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Virtual Cement and Concrete Testing Laboratory for Quality Testing and Sustainability of Concrete

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Synopsis: The NIST-Industry Virtual Cement and Concrete Testing Laboratory (VCCTL) Consortium has developed an integrated software package for performing simulations of a number of engineering test measurements, including isothermal calorimetry, adiabatic temperature change, chemical shrinkage, elastic moduli, and compressive strength. In the last two years, the software interface has been redesigned to be easier to navigate, with online tutorials and documentation for easy reference. As a result, VCCTL is now ready to be integrated in industrial settings as a supplemental tool to accelerate research on mix designs and to streamline routine quality testing procedures. This paper will demonstrate the software interface, and two applications will be described to illustrate the utility of the software to help solve practical problems. In the first application, we address sustainability issues by investigating the replacement of coarse clinker particles with limestone and its effect on elastic moduli and compressive strength. In the second application, we illustrate VCCTL's potential for screening the quality of incoming cement clinkers by providing rapid estimates of compressive strength development in mortar specimens.

Keywords: building technology; hydration; microstructure; strength; sustainability; virtual testing.

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INTRODUCTION

Design, optimization, and quality testing of concrete typically are accomplished by intensive physical testing procedures that consume large amounts of labor and materials. This task is becoming increasingly difficult as requirements for sustainability and performance begin to necessitate the use of blended cements and chemical admixture cocktails that may have significant interactions affecting durability and service life. As a result of this complexity, the performance of concrete over its intended service life is difficult to predict without extensive physical testing, since simple empirical relations cannot be universally applied with confidence. A promising alternative is to use scientifically based computer models to guide testing and development efforts. Properly validated computer models can supplant much of the current empirical design and testing procedures with computer simulations that are relatively quick and inexpensive. This paper describes an integrated package of computer-modeling software, called the Virtual Cement and Concrete Testing Laboratory (VCCTL), the goal of which is to enable a rapid exploration of concrete design space at significantly reduced cost. This software has been developed for the last eight years through a consortium led by the National Institute of Standards and Technology (NIST) and with a membership that has included cement manufacturers, chemical admixture suppliers, aggregate suppliers, and industry associations. The computer models in VCCTL continue to be refined to include a wider range of materials and curing conditions. Nevertheless, the models—and the user interface that ties them together—have advanced to a point where they can provide immediate benefits to industry in a number of ways. Features of the VCCTL software will be described in this paper, and then two case studies will be presented to demonstrate how the software can assist in mixture design and quality testing.

RESEARCH SIGNIFICANCE

Scientifically based computer models of concrete materials have existed for several decades but, so far, they have not been widely adopted in industry. To gain better acceptance in concrete research and testing environments, computer models must (1) be supported by adequate characterization of the materials that are being used, (2) provide the materials engineer with reliable guidance about the likely effects of different material characteristics and realistic curing conditions on concrete performance, and (3) be accessible through an intuitive, easy-to-use software interface. VCCTL software has been designed and developed with these objectives in mind. By adopting such modeling software, the concrete industry can streamline mixture design and testing activities.

DESCRIPTION OF VCCTL MODELING SOFTWARE

Materials Characterization

To be as accurate as possible, VCCTL models have been built upon principles of chemistry, physics, and materials science. As a result, these models require accurate input about the materials that are being simulated, including the cement powder as well as the physical properties of the fine and coarse aggregates. Therefore, we begin this section with a cursory description of the materials characterization that is required for VCCTL use.

The VCCTL module for cement hydration is based on the CEMHYD3D model developed at NIST[1]. This module simulates microstructure development at the micrometer scale and, as such, requires knowledge of the cement particle size distribution (PSD) and spatial distribution of solid phases at this length scale. The PSD of cement powder, which typically ranges from less than 1 μm to more than 50 μm, can be characterized by a number of techniques, although no universally recognized standard test method currently exists.[2,3] By far the most common method employed in industry is laser diffraction.[2,3] Ideally, for blended cements, the PSD of each component (cement, limestone, fly ash, etc.) should be measured separately, although if these components are interground then the overall PSD of the final composite powder may be the only feasible measurement. Regarding the spatial distribution of solid phases in cements, the most accurate procedure is a combination of backscattered scanning electron microscopy (SEM), X-ray microprobe analysis, and quantitative X-ray powder diffraction (QXRD) using Rietveld refinement.[4] QXRD provides an accurate measurement of the mass fraction of most of the solid phases, while SEM gives an indication of the spatial distribution of those phases in the microstructure, which can be quantified for isotropic powders by using two-dimensional (2-D) autocorrelation functions. From this information, a three-dimensional (3-D) digital-image representation of the microstructure can be generated and used as input to the VCCTL microstructure model[5] (see Fig. 1). In recent years, the SEM/QXRD procedure has been simplified considerably by using modern image processing software. Even so, proper specimen preparation for SEM imaging can be time consuming and requires considerable experience. We have gathered evidence that QXRD alone, which is a widely available and simple technique, combined with reasonable assumptions about the surface area fraction of each component phase, is typically sufficient to make accurate predictions with VCCTL software.

For the fine and coarse aggregates, the density, size distribution, and shape of the individual aggregate particles must be measured. The density and size distribution are easily measured using standard methods. The particle shapes can be accurately quantified using X-ray computed microtomography (μCT) and the shape data can be stored efficiently for thousands of particles as spherical harmonic coefficients.[6] μCT also can be used to characterize the shapes of cement particles.[7]

Computer Models and User Interface

The VCCTL software is a web server application, which means that it can be installed on a web server and then accessed through a web browser on any client computer with internet access to the server. The user interface has been designed to represent different parts of a cement and concrete testing laboratory, with modules for storing and accessing materials, mixing concrete, curing concrete, and performing tests of mechanical and transport properties. Figure 2 shows the basic work flow for virtual testing with VCCTL. Figure 3 shows a portion of the Lab Materials module, from which the user can browse the characteristics of available cements and supplementary cementitious materials (SCMs). Existing materials can be edited in place to change their properties, and newly characterized materials can be easily uploaded to the database. These features make it easy to customize the VCCTL to a particular organization's material inventory.

The Mix Preparation module, a portion of which is shown in Figure 4, enables the user to select the cement, aggregate sources, and any SCMs that are to be included. The proportions of these materials, water, and entrained air volume can be entered in text fields. The entered data are sent to a program that generates a 3-D digital image of the virtual cement paste microstructure and of the larger-scale virtual mortar or concrete aggregate packing.

The user can perform virtual curing on the resulting virtual cement paste microstructure, by entering the curing conditions in the Curing module (also shown in Figure 4). The thermal conditions (isothermal, adiabatic, or semi-adiabatic) and initial temperatures of the paste and aggregate can be specified, together with the moisture conditions provided for curing (saturated or sealed). The time scale of the curing simulation can be calibrated either to an empirical parameter or, more realistically, to isothermal calorimetry or chemical shrinkage data that can be conveniently uploaded in the Lab Materials module. The input data are sent to a program that simulates the 3-D microstructure development and tracks the value of several properties such as heat release, temperature, chemical shrinkage, and Powers' gel-space ratio.

Once curing is completed, an online graphing package in the Measurements module enables the user to plot the hydration properties (temperature, chemical shrinkage, degree of hydration, etc.) as a function of time or any other independent variable. The Measurements module can also be used to calculate several mechanical properties of the cement paste, mortar, or concrete, such as the linear elastic moduli and the compressive strength. A finite element program[8] is used to calculate the effective linear elastic moduli of the hydrated cement paste, and these computed data are then used in a second C program, based on differential effective medium theory, to compute the elastic properties and to estimate the compressive strength of the mortar or concrete.[9]

VCCTL APPLICATIONS

As the VCCTL software has matured, members of the VCCTL consortium have used the models in short-term quality testing and troubleshooting efforts, and have reported significant savings in materials and labor costs for each project. In each case, the VCCTL models were used to identify *likely* (not guaranteed) results or troubleshooting solutions, which were then validated using a small set of physical tests. In this section, we describe two applications of VCCTL software that illustrate the types of practical problems that the models can address.

Application 1: Limestone additions for increased sustainability

In concretes made with cement binders having a low water-cement mass ratio (*w*/*c*), the coarsest cement particles often remain largely unhydrated for the life of the material because of insufficient water in the mix and/or insufficient space in which the hydration products can form. Permanently unhydrated clinker particles are generally undesirable (except in cases where autogenous healing of cracks is an issue) because their contribution to the strength of the concrete is the same as a less expensive inert filler material, such as limestone. In addition, a filler such as limestone can be used with zero CO_2 emission, while the manufacture of cement clinker releases about 1 metric ton of CO_2 for every metric ton of clinker produced. If one were to replace the coarsest fraction of a cement powder with coarse limestone particles, then nearly the same mechanical properties should be obtainable with a less expensive, more sustainable binder. In fact, the concept of limestone replacement has been demonstrated previously with both experiments[10] and computer modeling studies with CEMHYD3D.[11]

Figure 5 shows 2-D slices through 3-D virtual cement pastes made with and without coarse limestone replacement. In both cement pastes, the cement used was Proficiency Sample Cement 152 issued by the Cement and Concrete Reference Laboratory (CCRL). Both pastes were generated with *w*/*c* = 0.35. In the microstructure with limestone replacement, all cement particles with equivalent spherical diameter exceeding 40 μm were replaced with limestone particles (green in the images) of the same equivalent spherical diameter. This amounts to a replacement of 12.5% by mass on a total solids basis. Also shown in Figure 5 are the simulated microstructures after isothermal hydration at 20°C (68°F) under saturated conditions for 28 d. These microstructures demonstrate that the coarse limestone particles, which are relatively inert during hydration, take the place of the unhydrated cores of large clinker particles, since there are fewer unhydrated clinker particle cores in the microstructure with limestone replacement after 28 days. The mechanical properties are also predicted to not be adversely affected by the limestone replacement. In Figure 6, the predicted Young's modulus and compressive strength are plotted at 2, 7, and 28 days. At all three ages, both the Young's modulus and the compressive strength are predicted to be modestly greater for the material with 12.5% replacement with coarse limestone. These results are qualitatively consistent with previous numerical and experimental studies on the effects of coarse limestone replacement,[10,11] although those earlier studies predicted a slight decrease in strength, instead of a slight increase, upon limestone replacement. The strength increase predicted in the current simulations are largely due to a decrease in capillary porosity that must accompany mass-based replacement of a higher-density material (cement powder) with a lower-density material (limestone). In any case, the VCCTL predicts, and experiments confirm, that for low *w*/*c* ratios, partial replacement of coarse clinker particles with coarse limestone particles can yield a less expensive and more sustainable concrete binder without significantly diminishing the mechanical properties. Further work using the VCCTL and supporting experiments could seek to find optimum replacement levels as a function of *w*/*c* ratio and of the particle size distribution of the original cement powder.

Application 2: Screening clinker quality based on 28-day compressive strength

The concrete industry uses cement that is produced in numerous regions, such as North America, Western Europe, China, and India. The available raw materials for clinker production can be highly variable from region to region. Therefore, it would be desirable to find a rapid test method for screening the quality of incoming clinkers, based on some convenient metric, so that potential problems can be rapidly identified without using excessive amounts of labor and materials. As a first step toward this goal, we report on a simple test procedure that can be used in conjunction with VCCTL simulations to directly compare different clinkers according to the 28-day compressive strength.

The following simple screening procedure is proposed here. As already described, each clinker powder is characterized using either SEM/X-ray image processing or QXRD to obtain the mass fractions of each mineral in the clinker. If SEM/X-ray imaging is used, then the surface area fractions and autocorrelation functions can be obtained directly from a sampling of segmented images. If QXRD alone is used, then reasonable assumptions, based on research on many different cements, can be made about the proportionality of the surface fraction to the volume fraction of each clinker mineral. The PSD of the clinker also must be measured. Once characterized, each clinker is blended with 5% replacement of gypsum from a fixed consistent source with a well-defined PSD. A small volume of paste is produced for obtaining isothermal calorimetry data up to 2 days of hydration. With these calorimetry data on pastes for calibration, the VCCTL models can simulate the microstructure development and mechanical properties of virtual mortars of the same materials mixed with a standard sand (ASTM C778-02 or EN 196-1, for example) out to 28 d or later.

In this case study, this test procedure was used to predict the mechanical properties of mortars made from a coarse clinker originating in China. Isothermal calorimetry on a cement paste formed from this clinker (w/c = 0.4) was performed at 20°C (68°F) for 48 h. A representative phase-segmented SEM image of the clinker is shown in Figure 7, and the mass fractions of clinker minerals are given in Table 1. Although not shown here due to space limitations, the PSDs of both the clinker and the gypsum were measured. The median particle diameters of the clinker and the gypsum are 18.7 μm and 20.7 μm, respectively. The European EN 196-1 sand was used to make virtual mortars with a mass ratio of sand:cement:water equal to 3:1:0.5. The specimens were assumed to include 4% entrained air by volume. Hydration of the mortar specimens was simulated at 20°C (68°F) under saturated conditions for 28 d. For comparison, actual mortar specimens were prepared using the EN 196-1:2005 test method,[12] which uses the same mixture proportions and curing conditions as employed in the VCCTL simulations. Figure 8 compares the predicted and measured compressive strengths at 2, 7, 14, and 28 days. The specimen-to-specimen variability in the experimental measurements is about ±1 MPa (145 psi), and the uncertainty in the VCCTL prediction, due to assumptions about the entrained air volume fraction and statistical sampling from the particle size distribution, is about the same. Therefore, the differences between measured and predicted strengths at intermediate ages are probably significant. Similar differences have been observed for several other cements over the last several years,although application of the VCCTL to most Type I Portland cements has yielded better agreement to experiment than that shown in Fig 8. Further research needs to be conducted to determine the source of these differences and to determine whether the VCCTL strength predictions for other clinkers show a comparable agreement with experimental measurements.

SUMMARY AND PROSPECTUS

The VCCTL modeling package has been developed and refined over the last 10 years. It now enables users to quickly simulate the hydration and to calculate a number of important engineering properties of cement paste, mortar, and concrete. Improvements in the graphical user interface now make it possible for knowledgeable materials engineers to obtain useful data with only a modest amount of training. In this paper, we have demonstrated two potential applications—exploring new and more sustainable concrete mixture designs, and quality testing of clinker sources—that could begin producing immediate dividends in several sectors of the concrete industry. Other features of the software not described here, such as the ability to specify initial aggregate temperature and monitor heat evolution in hydrating cements, have immediate application for predicting and controlling the maximum temperature excursions at a job site. Research for improving the computer models that comprise the VCCTL is ongoing and should enable even more practical utility. Short-term improvements planned for the software include the addition of component costs to the mixture proportioning modules and more flexibility in mixing different clinker sources to customize a mixture. Longer-term research is already underway to improve the predictions of mechanical and transport properties, and to integrate new models of early-age hydration and the rheological behavior of fresh cement paste, mortar and concrete.

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Table 1—Mass fraction of clinker phases determined by QXRD using Rietveld refinement

Phase	Mass Fraction
C_3S	0.543
C_2S	0.259
C_3A (Cubic)	0.067
C_3A (Orthorhombic)	0.011
C_4AF	0.106
$K\underline{S}$	0.011

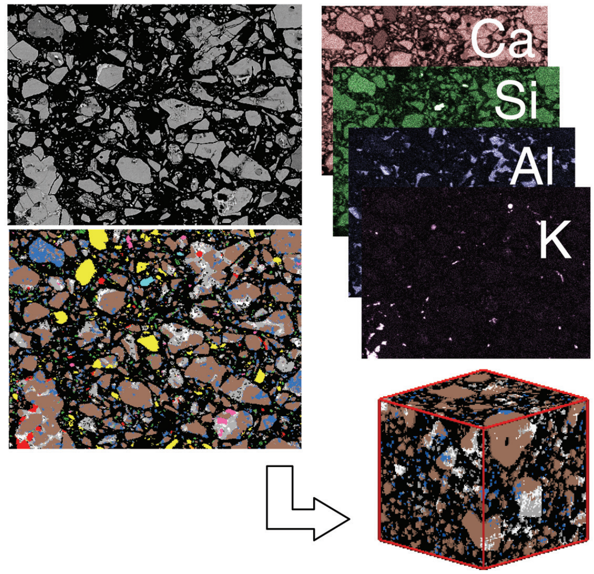

Fig. 1—SEM/X-ray microprobe characterization of 2-D cement microstructure images. A backscattered electron image (gray scale upper left) is filtered through element maps (upper right) to produce a phase segmented 2-D image (lower left). Autocorrelation functions measured on this image can be used to generate the final microstructure as a 3-D digital image (lower right). In the color images, alite is brown, belite is blue, tricalcium aluminate is gray, ferrite is white, potassium sulfate is red, periclase is pink, and calcium sulfate is yellow.

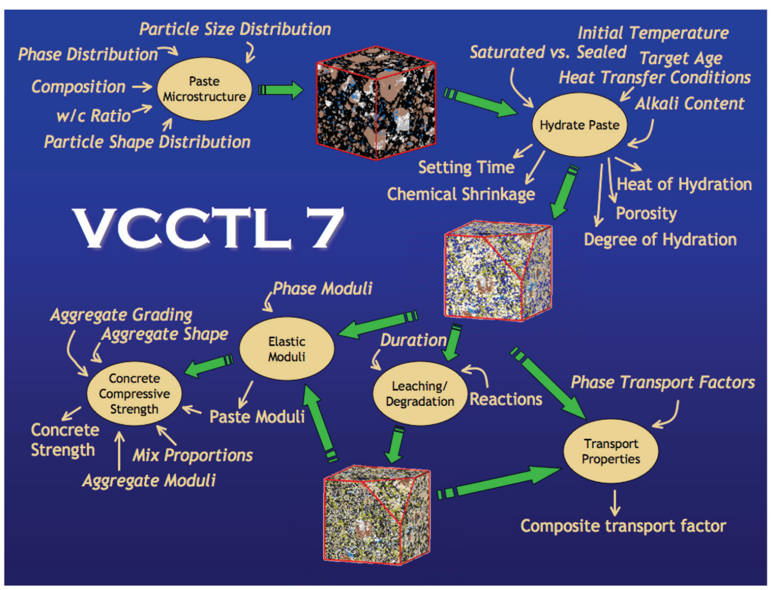

Fig. 2—Pictorial representation of the VCCTL software work flow, showing the various steps of virtual testing and the inputs (in italics) and outputs.

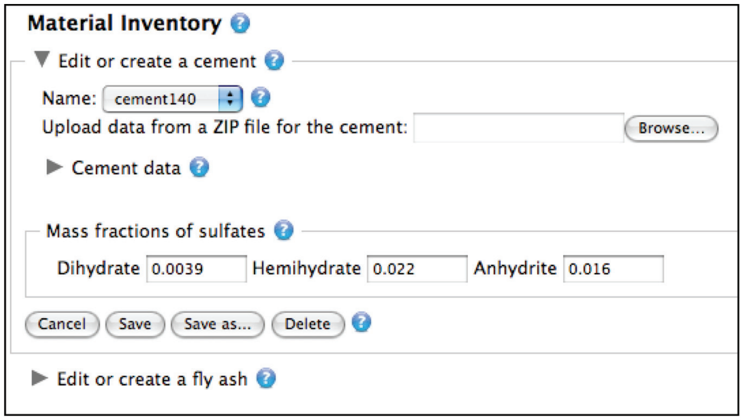

Fig. 3—Laboratory materials module in the VCCTL software interface can be used to view or edit the properties of cements or SCMs in the database, or to upload newly characterized materials.

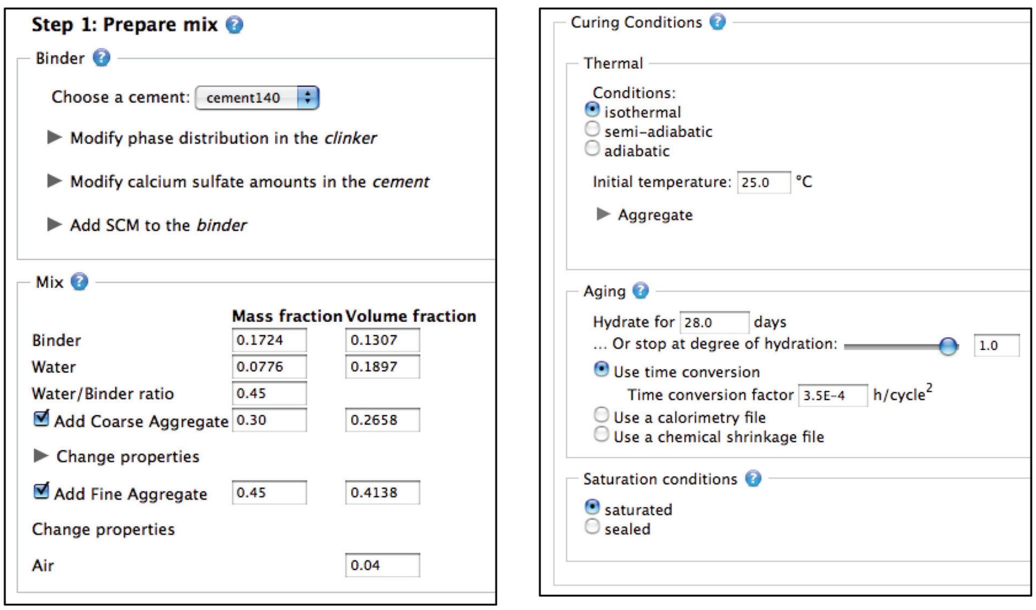

Fig. 4—A portion of the VCCTL Mix Preparation module (left) and Curing module (right). The Mix Preparation module enables the user to select cement, SCMs, and aggregate sources from the database, choose the gradings and proportions of these components with water, and entrained air volume. The Curing module enables the user to choose the thermal and moisture conditions of the fresh concrete, in addition to the duration and more detailed parameters, such as the hydration activation energy, heat transfer coefficients, and aggregate temperature.

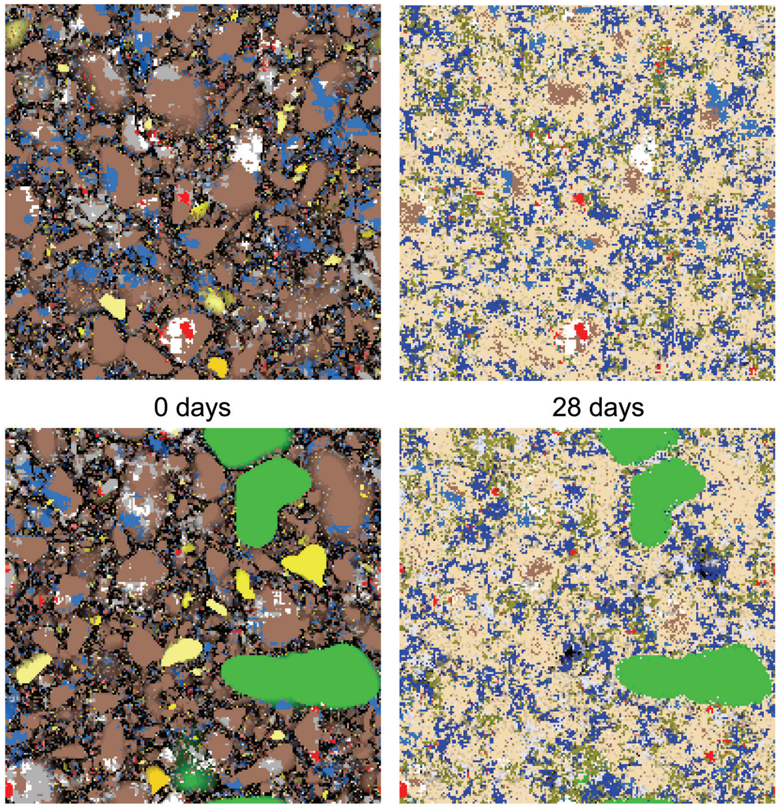

Fig. 5—Virtual mortar microstructures of CCRL Cement 152 at 0 days and 28 days of hydration without limestone (top) and with 12.5 % limestone replacement by mass. The scale is 200 µm on a side. In both microstructures, *w/c* = 0.35 initially, and curing was simulated at 20°C (68°F) under saturated conditions The phases are colored as follows: brown = C_3S, light blue = C_2S, gray = C_3A, white = C_4AF, yellow = $C\underline{S}$, red = $K\underline{S}$, green = limestone, light brown = C-S-H, dark blue = CH, and olive = ettringite. Here and throughout, conventional cement chemistry notation is used (C = CaO, S = SiO_2, A = Al_2O_3, F = Fe_2O_3, $\underline{S}$ = SO_3, K = K_2O, H = H_2O)

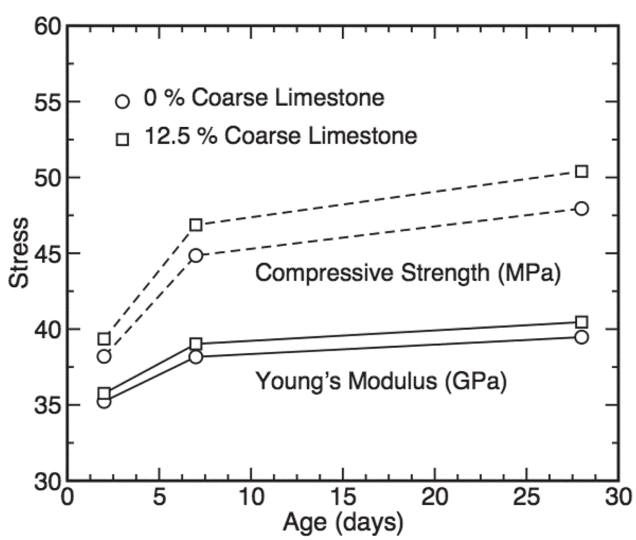

Fig. 6—Comparison of predicted Young's modulus (solid curves) and predicted compressive strength (dashed curves) for the two mortar microstructures shown in Figure 5. The variability in predictions due to both statistical sampling and uncertainty in the entrained air volume fraction (assumed ±1%) is about the size of the symbol.

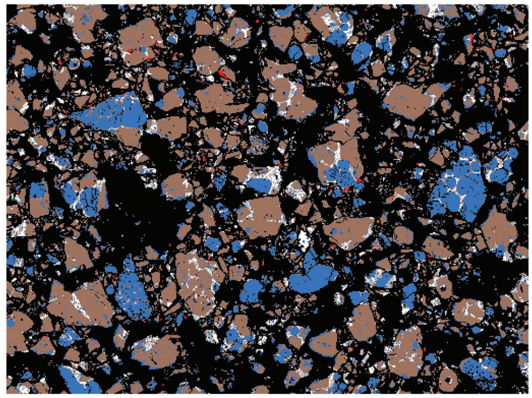

Fig. 7—Representative phase segmented SEM image of coarse clinker from China. The color scheme is the same as in Fig. 1 and 5. The image is 512 µm × 384 µm.

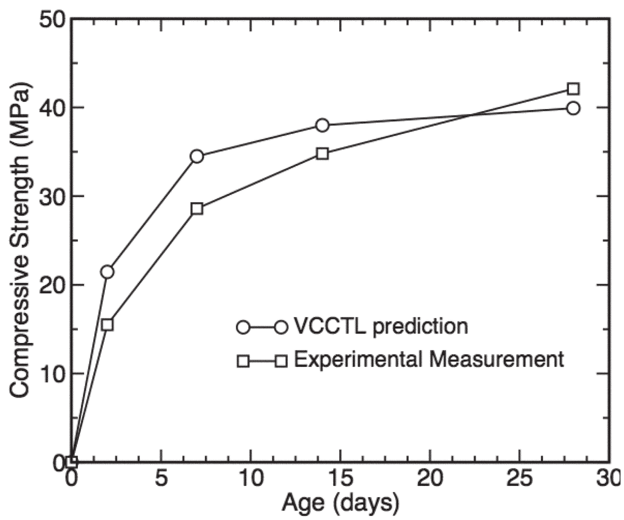

Fig. 8—Comparison of experimental and simulation values for the compressive strength of mortar specimens made according to the EN 196-1:2005 standard test method, using coarse clinker imported from China and mixed with 5% coarse gypsum by mass. The variability in predictions and experimental measurements due to both statistical sampling and uncertainty in the entrained air volume fraction (assumed ±1%) is about ±1 MPa (145 psi). (Note: 1 MPa = 145 psi.)