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Testing Geothermal-Well Cements: Strength Measurements Following Hydrothermal Exposures

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Center for Materials Science
National Bureau of Standards
U.S. Department of Commerce
Washington, DC 20234

April 1979

Interim Report

Issued July 1980

Prepared for

Division of Geothermal Energy
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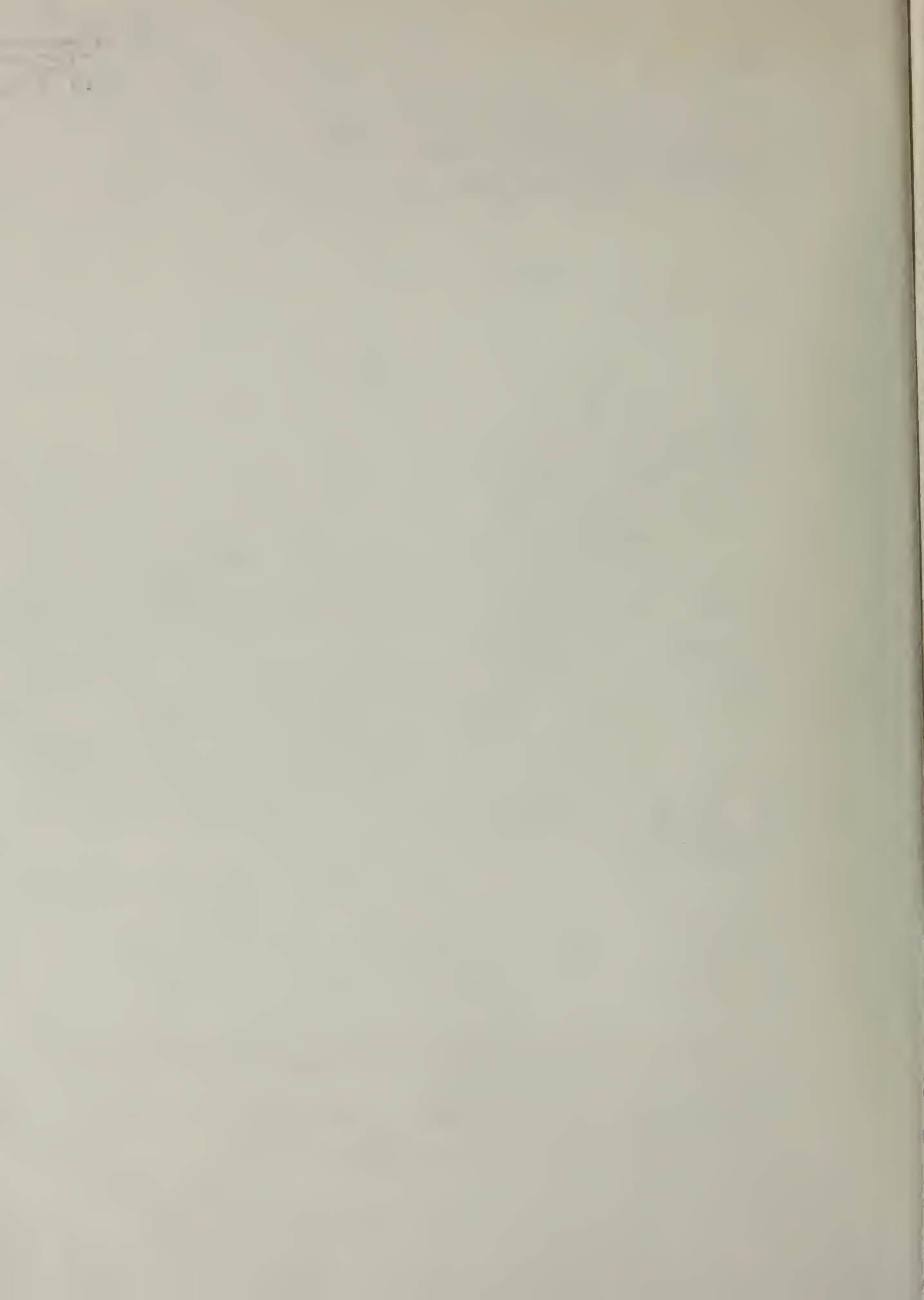


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Abstract

Laboratory data were obtained for the compressive and tensile strength of some candidate cements for geothermal wells after they were exposed to water at a temperature of 195°C and a pressure of 17 to 21 MPa for various periods. Some of these cements were being considered for use in the remedial cementing of a hot-dry-rock well at Los Alamos Scientific Laboratory. The shear-bond strength to steel of the prime candidate for this remedial cementing was also examined. The methods used are preliminary to a standard property verification program, now being developed at the National Bureau of Standards.

Introduction

In an earlier report [1] the results of compressive and tensile strength tests at room temperature and pressure were given for five cements which had been treated according to a certain procedure in water at 195°C and 21 MPa. The cements were first cured in molds for about 40 h and then were exposed out of the molds for about another 136 h. Additional tests have since been made on these same cements after a succession of even longer periods of exposure, extending up to a total of 950 h for some cements.

Due to specimen numbers and size restrictions, these tests did not specifically follow recommended practice of the American Petroleum Institute [2] for testing oil-well cements. Instead, specimens were molded into 18 mm diameter rods rather than the standard two inch cubes. These smaller specimens were chosen because a large number of specimens had to be handled in a short time under the space restraints of our equipment. In addition, inordinate delays often occurred between the time specimens were removed from the autoclave, detached from their molds, cut to size, and eventually tested in a compressive strength test: In an effort at giving a clear history of all specimens tested, the earlier results are repeated here along with the recent results.

Major emphasis was given to testing cements which consisted of sodium silicate, silica, and either ZnO or Al(OH)₃ (cementing materials C and D, respectively, in Table 1) because these cements were prime candidates for the remedial cementing of a hot-dry-rock well at Los Alamos Scientific Laboratory. Initial tests [1] on these cements indicated strengths which were less than those reported by another laboratory [3]. Newly formulated batches of these cements were tested to determine the influences of segregation of dry components, mold material, release agent, curing in a mold under saturated steam versus liquid water, and the wetness or dryness of the test specimens. In addition, specimens of the ZnO cement which had been cured elsewhere [3] were tested for comparison. Prior to the remedial cementing, a measure of the shear-bond strength to steel was attempted for the Al(OH)₃ cement.

After completing the work of interest to the remedial cementing, two additional cements (cementing materials F and G, Table 1) were tested for compressive and tensile strengths. In these two cases, techniques were modified so that freshly molded specimens were cut to size and tested wet without undue delay.

Compressive and Tensile Strength Tests

Slurries

The cementing materials that have been used thus far are listed in Table 1. Batches of 0.6 to 1.0 kg were weighed in the proportions prescribed. Slurries of cementing materials A, B, F, and G were prepared by the API recommended practice [2] except that a two liter high-speed blender was used. Cementing material G was very difficult to blend with water because its slurry was very viscous. Cementing materials C and D were blended with water in a paddle mixer for 10 min, resulting in a workable mortar. The components of cementing material E were preheated separately to 55°C and mixed by hand; this slurry also seemed very viscous.

Molds

Molds for the majority of the tests described below were 20 mm diameter Pyrex tubes with a one mm wall thickness and 150 mm length. Silicone grease, Kraxo, or Teflon powder was used as a mold release. Slurries of the cementing materials were placed into these tubes and puddled; the tubes were then capped with Teflon plugs, which had a one mm leak hole. In addition, slurries of cementing material D were placed into similarly-sized brass tubes with a thin-walled teflon tube lining because this cementing material was suspected to react with Pyrex.

Set-Cure

All of the cementing materials except E were set-cured in their molds for nominally 40 h under autoclave conditions. Usually nine molds of two cementing materials were submerged together in water at 25°C in a stirred autoclave. At the start of the cure the autoclave was pressurized to 8 MPa, and automatic heating brought the contents up to 195°C and 21 MPa in 75 min. These conditions were maintained for the remainder of the curing period. At the end of the cure the inside of the autoclave was water-cooled gradually to about 50°C in 45 min and then depressurized slowly. In contrast the setting of cementing material E was done at one atmosphere in an oven: first, for four h at 55°C; then, for 18 h at 110°C. The set-cure time for a batch of a given cementing material is the first entry of each group of exposure times in Table 2.

The formulations used in preparing the first and second batches of cementing material B did not yield set cements. After each unsuccessfully set batch, the formulation mix was altered as described in Table 1 at the advice of the supplier. Finally, the third formulation provided adequate firmness, for all except approximately the upper ten percent of the material in the mold.

The volume of a set rod of cementing material F appeared about 15 percent smaller than the original volume of its slurry, perhaps due to segregation of water. The corresponding volume change for the other cementing materials appeared slight.

Specimen Sizes

Upon cooling to room temperature, the Pyrex tubes were carefully fractured and broken away from the set cement. The resulting rods of nominally 18 mm diameter were sawed perpendicular to their longitudinal axes into several pieces. Generally, the rods of a given set cement yielded 20 specimens of 36 mm length for compressive strength tests and 20 specimens of 9 mm length for tensile strength tests. The batches prepared prior to the last report [1] encountered considerable delays between the time of removal from the autoclave and testing. These delays have since been brought under control so that specimens from the recent batches were tested wet within 2 to 4 h after the end of their cures.

When cementing materials C and D were first removed wet from the autoclave, they were so friable that specimens of appropriate length were difficult to obtain without fracturing. When they were dried in the room for awhile, these two materials were sufficiently stronger that less breakage was incurred upon the removal of their molds. Even so, several of the rods of cementing material D had to be sawed to size within their molds to provide enough specimens.

Two two-inch cubes of cementing material C which had been set-cured elsewhere [3], were sawed into smaller pieces (15 mm by 15 mm by 23 mm) to provide enough specimens for statistically meaningful test results. Both cubes had been cured initially in saturated steam at 120°C for 6 h; then one cube was cured further in an open autoclave at the same temperature for another 6 h. Both cubes had been received dry with no history of the time between curing and receipt.

Load Tests

Generally, five compressive specimens and five tensile specimens of each set cement were used for the respective strength tests. In the case of a compressive strength test, the diameter (d) of a rod specimen (or the sides of a rectangular specimen) were measured, and its sawed faces were carefully aligned parallel to the bearing blocks of a strength testing machine. The machine was operated at a displacement rate of 0.5

mm/min to determine the force (F_c) required to crush the specimen. In the case of a tensile strength test, both the diameter (d) and the length (L) of a rod specimen were measured; then the test machine was used at the same displacement rate to apply diametral compression to the specimen, until the force (F_t) required to break the specimen was observed. Cardboard cushions were used in these diametral tests to distribute evenly the applied load. The compressive strength (σ_c) and the tensile strength (σ_t) of the rod specimens were calculated from $4F_c/\pi d^2$ and $F_t/\pi dL$, respectively. Values of these strengths for the various set cements are given in Table 2.

Extended Exposures

The remainder of the sawed specimens were exposed to liquid water for extended periods under the same autoclave conditions as used in the set-cure. The autoclave was occasionally depressurized and cooled so that selected specimens could be removed for testing. The remaining specimens were then subjected to another exposure period. Table 2 gives the accumulated time that specimens of each batch of cement was exposed to the autoclave conditions, including the initial cure in the mold, before the strength tests were conducted. Because only one autoclave of a limited capacity was available for either curing specimens in their molds or exposing them out of the molds, several of the set cements were kept at room temperature and pressure for many days before the strength tests. These interim times are also given in Table 2.

During one period of exposure, the specimens were subjected to a sudden loss of pressure due to the fatigue of a safety rupture disc. Consequently the specimens were cooled suddenly and eventually dried upon being reheated automatically to 195°C for an unknown duration over a weekend. All of the specimens which had been prepared from cementing material A, Table 1, and which had been cut to size for compressive strength tests broke apart as a result of this abnormal exposure condition. In contrast almost all the specimens of the other four cementing materials (B, C, D, and E) survived without breaking apart.

Strength Retrogression

The strength of set cement C retrogressed significantly when it was exposed to liquid water at the autoclave conditions. Even the strength of the freshly set cement did not develop adequately despite the time of cure, the wet rods being too fragile to withstand removal of the Pyrex molds. A drying period after the set-cure facilitated the removal of the mold. In our first formulations of this cement only a portion of a large powder supply was used to prepare the batches. Since the observed strength retrogression could have been caused by a segregation of components in the powder mixture, a second shipment was sent from which the entire contents of a one kg package could be prepared. Since the strength of specimens regressed in both instances, possible segregation of the components in the powder mixture appeared to have a negligible influence.

When the abrupt depressurization and drying occurred near the end of the 130 h exposure, the decrease in strength was not as serious as before; but became worse upon subsequent exposure to liquid water at the autoclave conditions. This strength retrogression behavior was also observed for specimens which were prepared from cubes that had been set-cured elsewhere [3].

When set cement D was exposed to liquid water at the autoclave conditions, its strength did not retrogress proportionately as much as that of cement C; but neither was cement D ever as strong as cement C. Even though cement D was allowed to dry after the set-cure, sufficient strength did not develop to withstand removal of the Pyrex without breaking many of the rods. In order to prepare specimens for the extended exposures, they had to be sawed while in their molds. Their strength appeared to increase somewhat after subsequent exposure in their molds. When the abrupt depressurization and drying occurred near the end of 456 h of exposure, the Pyrex molds cracked and were removed before continuing the exposure. After 814 h of exposure, the strength decreased by almost half. Table 2 shows that specimens, which were prepared by different methods to examine possible influences on strength development, were all weak immediately after the set-cure; however, when the specimens were allowed to dry for several days at room temperature, they strengthened considerably.

Some retrogression in strength of set cements A and B was observed upon extended exposures; however, their strengths remained relatively high. The strength of set cement E remained very high throughout all the tests. The strength of set cements F and G were moderately high, although exposures have not been conducted sufficiently long to date to test for any retrogression in strength.

Shear-Bond Strength Tests

Cementing Material

Since a limited time was available prior to the remedial cementing at Los Alamos, the shear bond strength to steel was examined only for cementing material D, the prime candidate.

Molds

Commercial one inch pipe couplings of 316 stainless steel were used to mold the test specimens. Stainless steel end plates were fabricated to center a steel rod (19 mm diameter by 48 mm length) within each coupling. The couplings and rods were cleaned with trichloroethylene and dried. Teflon sheet (0.2 mm) was used as a release agent at the end plates. The cement slurry was poured into and puddled within the molds, and the molds were then stacked inside a pressure vessel.

Thermal Cycling

The cement was cured near 200°C with saturated steam and nitrogen at pressures from 6 to 13 MPa. About 75 min was required to heat the previously pressurized vessel to the operating temperature. The curing conditions were maintained for about two days; then, the vessel and contents were cooled by compressed air to about 60°C in 2 h. The vessel was opened, one specimen was removed, and the above process was repeated with the other specimens for a total of four cycles.

Load Tests

After a specimen was cooled to room temperature, the diameter (d) of the steel rod and the height (L) of the cement and steel rod interface were measured. Then a strength testing machine was used at the displacement rate of 0.5 mm/min to determine the force (F_s) required to displace the steel rod in the cementing-material. Because the cementing-material itself failed near the boundary of the coupling, only a minimum value of the shear-bond strength (σ_s) could be calculated from $F_s/\pi dL$. Table 2 gives these values which were obtained after each cycle in a series of four cycles.

X-Ray Diffraction Examinations

In order to obtain a better understanding of the mineral and micro-structural changes that might be accompanying the observed strength development of the set cement, D, x-ray diffraction patterns were obtained for specimens which had been subjected to the select autoclave and room exposure periods. The results are listed in Table 4 with the corresponding compressive strengths. These preliminary results indicate that the strength of this cement is related to the formation of a series of zeolites which range from analcime with a Si/Al ratio of 2:1 to those with Si/Al ratios of 5:3 and 1:1. The water content of the zeolite increases concurrent with this decreasing silica content.

Acknowledgement

We wish to thank C. R. Robbins for his assistance both in obtaining and in interpreting the x-ray diffraction patterns.

Table 1. Cementing-materials with mass proportions of components.

Code	Mass proportions	Reference
A	1.00 class G cement, 0.40 silica flour, and 0.60 water.	6, 7
B	1.00 class H cement, 0.35 silica flour, 0.045 lignin, 0.015 borax, and 0.56 water. (a)	8
C	1.00 cement (ZnO, sodium silicate, and silica) and 0.18 water. (b)	3
D	1.00 cement [Al(OH) ₃ , sodium silicate, and silica] and 0.18 water. (b)	3
E	0.30 class C cement; 0.70 silica (0.35 mesh 16, 0.175 mesh 30, and 0.175 mesh 100); 0.13 liquid monomers (0.065 styrene, 0.0455 acrylonitrile, 0.013 divinyl benzene, and 0.0065 acrylamide); 0.0013 silane A-174; and 0.00065 catalyst AIBN.	9
F	1.00 cement (β -2CaO·SiO ₂ and silica with 0.65 mole ratio CaO/SiO ₂ , 0.03 bwc Al ₂ O ₃ , and 0.0225 bwc CaSO ₄ ·2H ₂ O); 0.045 bwc perlite; 0.011 bwc bentonite; and 0.85 bwc water.	4
G	1.00 cement (0.3 class J cement, 0.4 pozzolan, and 0.3 blast furnace slag); 0.005 carboxy methyl cellulose; and 0.50 water.	5

(a) Two other formulations were also used; firstly, the mix was altered to have 0.40 bwc water; and secondly, the mix was further altered to have 0.025 bwc lignin, 0.025 bwc borax, and 0.45 bwc water.

(b) Two other formulations in which the mix was altered to have 0.19 and 0.205 bwc water were also used. This cement was received in three separate shipments.

Table 2. Compressive strength (σ_c) and tensile strength (σ_t) of selected geothermal cementing-materials after exposure under liquid water in an autoclave at 195°C and 17 to 21 MPa for various accumulated times. Entries for σ_c and σ_t are the mean value and standard deviation for the N samples.

Cementing materials (a)	Exposure time (h)	Interim (da)	N	σ_c /MPa	N	σ_t /MPa
A	40 (f)	3(dry)	5	90 \pm 8	5	9.4 \pm 1.0
	176	4(dry)	5	64 \pm 6	5	7.8 \pm 0.4
	592 (n)	22(dry)			4	10.7 \pm 1.9
	950	16(wet)			3	11.6 \pm 0.5
B	24 (f)	0.1(wet)				
B (b)	40 (f)	0.1(wet)				
B (c)	40 (f)	4(dry)	5	102 \pm 10	5	12.5 \pm 0.3
	178	0.2(wet)	5	71 \pm 5	5	9.8 \pm 1.0
	456 (n)	22(dry)	3	88 \pm 5	2	12.3 \pm 0.7
	814	16(wet)	3	58 \pm 12	1	12.9
C1	24 (f)	0.1(wet)				
C1	40 (f)	8(dry)	5	34 \pm 3	5	5.0 \pm 0.6
	176	4(dry)	5	4.0 \pm 1.4	5	0.83 \pm 0.15
	502	63(wet)	4	4.6 \pm 1.2	4	0.74 \pm 0.06
C2	40 (f)	3(dry)	5	35 \pm 8	5	7.1 \pm 1.2
	130 (n)	22(dry)	4	11.0 \pm 1.7	5	1.80 \pm 0.13
	488	16(wet)	4	3.2 \pm 0.3	4	0.54 \pm 0.10
C2 (d)	70 (g)	0.1(wet)				
C3	6 (k)	?(dry)	4	27 \pm 4		
	166	0.1(wet)	4	3.5 \pm 0.4		
C3	12 (k)	?(dry)	4	23 \pm 4		
	166	0.1(wet)	4	3.6 \pm 0.5		
D1	40 (f)	3(dry)	2	13.2 \pm 2.4	5	2.6 \pm 0.6
	176	4(dry)	8	20.3 \pm 6.3	5	3.0 \pm 0.7
	592 (n)	22(dry)			5	3.3 \pm 0.4
	950	16(wet)			4	2.6 \pm 0.4
D1	40 (f)	4(dry)	3	17.5 \pm 3.7		
	456 (n)	22(dry)	5	17.1 \pm 1.8		
	814	16(wet)	3	9.8 \pm 1.3		
D2	40 (f)	30(dry)	4	8.6 \pm 0.3		
D2 (d)	70 (g)	32(dry)	5	9.8 \pm 2.3	5	1.71 \pm 0.27
	158 (j)	0.1(wet)	5	7.2 \pm 2.6	5	1.42 \pm 0.44

Cementing materials (a)	Exposure time (h)	Interim (da)	N	σ_c /MPa	N	σ_t /MPa
D3 (e)	36 (i)	0.2(wet)	1	0.9		
	36 (i)	3(dry)	1	7.8		
	101 (i)	0.2(wet)	3	2.4 + 1.6		
	101 (i)	9(wet)	5	2.3 + 0.7		
	101 (i)	9(dry)	5	6.1 + 1.3		
D3 (e)	65 (i,j)	0.2(wet)	5	1.3 + 0.2		
	65 (h,j)	0.2(wet)	2	6.0 + 1.5		
	153 (j)	0.1(wet)	6	3.5 + 0.5		
E	18 (g,m)	14(dry)	5	175 + 3		
	18 (g,m)	27(dry)	5	172 + 2	5	28.2 + 1.8
	176	4(dry)	5	178 + 2	5	19.7 + 2.6
	592 (n)	22(dry)	5	220 + 11	5	17.2 + 0.9
	950	16(wet)	4	175 + 5	4	20.0 + 1.0
F	63 (f)	0.3(wet)	5	22.1 + 1.5	5	2.27+ 0.31
	199	0.1(wet)	5	25.1 + 6.3	5	2.95+ 0.30
G	63 (f)	0.3(wet)	5	35.8 + 1.8	5	3.16+ 0.51
	199	0.1(wet)	5	31.2 + 2.5	5	3.57+ 0.88

- (a) See Table 1. Numerals denote a particular shipment. Line-spaces separate the various batches prepared.
- (b) The mix was altered to have 0.40 bwc water.
- (c) The mix was further altered to have 0.025 bwc lignin, 0.025 bwc borax, and 0.45 bwc water.
- (d) The mix was altered to have 0.19 bwc water.
- (e) The mix was altered to have 0.205 bwc water.
- (f) Material was held in Pyrex molds using Silicone grease as release agent.
- (g) Material was held in Pyrex molds using Kraxo release agent.
- (h) Material was held in Pyrex molds using Teflon release agent.
- (i) Material was held in Teflon-lined brass molds.
- (j) Saturated steam was used in the autoclave instead of liquid water.
- (k) The material was set cured elsewhere [3] in brass molds in saturated steam at 120°C.
- (m) This portion of cure was conducted in room air at 55 and 110°C.
- (n) Specimens were subjected to an abrupt depressurization and subsequent drying-out the last 1 or 2 days of the exposure.

Table 3. Shear-bond strength (σ_s) of the interface between a steel rod and the $Al(OH)_3$ cement (a) after successive thermal cycles between 25 and 200°C (saturated steam and nitrogen at 6 to 13 MPa).

No. of Cycles	Accumulated time, (hr.) at 200°C	σ_s /MPa
1	47	>3.6
2	114	>4.6
3	154	>4.1
4	202	>4.0

(a) Cementing material D from the third shipment was mixed with 0.205 bwc water

Table 4. Mineral phases as determined by x-ray diffraction analysis and compressive strengths (σ_c) of the $\text{Al}(\text{OH})_3$ cement (a) after select exposures to water at 195°C and 21 MPa.

Exposure time (h)	Interim (da)	σ_c /MPa	Mineral phases
65 (b)	0.18(wet)	1.3 ± 0.2	$\alpha\text{-SiO}_2$ $\text{NaAlSi}_2\text{O}_6 \cdot \text{H}_2\text{O}$
814 (c)	16(wet)	9.8 ± 1.3	$\alpha\text{-SiO}_2$ $\text{NaAlSi}_2\text{O}_6 \cdot \text{H}_2\text{O}$ $\text{Na}_3\text{Al}_3\text{Si}_5\text{O}_{16} \cdot 6\text{H}_2\text{O}$
70 (d)	32(dry)	9.8 ± 2.3	$\alpha\text{-SiO}_2$ $\text{NaAlSi}_2\text{O}_6 \cdot \text{H}_2\text{O}$ $\text{Na}_3\text{Al}_3\text{Si}_5\text{O}_{16} \cdot 6\text{H}_2\text{O}$ $\text{NaAlSiO}_4 \cdot n \text{H}_2\text{O}$

(a) Cementing-material D.

(b) Cement from third shipment was mixed with 0.205 bwc water and cured in Telfon-lined brass mold under saturated steam.

(c) Cement from first shipment was mixed with 0.18 bwc water, cured 456 h in a Pyrex mold, and exposed to liquid water for the remaining time.

(d) Cement from second shipment was mixed with 0.19 bwc water and cured in a Pyrex mold under liquid water.

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