

## Observation of Water Penetration into Water Repellent and Cracked Cement-based Materials by Means of Neutron Radiography

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### Abstract

Service life of reinforced concrete structures is often limited by penetration of water and chemical compounds dissolved in water into the pore space of cement-based material. It is well known that cracks in concrete are preferential paths for ingress of aggressive substances. In this paper, time dependent moisture distributions in mortar in direct contact with water have been determined by means of neutron radiography. The influence of water repellent treatment on water ingress has been investigated in particular. For this purpose the surface of some mortar specimens has been impregnated with different amounts of silane gel and in addition integral water repellent specimens have been prepared by addition of silane emulsion to the fresh mix. In the second part of this paper the role of cracks in the process of water absorption has been investigated. Cracks are instantaneously filled if the surface of a cracked specimen is put in contact with water. Water from the cracks is absorbed by capillary suction into the porous space of concrete. The interface between steel and concrete is mechanically damaged in the vicinity of cracks. This damaged zone is also instantaneously filled with water from the crack. Cracks serve as water duct. They feed the crack surface and the damaged interface with water for further migration into the material by capillary suction.

**Keywords:** Cracks; Water penetration; Surface impregnation; Silane; Water repellent concrete; Neutron radiography.

### Beobachtung des Eindringens von Wasser in hydrophobierte Zement gebundene Werkstoffe mit Rissen mit Hilfe der Neutronenradiographie

#### Zusammenfassung

*Die Nutzungsdauer von Stahlbetontragwerken wird oft durch Eindringen von Wasser und darin gelöster Verbindungen in den Porenraum des Zement gebundenen Werkstoffs begrenzt. Es ist hinreichend bekannt, dass Risse bevorzugte Pfade für das Eindringen von aggressiven Substanzen bilden. In diesem Beitrag werden zeitabhängige Feuchteverteilungen in Mörtelproben beschrieben, bei denen eine Seite in direktem Kontakt mit Wasser stand. Die Verteilungen wurden mit Hilfe der Neutronenradiographie sichtbar gemacht. Der Einfluss einer Hydrophobierung auf das Eindringen des Wassers wurde insbesondere untersucht. Hierzu wurde die Oberfläche eines Teils der Mörtelproben durch Imprägnieren mit unterschiedlichen Mengen von Silangel hydrophobiert. Außerdem wurden integral (in der Masse) hydrophobierte Proben durch Zugabe einer wässrigen Silanemulsion beim Mischen der Komponenten hergestellt. Im zweiten Teil dieses Beitrages wurde die Rolle von Rissen bei der Wasseraufnahme untersucht. Es zeigte sich, dass die Risse selbst unmittelbar nach dem Kontakt mit dem Wasser gefüllt sind. Daran anschließend wird Wasser aus dem Riss in das angrenzende Material durch Kapillarkraft absorbiert. Die Risse wurden in einem Dreipunkt-Biegeversuch erzeugt. Dadurch wurde die Grenzfläche zwischen Mörtel und Bewehrungsstahl mechanisch beschädigt. Die beschädigte Zone wird auch unmittelbar nach dem Kontakt mit Wasser gefüllt. Die gefüllten Risse und die geschädigten Grenzflächen erhöhen die Fläche, die zur kapillaren Wasseraufnahme zur Verfügung steht erheblich und folglich wird wesentlich mehr Wasser pro Zeiteinheit vom Bauteil aufgenommen. Über die hohe Wasserleitfähigkeit der Risse können Wasser und Schadstoffe in kurzer Zeit bis tief in ein Bauteil gelangen.*

**Stichwörter:** Risse; Wasseraufnahme; Oberflächenimprägnierung; Silan; Hydrophobierter Beton; Neutronenradiographie.

## 1 Introduction

Cement-based materials such as mortar and concrete are extremely durable if placed in a dry environment. Under these conditions concrete from the Roman Empire has survived more than 2000 years. If cement-based materials are exposed permanently or temporarily to water, however, they may be seriously damaged in a short period. Water saturated concrete is frost sensitive. Binding compounds such as  $\text{Ca}(\text{OH})_2$ ,  $\text{CaCO}_3$ , and CSH gel may be leached out by hydrolysis. Aggressive chemical compounds may be transported deep into the pore space of concrete by capillary suction and deteriorate the porous material by accelerated leaching or by internal pressure after ion exchange. The rate of corrosion of steel in reinforced concrete depends on the moisture content. These are just a few more or less arbitrarily chosen examples of direct or indirect damaging mechanisms of water in concrete. In order to ascertain sufficient service life of reinforced concrete structures in aggressive environment we have to limit moisture ingress and moisture exchange of concrete.

Design of reinforced concrete structures allows crack formation until a critical crack width. But cracks are preferential paths for ingress of water and salt solutions. Therefore durability of cracked reinforced concrete structures depends essentially on the well-known self-healing effect. If self-healing is suppressed in one way or another, service life can be reduced considerably by the presence of cracks. So far penetration of water or chlorides or sulphates dissolved in water into cracked structural concrete elements has hardly been studied in a quantitative way.

We have applied neutron radiography to investigate moisture ingress into and moisture movement in cracked and uncracked concrete elements. First results obtained on neat concrete samples have been published recently [1, 2]. Water repellent treatment of concrete is by now a well established technology to minimize water uptake of concrete in aggressive environment by capillary suction [3, 4]. In this contribution results obtained on water repellent mortar will be described. Cracked and uncracked mortar specimens have been surface

impregnated with silane gel. In addition water penetration into integral mortar specimens has been studied. Results will be presented and discussed.

## 2 Experimental

### 2.1 Preparation of Mortar Samples

Test specimens have been prepared with one type of mortar. The composition is shown in Table 1. Ordinary Portland cement, type 42.5 (C) and local river sand with a maximum grain size of 2.5 mm from Qingdao area (S) have been mixed with tap water. The maximum grain size has been limited in order to have a representative volume in slices with a thickness of approximately 25 mm. Prismatic specimens with the following dimensions have been cast in steel forms:  $100 \times 100 \times 300$  mm.

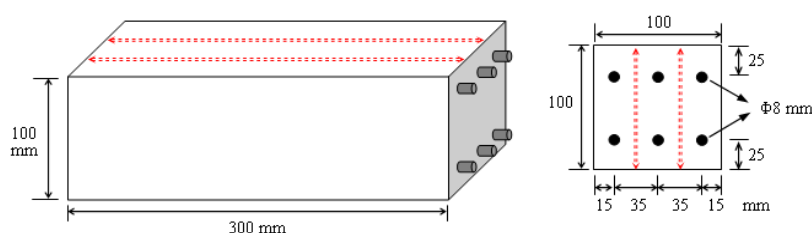
### 2.2 Water Repellent Treatment

After 14 days of moist curing ( $\text{RH} > 95\%$ ) specimens have been taken out of the curing room and placed in a laboratory with relative humidity of 60% at a temperature of 20 °C for 7 days. The bottom surface of some specimens has then been impregnated with silane gel. Silane gel consists essentially of liquid silane with fine clay added as stiffener. A well defined quantity of silane paste can be spread out on the surface. In this way sufficiently long contact between the surface and liquid silane for deep impregnation can be achieved. Two different amounts of silane gel have been applied:  $100 \text{ g/m}^2$  and  $400 \text{ g/m}^2$ . The corresponding test specimens will be designated G100 and G400 respectively. The penetration depth of silane has been measured to be 2.2 mm for G100 and 4.4 mm for G400. The silane in all surface impregnated specimens was allowed to react for seven days before further handling.

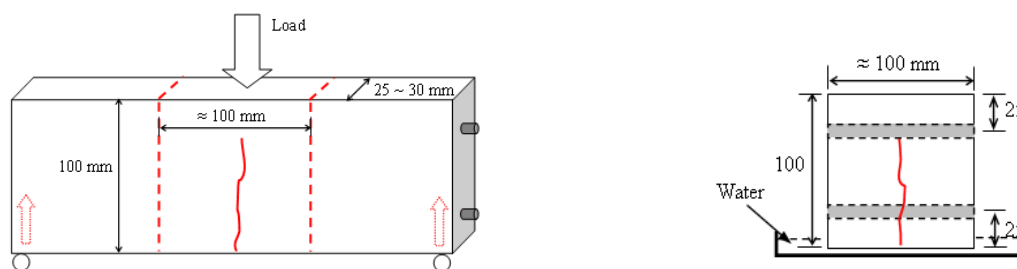
In addition to surface impregnated mortar specimens, integral water repellent mortar samples were prepared for comparison. For the production of integral water repellent samples (IW), silane emulsion (silane in water) has been added to the fresh mortar during the mixing process. Otherwise the composition of the mortar was as indicated in Table 1. Curing was as indicated for the surface impregnated samples.

**Table 1:** Composition and compressive strength of the mortar

W/C	C ( $\text{kg/m}^3$ )	S ( $\text{kg/m}^3$ )	W ( $\text{kg/m}^3$ )	Compressive strength (MPa)		
				3 days	7 days	28 days
0.6	500	1650	300	10.3	20.0	29.2



**Figure 1:** Position of the steel reinforcement in the mortar prisms and cutting lines



**Figure 2:** Formation of a centre crack under three point bending (left) and detached specimen with a crack in contact with water for observation of capillary suction (right)

Then slices with a thickness of approximately 25 mm have been cut from the original prisms. These slices were further dried in a ventilated oven at 50 °C for four days. After this drying period constant weight had been achieved. The square surfaces (100 x 100 mm) and two opposite small surfaces (25 x 100 mm) were then covered with self adhesive aluminium foil.

### 2.3 Crack Formation

Part of the mortar prisms have been reinforced with six steel bars having a diameter of 8 mm each, as shown in Fig. 1. These steel reinforced prisms were cut with a diamond saw into three slices along the long axis of the prisms (300 mm). The slices obtained in this way have been loaded by three point bending under well controlled conditions in order to induce one single crack in the centre (see Fig. 2). In the following we will call cracked specimens with a crack width of 0.4 mm and surface impregnated with 100 g/mm<sup>2</sup> silane gel G100-4. From the centre part around the induced cracks slices with a width of 100 mm have been cut as shown in Fig. 2 (right part). These slices (25 x 100 x 100 mm) have been oven dried until constant weight was achieved and then covered with self adhesive aluminium foils as described above for the uncracked samples.

### 2.4 Neutron Radiography

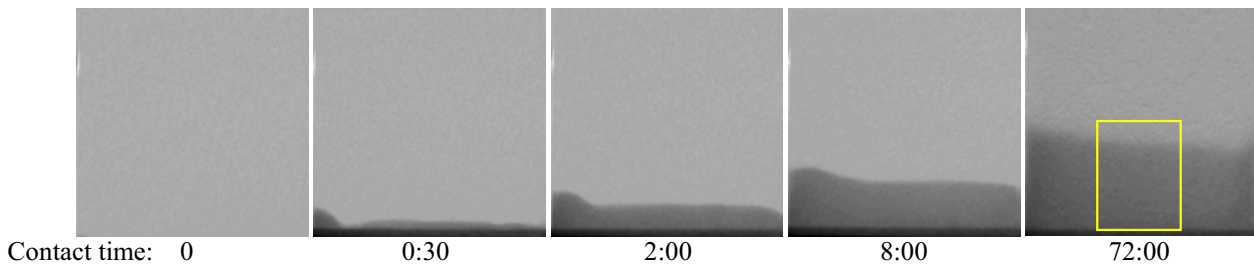
Neutron radiography has been applied by several authors to study moisture movement in mortar or concrete [5-14]. The mathematical and physical basis of the method is described in detail in [15].

The water absorption of water repellent treated and untreated mortar specimens with and without a crack has been measured as function of time by neutron radiography. All specimens were placed in contact with water in the neutron beam. The neutrons were registered with a position sensitive detector behind the mortar sample. The digital image has to be evaluated by means of transmission analysis. The mass of absorbed water has been measured on similar specimens gravimetrically in parallel.

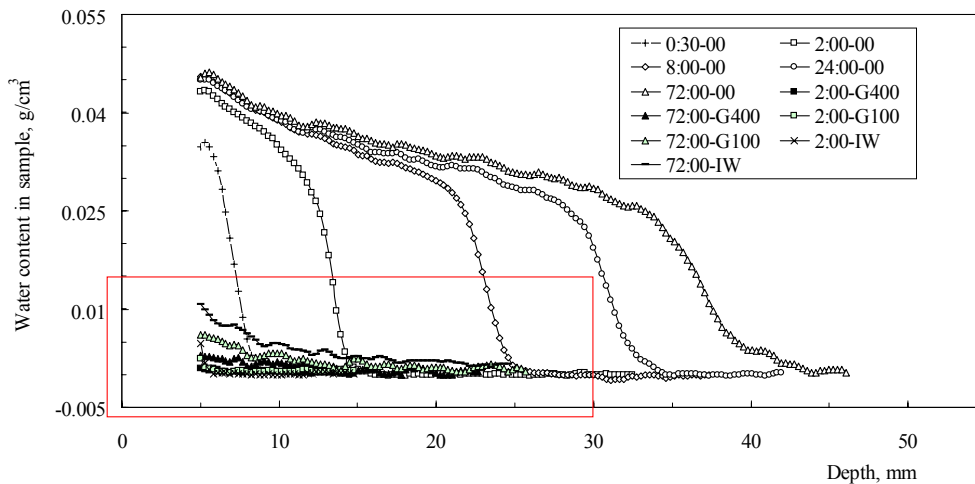
## 3 Results and Discussion

### 3.1 Water Penetration into Neat and Water Repellent Mortar

Typical results obtained on a specimen without crack are shown in Fig. 3. The suction time is indicated in hours. At time  $t = 0$  no moisture can be observed in the pre-dried sample. After 30 minutes of contact with water a penetration front becomes visible. This irregular front gradually moves into the material. It seems that the mortar samples are not totally homogeneous, penetration rate is higher in the left part of the sample. This can most probably be explained by the manufacturing process of the samples.



**Figure 3:** Direct observation of the penetration front of water into untreated mortar by neutron radiography up to a contact time of 72 hours



**Figure 4:** Water penetration profiles as determined on water repellent (G100, G400 and IW) and untreated mortar specimens (00) by neutron radiography after contact time with water of 0.5, 2, 8, 24 and 72 hours

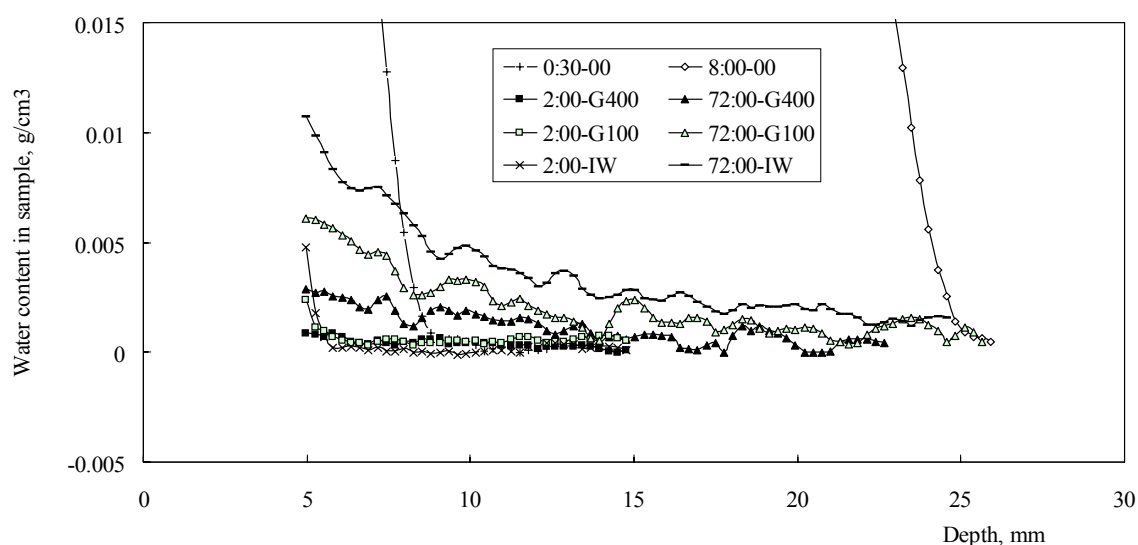
Water uptake of the surface impregnated and the integral water repellent samples could not be observed with the naked eye. The time-dependent moisture profile, however, could be determined by transmission analysis.

The digital images as obtained on untreated and on water repellent mortar have been further evaluated by means of earlier developed software IDL. In this way penetration profiles as shown in Fig. 4 have been obtained. For the determination of the profiles the quasi homogeneous centre part of the specimens, i.e. the rectangular area marked in Fig. 3, has been selected. The resulting profiles are shown in the following figure 4.

It is obvious that untreated mortar absorbs a big amount of water in a comparatively short time. The penetration depth reaches a value of about 38 mm after 72 hours. This situation corresponds in practice to a structural element, which is exposed to driving rain for 72 hours. From Fig. 4 we can see

immediately that water repellent mortar absorbs a small fraction in the same time only and the penetration depth remains limited. In order to be able to distinguish the water uptake of the differently treated mortar samples the part in Fig. 4 marked with a rectangular frame has been re-plotted on bigger scales in Fig. 5.

From Fig. 5 we learn that surface impregnation with  $400 \text{ g/m}^2$  is an effective moisture barrier. Even after 72 hours of direct contact with water, traces of water can be observed in the porous material only. In this case the penetration depth of the water repellent agent was measured to be 4.4 mm. Obviously no liquid water can penetrate these samples but water vapour migrates through the open pores and will be captured by surface adsorption and capillary absorption in the untreated zone. If  $100 \text{ g/m}^2$  are applied instead of  $400 \text{ g/m}^2$  the thickness of the water repellent layer is 2.2 mm. In this case an increased amount of water vapour can penetrate into the porous space of mortar. The integral water



**Figure 5:** Moisture distribution in water repellent samples after different durations of contact between the surface of the samples and water (see marked area in Fig. 4). For comparison lower parts of the profiles measured on untreated mortar samples after 2 and 8 hours are shown again

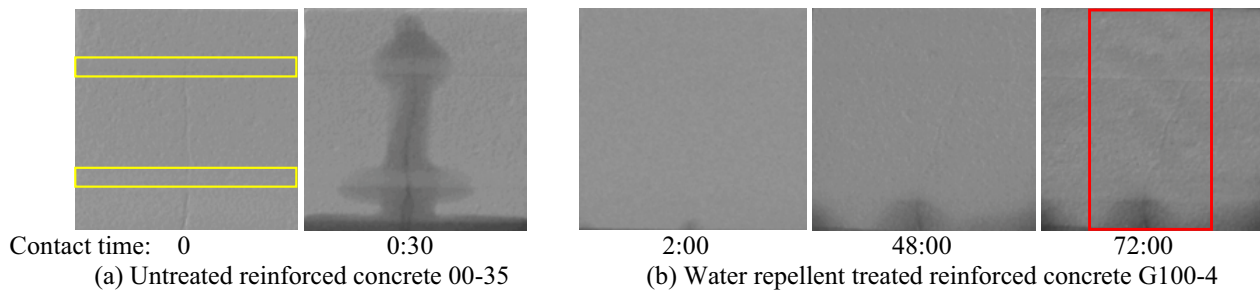
repellent mortar absorbs under the chosen conditions the biggest amount of water but as can be seen from Fig. 4 it is still a small amount compared to the water taken up by untreated specimens. These results are in good agreement with recent measurements of chloride penetration into concrete. Chloride penetration into integral water repellent concrete is significantly reduced but correct surface treatment leads to a more efficient chloride barrier [3, 15].

### 3.2 Water Penetration into Water Repellent and Cracked Mortar

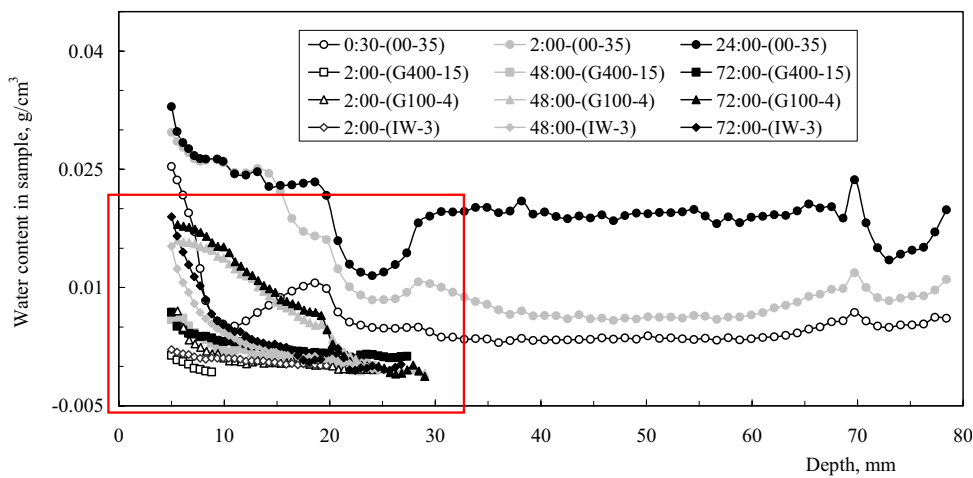
In this section we will present results obtained on mortar samples with a centre crack. In order to be able to induce central cracks under controlled conditions steel reinforcement has been placed in the mortar prisms (see Fig. 2). The position of the steel reinforcement is marked in the neutron image of the dry specimen ( $t = 0$ ). Typical images of the time dependent moisture distribution as obtained by neutron radiography on cracked discs are shown in Fig. 6. In Fig. 6 (a), the left part of Fig. 6, the moisture distributions in an untreated cracked specimen before and after contact with water for half an hour are shown for comparison (see also [1, 2]). The width of the crack mouth in this case was 0.35 mm. It was found that cracks with a width  $w \geq 0.1$  mm are filled with water immediately after contact of the surface with water. Further research is needed to find the maximal tolerable width of cracks, which will not lead to immediate filling of the crack with water.

Once the crack is filled, water is migrating normal to the crack surface into the porous material under the influence of capillary action. Water also penetrates into the fracture process zone (fictitious crack) ahead of the real crack. Capillary suction and water capacity in the fracture process zone have increased due to micro-crack formation [1, 2]. It is of particular interest to learn that water also penetrates immediately into the damaged interface between steel reinforcement and mortar. From the water filled interface water migrates further into the surrounding mortar. The centre crack and the interfaces serve in this case as water reservoir which remains filled by continuous transport of water from the outside by capillary action. With respect to durability of reinforced concrete structures this efficient transport mechanism is of particular importance if salt solutions such as sea water are taken up by cracked reinforced concrete elements.

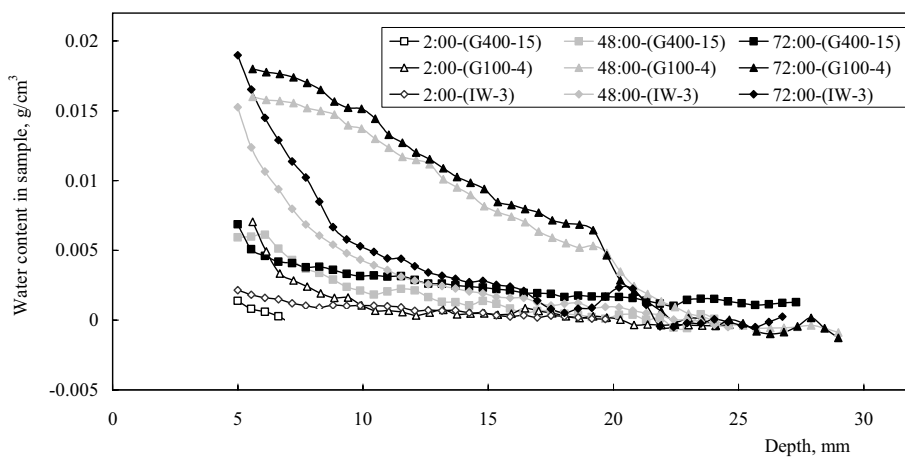
In Fig. 6 (b) three digital images as obtained on a mortar disc with a centre crack of 0.4 mm and treated with  $100 \text{ g/m}^2$  of silane gel are shown. The contact time is indicated in hours. In comparison with the untreated sample very little water is taken up by the surface impregnated sample G100-4. Samples impregnated with  $400 \text{ mg/m}^2$  of silane gel and a crack width of 0.15 mm (G400-15) as well as samples produced from integral water repellent concrete and a crack width of 0.3 mm (IW-3) the water distribution could not be observed visually from the images.



**Figure 6:** Time-dependent water distribution in the vicinity of a crack in untreated (left) and water repellent samples (right)



**Figure 7:** Time dependent moisture distribution in water repellent (G400, G100, and IW) and untreated (00) cracked reinforced mortar as observed by neutron radiography



**Figure 8:** Moisture distribution after different time in cracked water repellent treated reinforced concrete

The obtained digital images shown in Fig. 6 have also been evaluated by means of the computer program IDL. The rectangular area, which has been selected for the further evaluation is marked in Fig.

6 (b). This detailed evaluation provides us with quantitative moisture distributions. Results are shown in Figs. 7 and 8.

The induced crack in the untreated specimen with a centre crack with a crack mouth opening of 0.35 mm (00-35) is instantaneously filled when the surface is put in contact with water. After contact with water for 30 minutes the capillary absorbed water has already reached the upper surface of the specimen. Later the overall moisture content in the sample increases as water is absorbed normal to the crack surface. The striking difference of water penetration into water repellent mortar is obvious from Fig. 7. In order to visualize the differences in more detail, the marked area in Fig. 7 is shown again with increased scales in Fig. 8.

If the surface of mortar specimens has been impregnated with 400 g/m<sup>2</sup> of silane gel and the crack mouth opening is 0.15 mm (G400-15) hardly any water is absorbed even after a contact time of 72 hours as shown in Fig. 8. In case the surface of mortar has been impregnated with 100 g/m<sup>2</sup> and the specimen has a crack with a crack mouth opening of 0.4 mm (G100-4) the crack below the first layer of reinforcement absorbs a certain amount of water. This combination cannot be considered to be a reliable barrier for chloride ingress. The integral water repellent mortar with a crack mouth opening of 0.3 mm (IW-3) allows water to penetrate into the porous material up to approximately 15 mm after contact with water for 72 hours. This may be considered to be at least a temporary protection of reinforced concrete elements in aggressive environment.

More research is needed to identify safe combinations of crack mouth opening and thickness of water repellent layer. This corresponds to combinations of crack mouth opening and amount of silane emulsion added for integral water repellent concrete.

#### 4 Conclusions

From the results presented and discussed in this contribution it can be concluded that:

- Neutron radiography is a powerful method to observe quantitatively moisture distributions and moisture movement in porous materials such as mortar and concrete.
- Capillary suction of concrete after surface impregnation with silane gel and of integral water repellent concrete is substantially reduced.
- Cracks in concrete are instantaneously filled with water whenever the surface of the cracked material is put in contact with water.

- Water migrates out of the crack into the adjacent porous material due to capillary action.
- The fracture process zone ahead of a real crack is a mechanically damaged zone. Capillary suction is enhanced by the presence of micro-cracks in the fracture process zone.
- The interface between steel reinforcement and concrete is damaged in the vicinity of a crack. Water penetrates from a water filled crack into the damaged interface.
- The synergetic influence of mechanical damage and moisture movement on durability and service life of reinforced concrete structures can be studied in detail by means of neutron radiography.

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