APPLICATION OF DIGITAL IMAGE CORRELATION FOR THE ANALYSIS OF THE FRACTURE BEHAVIOUR OF REFRACTORIES

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ABSTRACT

Refractories are heterogeneous materials designed to operate in harsh working environments that sometimes lead to their premature failure. Therefore, it is necessary to enhance their properties to ensure consistent furnace performance and operator safety. Among these properties, the thermal shock resistance of refractories is a parameter of significant interest, which is known to be closely related to their mechanical behaviour. In fact, a pre-existing network of micro-cracks within the microstructure of refractories often leads to a non-linear mechanical behaviour which is beneficial for the crack propagation resistance and thus, the thermal shock resistance.

In the present study, refractory materials with a non-linear mechanical behaviour were chosen in order to highlight their fracture process, with regard to their microstructure, by using Digital Image Correlation (DIC). The direct measurement of displacement fields between digital images of the reference state and the deformed one has provided valuable information on material deformation during loading, especially at the vicinity of the crack tip. Therefore, the aim of this work is to investigate the fracture behaviour of refractories through DIC by using a refined process, called 2P-DIC, based on a material transformation taking into account a discontinuity of displacement.

As highlighted by the results, the coupling of 2P-DIC with the wedge splitting test has proven to be effective in characterizing the fracture behaviour of the studied materials.

KEY-WORDS: Refractories, Digital Image Correlation, Wedge-splitting test, Fracture behaviour, Process zone

INTRODUCTION

The mechanical behaviour of refractories is generally not purely elastic as they exhibit a non-linear stress-strain curve accompanied by a relatively high strain to rupture (≃0.1%), which is not common for standard ceramic materials. Moreover, this non-linear mechanical behaviour is known to be closely related to pre-existing damage within refractory materials [1]. Indeed, the presence of damage, at the microstructural scale, actively dissipates the elastic energy stored in the material, leading to more strain before fracture. As such, pre-fabricated microcracks that originate from a mismatch between coefficients of thermal expansion (CTE) of refractory constituents can lead to an enhanced thermal shock resistance [2-3].

From this perspective, refractories are considered quasi-brittle materials with a complex fracture behaviour that is marked by several energy-consuming phenomena present within the microstructure [4]. In fact, such mechanisms are confined in the so-called frontal process zone and are responsible for a large part of the consumed elastic energy, leading to a relatively high total fracture energy. These toughening mechanisms are due to the heterogeneous composition of refractories aswell as pre-existing microcrack networks in the microstructure.

In the present study, a novel approach involving the application of a refined Digital Image Correlation technique, called 2P-DIC, coupled to the wedge splitting test will be used to study the fracture behaviour of three types of industrial magnesia-spinel refractories.

EXPERIMENTAL PROCEDURE

The Wedge Splitting Test

The wedge splitting test has been introduced by Tschegg as a method to study the fracture behaviour of coarse grained materials such as refractories [5-6]. This test provides an ideal setup to perform stable crack propagation and measure fracture parameters such as the specific fracture energy G_f. The testing setup is constituted of two metallic supports, two rolls and a wedge as shown in Fig. 1. The metallic supports act as load transmission parts from the action of the wedge on the two rolls, where the vertical load F_V applied by the wedge is converted into a horizontal load F_H. This configuration contributes to limiting the elastic energy stored in the testing machine and thus promotes stable crack propagation. Cubic samples of 100x100x70 mm^3 are machined in order to produce the groove and the starter notch for crack propagation.

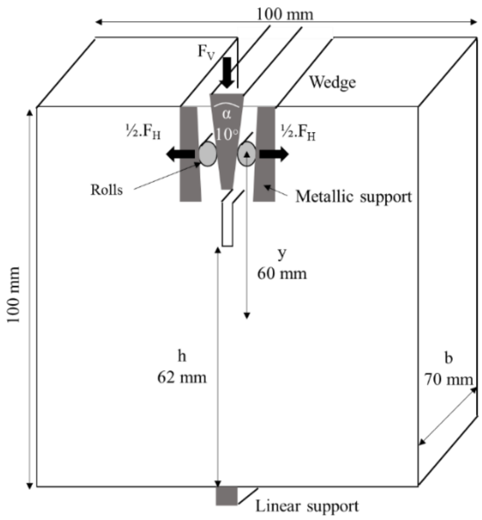

Fig. 1 Wedge splitting test setup

During loading, the applied vertical load F_V is recorded along with displacement. Horizontal load is deduced from the vertical load as:

$$F_H = \frac{F_V}{2.tan\left(\frac{\alpha}{2}\right)} \qquad (1)$$

Where α, equal to 10°, is the angle of the wedge. The specific fracture energy is calculated from the area under the horizontal load-displacement curve according to the following integral:

$$G_f = \frac{1}{A}\int_0^{\delta_{H\,max}} F_H \,.\, d\delta_H \qquad (2)$$

Where A is the fracture surface area, δ_H the horizontal displacement and δ_{max} the maximum horizontal displacement of the sample during the mechanical test.

Moreover, the nominal notch tensile strength can be calculated using the following equation:

$$\sigma_{NT} = \frac{F_{H\,max}}{b.h} + \frac{6F_{H\,max}.y}{b.h^2} \qquad (3)$$

Where $F_{H\,max}$ is the maximum horizontal force, b and h are the width and the height of the fracture surface and y the vertical distance between the loadpoint and the centre of gravity of the fracture area.

Materials

In this study, three types of industrial magnesia spinel bricks were characterized, namely, MS1, MS2 and MS3. These refractories are mainly composed of a magnesia matrix with different inclusions for each type of material. The elastic properties of these materials were measured by ultrasonic techniques, the values have been reported in Tab. 1 along with physical properties.

Tab. 1 Material data

Material	Main components		Young's modulus (GPa)	Porosity (%)
	Matrix	Inclusions		
MS1	MgO	$MgAl_2O_4$	35.41	16.37
MS2	MgO	$FeAl_2O_4$	42.39	15.23
MS3	MgO	$MgAl_2O_4$ and $FeAl_2O_4$	33.53	15.62

The industrial magnesia spinel materials studied herein are designed for thermomechanical applications involving thermal shocks. Therefore, an increased flexibility is expected from these materials to sustain the mechanical strain resulting from thermal solicitations. Moreover, the studied materials are most likely to contain micro-crack networks within their initial microstructure. In fact, the CTE mismatch between the different components of each material, namely, hercynite (10.3×10^{-6} °C^{-1}) or spinel (9×10^{-6} °C^{-1}) aggregates and the magnesia matrix (15×10^{-6} °C^{-1}) suggests that the matrix is prone to containing micro-cracks around the aggregates.

As such, these microstructural features can lead to the development of toughening mechanisms in the so-called process zone, which is divided into two regions, namely, the frontal process zone and the process wake zone. Fig. 2 illustrates some of the mechanisms that occur during crack propagation, mainly because of a pre-existing microcrack network within a refractory.

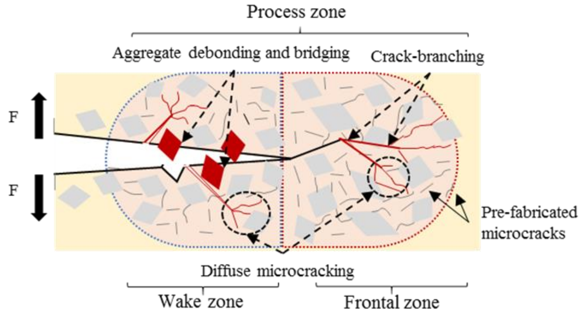

Fig. **2** Toughening mechanisms in the process zone

Wedge splitting samples have been produced by machining from refractory bricks. The sample surface was then prepared for the DIC matching process by spraying an opaque black layer then droplets of white paint. This way, an artificial speckle pattern was created to follow material displacements.

Digital Image Correlation

Digital Image Correlation (DIC) is an optical technique for displacement field measurements. It has been applied in diverse topics of experimental mechanics, including the study of the mechanical behaviour of refractory materials [7]. In this study, a refined DIC technique, named two-parts DIC (2P-DIC) has been used to study the propagation of vertical cracks in quasi-brittle materials. The refined DIC process uses the same principle as local DIC [8]. It consists in matching reference and deformed 2D images corresponding to two distinct mechanical states of a sample based on their grey level distribution. The matching process is performed on user-defined subsets by defining a plane material transformation between successive images, where a correlation coefficient is used to find the best values for the unknown displacement components according to an optimisation routine. This latter evaluates the resemblance of grey level distributions of pixels between initial and deformed states assuming the conservation of optical flow. With the refined 2P-DIC approach [9], material discontinuity can be detected following a subset splitting procedure leading to an increased crack resolution compared to standard DIC. Therefore, crack presence, position and opening can be determined per a user defined pseudo-strain threshold ε_s.

The experimental setup in Fig. 3 was used to acquire images, whereby a CMOS USB camera with a resolution of 2560x1920 px^2 was placed in front of a prepared WST sample. Additionally, LED lights were used as a cold light source to provide sufficient illumination without heating the samples. The images are then processed using the 2P-DIC software to measure the displacement fields as displayed on Fig. 2 for illustrative purposes.

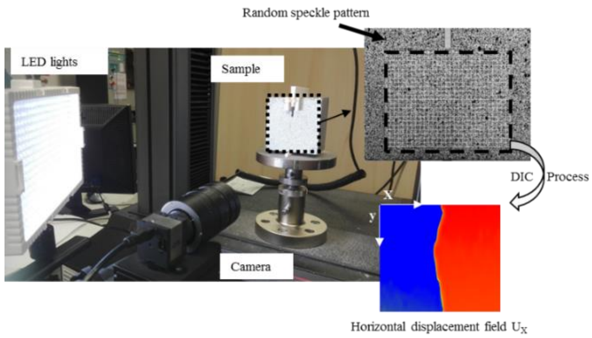

Fig. 3 Experimental setup and acquisition process

RESULTS AND DISCUSSION

Horizontal Load-Displacement curves have been reported in Fig. 4 for the three materials. The non-linearity of the curves as well as the existence of a rather large post-peak region indicate that the studied materials are, in fact, flexible and quasi-brittle unlike standard ceramics.

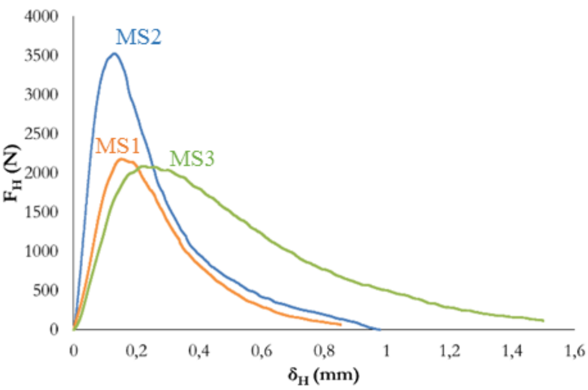

Fig. 4 Horizontal load-displacement curve of MS1, MS2 and MS3

Fracture energies were measured from the area under the horizontal load-displacement curves up to F_H= 15%.$F_{H\,max}$ for three samples of each material. Fracture parameters such as the

G_f/σ_{NT} ratio and the characteristic length l_{ch} were then computed to assess the brittleness of each material as reported in Tab. 2. The so-called l_{ch} is calculated to assess the brittleness of materials without considering specimen size, therefore, only material properties are taken into account [10]. The G_f/σ_{NT} ratio is similar to l_{ch} as it also relates to the brittleness of the materials, whereby a low value of the ratio indicates a brittle behaviour and vice-versa. The brittleness in this case is an evaluation of the crack propagation resistance of a material, where a reduced brittleness favours crack propagation resistance.

Measurements of the specific fracture energy indicate relatively high values for the three types of materials with a rather low nominal tensile strength. In conjunction with these results, the G_f/σ_{NT} ratio suggests a rather similar behaviour for MS1 and MS2 as they both exhibit a reduced brittleness. In comparison, MS3 has a much higher ratio, which means it is less brittle than the other two.

Tab. 2 Fracture parameters measured by WST

	MS1	MS2	MS3
G_f (J.m^{-2})	185.07	228.26	306.73
σ_{NT} (MPa)	4.13	4.99	3.53
G_f/σ_{NT} (µm)	45.15	45.83	87.51
l_{ch} (mm)	352.68	367.69	882.78

The reduced brittleness of these materials is mainly linked to their microstructure and the energy dissipating mechanisms that operate during crack propagation. In fact, toughening mechanisms are initially present in the microstructure and are activated during loading, which leads to the quasi-brittle behaviour of the studied magnesia-spinel materials. Ultimately, the composite-like structure of magnesia-spinel bricks, along with the existence of a CTE mismatch between the matrix and the inclusions, is the leading factor in developing energy dissipating phenomena during crack propagation.

From this viewpoint, the newly developed 2P-DIC method comes as a convenient technique to underline fracture mechanisms occurring in the process zone, as the crack propagates. Sample displacement fields were measured throughout loading and calculations were performed to extract features of crack propagation from the images. Cracks were identified for a strain threshold of ε_s=0.002, that was selected to be just above the noise level, in order to visualise all the important aspects of cracking. The graph in Fig. 5 describes the evolution of the dissipated and elastic energies, in orange and green colour respectively, during loading for MS 1.

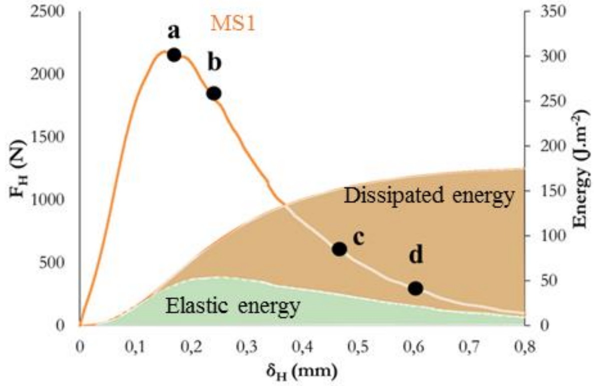

Fig. 5 Horizontal load-displacement curve and evolution of Energy versus horizontal displacement for MS1

As a result of 2P-DIC computations, crack propagation was followed throughout loading as reported in Fig. 6 for different loading states. Crack initiation occurs before state a, at the peak of the load-displacement curve, while crack propagation is associated with the post-peak region. In state b, the crack propagates and splits into two cracks before joining again. The main crack continues to propagate in state c while the progressive closure of the secondary crack occurs as the crack tip advances. Finally, in state d, the main crack propagates through the sample while the secondary crack closes.

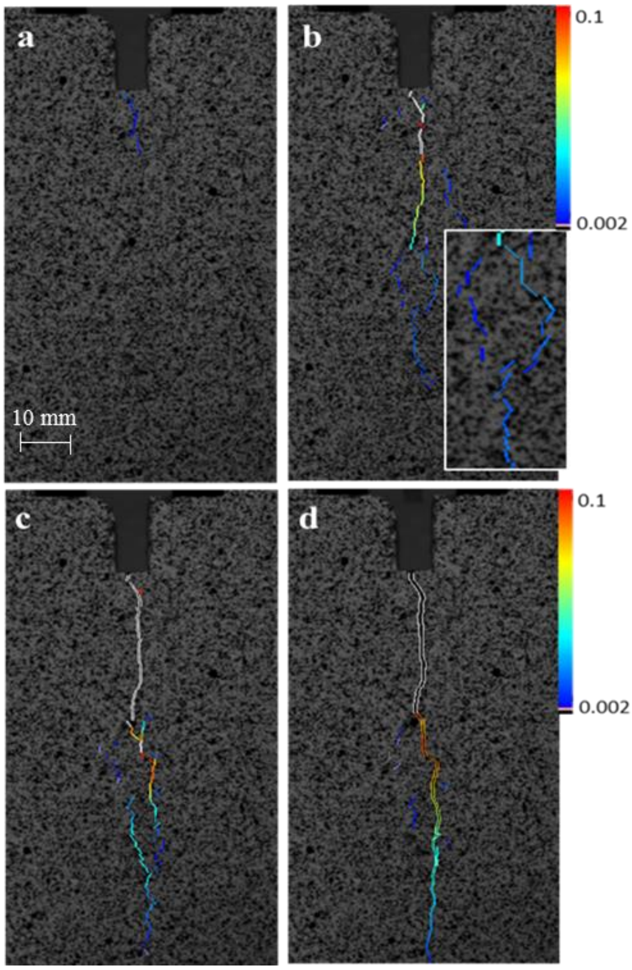

Fig. 6 2P-DIC images of MS1 for different loading states

Crack propagation in the case of MS1 is therefore, not enterily straight. In fact, it is marked by some tortuosity due to the initial presence of microcracks at the microstructural level, which lead to a decrease in brittleness.

Similarly, Fig. 7 highlights loading states that describe crack-propagation for MS3 as well as the evolution of the dissipated and elastic energies, displayed in orange and green colour respectively. Unlike MS1, MS3 exhibits a much higher specific fracture energy indicating the presence of an initially dense network of microcracks. Therefore, as the crack propagates, an increased amount of energy is consumed and a reduced brittleness is achieved.

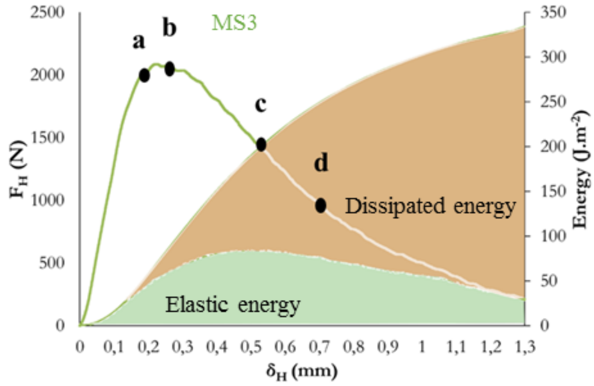

Fig. 7 Horizontal load-displacement curve and evolution of Energy versus horizontal displacement for MS3

Moreover, according to images in Fig. 8, 2P-DIC results reveal the occurrence of extensive crack branching during crack propagation. During loading, the main crack splits into two macrocracks in state b, which in turn split into more cracks in further states. This leads to the development of many more crack branches as the crack tip advances, as observed in the intermediate state c, before reaching the final fracture state of the sample in state d. These phenomena stem from the microcrack network initially present within the material because of a CTE mismatch between the magnesia matrix and the combination of hercynite and spinel inclusions.

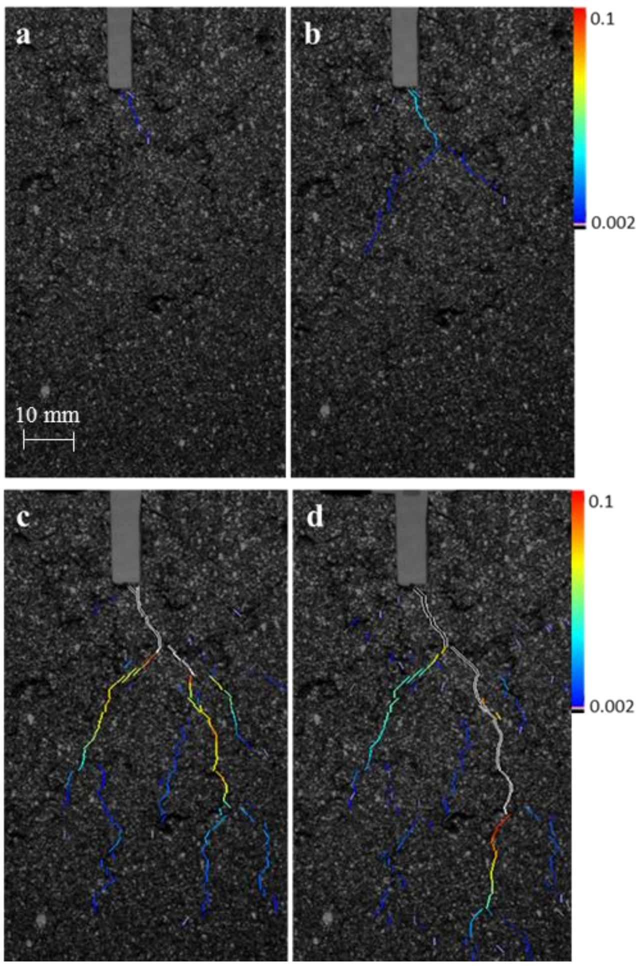

Fig. 8 2P-DIC images of MS3 for different loading states

In comparison, MS3 exhibits a much higher G_f/σ_{NT} ratio due to the extensive crack-branching during loading, as demonstrated by 2P-DIC analysis.

CONCLUSION

The fracture behaviour of three types of magnesia spinel refractories was studied in opening mode. Wedge splitting experiments were carried out to measure fracture parameters and characterise the brittleness of the materials. Additionally, a refined DIC process was applied to highlight fracture mechanisms occurring during loading as the crack tip advances. The main objective of this work was to study the relationship between the fracture behaviour and the microstructure of magnesia spinel materials used in thermomechanical applications. WST results have demonstrated that a reduced brittleness, characterised by a relatively high G_f/σ_{NT} ratio, was achieved for magnesia spinel materials thanks to a pre-fabricated microcrack networks associated to a CTE mismatch. Moreover, the approach used herein revealed some important, process-zone related, features during crack propagation as 2P-DIC was successfully employed to underline the crack-branching phenomenon with an enhanced spatial accuracy.

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