IDENTIFICATION OF THE MECHANICAL PROPERTIES OF AN ASYMMETRIC CREEP LAW APPLIED TO REFRACTORIES AT HIGH TEMPERATURES USING THE DIGITAL IMAGE CORRELATION TECHNIQUE

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ABSTRACT

Refractory materials are designed to work at high temperatures up to 1600°C at the hot face in vessels such as the steel ladle, and therefore creep strains play an important role in the mechanical behavior of the refractory lining. These materials often demonstrate asymmetric creep behavior, i.e., different creep strain rates are observed under tension and compression. To characterize the behavior of an alumina-spinel brick used in steel ladles, an innovative experimental setup composed of Brazilian tests associated with a digital image correlation (DIC) technique is proposed. The details of the optical techniques suitable to acquire high quality images are described, as well as the features present in the DIC software that allow the determination of the material parameters. Further, a four-points bending test is simulated using the identified parameters, and the numerical displacements fields are compared to experimental results.

INTRODUCTION

In experimental mechanics, optical full field methods for the characterization of displacements and strains are of great interest, since normally they allow to obtain a considerably higher volume of information from an experiment than classical point-wise techniques such as extensometry. Another advantage of such methods is the absence of direct contact of the measuring instrument with the sample, which is especially important in cases where this contact can result in noise on the experimental data, such as for measurements upon soft materials or in harsh conditions like high temperatures[1].

Several full field measuring techniques are currently available in the literature[2], and, among those, the Digital Image Correlation (DIC) technique has been receiving considerable attention from various research groups, due to its versatility and to the possibility to easily compare its output to numerical simulations performed, for example, using the Finite Element Method.

The fundamental assumption of the DIC technique is the conservation of optical flow, which states that the changes in the pixel values from an image obtained at instant t (reference image) to the next image obtained at instant t^* (deformed image) during a mechanical test are due uniquely to the deformation of the sample and consequent movement of the image surface texture. Based on a measurement of the difference between the reference and deformed image – for example, a cross-correlation criterion[3] – The displacement fields can be calculated. For a mathematical description of the DIC technique, see Besnard et al[4].

DIC at high temperatures

At high temperatures, DIC techniques can be particularly interesting, since contact measurement devices can be expensive and difficult to operate. Nevertheless, such techniques also present important inconveniences, mostly related to the acquisition of high-quality images. Leplay et al.[5] highlight the three main challenges related to this topic:

1. Maintain the stability of the speckle pattern, that needs to withstand the temperature and cannot react with the sample, while keeping an acceptable contrast.
2. The excess of black body radiation leads to a violation of brightness conservation.
3. The existence of a temperature gradient between the sample and the camera and the consequent variation of the refractive index of the air with the temperature, resulting in heat hazes.

Several authors have proposed experimental setups to improve the images' quality at high temperatures[5,6,7]. In this work, Brazilian tests are used to identify the parameters of an asymmetric creep law previously proposed by the authors[8] for an alumina-spinel material applied in steel ladle linings, at 1300 ºC, and four-point bending tests are used to validate the obtained results. Additionally, the identified compressive creep parameters are compared with data available in the literature for the material being studied[9,10].

ASYMMETRIC CREEP MODEL

The creep model whose properties are identified is based on the principle of split of the stress tensor into tension and compression parts, originally proposed by Blond et al.[11], with a further weighted sum of the contribution of each stress sign for the creep strain rate, as described in Eqs. (1), (2), and (3).

$$\underline{\underline{\dot{\varepsilon}}}^{cr} = w^+ \cdot \underline{\underline{\dot{\varepsilon}}}^{cr^+} - w^- \cdot \underline{\underline{\varepsilon}}^{c\dot{r}^-} \qquad (1)$$

$$w^{\pm} = \frac{\sigma_{eq}^{\pm}}{\sigma_{eq}} \qquad (2)$$

$$\begin{cases} \underline{\underline{\dot{\varepsilon}}}^{cr^+} = \dfrac{3}{2}\dfrac{\underline{\underline{s}}}{\sigma_{eq}} A^+ \langle \sigma_{eq} - f_y^+ \rangle^{n^+} p^{m^+} \\ \underline{\underline{\varepsilon}}^{c\dot{r}^-} = \dfrac{3}{2}\dfrac{\underline{\underline{s}}}{\sigma_{eq}} A^- \langle \sigma_{eq} - f_y^- \rangle^{n^-} p^{m^-} \end{cases} \qquad (3)$$

where + and – represent the contributions of tension and compression loads, respectively, σ_{eq} is the von Mises equivalent stress, $\underline{\underline{s}}$ is the deviatoric stress tensor, f_y is the yield stress, p is the accumulated viscoplastic (creep) strain, w are the weighting factors and A, m and n are temperature dependent material parameters. For more details about the constitutive model, see Teixeira et al[8].

In this work, secondary creep was considered under tension, which is consistent with experimental data available in the literature[10]. Therefore, $m^+ = 0$.

EXPERIMENTAL SETUP – BRAZILIAN TESTS

The Brazilian test was used to make the identification of the asymmetric creep properties due to its heterogeneous stress distribution, represented in Fig. 1, presenting tension, compression, and shear loads in the sample at the same time[8].

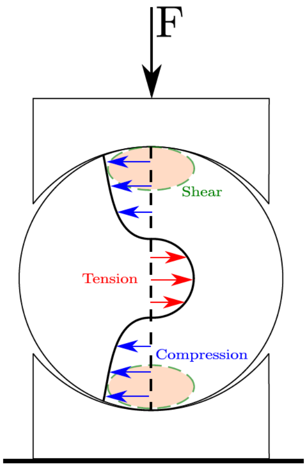


Fig. 1. Stress distribution in a Brazilian test sample[8].

Fig. 2 shows the experimental setup used to perform the Brazilian tests and to take the pictures at high temperatures. This setup is composed of the following parts:

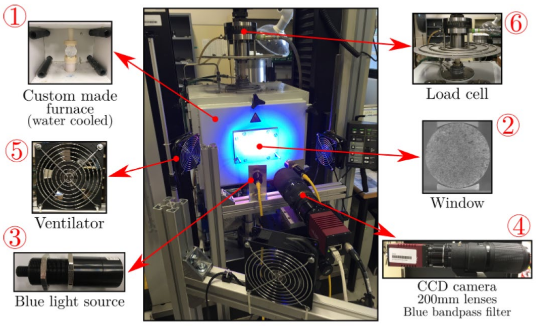


Fig. 2. Experimental setup for the Brazilian tests[8].

1. A water-cooled custom-made furnace, with the capacity to heat up to 1300 °C.
2. The furnace's door is equipped with a window made of a vitro ceramic material, so that the sample can be photographed.
3. To increase the amount of blue light available when taking the pictures, two blue light sources are used to enlighten the sample. In combination with the blue filters, it increases significantly the contrast of the pictures, to a point where the DIC analysis becomes possible.
4. The pictures are taken using high resolution CCD camera. A blue band pass filter, to decrease the amount of light being captured by the camera, since it blocks all parts of the optical spectrum that are not blue, avoiding the saturation of the sensor.
5. The camera and the lenses are cooled using two ventilators, since they stand close to the furnace and can overheat.
6. An universal testing machine equipped with a 30kN capacity load cell.

In order to increase the contrast in the pictures, a SiC speckle pattern was applied in the sample. Fig. 3 shows the considerable increase in the image's quality at 1300 ºC when the proposed experimental device is used, in comparison to images taken at high temperature and without using the blue light and the blue band pass filter.

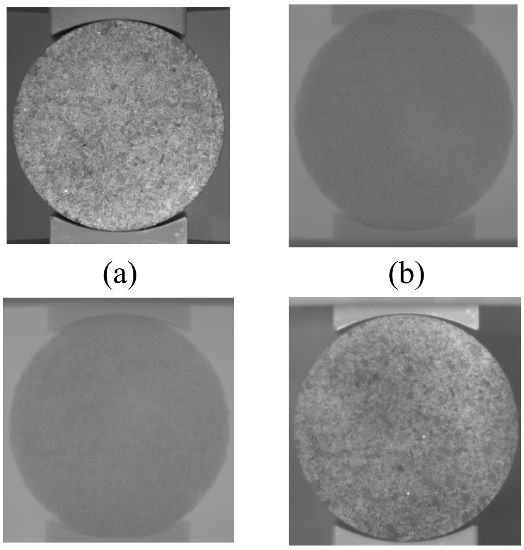

(a) (b)
(c) (d)

Fig. 3. Influence of the blue light and the blue band pass filter in the quality of the image. (a) Image at room temperature. (b) No blue light and no filter. (c) No filter. (d) Use of blue light and blue band pass filter[8].

In Fig. 4 the histograms of the images presented in Fig. 3 can be compared. When the proposed experimental design is not used, there is a high concentration of pixels values around a narrow range of values, which characterizes the lack of contrast, making the DIC calculations virtually impossible. When all devices are used, the histogram is closer to what can be obtained at room temperature, and the contrast is again recovered.

The test's procedure is composed of the following steps:

1. A pre-load of 50 N is applied on the sample, and the testing machine is put under force control to avoid thermal stresses during heating.

2. A heating rate of 10 ºC/min is applied from room temperature until 600 ºC, and 5 ºC/min are applied until the test temperature is reached.

3. A dwell time of approximately 2 h is applied, in order to homogenize the temperature in the furnace and in the sample. Fig. 5 shows that the recorded machine displacement stops to vary after around 1.5 h of dwell, indicating that the sample and the machine have stopped expanding and the temperatures have reached a steady state condition.

4. A load of 800 N is applied on the sample in 30 s, and held for 2 h.

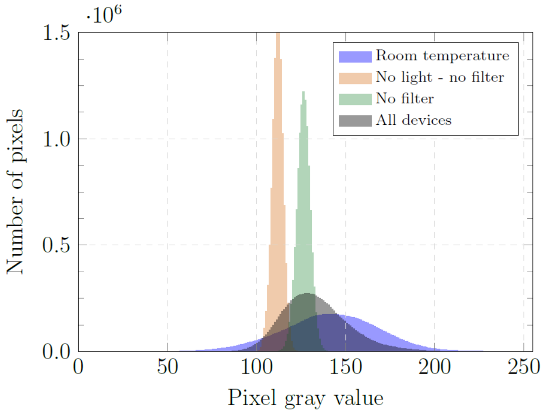


Fig. 4. Histograms of the images at room and high temperature[8].

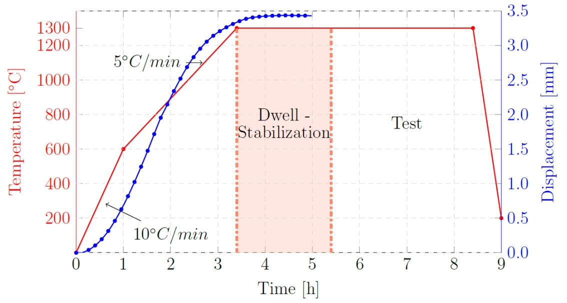
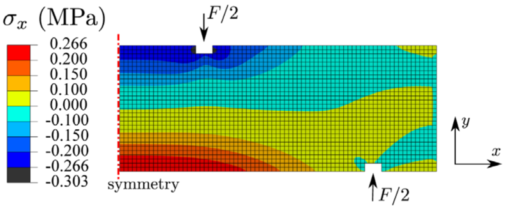


Fig. 5. Heating curve and evolution of machine displacements prior to the test[8].

EXPERIMENTAL SETUP – FOUR-POINT BENDING TESTS

Similar to the Brazilian test, the four-point bending test sample also presents a heterogeneous stress distribution, with compression at the upper part and tension at the lower part of the y axis, as shown in Fig. 6. This test has been used in association with the DIC technique to identify the mechanical properties of refractories[5]. One limitation of this test is the relatively low compression load that can be applied, since the failure of the sample is governed by the tensile stresses.



(b)

Fig. 6. Stress distribution in the four-point bending test for a linear elastic case.

Fig. 7 shows the experimental setup used to perform the four-point bending tests and to take the pictures at high temperatures. This setup was previously used by Leplay et al.[5] and is composed of:

1. A home-made furnace designed at Saint-Gobain Research Provence with capacity to heat up to 1600 °C.

2. The furnace's door is made of a lightweight insulating refractory, and is equipped with a sapphire window. If necessary, a double window can be used, to better insulate the furnace.

3. One blue light source is used to enlighten the sample and increase the contrast.

4. The pictures are taken using a RGB CMOS camera and a 300 mm macro telephoto lens with a 2x focal extension.

5. The interior of this furnace is subjected to considerable heat hazes, which generates noise in the images. To average the effect of the noise, the pictures are taken using large exposure times (for example, 20 s). To limit the amount of light arriving at the

camera's sensor, neutral density filters are used. Blue band pass filters are also used, to remove UV and IR radiations.

6. The load is applied using static weights deposited under a metallic rod, which transfers the load to an alumina rod that has one end placed inside the furnace.

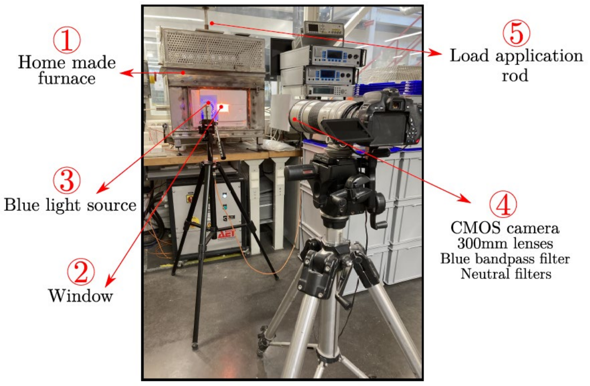


Fig. 7. Experimental setup for four-point bending tests at high temperature.

Fig. 8 shows the speckle pattern distribution in a sample at 1300 °C. As in the Brazilian test, a SiC material was used to create the speckle.

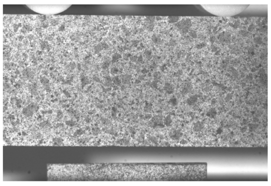


Fig. 8. Four-point bending: Example of speckle pattern at 1300 °C.

BRAZILIAN TESTS RESULTS – INVERSE IDENTIFICATIONS

To identify the tensile and compressive creep parameters, six Brazilian tests at 1300 °C were done. Two of the tests presented time vs displacement curves incompatible with the remaining four tests, and were not considered in the analysis. A finite element model of the experiment was built, and the material properties were varied until both results were similar.

Fig. 9 shows the vertical displacements at the point of load application in the sample, obtained by DIC, as well as an envelope of curves formed by possible sets of parameters of the asymmetric creep model that can fit the experiments. Fig. 10 shows the identified curve, and the identified parameters are presented in Table 1.

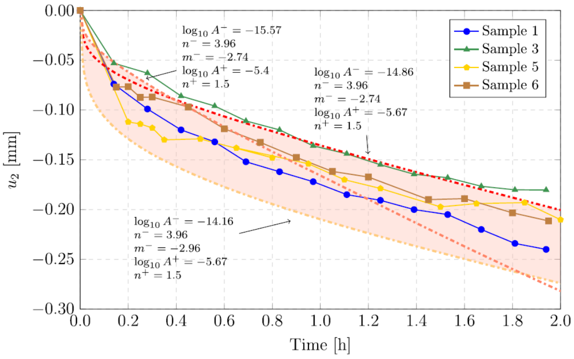


Fig. 9. Brazilian tests time vs displacement curves and parameters envelope.

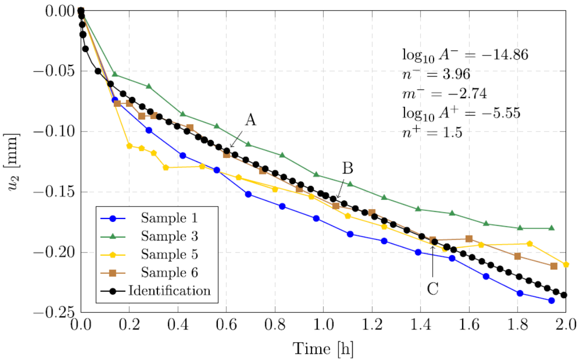
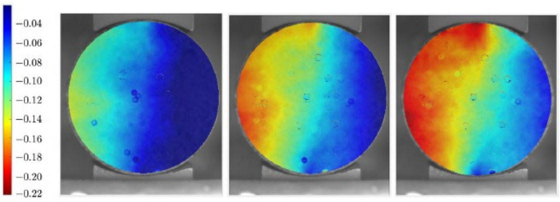


Fig. 10. Brazilian tests time vs displacement curves and inverse identification.

Table 1. Identified creep parameters.

Parameter	Comp.	Tension
$log_{10}A$[MPa^{-n}/s]	-14.86	-5.55
n [-]	3.96	1.5
m [-]	-2.74	0

Fig. 11 shows the vertical displacements in sample 6 at points A, B and C (Fig. 9), obtained by the DIC calculations. It is possible to see that the displacements are not symmetric around the vertical axis, as it would be expected in a Brazilian test sample, but the sample is rather rotated counterclockwise. This happens because of the imperfections of the experimental setup. The impossibility to obtain theoretically perfect results from experiments has been extensively reported in the literature[12], and it was expected for the current application.



(a) (b) (c)

Fig. 11. Vertical displacements in mm at 1300 °C – DIC sample 6. (a) Point A. (b) Point B. (c) Point C.

To accurately describe the Brazilian tests, the rotation of the sample needs to be included in the numerical model, and the horizontal force causing this rotation needs to be identified, together with the material parameters. Fig. 12 shows the displacement fields obtained by considering a horizontal force of –7 N at the top of the sample, which corresponds to an inclination of 0.5° in the load application piston. The displacements' maps of Fig. 11 and Fig. 12 are similar, which is a first indication that the correct material parameters were identified.

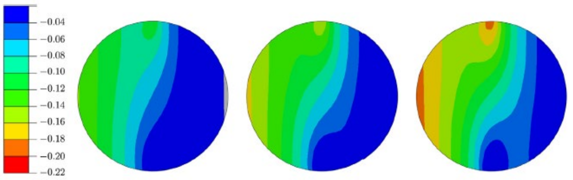


Fig. 12. Vertical displacements in mm at 1300 °C – Simulation. (a) Point A. (b) Point B. (c) Point C.

VALIDATION OF THE IDENTIFICATION RESULTS

Teixeira et al.[10] reported the compressive creep identification results for the alumina-spinel studied in this work, obtained using a one-dimensional test. In this work, the identified value for $log_{10}A$ was -14.16 MPa^{-n}/s, representing a difference of 4.8% between both results.

The advantage of the methodology proposed in this work over the one proposed in [10] is that, when the Brazilian test is used, only one test needs to be performed to allow for the identification of the creep parameters, due to the complex stress distribution in the sample; in the compressive creep test, three experiment with different stresses need to be done[13]. Obviously, due to the heterogeneities of the material, tests in each condition need to be repeated to allow for a statistical analysis of the results, which makes an identification procedure requiring less experiments even more advantageous.

To improve the quality of the validation, and particularly to validate the tensile creep parameters, four-point bending tests were performed, as described in the previous sections. Two experiments were made at 1300 °C and at a constant load of 59.3 N.

As it can be seen in Fig. 13, both tests presented problems at the beginning of loading. Sample 1 had a high rigid body motion upon the application of the load, which can be attributed to the loading procedure (use of manually positioned weights instead of a testing machine). In the second test, the loading was erroneously started before the temperatures in the furnace and in the sample were homogeneous, therefore leading to the measurement of a vertical displacement upwards (Fig. 14).

Because of these problems, the first four hours of the tests were neglected during the validation, and the slopes of the force x displacement curves were compared.

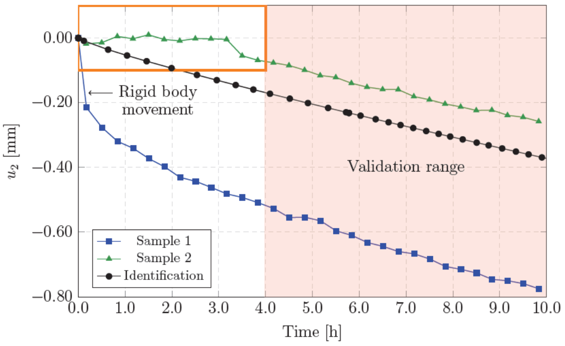


Fig. 13. Bending tests: Vertical displacements at 1300 °C and 59.3 N.

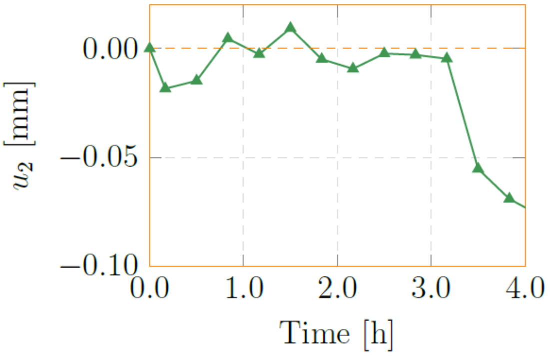


Fig. 14. Vertical displacements at 1300 °C and 59.3 N – Detail of the first 4 hours.

Fig. 15 shows the slopes of the curves relative to the two experiments, as well as the curve obtained with a finite element simulation of the experiments using the identified material parameters. The slope based on the identified parameters is comprised between the two experimental slopes. It enables us to validate the parameters identified with the Brazilian test. However, more tests would be required for a better statistical approach, since there is 30% of difference between the slopes of the two bending tests. The simulation results presented a similar slope as the first test, with a deviation of 3%.

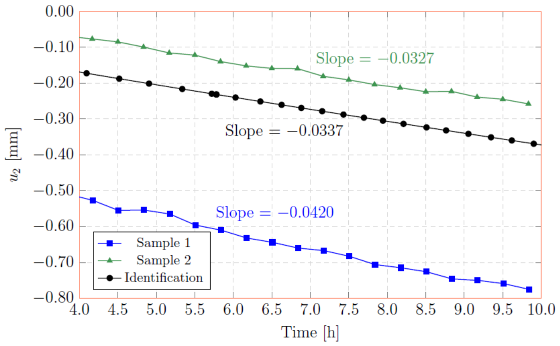


Fig. 15. Vertical displacements at 1300 °C and 59.3 N – Validation range.

CONCLUSIONS

In this work, the material properties of an alumina-spinel material were identified, related to an asymmetric creep law. An identification procedure composed of Brazilian tests and a DIC technique was used. This methodology requires less experiments for the identification of the material parameters, when compared to traditional one-dimensional techniques, which is a considerable advantage for the characterization of refractories at high temperatures.

The identified results were compared to data available in the literature and to the results of four-point bending tests, and the parameters could be validated. To increase the quality of the validation, more bending tests need to be performed, to allow for a better statistical treatment of the results.

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