Strain Fields' Measurements: Novel Engineering Approach to Enrich the Characterisation of Refractories with Non-Linear Mechanical Behaviour

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The present work aimed to apply digital image correlation (DIC) used for kinematic fields' measurements as a support for the experimental characterization of refractory materials with specific non-linear behaviour. Model and industrial materials with different degrees of flexibility were studied. The first type of materials was a single phase model flexible aluminium titanate material (AT VF) developed for academic purposes by improving the grain growth. Its non-linear mechanical behaviour was obtained thanks to the thermal expansion mismatch of its grains according to the different crystallographic axis. The second one is multi-phased magnesia based industrial materials, whose flexibility is less accentuated, and for which the non-linear mechanical behaviour is obtained thanks to the thermal expansion coefficients mismatch between spinel aggregates and magnesia matrix. In order to apply the optical methods on these materials which exhibit lower strain-to-rupture, it was necessary to optimise the accuracy of these techniques by improving experimental conditions.
In the case of AT VF, DIC has been applied on four-points bending test at room temperature to underline the material asymmetric mechanical behaviour which induces a significant shift of the neutral fibre and to evaluate the relative displacement of rolls. The application of DIC has been extended to other experimental testing method such as Brazilian and wedge splitting test using the multi-phased magnesia-based materials. This highlighted fracture mechanisms (crack occurrence and propagation) and the presence of crack branching phenomenon promoted thanks to an initial microcracks network voluntary introduced by thermal expansion mismatch between the different phases so as to improve their thermal shock resistance. From displacement experimentally obtained by DIC, a finite element method updating (FEMU-U) has been developed to determine material properties.

1 Introduction

Thermal shock resistance is a key parameter to enhance service life of refractories in their industrial applications. The improvement of this property is usually directly related to a specific mechanical behaviour induced by a voluntary microcracked microstructure. Indeed, depending on microcracking level, the mechanical behaviour will change from pure elastic to a "non-linear" one. This non-linear mechanical behaviour results of the complexity of the different inelastic phenomena occurring during fracture mechanisms and which consumes a large amount of energy as the crack front is progressing so as to improve the service life of the refractory.

In order to understand the behaviour of these materials, an accurate determination of their constitutive laws is necessary to feed finite element method (FEM). To do so, it was necessary, during the past 15 years, to develop uniaxial tests taking

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into account that these materials are characterised by a low level of strain-to-rupture. Nowadays, accurate instrumented tensile test using extensometers are available. However, such tensile devices involve some complexity for tests management (sample machining, well aligned loading grips and equipment maintenance). Fortunately, the occurrence of optical techniques such as digital image correlation (DIC) [1], which allows to obtain the overall strain fields on a given loaded sample by operating digital images obtained before and after deformation using different means of non-contact optical acquisitions, and can be coupled to FEM modelling, give today new opportunities for experimental investigations of refractory materials exhibiting a non-linear mechanical behaviour.

In this context, the challenge of the present work was to enrich the mechanical characterization of such refractory materials with kinematic fields by applying DIC on quite common mechanical tests in this scientific community: four-points bending test, Brazilian test and wedge splitting test. DIC was then used to contribute in proposing an innovative approach of refractory characterisation by combining energetic and mechanical aspects with strain fields' measurements.

2 Experimental

2.1 Materials

Different materials with a non-linear mechanical behaviour have been chosen: a "Very Flexible" (VF) model refractory material based on Aluminium Titanate (AT) developed for academic purpose has been considered. It has been chosen as it is a synthetic material of potential interest and it is suitable for high temperature applications where thermal shock resistance is required. For instance, it is used in Diesel Particular Filters (DPF). It is obtained by synthesis of an equimolar mixture of Al_2O_3 and TiO_2 at temperatures above 1280 °C. AT materials develops a non-linear mechanical behaviour due to microcracks induced by the high anisotropic thermal expansion of its grains after sintering (Along the *a* axis: $\alpha_a = -2{,}9 \times 10^{-6}\ K^{-1}$, along the *b* axis: $\alpha_b = 10{,}3 \times 10^{-6}\ K^{-1}$ and along the *c* axis: $\alpha_c = 20{,}1 \times 10^{-6}\ K^{-1}$) [2].

In addition to AT VF, industrial Magnesia-Spinel (MSp) refractories which are chrome-free materials were studied. They exhibit an improved thermal shock resistance unlike pure magnesia refractories, and it has been confirmed that two to three times longer service life can be obtained, compared to a conventional magnesia-chrome brick. The reason for the improved thermal shock resistance has been linked to the large difference in thermal expansion coefficient between magnesia (~$13{,}5 \times 10^{-6}$ °C^{-1}) and $MgAl_2O_4$ (~$7{,}6 \times 10^{-6} \times$ °C^{-1}). On cooling from sintering temperature (about 1700 °C), the thermal expansion mismatch leads to microcracks development around the spinel grains. Magnesia spinel refractory materials are used mainly in the cement kilns in particular in the cooling zone and the upper part of kilns.

2.2 Experimental techniques

2.2.1 Four points bending test

The mean purpose of the four-point bending test is to study the tension-compression mechanical behaviour. Unlike the three-points bending test, it has a uniform stress distribution between the higher supports of bending device and a zero shear in this area. According to ASTM C 1161-02c, the four-points bending device allows independent articulation and pivoting of all rollers about the specimen long axis so as to match the specimen surface. In fact, the upper or lower pairs of rollers are free to pivot to distribute force evenly to the bearing cylinders on either side; avoiding the measurement of additional frictional.

The bar-shaped samples with the rectangular cross-section of 30 mm × 40 mm × 160 mm were tested with a maximum effort of 50 kN. A constant cross-head displacement velocity of 0,05 mm/min was employed. To measure strain using conventional gages, two strain gages have been bonded on the top and the bottom faces in the middle of the sample. The bending stress σ (MPa) and the Young's modulus E (GPa) are deduced using the relationships established within the framework of linear elastic beam. It assumes a symmetric tensile-compressive behaviour and leads to incorrect estimations in case of asymmetric non-linear behaviour [3].

2.2.2 Wedge splitting test

Since the thermal shock resistance of refractories has to be improved, the brittleness of these materials has to be reduced and the deformation before cracking has to be higher. The critical energy release rate is one parameter that characterises the fracture resistance of the material. One possible technique to measure this specific fracture energy is wedge splitting test (WST). This technique is more adapted for refractories [4].

The WST consists of opening a crack using a wedge. It allows investigating the formation of a macrocrack and its stable propagation within the sample. The damage is supposed to be mainly concentrated in the plane section below the notch. The principle of this test is to apply a vertical force (F_V) received from the device which is transformed in a much higher horizontal force (F_H) causing a symmetrical opening mode of the crack. Symmetrical loading configuration and sample geometry allows a pure opening mode by avoiding shear mode.

2.2.3 Brazilian test

Brazilian test is one testing methods applied as an indirect tensile test. It was mainly applied to characterise rocks and concrete [5]. This test method consists of applying a diametric compressive force along the length of a cylindrical concrete specimen at a rate that is within a prescribed range until failure occurs. This loading induces tensile stresses on the plane containing the applied load and relatively high compressive stresses in the area immediately around the applied load [5]. Tensile failure occurs rather than compressive failure.

The main advantages of this test are: it is easy to set up and allows avoiding expensive and random machining of brittle samples unlike direct tensile test. When a compressive load is applied on a cylinder, a material element along the diameter coincident with the application of the load is subjected to a compression stress σ_c and a tensile stress σ_t (uniform along the diameter). Following ASTM testing method, tensile strength was calculated using eq. 1. This expression assumes the material to display linear elasticity until failure and that maximum tensile stresses are therefore induced normal to the loaded diameter such that failure is initiated at the centre of the disc.

$$\sigma_t = \frac{2.P}{\pi.e.D} \qquad \text{(eq. 1)}$$

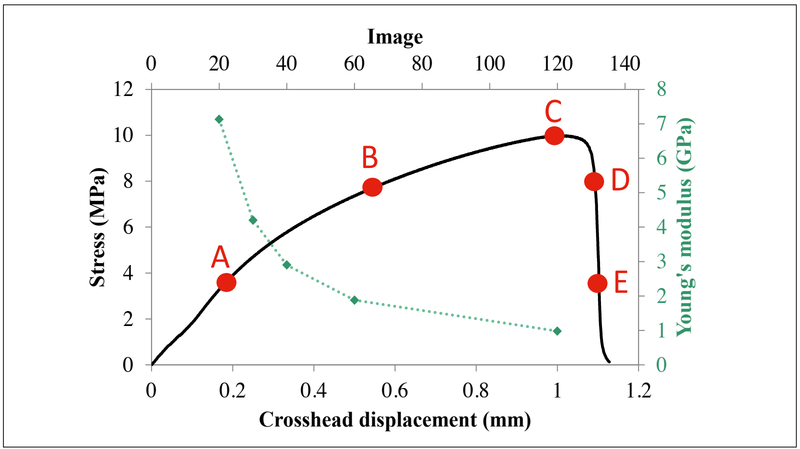

Fig. 1 Experimental stress-displacement curve and evolution of Young's modulus of AT VF obtained by FEMU-U

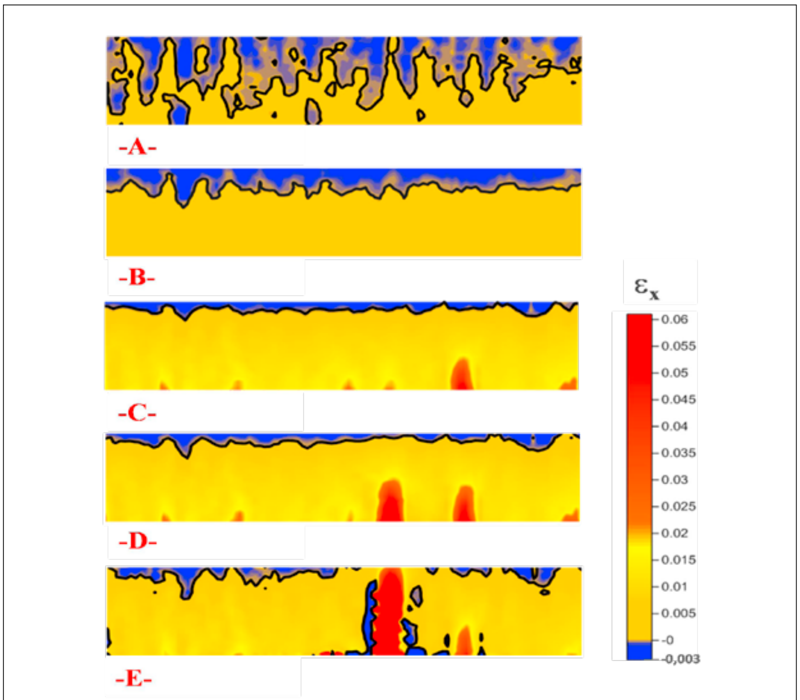

Fig. 2 Maps of strains along X-axis for the five times defined in the stress-displacement curve (a)

The cylindrical samples with a diameter of 50 mm were tested with a maximum effort of 50 kN. A constant crosshead displacement velocity of 0,05 mm/min was employed.

2.2.4 Digital Image Correlation (DIC)

Aside from the widely used strain gauge technique, various full-field non-contact optical methods have been developed. In this paper, the experimental measurements were obtained thanks to DIC for fields strain measurements [6, 7]. DIC is based on the analysis of successive digital images of a same sample during a mechanical test. The displacement fields are obtained by measuring the degree of similarity of series of subsets between the image corresponding to an unloaded state and the deformed image recorded during the test. Concerning the sample preparation, a white-on-black speckle pattern was applied on the prepared surface using fine aerosol paint. Upon DIC system setup, every droplet contains around 3–15 pixels and additional LED (cold source) lighting was used in order to maintain sufficient contrast of the pattern.

In addition to the mechanical experimental setups, a digital 8-bit CMOS camera (2560 pixels × 1920 pixels) is placed in front of the prepared sample surface to record images during the loading, in order to perform optical measurements to obtain kinematic fields. The acquisition frequency was 1 image per second. The principle of the technique and sample preparation are detailed elsewhere [8].

2.2.5 Finite Element Method Updating (FEMU-U)

FEMU-U [9] is one of the updating methods of identification. It consists in performing iteratively FEM calculation in order to minimize the difference between experimental and simulation data using an objective function. This latter determines the convergence satisfaction criterion. In our case, the compared results are experimental (U_{EXP}) and simulation (U_{FEM}) displacements. U_{EXP} corresponds to those obtained by DIC. The very first FEM calculation is computed thanks to an initial parameter set P_i (to be chosen by the operator) and then, taking into account the value of the objective function, new parameter set P_{i+1} is determined for the following calculation up to the satisfaction of the convergence criterion.

3 Results

The first step of the study was to demonstrate the efficiency of the DIC technique as an effective tool to enrich the mechanical analysis of refractories. To do so, the nonlinear mechanical behaviour of AT VF has been studied during four-points bending test at room temperature.

From Fig. 1, since the applied stress reaches 4 MPa, the mechanical behaviour becomes non-linear that leads a higher flexibility until the rupture. This latter is not brittle and the post-peak region is more marked. Thanks to DIC, maps of longitudinal strain have been obtained at different steps of the test and were represented in Fig. 2.

The upper part of sample is submitted to horizontal compressive stresses, while the bottom is subjected to a tensile one.

At the beginning of the test (stage A) which corresponds to the linear part of the mechanical curve, compression and tension are balanced and are symmetrically distributed with respect to the zero stress line well-known as "neutral fibre" represented with the black line. In stage B, which corresponds to the beginning of the non-linear behaviour, strains in the bottom of sample are becoming higher in comparison with the compressive strains inducing the asymmetric distribution of tensile and compressive stresses. In stage C, the asymmetric behaviour is stronger and there is localisation of zones with high strain level which are concentrated where the crack may appear on the surface. Red colour is not related to a real deformation, but corresponds to crack opening. Stages D and E have been chosen in order to describe the post-peak behaviour of the material which appears as representative of not brittle material rupture. In stage D, strain level around crack opening which will induce material rupture becomes higher in comparison with zones characterised with high strain level. In stage E, this crack will propagate until material rupture. High strain values localized in the zones become low as the applied load is decreasing. The evolution of the neutral fibre has been obtained by DIC and compared with an aluminium sample (AU4G) characterised with a symmetrical behaviour (Fig. 3).

At the beginning of the test, for both samples, neutral fibre is localized around the middle due to the symmetric tensile-compressive stress distribution. This part corresponds to an elastic behaviour. In the case of aluminium sample, this stress distribution still remains for the whole test and induces a constant neutral fibre position. However, neutral fibre position for AT VF tends to move up from a specific stress value (stage A in Fig. 3) due to the asymmetric distribution of compression-tension stresses. This can be justified by the equality between the areas under compressive and tensile stresses which has to be satisfied. This explains the reason why the classical equation for stress estimation and elastic properties determination are not valid in the case of such behaviour, and using Finite Element Method Updating (FEMU-U) based on coupling DIC with FEM and an optimization algorithm to determine elastic properties.

Indeed, from displacement fields obtained by DIC, a simulated geometry and meshing identical to the experimental one has been generated using Python script. The evolution of the obtained Young's modulus, which represents material rigidity, values

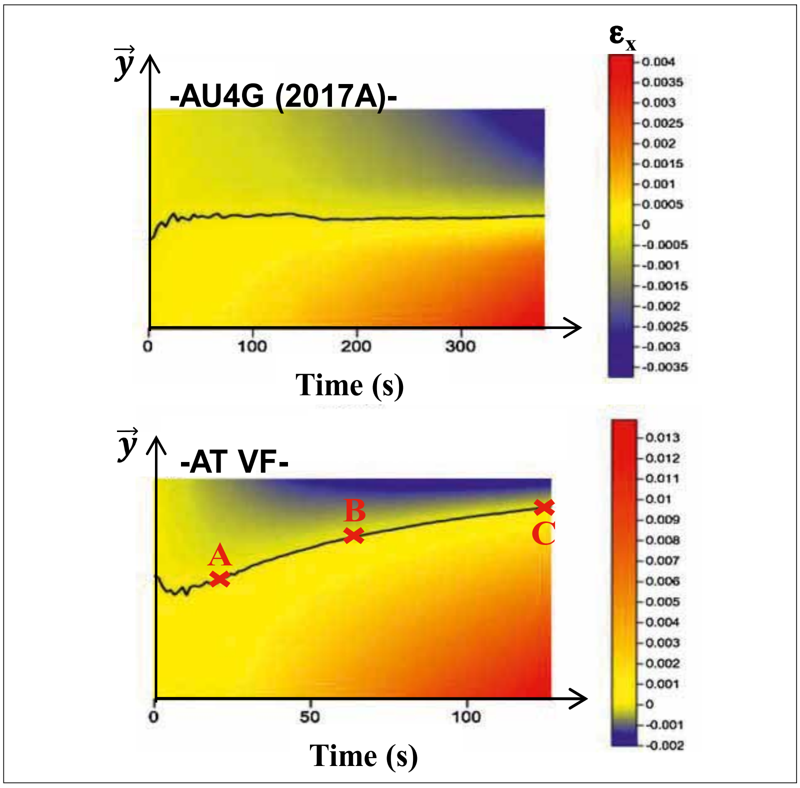

Fig. 3 Evolution of strain fields during the test for an aluminium sample (a), and AT VF sample (b)

Tab. 1 Fracture parameters of pure MgO and MSp obtained using wedge splitting test

	σ_{NT} [MPa]	G_f [N·m^{-1}]
Pure MgO	14,36	206,5
MSp	6,65	361

for five stages is represented in Fig. 1. As the applied load increases, Young's modulus decreases due to the development of microcracks network already present in the AT VF and so, damage level is enhanced.

The acquired DIC knowledge and expertise from this first part of the project has been used and improved in order to characterise industrial magnesia spinel refractories with a flexibility which is less accentuated than AT.

The wedge splitting test has been chosen to analyse crack propagation and evaluate fracture energy. It confirms that the nominal notch tensile strength decreases and the fracture resistance increases with the spinel rate (Tab. 1). Besides, the higher values of energy in the case of MSp are due to the dense network of microcracks created during the crack initiation and propagation. Different maps of strains along X-axis were obtained using DIC (Fig. 4).

In the case of pure MgO, there is only one crack that propagates in a vertical direction. However, the strain maps show that the crack in the case of MSp materials doesn't propagate only in a vertical direction and has a specific mode of propagation: "crack branching" phenomenon in the case of MSp resulting of many interacting microcracks or microbranches (coalescence of microcracks). This confirms the complexity of crack growth process in heterogeneous materials due to the presence of several phases with different properties (thermal expansion coefficient and stiffness). Then, this can explain the high value of dissipated energy exhibited by MSp materials.

Brazilian test has been enforced for many years as an industrial easy test to obtain reliable values of tensile strength for refractories. The idea here is to combine this test with DIC to characterise the tensile behaviour of MSp. The optimization of ex-

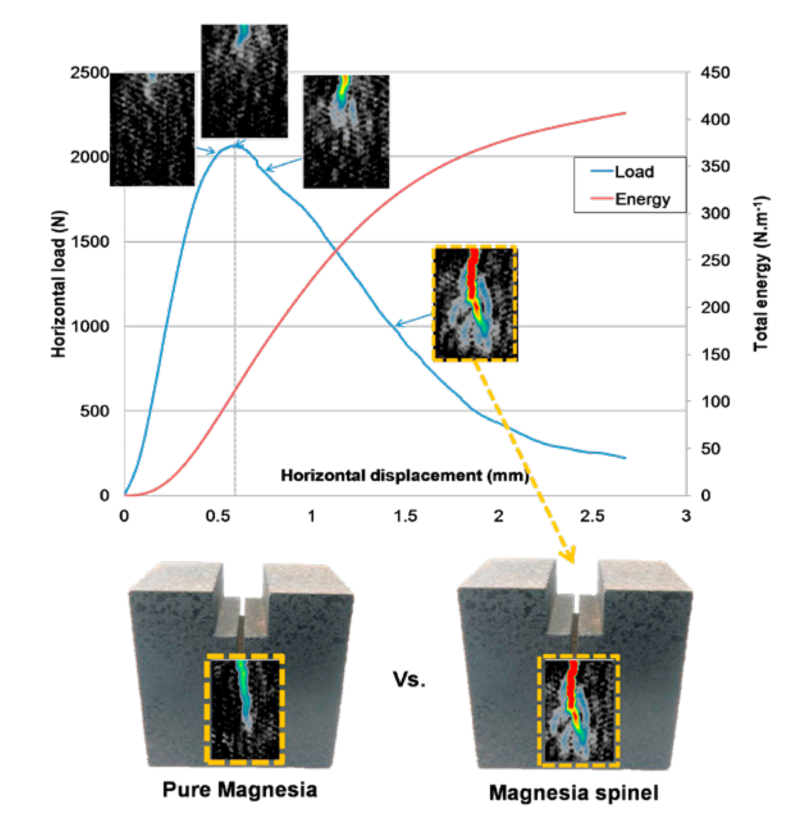

Fig. 4 Horizontal load-displacement and energetic curves for the MSp sample; the strain maps obtained along X-axis are represented in different steps during the wedge splitting test

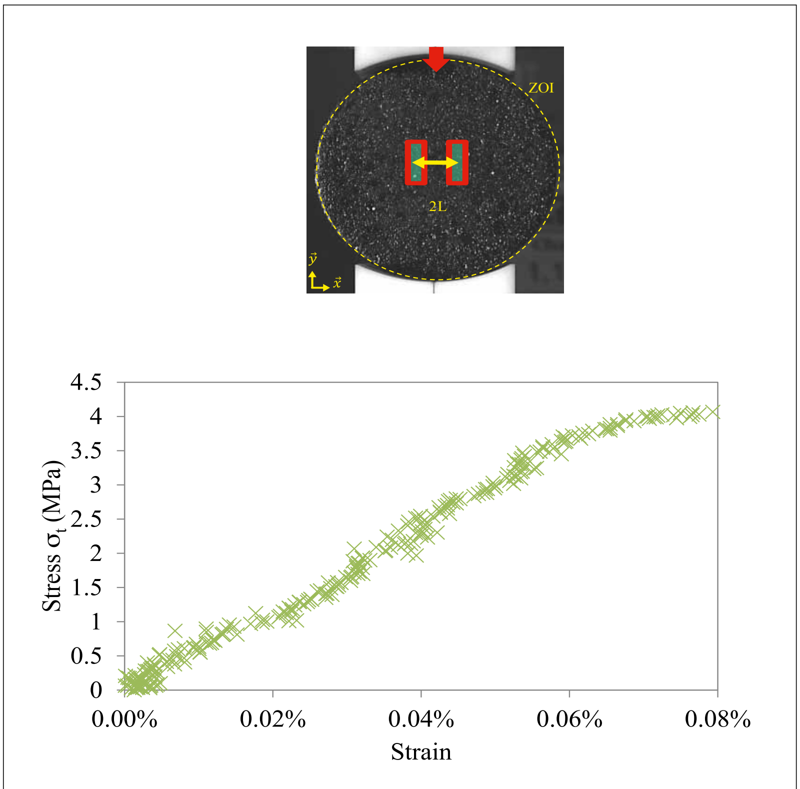

Fig. 5 Stress-strain curve obtained using DIC during a Brazilian test

perimental conditions leads (surface sample preparation, DIC process parameters, and external sources of perturbance) to improve the accuracy of measurements ($\Delta l/l \sim 10^{-5}$). In order to obtain strain-stress curve (Fig. 5) using DIC, the correlation windows have been localized symmetrically from the vertical axis of the sample.

The obtained results show a rather linear behaviour at the beginning of loading. Then, a non-linearity appears around an applied stress of 3 MPa, which leads to a tensile strength of 3,98 MPa. These results are in agreement with a reference value obtained by four-points bending test.

Considering the linear part, elastic properties of a material can be calculated taking into account the biaxial loading in the central part of the sample. Young's modulus has been determined, and the measured value was $E_t = 39{,}6$ GPa which is quite similar to the reference value obtained by ultrasonic measurements.

4 Conclusion

Due to their interesting properties for industrial applications and researchers, different materials with non-linear mechanical behaviour were investigated using different mechanical tests. Their microstructure could be used as a model for refractory materials to improve their thermal shock resistance and to enlarge their applications. This study highlights the asymmetric and non-linear behaviour during four-point bending test, Brazilian test and wedge splitting test at room temperature thanks to digital image correlation. This allows having access to material's properties during the experimental tests in one hand. On the other hand, digital image correlation could be considered as an efficient tool to measure strains and can replace traditional methods of measuring local strains such as electrical resistance strain gages and linear variable differential transformers (LVDT's), which are limited to a finite number of linear 2D-gauge lengths per sample due to space restrictions. Besides, DIC presents a more realistic strain behaviour which allows underlining the complexity of crack's propagation in refractories.

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