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MINES BRANCH INVESTIGATION REPORT IR 58-166

HIGH DENSITY CONCRETE WITH ILMENITE AGGREGATE

by

V. A. HAW and N. G. ZOLDNERS

INDUSTRIAL MINERALS DIVISION

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HIGH-DENSITY CONCRETE WITH ILMENITE AGGREGATES

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V.A. Haw* and N.G. Zoldners**

SUMMARY OF RESULTS

Tests were performed on ilmenite to investigate its potential value as an aggregate to be used in high-density structural concrete designed for nuclear reactor shields.

A series of concrete trial mixes were made to determine the best aggregate proportions, cement factor, and the best suitable admixture to be used for a workable and plastic concrete mixture to produce concrete of maximum density and adequate strength.

A good workable concrete mixture with 7 bags of cement per cubic yard of concrete and graded all-ilmenite aggregate was prepared with addition of a densifying agent. Concrete test specimens moist cured for 28 days showed an average of 5770 psi compressive and 782 psi flexural strength at a density of 240 lb/cu ft.

Investigations on 14 beams, 3 cylinders, and a slab were conducted to determine the physical properties of this type of concrete.

Thermal expansion and shrinkage characteristics were found to be within the normal range of a good quality concrete.

Wetting and drying tests have shown no evidence of reactions leading to destructive expansion of the ilmenite concrete within test limits.

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* Head, Construction Materials Section
** Senior Scientific Officer

Industrial Minerals Division, Mines Branch,
Department of Mines and Technical Surveys,
Ottawa, Canada.

CONTENTS

INTRODUCTION

The work described in the following report continues the investigations into the use of ilmenite aggregate for high density concrete. A previous report, I.M. Rept. No. 481, outlined the purpose of the investigation and contained the results of work on the properties of the ilmenite aggregate. Some preliminary work was also done on proportioning of concrete to determine strengths and densities obtainable.

It remained to complete proportioning studies of the concrete, particularly with reference to moulding, or placing methods, and to the use of additives for the purpose of improving the quality of the plastic concrete and increasing the density. The properties of the hardened concrete also remained to be investigated, particularly those related to its use in nuclear reactor shields where autogenous and other volume changes might result in serious damage. These investigations have now been completed to a point where a report can be prepared which can be of use to the firm producing the ilmenite aggregate and to others interested in the use of high density concrete.

The Industrial Minerals Division has been co-operating with the firm of Ensio, Whiton and Associates, Consulting Engineers, on the work outlined above. This firm is associated with Continental Iron and Titanium Mining Limited, Montreal, in providing the ilmenite aggregate for use in biological shields for nuclear reactors.

ILMENITE AGGREGATES

The 1 1/2 ton sample of crushed ilmenite, received here from Continental Iron and Titanium Mining Limited, Montreal, consisted of 2 lots - coarse aggregates with a maximum size up to 2 in, and sand minus 10 mesh.

Following are the results obtained on physical properties of this material.

(1) Unit Weight (ASTM Design. C 29-55T)

The jigging procedure was used to determine the unit weight of graded coarse and fine aggregates.

	Unit Weight
Coarse aggregates (-2 in. + No. 4 M)	186 lb/cu ft
Fine aggregates (-No. 4 M)	180 lb/cu ft

(2) Specific Gravity (ASTM Design. C 127 & 128-42)

	Coarse	Fine
Bulk specific gravity, oven dry	4.63	4.65
Bulk specific gravity, saturated, surface dry	4.65	4.67

(3) Absorption (ASTM Design. C 127 & 128-42)

	Absorption
Coarse aggregate (-2in. + No. 4 M)	0.27%
Fine aggregate (-No. 4 M)	0.50%

(4) Sieve Analysis (ASTM Design. C 33-55T)

All material was separated by screening into sized fractions which were recombined together in proportions to meet the selected median values given in table 1 from the ASTM standard grading limits.

TABLE 1

Gradings

Sieve No	Coarse Aggregates: -3/4 in. Passing %	Coarse Aggregates: -2 in. Passing %	Fine Aggregates: Sieve No	Fine Aggregates: Sand Passing %
2 in.	-	100	4	100
1 1/2 in.	-	81	8	90
1 in.	-	52 1/2	16	67 1/2
3/4 in.	100	34	30	42 1/2
1/2 in.	56	20	50	20
3/8 in.	37 1/2	12 1/2	100	6
No 4 M	0	0		

TRIAL MIXES

To establish the optimum ratio of fine to coarse aggregates as well as the proper cement factor to produce a plastic and good workable concrete of the highest density, three series of trial mixes were made, shown in table 2.

TABLE 2

Concrete Proportions

	Maximum Aggregate Size	F:C Aggregate Ratio	Cement Factor Bg/cu yd	Concrete Slump
A Series	3/4 in.	35:65%	6 bags	1 in.
B Series	3/4 in.	40:60%	7 1/2 bags	2 in.
C Series	2 in.	35:65%	7 1/2 bags	2 in.

In each series, three mixes were compared; one plain, one with Pozzolith, and one with Plastiment.

The size of batches were designed to make a slump test, unit weight, air content, and to mould two standard test cylinders 6 x 12 in. for 28-day compressive strength tests.

The unit weight of fresh concrete was determined in the airmeter container (vol. 0.25 cu ft) by placing concrete mixture in three layers and consolidating the two first layers for 10 seconds each and the top layer for 20 seconds on a vibrating table.

To determine the amount of air in the fresh concrete mixture, a Techcote airmeter was used.

Concrete test cylinders (6 x 12 in.) were moulded in three layers using the vibrating table. It was found that the same concrete mixture moulded by vibration produced concrete with a density 3 to 4% higher than by standard rodding procedure.

The density of hardened concrete in saturated, surface-dry condition at 28-day age was measured by the immersion method, using the standard test cylinder before it was capped for compressive strength test. The same cylinder, still in saturated, surface-dry condition was used to determine the pulse velocity in the hardened concrete by an ultrasonic concrete tester.

All mix data and test results are shown in table 7.

Series A Mixes were harsh with high bleeding and unworkable, except Mix No 55, made with Pozzolith, which introduced some air entrainment into the mixture, making it more plastic than the other two.

Series B Mixes were improved by increasing the sand portion from 35 to 40% of the total aggregates, at the same time increasing the amount of cement from 6.1 to 7.3 bags per cu yd.

In heavy concrete mixes, cement has a dual function -- it is not only the strength element, but it is needed in the fresh concrete to float the heavy aggregate particles and to provide workability and cohesion to the mix.

The changes made for the C series of mixes reduced segregation and improved the workability greatly, but simultaneously reduced the density of plain and Pozzolith concretes.

Series C Mixes were made with 2 inch maximum size of aggregates and 35% sand by volume of the aggregate. These mixes produced concrete with a density of 243 lb per cu ft of hardened concrete in saturated, surface-dry condition at 28-day age.

CONCRETE DENSITY

The results of our trial mixes show that concrete using an all-ilmenite aggregate up to 3/4 in. maximum size, can be produced with a density of 241 lb/cu ft after 28 days of moist curing.

This is a higher density than any hitherto reported in the literature for natural aggregate without any addition of iron or steel slugs. Some figures for other types of aggregates which have been reported are shown in table 4.

TABLE 3

TESTS		CONCRETE MIX DATA									TESTS ON HARDENED CONCRETE					
		Batch Weights per 1 Cu Yd					Mix Characteristics					7-d. age		28-d. age		
			SSD* AGGR.													
Ser.	No	Cem. lb	Fine lb	Cse lb	Wat lb	Admixt. type	W/C Ratio	Slump in.	Air %	Unit wt. lb/cu ft	Cem fact. bag cu yd	Comp. str. psi	Pulse veloc ft/sec	Comp. str. psi	Pulse veloc. ft/sec	Density of SSD conc. lb/cu f
			35%	65%												
A	54	534	1987	3673	326	Nil	0.61	1.0	1.0	241.6	6.1	2500	14950	3810	15975	242.0
	55	534	1991	3680	295	Pozzol. 3 lb	0.55	1.2	1.6	241.0	6.1	3750	14990	5290	15700	237.0
	56	531	1986	3680	308	Plastim. 30 oz	0.58	1.0	1.1	240.8	6.1	4100	15540	5575	16180	241.0
			40%	60%												
B	57	641	2245	3207	327	Nil	0.51	2.0	1.0	238.0	7.3	3890	14780	5720	15925	239.6
	58	641	2185	3154	300	Pozzol. $3\frac{3}{4}$ lb	0.47	2.0	2.6	232.8	7.3	4550	14755	5415	15325	234.0
	59	650	2280	3285	335	Plastim. 30 oz	0.52	3.5	0.5	242.8	7.5	4830	15045	6740	16180	241.0
			35%	65%												
C	60	655	1963	3600	382	Nil	0.43	2.0	1.2	241.0	7.5	4660	14360	5290	16490	243.0
	61	639	1917	3534	270	Pozzol. $3\frac{3}{4}$ lb	0.42	3.5	3.2	235.8	7.3	4455	13740	5510	16880	238.0
	62	659	1979	3640	282	Plastim. 30 oz	0.43	2.0	0.5	243.0	7.5	5040	13665	6320	17950	243.0

* SSD - Saturated surface dry

TABLE 4

Concrete Densities Using Heavy Aggregates

Aggregate			
Coarse	Fine	Density lb/cu ft	Placement Method
Barite	Barite (3)*	215	Conventional
Magnetite	Magnetite (3)	228	Conventional
Limonite	Limonite (4)	185	Conventional
Barite	Barite (4)	227	Prepacked
Magnetite	Magnetite (4)	232	Prepacked
Magnetite	Limonite (5)	205	Conventional
Barite	Barite (6)	233.8	Conventional
Barite	Barite (6)	230.3	Prepacked

* The numbers in brackets refer to the references on page 20.

The figures in table 4 are based mainly on the use of 1 1/2 in. aggregate.

It is of course to be expected that higher densities are to be obtained when using the maximum amount of aggregate, keeping the combined cement and water to a minimum. A higher coarse to fine aggregate ratio is also beneficial. However, other requirements limit the proportions of aggregate, cement and water which may be used. These requirements are - workability of the mix, the rate of bleeding, and finally, strength characteristics.

Our trial mixes show that a good workable and plastic concrete mixture with a minimum amount of bleeding required at least 7 bags of cement per cubic yard. The minimum amount of sand in the mix was 40% for 3/4 in. and 35% by volume for 2 in. maximum aggregate size.

Other features which have been found important in obtaining increased densities are:

Maximum size of aggregate - by increasing the size from 3/4 to 2 inches, density increased by 2 to 3 lb/cu ft;

Placement by vibration was found to increase the density by 3 to 4% as compared to moulding of concrete in forms by rodding;

Addition of a plasticizing densifier was found to give better results, and also improved the quality of the plastic concrete mix (Plastiment, a Sika Chemicals Corporation product, was used with success in our trial mixes).

To carry out a complete investigation on the physical properties of ilmenite concrete, test specimens in the form of beams and cylinders were prepared.

The right proportions for a suitable test mix of highest density and good workability were found from the trial mixes made before with all-ilmenite aggregate up to 3/4 in. maximum size.

The proportions for 1 cu yd of the test mix concrete are as following:

Cement, 7 bags	- 612 lb
Ilmenite Sand (Saturated, surface dry)	- 2230 lb
Ilmenite C.A. - 3/4 in. (" ")	- 3324 lb
Water (for 2 in. slump)	- 324 lb
Batch weight per 1 cu yd	- 6490 lb

Unit weight of fresh concrete - 240.2 lb/cu ft

Plastiment in solution - 18 oz per cu yd

Two batches of concrete were made of the same proportion and two series of concrete test specimens were prepared as follows:

No. 90 - 11 beams - 3 1/2 x 4 x 16 in.
No. 96 - 3 beams - 3 1/2 x 4 x 16 in.
3 cylinders 4 x 8 in.
1 slab 3 x 13 x 17 1/2 in.

PROPERTIES OF ILMENITE CONCRETE

Using the concrete specimens prepared, a number of physical tests were conducted to determine strength and thermal properties, and the volume stability under varying conditions of humidity and temperature of the ilmenite concrete.

Strength Properties

From the test mix No. 96 three cylinders, 4 x 8 in., and three beams, 3 1/2 x 4 x 16 in., after 28 day standard moist curing were used for compression and flexural strength determination. The results obtained as average from three samples are as follows:

Compressive strength - 5770 psi
Flexural strength - 782 psi

Thermal Coefficient of Expansion

The thermal coefficient of expansion was determined using 3 1/2 x 4 x 16 in. beams in a saturated condition after 28 days moist curing. A Whittemore strain gauge with a dial reading to .0001 in. was used to measure the change in lengths. The readings were taken on a 10 in. gauge length measured between two stainless steel reference plugs which were placed flush to the surface of the concrete. Two beams were placed in a constant temperature water bath, and readings were taken at 24-hr intervals between a temperature range of 75 and 170°F. The beams were given four cycles of heating and cooling. The coefficients of expansion as determined for the two beams varied between 3.94×10^{-6} and 4.60×10^{-6} in/in/°F, with an average of four readings being 4.28×10^{-6} in/in/°F. As a basis of comparison, linear coefficients of expansion in table 5 have been reported for other types of concrete in the literature.

TABLE 5

Coefficients of Expansion of Heavy Concrete

Aggregate	Linear Coefficient of Expansion, in/in/°F
Magnetite (7)	4.8 to 5.1×10^{-6}
Barite (8)	9.8×10^{-6}
Iron-limonite (9)	10.1×10^{-6}
Magnetite-limonite (9)	$8.6 \times 10.1 \times 10^{-6}$
Gravel and stone (10)	$3.5 \times 6.5 \times 10^{-6}$

Moisture Movement and Initial Drying Shrinkage

The determination of moisture movement and initial drying shrinkage is a determination of volume changes in concrete when first subjected to drying under controlled conditions followed by saturation. The test is given in British standards 1881:52 and provides a measure of comparison with concrete of good quality.

The tests were conducted on two 3 1/2 x 4 x 16 in. beams which had been removed from moulds 24 hr after being made and then cured in water for a further 27 days. Measurements were taken at this point between stainless steel reference plugs on two opposite sides of the beams at a gauge length of 10 in., which is the wet measurement. The beams after being removed from the water were placed in an oven at 50°C which contained a saturated solution of $CaCl_2$ to give a relative humidity of 17% for a period of 44 hr. The beams were then cooled in a container with dry $CaCl_2$ for a period of four hours, after which they were again measured. The drying procedure was repeated until the difference between two consecutive measurements was less than .0002 in. per six inches of length. The difference between the final dry measurement and wet measurement is known as the initial shrinkage.

The difference is expressed as a percentage of the gauge length of 10 in.

For the determination of the moisture movement, the dried specimens were submerged in water for four days, after which they were removed and measured. The difference between the final wet measurement and dry measurement is the moisture movement.

Test Results

Eight cycles of drying and cooling were completed before the drying shrinkage remained constant. Measurements were taken on both sides of each beam and averaged.

Initial drying shrinkage	-	.034%
After four days immersion moisture movement	-	.027%

In the new revised edition of "The Chemistry of Cement and Concrete" by Lea and Desch, concrete shrinkages of .02 to over 0.10% are reported depending on the mix proportions. The lower figures are for low water/cement ratios and low cement factors. These figures are for concretes with conventional type aggregates. For concretes with about the same water/cement ratio and cement to aggregate proportions by volume as the ilmenite concrete, the drying shrinkage runs between .04 and .06%, and the moisture movement about .03%. It will be observed, therefore, the drying shrinkage of .034% and moisture movement of .027% obtained in this work compares favourably with similar volume changes for good quality conventional type concrete.

Drying Shrinkage and Loss of Moisture

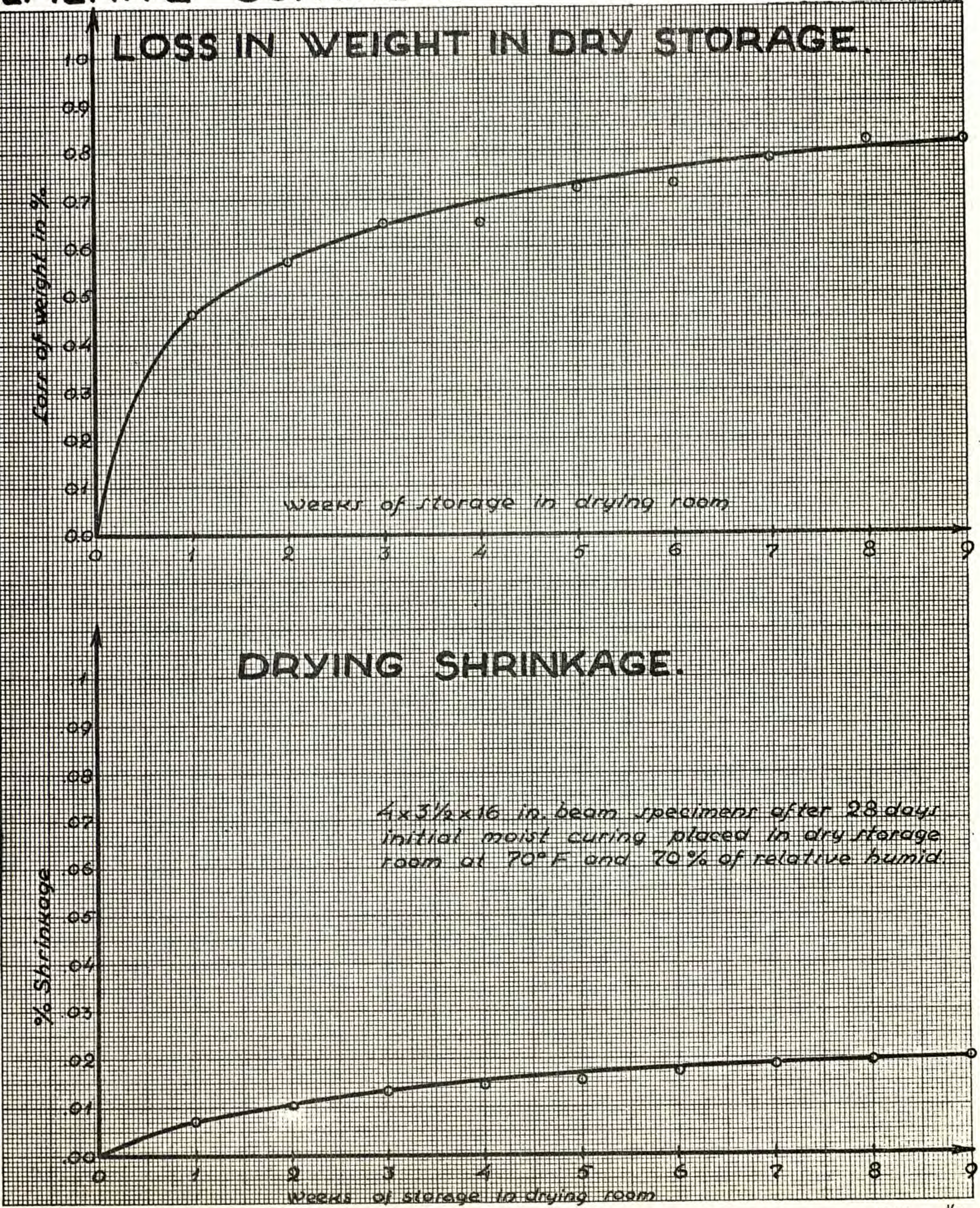
In addition to the British Standards test for initial drying shrinkage, two 3 1/2 x 4 x 16 in. beams were subjected to normal room conditions of temperature and humidity for determination of drying shrinkage and moisture loss. The results of changes in these properties are shown in figure 1. The somewhat irregularly distributed points for moisture loss reflect variations in the relative humidity of the storage room.

Volume Change Due to Wetting and Drying

The 3 1/2 x 4 x 16 in. beams were subjected to alternate wetting and drying with variations of temperature to detect potential reactions in the concrete which might result in abnormal expansion. The wetting and drying procedure has been used in the past as an accelerated method to determine some varieties of cement-aggregate reactions leading to abnormal expansion.

ILMENITE CONCRETE

FIG.1.



HUGHES OWENS 315L -METRIC

1958

This is apart from the alkali-aggregate reaction for which a standard and different type of test is available. The agency which results in destructive expansion of concrete when exposed to wetting and drying has not been definitely established, although C.H. Scholer(1) has suggested that a combination of moisture and temperature change frequently repeated is conducive to distress in any non-homogeneous absorptive material. The distress is caused by:

1. Acceleration of chemical reaction and resulting crystal growth.

2. Weakness of bond between aggregate and paste will develop failure.

3. Lack of thermal compatibility is accentuated.

4. Repetition of (1) and (3) results in a jacking action which develops increasing expansion.

A.D. Conrow(2) has also shown that abnormal expansion of concrete occurs which cannot be related to alkali-aggregate reaction. Test procedures have been described by both Scholer and Conrow designed to detect certain combinations of aggregate and cement which could result in undue expansion with accompanying loss of durability of the concrete.

As concrete used in nuclear shields is exposed to elevated temperatures and possibly to variations in moisture content, it appeared advisable to subject the ilmenite concrete to some type of wetting and drying test involving elevated temperatures. Further, it seemed desirable to use a test which has proved successful in the past and for which some comparable results would be available. It was, therefore, decided to use both the Scholer and Conrow tests, and these are described below.

Scholer Test

Two beams were placed in the moist room for three days after moulding and then immersed in water for a period of 28 days after moulding. After removal from the water at 28 days, the beams were measured and placed in an oven at 130°F for a period of 8 hr and then placed in water at 70°F for 16 hr. This cycle has been continued up until the writing of this report (about two months) and will continue for a period of 12 months. The beams are left immersed in water over week ends. Measurements are taken every 10 cycles. The results of this test are shown in figure 2. At 50 cycles the beams had expanded an average of .016%.

Conrow Test

Two beams were given the same initial curing as for the Scholer test after which they were subjected to the following heat treatment:

(a) placed in water at 130°F for 7 days.

(b) cooled in water for 24 hr at 70°F.

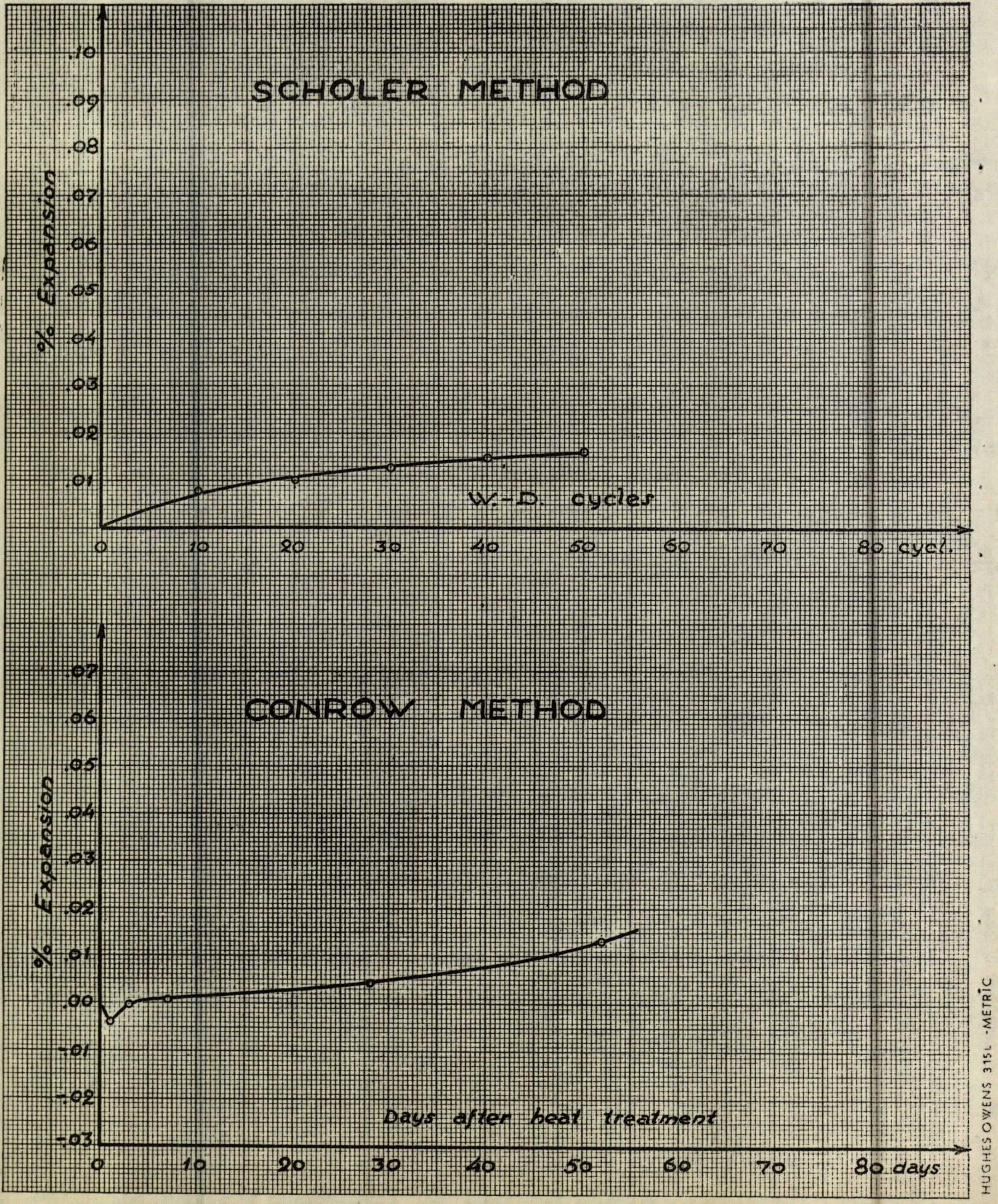
(c) oven dried at 130°F for 7 days

(d) cooled in air at 70°F for 24 hr.

(e) placed in water for indefinite storage at 70°F.

The length measurement at 28 days (after initial curing) is the base length. Measurements were made after each of the steps from (a) to (d) and the results are shown in table 6 as percent length increase of the base length.

WETTING AND DRYING TESTS. FIG. 2.



Length changes in step (e) during the water storage were measured at 24 hr, 3 days, 7 days, and thereafter at every 28 days. The length increase, expressed as percent expansion of the base length at 28 days age is shown in figure 2.

TABLE 6

Conrow Test

Temperature and Moisture Condition	Length Increase
(a) Percent length increase after 7 days in water at 130°F	.029%
(b) After cooling in water for 24 hr at 70°F	.005%
(c) Oven dried at 130°F for 7 days	.023%
(d) Cooled in air for 24 hr at 70°F	-.010%

The volume changes resulting from changes in temperature and moisture as indicated from the length changes shown in table 6 are about what would be expected for good quality concrete. After 52 days of continuous water storage at 70°F, the volume expansion amounted to .013%.

Consideration of the results of the two wetting and drying tests indicates that no evidence of reactions leading to destructive expansion of the ilmenite concrete has appeared to this date. For the Scholer test, the Bureau of Reclamation has placed a figure of .07% expansion after 300 cycles as being a safe limit for good quality concrete. For the Conrow test we have no information on acceptable limits for expansion, but reference back to the original paper by Conrow indicates values obtained for poor cement-aggregate combinations. For type 1 cements (as used here), expansions of over 0.6% were obtained at 300 days, the range being 0.1 to over 0.6%. At 50 days expansions varied from .05 up to as high as 0.30%. However, where the reaction was inhibited by pozzolanic materials, the expansions were reduced to less than 0.10% at 300 days.

Thermal Conductivity

The thermal conductivities at the mean temperatures reported below were determined by J.G. Brady of the Ceramic Section. The equipment employed was a high temperature thermal conductivity apparatus of the type used for testing full-sized refractory bricks or castable refractory slabs. Essentially it involves a high temperature electric furnace, a copper calorimeter and guards, and suitable control equipment. This apparatus conforms to the details outlined in ASTM Designation C 201-47 - Standard Method of Test for Thermal Conductivity of Refractories.

TABLE 7

Thermal Conductivity of Ilmenite Concrete

Mean Temp °F	K btu/sq ft/hr/°F/in
113	12.9
197	13.4
498	14.9

CONCLUSIONS

A concrete density of 240 lb/cu ft can be obtained using 3/4 in. all-ilmenite aggregate by conventional placement with vibration, provided that correct mix proportions and a plasticizing additive are used.

Strength properties of this concrete proved to meet the usual specifications for structural concrete in reactor shields.

The investigations conducted into volume changes resulting from variations in temperature and moisture indicate the ilmenite concrete to be volume stable to a high degree compared to other Portland cement concretes. The thermal coefficient of expansion is in the low range, and both drying shrinkage and moisture movement are within the range for good quality concrete. The wetting and drying tests are still under way and will be for another ten months. So far, no cement aggregate reactions have been detected which would lead to abnormal expansion.

Finally, the results of this investigation show that the ilmenite from the St. Urbain, Quebec, area supplied by the Continental Iron and Titanium Mining Limited is superior to other natural aggregates on record for production of high density concrete. Concrete density is higher, strengths are more than adequate, and volume stability is comparable to the best quality concrete.

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