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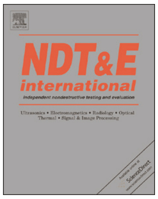

# Correlation between thermal deformation and microcracking in concrete during cryogenic cooling

Reginald B. Kogbara [a,*], Srinath R. Iyengar [a], Zachary C. Grasley [b], Syeda Rahman [b], Eyad A. Masad [a,b], Dan G. Zollinger [b]

[a] Mechanical Engineering Program, Texas A&M University at Qatar, P.O. Box 23874, Education City, Doha, Qatar
[b] Zachry Department of Civil Engineering, Texas A&M University, College Station, TX 77843, USA

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ABSTRACT

Thermal deformation behavior of concrete mixtures from limestone and trap rock aggregates has been related to microcracking during cryogenic cooling. The study was aimed at comparing the suitability of the concretes for direct containment of liquefied natural gas (LNG). The results showed strong correlation between the thermal strain rate and the acoustic emission (AE) cumulative hits rate in the concretes. The closeness of the average thermal expansion coefficient of the trap rock mixture over the ambient to cryogenic temperature range to that of 9% Ni or carbon-steel, and its lower cumulative energy emission corroborates previous observations on its porosity, permeability and microstructural behavior. These likely make it more suitable for direct LNG containment.

## 1. Introduction

Traditional liquefied natural gas (LNG) storage tanks utilize 9% Ni steel for the primary containment tank as it has greater ductility at cryogenic temperatures (i.e. $\leq -165$ °C) compared to normal carbon-steel. However, 9% Ni steel is becoming increasingly expensive. Literature review shows that concrete properties generally improve at cryogenic temperatures [1,2]. Utilizing concrete for conventional 160,000 $m^3$ capacity LNG tanks, which costs US$130 million or more, would lead to at least 10–15% cost savings [3]. The development of the standard on concrete structures for containment of refrigerated liquefied gases, ACI 376-11 [4] may increase the impetus for tank designs utilizing concrete for primary LNG containment. However, concrete behavior at cryogenic temperatures is not fully elucidated. Thus, this work seeks to study damage evolution in concrete during cooling due to stresses associated with coefficient of thermal expansion (CTE) mismatch between concrete components.

Studies have shown that concrete cured at 20 °C and 65% relative humidity (RH) exhibits an almost linear strain behavior when cooled below 0 °C. In contrast, water-saturated (wet cured) concrete exhibits a three-stage behavior with expansion between $-20$ °C and $-70$ °C preceded and followed by contraction [1,2,5,6]. Similarly, there is a sudden decrease in the CTE between 0 °C and $-75$ °C depending on the moisture content. A critical RH of 86% has been identified, with the CTE of concrete stored below this value being governed by aggregate type and those stored above 86% RH governed by moisture content [1,7]. Majority of previous studies on damage in cryogenic concrete focused on thermal strains. Thus, there is a dearth of information on measures of concrete damage like acoustic emission (AE), microstructure examination, and changes in porosity and permeability due to internal cracking. These have been the subject of recent related studies [8,9]. Thermally generated stresses could induce AE through microscopic deformation. AE signals are transient elastic waves emitted as a consequence of crack initiation and propagation or friction activation in existing cracks. Therefore, AE is a valuable tool for damage monitoring as it is capable of identifying failure mechanisms [10,11].

In the design of a concrete LNG tank, which is subjected to large temperature swings, the stresses and strains due to differences in CTE between concrete and steel must be considered. For instance, a drop of about 188 °C (338 °F) during cooling of the tank wall at $\sim 1$ °C/h is reported to cause contraction such that the composite concrete wall of a 160,000 $m^3$ capacity tank moves approximately 64 mm inward. The 9% Ni steel tank bottom, which is attached to the tank wall at its base, also contracts. Thus, the more similar the CTEs of the tank wall and bottom materials, the less tension is developed in the tank bottom plating and this must be considered in design [12]. The CTE of carbon-steel and 9% Ni steel are similar over the ambient to cryogenic temperature range [12]. *ArcelorMittal* reports a mean CTE value of 8.8 μstrain/°C for the

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* Corresponding author. Tel.: +974 4423 0289.
*E-mail address:* regkogbara@cantab.net (R.B. Kogbara).

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−196 °C to 21 °C range, and 9.9 μstrain/°C for the −129 °C to 21 °C range, for 9% Ni steel [13]. In contrast, the CTE of concrete could vary from 7 to 13 μstrain/°C at ambient temperature and may even decrease to negative values followed by subsequent increase during cryogenic cooling [2]. The extent of the variation depends mainly on the aggregate type, with significant influence from the degree of water saturation of the concrete. Hence, it was recommended that aggregates with a low CTE that is compatible with the cement matrix and a water/cement (w/c) ratio $\leq 0.45$ be used in concrete LNG tanks [4].

In light of the above, this work sought to evaluate and compare the suitability of two concrete mixtures produced with limestone and trap rock aggregates for use in direct LNG containment. The mixtures were shortlisted after testing different concrete mixtures subjected to cryogenic cooling for changes in porosity, mean pore size and internal microstructure using different non-destructive techniques, and water permeability [8,9]. The objective of this research was to investigate whether AE parameters such as cumulative hits and energy rates could provide a good indication of the strain rate in concrete during cryogenic cooling. It also investigated the existence of a relationship between the change in strain per unit temperature drop and cumulative hits and energy per unit temperature. The cumulative hits and energy per unit temperature are AE emission rate per temperature decrement parameters [14], which refer to the cumulative hits and cumulative energy build-up within a given temperature range during cooling. A very large increment in cumulative energy and hits per unit temperature change in a given interval could be related physically to a high damage growth rate in the concrete during cooling. Both parameters are introduced here to evaluate how they vary with the thermal strain within selected temperature ranges that are crucial during water freezing and frost damage in concrete [1]. The research also sought to compare the closeness of the CTE behavior of the concrete mixtures to that of 9% Ni or carbon-steel over the ambient to cryogenic temperature range.

## 2. Experimental methodology

### 2.1. Production of concrete specimens

The concrete mixtures were prepared with river sand as fine aggregate using limestone and trap rock as coarse aggregates. The aggregates were obtained from quarries in Texas, USA. The physical properties and mineralogical composition of the aggregates have been documented in a related publication [8]. The maximum coarse aggregate size employed was 19 mm. Type I portland cement was used for casting of the 75 mm diameter and 150 mm long cylindrical concrete specimens. The w/c ratio was 0.42. Table 1 shows key details of the mixture design used. The 28-day compressive strength values [15] correspond to the minimum specified for concrete for refrigerated liquefied gases when containing liquids (34.5 MPa) in the ACI 376 code [4]. The specimens were cured under water until preparation for testing.

### 2.2. Specimen preparation for strain gage installation

After 55 days of water curing, the concrete specimens were air-dried for about 2 h in the laboratory at 20 °C and 50% RH. The specimens were then cleaned to remove any laitance or other soiling from the gage installation area. Thereafter, grade 120 abrasive papers were used to abrade an area for strain gage installation. The specimens were abraded continuously for 8–10 min, and then thoroughly cleaned with tissue paper until the final tissue used was stain-free. This step was repeated twice and the entire abrading and cleaning process lasted about 25–30 min. The result was a polished surface, which exposed the smaller aggregates of the concrete. It should be noted that this step is quite critical to correct strain measurement by the bonded gage, especially during soaking at a given temperature. Preliminary testing showed that inadequate abrading of the concrete specimen leads to delayed thermal behavior where the thermal output decreases continuously, as the chamber temperature is kept constant, irrespective of the temperature in question.

The abraded surface of the gage installation area was precoated with M-bond 43-B adhesive/coating material (*Vishay Precision Group-VPG*, USA). The coating acts as a barrier against any dampness that is exuded from the surface of the concrete, thereby preventing absorption of moisture by the underside of the strain gage. Thereafter, a thin layer of cyanoacrylate (CN) adhesive (*TML*, Japan) was applied uniformly over the entire back of the strain gage. The gage was then firmly bonded to the concrete surface. A layer of K-1 coating material (special rubber for moisture proofing, *TML*, Japan) was then applied over the gage installation area. The whole assembly was then left to cure for 20–22 h. Thereafter, the coated gage installation area was covered with a waterproof film before deployment of the gage for CTE measurements in cryogenic cooling tests. A similar procedure was used for a 174 mm long by 25.4 mm diameter Invar 36 cylindrical specimen used as reference material in the CTE testing. The gage type employed was WK series gage, WK-00-250AF-350/W (*VPG*, USA), connected to a portable USB-powered Model D4 data acquisition conditioner (*VPG*, USA) via an RJ-45 connector. The gage has matrix length and width of 14.5 mm and 9.1 mm, respectively. It has a resistance of 350 Ω and a gage factor of 2.00 at ambient temperature. Three gages were used for each concrete mixture. These were bonded to the upper, middle and lower portions on different sides of the concrete specimens.

### 2.3. Cooling of concrete specimens

The concrete and invar specimens, on which were bonded strain gages were placed in a *Cincinnati Sub Zero* temperature chamber with internal dimensions, 609 mm × 609 mm × 609 mm. The specimens were cooled from ambient to cryogenic temperatures by liquid nitrogen ($LN_2$) injection from an attached 110-liter dewar (Fig. 1a and b). The moisture condition of the concrete mixtures just before cryogenic cooling was determined as 62% and 69% of the saturation moisture content for the limestone and trap rock mixtures, respectively. A ramp rate of 3.3 °C/min with soaking at selected temperatures for 65 min was employed for the cooling program. The ramp rate chosen was the highest possible cooling rate the temperature chamber can easily accommodate. The selected temperatures were 15 °C, −20 °C, −55 °C, −70 °C, −120 °C and −180 °C, although not all temperatures were used in a given experiment. The soaking time of 65 min (except at 15 °C, for which 60 min was used) was chosen after trials showed that the concrete specimens could attain temperatures

**Table 1**
Mixture design and properties of the concrete mixtures.

| Concrete mixture | Cement (kg/m$^3$) | Coarse aggregate (kg/m$^3$) | Fine aggregate (kg/m$^3$) | Water (kg/m$^3$) | Bulk density (kg/m$^3$) | 28-day compressive strength (MPa) |
|---|---|---|---|---|---|---|
| Limestone | 512 | 868 | 694 | 215 | 2460 | 36 |
| Trap rock | 512 | 1056 | 670 | 215 | 2650 | 40 |