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PROCEEDINGS OF THE THIRTY-THIRD INTERNATIONAL CONFERENCE ON CEMENT MICROSCOPY

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MICROSCOPIC CHARACTERIZATION OF AUTOGENOUS HEALING PRODUCTS IN ENGINEERED CEMENTITIOUS COMPOSITES (ECC)

Aaron Richard Sakulich[1], Victor C. Li[2]

1 – Engineering Laboratory, National Institute of Standards and Technology, Gaithersburg MD
2 – Department of Civil and Environmental Engineering, University of Michigan, Ann Arbor MI

ABSTRACT: Autogenous self healing in cementitious systems occurs when cracks expose unhydrated cement grains and $Ca(OH)_2$ to the environment and sufficient quantities of water, producing 'healing products' that partially or completely fill the crack. Robust autogenous healing has been observed in laboratory conditions only when the crack width is less than roughly 150 micrometers, and possibly lower under field conditions; the micromechanical design of Engineered Cementitious Composites (ECC), a class of fiber-reinforced cement-based composites, allows for such crack width control. Electron- and optical microscopies, complimented by Raman spectroscopy and EDS techniques, are used to show that the healing products are a mixture of low-Ca C-S-H and calcite, and that the surfaces of the fibers exposed by cracking act as nucleation sites for healing. Nanoindentation is used to demonstrate that the modulus of the healing product is roughly equivalent to that of hydrated cementitious matrix. Finally, 3D X-ray microtomography has been used to investigate the microstructure of cracked ECC.

AFm-phase in cementitious materials

Herbert Pöllmann
University of Halle/Germany
Von Seckendorffplatz 3
06120 Halle/Saale
Germany

Abstract

Ettringite and Afm-phases are the main hydration products of the aluminate and ferrate phases during the reaction of cementitious materials. The different AFm-phases can incorporate many different anions and structurally not necessary water and are very often quite difficult to identify.
The paper describes the whole family of potentially occurring AFm-phases in cement and their role during hydration of cements but also their role as new formed phases during cement deterioration reactions.
Afm-phases can also play a very important role as materials used for immobilization of hazardous materials.

PETROGRAPHIC STUDY OF CONCRETE: TWO CASE STUDIES INVOLVING INTERNAL AND EXTERNAL SULFATE ATTACKS

Hugh (Xiaoqiang) Hou (xhou@ctlgroup.com) Ann Daugherty (adaugherty@ctlgroup.com)
CTLGroup, 5400 Old Orchard Road, Skokie, IL 60077

ABSTRACT

This paper will illustrate the value of concrete petrography through two case studies. The first case involves a municipal concrete building in Michigan that exhibits substantial foundation expansion and cracking. Petrographic examination indicates that the distress is attributed to thaumasite sulfate attack (TSA), based on the presence of cracks, microcracks and ring gaps around aggregates filled with abundant thaumasite secondary deposits, and observed paste alteration to thaumasite. Petrographic examination also reveals the sulfate is internally sourced, from the dolomite coarse aggregate in the concrete. To the best of our knowledge, TSA due to an internal sulfate source from aggregate has not been reported so far.

The second case involves concrete walls of a sewer tunnel about 100 years old. The exposed sides of the inner walls have experienced significant deterioration and spalls. Petrographic examination revealed that concrete deterioration is due to sulfate attack associated with accumulation of hydrogen sulfide (H_2S) gas within the structure. Distinct physical, compositional, and textural variations in paste along the exposed surface are observed. Depth of deterioration has been determined to be shallow from the existing surface, although the near-surface damage is severe.

EVALUATION OF MICROSTRUCTURAL PROPERTIES OF WELLBORE CEMENTS SUBJECTED TO CARBON SEQUESTRATION CONDITIONS

Mileva Radonjic

Craft & Hawkins Petroleum Engineering Department, Louisiana State University, 3526D P. F. Taylor, Baton Rouge LA70808

ABSTRACT

Carbon Capture and Storage (CCS) is a novel technology, involving capturing of CO_2 and storing it in the subsurface – rock formations, for hundreds of years. The risk number one in this new technology is that the stored CO_2 will not be contained and will leak back into the atmosphere or/and to drinking water aquifers.

Two most likely pathways for this to occur are: existing wellbores and geologic imperfections. In this paper the focus is only on oil and gas wells and their integrity. Design of wellbores requires cementing of metal casing to the rock formation primarily to ensure zonal isolation. However, this cement design is meant to last only for the lifetime of a well - in most cases tens of years. In addition, during well operations cement is subjected extensively to pressure/temperature oscillation, causing expansion, cracking and often de-bonding at interfaces with casing or formation.

Apart from potential channels at the interfaces, Portland Cement Microstructure is the most important property that will determine whether wellbore cements can provide zonal isolation for hundreds of years and provide effective storage of CO_2. Thorough understanding of cement matrix changes under acidic environment applicable to carbon sequestration, are essential for risk assessment.

The cement samples retrieved from a wellbore exposed to carbonic acid rich brine are compared to the lab cement samples exposed to acidic brines at static and dynamic conditions. It appears that the initial cement slurry design, *insitu* conditions, flow rates and time are the primary parameters that will impact the reaction rate of cement alteration. So the question is not whether cement will react under low pH conditions but how fast these reactions are under geologic sequestration conditions.

The Carbon Footprint of the US Cement and Concrete Industries

Julie Rapoport, PhD, PE, LEED AP
CalStar Products, Inc.

Abstract: Concrete is perhaps the most used material in the world, after water. It's durable, easy to place, and can form any variety of shapes. It is produced throughout the world using mostly local materials, and it has a long track-record of excellent performance. It's also responsible for about 5% of anthropogenic carbon emissions, because of the cement in it. This talk will discuss the current and projected carbon footprint of the US cement and concrete industries, as well as current mitigation methods.

Sequestering CO_2 in the Built Environment

Irvin A. Chen*

*Calera Corporation; Los Gatos, California USA; ichen@calera.com

Abstract

Numerous technologies are being developed for the capture and sequestration of anthropogenic CO_2. Mineralization via Aqueous Precipitation (MAP) is a process that can capture CO_2 directly from fossil fuel power plants and other industrial sources and convert the CO_2 into carbonate minerals that can then be used in the building industry. This talk will describe in detail the processes and chemistry associated with the conversion of CO_2 to highly engineered carbonate products and the subsequent use and optimization of the materials in the construction industry.

INTERNAL SULFATE ATTACK IN SLAG AGGREGATE CONCRETE PAVEMENTS

David Lankard
President & Petrographer
Lankard Materials Laboratory
Columbus, Ohio

ABSTRACT

Air cooled blast furnace (ACBF) slag has been used as the coarse aggregate in portland cement concrete pavements since the early 1900s. Many of these pavements have performed satisfactorily. In recent times unusual cracking problems have been reported in some slag aggregate pavements in Michigan and Ohio. This paper describes petrographic examinations of cores taken from distressed slag aggregate pavements in the Ohio Department of Transportation system. Both internal sulfate reactions and alkali silica reactions (ASR) are involved in the distress. There is compelling petrographic evidence to support a conclusion that, in these pavement cores, internal sulfate attack is the primary distress mechanism. The precursor source of the internal sulfates is sulfides derived from the slag aggregate particles. The ASR activity is associated with chert and other siliceous aggregates in the fine aggregate phase of the concretes.

MICROSCOPICAL STUDIES OF MORTAR MADE WITH LUNAR AGGREGATE

Laura Powers, Associate Principal
Wiss, Janney, Elstner Associates, Inc.
Northbrook, IL
lpowers@wje.com

ABSTRACT

Microscopical studies were conducted on a mortar cube made with calcium-aluminate cement binder and lunar regolith aggregate. The mortar cube is considered to be a 'stand-in' for lunar concrete. The principal objectives of the studies were to determine the chemical stability of the lunar aggregate and the microstructure of the paste after prolonged storage in an environment free of oxygen and water. The cube had been made in 1986 by Dr. T. D. Lin at Construction Technology Laboratories, Inc. (CTL Group) and had been stored under nitrogen in a sealed container at NASA Johnson Space Center Curatorial Center between 1986 and 2000. A request to study the cube, designated No. 69999, 76, was granted by NASA in 2000.

The cube was tested for compressive strength. A thin section of the tested cube was prepared and then examined with a petrographic microscope. The examination revealed no evidence of chemical reaction between the lunar aggregates and the calcium-aluminate cement binder. Cracks and microcracks induced by the compressive strength test suggested no inherent weakness in the aggregates or in the binder-aggregate interfacial region. Microstructural characteristics of the binder appeared typical for unaltered, low water content, calcium-aluminate cement binder.

These studies could be of interest to individuals and agencies exploring the use of cement and local materials for infrastructure in space.

ALKALI ACTIVATION OF GRANULATED BLASTFURNACE SLAG AND FLY ASH

Pelin AYTURAN and Mustafa TOKYAY
Turkish Cement Manufacturers' Association - Research and Development Institute
PK2 06582 Bakanliklar / ANKARA – TURKEY
pelint@tcma.org.tr

ABSTRACT

Ground-granulated blastfurnace slag (GGBS) and fly ash (FA) have been widely used as a partial replacement material for cement in concrete. The utilization of GGBS and FA as a component of cement-based materials is very important due to economical, technical and environmental reasons. Because of their low hydraulic reactivity especially at early ages, they sometimes need to be activated either by chemical (the addition of activators) or mechanical (a fine grinding) and/or thermal means. In recent years, alkaline cements or alkali-activated materials have been produced by alkali activating silico-aluminous materials known as mineral additives in the cement and concrete industry. Although, alkaline cements may be good alternatives to portland cement due to their much reduced CO_2 emission and energy consumption, the exact setting and hardening mechanism of them is not yet fully understood. Based on the early age behavior of alkali activated materials, the current study was designed to investigate the properties of alkali-activated GGBS and FA (Class F) in view of the microstructure, mineralogy and strength development. For this purpose, GGBS and FA were mixed with different types of alkaline activator ($NaOH+Na_2SiO_3$, $KOH+K_2SiO_3$, NaOH, Na_2CO_3). Alkaline activator incorporated mixtures of granulated blastfurnace slag or fly ash prepared to contain three concentration levels of alkali oxide (4-6 % by wt.). Alkali silicate and alkali hydroxide mixtures were prepared with silica-alkali oxide ratio in ranging between 0.5 and 1.25. For the other two alkaline activators (sodium hydroxide and sodium carbonate), Na_2O in the mixture was used in as 4 % by wt. In the study, to investigate the properties of alkali activated GGBS and FA at early age , two sets of specimens of the pastes and mortars were prepared. One set was cured at room temperature for 2-day and the other was cured at room temperature for 24 h and then cured at 65^oC, until the time of test. Physical-mechanical tests, microstructural studies and mineralogical analyses were performed on the 2-day samples. For comparative purposes, same tests and analyses were carried out on portland cement specimens hydrated for 2 days.

Key words**:** Ground granulated blastfurnace slag; Fly ash; Alkali activation; Early age strength development; Microstructure.

A MORPHOLOGICAL AND XRD STUDY OF EARLY-AGE SHRINKAGE IN A TERNARY BINDER SYSTEM: A CASE HISTORY

A. Lo Presti, T. Cerulli, D. Salvioni, M. De Santis, S. Carrà

Mapei S.p.A. R & D Central Laboratory, Milan , Italy

ABSTRACT

During the development of a product in a new plant, different and complex physical-chemical events have been observed when potentially interchangeable raw materials have been used.

In particular, our attention has been focused on an early-age shrinkage phenomenon that developed in a cement-based self-levelling compound, which is a ternary system characterized by low viscosity and usually applied as a thin layer to give a smooth and flat surface.

This event can significantly influence the ultimate shrinkage and thus the cracking risk, which is one of the most unwanted practical consequences.

All the formulations studied are CAC dominated; the hydration of a ternary binder system shows differences from the classic OPC one: first of all it is sensitive to OPC/CAC ratio and, when CA (calcium mono-aluminate) is the principal hydraulic mineral present, its hydration mainly contributes to the high early strength of the binder.

A Quantitative X-Ray Diffraction analysis (QXRD) showed that the consumption kinetics of binders phases strongly depends on the “reactivity” of the calcium aluminate cements.

To better understand the reasons of this behaviour, some experiments have been carried on.

Different formulations have been deeply investigated, in addition to XRD, on a morphological point of view by SEM and by isothermal calorimetric tests.

AGGREGATES AFFECTED BY ASR IN MORTAR BAR AND GEL PAT SPECIMENS

Šárka Šachlová

Department of Geochemistry, Mineralogy and Mineral Resources, Faculty of Science, Charles University in Prague, Albertov 6, 128 43 Prague 2, Czech Republic, lukschova@seznam.cz

Geodynamics, Institute of Rock Structure and Mechanics, Academy of Sciences of the Czech Republic v.v.i., V Holesovickach 41, 182 09, Prague 8, Czech Republic

ABSTRACT

Various metamorphic rock types (orthogneiss, phyllite, quartzite and schist) were sampled from active quarries as well as from natural outcrops situated in the Bohemian Massif, Fennoscandia and in the Transcandinavian Igneous Belt. Their alkali-silica reactivity (ASR) was investigated using accelerated mortar bar test (following the standard ASTM C1260) and modified gel pat test (following the standard BS 7943). Influence of ASR on aggregates was observed in mortar bar and gel pat specimens employing microscopic techniques (scanning electron microscopy combined with energy dispersive spectrometer and polarising microscopy).

Orthogneiss samples showed low degree of ASR (expansion < 0.1 %). Phyllite, quartzite and schist are medium to highly reactive (expansion > 0.1 %). Presence alkali-silica gels and microcracks confirms origin of ASR in the most of the mortar bar and gel pat specimens. Alteration rims were found surrounding quartz grains indicating variable content of CaO, Na_2O and Al_2O_3. Propagation of microcracks seems to be supported by aggregate microstructure (e.g. arrangement of mica particles, alteration of feldspars or presence of very fine-grained quartz).

Key words: aggregates, alkali-silica reaction, accelerated mortar bar test, gel pat test, microscopy, microstructure

MONTIGNY VIADUCT REVISITED: MICROSTRUCTURE OF AN ASR DAMAGED CONCRETE FROM 1959

Oguzhan Copuroglu and Guðbjartur J. Einarsson
Delft University of Technology, Materials and Environment (CiTG), Stevinweg 1, 2628 CN, Delft, The Netherlands. corresponding author email: o.copuroglu@tudelft.nl

ABSTRACT

Montigny viaduct (51°56'42.53" N, 4°51'36.01" E) in Schoonhoven, is the first confirmed concrete structure in the Netherlands which was severely damaged by alkali silica reaction. Since the initial investigation in 1991, the structure has been kept under control and has recently been demolished as it had reached a terminal stage due to ASR induced crack development. Therefore, the current investigation was initiated in order to document the final microstructural condition of the Montigny viaduct. To investigate and characterize the damage, several cores were extracted from the reinforced concrete deck of the structure. Microstructural features of the concrete and amorphous and crystalline ASR product compositions were studied by optical microscopy, electron microscopy and quantitative energy dispersive spectrometry (EDS). Crystalline product was found to have an empirical formula of $(Ca, Na, K)_{2.5}(Si, Al)_4O_{9.8}(H_2O)_{2.8}$, close to the mineral mountainite.

Keywords: alkali silica reaction, optical microscopy, electron microscopy, microanalysis, EDS

MICROSTRUCTURAL INVESTIGATIONS OF HIGH-STRENGTH AIR HARDENED FOAM CONCRETE

Bernhard Middendorf[1] and Armin Just[2]
[1]Professor, Dept. of Architecture and Civil Eng., TU Dortmund University, Germany
[2]Senior researcher, Dept. of Architecture and of Civil Eng., TU Dortmund University, Germany

ABSTRACT

Foam concretes are well established building materials for construction purposes. Based on European standards, foam concretes are divided into two groups: On the one hand a physically foamed concrete is mixed in fast rotating pug mill mixers by using foaming agents. This physically foamed concrete hardens under atmospheric conditions. On the other hand an autoclave aerated concrete is chemically foamed by adding aluminum powder, but has to be cured in a saturated steam atmosphere in an autoclave at a temperature of approx. 190°C and a pressure of 1.2 N/mm² for at least 4 hours.

A new type of foam concrete has been developed by the combination of chemical foaming and air hardening. Compared to autoclaved aerated foam concretes the manufacturing of this new foam concrete consumes less energy and the production is not stationary. The main advantage of this foam concrete is the combination of high thermal insulating properties with high strengths. These properties are the results of a calculated packing density of the cement paste and a dedicated pore size distribution of the macropores. The distribution and the amount of the macropores are controlled by using aluminum powder in defined amounts and particle sizes. The matrix of the hardened cement paste of the foam concrete can be optimized additionally by the specific use of supplementary cementing materials (SCM) or inert fillers as well as by the use of superplasticizers.

The mechanical properties depend on the microstructure which is strongly influenced by the used binders and filler types as well as by the used superplasticizers. The microstructure of the foam concrete was investigated by using petrographic and scanning electron microscopy (SEM) as well as mercury intrusion porosimetry (MIP). Results of the characterization of the optimized microstructure by combining different techniques will be presented in the paper.

SULFATE ATTACK AND MICROSTRUCTURAL CHANGE OF FLY-ASH MORTARS MADE WITH ORDINARY- AND LIMESTONE-CEMENTS

Robert Day[1] and Bernhard Middendorf[2]

[1] Professor, Dept. of Civil Eng., University of Calgary, Canada
[2] Professor, Dept. of Civil Eng., TU Dortmund University, Germany

ABSTRACT

Various mortar mixes were cast and standard mortar bars were subjected to 0.35M sodium sulfate solution (in accordance with the CSA/ASTM test methods for sulfate resistance), with continuous soaking at either room temperature (~20°C) or at ~5°C in a refrigerator. Bars were manufactured with ordinary or limestone cements with and without two types of fly ash. Bar expansion measurements were recorded at regular intervals for periods up to 3 years. During soaking selected mortar bars were removed from the environment and samples from the outer region (0-5 mm from the surface) and from the inner core of bars were examined for chemical and physical change by X-ray diffraction, SEM and EDXA.

Without the presence of fly ash, neither of the two ordinary (GU) cements nor the limestone (GUL) cement produced mortars that were resistant in the long term to sulfate attack at 20°C or at 5°C. When mortars were made with GU/ash and GUL/ash blends, average performance was greatly improved, showing only small expansions up to 3 years of soaking at 20°C. However, when mortars are soaked at 5°C, in some cases fly ash mortars show large expansions which can occur beyond the 1-year evaluation limit of the standard test. Expansion behaviour at 5°C, compared to 20°C, appears to be sensitive to even small changes in mortar composition and environment. Microstructural examination reveals the expected presence of ettringite, gypsum and portlandite in expanded and unexpanded mortars. XRD analysis did not detect significant quantities of thaumasite in any of the specimens tested. Large differences in microstructure and sulfate content were observed between the outer region and the inner core of specimens.

PETROGRAPHY OF CONCRETE DETERIORATED BY WEATHERING OF SULPHIDE MINERALS

J. Duchesne and B. Fournier

Université Laval, CRIB, Dep. of Geological Engineering, Québec, QC, Canada G1V 0A6

ABSTRACT Damages of concrete associated to the presence of iron sulphides in aggregates were recently recognized in the Trois-Rivières area, Quebec, Canada. More than 600 residential houses presented important deterioration in their concrete foundation walls. The concrete foundations deteriorated very quickly within 3 to 5 years after construction.

Petrographic examination of damaged concretes was carried out using a combination of tools including: visual inspection, macroscopic description, stereomicroscopic evaluation, polarized light microscopy (transmitted and reflected light), and scanning electron microscopy with energy dispersive spectroscopy. The aggregate used to produce concrete was an anorthositic gabbro containing calcic plagioclase feldspars, biotite, and pyroxene with various proportions of pyrite, chalcopyrite and pyrrhotite. The pyrite and pyrrhotite contents vary significantly from one particle to another and can reach up to 5-7%. The observation of polished sections of concrete samples show, in several cases, that the pyrite is intact while the pyrrhotite presents evident signs of oxidation with presence of iron oxyhydroxides and associated cracking. Some cracks ran through the aggregate particles and extended into the cement paste. Scanning electron microscopy examination of deteriorated concretes confirmed the presence of iron oxyhydroxides, gypsum and ettringite at the proximity of pyrrhotite.

Concrete represents an aggressive environment for natural aggregates and can favour accelerated weathering of some minerals. In the presence of water and oxygen, pyrrhotite oxidizes to form iron oxyhydroxides and sulphuric acid. This attacks the cement paste causing loss of adhesion and weakness. The acid then reacts with the phases of the cement paste, mainly portlandite and C_3A, and provokes the formation of gypsum and ettringite. The precipitation of iron oxyhydroxides, gypsum and ettringite cause a volume increase that creates expansion and cracking of the concrete.

TECHNICAL SERVICE INVESTIGATIONS OF CONCRETE BY MICROSCOPY: EXAMPLES OF THE GOOD (STRAIGHT FORWARD), BAD (COMPLEX) AND THE UGLY (BIZARRE).

Dr. Ted Sibbick and Callie LaFleur

W.R. Grace &Co- Conn, Cambridge, MA, USA

ABSTRACT
This paper describes the various types and rough proportions of technical field-related concrete problems typically encountered by the authors in their most recent investigations.

The paper shows the range of field problems typically encountered, initially providing examples of how ‘straight forward’ problems have occurred where only one factor results in an issue. Field problems are described of a considerably more complex nature where one or more factors or constituents may have been acting on each other concurrently to ultimately explain the particular problem encountered, the final explanation often being less obvious than would have been immediately apparent in a simpler issue. Finally, a number of examples are presented where multiple processes have worked in conjunction with and against each other to create a totally unexpected final result. Changing just one minor variable from the original situation can have a dramatic effect on the development of the problem, often resulting in the complete elimination of the issue.

This paper also describes the investigative process and how each problem was solved, from the simple right through to the down right bizarre. The paper also describes how Concrete Optical Microscopy can be an extremely useful tool, by itself or in combination with other techniques such as SEM-EDXA, XRD, DTA-TG, Calorimetry, and Chemical Analysis.

As a Concrete Petrographer, you never truly know which direction an investigation may take. Some times, something discovered in one portion of the analysis will lead you to use an investigative technique you hadn’t anticipated, at times leading to a totally surprising conclusion. At times, no conclusion can be drawn even at the end.

CARBONATION VS. CORROSION

Stella L. Marusin, Dr-Eng

ABSTRACT

Carbonation is a natural process and every concrete exhibits some degree of carbonation, depending on exposure conditions, temperature and humidity, and quality of the concrete during its service life. Although carbonation is a reaction between carbonic acid and calcium hydroxide, the process is not simple. It is accompanied by a decrease of pH in concrete and appears to contribute to the concrete deterioration by corrosion. On the other hand, some degree of the carbonation can have a beneficial effect by decreasing the concrete permeability and improving concrete strength.

Key Words:

Concrete, Carbonation, Corrosion of Reinforcement, Nitric Acid, Sulfuric Acid

INFLUENCE OF HYDRATE PHASE DEVELOPMENT ON EARLY SHRINKAGE IN TERNARY BINDERS

Thomas A. BIER[1], Adrian Bajrami[2], Torsten Westphal[1]

[1] Institut für Keramik, Glas- und Baustofftechnik, TU Bergakademie Freiberg, Leipziger Straße 28, D- 09596 Freiberg, Germany, thomas.bier@ikgb.tu-freiberg.de, phone +49-3731-39-4242

[2] Department of Materials Engineering, Polytechnic University of Tirana, Bul "Dëshmorët e Kombit" Sheshi "Nënë Tereza" 4,Tiranë, Albania, adrianbajrami_sr@yahoo.com , phone +355-69-29-69-277

ABSTRACT

Volume changes of mortars resulting from chemical and environmental factors are a well known phenomenon. In the majority of cases volume changes are assumed to begin at the time when a solid body is established. In reality, volume changes start immediately after cement and water come into contact during mixing. These early age volume changes are typically ignored in classical design of concrete structures, but can significantly contribute to the ultimate shrinkage and/or the cracking.

The goal of the presented work is to establish a clearer understanding of possible chemical/mineralogical mechanisms causing early dimensional changes such as shrinkage and expansion in mortars with ternary binders containing Calcium Aluminate Cement (CAC), Ordinary Portland Cement (OPC) and Calcium Sulfate (C$) as they are used in applications such as Self Leveling Underlayments.

Dimensional changes during the first 24 hours were measured by different methods and compared to the in-situ measurement of phase development by XRD. Several mineralogical compositions of the binders were tested, showing very different hydration kinetics and different behavior what dimensional changes are concerned. It turns out that the role of Aft and Afm phases and the sequence of their appearance are crucial to understand shrinkage as well as expansion.

The results are discussed in the light the mineralogical phase development and the resulting structure build-up in the cementitious matrix.

NEW METHOD BASED ON POLISHED AND THIN SECTIONS DEDICATED TO THE MICROSTRUCTURE ANALYSIS OF FRESH CEMENTITIOUS MATERIALS

Fouad Amin[1], Philippe Le Coustumer[2] & Bernard Martin[2]

[1] CTG, Italcementi Group, Rue des Technodes, BP 01, 78931 Guerville Cedex, France
[2] Université Bordeaux 1, UFR STM, B18 avenue des facultés, 33405 Talence Cedex, France

ABSTRACT

A new technique has been developed to prepare polished and thin sections of cementitious materials at fresh state. This technique is based on substitution of water by acetone, allowing to stop hydration and to fix microstructure organization of the phases at various stages of hydration. The technique has been used primarily by pedologists to characterize the structural pore space of moist soils. Recently geologists have adapted the method to study the microstructure of sediment cores collected by the seabed.

This technique applied to the cementitious materials is used to observe and analyze the microstructure of cement paste, mortar and concrete at different scales with flatbed scanner, stereo microscope, optical microscope or electron microscope. In addition, these observations can be made on polished sections or thin strips of various sizes.

The process of preparation consists to pour the cementitious material output mixer in a perforated alumina box with dimensions of 100 mm x 45 mm x 13 mm. Immediately, the sample is immersed in a solution of water and acetone to achieve the stage of dehydration. The water is discharged through a peristaltic pump that realizes a continuous flow of acetone through a molecular sieve.

After a total substitution of water by acetone in few hours, the sample is placed in a plastic box containing the impregnation solution. It consists of epoxy resin, catalyst and acetone. A fluorescent pigment is also added to the mix to allow examinations under UV light. Then the plastic box containing the sample is placed in a desiccator maintained under primer vacuum. Finally the sample is removed from the desiccator and left at room temperature for curing and hardening. This material now fixed can endure the classic level of preparation: cutting, thinning, polishing.

This process opens up new perspectives to study the influence of additives and admixtures on the microstructure of cement, mortar and concrete during hydration.

INVESTIGATIONS ON THE USE OF A FOAM GLASS CONTAINING METAKAOLIN IN A LIME BINDER SYSTEM.

Michael Schmidt[1] & Herbert Pöllmann[1], Armin Egersdörfer[2], Jürgen Göske[3], Susanne Winter[3]

[1] Martin-Luther-University Halle/Saale, Institute of Geological Science Mineralogy/Geochemistry Von Seckendorffplatz 3, 06108 Halle/Saale, Germany, michael.schmidt@geo.uni-halle.de
[2] Dennert Poraver GmbH, Mozartweg 1, 96132 Schlüsselfeld, Germany, egersdoerfer@poraver.de
[3] Zentrum für Werkstoffanalytik Lauf GmbH, Hardtstrasse 39b, 91207 Lauf, Germany, info@werkstoffanalytik.de

ABSTRACT

Main object of this project was to determine the usability of the product Metapor® in a lime binder system. For the investigations prisms of different mixtures (slaked lime, Metapor®, metakaolin, Metastar501®, limestone sand) were prepared according to EN DIN regulations. Slaked lime was substituted with 6.25 weight % Metapor® and 12,5 weight % Metapor®. Two qualities of the product were used (10 weight % foam glass and 25 weight % foam glass). Additional samples were prepared in which slaked lime was substituted with 6.25 weight % metakaolin (lab preparation) and 6.25 weight % Metastar501® for comparison reasons. Strength development of prisms was investigated over a time period of six months so far. All samples were stored in a climate chamber at 25°C under humid conditions. Reference samples of slaked lime were stored according to EN DIN 1015-11 in a desiccator. In addition to investigations on the binder system all raw materials were characterized in detail.
Bending-tensile strength as well as compressive strength increased with the addition of Metapor®. Highest strength values were observed on samples with 12,5 weight % Metapor®. A higher foam glass content leads to slightly higher strength values. Relatively high strength values were observed for samples with glass free metakaolin (lab preparation) and Metastar501® due to their determined smaller particle size and higher specific surface. No negative effects on expansion or shrinkage were observed. Shrinkage was lowest on samples with 12,5 weight % Metapor®. Samples with higher foam glass content in the Metapor® portion showed lowest shrinkage. Negative effects such as cracks and/or fissures on the surfaces of Metapor® containing prisms were not oberserved. XRD investigations on hydrated samples showed the expected phase composition. Portlandite decreased due to carbonation and the formation of calcium-silicate-hydrate phases as well as calcium-aluminate hydrate phases. SEM investigations on two different samples showed foam glass particles with partially deep corroded surfaces and pits. In addition new hydrate phases were formed on the surfaces of foam glass spheres.

KEYWORDS: Hydration, Metakaolin, Pozzolane, Slaked Lime, Strength Developement

SURFACE MODIFICATION OF PET FIBERS TO IMPROVE MECHANICAL PROPERTIES OF CEMENT COMPOSITES

Lubomir Kopecky[1], Vladimír Machovic[2], Vlastimil Králík[1], Zdeněk Bittnar[1], Jana Andertová[2], Vít Šmilauer[1], Miroslav Lhotka[2]

[1] Faculty of Civil Engineering, Dept. of Mechanics, Czech Technical University in Prague, Thákurova 7, Prague 6, CZ-166 29, Czech Republic
[2] Central Laboratories, Institute of Chemical Technology in Prague, Technická 5, Prague 6, CZ-166 28, Czech Republic

Key words: Inter facial transition zone, cement paste, PET, polymer's aging, Raman spectroscopy, micro-structure, phase analyses, electron microscopy, nanoindentation

ABSTRACT

The increasing amount of polyethylene terephthalate (PET) waste is a worldwide problem which has induced the recycling and new ways of reutilization of PET bottles. That is why the emphasis is put for new applications of PET fibers, e.g. as a micro-reinforcement in concretes. Usable properties of the micro-reinforcement depend on many factors such as cement matrix, fiber geometry, surface of fibers, size, quantity, and rheological parameters, as well. Concrete represents a three-phase composite in which the inter-facial transition zone (ITZ), i.e. the space between the fiber and the matrix, is considered as a critical factor determining the concrete strength. Good workability of the fibers in concrete mixture has been empirically find at the 3 wt% of PET fibers [2]. It has been proved that PET fibers are resistant in high alkaline cement paste environment and they compete successfully in price with that made of steel. At present time, the fibers are applied in concrete for the tunnel constructions in Japan [2].

Standardly used PET (or polymers) fibers are characterized by significant hydrophobicity of the surface and then the lower adhesiveness to hydrated cement matrix. The reason is the non-polar character of polymeric matter in the strong polar alkaline environment of the newly-forming C-S-H gels and Portlandite as well. The optimal chemical and thermal degradation of the surface of the PET fibers improve the adhesion of the fibers to the ITZ within a concrete matrix. Aging of PET fibers in the alkaline environment of cement matrix was simulated by modifying the fibers in saturated calcium hydroxide solution at an increased temperature. Recycled PET fibers reacted with calcium hydroxide forming Ca-terephthalates on the fiber 's surface. This newly-formed multi-molecular layers enabled real chemical bondings between the modified PET surface and the C-S-H gels of hydrated cement matrix.

The present work has studied chemical and thermal degradation of PET fibres within a cementitious matrix. The chemical degradation has been simulated by alkali hydrolysis of the PET fibres in an NaOH solution or a saturated $Ca(OH)_2$ solution, which as a product of cement hydration concentrates in the transition zone surrounding the micro-reinforcement. Thermal ageing of the PET fibres was realized by temperature cycles in a thermostatic chamber under constant humidity. This way, the chemical degradation of the fibres during hydration of the cementitious matrix under varying weathering temperatures has been simulated, and simultaneously the effect of changes in the surface quality and adhesion of the fibres to concrete mixture in dependence on mechanical properties of the PET - cement composite.

APPLICATION OF MICROSCOPIC TECHNIQUES FOR STUDYING MICROSTRUCTURE OF AIR-ENTRAINED CONCRETES CONTAINING HIGH CALCIUM FLY ASH

D. JÓŹWIAK-NIEDŹWIEDZKA*, M.A. GLINICKI, M. DĄBROWSKI

Institute of Fundamental Technological Research
Polish Academy of Sciences, Pawinskiego 5B, 02-106, Warsaw, Poland

ABSTRACT

The possibility of using the high calcium fly ash (HCFA) as type II concrete additive is not well established, especially in relation to the durability of concrete structures in aggressive environment. The paper presents results of microstructural characterization of air-entrained concrete containing high calcium fly ash from lignite combustion. Frost resistant concretes were designed with different content of fly ash use for cement replacement by 15% and 30%. Different kinds of HCFA were used: raw (unprocessed) and grinded during 10, 15 and 28 minutes. The evaluation of the microstructure was performed using SEM and optical microscopy on thin sections and plane sections. All thin sections were impregnated with epoxy containing fluorescent dye and were examined using ordinary light, crossed polarized light and UV light. The thin section evaluation involved petrographic characterization of aggregates and examination of paste quality. Automatic air-void analysis on plane sections revealed air-content, specific surface, spacing factor and the content of micropores in the hardened concrete. Significant differences in particle shape and size before and after grinding of the HCFA were revealed by SEM analysis. The results of thin section analysis of the high calcium fly ash concrete showed that its microstructure was more dense than that of the ordinary concrete. The influence of specific surface of fly ash on air void content in HCFA concrete was found.

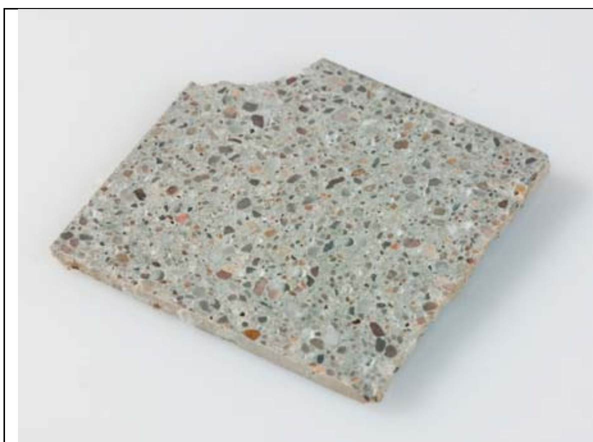
* Corresponding author: e-mail address - djozwiak@ippt.gov.pl, tel. +48 22 8261281 ext. 310

Multi-dimensional X-ray Investigation of Building Materials
Powder Diffraction to Computed Tomography Analyses

Herbert Pöllmann[1], Roger Meier [2], Uli Riedl[2], Gabriel Blai[2], Jennifer Anderson[3]

[1] University of Halle/Mineralogy/Germany
[2] Panalytical Almelo, Netherlands
[3] Panalytical Westborough, MA, USA

The careful characterization of mineral phases in building materials is an important indicator of the material stability in environment. Powder X-ray diffraction (PXRD) and the Rietveld method are well established in the field of building materials analysis. The distribution of mineral phases and pore spaces cannot be determined by diffraction techniques but adds valuable information to the material characterization. Computed tomography (CT) is a powerful 3D imaging technique that can provide information regarding mineral and pore size, shape and distribution within samples. Recent advances in detector technology (PiXcel3D) allow collection of both data types with the same detector in the same laboratory system. This poster presents the results PXRD and CT data for two different concrete samples, one with normal sand and another in which the sand was partially replaced by glass powder. Results of this study demonstrate the advantages of an X-ray platform that allows the correlation of nanoscopic features with material properties to estimate material durability.



Macroscopic view of the concrete sample
4 x 4 x 0.2cm

Microscopic sample description, pores, sand grains and hydrated cement