

Thermal Conductivity Measurement Study of Refractory Castables

API PUBLICATION 935
FIRST EDITION, SEPTEMBER 1999



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Downstream Segment

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Thermal Conductivity Measurement Study of Refractory Castables

1 Executive Summary

Thermal conductivity is a physical property that provides guidance in designing refractory systems for equipment in which heat loss and/or thermal behavior are important. The accuracy of reporting and understanding thermal conductivity is vital to developing the most cost effective, efficient, and reliable equipment.

The refractory industry uses various methods for measuring and reporting thermal conductivity that contribute to confusion in interpreting thermal conductivity data. The presence of chemically combined moisture in unfired castable masses complicates the measurement of thermal conductivity. The moisture contributes to higher thermal conductivity values until it is removed. Improper removal of the moisture during initial heat-up can also contribute to incorrect thermal conductivity data.

Temperatures associated with refining of petroleum products are considerably lower than other industries such as steel, foundries, aluminum, etc. At low operating temperatures (1000°F — 1400°F), removal of chemically combined water from refractory castable linings is incomplete, and castable products do not achieve the optimum thermal characteristics. Removal of chemically combined water is a function of temperature. The majority of chemically combined water—approximately 70%—is removed between 500°F and 850°F, with the remainder dissociating up to 1250°F. This is illustrated in a Thermo-Gravimetric Analysis (TGA), shown in Appendix A. Historically, thermal conductivity of castables was represented as a single value. More representative multi-point curves were later introduced as heat loss became more important but captured data while cooling a specimen fired to within 100°F of its use limit. Data collected during cooling of specimens is classified as descending data.

Thermal conductivity measured during initial heating of specimens is defined as ascending data and produces significantly different data than descending data. Ascending data provides a more accurate representation of a product's thermal conductivity for low temperature application typical in most hydrocarbon processing industry (HPI) applications.

A study was initiated to compare the thermal conductivity developed by different measurement techniques and assess the relationship between ascending and descending data. The study was designed to evaluate six products in six laboratories with five measurement techniques. The castable products were chosen to represent a specific category, including: lightweight, medium weight, moderate erosion resistant, dense, dense-extreme erosion resistant, and fused silica castables. The study was designed to show differences in measurement techniques and ascending and descending data. *There was no*

attempt to rank, classify, or assign accuracy to each of the measurement techniques.

The study concluded that the different thermal conductivity procedures/apparatuses yield very different results. Thermal conductivity of lightweight and medium weight insulating castables varied by 100%, depending on the measuring technique. As density increased, differences in thermal conductivity values attributed to measuring technique decreased but were still significant. Test results also indicate that differences in ascending and descending thermal conductivity data, for the castables studies, are considerable and worthy of design consideration.

It is recommended that users and designers utilize ascending thermal conductivity curves (data) in designing refractory lining systems, where heat transfer is a major consideration for applications below 1500°F. It is also recommended that users and designers evaluate thermal conductivity data and the method of measuring the data before using the data in designs when heat transfer and skin temperatures are important to successful equipment operation.

2 Introduction

Thermal conductivity is defined as the amount of heat transferred through a unit area of a material in a unit time, through a unit thickness, with a unit of temperature difference between the surfaces of the two opposite sides.

Thermal conductivity of refractory castables is difficult to measure accurately due to the presence of moisture (chemically combined water) in the matrix. When heated the first time, cementitious castables expel water (dehydration) from the hydrated cement. The moisture is responsible for affecting the identification of heat flowing through the refractory mass.

Manufacturers of refractory products use various measurement techniques to develop thermal conductivity of refractory castables. The following list identifies commonly used procedures.

- a. Water Calorimeter—ASTM C-201 apparatus; C-417 procedure.
- b. Calorimeter—Pilkington Method.
- c. Hot Wire Method—ASTM C-1113.
- d. Comparative Thermal Conductivity Method—Dynatech.
- e. Panel Test.

Each procedure addresses unique concerns about measuring thermal conductivity of unfired castable refractories.

This study was initiated to compare differences in the five test methods at six laboratories. The scope of the study was limited to one set of data for each of six products. Therefore, numeric relationships and direct evaluations between the various methods were not desired nor achieved.

The study concentrated on products with high to moderate cement contents. These products have distinct thermal conductivity curves during initial heating (ascending) and cooling (descending). Low and No Cement products were not evaluated and may or may not follow the same trends developed for the cementitious products.

Cement bonded castables develop physical properties through proper hydration of the cement. Upon heating, the hydrated cement dehydrates as the chemically bonded water dissociates from the calcium aluminate cement. Use of Thermo-Gravimetric Analyses (TGA) provides a good understanding of the dehydration process. Figure A-1 shows a TGA curve for a cement bonded castable refractory. Dehydration begins at approximately 425°F and continues through 1250°F. However, approximately 70% of the water loss due to dehydration occurs between 500°F and 850°F.

3 Test Methods

Thermal conductivity is a measure of heat flow through a medium. Various techniques of measuring thermal conductivity are employed by manufacturers and laboratories. The following is a brief description of the measurement techniques evaluated in this study.

3.1 WATER CALORIMETER

3.1.1 ASTM C-201 Apparatus (Conducted in Accordance with ASTM C-417)

3.1.1.1 The C-201 apparatus consists of a heating chamber, calorimeter assembly, water circulating system, and instrumentation. The heating chamber is capable of being heated electrically over a temperature of 400°F to 2800°F in a neutral or oxidizing atmosphere. Heating is controlled to $\pm 5^\circ\text{F}$. A silicon carbide slab $13\frac{1}{2} \times 9 \times 1$ in., with the $13\frac{1}{2} \times 9$ -in. faces plane and parallel, is placed above the sample for the purpose of providing uniform heat distribution. A layer of insulation equivalent at least to 1-in. Group 20 insulating firebrick is placed below the calorimeter and guard plates.

A copper calorimeter assembly is used for ensuring the quantity of heat flowing through the test specimen. The water circulation is such that adjacent passages contain incoming and outgoing streams of water. The calorimeter is 3×3 in.² and has one inlet and one outlet water connection. An inner and outer guard surrounds the calorimeter.

The water-circulating system provides the calorimeter assembly with water at constant pressure and at a temperature that is not changing at a rate greater than 1°F per hour. Instrumentation for measuring temperatures includes:

- a. Specimen temperature.
- b. Calorimeter water temperature.
- c. Temperature difference between calorimeter and inner guard.

The apparatus is modified for the C-417 procedure to reduce the affect of moisture released from the specimen. Ceramic fiber is used to ensure there is no contact between the specimen and calorimeter. Copper tubes are inserted through the furnace wall to the perimeter of the outer guard to facilitate removal of moisture during heating of the specimen. Compressed air supply with a flowmeter is also a part of this apparatus.

Thermal conductivity is determined by measuring the temperatures of the furnace and specimen, water temperature rise, and calculating thermal conductivity with the following formulation.

$$k = \frac{QL}{[A(t_1 - t_2)]} \quad (1)$$

where

k = thermal conductivity in Btu in./hr ft²°F,

Q = Btu/hr flowing into the calorimeter,

L = thickness (distance between hot junctions at which t_1 and t_2 are measured) in in.,

t_1 = higher of two temperatures measured in the test specimen in °F,

t_2 = lower of two temperatures measured in the test specimen in °F,

A = area of center calorimeter in ft².

3.2 CALORIMETER

3.2.1 Pilkington Apparatus MTP-103

3.2.2 The Pilkington apparatus is composed of a heating chamber, calorimeter assembly, and instrumentation. The refractory specimen is placed 2 in. above and parallel to the silicon carbide heating elements. The calorimeter is located in direct contact with the top surface of the specimen. The $2\frac{1}{4}$ -in. diameter calorimeter is surrounded by an inner and outer guard. Heating chamber temperature is controlled by a platinum-platinum/13% rhodium thermocouple located between the specimen and the heating elements. Platinum-platinum/13% rhodium thermocouples are attached to the calorimeter to measure the temperature gradient.

The refractory specimen is cut to form a solid octagon, 4.4 in. to 4.5 in. between parallel sides. The specimen should be cut/ground to a thickness between 1 in. and 3 in. based on density. The octagonal surfaces must be flat and parallel within ± 0.01 in. Platinum-platinum/13% rhodium thermocouples are cemented into grooves in the hot and cold face of the specimen to measure the temperature gradient.