Thermal Expansion Under Load and Creep of Refractories—An Evaluation and Comparison of Testing Methods

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Contents

Figures

Tables

Contents

Executive Summary

Refractory brick linings used in high-temperature applications need the ability to sustain structural loads and maintain volume stability at temperature. API is developing a recommended practice (API RP 565) for reaction furnaces in sulfur recovery units (SRUs) operating up to 1540 °C (2800 °F) for continuous use and 1650 °C for intermittent use. To meet these operating conditions in a horizontal cylindrical reactor vessel configuration as defined in the RP, self-supporting brick ring construction of dense high-alumina brick has proven to be the lining system of choice.

In support of the development of API RP 565, it is recognized that not all high-alumina refractories have the same physical properties. Historically, several standard tests have been developed to test and characterize hot properties under compressive load conditions[1–3]. While there are several papers that describe these methods[4–7], there is very little in the literature to compare these tests, their features, their benefits, and their deficiencies, including precision and repeatability.

In this study, a total of 15 commonly used high-temperature brick products for SRU applications were tested via ASTM C832 and DIN EN 993–9 at the Orton Materials Testing and Research Center in Columbus, Ohio, leading to an evaluation of the results as they pertain to service performance. This report documents an evaluation and comparison of available test standards for measuring high temperature physical properties of the brick to be used in selection and quality control to better meet performance expectations.

The testing results lead to several conclusions and recommendations. ASTM C832 and DIN EN 993–9 are shown to provide more useful information on the bricks in response to temperature and loading than ASTM C16, which measures only the before-and-after dimensions. Consequently, they provide for a better definition of the competing effects of creep compression due to load versus expansion due to crystalline growth as a result of exposure to heat of service magnitude. Based on test results and comparing results between the ASTM C832 and DIN EN 993–9 standards, opportunities are identified to combine the better features of each of the tests into a single, more optimized testing practice, including:

— standardizing on a 1650 °C (3000 °F) test temperature, with results reported separately for 1–50 hours and 50–100 hours;

— replacing push rods with a more reliable laser measurement;

— using the slower ASTM heating rate to allow mineralogical equilibrium to be reached before creep testing begins; and

— adopting the DIN cylinder for testing due to easier fabrication.

Based on reported findings, future testing should include:

— low load testing at different heat-up rates to better understand the condition of positive mineralogical change while avoiding those with negative impact; and

— cooperative testing between ASTM and DIN to set a more universal standard. In absence of an agreed-to universal standard, consider adding a modification to creep testing in API RP 565 similar to ASTM C704 in API 936.

NOTE This report includes references to the Orton Materials Facility and the registered trademark Lucalox, which are provided for purposes of disclosure of the methodology used in this research, and are not intended as an approval or endorsement of either as necessary for testing.

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1 Scope

1.1 This report documents the results of thermal expansion under load (TEUL) and creep in compression testing conducted by ASTM C832 and DIN 993–9 (same test as ISO 3187).

1.2 The prime objective for this study is to determine if one or both tests should replace the ASTM C16 method. ASTM C16, modified from 1.5-hour to 100-hour hold, has been widely used to qualify brick for sulfur reaction furnace use. In addition to deformation after 100 hours, the creep tests will provide additional information of TEUL and evidence of mineralogical changes during their early life cycle in process operation.

1.3 The second objective for this study is to better define a refractory specification protocol for premium brick used at high temperatures while supporting a load. Hold temperature in this testing was 1600 °C (3000 °F). This temperature is designed to simulate operating conditions of oxygen-boosted thermal reactors, as well those occurring during over-temperature excursions.

1.4 Orton Memorial Laboratory was selected to do all testing, as this laboratory can test both DIN and ASTM specimens in the same furnaces. Orton used a laser non-contact measurement method for both the ASTM and DIN samples. This allowed an evaluation only of the laser method while still facilitating evaluations of differences in heating rates, applied compressive load, and hold-time durations.

2 Reference Documents

M. K. Ferber, A. A. Weresczak, and J. G. Hemrick, "Final Technical Report – Compressive Creep and Thermophysical Performance of Refractory Materials," *ORNL/TM-2005/134*, Oak Ridge National Laboratory (2006).

S. Jin, H. Harmuth, and D. Gruber, " Compressive Creep Testing of Refractories at Elevated Loads – Device, Material Law, and Evaluation Techniques," *Journal of the European Ceramic Society*, 34 (2014) 4037-4042.

O. Buyukozturk and T. Tseng, " Thermomechanical Creep Behavior of Refractory Concrete Linings," *Journal of the American Ceramic Society*, 65 [6] (1982) 301-307.)

S. Samadi, S. Jin, D. Gruber, H. Harmuth, and S. Schachner, " Statistical Study of Compressive Creep Parameters of an Alumina Spinel Refratory," *Ceramics International*, 46 (2020) 14662-14668.

3 Background

3.1 Hot Load Deformation Test

The ASTM C16 hot load deformation test is a long-standing standard used to screen brick and shapes for use in sulfur reaction furnaces. In this test, bricks are placed in a furnace and loaded as in Figure 1. The test bricks are measured at ambient temperature before and after heating to determine a net permanent linear change. Previous investigators have shown the short 1.5-hour duration under load at the test temperature and volume changing mineralogical transformations can severely limit the usefulness of this test for high-temperature SRU applications.

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[1] ASTM International, 100 Barr Harbor Drive, West Conshohocken, Pennsylvania 19428, www.astm.org.

[2] DIN, Saatwinkler Damm 42/43, 13627 Berlin, Germany, www.din.de.