Working Groups 1 & 2: Minutes of kick off meeting 12th April 2018, UGent.

It was decided that Working Groups 1 and 2 would merge in the first instance, before maybe splitting later on once their respective foci became more clear.

WG	**topic**	**chairs**
WG1	Correlation between atmospheric carbonation and carbonation induced by accelerated testing at high CO2 concentrations	Barbara Lothenbach, Elke Gruyaert/Philip Van den Heede
WG2	Effect of SCMs on natural and accelerated carbonation of blended Portland cements	Karen Scrivener, Leon Black, Stefanie van Greve-Dierfeld

Name	Affiliation	email	expertise	facilities for carbonation	facilities for characterisation / testing
Stefanie von Greve-Dierfeld	TFB, Zurich, Switzerland	Stefanie.vonGreve@tfb.ch	Research fellow and technical consultant, concrete durability, concrete technology, modelling	6 Carbonation chamber HxLxW ~ 2x1x1m; 3 Chamber ~1x1x1 m: ~ 0.001 < CO_2 < 5%, 0 < RH < 100%, T = const. = 20°C Usually 1 chamber (2x1x1m) is for preconditioning 57% RH and 0.001% CO_2 and 1 chamber (2x1x1m) for carbonation testing according to SIA262/1 at 57% RH and 4 Vol.-% CO_2 All other chambers are used for preconditioning to specific moisture equilibrium conditions and/or for carbonation research 3 Stevenson-Screens for outdoor storage: Monitoring: RH, CO_2, T	XRF IC Thin section preparation and analyses (SEM and EDX resp. REM/EDX with facilities out house) TGA Oxygen and air permeability Mechanical testing Electrochemical testing Physical testing pH of powder suspension (preparation and measurement without or with nitrogen environment)
Leon Black	University of Leeds, UK	l.black@leeds.ac.uk	Analytical chemistry. Microstructure of cement Low-carbon cement, GGBS Durability Raman spectroscopy, SEM	1 chamber. Ambient temperature, RH controlled by saturated salt [CO2] 1-20% 4 controlled temperature and humidity cabinets (-30-90°C, 10-98% RH)	Environmental SEM-EDX Micro-CT TGA-MS XRD FTIR Solid-state NMR (Al, Si) Mechanical testing Air permeability
Francois Avet	EPFL, Switzerland	Frfancois.avet@epfl.ch		Glove box, natural carbonation with different RH	TGA, XRD, DVS, MIP, transport properties
Doug Hooton	University of Toronto, Canada	hoton@civ.toronto.ca		drying shrinkage setup (50% RH) and glove box	TGA, XRD, pore solution analysis, thin sections, DVS, MIP, transport properties
Tung Chai Ling (Bill)	Hunan University, China	tcling@hnu.edu.cn		CO2 chamber	
Maciej Zajac	Heidelberg Cement, Germany	Maciej.Zajac@heidelbergcement.com		CO_2 chamber (0-5%)	fully equipped lab for concrete analysis
Siham Kamali-Bernard	INSA, Rennes, France	siham.kamali-bernard@insa-rennes.fr		CO_2 chamber (0-20%, T and RH controlled)	TGA, XRD, SEM-EDX, mechanical incl. creep

Ivan Ignjatović	University of Belgrade, Serbia	ivani@imk.grf.bg.ac.rs	Analysis of concrete structures; Durability of concrete with and without SCMs (fly ash, slag)	CO_2 chamber (T and RH controlled)	mainly mechanical properties
Hanne Vanoutrive	KU Leuven, Belgium	hanne.vanoutrive@kuleuven.be			
Elke Gruyaert	KU Leuven, Belgium	elke.gruyaert@kuleuven.be	Assistant professor, structural elements with different types of concrete	CO2 chamber	FTR, MIP, TGA
Cesar Medina Martinez	Universidad de Extremadura, Badajoz, Spain	cmedinam@unex.es	Natural and accelerated carbonation Concrete, mortar and pastes with SCMs from several industrial wastes (CDW, clay - based materials, mining waste …	Natural carbonation	DTA/TGA, FT-IR XRD MIP 29Si and 27Al MAS NMR (solid state) BSE/ SEM-EDX, Mechanical properties Physical properties Gas permeability
Natalia Alderete	Ghent University, Belgium	NataliaMariel.Alderete@UGent.be	Concrete and mortar with SCMs, microstructure	2 chambers: 1 and 10% CO2	TGA, XRD, DVS, MIP,thin sections, SEM, mechanical testing, gas permeability, CT scan (cooperation with another department)
Charlotte Thiel	Technical University of Munich, Germany	charlotte.thiel@tum.de	10 years research experience in the field of durability of concrete and concrete technology	chamber can be set at low near natural concentrations; other chamber at 2% CO2 (German standard)	Si, Al, H NMR, MIP, N2 absorption, laser ablation, FTIR, Microscopy, gas permeability, SEM-EDX, climate chambers (can be flooded with N2, CO2 20-100 vol-%, RH 30-90%).
Kosmas Sideris	Democritus University of Thrace, Komotiní, Greece	kksider@civil.duth.gr		1% CO2, varying T and RH possible but usually, 20°C and 65% RH (for repair mortars)	TGA XRD MIP
Nele de Belie	Ghent University, Belgium	Nele.DeBelie@UGent.be		2 chambers: 1 and 10% CO_2	TGA XRD (quant) SEM-EDX, µCT, thin section microscopy, gas permeability, DVS
Susan Bernal/ John Provis	University of Sheffield, UK	s.bernal@sheffield.ac.uk		Chamber CO_2 1% up to 20% variable RH adjusted by saturated solutions	TG-MS, XRD, XRF, ESEM-EDX, MIP, BET, FTIR and Raman; in-house access to solid state NMR and LA-ICP-MS

Outputs

1. Systematic review of the factors influencing carbonation / carbonation resistance
2. Systematic review of tests to determine carbonation / carbonation resistance.
3. Round robin to assess carbonation resistance under accelerated and natural carbonation conditions of control specimens
4. Round robin to compare and contrast different tests for determination of carbonation / carbonated zone

Discussion

Starting point of the discussion was, to perform a round robin test, where different mixes are tested in order to assess the carbonation resistance. Herein it was identified, that in the first instance a systematic review of (a) the factors influencing carbonation resistance and of (b) test conditions is required.

(1) Systematic review of the factors influencing carbonation (-resistance)

The aim was, to perform a round robin test on carbonation resistance of different types of mixes (binders, w/b, aggregate, paste/mortar or concrete). It was agreed that there were still a number of questions regarding the factors influencing carbonation. There was some discussion to identify what are the key parameters controlling carbonation. The parameters mentioned were;
Influencing Parameter:

- CO_2 concentration (some discussion as to the "tipping point" where there is a change in mechanism).
- Relative humidity (50-70% was considered optimum for maximum carbonation dependent on porosity resp. water saturation degree Sd, but this is dependent on the maturity of the cement, with 85% being optimum for 3 day old samples, as well as from composition (mainly saturation degree (w/c, type of binder at specific RH))
- Temperature
- Mix design: w/b ratio, binder type (SCM type and replacement level: GGBS, FA, SF, MK, Calcined clay was discussed), respectively w/CaOreactive resp. degree of hydration and CaO content, phase assemblage, e.g. portlandite content
- $W/CaO_{reactive}$
- aggregate type and size, effect of ITZ
- curing time (degree of hydration/maturity because this controls permeability, porosity)
- compressive strength

Additionally it was decided, to review the following properties and property changes due to carbonation respectively effects of carbonation

- porosity and porosity changes (gas permeability, CO_2 diffusion, blockage of surface / calcite precipitation)
- microstructure and phase assemblage of different types of SCM
- weight changes
- release of water
- a review which parameter are needed for modelling (different model types / parameters)

<u>SCMs</u> people are working on at the moment: FA, BFS, limestone, calcined clays, bottom ashes, copper slag, RCA

It was agreed that the Working groups should together identify the key issues regarding carbonation and aim to produce a systematic review of the various parameters. People can self-nominate to prepare a short summary of the impact of one or more of the parameters. These can be presented in Delft at the next meeting.

The aim should then be to produce a journal article in Materials and Structures based on these findings. With a typical word limit of 8000 words and a maximum of 15 tables and figures, the precise length of each section will be defined by the number of parameters identified, but it will be about 500 words per parameter. If it appears that a comprehensive review is not possible within this word limit then we should consider approaching the Editorial Board to see if a slightly longer submission could be made.

Reference style of Materials and Structures should be followed. Decide on a software to be used like EndNote or Mendeley.

Parameter affecting carbonation and carbonation progress	Name
Mix design and effect on carbonation resistance as well as e.g. on Ca/Si carbonation of Portlandite/C-S-H, porosity, precipitation of calcite, pH (sound sample, partly carbonated, carbonated)	
$W/CaO_{reactive}$ resp. CaO content and age (degree of hydration) W/C	
Grain size: paste / mortar / concrete	
Other aggregates (type of aggregate, recycled aggregate, lightweight aggregate (?) or additions SAP, PP, SF)	
Curing and its effect on carbonation of concretes with different SCM / mix designs see above	
Effect on carbonation resistance	
on porosity and pore size distribution	
Interaction with CO_2 concentration (Ca/Si, calcite precipitation)	
Constant conditions: acceleration / slowing / in dependency of SCM / changes with time in porosity / portlandite content / aging	
Relative humidity/ degree of saturation effect on aging (Curing)	
Temperature	
CO_2 concentration and partial pressure	
Concrete / mortar / paste properties/characteristics affecting carbonation resistance of different SCMs	
Porosity and pore size distribution (uncarbonated/ partly carbonated/ carbonated)	
Permeability/ CO2 diffusivity (uncarbonated/ partly carbonated / carbonated)	

Test conditions currently applied:	Name
Temperature	
Relative humidity	
CO_2 concentration	
pH indicator, pH at color change, effect of e.g. temperature, moisture, pore solution on color change	
measurement points in time, amount of measurements	
Sample geometry, amount of samples (1D transport, radial transport, 2D…)	
data evaluation/ determination of carbonation resistance,	
standards	
preconditioning and curing time (majurity)	

ACTIONS:

- Consider whether the list of parameters above is comprehensive (it isn't), and suggest further additions. (ALL)
- Self-nominate to prepare a review of one or more parameters (ALL)

(3) Systematic review of tests to determine carbonation depth/extent of carbonation.

The presentations on April 11th showed that, while a number of methods have been used to assess the extent of carbonation, there is still some uncertainty over the lack of consistency between different methods. For example, while both phenolphthalein and thymolphthalein have been used as pH indicators to reveal carbonated zones in concrete, the results are not always consistent. The colour change for thymolphthalien occurs at pH 9.4, while for phenolphthalein it is 8.2. CT said that she had found systematic differences between the two indicators. While LB said otherwise, he has since checked and found that thymolphthalein showed slightly greater carbonation depths.

The presentations also showed that characterisation of carbonated samples by TGA and XRD showed discrepancies, with TGA generally revealing greater quantities of carbonates. This was possibly attributed to amorphous calcium carbonate. LB said that recent findings suggested that amorphous calcium carbonate was a product of carbonation of C-S-H. There were also some comments regarding the use of MIP to show carbonated zones.

This all suggested that there is a need for a second systematic review, running concurrently with the first, to assess the various methods used to probe carbonation and to assess the extent to which different methods gave supportive or contradictory results.

The methods identified were:

- thymolphthalien staining
- phenolphthalien staining
- alternative indicator
- TGA
- XRD
- Thin section analyses
- XRF
- FTIR
- Permeability
- Sorptivity
- MIP

The aim should then be to produce a second journal article in Materials and Structures based on these findings. Given the word limit of 8000 this should be possible without the need to approach the Editorial Board to see if a slightly longer submission could be made.

ACTIONS:

- Consider whether the list of parameters above is comprehensive (it isn't), and suggest further additions. (ALL)
- Self-nominate to prepare a review of one or more parameters (ALL)

(3) Round robin to assess carbonation resistance under accelerated and natural carbonation of control specimens

There was consensus over the need for a round robin test to examine accelerated carbonation.

There is a need for a single lab to prepare the samples, using the same batch of materials. SK-B reported on a round robin test in France where results were consistent for mortars, but not so for concretes, which may question a round robin test on paste or mortar samples.

There is a need to decide as to whether to perform tests on pastes, mortars or concretes (aggregates do not matter too much, carbonation speed will be similar); mortars are difficult to measure in XRD or TGA; on the other hand paste may shrink; paste has 10x more volume change than mortar

Precise carbonation conditions would depend on the outcome of the systematic review.

There was also general agreement that a test of natural carbonation would also be beneficial. This would need to start sooner to allow the results to be gathered before the end of the committee.

Final: Recommendation for test method

ACTIONS:

- Consider whether groups would wish to participate. (ALL)
- Consider possible experimental considerations, e.g. paste vs mortar vs concrete, [CO_2], RH, binder type (ALL)

(4) Round robin to compare and contrast different tests for carbonation depth determination / assess carbonated and partly carbonated zone

A somewhat ambitious suggestion was that the samples from the carbonation round robin could be used to conduct a second set of tests, building on the second systematic review, examining the consistency of various testing methods.

Each group therefore gave details of their characterisation capabilities.

Concern was raised that it might be difficult to coordinate and there might be problems introduced during shipping of samples.

ACTIONS:

- Consider whether groups would wish to participate. (ALL)
- Consider which techniques would be worth considering (ALL)