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Tensile creep measurements of ordinary ceramic refractories at service related loads including setup, creep law, testing and evaluation procedures

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Abstract[1]

The performance of refractories under operating conditions is often influenced by tensile creep. This paper proposes a high-temperature tensile testing device which allows for long term creep measurements at operating loads. The chosen specimen geometry is more adequate for the testing of ordinary ceramic refractories which may be heterogeneous with respect to grain size (maximum grain size e.g. 5 mm) and chemical composition. For this application the innovative design exhibits not only improved specimen and loading alignments, but also reliable specimen holding and cooling systems. Specific procedures were followed to avoid uneven stress distribution along the specimen gauge length. The testing procedure was optimized by simulating experimental creep conditions with a finite element (FE) model built with the software Abaqus. Measurements were performed on magnesia–chromite material at different temperatures and applied stresses. Under these testing conditions, three creep stages emerged. The Norton-Bailey creep rate equation was employed to describe the creep behavior for the three stages. An evaluation using the Generalized Reduced Gradient (GRG) algorithm was then performed in order to identify the three creep stages and inversely estimate the Norton-Bailey creep parameters *n*, *a* and *K* for each stage.

Keywords: Refractories; Tensile creep; FE simulation; Norton-Bailey; Inverse estimation

1. Introduction

In industrial applications, refractories may be subjected to frequent thermal cycling during important mechanical loadings [1]. Both compressive and tensile loads under high temperature conditions may lead to creep of the refractory materials, altering their mechanical properties and performance [2]. Creep tests are performed in order to understand the behavior of refractories at high temperatures and to design failure resistant systems. The expected creep deformation may influence the lining design. Two standard testing methods are available for the assessment of refractory materials; refractoriness under load (RUL) and creep in compression (CIC) [3,4]. The RUL (EN 993/ISO 1893) method measures the deformation behavior when the specimen is exposed to a constant load with increasing temperatures. The test is performed with heating being applied at an increasing rate of 5 K/min and a maximum loading of 0.2 MPa. The specimens are cylindrical shapes. The CIC (EN 993/ISO 3187) method determines the deformation of a specimen under a constant load and at a constant temperature. The specimen shape, the test apparatus and the maximum load are the same as those for the RUL method. Both approaches are suitable for compressive loading only. A maximum load of 0.2 MPa is required for creep observation at lower temperatures but can be insignificant in practical applications of refractories, which are generally subjected to greater loads [5]. Additionally, because deformation occurs during the heating up procedure as well, the beginning of material creep cannot be exactly determined.

Previous studies on the tensile creep process mainly investigated numerous advanced ceramics such as silicon

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[1]Finite Element (FE), Generalized Reduced Gradient (GRG), Refractoriness Under Load (RUL), Creep In Compression (CIC), Ceramic Matrix Composite (CMC), Zircon–Mullite–Zirconia (ZMZ), Alumina–Zirconia–Silica (AZS), International Organization for Standardization (ISO), American Society for Testing and Materials (ASTM), European Committee for Standardization, JISC (Japanese Industrial Standards Committee).

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nitride ceramics, ceramic matrix composites (CMC), mullite and zircon–mullite–zirconia (ZMZ) [6–12], or ceramics for glass industry belonging to the AZS system [13–15]. Many tensile testing facilities have been developed in previous years [16–18] and considerable efforts have been made in the standardization of fine ceramics (ISO, ASTM, CEN and JISC) illustrating the range of specimen geometries or testing methods [19,20]. However, there are currently no standardized methods that describe a high temperature tensile creep testing of heavy ceramic refractory materials with heterogeneous microstructures and different grain sizes. In addition, literature research provides limited tensile creep data of heavy ceramic refractories [21,22]. Lewis E. Mong [22] carried out tensile and compressive creep tests on numerous refractory bricks up to 950 °C. The specimens, of cylindrical shape ($228.6 \times 25.4\ mm^2$), were attached to porcelain load links. Few research laboratories report on devices for tensile creep measurement of refractories because it is often problematic to fulfill all the specifications necessary for worthwhile measurements. Accurate and long term measurements of small strains at high temperatures (up to 1600 °C) with a suitable alignment, while avoiding bending of the specimen and ensuring uniform uniaxial stresses, are essential. Moreover, suitable fixtures for relatively brittle materials at high temperatures are difficult to implement. Nevertheless a number of studies referring to uniaxial tensile tests of heterogeneous refractories, without considering creep, were available [23–27]. For instance Nazaret et al [26] performed tensile tests at high temperatures up to 900 °C with specimens' geometry of $30 \times 25\ mm^2$ reduced cross section and 12.5 mm gauge length. Sample extremities were glued on water-cooled metallic plates. Ghassemi et al [27] also used cylindrical samples of 18 mm diameter with two metallic parts glued at each end and a gauge length of 25 mm. They reached temperatures of 1200 °C on refractory castables.

Several criteria had to be met for a reliable tensile creep measurement of refractory materials. Finite element (FE) models simulating experimental conditions were built using the commercial software Abaqus in order to optimize the

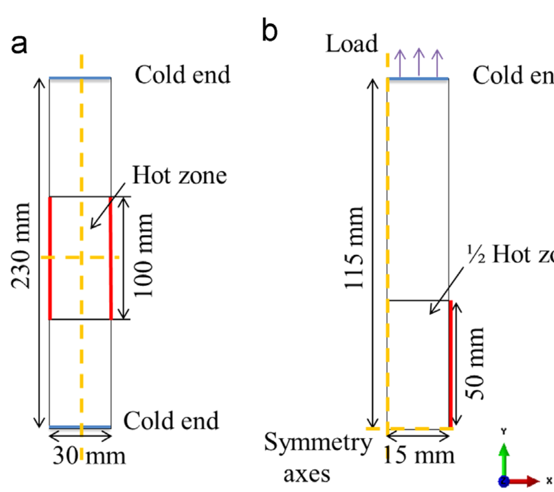

Fig. 1. (a) Specimen dimensions and (b) axisymmetric finite element model.

testing procedure. A new testing device with an innovative design was developed to fulfill all the requirements for tensile creep detection and measurements. The rigid setup included a strong specimen holding mechanism that provided for an accurate alignment and permitted a wide temperature range. Specific measures were taken so as to achieve homogeneous isothermal loading. The new testing design and the specific characteristics are detailed within this paper. The material studied was a commercial burnt magnesia chromite refractory. The experiments were performed at loads between 0.2 and 1.75 MPa and at temperatures up to 1600 °C. An evaluation method was implemented for identifying the creep stages while inversely estimating the creep parameters for each stage based on the Norton Bailey creep law [28,29].

2. Innovative setup design and procedures

2.1. Choice of the specimen shape and the heating up schedule

The design of the testing procedure first needed to determine a specimen shape suitable for coarse-ceramic refractories. Different specimen types for tensile testing were previously investigated [30–32]. Several studies utilized the flat dog-bone or rectangular specimen shape with various dimensions at high temperatures [33–36]. Those shapes are complex and manufacturing is limited to coarse-ceramic refractories having grains ranging in size from few micrometers up to approximately 5 mm. Because of the size of intrinsic defects in the material, as well as its microstructure, tensile specimens of sufficiently high volume were needed to be representative. Therefore cylindrical specimen shape, with a height of 230 mm and a diameter of 30 mm, was chosen. Smaller specimen dimensions would not be adequate due to the size of the biggest grains. Additional stresses and defects were thus avoided and the parallelism enhanced contrary to the rectangular or dog-bone shapes. The manufacturing of cylindrical shape was in that case advantageous and additional pre-cracking was avoided. The heated section of the specimen is 100 mm long (Fig. 1a). Both specimen ends were glued to steel adapters which are secured by water-cooled grips. A FE simulation was carried out in advance using the software Abaqus in order to determine the viability of the tensile creep measurements with the proposed specimen design and to estimate the temperature distribution within the specimen. It allowed for the prediction of the temperature and stress distributions in the specimen during the preheating and the creep test phases. It also allowed for more extensive observations of these distributions in the hot central zone and at the cold ends.

A simplified two-dimensional axisymmetric plane was considered for the model since the specimen geometry exhibited radial symmetry. The mesh consisted of 1740 quadrilateral elements of the coupled temperature–displacement type. The cylinder was supported by a simple symmetrical boundary condition at the lower end of the model (Fig. 1b). The preheating in the simulation ranged from 20 °C to 1600 °C at various heating rates (2.5 K/min, 5 K/min and