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Borehole Plugging-Materials Development Program

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BOREHOLE PLUGGING-MATERIALS DEVELOPMENT PROGRAM

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ABSTRACT

This report discusses the background and first year's results of the grouting materials development program for plugging boreholes associated with the Nuclear Waste Isolation Pilot Plant. The grouts are to be pumpable, impermeable, and durable for many thousands of years. The work was done at the Concrete Laboratory of the U.S. Army Engineer Waterways Experiment Station (WES), Vicksburg, Mississippi. The workability, strength, porosity, bonding, expansion, and permeability data are summarized and discussed. The work is continuing at WES.

ACKNOWLEDGMENTS

The grouting materials development work was conducted by the Concrete Laboratory of the U.S. Army Engineer Waterways Experiment Station, Vicksburg, Mississippi. Bryant Mather, Chief of the Concrete Laboratory, and many members of his staff have actively participated in the many coordination meetings. J. A. Boa, Jr., under the supervision of R. A. Bendinelli, head of the Grouting Branch, has been responsible for conducting the studies and reporting the results.

The study program is funded by the Department of Energy, Waste Isolation Pilot Plant program. Sandia's Waste Management Technology Department has technical responsibility. L. R. Hill and S. J. Lambert of the Nuclear Waste Technology Division have participated in coordination meetings at WES and in other discussions related to the program.

CONTENTS

TABLES

BOREHOLE PLUGGING-MATERIALS DEVELOPMENT PROGRAM

I. Introduction

Representatives of Sandia Laboratories attended a Borehole Plugging Program Review meeting in July 1975, conducted by the Office of Waste Isolation (OWI) at the Oak Ridge National Laboratory (ORNL). Industrial experts and ERDA personnel reviewed the accomplishments of the program to that date.

The purpose of the program is to

- Develop techniques for plugging boreholes through and adjacent to underground waste disposal facilities
- Seal boreholes with plugs which will prevent movement of fluids and gases toward or through the salt beds
- Provide plugs which will maintain their integrity for time periods comparable to the life of the rock formations in which they are used.

The concensus of the review meeting was that the only material currently available for plugging boreholes is high-quality cement. The desired approach is to improve the cementing technology, primarily materials, to meet the stringent quality standards required for waste storage sites.

Subsequent to that meeting, a state-of-the-art report[1] for borehole plugging was completed and distributed. A second Borehole Plugging Review Conference was held in Tucson in November 1976. Again, the Ad Hoc Committee concluded that existing state-of-the-art methods, machinery, materials, and knowledge are adequate to ensure that boreholes can be reentered and that borehole plugs comparable to or stronger than the original rocks can be installed to prevent vertical movement of fluids and gases. The committee further concluded that the problem of credibility remains and must be addressed.

In a letter dated January 7, 1977 to the US ERDA, Division of Waste Management, the Director of OWI listed three major programs for FY77 and FY78:

1. Cement and Concrete Research and Development. Continue to support cement research at Penn State and at ORNL. It was noted that this work is complemented by a Sandia-supported project at the U.S. Army Engineer WES, Vicksburg, MS.
2. In Situ Borehole Plugging Demonstration Project.

3. Develop instrumentation to monitor the physical and chemical properties of the plug and wall rock materials with telemetering systems and remote power supplies for many years.

Sandia commented that several years of development are needed for the third program and its successful completion is needed for the long-range monitoring of the second program to develop the credibility needed for the Borehole Plugging Program.

The initial conclusions reached in 1975 were the impetus for initiating a long-range development and testing program at the WES Concrete Laboratory. The program is to study and improve cementing materials and to conduct exposure tests of materials over an extended time period. This report will summarize the preliminary discussions and the various activities both in the laboratory and in the field which have taken place in support of the Borehole Plugging Development Program. It will amplify the first WES report of studies for the Borehole Plugging Program.[2]

II. Background

Discussions concerning plugging boreholes and durability of cementing materials have been held since July 1975 with individuals from the following organizations:

- B. J. Hughes, Inc.
- Concrete Laboratory, WES
- Dowell Division of DOW Chemical U.S.A.
- Haliburton Services
- Materials Research Laboratory, Pennsylvania State University
- Portland Cement Association
- Southwestern Portland Cement Company
- U. S. Navy Civil Engineering Laboratory

These discussions have been useful in finding previous and current studies about the behavior of cementing materials and in obtaining ideas for possible improvements.

A Dowell study[3] evaluated existing materials and techniques (in 1973) to determine the most suitable system for plugging boreholes. The recommended cements for the New Mexico area were two from Southwestern Portland Cement Company: El Toro Type V and El Toro ChemComp (expanding) sulfate-resistant cement. Both cements produced excellent bonds to formation materials and are resistant to sulfate-water. The report stated that the trend was to use expanding cement (ChemComp) in critical areas; however, the Bureau of Mines was the only one publishing data to back up the statement.[4] Preliminary laboratory testing reported by the Bureau of Mines showed lower values of porosity and permeability and higher bulk density for the ChemComp grout mixture than for normal portland cement and a flyash-and-portland cement mix.

The recommended cementing material in Reference 1 for the New Mexico area is a special Class C (SR) cement which contains no C_3A and, therefore, has high-sulfate resistance. The only two cements manufactured for the oil industry and meeting these requirements are Lone Star Cement "Incor" from Maryneal, Texas and Southwestern Portland Cement "El Toro 35" from Odessa, Texas. The addition of flyash and dispersant to lower the water content are also recommended.

The Portland Cement Association (PCA) conducted studies of the resistance of portland-cement mortar and concrete to attacks by sulfate and other chemicals for several decades.[5, 6] The laboratory studies of mortar bars in Reference 4 showed that El Toro Type V cement (calculated C_3A content of 3.7%) is more resistant to sulfate attack than the other cements. The field exposure studies[5] showed that cements with less than 11% C_3A content performed without concrete deterioration through 25 years of exposure to sulfate soils.

Lea discusses the resistance of concretes to organic and inorganic chemicals, natural waters, and sulfate soils.[7] In addition to the importance of low C_3A content to resist sulfate attack, he stresses the pozzolanic cements to increase resistance to chemical attack.

Tuthill stated that, in addition to low tricalcium aluminate content being the most important factor in improving resistance of concrete to sulfate, several other factors provide improvement: (1) reduce water/cement ratio, (2) reduce the leaching of hydrated lime by using an active pozzolanic material as a 15% to 30% substitution of the cement content by weight.[8] Tynes reported data on the effect of fly-ash replacement in concrete mixtures;[9] he compared 25% and 35% replacement by volume (approximately 21% and 29% by weight). For the 90-day age comparisons, the permeability was reduced for the fly-ash-replacement mixtures compared to the all-portland-cement mixture. Both amounts of fly ash gave about the same permeability while compressive strength decreased slightly and abrasion loss increased for the higher fly-ash content. WES concluded that for fly-ash-portland cement having a fineness of about 4000 cm^2/g (Class C), the data suggests an optimum fly-ash content of about 25% by weight.[10]

Mehta found that expansive cement concretes made with shrinkage-compensating Type K cements were similar to the concrete made with Type V portland cement.[11] The experimental results and theoretical computations showed that the resistance to sulfate attack is related to the amount of Al_2O_3 (the A in C_3A) available.

Mikhailov studied the durability and permeability of concrete made with self-stressing expansive cements.[12] He found that the great compactness of stressing-cement concrete gives it an increased resistance to the passage of fluids and gases. Permeabilities of pipe made with stressing cement were reduced by an order of magnitude or greater than with normal portland cement. When the properly restrained specimens of stressing cement were exposed to sulfate attack, they exhibited comparable sulfate resistance to low-aluminate portland cement.

The data in the references and the discussions suggested the following approach to the problem of providing maximum long-term durability, high resistance to groundwater attack, low permeability, and adequate working time for pumpable grout mixtures:

1. Select a Type K shrinkage-compensated expansive cement with a no tricalcium aluminate content for the "normal" portland portion of clinker.
2. Include more highly expansive Type K self-stressing cement to possibly improve bonding to the rock, reduce permeability, and overcome the destressed rock adjacent to the borehole.
3. Include both fly ash and natural pozzolans to study their effects on permeability and durability.
4. Study water reducing admixtures (super plasticizers) to find those most efficient in reducing the water/cement ratio.

5. Include a retarder to achieve a working time of 3 hours for any mixture selected for extended testing.

6. Add salt, both dissolved in the mixing water and as salt sand, to some mixtures for the grouts to be placed through the "salt section" of a borehole plug.

As the study progressed, additional grout mixtures were added which typify the best mixtures currently available for the southwest New Mexico area - Class C (SR) cement. Both laboratory and field samples are included in the exposure studies to extend the data base for future dec ons whenever a borehole is to be permanently plugged.

To set this study in the proper perspective, the following is quoted from Bryant Mather, Chief of the Concrete Laboratory at WES:[13] "Concrete is a construction material that is made by mixing to a degree of homogeneity ingredients having an infinite variety of properties of widely varying significance, proportioned in accordance with a recipe selected according to poorly defined rules." The same idea is applicable to grouts in the cementing industry. Even with the large amount of laboratory research including techniques to simulate conditions underground, the present knowledge of actual behavior in situ is still far from complete. The goal of the Borehole Plugging Materials Development Program is to

1. Combine an extensive laboratory program with field supervision and quality control of grouting operations

2. Continue study of representative field samples of grout mixtures including samples prepared at the surface and, when possible, cores of the downhole plugs.

III. Materials Development Program at WES

The initial phase of the work at WES consisted of proportioning several hundred grout mixtures with varying combinations of materials. Included in these preliminary mixtures were cements, fly ash and natural pozzolans, gel, sand, salt, super plasticizers, retarders, and water. This effort relied on the experiences of the personnel in the grouting branch at WES to reduce the many possible combinations down to a practical number which could be selected for preparation and testing. The initial proportioning included both calculations of mixture weights and mixing of fluid samples to check viscosity (potential pumpability) with a setting time of up to 3 hours, and for general appearance.

The 59 mixtures listed in Tables 1, 2, and 9 of Reference 2 were developed to systematically look at differing amounts of each material in a mixture. Table 1 (Reference 2) lists the weight of each material, density, cement content, and water/cement ratio for comparison. Table 2 (Reference 2) lists the freshly mixed and hardened data. The behavior in the freshly mixed state--viscosity and time of initial and final set--provided data for selection of mixtures for further testing. The strength data at the standard 28-days age were compared with a reasonable lower limit of 20.7 MPa (3000 psi) unconfined compressive strength to further narrow the selection of mixtures. This strength level is comparable to the expected upper level of in situ conditions for plugging holes at the WIPP site. Table 9 (Reference 2) lists the replacement of cement with fly ash and other pozzolans for comparison within this group and with data available from previous studies at WES and elsewhere.

As soon as most of these data were available, the mixtures for long-term durability and comprehensive testing of physical properties were selected. The mixture proportions of the five grouts selected are shown in the first five columns of Table I.

The first four mixtures have Type K shrinkage-compensated sulfate-resistance ChemComp as the basic cement. The fifth mixture has the normal Class C (SR) (Type III) portland cement. Mixtures 3 and 4 include the more highly expansive ChemStress cement to study the effect of self-stressing levels of expansion. All five mixtures have 23% to 30% by volume fly ash replacement (19% to 25% by weight). Mixture 4 includes the natural pozzolan (a calcined tuff) to study the effects of a grout with the maximum practical content of pozzolan (fly ash plus natural pozzolan). The fly-ash/pozzolan replacement is 55% by volume.

TABLE I

Grout Mixture Proportions and Strengths

	Metric Units	1	2	3	4	5	English Units	1	2	3	4	5
		BPN-FA-SP-P	BPN-FA-BS-SP-P-1	BPN-CS-FA-1	BP-521 25MP	BPN-FA-BS-SP-P-1 (Type III)						
Class C (Type III) Cement	kg/m^3	-	-	-		884	lb/ft^3	-	-	-	-	55.21
ChemComp Cement	kg/m^3	979	884	993	697	-	lb/ft^3	61.12	55.21	62.02	43.54	
ChemStress Cement	kg/m^3	-		144	144	-	lb/ft^3		-	9.00	9.00	-
Fly Ash	kg/m^3	329	298	268	199	298	lb/ft^3	20.56	18.58	16.76	12.40	18.58
TUFA (pozzolan)	kg/m^3	-	-	-	158	-	lb/ft^3	-	-		9.84	
Fine Salt (dissolved)	kg/m^3	-	183	-		183	lb/ft^3	-	11.43		-	11.43
Melment-L10	kg/m^3	26	24	28	34	24	lb/ft^3	1.63	1.48	1.72	2.10	1.48
Plastiment	kg/m^3	2.6	2.9	3.0	2.8	2.9	oz/ft^3	(2.60)	(2.94)	(3.02)	(2.76)	(2.94)
Water	kg/m^3	550	508	523	579	508	lb/ft^3	34.31	31.73	32.66	36.14	31.73
Density	gm/cm^3	1.86	1.87	1.93	1.78	1.87	lb/ft^3	116.0	116.9	120.4	110.9	116.9
							lb/gal	15.5	15.6	16.1	14.8	15.6
Cement Factor All Cements	kg/m^3	979	883	1137	841	884	lb/yd^3	1650	1491	1918	1419	1491
Cement + Fly Ash	kg/m^3	1308	1182	1405	1040	1182	lb/yd^3	2205	1992	2370	1753	1992
Fly Ash Replacement	% Vol	0.30	0.30	0.23	0.23	0.30	% wt	0.25	0.25	0.19	0.19	0.25
Water/Cement Ratio Water/Cement		0.56	0.57	0.46	0.69	0.57		0.56	0.57	0.46	0.69	0.57
Water/Cement + Fly Ash		0.42	0.43	0.37	0.56	0.43		0.42	0.43	0.37	0.56	0.43
28-Day Unconfined Compressive Strength	MPa	52.2	37.1	61.9	41.0	34.9	psi	7570	5380	8980	5940	5060

Mixtures 2 and 5 used saturated brine mixing water. Mixture 2 is a direct comparison with Mixture 1 to study the effect of saturated-salt grouts which may be required through the salt sections of borehole plugs. The effect on strength is notable for the same water/cement ratio. The reduction in the unconfined compressive strength from 52.2 MPa to 37.1 MPa (7570 psi to 5380 psi or 29%) is probably more severe than the reduction in cement content of 10 percent for Mixture 2 compared to Mixture 1 with fresh water.

All five mixtures have the superplasticizer Melment L-10 included to reduce the water content to the minimum while still maintaining adequate pumpability. This is expected to improve strength, bonding, and durability, and to reduce permeability. The addition of the retarder, Plastiment, maintains the pumpability (for these high-cement-content grouts) for the required 3 hours.

Mixture 3 had the highest fluid density of all the grouts without salt or sand in the mixture. It also had the highest 28-day unconfined compressive strength. The highest cement content and lowest water/cement ratio accounts for this very high strength which is not normally found in pumpable mixtures.

A comparison of ChemComp cement with Class C (SR) (Type III) cement in brine grouts can be seen in Mixtures 2 and 5. With all weights of materials being equal, the strength for Mixture 2 is about 6% greater.

Some of the specimens of each of the five mixtures have been submerged in water with a chemical content similar to the underground water samples of the WIPP site. The first criteria and method of preparation for the simulated groundwater are listed in Table 8 (Reference 2). The criteria was revised as listed in Table 10 (Reference 2), to simulated groundwater for the exposure and permeability testing after the first year of exposure for the current specimens.

The remainder of this section will discuss the data in Tables 3 through 7 (Reference 2). All of the data and observations of the specimens confirm the excellent quality of the five selected laboratory mixtures. There is no apparent deterioration of any specimen either under the exposure to simulated groundwater or protected to prevent moisture loss in the laboratory environment.

Table 3 (Reference 2) lists dynamic modulus and compressional-wave velocities for 7.6-cm-diam x 50.8-cm-long cylinders (3 x 20 in.). Even with the elevated curing temperature of 49°C (120°F) for 28 days, the initial representative values for mixtures with 30% or higher pozzolan content should be the 56-day age values. Mixture 3 consistently has the highest unconfined compressive strength, the highest dynamic modulus, and the highest compressional-wave velocity. Conversely, Mixture 5 has the lowest values. The other three mixtures have consistent numbers based within that range consistent with the relative strengths. The variation in dynamic modulus from 56 days to one year for the same exposure condition is less than 5% and generally has a slight increasing trend as expected for the continuing normal hydration of the cement content of the grout.

The highest value is 23.6 x 10^3 MPa (3.4 x 10^6 psi) for Mixture 3 with the highest strength. The lowest values are about 13.8 x 10^3 MPa (2.0 x 10^6 psi) for the two lowest strength mixtures, 2 and 5. These are the two mixtures with saturated-salt mix water.

The compressional-wave velocity data show a similar pattern of the highest values for Mixture 3. The variation with time for each mixture is less than 5% and does not show an increasing trend for all mixtures at later ages. This variation is about the expected accuracy of the soniscope for the 51-cm (20 in.)-long specimens.

The resonant-frequency method for dynamic modulus and the soniscope method for compressional-wave velocity are nondestructive tests. The same specimens will continue to be tested at 1-year intervals to monitor improvement or deterioration. There are three specimens for each mixture submerged in the simulated ground water. Unpublished weight-change data (which will be included in the next WES report) show the expected slight gain in weight during the first year of about 5% to 8% for Mixture 1 and less than 4% for the other four mixtures. The dry specimens were covered with a plastic coating at demolding and encased in double-plastic bags to prevent moisture loss. The weight loss is less than 1% for these specimens, most of which probably occurs when they are removed from the plastic bags for periodic measurements.

All of the above data indicate good quality grout with no evidence of deterioration during the first year of exposure to the simulated groundwater.

The bond-strength data in Table 4 (Reference 2) was intended to provide a preliminary indication of bonding characteristics for each of the mixtures. The test of bonding to a smooth steel surface is probably more severe than the bonding to rock with irregular surfaces. The values of bond strength which range between 5.9 and 1.7 MPa (855 and 250 psi) show a good tight interface. The successive cutting of specimens from the grout-filled pipe and drying of the grout can explain the lack of definite trends. The current bond studies are concentrating on bonding to the formation materials.

In Reference 3, bond strengths of grouts to sandstone, salt, and anhydrite were determined. Drilling-mud contamination of the surface and flushing with various agents preceded the grouting of the 3.3-cm (1.3 in.)-diam holes in formation cores slightly less than 10.2 cm (4 in.) in diameter. Values of bond strength to sandstone were 2.8 MPa to about 8.45 MPa (400 to 1225 psi). Some of the five study grout mixtures may show higher values of bonding to the stronger formation rocks because of surface roughness and irregularities compared to smooth steel pipe. Cleaning the drilling mud from formation surfaces will be a key factor in achieving good bond.

The volume-change data in Table 5 (Reference 2) is a measurement of the length change of unrestrained 0.075- x 0.075- x 0.25-m (3- x 3- x 10-in.) prisms. The set of specimens coated with a protective "paint-on" skin and stored in double-plastic bags at 24-hour age still show a "drying out" length reduction for all five mixtures. Mixture 3 with ChemStress expansive cement showed a peak expansion at 14-day age and had the least length reduction of all five mixtures. The other mixture with ChemStress and with the additional natural pozzolan (Mixture 4) reached a lower peak expansion of 6 days and showed the next lowest length reduction through 6 months. Mixture 5 with brine mixing water and the normal Class C (SR) cement showed a slight expansion through 3 days and a slightly greater length reduction through 6 months than Mixture 4. Mixtures 1 and 2 showed no expansion and about 50% greater length reductions than the other three mixtures through 6-months age. The data for Mixtures 1 and 2 show a continuing length reduction through 12 months of data.

The second set of specimens which had the initial length reading after remaining in the steel molds for 28 days were then submerged in the simulated groundwater. Mixture 4 kept its initial length through 6 months. Mixture 2 (with brine mixing water) showed the greatest expansion for the submerged specimens. All of the expansions stabilized by 6 months and have maintained the length through 12 months. There is no apparent deterioration of the surface of these submerged samples. The effect of the expansion on bond strength will be evaluated during the bond-to-formation testing since a definitive trend for the bond-to-steel pipe specimens is not apparent at this time.

Boa concluded that the data on porosity may be misleading since Mixture 5 had the lowest porosity and was the only mixture which had a measurable permeability for flow through the specimen. All mixtures had water flow initially into the specimens. Mixtures 1 through 4 showed permeability rates of less than 0.0002 md during the initial flow of water into the specimens through about 1 to 2 weeks time. The data for Mixture 5 in Table 7 (Reference 2) shows permeabilities of 0.0006 md and less for the four specimens. The effective porosity is receiving additional study. Gas permeability testing of samples has recently been initiated; also porosity determinations by grinding of specimens to a fine grain size and comparison with the pressure pycnometer data in Table 6 (Reference 2) are underway. These studies will receive more emphasis during the current year and will be discussed further in the next report.

IV. Long-Term Development Program

The studies at WES and numerous discussions with individuals have generated ideas for the long-term materials-development program. Part V (Reference 2) lists many of the ideas which have been discussed and seem to be practicable. They will be considered during each periodic review of work in progress and in planning for the program.

At the present time, work is underway or planned during FY78 for the following areas listed on pages 13 through 16 of Reference 2: a, b, c, d, e, i, j, k, m, n, o, p, q, r, t, v, x, y, z, aa, bb, and cc. The items to be initiated during FY79 are f, g, l, s, u, and w. The idea of possible use of chemical matching grout (item h) is not being actively pursued under this program. However, WES maintains an up-to-date knowledge of chemical grouts available throughout the world for their many other R&D programs. New products with properties which seem advantageous will be discussed at periodic review meetings.

This report described the background and some of the thinking and discussions which generated the work underway, the data available,[2] and a list of activities proposed for initiation during the next year and a half.

An important part of the overall program for any borehole plugging project is the quality control program. Personnel from the grouting branch of WES will participate in all field activities and supervise the operations of the cementing contractor. Quality control will also involve preliminary inspections of the equipment to be used at the job site and in the batch plant. Samples of all grouting will be obtained in addition to samples of the blended materials before mixing. Samples of the mixed grouts will be cast and tested at the job-site field laboratory for rapid evaluation. Additional samples will be shipped to the WES Lab and will be included in the long-term durability studies. Physical properties of the field samples will be evaluated for a data base of field materials compared with laboratory-prepared specimens.

The first field-learning activity took place in October 1977, during the plugging of ERDA 10 borehole. Data from the studies of these field samples continue to be generated. Samples cast at the surface of the three different grout mixtures placed in a total of four plugs, cores from hardened grout in the bottom plug, and return grout during the placement of the third plug were obtained. These studies are considered a part of the development program and are included in the long-term-durability testing. A report of the data from these field samples and the significance related to the development program is in preparation.

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