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Compressive creep testing of refractories at elevated loads—Device, material law and evaluation techniques

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Abstract

In order to cost-effectively characterize the high temperature compressive creep behaviour of refractories a testing device was designed for application at elevated loads. Special measures have been taken necessary to enable an even stress distribution within the specimen. To identify Norton-Bailey strain hardening creep law parameters a general inverse procedure using a Levenberg–Marquardt algorithm was developed. Satisfying experimental results could be received from the creep measurement in a wide range of temperatures and loads for both shaped and unshaped materials. By fitting the strain/time curves the creep law parameters of refractories under various temperatures can be precisely identified. The measurements also reveal that at elevated loads all three creep stages can be observed.

Keywords: Compressive creep; Elevated loads; Norton-Bailey; Inverse estimation; Refractories

1. Introduction

During service refractories are frequently exposed to intense thermomechanical loads resulting from the collaborative effects of severe thermal shock, thermal expansion of refractories and external mechanical constraints.[1,2] Under these conditions refractories may respond in elastic or inelastic manner which depends on the magnitudes and types of loads as well as on the material behaviour at high temperatures. Both material failure under Mode I–III conditions and creep may account for this inelastic behaviour and bring about an irreversible deformation of refractories.[3–7]

To characterize the creep behaviour of refractories, conventionally two types of testing procedures are employed. One is so called creep-in-compression (abbreviated as CIC) which applies the same equipment as the testing procedure for refractoriness-under-load (abbreviated as RUL). The standards of CIC in Europe, USA, Japan and China are more or less similar and mainly differ in specimen dimension, testing load and heating rate.[8] According to the European standard EN 993-9,[9] a fixed compressive stress of 0.2 MPa is loaded on the specimen during the heating-up and dwell periods. The change in length of the specimen is measured from the differential displacement of two corundum tubes through linear variable differential transducers (LVDTs). The deformation at certain dwell time relative to the maximum expansion occurring during the heating-up period is determined. This method is applied to compare the creep resistance capacity of refractories and to receive guidance for material selection and development in a rather phenomenological manner.[10–13] However, a deeper understanding of the creep mechanisms of refractories is demanded as well as extracting creep data for further thermomechanical modelling. For this purpose various loading conditions were also considered.[14–19] Formulating the secondary creep rate in dependence of temperature and stress offers one way to reveal the creep mechanisms of refractories, from which measures for material improvement may follow.

What are the tasks a satisfactory creep testing equipment should fulfil? The CIC measurement according to the European standard EN 993-9 is not suitable for determination of creep

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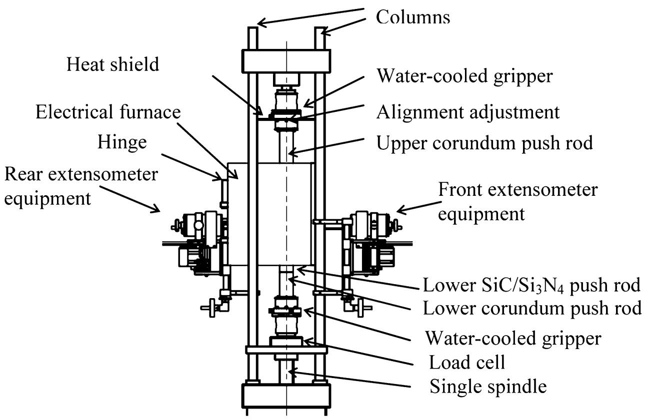

Fig. 1. Schematic diagram of the testing machine.

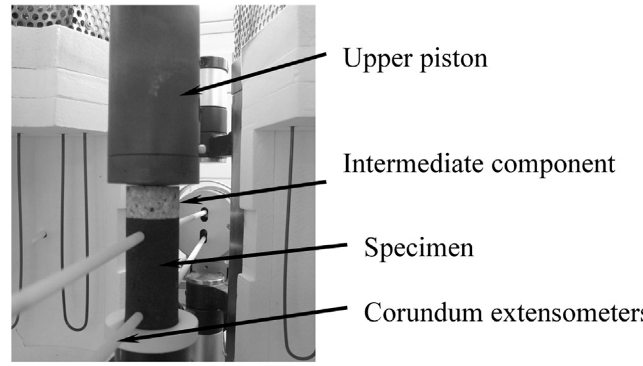

Fig. 2. Setup of the high temperature compressive creep test in the furnace.

laws. One reason is that the onset of creep is not defined. Creep will start during the heating up procedure before the beginning of the dwell time because the specimen is heated up under load. Moreover the restriction of the maximum load to 0.2 MPa does not allow investigation of load levels significant for practical use of refractories in many cases. A newly designed testing equipment should especially allow the following features of the measurement: The start of the loading under isothermal conditions must be defined exactly. Moreover, displacement has to be measured on the specimen surface itself, preferably at least at two diametrical locations, in order to assess even creep of the material. Further alignment of the specimen and the testing machine is of high importance: the line of action of the imposed load has to coincide with the specimen axis. Otherwise a bending moment would be caused and result in an uneven stress distribution within the specimen. Moreover a homogenous load distribution will depend on the quality of the specimen preparation; especially parallel end faces perpendicular to the specimen axis are desired. Furthermore the application of sufficiently high stresses related to service conditions is necessary. Only sufficiently high load levels will allow observing secondary and tertiary creep in reasonable testing times.

To fulfil these missions a testing device for high temperature compressive creep application was newly developed and is presented in this paper. Efficient inverse procedure to identify the creep law parameters is also introduced with two case studies of shaped and unshaped materials.

2. Construction and main features of the testing machine

A schematic diagram of the device for high temperature compressive creep testing is shown in Fig. 1. The whole setup is based on a spindle-driven universal testing machine of sufficient stiffness. An electrical furnace is inserted and equipped with silicon carbide heating elements. The furnace is composed of two symmetrical parts, held together by means of hinges in the rear and can be closed tightly with two buckles in the front. Two platinum-rhodium thermocouples are used to measure the temperatures, one of which is located near the specimen to monitor its temperature and the other one is placed close to the heating elements to control the furnace temperature. Two pairs of corundum extensometers are placed in the front and rear of the furnace, respectively. The loading is realized by a single spindle connected to a load cell. Water-cooled grippers are placed at the cold ends of corundum push rods to protect the metal components. To guarantee coincidence of the axis of the lower and upper piston an alignment adjustment is provided.

Compared to conventional creep testing machines, this device possesses several advanced characters: compared with the CIC test it allows application of by far higher loads up to app. 20 MPa on a specimen with a diameter of 35 mm and a height of 70 mm. The height/diameter ratio of 2 is suitable for deformation measurement in a zone of the specimen which is not affected from friction of the end faces (here especially the lower end face) any more. The displacement of the specimen under load is measured directly on its surface utilizing two pairs of corundum extensometers, as seen in Fig. 2; the onset of deformation measurement is documented while the loading procedure starts, and therefore the point of creep origin is well defined. Uneven loading is tactically avoided by modifying the end face of the upper piston into a slight spherical surface. Both end faces of the cylindrical specimens are prepared by diamond grinding. Point-surface contact between the upper rod and the specimen would result in a concentrated load on both surfaces. In order to protect the head of upper ceramic rod and load the force evenly onto the specimen, a less brittle refractory material is placed between the ceramic rod and specimen, as labelled as intermediate component in Fig. 2.

Alignment of specimen and testing machine is essential to achieve similar readings on both extensometers. Therefore the specimen axis has to coincide with the line of action of the applied load. This can be tested by preloading at room temperature before starting the test. Necessary alignment can be done by adjusting the position of the upper piston (alignment adjustment in Fig. 1) or of the specimen itself. The creep tests will normally be performed under isothermal conditions after the furnace is heated up to a defined temperature with 10 K/min. In order to fix the specimen a small preload is applied before the temperature is achieved. Afterwards, the loading procedure is commenced by ascending the lower crosshead; a heading of 0.3 mm/min is mainly applied. The creep test under constant load will last several hours. The extensometer reading starts from the beginning of loading with an initial leg distance of up to 50 mm between