OPTICAL AND ELECTRON MICROSCOPY ON THE MICROSTRUCTURE OF TRADITIONAL AND SELF-COMPACTING CONCRETE

V. Boel and G. De Schutter

Magne Laboratory for Concrete Research, Ghent University, Ghent, Belgium

Abstract
Self-compacting concrete (SCC) can be placed without any compaction. Because of this, some health risks as well as environmental problems could be avoided. The two essential properties of SCC are a high flowability and a high segregation resistance obtained by a large amount of fine particles and the use of superplasticizers. Self-compacting concrete and traditional concrete (TC) are based on different mix designs. As such, a different pore and microstructure of the cement matrix might occur causing a different durability. By means of optical microscopy and electron microscopy the microstructure of SCC and TC has been investigated and compared. The influence of a higher water or cement content has been investigated as well. The results are obtained from several mixtures of SCC and TC. The overall conclusion is that SCC has a more dense microstructure than TC and also the interfacial transition zone seems to be more dense.

1. INTRODUCTION
A lot of knowledge about composition and workability of SCC has been gathered, however there are still questions regarding long-term durability due to significant difference in the mix proportions of SCC in comparison to traditional concrete. There is an important relation between the ‘pore structure’ of solids, fluid transport properties and degradation. If the pore structure of SCC turns out to be different from traditional concrete, some changes in durability behaviour might be expected as well.

The transport properties and the degradation of several mixtures of hardened self-compacting and traditional concrete have already been investigated as a part of an extensive research program [1, 2]. By means of mercury intrusion porosimetry (MIP) the pore structure of 22 mixtures of hardened cement paste has been studied (16 self-compacting and 6 traditional pastes). Besides the difference between self-compacting and traditional paste, several parameters were studied: water/cement, cement/powder and water/powder ratio, type of cement and filler (limestone and quartzite). Due to the presence of the filler, the pore structure of self-compacting paste turns out to be denser than traditional paste and the water content is a very important parameter. A theoretical calculation of the capillary porosity is linked with the experimental results obtained by MIP and gives good results [2, 3]. In this
paper it is checked whether the dense microstructure and improved ITZ can also be noticed by optical and electron microscopical analysis.

2. MIXTURE DESIGN

Mixtures SCC1 and TC1 are the reference mixtures of respectively self-compacting and traditional concrete. Both mixtures have the same cement content (360 kg/m³) and the same W/C (0.46). Portland cement type CEM I 52.5 N, according to EN-197-1 Standard Specification, has been used for these reference mixtures. In order to obtain the self-compacting properties, limestone filler (240 kg/m³ Betocarb P2) has been added together with a polycarboxylic ether (Glenium 51).

3. EXPERIMENTAL PROGRAMME

3.1 Optical microscopy

The analysis on thin slices of self-compacting and traditional concrete have been performed by means of two types of microscopes: Zeiss Axiosplan 2 (VITO: Flemish Institute for Technological Research) and Leica DM LP (MagneL Laboratory). The results obtained by both apparatus are summarized in this paper. The concrete samples are impregnated with a fluorescent epoxy, all voids including the capillary pores are filled. The thickness of the slices is 25 μm. First of all the reference mixture SCC1 has been investigated thoroughly. Analysis of the cement matrix confirms the use of Portland cement with the addition of a filler, mainly containing lime and calcite grains. The lime and calcite grains which are clearly visible have a diameter within the range of 40 μm to 120 μm. In fact, from sieve diagrams it is known that the matrix contains even smaller limestone grains till sizes of 1 μm as well. In Figure 1(a) some anhedral grains of belite C₂S (1) and subhedral grains of alite C₃S (2) are clearly visible beside the angular fragments of calcite grains and rounded fragments of lime grains (3). The photograph has been taken under plane polarized light. In Figure 1(b) and 1(c) photographs are presented which are taken under fluorescent light. From these images it can be seen that the capillary porosity of the cement matrix is not homogeneously/uniformly distributed which could indicate an heterogeneous hydration. In some cases there is a minor increased capillary porosity in the direct vicinity of an aggregate. This could be due to an irregular moistening of the aggregates. Some of the aggregates are rather porous and admit the cement paste to intrude into the aggregate. The overall conclusion however is, that from the low (green) intensity it can be concluded that the cement matrix is dense. Regarding the texture, the adhesion between cement matrix and aggregates seems to be optimal. No defects are noticed and there is a complete lack of shrinkage cracks.

Several photographs are made in order to compare the (green) intensity of the cement matrix of a self-compacting SCC1 and a traditional concrete mixture TC1. It can be clearly seen that the capillary porosity of TC1 is higher. Also the interfacial transition zone between aggregate and cement matrix (ITZ) is more porous compared to SCC1. Speaking about capillary porosity it has to be mentioned that the paste content of a self-compacting concrete is higher compared to a traditional concrete. As such, it is possible to obtain a similar capillary porosity of both concrete types although the microstructure of the self-compacting concrete is more dense. As an example, the paste content of SCC1 amounts 39 % and of TC1 29 %, the capillary porosity of both mixtures is more or less the same.
A thin slice of SCC1 has also been studied on the presence of air voids. The air voids are mainly circular and occur in dimensions between 60 μm and 3.2 mm. On occasion locally there is a presence of overdimensioned air voids, probably formed by merging of several air voids. The air content seems to be considerably low. A more detailed study on the air void system has been performed on larger samples by means of automatized air void analysis [2].

Typically under plane polarized light and crossed nicols, a cement matrix looks “dark”. When carbonation has occurred the cement matrix is brownish due to the presence of CaCO₃. When a thin slice of a traditional concrete is compared to one of a self-compacting concrete, one can see that the cement matrix of the self-compacting concrete looks “brownish” (Figure 2, magnification 20 x). When someone does not know the actual composition of the concrete, the conclusion could be that the cement matrix is carbonated. The limestone filler used in these mixtures consists of 95 % CaCO₃. Due to the important amount of limestone filler, 44 volume % of the total volume of powder, there is already a significant amount of CaCO₃ present in the non-carbonated concrete.

In order to determine if the material really is carbonated one should look for the non-presence of portlandite Ca(OH)₂. When the cement matrix is carbonated, the phases of Ca(OH)₂ are converted into CaCO₃, causing a brownish color as mentioned before. The presence of Ca(OH)₂ has been investigated for several mixtures of self-compacting and
traditional concrete. From this it was concluded that portlandite CH was present in all mixtures, SCC as well as TC. However, there is an important remark to make. When SCC1 is compared to TC1, the same W/C and cement content, the amount of formed CH should be more or less the same. This is confirmed by thermogravimetric analysis on hardened cement paste, self-compacting as well as traditional [2]. During the hydration processes portlandite is typically formed where space is available to deposit. Knowing that the paste volume of SCC1 is higher compared to TC1 it is obvious that for SCC1 the amount of CH per unit volume of paste is smaller. Besides the microstructure of SCC1 is denser compared to TC1, which means that the available space for CH to deposit is smaller. For these reasons, CH appears in smaller concentrations and in smaller amounts per unit of paste volume. This was noticed on the photographs made under plane polarized light and crossed nicols. The small concentrations make the CH phases less clearly visible during the microscopic analysis (Figure 2). Moreover, the ITZ of the traditional concrete is more porous, which causes a lot a favourable space for CH to deposit. From a range of self-compacting mixtures, it was concluded that the higher the W/C and water content, the higher the concentrations of CH are. Higher concentrations enhance the visibility of the CH phases by microscopic analysis. From all this it can also be concluded that the amount of carbonatable material can be calculated in the same way for self-compacting concrete as it can be for traditional concrete. This is of importance when is comes to the assessment of the carbonation depth of concrete [1].

3.2 Backscattered electron microscopy

Compared to light microscopy, BSEM makes it possible to have a more detailed look on the microstructure. All images presented in this paper have been made by means of an Philips-XL30-ESEM apparatus. Due to the differences in atomic number various structures in concrete can be discerned. The distinction between different phases is based on threshold values of the grey values. In the cement matrix, typically the following phases can be noticed: unhydrated cement, calcium hydroxide, calciumsilicate hydrate, pores, .... When limestone filler is present as well in the cement matrix, it becomes a lot more difficult to define the threshold values for the different phases. Besides, a minor change in threshold value can lead to significant other proportions of the phases. For this reason the study of SCC, containing an important amount of limestone filler, is confined to merely a descriptive study rather than a “quantitative” study.

First of all, mixtures SCC1 and TC1 are compared to each other. In Figure 3(a) the ITZ of TC1 between a gravel grain (left bottom corner) and the cement matrix can be seen. In the cement matrix several sand grains are present, observable as dark grey elements. The aggregates are porous, but the voids can be considered isolated. As such, their contribution to the transport properties of concrete can be neglected. In the cement matrix some micro cracks can be observed. The cracks form connections between the sand grains and are probably due to the hindered shrinkage of the cement matrix. On the spot of the ITZ there is a strong perceptible increased porosity. Along the perimeter of the gravel grain there is hardly any attachment of the cement matrix. This transition zone forms an ideal canal for the transport of fluids. Mostly around the sand grains there is no attachment at all of the cement matrix. On places where the aggregates are packed closely together, the cement matrix is more porous than the bulk cement matrix. There is a clear presence of non-hydrated cement grains, small as well as large grains. In the cement matrix the macro pores are visible and they seem to be evenly distributed. In Figure 3(b) sand grains inbedded in the cement matrix are presented.
Besides all this also a higher concentration of CH is found on the places where the interfacial transition zone is more dense. Portlandite typically deposits where space is available, namely porous zones. Also on this figure it can be noticed that the cement matrix turns out to be more porous when it is positioned in between aggregate grains which are closely packed to each other. In some places even there is no cement matrix at all.

Figure 3: BSEM-images (a) and (b) of TC1, magnification 125x

The images in Figure 4(a) and 4(b) are taken from a sample originating from SCC1. No micro cracks can be observed. On both images some large voids are noticeable. These voids are surrounded by a dense cement matrix. In the ITZ located at the large aggregate grain, the cement matrix is better attached to the grain than it was in the case of TC1. Also the sand grains seem to be better surrounded by the cement matrix in the case of SCC. The presence of limestone grains and non-hydrated cement is clearly visible.

Figure 4: BSEM-images (a) and (b) of SCC1, magnification 125x

Several images have been analysed originating from different SCC mixtures. From these analysis it followed that there is indeed an interfacial transition between aggregate grains and cement matrix. The extent and structure of this transition zone is depending on the type of concrete and the amount of water present in the initial fresh concrete. Compared to traditional concrete, the cement matrix of self-compacting is more dense and the ITZ does not differ as much from the bulk cement matrix. This is probably due to less bleeding and the filling effect of the limestone filler. Also the lack of vibration influences in a good way the microstructure. As the W/C ratio increases the extent and the porosity of the ITZ increase as well. For all mixtures the content of non-hydrated cement grains lays within the expectations based on the initial amount of cement in the concrete composition. Namely, large concentrations at larger initial cement contents.
The question is whether the ITZ zone is still as important for SCC compared to TC when it comes to the transport properties. The importance of the transition zones is depending on the denseness of the intervening cement matrix. When the matrix has a dense structure, it is difficult for the potential aggressive agentia to move from one transition zone to another one. Besides the agentia do not purposively move from one zone to another. In self-compacting concrete even, the paste volume is larger which reduces the chance to meet transition zones. This interpretation leads to the thought that the transport properties and durability aspects of self-compacting concrete can be linked very well to the capillary porosity of the cement matrix, without taking into account the interfacial transition zones. This statement has been confirmed yet in [1, 2]. It was found that the relationship with the capillary porosity is better in the case of SCC than of TC. This can be explained by the improved microstructure of SCC, as mentioned before.

4. CONCLUSION

Analysis by means of optical microscopy on SCC1 reflects an optimal adhesion between the cement matrix and the aggregates. No defects or plastic cracks can be observed. The macro porosity is due to the presence of small air voids. Some of the aggregates are porous and enable the cement matrix to locally and partly penetrate into the grain. The biggest part of the cement matrix is very dense. Due to the presence of an important fraction of limestone filler the cement matrix looks brownish under polarized light with crossed nicols, even when no carbonation has yet occurred. This is in contrast with traditional concrete where the cement matrix is observed as dark. The amount of CH, development during the hydration process should be more or less the same for traditional concrete as for self-compacting concrete. In self-compacting concrete the paste content is larger which leads to a lower amount of CH per unit volume of cement matrix at the same initial cement content. The space available for CH is smaller and more spread over the cement matrix. As such the CH is present in smaller concentrations.

By means of BSEM-images it is concluded that there is an interfacial transition zone (ITZ) between aggregates and cement matrix. The size and structure of these zones are depending on the type of concrete and the initial amount of water. A higher W/C ratio increases the extent and porosity of the ITZ. The content of non-hydrated cement grains is within the expectations based on the concrete compositions.

REFERENCES