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National Bureau of Standards
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This report includes test results conducted at ambient (295 K), dry-ice and alcohol (195 K), liquid nitrogen (76 K), and liquid helium (4 K) temperatures. The compressive properties reported are Young's modulus, yield (at 0.2% offset) and maximum strengths, and elongation (elastic and plastic). Test specimens (5.1 cm diameter x 10.2 cm) were instrumented with a specially designed, diametrically opposed, cryogenic strain-gaged extensometer that minimizes possible errors due to specimen bending during the test.

Key words: Compressive strength; concrete mortar; elongation; low temperature; maximum strength; mechanical properties; yield strength; Young's modulus.

1. Introduction

Concretes are attractive materials for construction of large low-temperature installations because of lower material cost and economies of fabrication compared to alternative construction methods. Furthermore, they exhibit favorable mechanical and thermal properties for use in low-temperature applications. Although these materials are presently used to some extent in LNG and LPG construction, lack of confidence in their cryogenic behavior has restricted their use or has resulted in overly conservative and expensive designs. This reflects the lack of dependable cryogenic thermal and mechanical properties data and a poor understanding of the effect of field-fabrication variables on cryogenic performance.

Potential LNG- or LPG-oriented applications of concretes are diverse. Concrete is currently used for barge hulls and for ground-based storage facilities [1,2]. Proposals and designs for construction of large seagoing tankers and floating terminals, in which both the hull and the primary LNG containment systems are made of concrete, are also being considered. Possible applications of special concretes such as lightweight, cellular, fiber reinforced, and polymer will depend on their cryogenic properties which are not known at this time.

The research described below is part of a broader program to determine the thermal and mechanical properties of aggregate composites. The mechanical properties of two concrete mortars were determined at ambient (295 K), dry-ice and alcohol (195 K), liquid nitrogen (76 K), and liquid helium (4 K) temperatures. The properties reported here are compressive Young's modulus, yield strength (at 0.2% offset), maximum, ultimate, flexural, and splitting strengths, K_{Ic} , and elastic and plastic elongations.

2. Materials Characterization

The physical properties of two concrete mortars (designated mix 1 and mix 2) were studied in this program. Mix 1 was made by a commercial-cement company and mix 2 was made at the National Bureau of Standards. Both mixes utilized portland type I cement and a resin air-entraining agent. The aggregate used in mix 1 is commonly known as Elgin sand and that used in mix 2 is commonly referred to as Clear Creek sand. The chemical makeup and sieve analysis of each type of aggregate are shown in tables 1 and 2. Both mortars were mixed using the following ratios (by weight): water/cement = 0.5, aggregate/cement = 3.4. The air content of the hardened mortars was 11.0 and 17.7 vol.%, respectively, and the evaporable moisture contents were 8.1 and 10.8 wt.% relative to the oven-dry weight. The mixing procedure described in ASTM C192 was utilized for both mixes.

Mix 1 was designed to produce a mortar with a compressive strength of approximately 17.2 MPa (2500 psi). To maintain this strength until testing, the 10-day-old specimens were placed in a freezer held at 0°C. The hydration process, which causes concretes to increase in strength as they age, is slowed considerably at this temperature. Prior to the 0°C aging, the specimens were aged in covered glass molds for 24 h at 23°C and then removed from the molds and placed in saturated-lime water at 23°C for 7 days. Upon removal from the lime-water bath, they were wrapped in plastic and aluminum foil for shipment.

†This work was done for the Department of Commerce, Maritime Administration, Department of Commerce Building, Washington, D.C. 20235, under Program 193000, Project 12-410-54-425.

Table 1. Primary components of aggregates by percentage of particle count remaining on standard sieves.

Mix	Component	Standard Sieves		
		No. 8 (2.36 mm)	No. 16 (1.19 mm)	No. 30 (0.60 mm)
1	Carbonate	80.3	79.8	64.3
	Chert	8.9	11.0	----
	Granite	3.6	2.4	----
	Basalts	3.4	2.0	1.2
	Quartzite	3.0	4.3	----
	Quartz-Chalcedony	----	----	26.5
	Feldspar	----	----	7.6
2	Granite	67.4	66.4	33.2
	Metamorphics	7.2	3.7	1.5
	Basalts	6.8	2.7	1.8
	Quartz	12.4	17.2	44.9
	Feldspar	5.7	7.2	15.9

Table 2. Sieve analysis of the aggregates used in mix 1 and mix 2.

Number	Standard Sieve Size (mm)	Percent Passing	
		Mix 1 (Elgin)	Mix 2 (Clear Creek)
4	4.75	100	99
8	2.36	78	80
16	1.18	63	59
30	0.60	46	29
50	0.30	10	7
100	0.15	0	0

Mix 2 was made in two batches separated by 8 months. Mix design and conditioning for both batches were identical. The mortar was formed in airtight plastic molds and aged in the mold for 2 days at 23°C. After removal from the molds, the specimens were immersed in a saturated lime-water bath at 23°C. They remained in the bath until tested. The age of the specimens at the time of testing was from 79 to 215 days.

3. Specimens

The solid core specimen geometry was chosen for the compression and splitting strength tests; specimens measured 5.1 cm in diameter and 9.9 cm long, as recommended by ASTM C469-65, C496-71, and C873-77T. The flexural strength test specimens were 5.3 cm x 1.8 cm x 4.6 cm per ASTM C78-75.

The ends of all specimens to be tested in compression were ground parallel and perpendicular to the longitudinal axis to within $\pm 6.3 \times 10^{-4}$ cm. All mix 1 samples were completely saturated with lime-water solution and then kept at 0°C until the time for testing. All mix 2 specimens were stored in a lime-water solution and were removed just prior to testing.

4. Test Procedure

Tests were performed using a commercial 1.0 MN (2.2×10^5 lbf) servohydraulic static/fatigue test machine. The manufacturer's specifications indicate that the load

weighing system accuracy is $\pm 0.5\%$ of indicated load or $\pm 0.25\%$ of the load range in use, whichever is greater, on all load ranges. The crosshead speed used for all tests was 0.005 cm/min and is accurate to $\pm 0.1\%$ of set speed at all loads and speeds. Compression specimens were instrumented with specially designed, diametrically opposed cryogenic strain-gage extensometers. This method for sensing strain worked at all temperatures, including total immersion in liquid nitrogen and liquid helium. Prior to each test, particular care was taken to insure coaxial alignment of all test components (e.g., actuator ram, specimen, dewar) to insure uniaxial loading.

Specimen failure modes indicated a well-aligned system with a minimum of bending, i.e., uniform, conically shaped fractures emanating from the specimen ends to the center.

Some testing was carried out to determine plane strain fracture toughness of mix 2. Short-rod specimens with a single chevron in the configuration proposed by Barker and Baratta [3] were prepared. The diameter was 5.1 cm and the length was 10.2 cm. Testing at all temperatures was carried out in a newly designed fixture that operates like a pinch-type clothespin to load the chevron-notched specimen in tension.

This fixture has a lever-arm ratio of 2:1 and can be fitted into most universal-testing machines. The plane strain fracture toughness, K_{Ic}^{SR} , was calculated using the following equation proposed by Baker and Baratta [3] and modified by Beech and Ingrassia [4]:

$$K_{Ic}^{SR} = \frac{AP_c}{B^{3/2}}$$

where A is a dimensionless calibration constant $\sim 22.5 \pm 15\%$ (dependent on specimen geometry), P_c is the load at crack instability, and B is the specimen diameter.

With one exception the specimens fractured across the chevron; one specimen fractured in the body of the specimen.

5. Results

The compressive Young's modulus for mix 1 increased in value from room temperature to 195 K, and then at 76 and 4 K it decreased by approximately 30 percent (table 3). The compressive strength, however, increased as the test temperature decreased. Also, only at 195 K did mix 1 show a maximum compressive strength before reaching its ultimate.

The compressive modulus, proportional limit, and ultimate compressive strength for mix 2 all increased in value as the test temperature decreased (table 4). As shown in table 5, the splitting tensile strength increased from 295 to 195 K and then remained the same down to 76 K. The modulus of rupture (flexural strength), however, shows a peak value at 195 K.

The plane strain fracture toughness, K_{Ic}^{SR} , for short-rod specimens of mix 2 are tabulated vs. temperature in table 6. The room temperature results on moist mix 2 mortar are comparable to the fracture toughness reported for two kinds of rock [5]:

TYPE OF ROCK	K_{Ic}^{SR}	
	$\text{MPa}\cdot\text{m}^{1/2}$	$\text{psi}\cdot\text{in}^{1/2}$
Limestone	0.99	900
Granite	2.48	2250

The results in table 6 show that there was a substantial increase in K_{Ic}^{SR} as temperature was lowered initially, but no additional increases as temperature was lowered further. Splitting strength showed the same trend with decreasing temperature. It is likely that the pronounced increase in toughness with decreasing temperature is related to changes in the internal structure of the moist specimens as the water entrained in the pores freezes

and expands and then contracts with further cooling. Monfore and Lentz [6] discuss possible effects of freezing on concrete.

The plane strain fracture toughness of mix 2 mortar (table 6) is low compared to the fracture toughness of structured steels i.e., $54.9 \text{ MPa}\cdot\text{m}^{1/2}$ ($50 \text{ ksi}\cdot\text{in}^{1/2}$). This comparison suggests that concrete is not a suitable structural material under tensile loading. Nevertheless, the low cost and widespread availability of concrete may favor its application in compressive-loading situations.

Table 3. Effect of temperature on compressive Young's modulus and compressive strength for moist concrete mortar (mix 1).

Test Temperature K	Compressive Young's Modulus		Compressive Strength			
	GPa	ksi	σ Maximum†		σ Ultimate†	
			MPa	ksi	MPa	ksi
295	15.9	2300	----	----	23.0	3.34
	8.6	1250	----	----	21.8	3.16
	22.4	3250	----	----	23.4	3.40
	$\bar{x} = 15.6$	2270			22.7	3.30
195	27.2	3950	----	----	----	----
	----	----	56.5	8.20	53.8	7.81
	38.1	5530	49.1	7.12	47.6	6.90
	----	----	57.4	8.32	56.9	8.25
	$\bar{x} = 32.7$	4740	54.3	7.88	52.8	7.65
76	14.3	2070	----	----	71.1	10.31
	31.0	4490	----	----	107.8	15.64
	17.7	2570	----	----	90.9	13.18
	22.8	3300	----	----	87.9	12.75
	$\bar{x} = 21.5$	3110			89.4	12.97
4	23.0	3340	----	----	----	----

† σ Maximum: maximum stress the specimen reached during the test.

† σ Ultimate: stress at failure.

Table 4. Effect of temperature on compressive properties for moist concrete mortar (mix 2).

Test Temperature K	Compressive Young's Modulus		Proportional Limit		Ultimate Compressive Strength	
	GPa	ksi	MPa	ksi	MPa	ksi
295	15.25	2210	14.45	2.10	31.23	4.53
	19.80	2800	15.65	2.27	28.40	4.12
	$\bar{x} = 17.26$	2510	15.05	2.19	29.82	4.32
195	30.65	4450	33.04	4.79	89.19	12.94
	33.78	4900	28.87	4.19	93.22	13.52
	$\bar{x} = 32.22$	4680	30.96	4.49	91.21	13.23
76	38.59	5600	-----	----	120.9	17.53
	44.71	6480	-----	----	102.5	14.87
	$\bar{x} = 41.65$	6040			111.7	16.20

Table 5. Effect of temperature on splitting and flexural strength of moist concrete mortar (mix 2).

Test Temperature K	Splitting Tensile Strength*		Modulus of Rupture†	
	MPa	ksi	MPa	ksi
295	2.32	0.34	5.25	0.76
	2.64	0.38	5.25	0.76
	$\bar{x} = 2.48$	0.36	5.72	0.83
			$\bar{x} = 5.41$	0.78
195	6.62	0.96	13.44	1.95
	9.69	1.41	13.88	2.01
	$\bar{x} = 8.15$	1.19	13.66	1.98
76	5.96	0.86	9.26	1.34
	8.54	1.24	11.91	1.73
	$\bar{x} = 7.25$	1.05	10.59	1.54

*ASTM C496-71 splitting tensile strength = $T = 2P/\pi ld$

where: T = splitting tensile strength, MPa (or psi)

P = maximum applied load, N (or lbf)

l = length, mm (or in)

d = diameter, mm (or in)

†ASTM C78-75 modulus of rupture = $R = 3Pl/2bd^2$

where: R = modulus of rupture, MPa (or psi)

P = maximum applied load, N (or lbf)

l = span length, mm (or in)

b = average width of specimen, mm (or in)

d = average depth of specimen, mm (or in)

Table 6. Fracture toughness of short rod moist concrete mortar specimens (mix 2)*

Temperature, K	K_{Ic}^{SR}		P_c	
	MPa·m ^{1/2}	(psi·in ^{1/2})	kg (lb)	
295	0.88	(800)	41.0	(90.28)
	0.95	(860)	41.8	(92.16)
	0.88	(800)	39.3	(86.54)
195	2.26	(2060)	101.0	(222.56)
	2.24	(2040)	99.9	(220.30)
	2.04	(1860)	91.8	(202.32)
	2.20	(2000)†	97.9	(215.80)
76	1.91	(1740)	85.1	(187.70)

*All test specimens were 5.08 $\begin{smallmatrix} +0.00 \\ -0.05 \end{smallmatrix}$ cm in diameter.

†Specimen fractured in body away from chevron.

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