The Mechanical Fracture Characterization of Non-Linear Flexible Ceramics Using Digital Image Correlation

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Abstract During their application, refractory materials are submitted to several thermal shock attacks. That is why; improving their thermal shock resistance has a great importance and can enhance their lifetime. This characteristic is directly related to strains to rupture.

The non-linear mechanical behavior which characterizes specific developed ceramics, and is a consequence of their micro-cracked microstructure, leads to the increase of their strain to rupture level. This limit allows a better resistance to thermal shock solicitations.

Magnesia spinel materials are good candidates to characterize such non-linear mechanical behavior dependent on their microstructure. This non linearity is due to the presence of micro-cracks network. Indeed, mechanical behavior can vary from a fragile one to a large non-linear one according to the degree of micro-cracking present within the material.

In this paper, to investigate the crack propagation and the non-linear mechanical behavior of this material, wedge splitting test was used and coupled to digital image correlation technique. Results underline the influence of the addition of spinel on the non-linear mechanical behavior by comparing the non-linear mechanical behavior of magnesia spinel with pure magnesia. Besides, results clarified also the crack propagation mechanisms which are specific to these heterogeneous materials.

Keywords Magnesia spinel, Flexibility, Digital image correlation, Crack branching

1. Introduction

Due to their properties, pure magnesia and magnesia spinel materials showed a great interest for commercials and researchers [1-4]. Pure magnesia bricks are known by their resistance to corrosion and their relatively low thermal shock resistance. Besides, the incorporation of spinel improves the thermal shock resistance and can extend to three times longer service life of cement rotary kiln. Indeed, the addition of spinel, leads to a better adaptability of the material to severe solicitations generated during high and quick temperature variations. Even if their process of elaboration can affect the economy, but they still preferable to magnesia chromite refractories which have nearly the same properties such as their high resistance against thermal shock damage, erosion and corrosion but are not recommended due to the toxicity of waste refractories containing chrome.

The behavior of magnesia spinel is related to their microstructure and especially to the difference between the thermal expansion of magnesia and spinel which is behind the development of micro-cracks around the spinel grains and can prevent crack propagation generated by thermal shock [5-6]. Moreover, their microstructure induces a significant non-linear stress-strain mechanical behavior allowing a high strain to rupture, high fracture energy and as a consequence improves their thermal shock resistance. It has been reported that micro-cracking caused by the addition of spinel reduces both strength and stiffness, and affect the fracture behavior [7]. Both the knowledge of the mechanisms initiating such behavior and the possibility to characterize those in the best accurate way are essential.

From these considerations, this study is a part of a work aiming to understand the relationship between microstructure and the non-linear mechanical behavior of magnesia-spinel materials and compared to pure magnesia materials.

The fracture characterization is done using wedge splitting test (WST) which affords stable crack propagation [8-11].

To assess to strain fields on the surface of the material, digital image correlation (DIC) technique has been chosen. This full-fields practical and effective tool for quantitative deformation measurement of a planar sample surface is now widely accepted and commonly used in the field of experimental mechanics. This non-contact optical method directly provides full-fields strains by comparing the digital images of the sample’s surface obtained before and during deformation [12-14]. Using DIC, the propagation of the crack was studied thanks to kinematic fields approach in terms of strains. It allows highlighting the “crack branching” phenomenon due to the particularity of the microstructure of magnesia spinel materials.

2. Experimental

2.1. Materials

Pure magnesia and magnesia 15%-spinel (MSp) used in this study were elaborated from different grain distribution of industrial magnesia with low iron content (fines<0.1 mm ; 0-1 mm ; 1-3 mm ; 3-5 mm) and additional sub-stoichiometric spinel (1-3 mm) in the case of MSp. Spinel grains replaced the same content of magnesia aggregates having the same size.

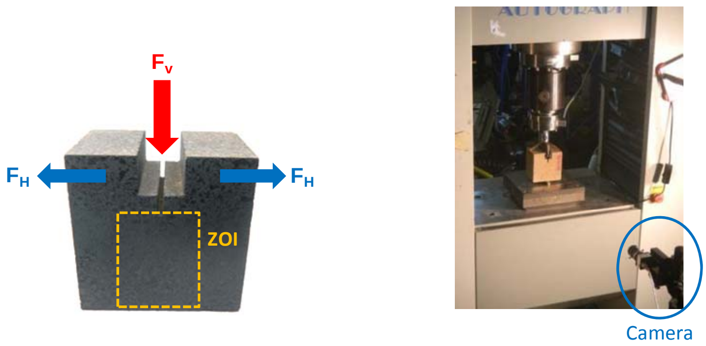
Their elaboration was done using a maximum pressure of 140MPa and a maximum firing temperature of 1600°C.

Due to the thermal expansion mismatch existing between the magnesia matrix (“the whole material excepting spinel inclusions”) and the spinel inclusions (α_{MgO}=13.3 x 10^{-6} K^{-1} and $\alpha_{MgAl2O4}$=8.9 x 10^{-6} K^{-1}), it appears that there are some micro-cracks around the spinel inclusions. These micro-cracks appear during the cooling stage of the process conferring the material a “thermally damaged” character [.

2.2. Wedge Splitting Test

A more recent and widely accepted test, which has been used frequently for materials with coarse microstructures, is the method patented by Tschegg under the name “wedge splitting test”. This technique is being used by a numerous research centers and universities, as well as by manufacturers of refractories.

As shown in figure 1a the WST consists of opening a crack using a wedge. 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 (Fig.1b). Samples were equipped with a groove in order to apply the splitting load and a starter notch. The specimen geometry and dimensions are illustrated in fig.1a.



- a -   - b -

Figure 1: The principle of wedge splitting test with sample's geometry (a) and image acquisition setup (b).

Since the thermal shock resistance of these materials has to be improved, the brittleness of these materials has to be reduced and the deformation before cracking has to be higher by modifying the microstructure of the refractory. In fracture mechanics, the fracture energy is one parameter that characterizes the fracture strength of the material.

Then, from the experimental data in terms of load-displacement, the specific fracture energy $\mathbf{G}_f$ ($N.m^{-1}$) is defined as the mean work per unit of projected fracture area required to propagate a crack, and represented by the sum of distinct energies consumed during the crack propagation process. $\mathbf{G}_f$ is calculated using equation (1).

$$G_F = \frac{1}{A} \cdot \int_0^{\delta_{max}} F_H \cdot d\delta_H \tag{1}$$

Where A is the crack area, F_H is the horizontal force and δ_H is the horizontal deformation. This specific fracture energy is calculated for δ values from zero up to the displacement δ_{max} corresponding to 10% of the maximum load.

The horizontal force (F_H) is calculated from the vertical force (F_v) neglecting any friction effects using the equation (2).

$$F_H = \frac{F_v}{2 \cdot \tan(\alpha / 2)} \tag{2}$$

Where α is the wedge angle, which was 19.5° in this investigation. The fact that (F_H) is much higher than (F_v) reduces the load applied on the machine's frame and storing less elastic energy in it and helping the propagation of the crack.

Another mechanical property of material estimate from the WST is the nominal notched tensile strength (σ_{NT}), which represents the stress at the point of beginning of rupture, is calculated by the equation (3).

$$\sigma_{NT} = \frac{6 \cdot y \cdot F_{H,max}}{b \cdot h^2} + \frac{F_{H,max}}{b \cdot h} \tag{3}$$

Where b (mm), h (mm) are sample's dimensions and y (mm) is the distance between $F_{H,max}$ and the middle of h.

In addition to the experimental setup, a 8-bits CMOS camera (1600 x 1900 pixels^2) used to record images is placed in front of the speckled surface in order to perform optical measurements thanks to digital image correlation (Fig 1b). This will stress crack growth during mechanical tests and the strain state around the crack tip. The acquisition frequency was 1 image per second.

2.3. Digital image correlation

The damage level neighborhood to the crack tip and the crack advancement are estimated from the strain cartographies obtained by digital image correlation (DIC). This full fields optical method 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. The zone of interest, on which the calculation is done, is represented in figure 1a. Each pixel of these images stores a grey level value due to a pattern at the surface. To avoid ambiguities in the similarity process a random distribution of grey levels can be used called speckle pattern. This pattern can be the natural texture of the specimen surface or artificially made by spraying black and/or white paints. As an example for sample's surface preparation, a black opaque paint layer is deposited on the surface of sample then dried. After that, white speckles will be carefully projected on using spray paint.

The used DIC process consists in calculating displacements and strains on specific points several subsets which constitute a grid. The principle of this technique is explained in figure 2

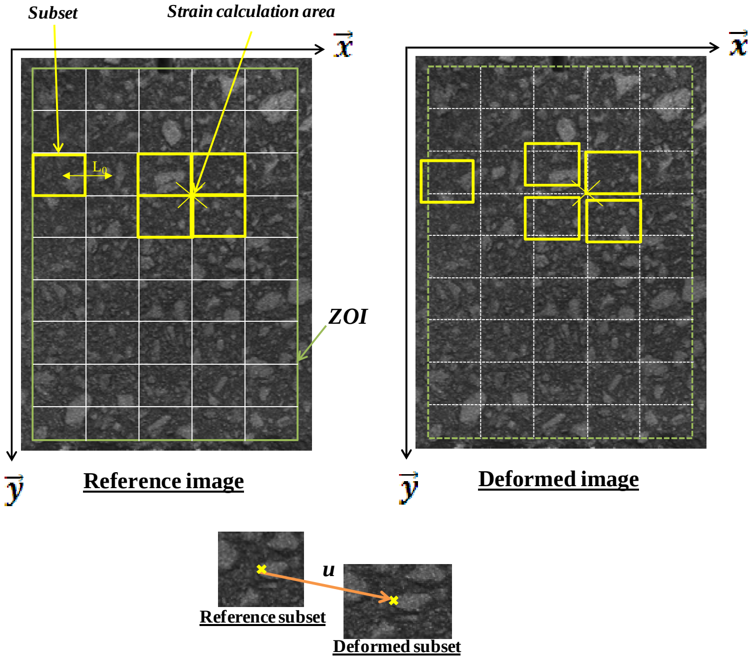


Figure 2: The principle of digital image correlation

3. Results

3.1. Mechanical properties

Figures 3, 4 represent the non-linear load-deflection curves of pure magnesia and magnesia spinel materials obtained by wedge splitting test.

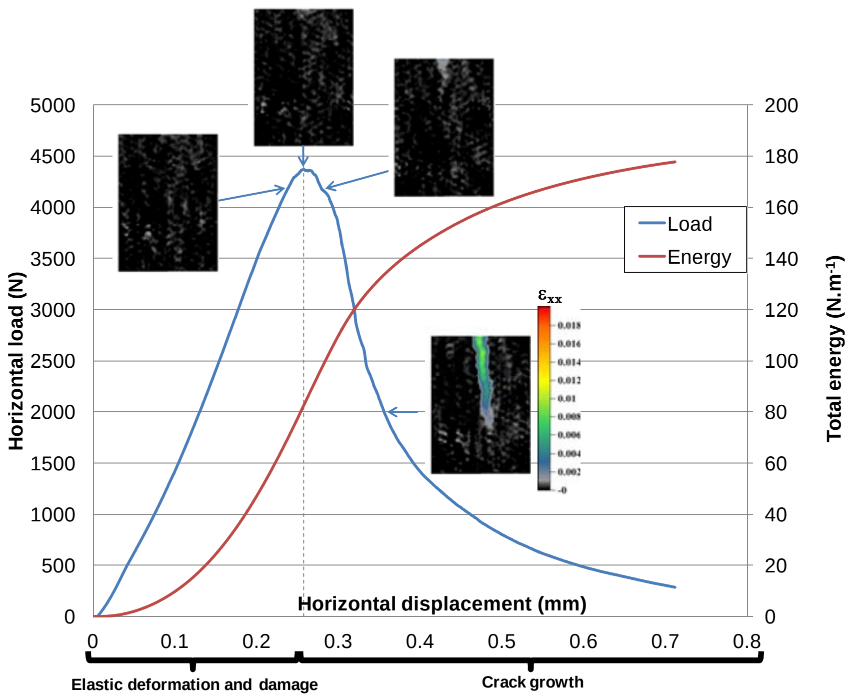


Figure 3: Horizontal load-displacement curve and total energy of pure magnesia material obtained thanks to

WST. Four maps of strains along X axis are represented at four times

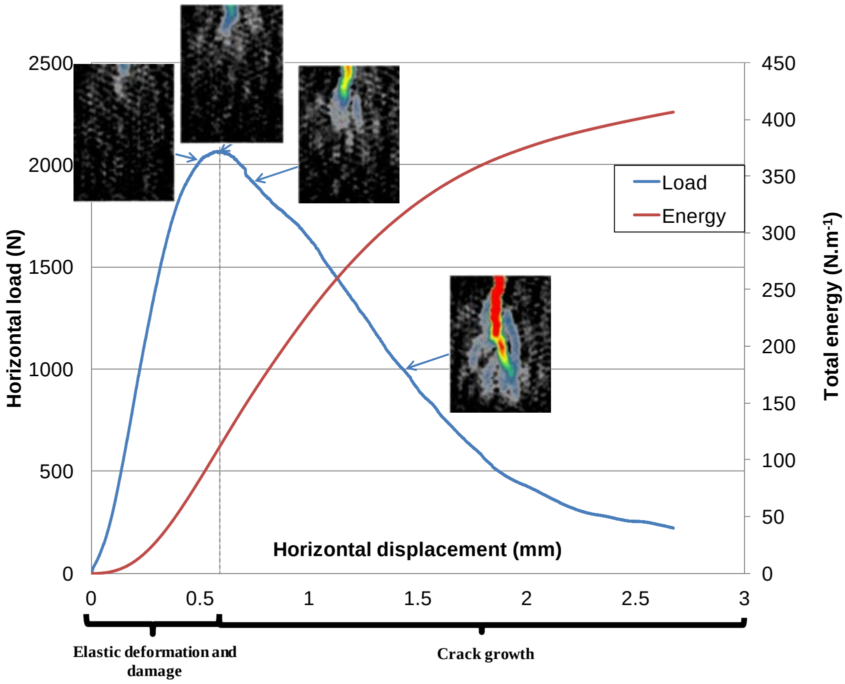


Figure 4: Horizontal load-displacement curve and total energy of magnesia spinel material obtained thanks to WST. Four maps of strains along X axis are represented at four times

The pure magnesia material presents a non-linear behavior (rather small) before the peak with a significant post-peak region and a high strain to rupture. It exhibits a much higher maximum load corresponding to the crack initiation, a lower strain at the peak, and a thinner peak due to a smaller post-peak region in comparison with MSp. This behavior is not common at room temperature for homogeneous ceramic materials. A possible explanation of this behavior is the coarse grain size (1-3 mm and 3-5 mm) distribution of this material which was reported to potentially improve fracture and thermal shock resistance of magnesia refractories.

Besides, load-deflection curve of magnesia spinel presents a non-linear mechanical behavior up to the peak with a significant post-peak region and residual strain when unloading. Moreover, the increase of spinel content seems mainly to lower the maximum load but does not influence so much the end of the post-peak region since it is close to pure magnesia. By considering the very beginning of this curve, the addition of spinel, and therefore, the thermal damage introduction, allows to enhance the non-linearity of the mechanical behavior. This material has a higher strain and a low mechanical resistance in comparison with pure magnesia; this can be explained by the higher volume of microcracks in the case of magnesia spinel developed during cooling and toughening mechanisms occurring around the crack, especially in the following wake region, which may increase the resistance to crack propagation. The higher volume of microcracks in the case of magnesia spinel is due to the difference between the thermal expansion coefficient of magnesia (α_{MgO}=13.3 x 10^{-6} K^{-1} and $\alpha_{MgAl2O4}$=8.9 x 10^{-6} K^{-1}) which leads to the apparition of microcracks around the grain of spinel.

In the two cases, the mechanical behavior of these materials is characterized by three phases, one corresponding to the elastic behavior, a second one characterized by the elastic behavior coupled with damage (elastoplasticity), and the last one corresponding to the crack growth.

Only the main macrocrack can be detected by the naked eye, but the early apparition of the macrocrack, as well as all the stressed microcracks pre-existing around the middle plane, is not really visible. That is why; digital image correlation has been used in order to obtain the strain fields so as to analyze the crack propagation and the presence of damage process.

Thanks to digital image correlation, the evolution of the strains along X-axis has been represented for different instants corresponding to different loading states (fig 3, 4). These maps correspond to the zone of interest defined in figure 1a .According to DIC principle, this area is subdivided using subsets of 32 x 32 pixels^2 with a gap of 8 x 8 pixels^2 and a scale factor of 0.067295 mm/pixels. In the two materials, we notice before reaching $F_{H,max}$ the development of the damage zone in the neighbor of the crack tip. The initiation and the propagation of the crack take place after this critical load ($F_{H,max}$). Due to the higher ductile behavior of magnesia spinel, the damaged zone in the neighbor of the crack tip is larger than the case of pure magnesia. Besides, this zone is developing and moving with the crack tip advancement. This confirms the complexity of crack growth in heterogeneous materials which feature many physical mechanisms such as crack branching phenomenon.

Nominal notched tensile strength and specific fracture energy for each sample have been calculated thanks to the equations (2) and (3) and represented in Table 1.

Table 1: Nominal notched tensile strength and specific fracture energy values

	σ_{NT} (MPa)	G_F (N.m^{-1})	G_F/σ_{NT} (mm) x 10^{-3}
Pure MgO	14.36	206.50	14.39
	14.38	182,00	12,66
MgO-15% spinel	6.65	361.00	54.25
	6.75	396.00	58.63

These intrinsic fracture parameters introduced in table 1 stress the nominal notched tensile strength shows that mechanical resistance decreases whereas the energy stresses that the fracture resistance increases with the increase of the spinel rate. The ratio G_F/σ_{NT} indicates the brittleness of the material: the more brittle material is, the lower value of the ratio is.

Figures 10 and 11 represent also the evolution of the total energy during the test. The total energies are deduced from the total area under the horizontal load-displacement curves obtained by wedge splitting test divided by the section of fracture and represent the sum of the elastic energy and dissipated one. The curves confirm that during the test, the total energy is higher in the case of magnesia-15% spinel than pure magnesia. The dissipated energy is mainly due to the dense network of microcracks created during the test.

4. Conclusion

The impact of microstructure on the mechanical behavior has been clarified. Indeed, studying mechanical behavior is important to have an idea on the thermal shock resistance of materials. In this paper, mechanical behavior of magnesia spinel products were analyzed and compared to pure magnesia. The difference between mechanical behaviors of these materials is due to the development of the network of micro-cracks in the case of magnesia-spinel materials. This is a consequence of the thermal expansion mismatch between magnesia and spinel grains. To study the mechanisms of micro-cracking and the influence of damage on the non-linearity, wedge splitting test has been used and coupled to digital image correlation. Indeed, by the calculation of mechanical properties such as nominal notched tensile strength and fracture energy in addition to the measurement of strain fields, the influence of the addition of spinel is clarified. In fact, it was confirmed that the addition of spinel influences clearly mechanical properties and the non-linearity of the mechanical behavior due to the presence of high level of damage induced by micro-cracks as shown thanks to digital image correlation. This microstructure optimization allows an increase of strain to rupture, which is considered as a key parameter for improving thermal shock resistance of refractories.

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