Creep Characterization of Refractory Materials at High Temperatures Using the Integrated Digital Image Correlation

Lucas Teixeira*, Jean Gillibert, Eric Blond and Thomas Sayet
Univ. Orléans, Univ. Tours, INSA-CVL, LaMé

Abstract

The study of the nonlinear mechanical behavior of materials at high temperatures is still a challenge for the refractories industry, since the applied tests are expensive and time consuming. Moreover, the characterization of the tensile behavior of such materials is especially difficult, due to requirements related to the load application and alignment, to avoid premature failure of the samples. In this paper, the Integrated Digital Image Correlation (I-DIC) in association with Brazilian tests is proposed as an alternative to classic unidimensional tensile and compressive creep tests to obtain the mechanical properties of refractories at high temperatures. The difficulties on the application of this technique, related to the experimental and computational parts, are explained and solutions are proposed to overcome them. An example of identification of creep parameters at high temperature is provided to show the potentialities of this technique.

1. Introduction

The aim of this work is to propose an alternative to the most common procedures for the characterization of the creep behavior of materials, i.e., uniaxial tensile and compressive tests, using an I-DIC technique and Brazilian tests.

Generally, the creep behavior of materials can be split in three stages. The first stage, called primary creep, presents a time-dependent strain rate which decreases with time. In the secondary creep stage, the strain rate is considered to be constant. Finally, in the third creep stage, the strain rate increases with time until the failure of the material[1].

For ceramic materials, including refractories, it has been demonstrated by Blond et al. [2] that the creep strain rate at one-dimensional tension load is considerably higher than the strain rate caused by a load with same absolute value in compression.

To study the creep behavior of refractories, Jin et al.[1] developed a high temperature compressive testing machine able to apply loads up to approximately 20kN. Later, Mammar et al.[3] developed a dedicated tensile testing device to measure creep strains at high temperatures.

Alternatives to the unidimensional tensile and compressive creep tests were already proposed in the literature. For instance, Dusserre et al.[4] used compression and bending tests to identify the parameters of a Drucker-Prager creep law for a fiber reinforced refractory concrete at 1200°C. Leplay et al.[5] used Finite-Element based DIC (Q4-DIC) and beam kinematics based DIC (Beam-DIC) in combination with bending tests to quantify the creep asymmetry of a zircon ceramic at 1350°C, but no identification of material's parameters was performed.

Gazeau et al.[6] used an I-DIC technique combined with Brazilian tests to identify the linear elastic behavior of an ion transport membrane at 900°C. In the current work, a similar approach will be used and applied to study the creep behavior of refractory materials at 1200°C.

In general, Digital Image Correlation techniques consist of taking one picture of the sample, normally coated with a speckle pattern to increase its visual contrast, before the beginning of the test, called reference image. Later, several other pictures are taken during the application of the load and consequent deformation. Using a specialized software, the deformed images are compared to the reference image, and the displacements' and/or strains' fields are calculated.

The I-DIC algorithm differentiates from other DIC techniques in the sense that the goal isn't to calculate strains or displacements in the sample's surface, but to identify the material's parameters suitable to fit a constitutive law in multidimensional stress states. Also, to overcome the problem that refractories present considerably low strains (in the range of 10^{-6}), which affects the accuracy of classic DIC techniques, I-DIC uses directly the images' pixels as input variables, since they are the raw data and, therefore, the less affected by previous calculations. The I-DIC algorithm will be explained in more details in the next sections.

Compared to traditional test methods to measure the creep behavior of materials, i.e., unidimensional tension and compression tests, the I-DIC method combined with Brazilian tests has two main advantages :

- It provides information about the complete deformation field, and not only about one point, such as the case of extensometers. This makes it ideal to compare with numerical simulation results.
- The number of tests required to characterize the material is considerably reduced. For example, to identify the behavior of a material following the Norton-Bailey law for primary creep, which contains three material's parameters[7], at three different

temperatures and repeating each test three times to improve the reliability of the results, it's necessary to perform 54 tests. On the other hand, to characterize the material under the same circumstances using I-DIC with Brazilian tests, it's necessary only 9 tests. This is explained by the fact that, in the latter case, the sample is subjected to a heterogeneous stress field, containing at the same time tension, compression and shear components, therefore the identification of the parameters in all stress states is done at the same time.

On the other hand, there are some difficulties related to the use of image treatment at high temperature. The most important of them is related to the difficulty to obtain suitable images that present a high contrast, what is mandatory to obtain good results from the DIC software.

Another problem is that it's necessary to have some previous knowledge about the behavior of the material before running an identification calculation, since I-DIC requires that a constitutive model is defined *a priori*. Generally, this previous knowledge is available for refractories.

Finally, it should be mentioned that both methods of identification face challenges when a constitutive law with many variables needs to be characterized, because normally an optimization algorithm is used to fit the experimental data to the model's equations. In the case of I-DIC in combination with Brazilian tests this problem is more critical, since the equations can't be simplified and integrated analytically, as it can be the case for the traditional tensile and compressive tests[1].

2. Methodology

In this work, the potentialities of the I-DIC technique will be demonstrated using two case studies:

- Case study 1: the reference image was generated using a software, therefore being called virtual image, to guarantee that the speckle pattern was optimized to generate the best contrast possible, which improves the quality of the calculations. A numerical simulation was done using a set of creep parameters, and the consequent deformation field was virtually applied to the reference image. The goal was to demonstrate that the I-DIC technique is able to identify this same set of material's parameters, given a certain tolerance, if high contrast images can be provided to the algorithm.
- Case study 2: it uses the same methodology to virtually deform the reference image, but this time real images, taken at 1200°C, were used as the reference image.

2.1 I-DIC algorithm

The I-DIC algorithm, illustrated in **Fig. 1**, is based on the idea of identifying the material's parameters at the same time as the displacements' field is calculated, therefore the name "Integrated".

As an input, one should provide the images obtained during the mechanical test (the Brazilian test, in the case of this work), as well as a reference image, which corresponds to the sample's state before the deformation starts. Also, the analyst provides the load applied at the sample and an initial guess for the material's parameters.

The next step is to create a numerical model using, for example, the Finite Elements Method, which reproduces the mechanical test. It should be noticed that experimental errors can influence the accuracy with which this numerical model represents the real test. Using the initial guess and the experimental load, one should run a simulation and obtain a displacements' field at the sample's surface.

This displacements' field is, then, applied to the reference image, using image treatment techniques to interpolate its gray levels in a way to represent the deformed state. This process results in the so called theoretically deformed image.

The pixels' levels of this image are compared to the ones of the experimental image, and a difference between them is calculated. If this difference is below a certain tolerance, it's considered that the material's parameters used to make the simulation are suitable. If not, a new guess for these parameters is calculated by an optimization algorithm, and the method starts the loop again.

In this work, we used a genetic optimization algorithm as a way to map the entire domain of the variables. For the sake of brevity, no details about this algorithm will be provided here.

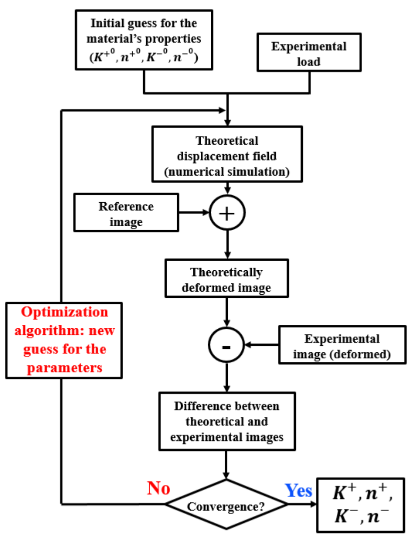

Fig. 1 I-DIC algorithm

2.2 Brazilian tests at high temperature

The experimental setup for the Brazilian test, shown in **Fig. 2**, consists of a regular INSTRON 4507 testing machine and a dedicated furnace that contains a window, allowing us to take the pictures of the sample.

Regarding the optical equipment, we use a CCD camera coupled with a 200mm lens. At high temperatures, the blackbody radiation coming from the sample is considerably high, and the image presents no contrast[5]. Therefore, we used a blue bandpass filter with useful range for wavelengths between 425 and 495nm to reduce the blackbody radiation being captured by the camera sensors, and at the same time we used two blue spotlights to enlighten the sample and to increase the contrast.

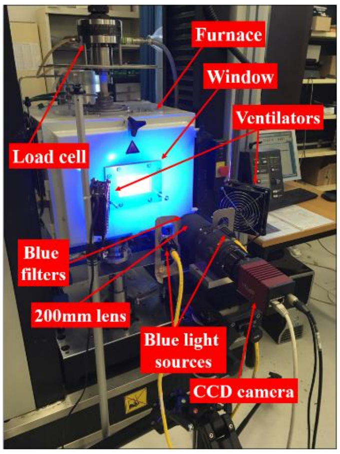

Fig. 2 High temperature test setup

Fig. 3a shows the virtual image used to perform the calculations, such as a zoom highlighting its pixels' gray levels' distribution. In the same figure, the real images are presented. To increase the sample's contrast, we used a speckle pattern made of a SiC powder. **Fig. 3b** shows the speckle at room temperature, **Fig. 3c** at 1200°C without the use of the blue lights, and **Fig. 3d** at 1200°C with the blue lights. It's possible to notice that, without using the blue lights, the sample presents a very low contrast at high temperature, which is enhanced when they are used.

Fig. 3e shows the histograms of the corresponding images, which is one indicator of the image's quality. It should be noticed that the histogram of an ideal image, i.e., a virtual image, covers all pixels' values with an equal number of pixels in each one. At 1200°C and without using the blue light, the histogram becomes very sharp, which is, to a certain extent, improved using the lightening.

2.3 Numerical simulations

In this work, we considered that the material presents an asymmetric creep behavior between tension and compression, which is consistent with refractories. To do that, we used a creep law developed by Blond et al. [2], which uses a split in the stress tensor to create one tensor with tensile components and one with compressive components, together with two distinct von Mises like yield surfaces to calculate the viscoplastic strain rate, given by Equation (1):

$$\dot{\boldsymbol{\varepsilon}}^p = \frac{3}{2}\frac{\boldsymbol{s}^+}{\sigma_{eq}^+}\left(\frac{\langle\sigma_{eq}^+ - \sigma_y^+\rangle}{K^+}\right)^{n^+} - \frac{3}{2}\frac{\boldsymbol{s}^-}{\sigma_{eq}^-}\left(\frac{\langle\sigma_{eq}^- - \sigma_y^-\rangle}{K^-}\right)^{n^-} \tag{1}$$

where $\boldsymbol{s}^+$ is the deviatoric part of the tensile stress component, σ_{eq}^+ is the equivalent stress, σ_y^+ is the yield stress and K^+ and n^+ are material's parameters. The same applies to the variable with negative index, with the difference that they represent the compression components.

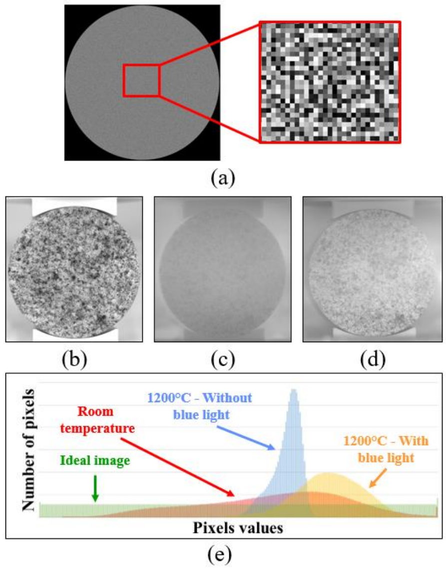

Fig. 3 Brazilian test samples used with I-DIC. (a) Virtual image. (b) Room temperature. (c) 1200°C without using the blue lights. (d) 1200°C using the blue lights. (e) Images' histograms.

In the present case, the yield stress was considered to be zero both for tension and compression. Therefore, four parameters are to be identified, being K^+, n^+, K^- and n^-, which is already challenging from the point of view of the optimization algorithm.

Table 1 presents the reference values used to generate the virtual images, as explained in Section 2. It also presents the range of variation for these values used in the optimization algorithm. In other words, it was considered that we know that the correct value is in between these ones, and that the work of the I-DIC algorithm is to find a set of parameters that will lead to more accurate simulations using this material.

The elastic properties of the material were considered to be fixed, and their values are Young's modulus $E = 30GPa$ and Poisson's ratio $\nu = 0{,}2$.

Table 1 Reference values for the creep properties of the material

	Reference values	Range of variation
Tension	$K^+ = 5773.5$	$K^+_{min} = 5000$
		$K^+_{max} = 6000$
	$n^+ = 2$	$n^+_{max} = 2.2$
		$n^+_{min} = 1.8$
Compression	$K^- = 1.0 \times 10^6$	$K^-_{min} = 5.0 \times 10^5$
		$K^-_{max} = 5.0 \times 10^6$
	$n^- = 1.5$	$n^-_{max} = 1.8$
		$n^-_{min} = 1.2$

3. Results and discussion

The results of the identification procedure using the virtual images are shown in **Table 2**. The asymmetric creep law has already proved to have several local minima, therefore making the identification process more difficult. In practice, this leads to the fact the results of the identification should be validated by another experiment, such as a bending test, for example. Therefore, the three best sets of parameters were selected as potential candidates. As it can be observed, all of them provide an accurate answer, which proves that the I-DIC method can be used to perform such kind of inverse identification. It is worth noting that the traditional direct and indirect tests should also be validated by a second load case.

Table 2 Results of the identification procedure – Virtual images

	Set 1		Set 2		Set 3	
	Value	Error	Value	Error	Value	Error
K+	5648.6	-2.16%	5431.3	-5.93%	5207.40	-9.81%
n+	2.00	-0.22%	1.96	-2.22%	1.99	-0.28%
K-	9.65E+05	-3.54%	9.41E+05	-5.85%	1.15E+06	14.80%
n-	1.52	1.11%	1.54	2.83%	1.49	-0.44%

Table 3 shows the results for the parameters' identification using real images taken at 1200°C. Such as in the case of virtual images, the algorithm was able to converge to accurate solutions. Therefore, we can conclude that the intrinsic loss of contrast at high temperatures didn't affect significantly its robustness.

Tables 2 and **3** show that, in general, the accuracy of the identification for the parameters n is higher than the ones for the parameters K. A sensitivity analysis was made, and it shows that these parameters have 99.3% of influence in the results and, therefore, are prioritized by the algorithm. The accuracy of the parameters K can be increased during the validation test.

Table 3 Results of the identification procedure – Real images at 1200 °C

	Set 1		Set 2		Set 3	
	Value	Error	Value	Error	Value	Error
K^+	5127.6	-11.19%	5543.3	-3.99%	5264.70	-8.81%
n^+	2.01	0.27%	1.99	-0.46%	1.99	-0.58%
K^-	9.71E+05	-2.91%	1.25E+06	24.97%	1.08E+06	8.14%
n^-	1.51	0.71%	1.48	-1.10%	1.51	0.35%

4. Conclusions

This work presented the I-DIC technique combined with Brazilian tests as an alternative to traditional tensile and compressive tests to characterize the creep behavior of refractory materials.

Through the presentation of two computational examples, it was possible to conclude that this method is promising, as it can reduce the number of total tests required in the characterization of the material, still preserving a considerable level of accuracy.

As a next step, our group will focus in the characterization of a refractory material using real experiments at high temperatures.

Acknowledgments

This work was supported by the funding scheme of the European Commission, Marie Skłodowska-Curie Actions Innovative Training Networks in the frame of the project ATHOR - Advanced THermomechanical multiscale mOdelling of Refractory linings 764987 Grant. The authors are thankful to the Artemis cluster of Centre Val de Loire region (France) for the computation time.

References

1) S. Jin, H. Harmuth, D. Gruber, Journal of the European Ceramic Society, **34** 4037-4042 (2014).
2) E. Blond, N. Schmitt, F. Hild, P. Blumenfeld, J. Poirier, Journal of the European Ceramic Society, **25**[11] 1819-1827 (2005).
3) A. S. Mammar, D. Gruber, H. Harmuth, S. Jin, Ceramics International, **42** 6791-6799 (2016).
4) G. Dusserre, O. Valentin, F. Nazaret, T. Cutard, Journal of the European Ceramic Society, **36** 2627-2639 (2016).
5) P. Leplay, O. Lafforgue, F. Hild, J. Am. Ceram. Soc., **98**[7] 2240-2247 (2015)
6) C. Gazeau, J. Gillibert, E. Blond, P.-M. Geffroy, N. Richet, Journal of the European Ceramic Society, **35** 3853-3861 (2015).
7) J. Lemaitre, J.-L Chaboche, Mechanics of Solid Materials, Cambridge University Press, (1994)