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DEPARTMENT OF CIVIL ENGINEERING

EFFECTS OF MICROSTRUCTURE ON DEFORMATION AND FRACTURE OF PORTLAND CEMENT PASTE

by

ROBERT BRADY WILLIAMSON and RAM PATI TEWARI

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SEPTEMBER 1971

STRUCTURAL ENGINEERING LABORATORY
UNIVERSITY OF CALIFORNIA
BERKELEY CALIFORNIA

Structures and Materials Research Department of Civil Engineering

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Structural Engineering Laboratory University of California Berkeley, California

September 1971

The constitution and hydration of portland cement is reviewed and the microstructure of hardened cement paste is introduced with scanning electron micrographs. The development of strength of monoclinic tricalcium silicate (i.e., alite) pastes both with and without the addition of gypsum is presented using scanning micrographs of fresh fracture surfaces. This experimental data is used to illustrate the general principles in the relationship between microstructure and mechanical properties for portland cement pastes.

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EFFECTS OF MICROSTRUCTURE ON DEFORMATION AND FRACTURE OF PORTLAND CEMENT PASTE

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INTRODUCTION

The solidification of portland cement paste by hydration is one of the most widely utilized reactions in all of Materials Engineering, and yet the effects of microstructure on mechanical properties is inexactly understood. In this paper the constitution and hydration of portland cement is reviewed and the microstructure of hardened cement paste is introduced with scanning electron micrographs. The development of strength of monoclinic tricalcium silicate (i.e., alite) pastes both with and without the addition of gypsum is presented using scanning micrographs of fresh fracture surfaces. This experimental data is used to illustrate the general principles in the relationship between microstructure and mechanical properties for portland cement pastes.

Constitution and Hydration of Portland Cement

Anhydrous portland cement contains four principle compounds, tricalcium silicate $(C_3S)^*$, β -dicalcium silicate $(\beta-C_2S)$, tricalcium aluminate (C_3A) , and a ferrite phase belonging to the $C_2F-C_6A_2F$ solid solution series which until recently was believed to have the fixed composition C_4AF . Small amounts of MgO, CaO and alkali sulphates also occur in many cements, and

^{*}The compositions of cement compounds are often represented as the sum of oxides, the formulae of which are abbreviated: C=CaO, S=SiO₂, A-Al₂O₃, F=Fe₂O₃, H=H₂O; thus for example, C₃S=3CaO·SiO₂=Ca₃SiO₅. This system is used in cement chemistry interchangeably with ordinary chemical notation.

the presence of foreign ions changes the crystal structure of the calcium silicates from those of the pure compounds. The alite form of tricalcium silicate used in the experiments described in this paper is the monoclinic form which is the most common form in commercial cements.

When water is added to the anhydrous cement powder, the calcium silicates form a rather insoluble amorphous hydrous calcium silicate and calcium hydroxide (CH) which is also known by its minerological name Portlandite. The approximate chemical equation for the hydration of C_3S can be written

$$2C_3S + 8H \rightarrow (C_3S_2H_{2.5}) * H_{2.5} + 3CH,$$
 (1)

where the hydrous calcium silicate is represented as containing 2.5 moles of chemically bound water and 2.5 moles of weakly absorbed water. A graphical representation shown in Figure 1 can be used to describe the sequence of events as the cement minerals react with water. This reaction produces the continuous solid matrix in the space that was originally occupied by discrete particles dispersed in water. In Figure 1 the relative volumes of water and cement are represented by a bar graph labeled "Fresh Paste" for the case of 0.5 water-to-cement ratio based on weight, (note that the specific gravity of cement is 3.15). The subsequent hydration reaction is presented on the basis that one unit volume of cement yields two unit volumes of

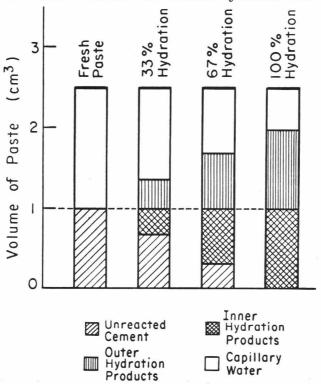


Figure 1. A graphical representation of the relative volumes of the hydration products of portland cement at three stages of hydration is shown for a water-to-cement ratio of 0.5.

hydration products; this is a widely accepted empirical relationship used by Powers. The nomenclature of inner and outer hydration products has been introduced into Figure 1 in order to discriminate between the products laid down within the boundaries of the original cement grains and the products laid down in the originally water-filled space. This nomenclature is due to Taplin, although others had come close to it previously.

Microscopic Observations

The most advanced microscopic techniques have always been applied to study the hydration of cement as they became available, and much of the current knowledge of the microstructure dates back to the classic work of the 19th Century such as that of LeChatelier. 4 Williamson has reviewed the microstructure of cement paste as observed in both optical and electron microscopes, and he suggests the distribution of hydration products shown schematically in Figure 2 in and around a single grain of cement. 5 This distribution of hydration products fits most microscopic observations, and it is presented here to acquaint the reader with the spatial arrangement of the hydration products shown in the bar graph of Figure 1. Williamson chose the term "columnar zone" because of its similarity to the columnar intent to restrict the possible origins of the morphology observed there. The column aspect of this zone is due to the growth perpendicular to the surface, and there is no single phase or chemical feature assumed for this zone.

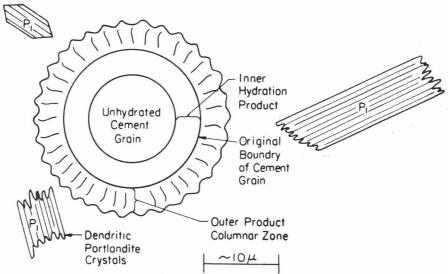


Figure 2. The hydration products formed inside and outside the cement grain are schematically represented as they appear through microscopic observation. The hydration process is continuing since the cement grain is represented as being suspended in water.

Mechanical Properties

The development of strength in hydrating cement pastes has been studied by many investigators since the original work of LeChatelier, but the first definitive study of the strengths of pastes made from the individual pure cement compounds was made by Bogue and Lerch. They determined the principal source of strength in the first thirty to sixty days was the C_3S , although the β - C_2S finally developed greater strength after two years. There has been a continuing study of the mechanical properties of cement pastes as hydration proceeds, but these have not shown any great differences from the Bogue and Lerch study nor has the role of microstructure been defined in more than a general fashion. In the experimental work reported below the deformation and fracture of alite paste samples is measured at ages from a few hours to ninty days using a hard testing machine, and the microstructure is shown with scanning electron micrographs.

EXPERIMENTAL TECHNIQUES

The alite used in this investigation was of high-purity monoclinic structure prepared in a pilot rotary kiln at the University of California, Berkeley. The details of the raw materials used and the process have been discussed by Mehta, Pirtz, and Polivka. The alite was ground to a fineness of 3800 cm²/gm Blain. Two mixes were prepared, one without gypsum and one with 5% analytical reagent grade gypsum. Pastes of 0.3 water-to-cement ratio were prepared and cast into 1 cm cubes for compression testing and into a special split mold for scanning electron microscopy. The cubes were demolded after 24 hours and kept in saturated lime solution until tested.

The compression tests of the cubes were performed using an MTS electronically servo-controlled hydraulic testing machine. In order to obtain the complete stress-strain diagram for the specimen the displacement of the ram was controlled to give a constant strain rate across the cubes with a first order correction for the displacement of the load cell and deflection of the frame. An important advantage of this correction is that when the specimen begins to fail, the energy stored in the frame and load cell is not "dumped" into the specimen causing premature failure. Since the movement of the ram is controlled to produce a constant rate of strain across the specimen, when the specimen begins to fail and the load on the test frame begins to decrease, the ram will move more slowly or even change direction momentarily. Thus the complete stress-strain diagram for the specimen can be determined.

Specimens for the SEM were cast in special split molds described by Williamson, which could be opened to reveal a fresh fracture surface on the paste specimen.⁵ The specimens were

fractured and placed in the evaporator as quickly as possible to prevent $\rm CO_2$ contamination with the fractured surface. A gold or platinum-palladium film of approximately 200 Å was evaporated on the surface. After being coated, the specimens were kept at 65-70° F with 50% relative humidity and viewed in SEM within a few days; these specimens do not appear to change under these storage conditions over periods of months.

OBSERVATIONS

Microstructure of Alite Pastes

The fracture surface of an alite paste after one day of hydration is shown in Figure 3 in a series of scanning micro-

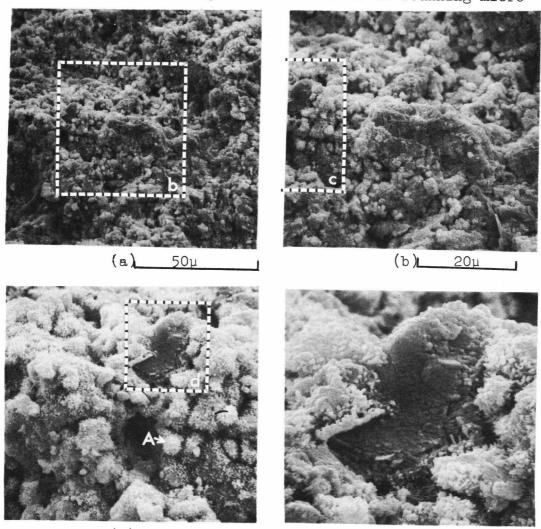


Figure 3. Scanning electron micrographs showing the fracture surface of a 1 day old 0.3 W/C atite paste. Note that the area covered in succeeding micrographs is indicated. In (c), A marks the typical partially hydrated cement grain with a columnar zone of outer hydration products on the outside.

graphs at increasing magnification. At this early age there is little strength and the individual grains of alite with their columnar zone of outer hydration products are clearly visible. An example is shown at A in Figure 3(c). The fracture surface is primarily made up of outer product zones since the fracture occurred through water-filled space. This gives a generally granular texture due to the presence of the individual grains. The fracture surface of alite pastes at 3, 7, 28, and 60 days are shown in Figure 4 and although one such micrograph cannot be definitive there is a general trend to a more solid structure. Figure 4(a) at 3 days still shows the granular texture but many of the grains are broken and there is less outer product columnar zone apparent in the fracture surface. This is interpreted as meaning that the outer hydration products from adjacent grains are growing together to make a bond which prevents them

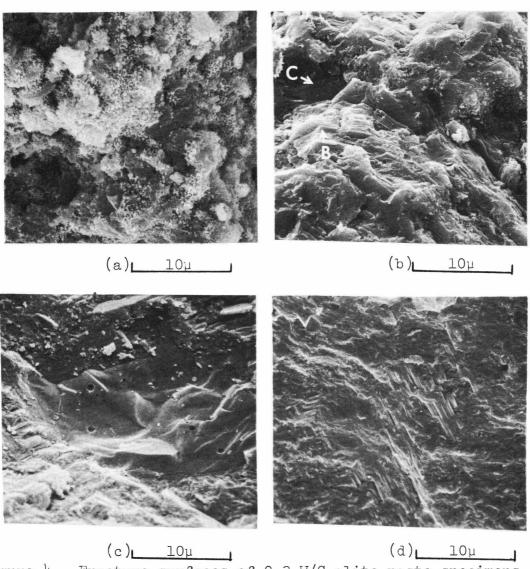


Figure 4. Fracture surfaces of 0.3 W/C alite paste specimens, (a) at 3 days, (b) at 7 days, (c) at 28 days, (d) at 60 days.

from appearing so often on the fracture surface. At 7 days the fracture surface as shown in Figure 4(b) has lost its granular texture and shows at B the cleavage steps of fractured portlandite crystals. There appears to be an unhydrated core of an alite grain at C in Figure 4(b) and a similar core takes up the central portion of the 28 day specimen shown in Figure 4(c). The 28 day old specimen and the 60 day specimen shown in Figure 4(c, d) appear to have become much more solid than the early fracture surfaces and the primary features are the unhydrated core in Figure 4(c) and a relatively large portlandite crystal in Figure 4(d). Williamson has noted that both C_3S and $\beta-C_2S$ pastes appear to gain their strength through the intergrowth of the outer product columnar zones of adjacent grains. 5 A similar observation has been made by Daimon, Ueda and Kondo⁸ who referred to earlier suggestions of this mechanism by Richartz and Locher. 9 Some of these concepts can be tested by observing the changes in microstructure accompanying the acceleration in the hydration process with the addition of gypsum.

The Strengthening of Alite Pastes at Early Ages

The effects of gypsum on the hydration of alite have been studied by various experimental means, and there is a general agreement that the hydration process is accelerated. This results in a strengthening of the alite pastes at early ages, and Mehta, Pirtz and Polivka have shown that the strengths of mortars and concrete made with alite show the same increase in strength at early ages. The effect can be dramatically shown by the stress-strain curves in Figure 5 for 1 day old specimens of alite with and without gypsum. Note that there has been a general increase in stiffness, yield stress, and general deformation behavior, and also note that the stiff testing machine allowed the recording of a complete stress-strain behavior for both materials.

The compressive stress-strain curves for alite pastes of 0.3 W/C are shown in Figure 6(a) for 1, 3, 7, 28, and 60 days,

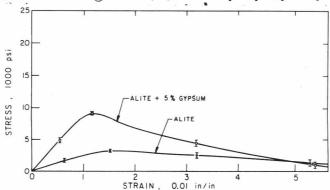


Figure 5. Compressive stress-strain behavior of 1 cm cubes of 0.3 W/C alite paste, with and without 5% gypsum.

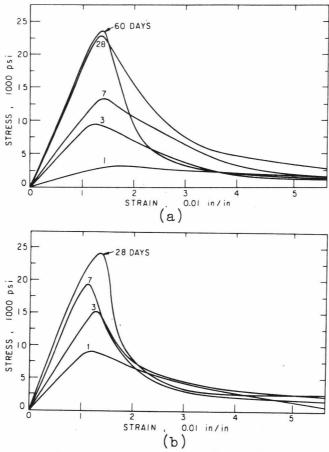


Figure 6. Compressive stress-strain behavior of 1 cm cubes of 0.3 W/C alite paste from age 1 to 60 days, with and without 5% gypsum.

and it is apparent that the paste continues to gain substantial stiffness and strength for the first 28 days. In the corresponding stress-strain curves shown in Figure 6(b) for the specimens with 5% gypsum the early acceleration is readily apparent, but at 28 days the difference is negligible. This early increase in strength is illustrated in Figure 7. It is interesting to note

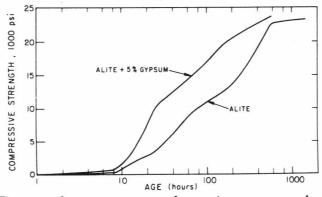


Figure 7. The maximum compressive stress carried by the 1 cm cubes of 0.3 $\rm W/C$ alite pastes, with and without 5% gypsum are shown as a function of age.

that the strength of the 1 day old alite specimens with gypsum is approximately the same as the 3 day old alite specimens without gypsum, and similarly the 3 day old alite specimens with gypsum are approximately the same as the 7 day old alite specimens without gypsum. These same trends are apparent in the microstructure. Representative micrographs of the fracture surface of the alite with 5% gypsum are shown in Figure 8 for ages of 1, 3, 7, and 28 days. As indicated above, the 1 day specimen shown in Figure 8(a) is very similar to the 3 day specimen without gypsum shown in Figure 4(a) with many grains fractured through but the general granular texture still apparent. The granular appearance is gone by 3 days age with gypsum as shown in Figure 8(b). The 7 day old specimen with 5% gypsum appears in Figure 8(c) to be just as solid as the 28 or 60 day specimens without gypsum shown in Figure 4 (c, d) respectively.

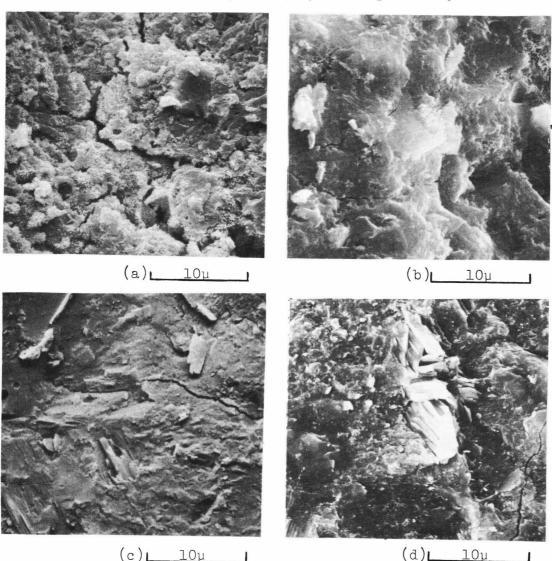


Figure 8. Fracture surfaces of 0.3 W/C alite paste specimens with 5% gypsum, (a) at 1 day, (b) at 3 days, (c) at 7 days, (d) at 28 days.

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Professor Robert H. Bragg (September 16, 1971)

- (1) <u>Comment</u>. Your proposal of "grain refinement" of the $Ca(OH)_2$ phase in cement pastes is the most refreshing idea I've heard in the cement field in a long time.
- (2) <u>Question</u>. How does your claim in an article in <u>Science</u>, that $Ca(OH)_2$ was a major strengthening phase, go with your present statement that the reverse is true?
- (3) Question. It seems to me that the addition of gypsum to cement retarded the setting and strength development whereas your strength vs alloying data (5% gypsum) indicates the opposite. Can you explain this?

Answer

- (1) In the oral presentation I made the proposal that "grain refinement" of the portlandite might be a way to improve the strength of mature cement pastes since I believe that the large portlandite crystals are "flaws" in the system because they cleave so easily. There are references in the literature to a finer portlandite crystal size under certain conditions such as limestone aggregate rather than silicious aggregate.
- (2) In an earlier paper* I noted that portlandite acted to transmit loads within the microstructure and that it was apparently responsible for the strength of the cement paste. I believe this is particularly true at early ages, and this is consistent with the observation that the cleaved portlandite crystals do not begin to appear on the fracture surfaces until later ages. As I indicated in the previous answer, I believe that the portlandite crystals represent flaws in mature pastes.
- (3) The gypsum is usually added to portland cement to prevent the hydration of C_3A with the characteristic "flash set." In the case reported above there was no C_3A so that we could see the effect of gypsum on alite.

^{*}R. B. Williamson, <u>Science</u> <u>164</u> (1969) 549.

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