NISTIR 6962

The Virtual Cement and Concrete Testing Laboratory Consortium

Annual Report 2002

Editor: Jeffrey W. Bullard

NIST
National Institute of Standards and Technology
Technology Administration, U.S. Department of Commerce

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Building and Fire Research Laboratory

January 2003

U.S. DEPARTMENT OF COMMERCE
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TECHNOLOGY ADMINISTRATION
Phillip J. Bond, Under Secretary of Commerce for Technology
NATIONAL INSTITUTE OF STANDARDS AND TECHNOLOGY
Arden L. Bement, Jr., Director

Contents

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Background

The Virtual Cement and Concrete Testing Laboratory (VCCTL) Consortium was formed in January 2001. Headquartered in the Building and Fire Research Laboratory (BFRL) at the National Institute of Standards and Technology (**NIST**), the consortium originally consisted of three **NIST** laboratories: BFRL, the Information Technology Laboratory (ITL), and the Materials Science and Engineering Laboratory (MSEL), and six industrial members: **Cemex**, **Dyckerhoff AG**, **Holcim Inc**., **Master Builders Technologies** (**MBT**), the **Portland Cement Association** (**PCA**), and **W.R. Grace & Co.- Conn**. The overall goals of the consortium were—and continue to be—to develop a virtual testing system that will reduce the amount of physical concrete testing needed and expedite the research and development process. This will result in substantial time and cost savings to the concrete construction industry as a whole.

The VCCTL Consortium Oversight Board, consisting of one representative from each member organization and the VCCTL Consortium Manager from **NIST**, governs the activities and direction of the consortium. The oversight board meets twice each year to review research progress and set the scope and agenda of future research. Within each industrial participant's laboratory, one researcher is assigned to participate in the research programs of the consortium. Once per year, the most recent version of the VCCTL system software is installed on a Linux-based PC[1] at each participating member's laboratory.

The ultimate goal of the consortium is to address durability and service life prediction, but the members recognized that first efforts must be concentrated towards enhancing the current microstructure models and property calculations. Therefore, the three major initial research topics of the consortium were: 1) enhancements to the cement hydration and microstructure development model to consider additional supplementary materials such as slags and limestones and the prediction of pore solution concentrations, 2) computation of the elastic properties (elastic modulus, creep, shrinkage) of three-dimensional microstructures,

[1] Commercial equipment, instruments, and materials mentioned in this report are identified to foster understanding. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology (NIST), nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

and 3) experimental measurement and computer modeling of the rheological properties (viscosity, yield stress) of cement-based materials.

This document reports the activities of the VCCTL Consortium in 2002.

Overview of Activities in 2002

The 2002 calendar year represents the second year (hereafter called Year 2) of the initial three-year consortium term. Year 2 witnessed significant growth in the membership of the consortium. Sponsored research expanded into new areas, while existing topics of investigation were further refined in response to major progress in research.

Three new members were welcomed into the VCCTL Consortium in Year 2:

- The International Center for Aggregates Research (ICAR), Austin, Texas, USA
- Verein Deutscher Zementwerke eV (VDZ), Düsseldorf, GERMANY
- Sika Technology AG, Zürich, SWITZERLAND (effective January 2003)

In addition, two other organizations have expressed interest in joining the consortium and are expected to become members in Year 3:

- Association Technique de l'Industrie des Liants Hydrauliques (ATILH), Paris, FRANCE
- National Ready Mix Concrete Association (NRMCA), Silver Spring, MD, USA

With these new and prospective members, the VCCTL Consortium is well represented by major research and service organizations, by every major constituent of concrete—cement, aggregate, and chemical admixtures, and by the major manufacturers of concrete.

One of our industry partners, **Degussa Construction Chemicals/MBT**, underwent major organizational changes throughout 2002. An unfortunate consequence of these changes was their decision to discontinue their participation in the VCCTL Consortium for the present. As a chemical admixture supplier, **MBT** was one of our more active participants over the past two years. Their VCCTL representative, Dr. Davide Zampini, was instrumental in modifying the CEMHYD3D hydration model to allow deactivation of clinker surfaces. The consortium will miss the contributions of **MBT**, and of Dr. Zampini in particular, and we sincerely hope that they would choose to renew their participation in the future.

Year 2 was also marked by major strides in research, which built upon the momentum established in the first year [1]. Collaborative investigations between **NIST** researchers and the industry members led to better understanding and improved computer models in several areas of research, as summarized in the table on the following page.

Materials Characterization	Calibration of PSD measurements, aggregate shape and size analysis/database
Hydration	Tracking pore solution composition, model for dependence of hydration kinetics on pH, influence of alkali salts on hydration
Elasticity	Elastic moduli at early ages
Rheology	Cross-calibration of rheometers, simulation of rheometer flows, simulation of suspensions with real aggregate shapes
Durability	Sulfate attack models and database

Considerable effort was placed on upgrading and enhancing the user interface to the VCCTL models. The flexibility and portability of a web-based interface was retained while at the same time adding considerable automation of data entry tasks that previously required tedious and time-consuming typing. The VCCTL User's Guide that accompanies the software has been completely updated, and the web forms in Version 3.0 are now extensively linked to the User's Guide to provide context-specific help for virtually every entry of every form. Users will also notice a new "look and feel" to the interface, some of which will be evident in this report.

Note that each of the major research sections in this report begin with a quote. Several of these quotes were taken from Hal Taylor's famous book, *Cement Chemistry* [2], to pay tribute to this great cement scientist who passed away in 2002.

References

[1] "The Virtual Cement and Concrete Testing Laboratory Consortium Annual Report 2001," NISTIR **6840**, U.S. Department of Commerce. Edited by D.P. Bentz, December 2001.

[2] H.F.W. Taylor, *Cement Chemistry*. 2nd Edition. Thomas Telford Publishing, London, 1997.

VCCTL Technical Notes and Software Notes

Technical Notes

- **Technical Note VCCTL-01**: Estimation of the Degree of Hydration of Portland Cement by Determination of the Non-Evaporable Water Content
- **Technical Note VCCTL-02**: SEM/X-ray Imaging of Cement Powders
- **Technical Note VCCTL-03**: Quantitative Determination of Calciumsulfate Dihydrate and Calciumsulfate Hemihydrate in Cement by Means of Thermogravimetric Analysis (from **Dyckerhoff**)
- **Technical Note VCCTL-04**: Estimation of the Degree of Hydration of Cement by Measurement of Chemical Shrinkage
- **Technical Note VCCTL-05**: Measuring the Viscosity of Cement Paste and Mortar (due in 2003)
- **Technical Note VCCTL-06**: Standard Method for Measuring the Particle Size Distribution of Cement (due in 2003)
- **Technical Note VCCTL-07**: Wet/Dry Cycling Method for Determining Resistance to Sulfate Attack (due in 2003)

Software Notes

- **Software Note VCCTL-01**: *CONCRETEVIEW* software
- **Software Note VCCTL-02**: Software for Hydration/Drying of 2-mm Thick Microstructures
- **Software Note VCCTL-03**: Computation of Cement Paste Elastic Properties

Manuals

- D.P. Bentz and G.P. Forney, "User's Guide to the NIST Virtual Cement and Concrete Testing Laboratory. Version 1.0". NISTIR **6583**. U.S. Department of Commerce, November 2000.

- J.W. Bullard, "User's Guide to the NIST Virtual Cement and Concrete Testing Laboratory. Version 3.0" to be published in early 2003.

Reports

- "The Virtual Cement and Concrete Testing Laboratory Consortium Annual Report 2001". Edited by D.P. Bentz. NISTIR **6840**. U.S. Department of Commerce, December 2001.

VCCTL Publicity

Presentation to the ACI Strategic Development Council as part of the New Technology Showcase, Orlando, FL (November 18, 2002). "VCCTL: A Web-Based Virtual Cement and Concrete Testing Laboratory."

Special presentation by E.J. Garboczi to the Research Committee of the National Ready-Mixed Concrete Association, Arlington, VA (October 6, 2002*)*. "VCCTL: A Web-Based Virtual Cement and Concrete Testing Laboratory."

Special 90-minute presentation by J.W. Bullard and E.J. Garboczi at the Portland Cement Association meeting, Chicago, IL (September 16, 2002). "VCCTL: A Web-Based Virtual Cement and Concrete Testing Laboratory."

Daily Commercial News and Construction Record (September 13, 2002). "Facility Provides Virtual Testing for Concrete."

Special symposium organized by G.J. Frohnsdorff and C. Ferraris, presented to ASTM Committees C-01 and C-09, Salt Lake City, UT (June 26, 2002). "The Virtual Cement and Concrete Testing Laboratory."

- *Concrete Construction* (November, 2001). "Virtual Concrete Laboratory"
- *The Concrete Producer* (October, 2001). "E-Concrete: Let's Try a Little Left Thinking"
- *Government Computer News* (August 27, 2001). "NIST Team Sees in Stereo" (available at http://www.gcn.com/20_25/news/16941-1.html)
- *Engineering News Record* (June 25, 2001). "Website Aims to Cut Concrete Analysis" (available at http://www.enr.com/itnews/it62501e.asp)
- *Civil Engineering* (June, 2001). "Virtual Concrete Lab Saves Time, Reduces Material Cost"
- *NIST Technology at a Glance* (Spring, 2001). "Co-Op Corner, Virtual Cement Laboratory"
- *NIST Update* (February 20, 2001). "Virtual Lab Consortium to Test Concrete and Cement Formulas"
- *Concrete Technology Today*- PCA (December, 2000). " The Future of Materials Testing? Virtual Testing of Concrete Will Save Time and Money"

VCCTL Newsletter. In Year 2, NIST began publishing a quarterly *VCCTL Newsletter*, which is distributed to members of the VCCTL Consortium and to users who login to the public domain site for the VCCTL software (Version 1.0). The newsletter highlights applications by users of the public domain version and also gives periodic tips for using the software. The VCCTL Newsletter is intended as a source of useful information as well as a publicity tool for the consortium.

VCCTL Consortium Members

NIST

Dr. Jeffrey W. Bullard (BFRL)
Dr. Judith Devaney (ITL)
Dr. Chiara F. Ferraris (BFRL)
Dr. Glenn P. Forney (BFRL)
Dr. Edward J. Garboczi (BFRL)
Dr. Vincent A. Hackley (MSEL)
Mr. John Hagedorn (ITL)
Mr. Peter Ketcham (ITL)
Dr. Nicos S. Martys (BFRL)
Mr. Steve Satterfield (ITL)
Mr. Paul Stutzman (BFRL)
Mr. John Winpigler (BFRL)

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Portland Cement Association

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W.R. Grace & Co.- Conn.

Dr. Vijay Gupta
Dr. David Myers

Dyckerhoff AG

Dr. Claus-Jochen Haecker

Association Technique de l'Industrie des Liants Hydrauliques (ATILH)

Dr. Alain Capmas
Prof. Denis Damidot (Mines de Douai)

International Center for Aggregates Research (ICAR)

Prof. David W. Fowler (U. Texas at Austin)

Materials Characterization

> *There is an underlying philosophy [in] the branch of engineering known as "materials science": To understand the **properties** (i.e. observable characteristics) of engineering materials, it is necessary to understand their structure on the atomic and/or microscopic scale. [1].*

Background

Without meticulous characterization of the starting materials, it is practically impossible to formulate fundamental microstructure models of the hydration and properties of a cement paste, mortar, or concrete. One of the signature features and strengths of the CEMHYD3D hydration model is its incorporation of microstructural information with unprecedented levels of detail.

Proper characterization of cement-based materials is not a trivial task. The starting powders typically consist of a wide distribution of particle sizes, each particle of which is composed of multiple phases with variable chemical compositions. Many products of hydration are amorphous or poorly crystalline, and these may be finely intermixed. An additional complication is that some microstructural characteristics have not been rigorously defined. In some cases it has not been possible even to properly calibrate measurements, like those of particle size distribution (PSD), because of the absence of an accepted reference material or standard methodology.

Recognizing the critical importance of materials characterization for predictions of microstructure development, physical properties, and durability, the consortium has continually sought to achieve greater accuracy and detail in these types of measurements, and to supply robust, calibrated measurement techniques where those have been lacking.

Activities in Year 1

Particle Size Distribution. In collaboration with ASTM Subcommittee C01.25, it was established that there is no standard test for measuring the PSDs of cements. **NIST** obtained data generated by an ASTM-sponsored round-robin test in 2000. **NIST** used these data to initiate an analysis of the type of techniques that are used most commonly, and to develop a method for determining a statistically valid mean PSD from the distributions generated by multiple laboratories. A small VCCTL round-robin test also helped determine a logical course of action: 1) establish a reference material, and 2) determine which parameters need to be controlled using the laser diffraction technique (wet or dry).

SEM/X-ray Imaging. **NIST** has pioneered the use of SEM/X-ray imaging for the quantitative characterization of cement powders [2]. This analysis produces a 2-D image of the cement powder in which each pixel is identified as one of the major phases of portland cement, as shown in Figure 1.

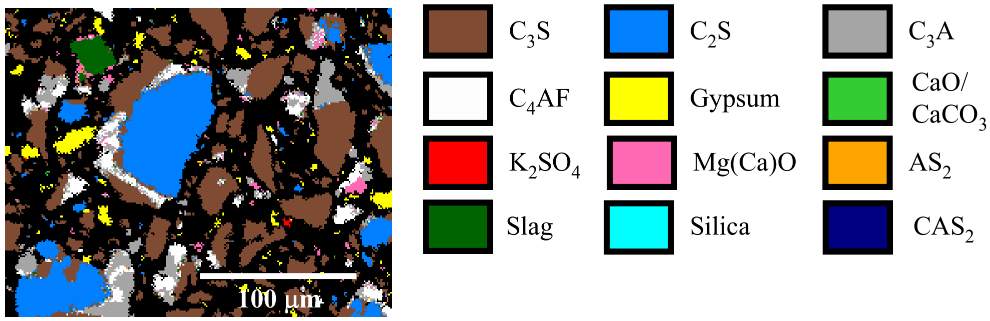

Figure 1: Processed 2-D image of CCRL Cement 140.

Research in Year 2

Particle Size Distribution. At the May 2002 meeting of the VCCTL Consortium, it was agreed that to improve measurements of cement particle size distribution, it is neccesary to develop a method for separating the individual PSDs of clinker and gypsum phases in interground cements. Initial efforts focused on physically separating gypsum from clinker in interblended or interground cements, by selectively sedimenting the powder in a liquid having a density between the average densities of gypsum and clinker phases. However, this method was unsuccessful, probably due to some degree of interphase particle agglomeration. Common methods to deflocculate a powder suspension, such as the use of a chemical dispersant and ultrasonic agitation, were investigated but also were unsuccessful. A more promising method may be to use a laser scattering technique to measure the particle size distribution of a cement that has been suspended in a liquid, the refractive index of which is accurately matched to that of the calcium sulfate phase(s). The close matching of refractive indices could effectively eliminate the gypsum particles as scattering centers, thus allowing the measurement technique to sample only the clinker particles. Preliminary results of this method indicate that benzyl alcohol/ethanol mixtures can be designed to closely match the average refractive index of gypsum.

With the recognition that a standard method for measuring PSD does not exist, the consortium took the lead in trying to establish such a standard. Working through ASTM Subcommittee C01.25, Dr. Ferraris (**NIST**) prepared two reports analyzing two ASTM round-robin tests [3,4]. The first round-robin was organized by ASTM and included data from 21 participants. Because no information was gathered during this round-robin about how the measurements were made (only the generic method was stated) it was deemed insufficient to establish a standard method or a reference material. Nevertheless, it helped develop the methodology to analyze the data. The second round-robin was planned by **NIST** in collaboration with ASTM and included laboratory members of the proficiency program from the Cement and Concrete Reference Laboratory (CCRL) as well as VCCTL members. There were 41 participants in this round-robin. As data were collected on the parameters used to prepare the specimens and to analyze them, it was used to define a reference material and some basic understanding of the standard method. Reference material SRM-114p, distributed by **NIST** and used for Blaine calibration, was selected. The PSD established in the second round-robin will be included in the SRM-114p certificate in early 2003. The reference material would be used to "calibrate" an instrument or to validate the procedure used in a laboratory. It was also established that most (93 %) of the cement laboratories use a laser diffraction technique, with either liquid suspension or aerosol handling of the cement powder. Therefore, **NIST** agreed to develop a standard procedure to be submitted to ASTM in Year 3, based on laser diffraction.

Aggregate Shapes. Ed Garboczi (**NIST**) began a joint project with **ICAR** to mathematically represent and analyze aggregate shapes. The project encompasses (1) developing ways to experimentally capture the three-dimensional images of aggregate assemblies, using X-ray computed tomography, (2) using image analysis to isolate individual aggregate pieces from the data, and (3) creating and storing a mathematical description of the aggregate shape using spherical harmonic expansion [3]. Several trials were made to validate the general procedure, and an assessment was made of the convergence of the spherical harmonic expansion. Figure 2 shows a comparison of the spherical-harmonic expansion of the initial data for a single rock particle.

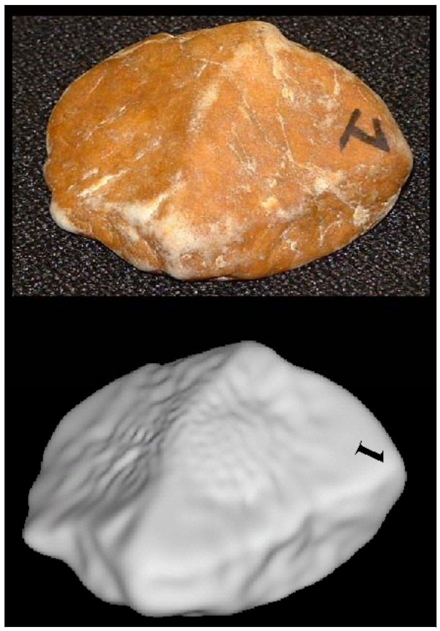

Figure 2: (Top) Photograph of a streambed rock (volume = $5x10^4$ mm^3). (Bottom) Spherical harmonic expansion, using 300 terms, of a computed tomography (CT) scan of the same rock.

The ultimate objective of this project is to provide a database of aggregate shapes for accurately modeling the macrostructure of concrete. A skeletal database of shapes will be included in Version 3.0 of the VCCTL software. Ongoing work is focusing on analyzing different types of aggregate (e.g. riverbed vs. crushed) for common characteristics and on enhancing the database. **Case Study II** in this report provides additional detail and validation of the spherical harmonic analysis of several rock samples by comparing the predicted volume of each to the true volume measured by immersion in a liquid.

SEM/X-ray Imaging. Much of the microstructural characteristics of cements are obtained by segmenting a series of 2-D SEM images into constituent phases, with the aid of X-ray maps for the relevant elements. Segmentation is accomplished pixel-by-pixel, using an algorithm that steps through a decision tree based on the intensity of the X-ray signal for each element at a particular location. In Year 2, the decision tree was expanded to include slag-like material and a calcium aluminosilicate compound as possible additional phases, as shown in Figure 1. In addition, the color scheme for representing phases was changed to more closely match the actual colors of the phases when they are HF-etched and examined by reflected-light microscopy.

Although it is fairly routine, the segmentation procedure involves a considerable amount of user intervention and somewhat subjective decisions, especially when thresholding the intensities of the signals for each element's X-ray map. Researchers in the Information Technology Laboratory (ITL) at **NIST** are actively pursuing applications of machine learning, which can be a powerful tool for analysis of complicated images. The SEM segmentation procedure could be made simpler and more automated if a way could be developed to implement machine learning. With sufficient testing and documentation, such a segmentation protocol could be distributed to the VCCTL Consortium members as part of the software within a year.

Future

Work in Year 3 will build on the research progress made in 2002, and will include the following research/development activities:

Particle Size Distribution Analysis

1. Validate refractive index matching to gypsum as a way to isolate the particle size distribution of clinker in interground/interblended cements.
2. Develop formulations for benzyl alcohol/ethanol mixtures that are index-matched respectively to gypsum, hemihydrate, and anhydrite forms of calcium sulfate.
3. Create and validate an algorithm for deconvoluting the individual contributions of two separate particle size distributions from a mixture in which one of the PSDs is known.
4. Complete documentation of reference data for PSD of SRM-114p
5. Develop next-generation SRM 114q as a standard reference material for calibrating particle size analyzers.
6. Draft a standard cement PSD measurement method as a VCCTL TechNote and for ASTM.

Aggregate Shape Characterization

1. Refine and optimize procedures of image and mathematical analysis of aggregates (joint work between **NIST** and **ICAR**)
2. Extend the project to characterize shapes of cement particles, and incorporate cement particle shape in CEMHYD3D hydration model.
3. Search for more generally available 3D imaging techniques, besides X-ray computed tomography, for use by consortium members.

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[1] J.F. Shackelford. p. 11 in *Introduction to Materials Science for Engineers*. Macmillan Publishing Company. New York, NY (1985).

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[3] C.F. Ferraris, V.A. Hackley, A.I. Aviles, and C.E. Buchanan, “Analysis of the ASTM Round-Robin Test on Particle Size Distribution of Portland Cement: Phase I”. NISTIR **6883**. U.S. Department of Commerce, May 2002. Available at http://ciks.cbt.nist.gov/~monograph/nist6883/nistir6883.htm .

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[5] E.J. Garboczi, “Three-Dimensional Mathematical Analysis of Particle Shape Using X-Ray Tomography and Spherical Harmonics: Application to Aggregates Used in Concrete,” *Cem. Conc. Res.* **32** [10], 1621-38 (2002). Available at http://ciks.cbt.nist.gov/monograph/paper134/mono134.html .

Hydration Research

> *Mathematical modelling [of hydration] has the objective of quantifying knowledge of the hydration process and microstructure of the resulting material ...thus allowing the effects of changes in inputs or assumptions contained in the model to be examined [1].*

Background

Because the physical, chemical, and durability properties of cement and concrete are ultimately dependent on their hydrated microstructures and phase composition, a microstructure-based model of hydration serves as the core of the Virtual Cement and Concrete Testing Laboratory software [2]. The 3-D model of hydration (**CEMHYD3D**) [3,4] is currently recognized as the most extensive, complete, and robust cement paste micro-structure model in the world. The general flow of the model may be summarized by several steps, as shown in Fig. 3. As hydration executes in the model, a series of output files is created to monitor specific properties—chemical shrinkage, heat release, setting, capillary porosity percolation, pH, and strength.

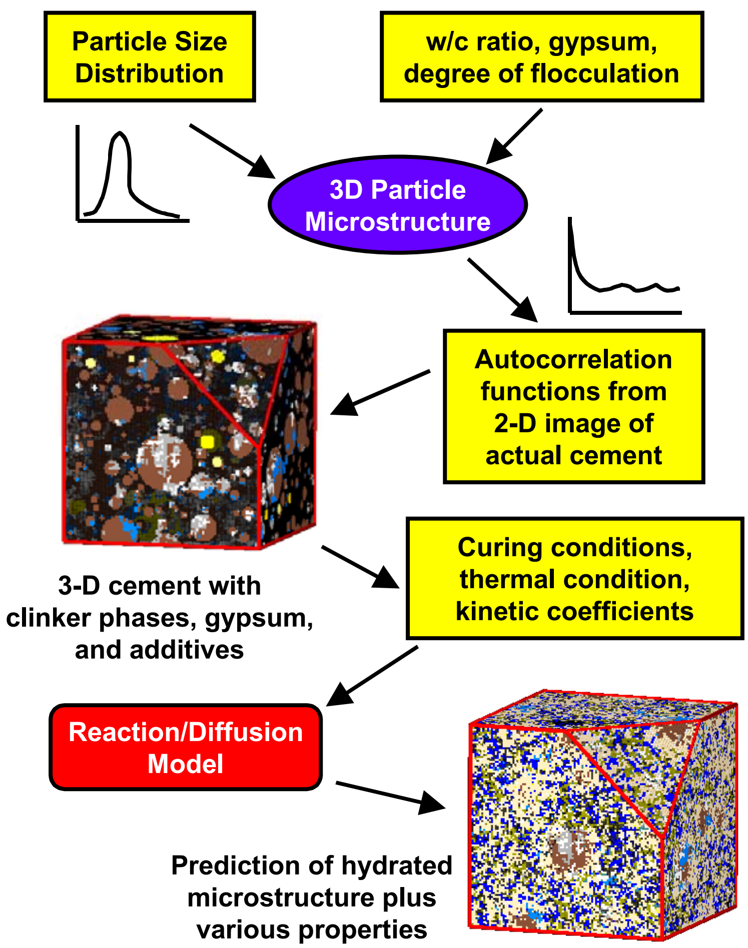

Figure 3: Schematic of CEMHYD3D models.

Activities in Year 1

During 2001, research focused on three subtopics: 1) modeling and experimental measurements on systems containing limestone fillers, 2) development of a framework for incorporating reactions for slag into CEMHYD3D, 3) modeling of the pH and ion concentration of the pore solution during hydration, 4) creation of options in CEMHYD3D for curing under sealed conditions and for specifying the nature of heat transfer between system and surroundings, and 5) addition of a supplementary model to monitor hydration/drying in 2 mm-thick slabs [5].

Research in Year 2

Influence of Alkali Species on Hydration. This project was initiated in late 2001 and has made substantial progress in Year 2, although several outstanding questions remain. The goal of this project is to link pore solution pH to hydration kinetics and to validate the modeling with inter-laboratory experiments on different cements with intentional addition of sodium and potassium salts. Modification of CEMHYD3D was undertaken at **NIST** to make a preliminary accounting of the acceleration in hydration with increasing pH, in which the dissolution probability of all four clinker phases was made to depend in the same way on pH. Preliminary experiments, also conducted at **NIST**, revealed several complications, such as the importance of experimental mixing procedure in establishing a homogeneous distribution of alkali species, and the possibility of syngenite precipitation in cements with high initial potassium content. Suitable modifications were made to CEMHYD3D to allow for syngenite formation, and modifications were also made to experimental mixing procedures to better homogenize the alkali species. Concurrent experiments for validation were conducted on three different cements at **NIST**, **Cemex**, and **Dyckerhoff** during Year 2. The three cements examined were CCRL Cement 140 (at **NIST**), a white cement (at **Cemex**), and a Type I Portland cement (at **Dyckerhoff**). The model predictions of degree of hydration closely matched experimental results for CCRL Cement 140, but were considerably poorer for the other two cements. Specifically, the predicted degree of hydration at later ages (e.g. 28 days) was much higher than the observed degree of hydration. The discrepancy is likely due to unrealistic or incomplete model assumptions, but the possibility of systematic differences in experimental procedure at the three laboratories cannot yet be discounted. Therefore, an investigation of the discrepancies in Year 3 will involve

1. repeating the experiments on identical equipment at **NIST** (to ensure minimal systematic error due to differences in procedure or equipment calibration),

2. repeating at **Cemex** and **Dyckerhoff** the experiments using CCRL Cement 140, and

3. making a critical reexamination of CEMHYD3D to better account for other influences of alkali species other than pH. This will also involve the incorporation of more realistic algorithms for altering the solubility of anhydrous phases.

Despite the mixed results of these experiments, Version 3.0 of the VCCTL software will include an option in CEMHYD3D to allow pore solution pH to influence hydration rates. As CEMHYD3D is improved in this respect, upgrades to the model will be distributed to the consortium members.

Future

In addition to this continuing work on alkali influences, Year 3 will see an effort to further refine CEMHYD3D for predicting hydration behavior of blended cements with slag and/or fly ash additions. Limestone reactions will also be further incorporated and validated. These are areas in which the expertise and collaboration of several consortium members, including **Cemex**, **Dyckerhoff**, **VDZ**, and **W.R. Grace**, will be essential for making rapid progress.

Specific Goals

1. Collect fundamental experimental data on solubility curves of clinker compounds as a function of pore solution composition/pH.

2. Modify CEMHYD3D to allow for possible pH influences on individual cement clinker phases and hydration product characteristics. Make preliminary tests of modified models with existing experimental data.

3. Repeat at **NIST** the alkali-effect experiments conducted by **Dyckerhoff** and **Cemex**, to separate the influences of different material sets from the possible influences of instrumentation and measurement techniques at these laboratories.

4. Repeat at **Cemex** and **Dyckerhoff** the alkali-experiments conducted by **NIST** on CCRL Cement 140.

5. Design and perform experiments to test the effects of soluble gypsum forms on the influence of alkali components.

6. Extend the model to include the dependence of the reactivities of slag and fly ash on pore solution pH.

References

[1] H.F.W. Taylor, p. 237 in *Cement Chemistry*. 2nd Edition. Thomas Telford Publishing, London, 1997.

[2] D.P. Bentz and G.F. Forney, "User's Guide to the NIST Virtual Cement and Concrete Testing Laboratory. Version 1.0," NISTIR **6583**, U.S. Department of Commerce, November 2000. Available at http://vcctl.cbt.nist.gov/vcctlman .

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Rheological Properties

> *...fresh concrete is no longer considered as a material with a single parameter, called consistency or workability [1].*

Background

At **NIST**, there is an ongoing program with the goal of predicting the rheological performance of concrete from its composition. A hierarchical approach, starting with cement paste and progressing to mortar and concrete, is used by which the rheological properties at one level are used as input to compute those at the next. This approach consists of the following steps:

1. measuring the rheological properties of the cement paste to determine the influence of chemical admixtures and supplementary cementitious materials,

2. measuring mortar rheological properties to determine the influence of air and sand content,

3. predicting rheological properties of concrete via computer simulation [2-4] using measurements on mortar and/or cement paste and information about the size and shape distributions of the coarse aggregates

A novel computer simulation approach allows **NIST** to take the rheological properties measured on mortar, add the coarse aggregates with their shape and PSD, and predict the concrete rheological properties. The advantage of this approach is that several tests could be done by one operator on cement paste or mortar in a day, while concrete testing requires several operators and much more material. The model is based on the theory of dissipative particle dynamics (DPD) [5-7], which combines aspects of cellular automata and molecular dynamics. The method described in Refs. [6,7] was modified by Martys at **NIST** for application to the flow of concrete. The model itself requires powerful computers, so **NIST** has built a database, included in Version 2.0 of the VCCTL software, that catalogues the relative viscosity[1] of a concrete with a coarse aggregate defined by its size distribution. The database at present does not include the effect of aggregate shape.

The goals of the VCCTL Consortium in this area are 1) to establish standard tests for the influence of air and mix proportioning on the viscosity of mortar, 2) to validate the modeling approach for a wide range of systems with different supplementary cementitious materials (SCM) and chemical admixtures, and 3) to extend the VCCTL database to include realistic shapes and wider size distributions of common aggregates.

[1] The relative viscosity is the ratio between the viscosity of the concrete and that of the mortar with the same composition as the mortar component in the concrete.

Activities in Year 1

A methodology to test mortar was developed by modification of the parallel plate rheometer used previously for cement paste. A database was created to collect aggregate size distributions and graphs of relative viscosity versus aggregate concentration. A partial validation of the modeling approach was made possible by rheological measurements on mortar and concrete, provided by **W.R. Grace** using a BML rheometer [8,9] and **Degussa/MBT** using an IBB rheometer.

The DPD model was modified to include air as a second fluid phase. Air was introduced as spherical bubbles, which may subsequently deform under shear forces. The two fluid phases were modeled as being immiscible to prevent dissolution of air into the paste. Validation of this model was an objective of Year 2 research.

Research in Year 2

Standard Measurement Method. One aspect of the project is to determine experimental procedures and materials that can be used for cross-calibration of results on different types of rheometers in different laboratories. Considerable progress has been made in pinning down the effects of rheometer geometry (the wall, plate gap, and specific effects of plate serration). A VCCTL TechNote, which describes the methodology developed to measure the rheological properties of cement paste and/or mortar, will be available in late January 2003. An interlaboratory experimental study, performed at **W.R. Grace** and **Degussa/MBT**, provided data on concrete and mortar viscosities and their dependence on air content. Plots of relative viscosity versus volume fraction of coarse aggregate were shown to be in reasonably good agreement across laboratories using different rheometer geometries, as shown in Figure 4. These results lend support to the claim that the influences of geometry and experimental procedure can be scaled out of the measurements to some extent, yielding truer measurements of the actual rheological properties of the material, by using relative viscosity instead of plastic viscosity [10]. The influence of air content on the flow of concrete was also investigated, and Fig. 5 shows the relationship between air content and relative viscosity while the coarse aggregate concentration in concrete or the sand concentration in mortar is kept constant. It seems that the relative viscosity increases with air content up to about 10 % and then decreases at higher values. The model predicts correct trends in the relative viscosity up to 10 % air content.

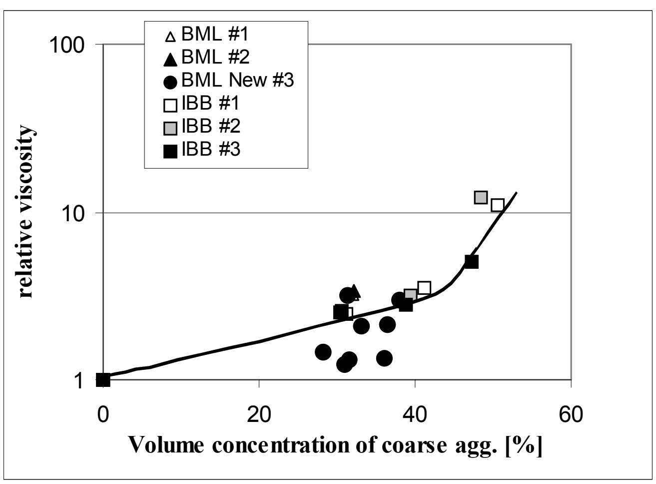

Figure 4: Relative viscosity vs. volume concentration of coarse aggregates in concrete. Comparison of results from various rheometers. The line is given only to guide the eye and it is not intended as a reference function.

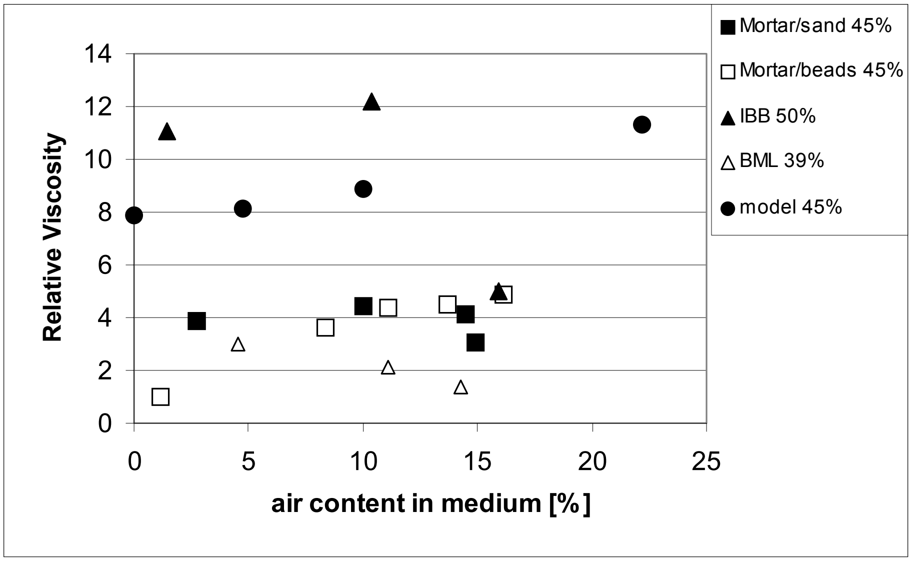

Figure 5: Influence of air on the relative viscosity. Comparison of results from modeling, mortar measurements and concrete measurements.

Modeling. The DPD model, modified to simulate the flow properties of fresh concrete, shows good agreement with experimental measurements of relative viscosity as a function of coarse aggregate volume fraction (see Fig. 6). This model has been used to incorporate realistic crushed aggregate shapes (see the section on *Characterization of Aggregate Shapes* on p. 5). The use of more realistic shapes appears to make qualitative and quantitative changes in the predictions of viscosity and of flow through rebar compared to that using spherical aggregate shapes (see **Case Study III**). The differences are probably at least partly due to differences in the packing efficiency of the aggregate. DPD models can be used to simulate the influence of entrained air, as well as to simulate the flow of fresh concrete within different types of rheometers.

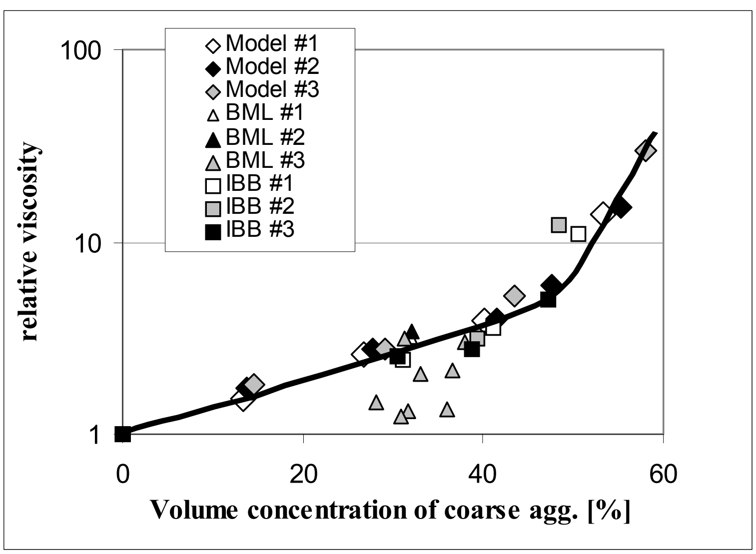

Figure 6: Comparison of DPD model to experimental data on the dependence of relative viscosity of fresh concrete on the volume fraction of coarse aggregate. The first three sets of data in the legend are the simulations, and the remainder are the experimental data as in Fig. 4.

Version 4.0 of the VCCTL software will include an expanded database of aggregate shape and size distributions to allow the user more flexibility in approximating an actual concrete for computation of relative viscosity. Version 4.0 also should include the air content as a parameter to consider in the prediction of relative viscosity.

Future

VCCTL research into rheological properties is poised for some important breakthroughs in 2003. A large part of the systematic work to establish standard measurement techniques has been completed. Further modeling work is necessary to determine the flow patterns formed in different rheometers, and to validate the DPD model with experimental data. A new 128-processor computer acquired by **NIST** in 2002 will greatly accelerate this work. With these foundations in place, future work will exploit the power of combined modeling and experimental research to reveal important relationships between mix proportioning and the flow properties of fresh concrete. Specific goals include:

1. Finalize TechNote that describes procedure for measuring viscosity of cement paste and/or mortar.

2. Gather more measurements on mortars and concrete with different air contents and air-entraining agents, in collaboration with **W.R. Grace**.

3. Develop the model and database to examine real-case scenarios

   - Simulate parallel plate rheometer with walls

   - Initiate work in collaboration with **SIKA** to model interactions between particles

   - Increase the database to include shape and more distributions of aggregates

   - Further validate and develop the model to include the influence of air content on flow. Modify the database to include the influence of air content.

4. Investigate the usage of the LCPC model to predict the yield stress from the concrete composition.

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Elastic Properties

> *Cement paste is extraordinarily complex elastically, with many… different chemically and elastically distinct phases and a complex microstructure. This complexity further increases in concrete, as aggregates are added [1].*

Background

The elastic behavior of engineering materials, especially those materials intended for structural applications, is a critical design factor for any project in which they will be used. For this reason, the elastic properties of metals, ceramics, and structural composites have been subjects of experimental and theoretical interest for many years. The same cannot be said for concrete, however. Though one of the most extensively used structural materials in the history of civilization, concrete has probably had the least attention paid to it by composite theoreticians and experimentalists. This situation is probably due, in part, to the enormous structural complexity of the material. Concrete is a random composite material over many length scales, consisting of phases, e.g. aggregate and capillary porosity, that differ in their intrinsic elastic moduli by many orders of magnitude. As a result, the effective elastic moduli of concrete cannot be predicted accurately by most of the analytic theories that are useful for other classes of materials.

Despite the little progress that has been made in *predicting* the elastic properties of cement and concrete, these properties are of great importance to industry. In addition to the significance of elastic response in the design of structures, there is growing interest in using the elastic moduli of a mortar or concrete as an estimator of other important mechanical properties such as its compressive strength. (Prediction of strength from theoretical models is even more difficult than the prediction of elastic moduli). As a result, a major component of research within the VCCTL Consortium is directed toward developing robust numerical procedures that can predict the effective elastic moduli of cement, mortar, and concrete.

Activities in Year 1

A finite element package for computing the elastic moduli of composite materials has been written at **NIST** [2] and validated on a diverse range of material classes [3-6], and was incorporated into Version 2.0 of the VCCTL software package. The finite element program operates directly on the same digital images that are generated by the other components of the VCCTL software, such as CEMHYD3D. This enables the true spirit of a “virtual lab” to be realized, because the user can create, hydrate and compute elastic properties of a microstructure, all within the same software package. It is important to realize, however, that the elastic calculations are only as good as the phase elastic moduli input. One ongoing task of the VCCTL Consortium is to measure the individual phase elastic moduli needed, along with cement paste elastic moduli as a function of degree of hydration, cement particle size, and w/c ratio. This work will help develop the easy-to-use elasticity software module into a tool that is even more accurate.

As part of that effort, **Dyckerhoff** provided experimental measurements of the elastic moduli of a series of cement pastes, with w/c = 0.25–0.60, and cured under saturated conditions for various times. **Dyckerhoff** also provided experimental data on the elastic moduli of polycrystalline ettringite. **NIST** researchers gathered published values of the intrinsic elastic moduli of minerals commonly found in cement/cement paste. Agreement between model and experiment was quite reasonable (usually within 5 %) for later-age specimens, but more serious discrepancies were evident for early-age specimens.

Research in Year 2

In Year 2, research focused on refining the predictions of the elastic moduli of cement paste at early ages (hours to several days after set). The finite element method used in the VCCTL software, by default, establishes an elastic connection between any two solid pixels that are in contact across faces, edges, or corners, even when the pixels in question are anhydrous cement phases belonging to two different particles. A compelling argument can be made for the elimination of connections between different anhydrous particles on the basis that such particles experience only weak van der Waals attractions that are easily broken in elastic measurements. When such connections are eliminated by removing the elastic stiffness from one of each pair of connecting pixels, remarkable improvements are made in the agreement between early-age predictions and experimental observations, as shown in Fig. 7.

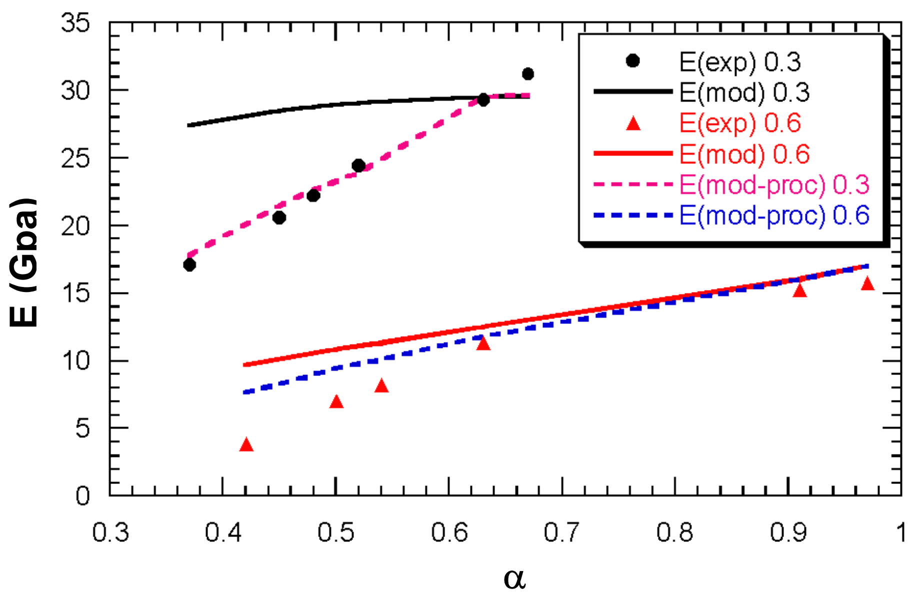

Figure 7: Comparison of measured and predicted values of the elastic modulus of a cement paste as a function of degree of hydration, α. The symbols represent measured values, the solid lines are model predictions when uncorrected for artificial connections between particles, and the dashed lines represent model predictions that are corrected by elminating such artificial connections. The upper set are for a paste with w/c = 0.3, and the lower set are for a paste with w/c = 0.6.

The experimental data provided by **Dyckerhoff** were obtained by dynamic acoustic resonance techniques. The accuracy of the model predictions were shown to improve from nearly 200% error, for the original model, to no greater than 5% error for the corrected model at low w/c ratios. For higher w/c ratios (≈0.6) the agreement is poorer, and current research effort is being placed on determining the source of the error and ways to improve those predictions.

Future

In Year 3, further validation is expected of the finite element code for elastic properties of cement. Additionally, research activity will broaden to begin to investigate reliable and accurate ways to address the elasticity of concrete. The following items will be undertaken in Year 3.

1. Establish validity of corrected elasticity calculations for other cement systems

2. Test the validity of the corrected elasticity calculations at increased system resolution

3. Investigate sources of error in the early-age calculations at higher w/c ratios, and modify calculation method as necessary

4. Document a procedure for making dynamic measurements of elastic moduli so that inter-laboratory comparisons can begin to be made.

5. Investigate applicability of effective medium theory to model the elastic properties of concrete.

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Durability Research

> *To design a concrete for a specified minimum life, it is necessary to understand the processes that cause deterioration, including the rates at which these will occur under the conditions to which it will be subjected [1].*

Background

One of the ultimate goals of the VCCTL Consortium is the prediction of service life of concrete based on fundamental knowledge of the starting materials, mixture proportioning, curing conditions, and environmental factors. A moment's reflection will reveal how complex an exercise it is to make accurate predictions. The field durability of concrete depends on a myriad of phenomena that interact and compound, beginning with its initial placement. One must have a reasonably complete understanding of microstructure development during curing, and of the mechanisms by which the microstructure deteriorates due to environmental exposure.

The VCCTL models have progressed to offer a comprehensive treatment of microstructure development and properties of cement pastes, and have begun to expand to include aspects of aggregate. Although our understanding of the mechanisms of microstructure development is by no means complete, the framework is in place to enable some preliminary modeling of durability. For example, Versions 1.0 and 2.0 of the VCCTL software include modules for simulating the leaching by water of portlandite and other phases from a hydrated cement paste microstructure. And beginning in Year 2, VCCTL research on durability expanded to include attack by sulfate salt solutions. These efforts are clearly at an early stage of development, and research in Year 3 is expected to continue at an increasing rate.

Research in Year 2

VCCTL research in durability has focused in Year 2 on the resistance of cement pastes to degradation by sulfate attack, and this work has proceeded along two complementary paths.

Sulfate Resistance Database. Researchers at **NIST** are developing a searchable database that documents experimental observations of the resistance of different cements to sulfate attack. For each cement, the database will include microstructural observations as well as data on the time dependence of expansion, elastic modulus, and other macroscopic properties as a function of exposure conditions. The database will continue to be developed and maintained at **NIST**, and in Year 3 it will be directly linked with the VCCTL software (Version 4.0). The development of the sulfate resistance database is supported by ongoing experimental investigations at **NIST**. This work involves monitoring deterioration of specimens prepared with various blended cements.

Extensive evidence from microstructure characterization indicates sharp fronts of reaction products (consumption of monosulfate, decalcification, and gypsum formation, for example), as shown in Fig. 8. The microscopic observations also reveal a potentially misleading artifact of the measurement technique: excessive reaction around the pins of the specimen can make the linear expansion appear higher than it actually is. This artifact has not been recognized previously, and earlier data should be revisited to determine their validity. It is possible that much smaller specimens will provide all the information necessary, thus accelerating the test greatly. This possibility is being explored currently.

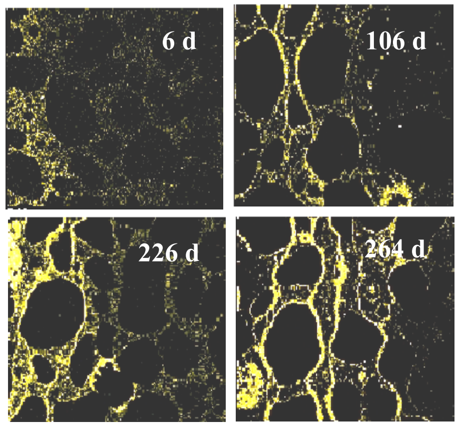

Figure 8: Mortar immersed in a solution of Na_2SO_4 for varying times. Yellow regions indicate replacement of CH by gypsum.

In the field, it is often reported that changes in temperature and/or wet-dry cycles accelerate the deterioration of concrete. A new test method was developed at **NIST** to investigate the validity of this observation and to determine the material parameter(s) that influence the deterioration rate. This study aims at elucidating the influences of thermodynamic phase stability, pore-size distribution, and crystallization pressure. **VDZ** helped characterize some of the specimens that are under investigation at **NIST**.

All of these observations are being used to formulate and implement improved experimental procedures for testing cement and concrete resistance to sulfate solutions. It should be noted that this experimental program is an excellent example of the ways that resources are leveraged at **NIST** to provide enhanced productivity to the VCCTL Consortium effort. The development of the sulfate resistance database and the associated experimental program were sponsored by a separate three-year project through the **Portland Cement Association** (PCA Project 99-07).

Modeling Sulfate Attack. A microstructure model, developed jointly by Dale Bentz (**NIST**) and Erik Pram Nielsen (Aalborg Portland A/S) to simulate attack of cement paste by $MgSO_4$ solutions was created in Year 2. The model operates according to principles similar to CEMHYD3D (agent-based reaction/diffusion events). Along with changes in the microstructure (see Figure 9), the model also tracks quantities like the volume of each phase that has reacted and the effective strain as a function of depth, which results from insufficient reaction space. This model is still in the development/validation stage of research, but will be included in Version 3.0 of the VCCTL software to encourage its testing by the consortium.

At larger length scales, a continuum model of ionic transport in cement pore solutions, called 4SIGHT, has been developed and validated over the past seven years [2]. More recently, the potential of modifying that program to include the types of reactions that occur during sulfate attack has been demonstrated persuasively [3]. With further work, 4SIGHT has the potential to accurately predict the velocity and nature of the reaction front during attack by sulfate solutions ($MgSO_4$ or Na_2SO_4). Another example of leveraging within **NIST**, this model was developed with support from the **Nuclear Regulatory Commission**, but may be used in Year 3 to supplement the VCCTL effort in this area. A powerful idea is to couple 4SIGHT with the microstructure model described in the previous paragraph. The result would be a multi-scale model of sulfate attack, combining the advantages of the well-established thermodynamic and kinetic principles of 4SIGHT with the ability to model changes in formation factor that accompany microstructure changes. **ATILH**, a new member in Year 3, is sponsoring that kind of research and will help with this part of the VCCTL research.

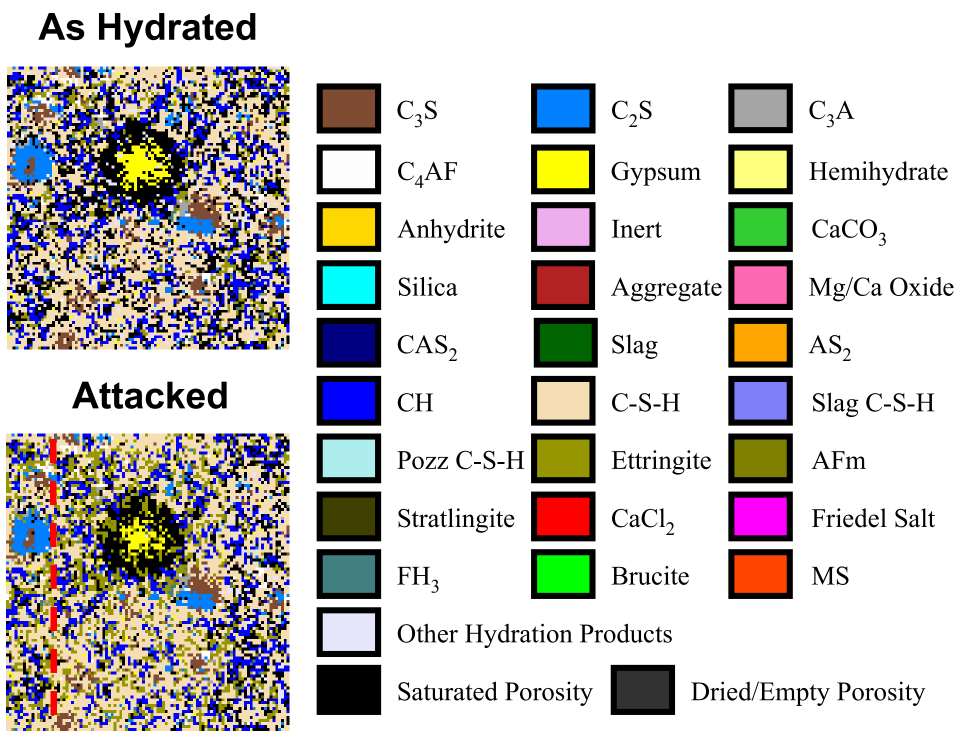

Figure 9: Microstructure changes predicted by sulfate attack model after 100K cycles. The as-hydrated cement is a CCRL 140 cement with w/c = 0.4, hydrated for 28 d before commencing sulfate attack.

Future

As the foregoing description indicates, research methods into durability, although not new, are being systematized in a way that enables proper comparison/validation of results. A portion of the VCCTL activities in this area, therefore, will concentrate on developing standard test methods for assessing resistance to sulfate attack. Other research will include ongoing work to develop and validate multiscale models of sulfate attack. Specific goals include:

1. Continue validation of 4SIGHT and VCCTL models of sulfate attack, possibly coupling these two methods together.

2. SEM/XRD analysis of all specimens currently under test at **NIST**

3. Publish a TechNote on how to determine the sulfate resistance of a hydraulic cement using a combination of measurements and modeling.

4. Complete the sulfate attack database containing all data collected at **NIST**; provide search tools and uniform structure

5. Sponsor sorptivity test through ASTM

6. Finalize wet/dry cycling method and sponsor it through ASTM, and prepare a TechNote describing the method

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Case Study I

Later-Age Elastic Properties of a Type I Portland Cement

This case study is a good example of the value of consortium collaboration. Version 2.0 of the VCCTL software included an initial version of the elastic code for directly computing the elastic properties of cement paste for any degree of hydration. The code was thought to be accurate for later ages, past 50 % to 60 % degree of hydration, although it had only been tested for one kind of cement, a cement from **Dyckerhoff**. Part of this year's work was to use another cement, from another company, to further validate the elastic model.

For this process, **Holcim** supplied the cement, a Type I portland cement, to **Dyckerhoff** and also provided particle size distribution analysis. **Dyckerhoff** made samples from the cement and measured elastic properties, non-evaporable water contents, and an analysis of the forms of gypsum present. **Dyckerhoff** sent samples of the cement to **NIST**, which did SEM and X-ray microprobe analysis to provide the basic information needed for the VCCTL models. This detailed chemical/particle analysis was then used to translate the non-evaporable water contents into degrees of hydration. Version 3.0 was used to hydrate the cement, and the elastic code in version 2.0 was used to compute elastic properties for models that matched the w/c ratios and degree of hydration used by **Dyckerhoff** in the experimental samples.

The agreement between experiment and model prediction is excellent; differences between measured and predicted values are on the order of only 5 %, as can be seen in Figures 10 (14 day old samples) and 11 (56 day old samples) below. The model predictions at w/c = 0.55 and 0.60 have been slightly modified to allow for the bleeding that was seen experimentally. Essentially, a small decrease in the effective w/c ratio was made to allow for this bleeding. In the **NIST** X-ray microprobe analysis, about 8 % of the cement was determined to be non-hydratable material, so this material in the model was treated as inert filler with the approximate elastic properties of gypsum (dihydrate).

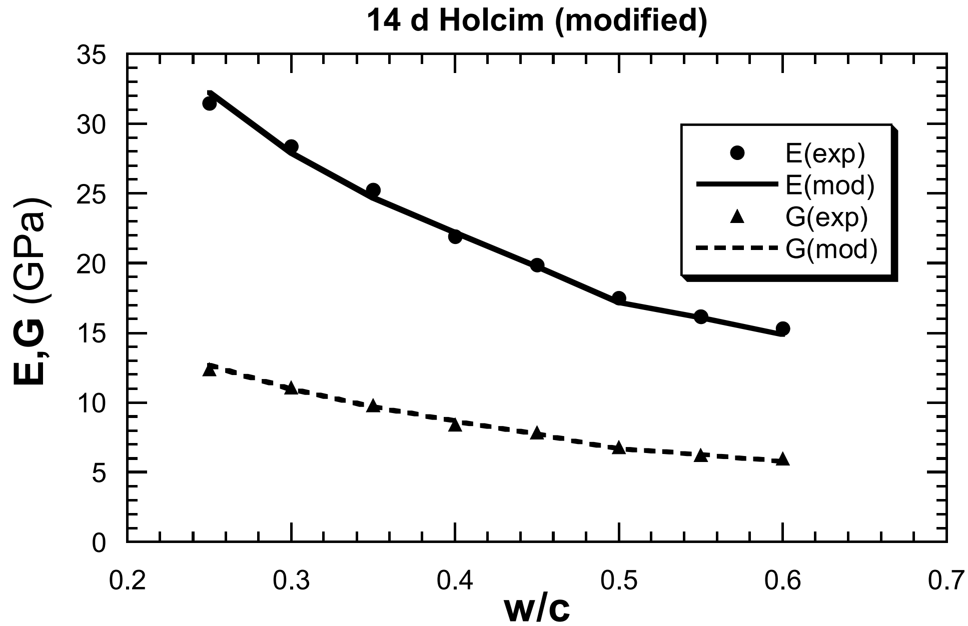

Figure 10: Comparison of calculated and measured elastic moduli for Holcim cement at 14 days.

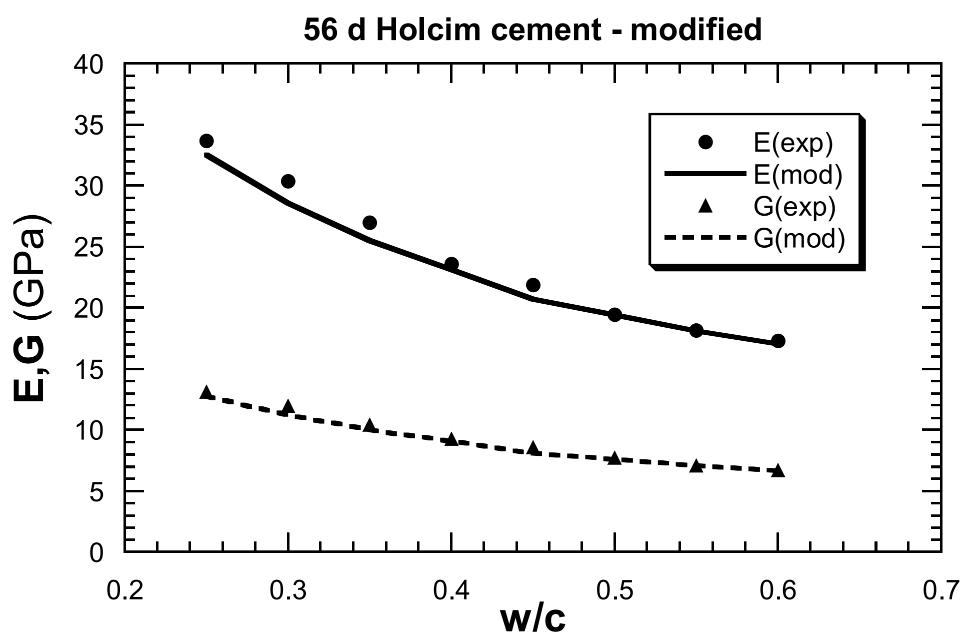

Figure 11: Comparison of calculated and measured elastic moduli for Holcim cement at 56 days.

Case Study II

Quantitative Test of X-Ray Computed Tomography and Spherical Harmonic Shape Analysis

Taking a 3-D X-ray computed tomography (CT) picture of a concrete cylinder, and then extracting and analyzing the gravel shapes with spherical harmonic techniques, will always produce a particle image of some sort. Deciding whether or not it is correct is a harder question. There are some obvious indirect checks, of course. If two particles were artificially connected together in the CT image, a very odd spherical harmonic shape will be produced. Visually checking can eliminate such gross errors, using the VRML (Virtual Reality Modeling Language) images in the aggregate database (VCCTL v.3.0). However, the best check of accuracy is a direct, "head-to-head" check – real rock vs. spherical harmonic image. That is impossible to do, however, when the source of particle images is a concrete cylinder. The particles cannot be seen inside the opaque cylinder, and are not easily available by breaking open the cylinder, as how does one find a given particle, especially when one is not sure of the shape one is looking for, since that is the very quantity to be checked!

To overcome this problem, a single rock, approximately 80 mm × 60 mm × 40 mm, was packed in a cylindrical container using cement powder and placed in the X-ray tomography unit at the Federal Highway Administration Turner-Fairbanks lab[*]. The same rock, after imaging was done, was removed, cleaned, and measured at NIST. Its linear dimensions were carefully measured and its volume was measured via water displacement, to be about $(5.0 \pm 0.5) \cdot 10^4$ mm^3. The spherical harmonic mathematical analysis was performed. Once the spherical harmonic coefficients are known, almost any shape quantity of the rock can be computed – volume, surface area, average curvature, etc. The volume of the rock was computed to be $5.4 \cdot 10^4$ mm^3, well within experimental error. Figure 12 on the next page shows four different views of the rock, comparing a real digital camera image to the equivalent view of the VRML image. VRML images are completely rotatable, so that an equivalent view could be found to the actual views of the real rock. The visual comparison is excellent, and along with the good quantitative agreement between the measured and computed volumes, implies that this method of image acquisition and analysis is accurate. An even more rigorous comparison, utilizing additional shapes, will be published in 2003 (S. Erdogan, E.J. Garboczi, H.S. Saleh, D. Fowler, and R. Livingston, unpublished).

[*]The actual imaging and image processing was done by Dr. Habeeb Saleh, under the leadership of Dr. Richard Livingston, at FHWA, and Mr. Sinan Erdogan, from ICAR at the University of Texas, who is a graduate student under the supervision of Prof. David Fowler.

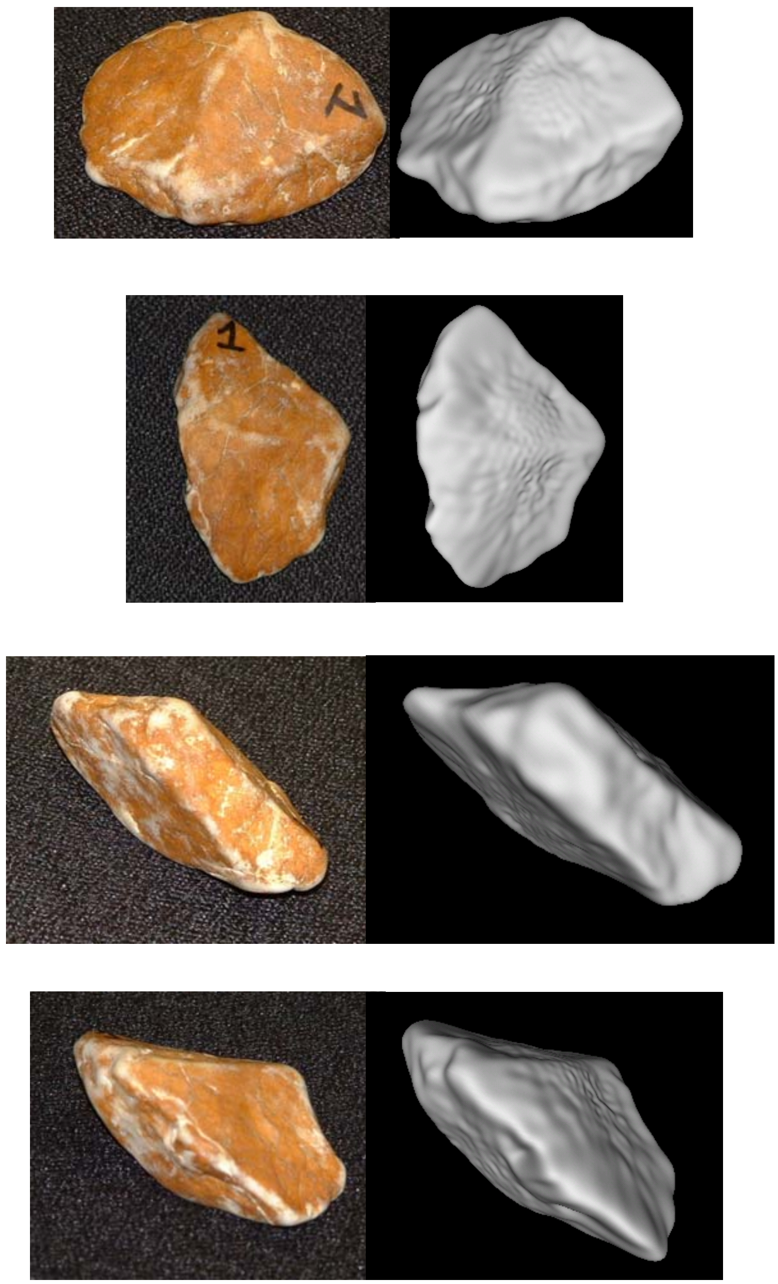

Figure 12: Four comparison views of a digital camera image of the real rock vs. the equivalent view of the VRML image of the rock produced from the spherical harmonic expansion.

Case Study III

Effect of Coarse Aggregate Shape on Concrete Plastic Viscosity

This case study shows three applications where the DPD simulation was used to directly compute the effect of real coarse aggregate shape on the plastic viscosity of a model concrete. This modeling study shows the potential for the virtual testing concept in concrete rheology.

Two model specimens were prepared, both with four different size particles, ranging over approximately a factor of two in volume. One concrete model used crushed aggregate shapes, based on X-ray computed tomography (see **Aggregate Characterization** section for details) of a concrete cylinder. The other model used spherical particles having identical volumes to the four real-shaped particles. A 3-D view of both models is shown in Figure 13.

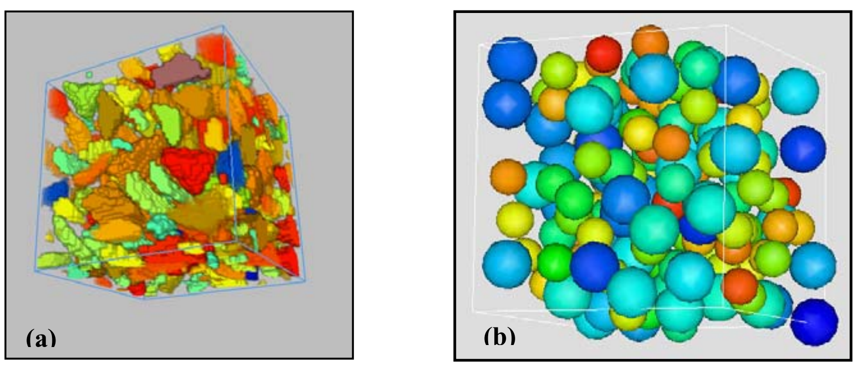

Figure 13: 3-D views of the two model concretes used in this case study: (a) four different real crushed aggregate shapes, and (b) equivalent spherical particles. The aggregate volume fraction in both cases is 45 %.

Rheological differences were found between the two models. For example, at a volume fraction of 45 %, the same as that shown in Figure 13, the relative viscosity (viscosity of the concrete compared to the viscosity of the mortar matrix) of the spherical particles was about 8. In contrast, the relative viscosity of the crushed aggregates was about 15, almost twice as high. This simple example shows the large effect that real particle shape can have.

By varying the number of aggregates in the simulation, the solid volume fraction can be controlled. Figure 14 illustrates a further comparison of the two concrete models, showing the difference in relative viscosity over several different coarse aggregate volume fractions. Clearly, the effect of the different shapes only becomes apparent for volume fractions greater than about 30 %, which is well below the practical range, anyway. At practical volume fractions, above 35 %, the particle shape plays an important role as the relative viscosity increases, for the crushed aggregates, at a much greater rate in comparison with the spherical aggregate concrete model.

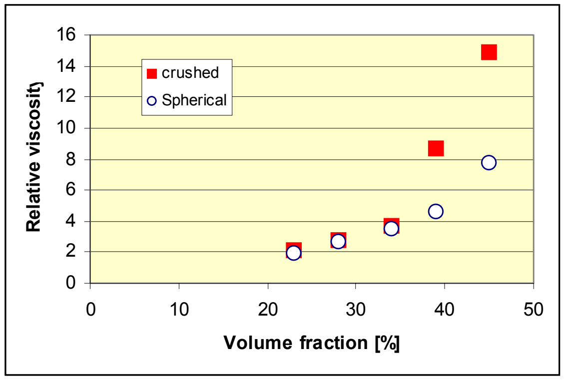

Figure 14: Plot of relative viscosity for the two concrete models as a function of the volume fraction of coarse aggregate.

More than just computing viscosity under simple shear flow, the DPD simulation can also be used to study and bring physical understanding to complex geometries, like those considered as candidates for standard tests for self-consolidating concrete. Figure 15 shows the same two concrete models in a simple simulation of this kind of test, at a coarse aggregate volume fraction of 45 %. In this test, which is generically similar to real tests used in the industry (unstandardized at present), concrete flows under a gravity-induced stress downward through a cage of reinforcing steel bars. There are four such rebars in the figure, oriented perpendicularly into the page. DPD simulation found that the flow rate in (a) was about one half that found in (b), a result consistent with the difference in the relative viscosity found for the two models. This last example illustrates the potential of the VCCTL DPD simulation as a design tool for self-consolidating and other kinds of concrete, and its ability to directly simulate test apparatus, giving basic physical understanding of empirical test results that are too complex for analytical interpretation.

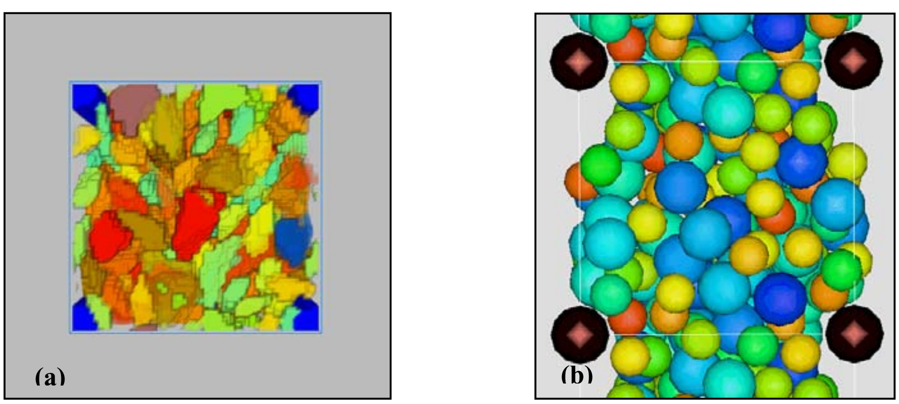

Figure 15: The two different concrete models being analyzed in a simple "self-consolidating concrete" type test, where the concrete flows under gravity through a cage of reinforcing bars. Rebars extend orthogonally into the page, and are shown at the corners in blue in (a) and in dark red in (b).

NIST/Industry VCCTL Funding

*The following **approximate** financial figures are based on the 2002 calendar year, consisting of nine months of fiscal year (FY) 02 (Jan-Sep) and three months of FY 03(Oct-Dec).*

Internal funding of Inorganic Materials Group by NIST	**$1.25M**
External funding generated by group[1]	**$0.50M**
Total operating cost of the Inorganic Materials Group at NIST (includes salaries, equipment, travel, etc.)	**$1.75M**
VCCTL Finances[2]	
External funding of VCCTL (from consortium members outside NIST)[3]	**$0.36M** (6 × $40K + 2 ×$60K)
Internal NIST funding of VCCTL research activities	$0.875M (BFRL) + $0.1M (MSEL) + $0.48M (ITL) = **$1.455M**
NIST equipment funding	BFRL – 128 processor computer - $250K BFRL – PSD analyzer - $90K **Total = $0.34M**
Total NIST funding of VCCTL project (labor + equipment)	**$1.795M**
Percentage shares of total VCCTL project funding	**NIST – 83 %** **Industry – 17 %** **Total cost/industrial share ≈ 5.9:1**

Note: Almost *all* research sponsored by externally funded sources *other than* VCCTL have the potential to be included in new versions of the VCCTL software. For example, the new sulfate attack simulation work being built into Version 4.0 is based on three years of funding by PCA on research into sulfate attack mechanisms (total of $225K). The Nuclear Regulatory Commission has provided long-term support of the development of 4SIGHT (see Durability Research section of this report for details).

[1] The Inorganic Materials Group always seeks outside research funding to complement internal projects, building on expertise to solve problems of interest to industry and other U.S. government agencies.

[2] The total amount of VCCTL support in our group, both internal and external, supports the majority of time of the main researchers, E.J. Garboczi, N.S. Martys, J.W. Bullard, and C.F. Ferraris. VCCTL funds are also used for some secretarial support and other research that is needed for the VCCTL project, like some of P.A. Stutzman's time characterizing cements for the VCCTL cement database.

[3] Of this amount, $60K was directed to ITL for support of visualization and parallelization services, and $60K was directed to MSEL for services in particle size distribution analysis and for research on the effect of chemical admixtures on cement/water suspensions.

NIST Equipment and Facilities

Materials and Construction Research Division

Microscopy

- **Scanning electron microscope** equipped with solid-state backscattered electron detector. Digital image capture, processing and analysis capabilities. X-ray microanalysis capabilities for qualitative and quantitative spot analyses as well as imaging element spatial distribution.

- **Automated X-ray powder diffractometer** with nine-position sample changer, adjustable optics, diffracted beam monochromator

- **Light Microscopy Facilities:**

    - Stereo microscope utilizing apochromatic objectives and single optic axis
    - Polarized light petrographic microscope
    - Reflected/transmitted light microscope

- **Atomic force microscope** utilizing an environmental chamber

- **Confocal microscope**

- **Specimen preparation facilities** for SEM, optical microscopy, XRD and XRF.

- **Image Processing**: including digital and optical camera. Semi-automated image analysis is available

Mechanical Properties

- Compressive testing machines
- Nano-indenter
- Stress measurements of a confined specimen for ASR or sulfate attack studies

Transport Properties

- Gas permeability equipment

- X-ray absorption unit for monitoring water movement in materials Gas permeability equipment
- Electrical conductivity measurement equipment
- Ionic diffusivity measurement equipment

Systems Characterization

- Malvern particle size analyzer, wet and dry capability (**new in 2002**)
- Particle classifier (**new in 2003**)
- Atomic absorption
- Infrared spectrometer
- Ion chromatograph
- Thermogravimetric analysis
- Differential scanning calorimeter (DSC)
- Differential thermal analysis (DTA)
- Automated high-temperature furnace to 1600 C
- Dunouy Tensiometer for measurement of the surface tension of solutions.
- Heiden Sorption Analyzer for measurement of absorption/desorption isotherms.

Rheology

- Parallel plate fluid rheometer: stress or strain controlled
- Controlled temperature and speed mixer: This mixer is designed according to the specifications developed by PCA/CTL and can be used for paste and/or mortar
- Standard tools for flow measurement in cement paste or mortar, like Marsh cone, flow cone, mini slump
- Slump and modified slump test.

Sample Preparation Laboratory

- Attritor mill (**new in 2002**)
- Fully equipped laboratory to prepare and cure mortar or cement paste specimens including Hobart mixers, flow table, vibrating table
- Environmental chambers:
  - three cabinets (temperature and RH controlled) (one **new in 2002**)
  - one walk-in (temperature controlled)
  - curing cabinet (temperature controlled and RH higher than 98 %) (**new in 2002**)
- Water baths: 4 table top and one floor unit
- pH controlled units
- Length measurement devices

Computational Facilities (Building Materials Division)

- Windows and LINUX-based PCs
- Linux-based cluster with 32 processors
- 128-processor cluster (in procurement process, expected April 2003)

Dispersion & Fine Particle Labs, Ceramics Division (MSEL)

- Matec Applied Science ESA-9800 (electroacoustic analyzer particle mobility)
- Colloidal Dynamics Acoustosizer (electroacoustic spectrometer for particle size and zeta potential)
- Dispersion Technology DT-1200 (acoustic spectrometer with CVI for particle size and zeta potential)
- Malvern Zetasizer 3000HS (microelectrophoresis and QELS particle sizer)
- Rheometrics 2000 (controlled stress dynamic rheometer)
- Brookfield DV-II+ Viscometer
- Coulter LS230 (laser diffraction particle sizer)
- Quantachrome AUTOSORB-1 (multi-point surface analysis system for BET surface area and porosity)
- Quantachrome AUTOSCAN 60 (mercury porosimeter)
- Rosemount Dohrmann DC-80 Total Organic Carbon Analyzer
- AMRAY 1830 Scanning Electron Microscope
- Beckman J2-series High Speed Centrifuge
- Brookhaven X-RAY Centrifuge Particle Size Analyzer

Information Technology Laboratory Central Hardware Facilities (ITL)

Computational Resources

- **Four SGI Origin 2000 machines**:
  - 8 196 Mhz R10000 CPUs, 8 GB of memory, 120 GB of disk space
  - 32 250 Mhz R10000 CPUs, 32 GB of memory, 96 GB of disk space
  - 32 300 Mhz R12000 CPUs, 32 GB of memory, 193 GB of disk space
  - 32 300 Mhz R12000 CPUs, 32 GB of memory, 193 GB of disk space

- **One SGI Cluster**
  - five dual processor R10000 CPUs, with 4 GB of memory

- **Three Linux Clusters**
  - sixteen 400MHz Pentium IIs connected by a Fast Ethernet network.
  - Each CPU has 256MB of RAM and 6GB of local disk storage.
  - forty-eight 500Mhz Pentium IIIs connected by a Fast Ethernet network.
  - Each CPU has at least 256MB of RAM and 6GB of local disk storage.
  - 128 Pentium IIIs connect by a Fast Ethernet network
  - Each CPU has 1GB of RAM and 6GB of local disk storage

Visualization Resources

- One SGI Onyx3000
  - Four Infinite Reality4 Graphics
  - Sixteen R14000 CPUs (500 MHz)
  - 16 GB memory

- One RAVE two-wall immersive environment with Crystal Eyes software/hardware with head tracking

For More Information

1. Examine Version 1.0 of the VCCTL software package, available at:

   http://vcctl.cbt.nist.gov .

2. Visit the VCCTL Consortium information web site at:

   http://www.bfrl.nist.gov/862/vcctl

3. Contact the VCCTL Consortium Manager at:

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