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U.S. DEPARTMENT OF COMMERCE National Bureau of Standards Center for Materials Science Fracture and Deformation Division Washington, DC 20234

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Interim Report **Issued December 1981**



Division of Geothermal Energy U.S. Department of Energy Washington, DC

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Abstract

Compressive and splitting tensile strengths were measured for several set cements at room temperature after they had been exposed for periods of 1 week and 1 month to a 20 wt percent salt solution pressurized to 20 MPa and heated to 300 °C. The compressive strength was also measured following exposures for identical periods to distilled water pressurized to 20 MPa and heated to 200 °C. Prior to the exposures, the cements had been set-cured for 2 days in molds immersed in water under the same pressure and temperature as the distilled water exposure. These measurements are part of a project being carried out to evaluate certain physical properties of cements which are candidates for use in finishing geothermal wells.

Introduction

In response to the U. S. Department of Energy (DOE) program to develop improved cements for use in geothermal wells, nine different laboratories submitted cementing materials between January and March 1980 to the National Bureau of Standards (NBS) for property verification tests. An invitation [10] to laboratories interested in participating in the NBS testing program had been issued June 1979 through the American Petroleum Institute (API) Task Group on Geothermal Well Cements, which is serving as a review group for the DOE program. Table 1 gives the 21 cement formulations that were received and subsequently assigned letters for ease of identification. The chemical composition of some of the cement components are given in table 2.

A standard practice [10] for testing geothermal well cements was generally applied in the present work except that the measurements proposed for water permeability and for shear-bond strength to steel were deferred. Only specimens that were destined for compressive or splitting tensile strength measurements were first set-cured in molds and then exposed to simulated geothermal fluids at elevated temperature and pressure. While a first series of tests, conducted between January and March 1980, involved only compressive strength measurements on cement specimens that had been exposed only to distilled water, a second series of tests, subsequently conducted between April and June 1980, also included compressive and splitting tensile strength measurements on cement specimens that had been exposed to 20 weight percent salt water. The practice was modified because the API Task Group had requested at its December 1979 meeting that as many of the candidate cements as possible be partially examined prior to its March 1980 meeting. We believe that the revised schedule of tests best utilized the limited pressure vessel space in the time available.

As of June 1980, 15 cement formulations, which include the first and second choice of each participating laboratory, were partially examined. In regard to the first series of tests, seven of the twelve cements tested had been prepared without their specified retarders because the retarder for some of them had not been submitted initially. Upon the advice of the API Task Group, the tests were repeated on most of these cements along with their retarders as part of the second series of tests. Also, two new cements (M and N), which were not received until March 1980, were included in the second series of tests. The results of the two series of tests had been presented at the March and June 1980 meetings, respectively, of the API Task Group.

Specimen Preparation

Using a truncated version of the proposed schedule of tests [10], the preparation of a cement slurry was initiated on separate occasions, usually twice, but as many as four times, instead of once weekly as originally proposed. On each occasion the weighed components of a given cement being prepared were mixed to form 600 mL slurry. After the dry ingredients were hand-blended, their mix was added during a period of about 15 s to the liquid in a two speed propeller blender (2 L capacity) operating at the lower speed; next the slurry was blended for 35 s at the higher speed. The slurry was then poured into as many as 18 glass tube molds (20 mm o.d., 150 mm length, 1.5 mm wall) that had been coated lightly inside with silicone grease beforehand. After the slurry in each tube was puddled to remove trapped air bubbles, each of the filled tubes was capped with a Teflon stopper (10 mm length, 1 mm hole); and the capped tubes were secured together in a stainless steel basket.

The molded slurry of each cement was set-cured nominally two days (42 h actually) at elevated temperature and pressure. Immediately upon

completion of the slurry preparation, the basket of molds of a given cement was immersed in distilled water at room temperature and sealed in an internally stirred autoclave of stainless steel (4 L capacity). The water level was adjusted so that the water occupied about 78 percent of the available fluid space (autoclave capacity less volume of specimens), and the autoclave was next pressurized to about 7 MPa with nitrogen gas. Curing was considered to commence when the contents of the autoclave began being heated at the rate of about 2.4 °C/min. In about 75 min the temperature of the contents increased to 200 °C, and the autoclave became pressurized to about and eventually adjusted to 20 MPa, mainly due to expansion of the water and compression of the gas. The initial charge of water was deliberately chosen so that the volume ratio of final gas phase to available fluid space would be about 10 percent. These conditions were maintained throughout the remainder of the curing period by automatic temperature control, using a sheathed thermocouple inside the autoclave. The cure was considered to terminate when cold water began to circulate through internal coils of the autoclave, cooling the contents at a rate about the same in magnitude as the heating rate. When the temperature of the contents decreased to about 50 °C, thermal contraction had reduced the pressure again to 7 MPa; and the autoclave was further depressurized slowly and dismantled.

Following the two day set-cure of a given cement, the glass tubes were carefully broken away from the rods of set cement; and each rod was diamond sawed into three specimens, the length being twice the nominally 17.5 mm rod diameter. The specimens were immediately immersed in room temperature water and maintained wet. At this stage six specimens of each set-cured batch of cement were subjected to strength tests to provide a data base, to which the results of testing the other specimens of the batch could be compared, following their exposures as described later.

Because the prescribed method of slurry preparation was not appropriate with some cements, special care was exercised as follows:

1. Cements F and G: The water/solid weight ratio of 0.50 given in table 1 produced a paste slurry. In consultation with reference [5] a weight ratio of 0.60 was used in the present work, giving a more pourable slurry. Cement H: As reference [1] specified, water was added to the solid 2. and they were mixed in a low-speed paddle mixer for about 5 min until the slurry became pourable. Following the two day set-cure, the rods of set cement tended to stick to the silicone greased glass tubes and were so friable that many rods did not withstand removal of the glass molds. In a search to produce sound specimens, other batches of this cement were set-cured alternatively either in glass tubes on whose inside surface a Teflon spray release agent had been baked, or in thin-walled Teflon tubes which were held in split tubes of stainless steel with Teflon-lined copper caps. In addition several specimens (15 mm square, 30 mm length) were diamond sawed from three rods (50 mm diameter, 50 mm length) that reference [1] had set-cured reportedly in saturated steam at about 200 °C.

3. Cement P: As reference [7] specified, this cement was set-cured at 240 °C instead of the customary 200 °C. Since the first shipment of liquid siloxane monomer failed to set, a second shipment of the liquid to which a catalyst had been added was used, resulting in set specimens.

4. Cement Q: As reference [3] specified, this cement was set-cured while the molds of slurry were immersed in diesel fuel oil. Although the autoclave was controlled at the specified temperature of 115 °C, the exothermic polymerization reaction of the cement occurred so suddenly that the temperature increased to about 170 °C. The resulting specimens were foam-like, having

expanded out of the molds to be twice their length; and they possessed about one-tenth the roughly 60 MPa strength of specimens that were cured successfully. A few specimens of this cement were set-cured successfully in another batch when the proportion of slurry being cured in the fuel oil was reduced to about one-tenth that ordinarily used, thus limiting the rise in temperature during the set-cure. In consultation with reference [3] further examination of this cement was deferred at this time because the preparation of set specimens proved too difficult.

Exposures

Two separate treatments were used to expose specimens of the cements at elevated temperature and pressure. Simulating light and heavy geothermal fluids, distilled water heated to 200 °C and 20 weight percent salt water heated to 300 °C constituted the two treatments, both being confined under 20 MPa pressure of nitrogen gas. While only distilled water was used in the first series of tests, a batch of freshly set-cured specimens of a given cement was usually divided into two separate groups of twelve specimens each for exposure in the two fluids, respectively, usually along with specimens of one to three other cements that had already been exposed for some time.

The chemical composition of the 20 weight percent salt solution was formulated to represent Salton Sea fluids, a somewhat arbitrary task since their salt content varies considerably. The salt solution consisted of 2.3 molal NaCl, 0.36 molal KCl, and 0.66 molal $CaCl_2$ (1 molal = 1 mol solute per kg water); also, it was equilibrated with 0.2 MPa partial pressure of CO₂, giving a pH of about 4.4 at 25 °C in a closed vessel.

The exposures were conducted in a recently constructed high pressure, high temperature fluid handling facility [11], which included a series of four Hastelloy alloy C-276 pressure vessels (5.1 cm i.d., 26 cm inside length). Demolded specimens of cement were installed within a given pressure vessel along with stainless steel blanks as necessary to keep constant the space that was occupied by as many as thirty specimens. The vessel was charged initially with a specified quantity of room temperature liquid and pressurized to 7 MPa with nitrogen gas so that the volume ratio of final gas phase to available fluid space be about ten percent, the same criterion as used in the set-cure. This corresponds either to filling 78 percent of the available fluid space of a vessel with distilled water that is eventually heated to 200 °C and pressurized to 20 MPa or to filling 64 percent, with 20 weight percent salt water that is eventually heated to 300 °C and also pressurized to 20 MPa (for example, about 220 or 180 mL, respectively).

Each pressure vessel with its contents was completely enclosed within a wire coil resistance, split tube furnace whose operation was automatically controlled by a thermocouple attached at the middle of the vessel exterior. A second thermocouple in a Hastelloy alloy sheath was used to indicate the temperature at the inside center of the pressure vessel. About 90 min was required from the time heating commenced for this internal temperature to attain 80 percent of its ultimate level. Weekly, the specimens of cement were cooled to about 50 °C in 2 h by directing compressed air on the pressure vessels in the furnace cavity. After the vessels were depressurized and dismantled, some specimens of cement were withdrawn and other specimens deposited as necessary to fulfill 7 or 28 day periods of exposure. Freshly made fluid was again installed in the pressure vessels and the cycle of operations was repeated. Meanwhile, the withdrawn specimens of cement were immersed in room temperature water until their strengths were measured.

The detailed procedure for testing the compressive strength (σ_c) and the splitting tensile strength (σ_{+}) of the set cements is described elsewhere [10]. In brief, the load bearing faces of the testing machine and of a test specimen were wiped clean. The length (L) and the diameter (D) of the specimen were measured within an accuracy of \pm 0.05 mm. Using a wooden guide, the specimen was centered directly under a freely rotatable, spherically seated compression tool which was suspended on a gear driven crosshead. In the diametral splitting tests, unused cardboard strips (0.3 mm thick) were placed between the specimen and the load bearing faces to distribute the applied force smoothly. A compression force was applied at a constant displacement rate (0.5 mm/min) until the maximum force (F) necessary to cause failure of the specimen was recorded. The values of F were observed in lbf units (1 lbf = $0.45359 \cdot 9.80665$ N) within an accuracy of ± 0.3 percent according to a recent calibration of the testing machine. Values of σ_{c} and σ_{t} were calculated from 4 F/ πD^{2} and 2 F/ πDL , respectively, in tests on separate specimens.

The results of compressive strength measurements that followed the initial set-cure and the distilled water exposures at 200 °C are given in Table 3 and Figures 1a, 1b, and 1c. The results of compressive strength measurements that followed the initial set-cure and the 20 weight percent salt water exposures at 300 °C are given in Table 4 and Figures 2a and 2b. Finally, the results of splitting tensile strength measurements that followed the initial set-cure and the same salt water exposures are given in Table 5 and Figures 3a and 3b. Line entries in the tables which show the same set-cure values for a given cement indicate specimens that originated from the same batch.

Since the present work, which was designed as part of a preliminary screening effort, involved a limited number of tests per event, any trends that the strength data portray with time of exposure should be considered as approximate. The mean values that are tabulated in Tables 3, 4, and 5 are not based evenly among the 686 specimens tested. Excluding duplicate entries, 85 of the mean values were based on 6 tests each (2 rods of set cement sawed into 3 specimens each), 5 of the mean values were based on 4 tests each, and 52 of the mean values were based on 3 tests each (3 specimens sawed from the same rod of set cement). Statistically meaningful trends would be based on larger populations than used here. One trend, however, was confirmed manyfold; the compressive strength of a rod of set cement, especially a lightweight cement, was found generally to increase with the depth that specimens were extracted from the rod, no doubt due to segregation of water from the heavier components prior to setting of the cement. The standard deviation of a mean value which is based on 3 tests generally reflects this trend for a given cement.

Very little distinction between the presence or absence of a retarder in cements A, B, D, F, K, and L is apparent within the precision of measurements. The presence of a retarder perhaps has made these cements slightly stronger immediately following their set-cure, but the differences could easily be due as much to the separate batches of set-curing.

The cements varied in their response to being exposed in distilled water at 200 °C. The high strength of cement P tended to increase with exposure time, the high strength of cement D held fairly steady within wide bounds, and the high strength of cements C and N began to decline somewhat following the four week period of exposure. Although cements A and B were

initially very strong at the one-day set-cure, their strengths retrogressed to lower levels upon exposure. The same held true to a lesser extent for the moderately strong cements F and M. The moderate strengths of cements E, J, K, and L held generally steady. Cements G and H were the weakest. Although the glass-molded specimens of cement H were moderately strong at the one-day set-cure, their strength retrogressed severely upon exposure.

The moderately strong cement L appears to have become slightly stronger in salt water at 300 °C than in distilled water at 200 °C. The respective strength trends of cements B, D, K, and P appear about the same with time of exposure in either fluid. Although the glass-molded specimens of cement H showed about the same strength retrogression in either fluid, the cement H specimens which were set-cured by reference [1] gave an increase in strength upon 4 week exposure to salt water at 300 °C; this inconsistency might possibly be due to the position these latter specimens occupied in the original stock material. The strengths of cements A, F, and M appear to have retrogressed slightly more when these cements were exposed to the salt water at 300 °C than to the distilled water at 200 °C. Drastic reductions in strength of cements C, E, J, and N were observed when these cements were exposed to the salt water at 300 °C. A reduction in strength was expected for cement C, for as reference [7] advised, it is useful at temperatures up to about 250 °C, above which the organic polymers decompose appreciably.

While the splitting tensile strength has about one-tenth the value of the compressive strength, both properties yielded parallel trends with time of exposure of the respective cements to the salt water at 300 °C.

Identity	Parts by Weight	<u>Components</u>	Reference
A	100 35 54 1	API ^a class G cement silica flour water lignin/sugar retarder	9
В	100 44 0.4	API class J cement water lignin/sugar retarder	9
С	100 22	solid aggregate liquid organic monomers	7
D	100 45 0.7	cement water retarder	2
E	100 4.5 1.1 85	modified _B -C ₂ S cement perlite bentonite water	4
F	30 40 30 50 0.5	API class J cement pozzolan blast furnace slag water carboxy methyl cellulose	5
G	30 40 30 50 0.5	API class J cement silica flour pozzolan water carboxy methyl cellulose	5
Н	100 20.8	hydrothermal cement (250 °F) water	1
J	100 50	ordinary Portland cement water	б
К	100 35 2 8.5 116 1	API class G cement silica flour bentonite perlite water lignin/sugar retarder	9
L	100 35 10 91 1	API class G cement silica flour diatomaceous earth water lignin/sugar retarder	9

Table 1. Formulations of geothermal-well cements submitted for testing at the National Bureau of Standards

Table 1. Continued.

Identity	Parts by Weight	Components	Reference
М	80 20 47.5 0.25	API class J cement calcined chrysotile (M ₃ S ₂) water D-28 Dowell retarder	8
N	100 100 89.1 0.9	system CA-CA ₂ cement 5 µm quartz water 100XR Pozzolithe	8
0	60 40 47.5 0.375	API class J cement calcined chrysotile (M ₃ S ₂) water D-28 Dowell retarder	8
Ρ	1 00 50	solid aggregate liquid siloxane monomer	7
Q	110 1	mixture containing furfuryl alcohol resin catalyst	3
R	100 35 20 54 1	API class B cement silica flour NaCl water lignin/sugar retarder	9
S	100 100 2 1 135 1	API class G cement silica flour sodium silicate extender NaOH water lignin/sugar retarder	9
Т	30 30 40 50 0.5	API class J cement silica flour blast furnace slag water carboxy methyl cellulose	5
U	30 30 40 50 0.5	API class J cement pozzolan blast furnace slag water carboxy methyl cellulose	5
V	30 30 35 5 50 0.5	API class J cement silica flour blast furnace slag bentonite water carboxy methyl cellulose	5

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Identity	Component	Composition (parts by wt)	Reference
A,K,L,S	API class G cement	By analysis: 64.2 CaO, 21.5 SiO ₂ , 3.9 Al ₂ O ₃ , 3.8 Fe ₂ O ₃ , 1.3 MgO, 2.1 SO ₃ , 0.7 alkalies, 0.6 free CaO	9
		By calculation: 60.3 C_3S , 16.2 C_2S , 11.5 C_4AF , 3.9 C_3A , 3.5 $CaSO_4$	
В	API class J cement (system β-C ₂ S and silica)	By analysis: 37.3 CaO, 54.2 SiO ₂ , 1.1 Al ₂ O ₃ , 1.0 Fe_2O_3 , 0.3 SO ₃ , 4.6 loss on ignition	9
C	solid aggregate	30 API class C cement, 70 sand (50 mesh no. 16, 25 mesh no. 30, 25 mesh no. 100)	7
С	liquid organic monomers	50 styrene, 35 acrylonitrile, 5 acrylamide, 10 divinylbenzene, 1 silane A-174	7
Ε	modified β-C ₂ S cement	100 (β-C ₂ S + silica with 0.65 mol ratio CaO/SiO ₂), 3 Al ₂ O ₃ , 2.25 CaSO ₄ ·2 H ₂ O	4
Н	hydrothermal cement (250 °F)	7 Al(OH) ₃ , 10 Britesil, 10 anhydrous sodium silicate, 25 silica flour, 50 sand, 0.5 bentonite	I
Μ	API class J cement (system β-C ₂ S and silica)	By analysis: 43.79 CaO, 49.25 SiO ₂ , 0.76 Al ₂ O ₃ , 0.59 Fe_2O_3 , 0.29 MgO, 0.27 SO ₃ , 0.46 alkalies, 3.50 loss on ignition	8

Table 2. Composition of cement components as given by sources

Table 2. Continued.

Identity	Component	Composition (parts by wt)	Reference
N	system CA-CA ₂ cement	By analysis: 28.4 CaO, 0.35 SiO ₂ , 70.5 Al ₂ O ₃ , 0.10 Fe ₂ O ₃ , 0.43 MgO, 0.32 SO ₃ , 0.27 alkalies, 0.38 loss on ignition	8
Ρ	solid aggregate	10 API class C cement, 90 silica flour	7
Ρ	liquid siloxane monomer	97 tetramethyltet ravinyl- cyclotetrasiloxane, 3 polydimethyl- siloxane, 0.5 di-tert-butyl peroxide	7

Cement abbreviations: $A = Al_20_3$, C = Ca0, $F = Fe_20_3$, M = Mg0, $S = Si0_2$

Table 3.	Compressive strength (σ_c) of set cements at 25°C, following
	the set-cure and subsequent exposures to distilled water,
	pressurized to 20 MPa and heated to 200°C. Except as noted,
	entries give the mean and standard deviation of 6 tests.

Cement		σ _c /MPa		
	2 da set-cure	7 da exposure	28 da exposure	
A ^a A	67.8 + 6.4 77.1 + 10.2	34.0 + 7.8 31.3 + 3.6	$\begin{array}{r} 36.3 \pm 4.2 \\ 24.4 \pm 4.1 \end{array}$	
B ^a B	$54.0 + 7.9 \\ 62.1 + 4.1$	$\begin{array}{rrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrr$	$\begin{array}{r} 32.8 + 2.5 \\ 30.1 + 2.6 \end{array}$	
С	155.1 <u>+</u> 9.9	175.8 <u>+</u> 13.4	133.6 <u>+</u> 7.7	
D ^a D	58.3 ± 7.8 57.9 ± 18.7	52.4 + 12.552.0 + 7.7	$\begin{array}{r} 63.9 + 15.8 \\ 59.1 + 7.2 \end{array}$	
E	21.3 <u>+</u> 2.8	25.4 <u>+</u> 2.0	24.5 <u>+</u> 3.5	
F ^a F	$\begin{array}{rrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrr$	$\begin{array}{rrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrr$	15.5 ± 0.5 14.4 ± 1.2	
G ^õ	1.6 <u>+</u> 0.3	5.6 <u>+</u> 0.4	3.6 <u>+</u> 0.2	
H H H H H H H	$\begin{array}{rrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrr$	5.6 + 0.4 $4.6 + 1.7$ $6.1 + 1.5$ $3.2 + 0.4$	$\begin{array}{r} 4.4 + 1.6 \\ 2.2 + 1.4 \\ 8.3 + 1.6 \\ 2.4 + 0.5 \end{array}$	
J	16.1 <u>+</u> 1.9	14.2 <u>+</u> 2.1	17.9 <u>+</u> 2.2	
к ^а К	$\begin{array}{rrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrr$	$\begin{array}{rrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrr$	16.2 + 1.9 14.1 + 4.0	
L ^a L	$\begin{array}{rrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrr$	$\begin{array}{rrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrr$	$26.9 + 1.6 \\ 25.0 + 2.1$	
М	37.6 <u>+</u> 5.1	26.9 <u>+</u> 0.8	25.0 <u>+</u> 2.1	
N	88.9 <u>+</u> 11.3	99.4 <u>+</u> 10.4	58.2 <u>+</u> 6.9	
Р	60.7 <u>+</u> 2.1	73.4 <u>+</u> 9.1	76.2 <u>+</u> 3.4	

1 MPa = 145 psi

^aRetarder not added.

^bSet-cured in teflon-coated glass tubes.

^CSet-cured in teflon tubes.

^dSet-cured by reference 1; 4 tests conducted per entry.

Table 4. Compressive strength (σ_c) of set cements at 25°C, following the set-cure and subsequent exposures to 20 wt per cent salt water, pressurized to 20 MPa and heated to 300°C. Except as noted, entries for the set-cure and exposures give the mean and standard deviation of 6 and 3 tests, respectively.

Cement ^a		σ _c /MPa	
	2 da set-cure	7 da exposure	28 da exposure
А	77.1 <u>+</u> 10.2	21.6 <u>+</u> 1.0	19.9 <u>+</u> 2.3
В	62.1 <u>+</u> 4.1	30.0 <u>+</u> 2.0	32.4 <u>+</u> 0.9
С	141.8 <u>+</u> 7.0	80 <u>+</u> 28	6.8 <u>+</u> 2.1
D	62.3 <u>+</u> 7.4	37.1 <u>+</u> 4.1	55.9 <u>+</u> 2.5
E	20.0 <u>+</u> 3.0	11.4 <u>+</u> 1.6	1.0 <u>+</u> 0.2
F	25.1 <u>+</u> 2.1	17.1 <u>+</u> 0.9	12.2 <u>+</u> 1.1
н ^b н ^d	$\begin{array}{rrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrr$	5.6 + 0.64.2 + 0.2	5.9 <u>+</u> 1.6 14.5 <u>+</u> 2.5
J	15.2 <u>+</u> 1.3	9.1 <u>+</u> 1.0	1.9 <u>+</u> 0.6
К	20.1 <u>+</u> 5.0	12.1 <u>+</u> 3.9	11.4 <u>+</u> 6.1
L	28.5 <u>+</u> 3.3	37 <u>+</u> 12	33 <u>+</u> 19
М	34.6 <u>+</u> 5.5	18.7 <u>+</u> 1.6	19.2 <u>+</u> 1.1
N	81.6 <u>+</u> 7.4	11.8 <u>+</u> 0.3	19.1 <u>+</u> 0.4
Р	60.7 <u>+</u> 2.1	75.7 <u>+</u> 8.4	84.5 <u>+</u> 10.9

1 MPa = 145 psi

^bSet-cured in teflon-coated glass tubes.

^dSet-cured by reference 1; 4 tests conducted per entry.

Table 5. Splitting tensile strength (σ_t) of set cements at 25°C following the set-cure and subsequent exposures to 20 wt per cent salt water, pressurized to 20 MPa and heated to 300°C. Entries for the set-cure and exposures give the mean and standard deviation of 6 and 3 tests, respectively.

Cement	t	σ _t /MPa		
		2 da set-cure	7 da exposure	28 da exposure
А		7.00 <u>+</u> 1.68	2.72 <u>+</u> 0.24	2.74 <u>+</u> 0.20
В		5.70 <u>+</u> 1.54	4.78 <u>+</u> 0.51	4.85 <u>+</u> 0.66
С		18.0 <u>+</u> 2.4	8.3 <u>+</u> 2.4	1.41 <u>+</u> 0.39
D		5.14 <u>+</u> 1.29	4.38 <u>+</u> 1.27	5.71 <u>+</u> 0.91
Ε		2.30 <u>+</u> 0.36	2.26 <u>+</u> 0.14	0.28 <u>+</u> 0.08
F		3.37 <u>+</u> 0.61	1.92 <u>+</u> 0.12	1.50 <u>+</u> 0.22
н ^р		3.00 <u>+</u> 0.99	0.76 <u>+</u> 0.37	0.66 <u>+</u> 0.09
J		1.81 <u>+</u> 0.38	1.44 <u>+</u> 0.37	0.34 <u>+</u> 0.01
К		2.55 <u>+</u> 0.36	2.31 <u>+</u> 0.68	1.70 <u>+</u> 0.46
L		3.49 <u>+</u> 0.65	5.77 <u>+</u> 1.00	6.76 <u>+</u> 0.56
М		4.69 <u>+</u> 0.57	3.23 <u>+</u> 0.25	2.46 <u>+</u> 0.18
N		8.66 <u>+</u> 1.41	1.60 <u>+</u> 0.53	2.96 <u>+</u> 0.13
Р		4.22 <u>+</u> 0.76	6.35 <u>+</u> 0.39	6.26 <u>+</u> 1.84

1 MPa = 145 psi.

^bSet-cured in teflon-coated glass tubes.





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- 9. B. E. Simpson and L. H. Eilers, Dowell Division of Dow Chemical, P.O. Box 21, Tulsa, Oklahoma 74102.
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