23rd INTERNATIONAL CONFERENCE ON CEMENT MICROSCOPY

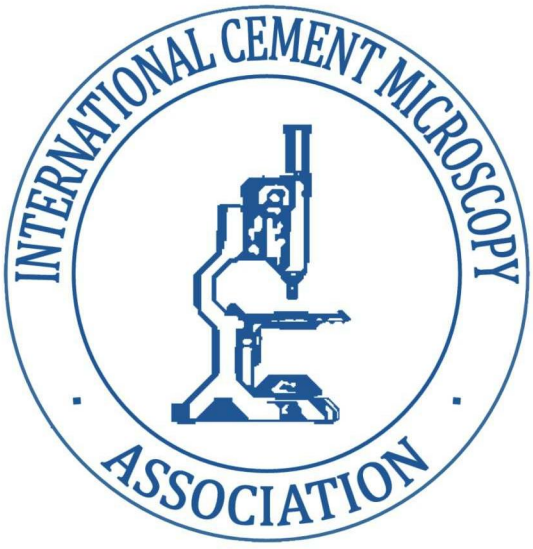

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Table of contents

	Beads Technique
J. K. Galinat, V. Ramakrishnan, S. S. Bang	Concrete Crack Remediation by Polyurethane-Immobilized *Bacillus Pasteurii*
B. Walk-Lauffer, D. Knofel, S. G. Zurn, D. Hums, M. Huber	SEM-Investigations on the Microstructure of Steam Cured Building Materials
R. Uribe Afif, R. Rodriquez Camacho, J. Flores Martinez, H. Montano Roman	Physical and Chemical Characteristics of Aggregate Material Passing the Mesh 75 µm (#200) and its Influence in the Inhibition of the Alkali-Silica Reactivity in Mexico City
C. W. Baumgart, K. D. Linder, S. P. Cave	An Automated, Image Processing System for Concrete Evaluation
D. Herfort, I. A. Juel	The Mineralogy of Sulfate and Sea Water Attack on Concrete Interpreted From EPMA Analysis
M. A. Al-Shafei	A Case Study of Ettringite Induced Microcracking of Concrete at a Waste Water Treatment Plant
U.H. Jakobsen, N. Thaulow	Sulfate Attack as Observed by Optical and Scanning Electron Microscopy
S. Sahu, A. Snyder, S. Badger, R.J. Lee	Depth Profiling and Phase Discrimination in Deteriorated Concrete Utilizing Scanning Electron Microscopy with Automated Point Count Analysis
J. Carrillo-Morris	Impact of Amorphous and Crystalline Silica on Pyroprocessing
A. Blanco, A. Centeno, C. Cadenas, J. Soto	Relationship Between Mechanical Properties and Microstructure of Cement with Fiber for Multilateral Wells
A. Santos-Arceo, C. Gaytán	"In-House" Image Analysis Software: A Comparison Study, For a Fast and Reliable Automatic Microscopy Analysis
A. K. Crumbie	SEM Microstructural Studies of Cementitious Materials: Sample Preparation of Polished Sections and Microstructural Observations With Backscattered Images – Artifacts and Practical Considerations
M. Radonjic, G. Allen, A. Hughes, N. Elton, J. Hooper	In-Situ Observation of Lime Paste Carbonation in CO_2 Environment Scanning Electron Microscopy (ESEM)
M. Davidts	Fused Beads for Better XRF Analytical Results in Cement Application

EFFECT OF KILN SPEED ON SIZE RELATIONSHIPS OF THE SILICATE CRYSTALS IN TYPE I PORTLAND CEMENT CLINKER AT ASH GROVE CEMENT'S DURKEE, OREGON PLANT

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ABSTRACT

The time that a clinker nodule spends in the burning zone of the kiln, also known as the residence time, can have quite an effect on the microstructure of the resulting clinker. The kiln speed, expressed in rpm, or revolutions per minute, controls the residence time. This means that the faster the kiln speed, the lower the residence time will be. Reflected light microscopy can be used to track resulting changes in the clinker microstructure as the kiln speed is increased, and estimates can be made as to the possible effects these changes may have on the performance of the cement made from that clinker. Crystal size, reactivity, and structure, as well as porosity of the nodule itself, can all be improved with an increase in kiln speed, and some detrimental conditions can also be at least partly remedied. Overburning can, for example, be at least partly counteracted by increasing the kiln speed and decreasing the residence time. However, there appears to be a point of diminishing returns which is eventually reached, after which no apparent structural changes will be observed if kiln speed is increased further. A marked change in crystal size relationships was observed in the clinker from Ash Grove Cement's Durkee, Oregon plant in the latter part of 1999 and the early part of 2000, and the above mentioned "point of diminishing returns" seemingly reached.

FLUX PHASE COMPOSITIONS IN PORTLAND CEMENT CLINKER

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ABSTRACT

Electron optical analyses of the flux phases in Portland cement clinker have frequently shown the presence of three distinct areas within the flux, corresponding to the aluminate phase, the ferrite phase and a phase which appears from x-ray microanalysis to be chemically between the two.

The phases have been examined in a variety of industrial clinkers produced under known conditions, using methods including optical and x-ray microanalysis. From the results some interpretations concerning the likely origin and conditions for formation of the intermediate composition material are considered.

Chemical Admixture for Sulfate Resistance of Hydrated Oilwell Cements: Part II,

Conclusion of Study

Prepared for presentation at the Twenty-Third International Conference on Cement Microscopy, Albuquerque, New Mexico, April 29-May 3, 2001.

Carole Fleming, Chevron Petroleum Technology Company; Bill Caveny and Rickey Morgan, Halliburton Energy Services, Inc.

Abstract
This paper continues to explore the feasibility of using chemical admixture to give hydrated oilwell cements resistance to sulfate attack, and presents final and favorable results of tests performed with the admixture. Results observed after 1 year of exposure to sulfates indicate that treating cements with 1% of the admixture by weight of cement (BWOC) helps improve their resistance to sulfate attack.

Light microscopy, environmental scanning electron microscopy (ESEM), expansion bars, and compressive-strength measurements were used to study sulfate attack on several hydrated-cement designs. Samples were aged in a 5% sulfate solution at 39°C, and were removed periodically over 12 months for measurement and evaluation. The paper reports findings of the study and shows (1) photomicrographs of samples and (2) the physical measurements recorded. This concludes the work that was published in the proceedings of the Twenty-Second International Conference on Cement Microscopy.

DEPTH PROFILING AND PHASE DISCRIMINATION IN DETERIORATED CONCRTE UTILIZING SCANNING ELECTRON MICROSCOPY WITH AUTOMATED POINT COUNT ANALYSIS

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ABSTRACT

Automated point count analysis utilizing scanning electron microscopy (SEM) associated with energy dispersive spectroscopy (EDS) allows for the discrimination of various phases by chemical composition. Phases are discriminated by energy dispersive spectroscopy with "rule" files developed for all phases of interest. Automatic control of the SEM stage and recording of coordinates also allows for the mapping of individual phases by location within a polished concrete section. In this manner, relative quantities of each phase (i.e. ettringite, monosulfate, CSH) may be plotted with respect to depth of the concrete from the surface. Automation of the analysis is achieved by fixing an array of points over selected "standard" magnified paste images. The computer then obtains EDS data from each point within a given image. Images and EDS point data can be electronically stored for subsequent review. Therefore, the frequency with which a given phase coincides within the regular grid system of points allows variations in paste composition to be observed. Comparisons of automated to manual SEM analysis are presented.

MICROSTRUCTURAL AND CHEMICAL DEVELOPMENT OF HEAT-CURED CONCRETES AND MORTARS

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ABSTRACT

Ordinary Portland cement mortars subjected to elevated temperatures (>70°C) may occasionally exhibit expansion on exposure to moisture at room temperature. This deterioration process is widely referred to as delayed ettringite formation or DEF because ettringite, which normally forms at early ages of hydration, is formed on a delayed basis at much later ages.

It has been claimed that the expansion is a result of the delayed formation of ettringite. However delayed ettringite is observed in heat-cured mortars that are not expansive. Thus the presence of ettringite in a damaged mortar is not a sufficient condition to show that ettringite has caused the damage since ettringite formed at late ages is generally present, whether or not expansion occurs.

This paper presents the changes occurring in the chemistry and microstructure of expanding and non-expanding heat-cured mortars made in laboratory. A mechanism of expansion is proposed and the necessary conditions for expansion to occur in heat-cured mortars or concretes are discussed.

EFFECTS OF POST-HYDRATING PORTLAND CEMENT PARTICLES IN THE HARDENED CEMENT MATRIX

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ABSTRACT

In this research project different Portland cement pastes with low water/cement ratios - as used in high performance concretes - were stored under water at 8°C, 20°C and 50°C for up to 360 days. Afterwards the samples were investigated with regard to a possible expansion and change in microstructure due to secondary hydration of clinker relicts with additional water in the hardened cement paste. Industrial and laboratory made Portland cements with lime saturation factors between 0.85 and 0.99 and specific surfaces between 2300 and 4100 cm^2/g were used to prepare cement pastes with w/c-ratios of 0.35 and 0.20. Beside the length change the following examinations were carried out: X-ray diffraction, mercury intrusion porosimetry, scanning electron microscopy, metallographic UV-microscopy.

ZUSAMMENFASSUNG

In diesem Forschungsprojekt wurden verschiedene Portlandzementsteine mit niedrigen Wasser/Zement-Werten – wie in Hochleistungsbeton verwendet – für bis zu 360 Tage bei 8°C, 20°C und 50°C unter Wasser gelagert. Danach wurden die Proben im Hinblick auf eine Expansion und Änderung der Mikrostruktur untersucht, die darauf zurückzuführen ist, daß nicht hydratisierte Portlandzement-Partikel mit von außen zugeführtem Wasser in der erhärteten Zementsteinmatrix reagieren. Es wurden industriell hergestellte Zemente und Laborzemente mit unterschiedlichen Kalkstandards (85 bis 99) und Feinheiten (2300 bis 4100 cm^2/g) verwendet um Zementsteinproben mit w/z-Werten von 0.35 und 0.20 herzustellen. Neben der Längenänderung wurden folgende Untersuchungen durchgeführt: Röngenbeugungsanalyse, Quecksilberdruck-porosimetrie, Rasterelektronenmikroskopie und Auflicht-UV-Mikroskopie.

EFFECT OF SP TYPE ON ETTRINGITE MORPHOLOGY AND ON THE RHEOLOGY OF PORTLAND CEMENT MORTARS AS A FUNCTION OF TYPE OF ADDED CALCIUM SULFATES

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ABSTRACT

Concrete technology has improved, widening the range of applications of this material. For instance, high performance properties such as high fluidity have been in great demand in concrete technology over the last few years. Therefore, a number of new superplasticizers (SP) have been developed and applied to important construction projects. In this regard, polycarboxylate-type superplaticizers (PCP) have been more widely used recently.

In order to have more insights on the effect of the PCP-type SP as a function of the nature of added sulfates on Portland cement fluidity, a comparative study has been carried out in 2 steps.

In the first part of the study, the effects of a common polynaphtalenesulfonate (PNS) - type SP and of two PCP - type SP on the formation of ettringite have been compared. The ettringite has been prepared on mixing solutions of $Al_2(SO_3)3$ and $Ca(OH)_2$ in presence or not of SP. The morphology of ettringite been examined by SEM and the SP adsorption has been monitored.

In the second part of the study, the effect of the 3 SP's as a function of the nature of added calcium sulfates (gypsum, plaster, anhydrite) and C_3A content (2 levels considered) in Portland cement has been compared through the early rheological behavior of mortars at a W/C of 0.5. Concurrently, early SP adsorption has been measured in the mortars.

Overall, the disturbance of the ettringite morphology is somewhat different between the SP's : an ill-defined mass being observed in presence of PNS and small needles in presence of PCP-type SP compared to long and sharp needles in the reference mixture without SP. Moreover, the adsorption of the PCP-type SP on the ettringite is lower when compared to the PNS type SP.

The rheological results on mortars indicate that, for high C_3A level, the early fluidity increases as a function of the calcium sulfates solubility rate regardless of the type of SP. When the C_3A level is lower, dosage of SP is reduced and the cement dispersion is less sensitive to the nature of added calcium sulfates regardless of the type of SP.

Effects of Shrinkage Reducing Admixtures on the Physical Mechanical Properties of Mortars

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Abstract

The shrinkage is one of the main reasons of failure of cementitious building materials (concrete, mortars, adhesive for tiles). Cracks formation or detachment are normally related with the high shrinkage of these products. Shrinkage reducing admixtures (SRA) improve the dimensional stability of hydrated cement. A study was carried out in order to better understand the effects of such admixtures on the physical mechanical and chemical properties of cementitious mortars. On this purpose specimens of mortars mixed with 10%, 5% and 0,5% (on the cement weight) of two different SRA were prepared. The samples were analysed by mechanical (compressive strength, total shrinkage, setting time, water evaporation), morphological (surface area determination by B.E.T. method, ESEM) and chemical (TG-DSC) tests.

THE INDIRECT EFFECTS OF THE USE OF MINERAL ADDITIVES IN THE DURABILITY AND MICROSTRUCTURE OF CONCRETE PASTE, EXPOSED TO ABRASION

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ABSTRACT

At the present, the use of mineral additives in the manufacture of concrete, has become a common and familiar practice for people who are involved in structure design of this type of additives. This condition in certain cases has favored the relaxation in the measured preventive in the use of this type of additive when this material is considered like raw material in the concrete's production. This paper reviews and analyzes the structure concrete case and as consequence of its exposition conditions and service and the use of an addition mineral inorganic type. It presented a premature deterioration.

In the same one an analysis of the microstructure is presented and it permits certainly identify that is has been importantly affected by the inclusion in the concrete mixture of a specific component. The evaluation results and the employed criteria in the identification of the microstructure and their influence in the decrease of the durability are presented.

In the present job, was reviewed and analyzed a concrete one, in which for it was employed an addition of inorganic material to give it a specific color. The concrete one was placed as a floor that served as a bearing surface in a workshop, where the service condition consisted in automotive vehicles and persons traffic. In a period lower than 6 months the material presented an important deterioration that consisted in a mortar paste superficial disintegration and a concrete delamination.

THE STABILITY OF PYRITE IN CALCAREOUS AGGREGATE : INVESTIGATIONS IN OLD CONCRETE STRUCTURES

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ABSTRACT

The literature has reported some cases where iron sulfide-bearing aggregates have contributed to threaten the concrete durability. The Tournasian silicified limestone has been suspected to cause secondary expansive reaction in concrete as a consequence of the presence of pyrite. A chemical analysis was performed on samples from old concrete structures, sound or affected by alkali-silica reaction (ASR). The objective was to quantify the sulfide and the sulfate concentrations, expressed as a function of the aggregate and cement content respectively, and to examine if those amounts could be considered as consistent with the expected values for cement and limestone. No tendency for increasing in sulfate and decreasing in sulfide was observed: keeping in mind that the oxidation would have, for certain, created a depletion in sulfide on behalf of a higher sulfate concentration, it could be suggested that the stability of pyrite in the heart of concrete was clearly established.

A further systematic microscopical study, in backscattered electrons mode, has been performed on polished sections taken out from those concretes concerned by chemical analysis, as well as additional ones, to investigate not only the state of pyrite present in aggregates, but also to identify any indications, which could point out some pyrite deterioration.

The cubic and "framboïdal" pyrite microformations give neither evidence of morphological change, nor any modification of the S/Fe ratio that would have indicated chemical transformation. No trace of gypsum, ettringite, of other typical components resulting from pyrite weathering (ferrous and/or ferric sulfates, iron hydroxides) was detected. The SEM study confirms the diagnosis of chemical analysis, to know the resistance to oxidation of pyrite in aggregate when those are embedded in mortar protective matrix.

A COMPREHENSIVE PETROGRAPHIC INVESTIGATION OF A SEVERELY DETERIORATED CONCRETE UTILITY VAULT

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ABSTRACT

Petrographic examination is a powerful and indispensable tool in assessing the quality of concrete and trouble shooting concrete problems. However, in dealing with complicated deterioration problems, traditional optical petrography is often not enough, and a comprehensive approach using petrography, chemical analysis, X-ray diffraction and scanning electron microscopy is required.

A more than 20-year old concrete utility vault was severely deteriorated. Corrosion of steels, sulfate attack and/or delayed ettringite formation (DEF) have been suspected as the causes of the deterioration. However, the evidence from a comprehensive investigation using thin section optical microscopy, X-ray diffraction (XRD) of secondary deposits, scanning electron microscopy (SEM) with energy dispersive X-ray analysis (EDX) and other techniques strongly suggests that acid leaching was the primary cause of the deterioration. Many details will be presented.

KEY WORDS: Petrography, acid leaching, sulfate attack, thin section, SEM

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FINITE ELEMENT STRESS COMPUTATIONS APPLIED TO IMAGES OF DAMAGED CONCRETE: A POSSIBLE NEW DIAGNOSTIC TOOL FOR CONCRETE PETROGRAPHY

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ABSTRACT

Concrete petrography is used to assess damaged concrete in order to determine the probable mechanism of damage. However, using traditional petrographic analyses, it is often difficult to say what mechanisms caused the crack damage, since many deterioration mechanisms produce cracks. This article describes a hybrid imaging-finite element modelling technique that can be used to help determine the mechanism of damage in such cases. This application is intended to assist concrete petrographers in the assessment of degradation mechanisms.

INVESTIGATION OF FIRE-DAMAGED CONCRETE WITH DIFFERENT TYPES OF AGGREGATES

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ABSTRACT

Inspite of the fact that portland cement concrete is incombustible and give good resistance to elevated temperatures, heating due to fire or different causes will bring about physical and chemical changes in the structure of concrete. To repair fire damaged concrete, firstly, it is necessary to understand the extend of the effects of high temperatures on the concrete. Considering this, the current study is designed to visualise the effects of elevated temperatures on concrete microstructure and compressive strength. For this purpose, mortars with normal portland cement and three different aggregates were prepared. These aggregates were quartzite, limestone and pumice. The mortar specimens for compressive strength tests and SEM investigations were subjected to 100, 250, 500, 700 and 850°C for period of 4 hours including the temperature increase period. For SEM observations the same places in the samples before and after heating were investigated. This provides with the main advantage of the study which clearly shows the effect of heating on microstructure. As a result, changes in CSH, CH and aggregate, crack propagation and formation can be exactly identified on the same samples.

ÖZET

Portland çimentosu içeren beton, yüksek sicakliklara karsi iyi dayaniklilik gösteren ve yanmaz bir malzeme olmasina ragmen, yangin ya da farkli sebeplerle olusan sicaklik artislariyla yapisinda bazi fiziksel ve kimyasal degisiklikler gösterir. Yangindan zarar görmüs betonun onarilmasinda ilk olarak yüksek sicakliklarin beton üzerinde ne mertebelerde etkili oldugu incelenmelidir. Bu durum göz önüne alinarak, bu çalisma yangina maruz kalan betonlarda mikroyapi ve dayanim degisikliklerini göz önüne sermek amaciyla yapilmistir. Bu amaçla normal portland çimentosu ve 3 tip farkli agrega içeren harçlar hazirlanmistir. Kullanilan agregalar; kuvartzit, kireçtasi ve bims'tir. Basinç dayanimi ve SEM incelemeleri için harç numuneleri, sicaklik artis periyodu da dahil olmak üzere 4 saat süreyle 100, 250, 500, 700 ve 850°C sicakliklara tabi tutulmustur. SEM incelemeleri için ayni numunenin ayni bölgeleri sicakliga tabi tutulmadan önce ve sonra incelenmistir. Sicakligin mikroyapiya etkisinin ayni bölgede gösterilmesi bu çalismanin sundugu bir avantajdir. Böylelikle CSH, CH, agrega ve çatlak gelismeleri net olarak incelenebilmistir.

EFFECTS OF LITHIUM SALTS ON ASR GEL COMPOSITION AND EXPANSION OF MORTARS

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ABSTRACT

Suppression of ASR expansion in mortar and concrete by the addition of lithium salts has been confirmed by some workers. It has been revealed that lithium hydroxide tended to reduce the reaction between sodium or potassium hydroxide and silica, and ASR gels incorporating lithium was less expansive. However, it has not been reported how the addition of a lithium salt influenced the composition of ASR gel. The calcium in ASR gels is considered to play an important role in expansion of ASR gel. Thus, it is significant to reveal the characteristic of ASR gels composition in mortars containing lithium salts by the BSE-EDS analysis. This study aims at discussing the mechanisms of suppression of ASR expansion in mortar by lithium salts from the viewpoint of ASR gel composition. The average CaO/SiO_2 ratio in ASR gels decreased with increasing amounts of added lithium salts. It should be noted that the extent of variations in the CaO/SiO_2 ratio in ASR gels significantly decreased with increasing amounts of lithium salts. The addition of relatively small amounts of LiOH and Li_2CO_3 resulted in increased expansion. We also obtained an unexpected result that ASR gels became homogeneous in their CaO contents at high dosage levels. However, the reduction in average CaO/SiO_2 ratios and the homogenization in CaO contents of ASR gels due to the addition of lithium salts may not be related to the expansion of mortars.

CONNECTION BETWEEN RHEOLOGICAL AND MICROSTRUCTURAL CHANGES DURING HYDRATION OF CEMENT PASTES.

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ABSTRACT

One of the most important quality requirements to a pasty building material is an easy workability which is described by the rheology. By rheology one understands the motion under operation of a force and/or the result of its interaction with the microstructure of the material. In order to affect the workability purposefully it is important to know exactly about the microstructure and their reaction to outside forces. A characteristic of cementitious building materials is the change of their microstructure by cement hydration. This stiffening is of crucial importance for the working time.

A aim of this work was the observation of the stiffening of cement pastes containing chemical admixtures both with rheological and with microscopic methods and from this the development of an improved understanding of the microstructure of cement pastes. For this - with oscillating rheometer measurements - the development of the microstructure was tested and the transition from the liquid state to a solid similar state was observed. With a rotational rheometer the increase of the yield value during the hydration was measured and the influence of mixing on the microstructure of partly hydrated cement pastes was tested. With microscopic methods the appearance of the microstructure was observed. Since the microstructure of soft pastes is easily destroyed, the samples were shock-frozen by liquid nitrogen and the pore solution was removed from the sample by sublimation. The results are SEM micrographs of a hardly changed three-dimensional microstructure.

MORPHOLOGICAL VARIATION OF CEMENT PASTE MICROSTRUCTURE DUE TO THE USE OF ADMIXTURES.

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ABSTRACT

It is our opinion, in agreement with several authors, that the final mechanical performances of cement based products depend mainly on the "reactions" that take place during the early hydration phase. The aim of this work is the study of the cement paste microstructure in the first 24 hours. We have considered several types of admixtures, including accelerators, retarders and superplasticizers, and we have observed their effect on the cement microstructure. The study has been carried out using an ESEM-FEG to characterize the morphology of the samples. The hydration kinetic and thermodynamic have been studied by XRD analysis and temperature variation, thus following the growing of hydration products and their thermal profile.
A dramatic improvement of the morphological details of the hydrating cement has been possible only with the use of ESEM-FEG, which allows the analysis of untreated samples, in the presence of significant amounts of residual water.

MISINTERPRETATION OF X-RAY ELEMENTAL ANALYSES OF CONCRETE DUE TO FA AND SF CONTENTS

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ABSTRACT

Fly ash (FA) and silica fume (SF) are widely used as concrete admixtures. Their additions influence many physical and mechanical properties of fresh and hardened concrete and improve concrete durability. However, if studies of concrete failures are based only on scanning electron microscopy locations containing accumulated or fractured FA or SF can be misinterpreted, and based on X-ray elemental analysis, may erroneously be used to identify concrete composition and condition, perhaps leading to future failures. I have encountered such misinterpretation; examples are presented.

Key Words: Concrete, silica fume, fly ash, X-ray elemental analysis

ASTM C-114 QUALIFICATION TEST METHOD TO IMPROVE ANALYTICAL RESULTS USING THE FUSED BEADS TECHNIQUE

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Abstract

The 21st century offers a host of new challenges, especially in analytical chemistry, where quality standards become increasingly demanding. To meet those challenges, cement industries are among the first to have understood the interest of XRF analysis using fused beads technique. This method is well established as a quick and reliable method, which yields highly accurate and precise results. The purpose of this paper is to show how to qualify for ASTM C-114 certification using the fused beads technique for chemical analysis of hydraulic cement. Important considerations and most common problems that may be encountered will be discussed. A particular attention will be given to several reproducibility issues, often overlooked and misunderstood.

CONCRETE CRACK REMEDIATION BY POLYURETHANE-IMMOBILIZED *BACILLUS PASTEURII*

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ABSTRACT

This paper reports the results of the scanning electron micrography (SEM) analysis of concrete crack remediation by polyurethane (PU)-immobilized *Bacillus pasteurii* whole cells. In SEM, encapsulated bacterial cells were identified throughout PU polymer matrices with some clumps. During remediation, calcite precipitation appeared in PU matrices as well as between the polymer and the concrete, in which cells were embedded in growing calcite crystals. Calcite in PU showed little effect on the elastic modulus and the tensile strength of the polymer, but increased the compressive strengths of concrete cubes whose cracks were remediated with PU-immobilized cells.

Key words: SEM, polyurethane immobilization, calcite, concrete remediation, *Bacillus pasteurii*

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SEM-INVESTIGATIONS ON THE MICROSTRUCTURE OF STEAM CURED BUILDING MATERIALS

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ABSTRACT

Hydrothermally hardened building materials are characterized by high compressive strength and –if of porous texture- good thermal insulation properties and workability. By the efforts to lower the energy consumption at the expense of fossil fuels heat insulating building materials gain increasingly in attractiveness.

In addition to the characteristics specified above further demands on the building material are placed. The quality of hydrothermally hardened building materials depend substantially on the chemical and mineralogical composition of the raw materials.

Previous investigations on these topics [1-3] showed that shrinkage (as one example) is strongly determined by the crystallinity of the 1,13 nm tobermorite, which is the main binding phase in most hardened materials. The scope of this work is twofold:

- Influence of the foreign cations Al^{3+} and K^{+} on the basic system $CaO\text{-}SiO_2\text{-}H_2O$.
- Application to industrially relevant systems

The samples were investigated by means of scanning electron microscopy (SEM), energy disperse x-ray-fluorecense (EDX) and electron microprobe analysis (EMPA).

Additionally x-ray diffraction and mercury intrusion porosimetry were applied to the hardened material.

The influence of the mineralogical phase composition and the microstructure on the physico- mechanical properties of the material is presented by selected examples.

PHYSICAL AND CHEMICAL CHARACTERISTICS OF AGGREGATE MATERIAL PASSING THE MESH 75 μm (# 200) AND ITS INFLUENCE IN THE INHIBITION OF THE ALKALI – SILICA REACTIVITY IN MEXICO CITY

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ABSTRACT

Alkali-silica reaction has always been considered a potential problem in concrete manufactured in Mexico City. However, a problem of this type has not been identified in this region in any structures made with these materials. This is surprising, especially when considering that all of the conditions necessary to promote ASR seem to be present. The present work characterizes the aggregates used and presents some case studies of concrete elements with more than 25 years of age. A series of hypothesis are presented to explain the absence of any significant ASR in concrete structures that appears to have all of the necessary elements for the development of ASR.

AN AUTOMATED, IMAGE PROCESSING SYSTEM FOR CONCRETE EVALUATION *

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ABSTRACT:
The determination of the microscopic properties, hence air-void system, in hardened concrete provides a valuable link to the durability and performance of concrete. Because of this, knowledge of the air-void structure in hardened concrete is a valuable tool. Current manual methods (ASTM C 457 linear-traverse or modified point-count) used to determine the air-void structure in hardened concrete are extremely time consuming, tedious, and require highly skilled, and experienced personnel. Automating the system saves both time and effort and improves the consistency and repeatability of the operation and its results. Previous efforts to develop an automated system, as such, have not produced to the degree of accuracy needed and required for total confidence. As a result, conventional, manual methods of evaluating hardened concrete remain in common use.

Since 1998, a cooperative effort between the Missouri Department of Transportation and Honeywell Federal Manufacturing & Technologies** has made significant progress developing an automated, image analysis system with a baseline capability to determine the air-void structure in hardened concrete while addressing all inevitable conditions that can be encountered during hardened concrete analysis. This system automatically scans and collects microscope images of the surface of a sample of polished concrete, with very little interaction from the human operator. Analysis of the acquired imagery is then performed using customized image processing techniques to detect voids (and other components) in the concrete. Sixteen microscopic properties of the concrete, as defined by the ASTM standard, are estimated and reported to the user assessing the quality of the test sample. Complete development of this system is proposed enabling field implementation of concrete materials, construction, and research.

Keyword I: Machine Vision and Image Processing
Keywords II: Classification-Fusion, Computer Vision, Data-Analysis, Data-Fusion, Data-Visualization, Design-Automation, Feature-Extraction, Feature-Identification and Classification, Image-Data Acquisition, Image-Interpretation, Image-Processing, Machine-Vision, Machine-Vision and Image Processing, Matching-Fuzzy, Texture Classification

*Patent Pending
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THE MINERALOGY OF SULFATE AND SEA WATER ATTACK ON CONCRETE INTERPRETED FROM EPMA ANALYSIS

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ABSTRACT

A three year project investigating the mineralogical processes of sulfate and sea water attack of concrete is described. The aim of the project is to experimentally test a model which predicts that the mineralogy of concrete altered by chemical attack is governed by the existence of stable or metastable equilibrium within localized zones, even though a state of equilibrium does not exist across the entire system. The model is tested experimentally in the laboratory, and by detailed examination of concretes of wide ranging composition exposed to sea water for 18 years at an exposure site on the west coast of Denmark. Electron Probe Microanalysis (EPMA) results are presented in this paper which, although not providing independent confirmation of the above model, indicate the AFm phases to consist of monosulfate intimately mixed with the C-S-H phase, and a solid solution of Friedel's salt and monocarbonate. Ettringite, although regarded as part of the same phase assemblage, is found almost exclusively in original air voids. Ingress of chlorides to a depth of 100 mm from the concrete surface is markedly reduced in concrete containing fly ash. This seems at least in part to be the result of the greater binding capacity of the AFm solid solution phase.

A CASE STUDY OF ETTRINGITE INDUCED MICRO-CRACKING OF CONCRETE AT A WASTE WATER TREATMENT PLANT

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Summary. Saudi Aramco engineers observed micro-cracks at walls and slabs of Industrial Waste Water Treatment Plant after cementing jobs had been completed. These micro-cracks were also found to propagate as a function of time, thus indicating that the reactions causing the failure are also time dependent. Surface analyses instruments at R&D Center were used to determine the causes of concrete deterioration, and it was observed that ettringite, which is a hydrated calcium sulfoaluminate, formed after the cement was cured and caused the concrete to expand and crack. This delayed ettringite formation (DEF) results from high ambient temperature and high salinity water used during cementing and adversely affects cement/concrete strength, durability and integrity.

This paper discusses the nature of DEF in cement paste and at the aggregate /paste interfaces as observed by scanning electron microscopy (SEM) and computed X-ray tomography (CT Scan). Based on these observations, recommendations are made to minimize DEF induced micro-cracking of cement and concrete.

SULFATE ATTACK AS OBSERVED BY OPTICAL AND SCANNING ELECTRON MICROSCOPY

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ABSTRACT

Sulfate attack can be divided into two groups, depending on the source of the sulfate, namely internal sulfate attack and external sulfate attack. Internal sulfate attack is a process where the sulfate is supplied from the concrete itself e.g. released from gypsum in aggregates or sulfate made available in the cement paste due to too high internal temperatures at early ages (Delayed Ettringite Formation, DEF). External sulfate attack occurs when sulfate penetrates the concrete from an external source such as ground water, sea water or sewage water. Combining optical and scanning electron microscopy in the study of the two types of sulfate attack elucidates different micro-structural features. An internal sulfate attack such as DEF can be recognized by the presence of gaps around aggregates, whereas an external sulfate attack leaves a pronounced zoning through the surface near part of the concrete. The paper describes case studies of the two main types of sulfate attack illustrated by photographs taken in the optical microscope and in the scanning electron microscope.

DEPTH PROFILING AND PHASE DISCRIMINATION IN DETERIORATED CONCRTE UTILIZING SCANNING ELECTRON MICROSCOPY WITH AUTOMATED POINT COUNT ANALYSIS

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ABSTRACT

Automated point count analysis utilizing scanning electron microscopy (SEM) associated with energy dispersive spectroscopy (EDS) allows for the discrimination of various phases by chemical composition. Phases are discriminated by energy dispersive spectroscopy with "rule" files developed for all phases of interest. Automatic control of the SEM stage and recording of coordinates also allows for the mapping of individual phases by location within a polished concrete section. In this manner, relative quantities of each phase (i.e. ettringite, monosulfate, CSH) may be plotted with respect to depth of the concrete from the surface. Automation of the analysis is achieved by fixing an array of points over selected "standard" magnified paste images. The computer then obtains EDS data from each point within a given image. Images and EDS point data can be electronically stored for subsequent review. Therefore, the frequency with which a given phase coincides within the regular grid system of points allows variations in paste composition to be observed. Comparisons of automated to manual SEM analysis are presented.

IMPACT OF AMORPHOUS AND CRYSTALLINE SILICA ON PYROPROCESSING

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ABSTRACT

Two industrial raw mix test burns were performed on a long, dry (16 ft. by 19 ft. by 540 ft.) Type I cement kiln with a 1 stage preheater; this system burns a coal/coke blend delivered by an indirect firing system. An amorphous silica and microcrystalline silica were substituted for the standard quartz silica in the mix.

This paper provides an evaluation of the impact on the pyroprocessing of the two raw mixes and the affect of quartz greater than 45 μm on the raw mix burnability. To evaluate the raw mix, the parameters of specific heat consumption, production rate, rate of dust wasting, and microscopy are discussed.

RELATIONSHIP BETWEEN MECHANICAL PROPERTIES AND MICROSTRUCTURE OF CEMENT SLURRIES WITH FIBER FOR MULTILATERAL WELLS

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1. ABSTRACT

Multilateral wells are more prone to problems of loss zone isolation during their productive lives than normal wells. Cement systems used in this case have to withstand additional loads and may suffer critical disintegration and/or debonding from the tubular or formation. Several special additives are under test and employed in the petroleum industry, all aiming to improve the mechanical properties of cement sheaths in multilateral wells, and thus assure the hydraulic integrity along the well over its production life.

In this study, mechanical properties such as Young's modulus, flexural strength and Poisson's ratio of cement systems containing fibers are evaluated and correlated with the resulting cement crystallographic microstructure, at different curing temperatures and pressure loads.

INTERNATIONAL CEMENT MICROSCOPY ASSOCIATION
MICROSCOPY OF CEMENT AND CONCRETE

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"IN-HOUSE" DIGITAL IMAGE ANALYSIS SOFTWARE A COMPARISON STUDY, FOR A FAST AND RELIABLE AUTOMATIC MICROSCOPY ANALYSIS.

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ABSTRACT

This paper presents the results of a deep study to evaluate the feasibility of the Image analysis using software tools developed within CEMEX. MICRODIP (Microscopy Digital Image Processing), was programmed few years ago, after a series of improvements and tests, and with a strong program to promote the microscopic methods in the plants, it has been more accepted in the operations.

Right now this tool is more mature and proven and it has been installed in different plants in Mexico and Spain; the study comprised here, describes how this working tools align with the company's direction and it also shows the results of evaluating this tool against normal manual microscopy analysis in different plants and with different clinker samples, highlighting facts such as analysis time, standarization of the analysis and error analysis.

The purpose of this study was to prove the functionality and advantages of the software against normal procedures and commercial softwares, as part of one of the big efforts within the company: the process control through microscopic methods.

SEM MICROSTRUCTURAL STUDIES OF CEMENTITIOUS MATERIALS: SAMPLE PREPARATION OF POLISHED SECTIONS AND MICROSTRUCTURAL OBSERVATIONS WITH BACKSCATTERED IMAGES – ARTEFACTS AND PRACTICAL CONSIDERATIONS

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ABSTRACT

For successful SEM studies of cementitious materials the processes involved in the sample preparation of polished sections need to be carefully considered and applied. Significant artefacts can result from the preparation processes and specific examples of this are given. Some practical considerations are discussed for SEM bse observations of the «real» microstructure and also for reliable EDS microanalyses.

In-situ Observation of Lime Paste Carbonation in CO_2 Environment Scanning Electron Microscopy (ESEM)

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The setting of lime-based mortars is attributed to the process of carbonation but the rate and mechanism of this reaction has yet to be accurately defined experimentally.

Here we report an *in-situ* observation of the carbonation of lime paste within the analysis chamber of an Environmental Scanning Electron Microscope (ESEM). Changes in the microstructure of the lime paste surface induced by the flow of CO_2 or CO_2-rich water vapour have been carefully monitored during initial stages.

Initial results show not only the rate at which Portlandite converts to calcium carbonate but also the parameters influencing the mechanism of the reaction. Environmental scanning electron microscopy can provide in-situ microstructural and micro-chemical characterisation of the dynamic surface changes of materials exposed to various environments.

Fused Beads for Better XRF Analytical Results in Cement Application

Michel Davidts, Lic.Sc.Chim.