crack propagation but cannot be used to determine the initial state of the material, do not qualify. Finally, the ideal method has to be easily coupled to an image analysis system in order to yield quantitative information.

The various techniques designed to study cracks in cement-based materials can be divided into three categories: radiography techniques, replica techniques and impregnation techniques. X-radiography is probably among the oldest methods developed to study the internal structural

features of concrete. Originally introduced by SLATE and OLSEFSKI [10], the technique was later used by several of their co-workers at Cornell University to study the crack formation process of mortar and concrete samples [11]. The principle of the technique rests on the fact that the ability of materials to attenuate X-rays continuously increases with atomic number. Therefore, X-rays can be used to identify the differences in density existing between cracks and the surrounding material. Relatively thin samples (≈ 4-mm thick) of wet concrete are exposed to a X-ray flux normal to the plane of the sample. A picture of the sample is taken. On the micrograph, cracks appear as discrete locations in which X-ray penetration was greater than in surrounding areas. Although relatively simple, this method is plagued by a poor resolution. A comparative study carried out by NAJJAR et al. [12] has clearly indicated that X-radiography systematically overlooks thin cracks.

Originally used to measure the moisture content of building materials [13, 14], neutron radiography has been recently used to observe cracks in concrete [12, 15]. The principle of the technique is quite similar to that of X-radiography. A thin slice of concrete is exposed to a flux of neutron, and a picture of the sample is taken. However, since the attenuation of neutron does not correlate with the atomic number, the cracks present in the sample have to be impregnated with a solution of gadolinium nitrate prior to the test. Gadolinium is one of the few materials to have very high neutron attenuation capacities. Although this technique has been found to have a good resolution [12], its main drawback is that the concrete samples have to be air dried prior to the impregnation with the gadolinium nitrate solution. Microcracks are thus induced to the material during the preparation operations.

The replica method is probably the first high-resolution technique designed to observe cracks in concrete without requiring any predrying of the sample. First introduced in 1985 by OLLIVIER [16], the method has since been successfully used to study the crack formation process in various cement-based materials [6, 9, 17, 18]. The technique is quite simple, and consists essentially in taking a "print" (or a replica) of a polished surface. The replica is obtained by placing an acetylcellulose film on the studied surface using methyl acetate. Once the solvent is evaporated, the film is taken off and observed. The examination of the film can be easily carried by means of an optical microscope or using a scanning electron microscope since the replica is not sensitive to high-vacuum drying. The resolution of the technique is quite good since it can detect cracks with an aperture of 0,5 μm. A significant advantage of this method is that it can be used to follow crack development on the external surfaces of a loaded sample. The main drawback of the technique is that the area of the acetylcellulose film has to be limited to approximately ≈ 2 cm^2 in order to be able to peel it off. Several replica have to be done to cover a significant surface, and the method might be quite time consuming for regular quality control check ups.

Impregnation techniques have been used to assess the microstructure of cement-based materials for more than three decades. As soon as 1963, SLATE and OLSEFSKI [10] developed a dye-impregnation technique to study cracks in concrete. The technique was subsequently modified by other researchers and concrete samples were impregnated with a fluorescent dye in order to facilitate the detection of cracks [19]. Although these techniques offer interesting possibilities to investigate the microstructure and the air-void characteristics of cementitious materials [20-23], they usually require drying of the sample prior the impregnation [24].

An alternative way to impregnate concrete samples with a fluorescent dye has recently been developed by STRUBLE et al. [25] and by GRAN [26]. In order to avoid the predrying of the samples, the fluorescent dye is dissolved in an organic solvent such as ethanol. The water saturated concrete sample is immersed in a bath filled with the fluorescent dye solution, and the

pore water is slowly replaced by the colored solvent by a counter-diffusion process. After a few days of immersion (usually 4 days), the sample is taken out and excess fluorescent dye is removed by slightly polishing the surface. The technique has been found to be well suited for the study of cracks in concrete [25-27], and might even be used to determine the water/cement ratio of hydrated cement-based materials [26]. Its main drawback is that it takes many days to prepare a sample.

Description of the Dye Impregnation Technique

The new dye impregnation method presented in this report is quite similar to the technique developed by STRUBLE et al. [25] and by GRAN [26] since it relies on a solvent replacement process to impregnate the sample. However, it has been designed to minimize sample preparation time. Although this technique has been developed a few years ago [28], it has never been described in detail in any publications.

Surface Preparation. The preparation starts by cutting a 15-mm slice from a concrete element. After cutting, one surface of the sample is ground to 1000 grits in order to remove surface irregularities produced by the saw. At the end of the grinding operations, the sample is carefully cleaned to remove debris. The surface is then carefully polished to 6 μm using a diamond paste. During all the sawing and polishing operations, special care is taken to constantly keep the sample humid.

Dye Impregnation. After the polishing operations, the surface of the sample is cleaned, and gently wiped in order to remove excess water. The surface is then impregnated with a red dye diluted in an ethanol solution. The red dye comes under the form of a water-insoluble red powder (commercially available under the brand name Irgacete). To prepare the solution, 4 grams of powder are diluted in 100 ml of ethanol. In the course of the development of the technique, numerous commercially-available dye were tried. The Irgacete red powder was the one which was found to yield the best results. The red dye was chosen preferably to fluorescent dye because it tends to penetrate the hydrated cement paste porosity more readily. Several organic solvents, such as methanol and acetone, have also been tested. Ethanol was selected because it easily dilutes the red dye and facilitates the impregnation operations.

After a first impregnation, the red dye solution is let to penetrate the sample for approximately 5 minutes. Then a second impregnation is performed. After another five-minute impregnation period, the excess dye is removed by gently polishing the sample under water using a 6-μm diamond paste. The sample is subsequently polished to 3 μm and 1 μm. The polishing operations have to be carefully controlled. Insufficient polishing will leave an excess of colored dye and the cracks will not be easily discernible during the microscopic observations. On the other hand, an excessive polishing will contribute to remove the colored dye from impregnated cracks, and will affect the subsequent quantification operations.

Microscopic Observations. Direct observations of the polished sections can be made using a microscope in reflected light at 10X to 300X. The microscopic observation of impregnated samples reveals several details of the concrete internal structure. First of all, the red dye impregnation facilitates the detection of microcracks (Fig. 1). Unfortunately, color pictures can not be reproduced in this document, and the reader will not be able to appreciate the full potential of the impregnation technique. Systematic observations of impregnated concrete

samples at a magnification of 100X have clearly indicated that cracks of widths in the range of about 1 μm and less can be easily detected with this technique. The resolution depends on the type of concrete and particularly on the contrast between the cement paste and the impregnated cracks.

As can be seen in Fig. 2, the impregnation technique also permits the detection of discontinuities at the paste-aggregate interfaces. These discontinuities may either be under the form of porous zones of hydrated cement paste localized at the vicinity of an aggregate or a crack going through this porous zone. Numerous studies have clearly indicated that these discontinuities play a important role in the mechanical behavior of concrete [29, 30].

Finally, the impregnation technique facilitates the detection of porous zones of hydrated cement pastes and that of entrained spherical air voids. As previously mentioned, the red dye readily penetrates the hydrated cement paste porosity after a few minutes of impregnation. Even after the subsequent polishing operations, there always remain colored areas of greater porosity where the dye had penetrated more deeply. This capacity of the technique to detect porous zones can be used to assess the quality and the homogeneity of a given concrete mixture.

Verification of the Reliability of the Technique

In the development of the dye impregnation technique, the reliability of the method was tested in three different ways. Since a previous investigation by FELDMAN [31] had indicated that pore water solvent replacement could produce significant length changes and could eventually lead to microcracking, thin (1-mm thick) discs of hydrated cement pastes were polished and impregnated according to the procedure described above. Optical observations of the paste discs were conducted at a magnification of 100X using a conventional laboratory stereomicroscope [32].

As can be seen in Fig. 3, no visible microcrack could be detected on any of the discs observed. The absence of microcracks can not be related to an insufficient penetration of the colored dye during the sample preparation since macrodefects and air bubbles all appeared to be well impregnated.

The ability of the technique not to induce any microcracking was also checked for concrete samples. Since preliminary investigations of the initial cracking state of various concretes had

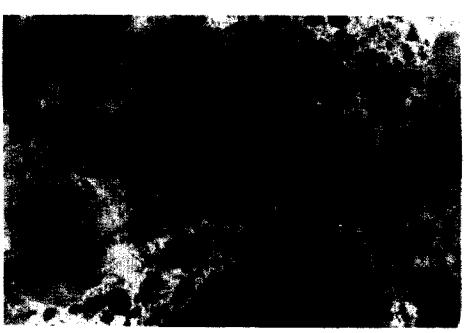

FIG. 1.
Crack impregnated with the red dye.

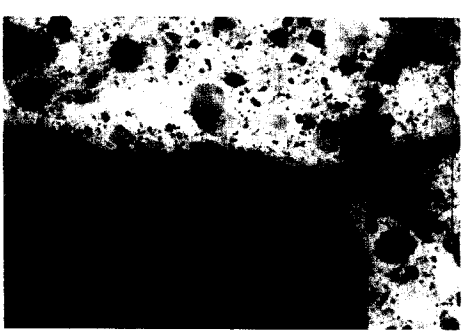

FIG. 2.
Discontinuity at the paste-aggregate interface.

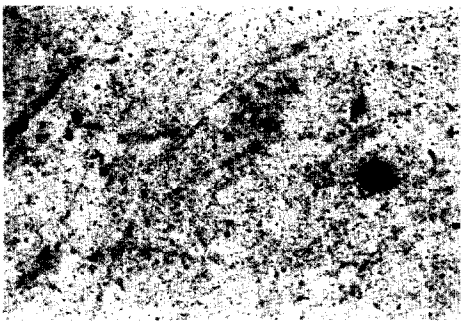

FIG. 3.
Thin paste disc impregnated with the red dye.

indicated that virgin (i.e. never dried) concretes always contained a certain (minimal) number of microcracks [32, 33], it was decided to verify if these discontinuities did not originate from the sample preparation technique itself [34].

The proportion of the concrete mixture used for this series of tests is given in Table 1. A 75 × 225 × 300 mm slab was cast and water cured for 14 days. At the end of the curing period, six 100-mm diameter cores were taken from the slab. From each core, a 10-mm thick disc was sawn and polished under water. Two discs (always kept saturated) were then impregnated according to the procedure described above. Two other discs were placed in a room kept at 20°C and 50% relative humidity and allowed to dry for 14 days. A third series of two discs were oven-dried at 40°C for 14 days. At the end of the drying period, the four discs were impregnated with the red dye.

The crack density of each impregnated disc was determined by microscopic observations at 100X. The quantification of the crack density was carried out according to the oriented secants technique originally developed by SALTIKOV [35] and described by STROEVEN [36]. Since trial observations had indicated that, for these samples, cracking was isotropic, measurements were performed for only one secant orientation. During the sample microscopic observations, cracks going through the hydrated cement paste phase were distinguished from discontinuities at the paste-aggregate interfaces. The results of the crack density determination are summarized in As can be seen in Table 2, a limited number of cracks and paste-aggregate discontinuities were observed for the virgin discs (i.e. those which had been always kept humid). Although it cannot

TABLE 1

Mixture Proportions [34]

Constituents	Proportions
ASTM type I cement	363 kg/m^3
Water	163 kg/m^3
Fine agg.	720 kg/m^3
Coarse agg.	1080 kg/m^3
AEA	18 ml/m^3
WRA	363 ml/m^3

TABLE 2

Results of the Crack Density Measurements [34]

Sample conditioning	Crack density (n/cm)*	
	Paste-aggregate discontinuities	Cracks
Virgin	0.09	0.06
Dried at 20° C	0.15	0.31
Dried at 40° C	0.22	0.35

* Number of cracks intersected per centimeter - Mean value of two discs

be totally excluded that some of these cracks were induced by the preparation technique, it is rather probable that they were already present within the material prior the sample preparation operations. This hypothesis is supported by the fact that the cracking density of the two series of discs dried at 20° C and 40° C are significantly higher than that of the virgin samples. Results of Table 2 also indicate that the crack density increases with the intensity of drying. These results are in good agreement with those of a previous investigation [32].

In a last attempt to verify the reliability of the impregnation technique, observations carried out on impregnated samples were systematically compared to observations made on replica obtained according to the method developed by OLLIVIER [16]. These comparisons revealed that if the impregnation technique is characterized by a good resolution, the resolution of the replica technique is even higher. The resolution of both techniques can be easily compared in Figure 4. As can be seen, if the vast majority of the cracks present in the material can easily be observed with both methods, the replica technique permits the detection of very fine cracks and other structural details that are not revealed by the impregnation technique.

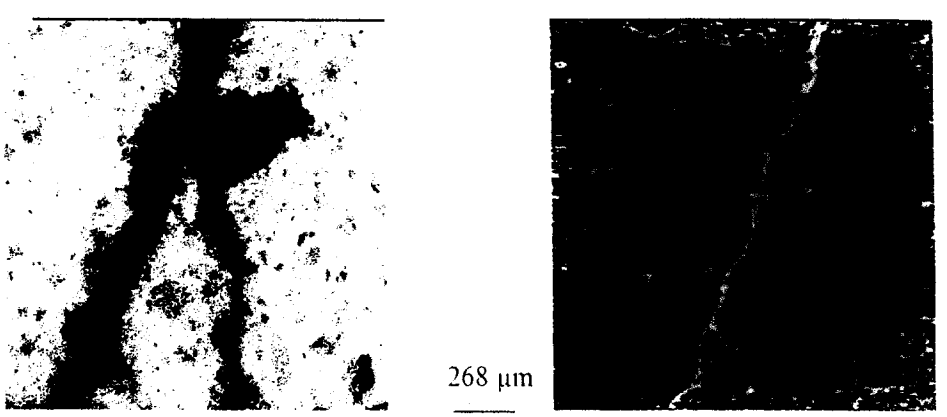

FIG. 4.

Comparison of the resolution offered by the dye impregnation technique (a) to that of the replica technique (b).

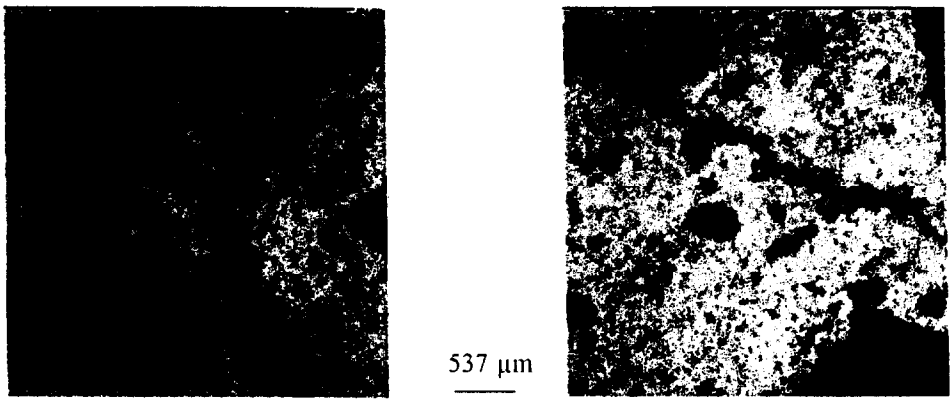

FIG. 5.
Digitized images of impregnated concrete sections.

Image Analysis of Impregnated Samples

As previously mentioned, the characterization of the microcracking state of dye impregnated samples can be easily carried out by microscopic observations using stereological methods such as the oriented secants technique [35, 36]. Although these methods are quite simple, they rapidly become tedious as soon as the number of views to analyze increases. Furthermore, the reliability of the results obtained are directly influence by the operator's experience, and results may vary significantly from one operator to another.

In order to develop an automatic procedure, the application of image analysis techniques to quantify cracks in impregnated concrete samples is presently being investigated. Digitized

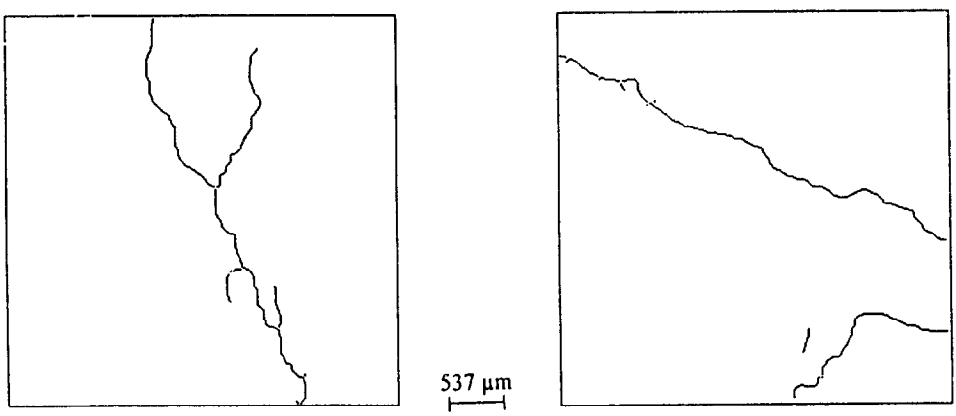

FIG. 6.
Extraction of the crack networks.

images of impregnated concrete samples are given in Figure 5. These images were acquired by a TRI CCD color camera mounted on an optical microscope. The image digitization was performed using a Matrox Image 640 image analysis system coupled to the camera.

Using the image analysis system, the microcracks appearing in a given field can be extracted after a series of operations on the binarized image (see Figure 6). These operations first involve a filtering and a binary segmentation of the digitized image. Then a series of erosion, dilation and hole filling operations are performed. The remaining objects can be classified according to their shapes. By applying appropriate shape criteria, cracks can be distinguished from porous zones and spherical air voids. Once the cracks are extracted, the quantification of the crack network can be determined using the total projection method described by RINGOT [17].

Although the use of image analysis techniques to quantify cracks in dye impregnated samples has yielded promising results, numerous problems have yet to be solved. For instance, the binary segmentation of the digitized images will have to be fine tuned. This operation appears to be particularly sensitive to local variations of the crack impregnation. These variations might be related to local defects induced to the surface during the polishing operations or to sudden modification of the crack aperture. Numerous aspects of the crack extraction also need to be improved. As previously mentioned, the red dye tends to impregnate certain porous zones. A crack going through an impregnated porous zone is generally extremely difficult to extract.

Conclusion

The red dye impregnation technique appears to be a reliable procedure to prepare concrete sections for crack observation and quantification. The technique is simple and quick. Furthermore, contrary to most dye impregnation techniques, the method does not involve any drying of the sample. Observations of cracks using optical microscopy can therefore be made without concern about drying shrinkage-induced cracking.

The use of the dye-impregnation technique in combination with an image-analysis system is presently being investigated. Although the extraction of the crack network can be in some cases problematic (especially for very porous zones deeply impregnated by the red dye), the combination of these two techniques appears to be extremely promising.

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