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# Investigation of the impact of micro-cracks on fracture behavior of magnesia products using wedge splitting test and digital image correlation

Y. Belrhiti<sup>a,\*</sup>, O. Pop<sup>b</sup>, A. Germaneau<sup>c</sup>, P. Doumalin<sup>c</sup>, J.C. Dupré<sup>c</sup>, H. Harmuth<sup>d</sup>, M. Huger<sup>a,\*</sup>, T. Chotard<sup>a,e,\*\*</sup>

<sup>a</sup> Science des Procédés Céramiques et Traitements de Surface (SPCTS UMR CNRS 7315), CEC, 12 rue Atlantis, 87068 Limoges Cedex, France

<sup>b</sup> Groupe d'Etude des Matériaux Hétérogènes (GEMH), Université de Limoges, 12 rue Atlantis, 87068 Limoges Cedex, France

<sup>c</sup> Institut Pprime, CNRS, Université de Poitiers, 11 Boulevard Marie et Pierre Curie, 86962 Futuroscope Chasseneuil Cedex, France

<sup>d</sup> Montanuniversität Leoben, Department of Ceramics, Peter-Tunner-Strasse 5, A-8700 Leoben, Austria

<sup>e</sup> Institut Universitaire Technologique du limousin, Département GMP, 2 Allée André Maurois, 87065 Limoges Cedex, France

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#### Abstract

For refractory materials, a large strain behavior before failure is desired to improve their thermal shock resistance during their service life in industry. Their microstructure (micro-cracked) induces a non-linear mechanical behavior.

The present paper aims at applying digital image correlation (DIC) during wedge splitting test on magnesia spinel sample and comparing it to pure magnesia sample so as to study the influence of spinel addition on fracture behavior. From strain measurements based on DIC, the initiation and the propagation of the macro-crack are investigated and the development of micro-cracks network in the case of magnesia spinel which is behind crack branching phenomenon is analyzed. The evolution of the fracture energy required in crack propagation is studied for each material. The experimental results highlight also their mechanical properties dependent on their microstructure.

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### 1. Introduction

In refractory industry, the ability to sustain severe thermal shocks is of prime importance in order to increase the length of the refractory material life and to avoid their failure. These "characteristics" are generally obtained by the development of specific microstructure patterns on well-chosen refractory materials. Among the materials used in refractory industry magnesia refractories are to be good candidates due to their thermo-mechanical properties at room and elevated temperature. Due to their properties, pure magnesia and magnesia

http://dx.doi.org/10.1016/j.jeurceramsoc.2014.09.001 0955-2219/© 2014 Elsevier Ltd. All rights reserved. spinel materials showed a great interest for commercials and researchers.<sup>1,2</sup> 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<sup>3</sup> compared to conventional magnesia chrome bricks. Indeed, spinel addition, leads to a better adaptability of the material to severe solicitations generated during high and quick temperature variations. Magnesia spinel refractories still preferable to magnesia chromite ones which are not recommended because of their toxicity due to chromite contain. These two types of refractory have nearly the same properties such as a high thermal shock resistance.

Even if their process of processing 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.

<sup>\*</sup> Corresponding authors. Tel.: +33 5 87 50 25 60.

<sup>\*\*</sup> Corresponding author at: Science des Procédés Céramiques et Traitements de Surface (SPCTS UMR CNRS 7315), CEC, 12 rue Atlantis, 87068 Limoges Cedex, France. Tel.: +33 5 87 50 25 60.

*E-mail addresses:* younes.belrhiti@etu.unilim.fr (Y. Belrhiti), marc.huger@unilim.fr (M. Huger), thierry.chotard@unilim.fr (T. Chotard).

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.<sup>4–7</sup> 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 microcracking caused by the addition of spinel reduces both strength and stiffness, and affect the fracture behavior.<sup>6–8</sup> 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 conducted through wedge splitting test (WST) results. This test is well known for its ability to produce reliable data on stable crack propagation.<sup>9–15</sup> This means that material characterization quality depends on test evaluation.

To assess to strain measurements based on DIC on material surface, digital image correlation technique has been chosen. This full-fields practical and effective tool for quantitative displacement and strain measurement of a planar sample surface is now widely accepted and commonly used in the field of experimental mechanics. This non-contact optical method compares digital images of the sample surface obtained before and during deformation.<sup>16–26</sup> It allows highlighting the "crack branching" phenomenon. It is a complex process of heterogeneous materials in which crack propagation induces the coalescence of existing "branches" that correspond to existing micro-cracks. Such phenomenon occurs due to the particularity of the microstructure of magnesia spinel materials.

#### 2. Materials and methods

#### 2.1. Materials

Pure magnesia and magnesia 15%-spinel (MSp) used in this study were processed 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



Fig. 1. Microstructure of magnesia-spinel material.

spinel (1-3 mm) in the case of MSp. Spinel grains replaced the same content of magnesia aggregates having the same amount.

Their processing was done using a maximum pressure of 140 MPa and a maximum firing temperature of  $1600 \degree \text{C}$ .

Information concerning these materials such as granulometry, density and porosity are given in Table 1.

Due to the thermal expansion mismatch existing between the magnesia matrix ("the whole material except spinel inclusions") and the spinel inclusions ( $\alpha_{MgO} = 13.3 \times 10^{-6} \text{ K}^{-1}$  and  $\alpha_{MgAl_2O_4} = 8.9 \times 10^{-6} \text{ K}^{-1}$ ), it appears that there are some micro-cracks around the spinel inclusions (Fig. 1). These microcracks appear during the cooling stage of the process conferring the material a "thermally damaged" character.

From each type of material, two cubic-shaped specimens with cross-section of  $100 \times 100 \times 100 \text{ mm}^3$  were tested, although, in this paper only one will be presented since the results are quite similar.

#### 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".<sup>10</sup> This technique is being used by a numerous research centers and universities, as well as by manufacturers of refractories.<sup>27,28</sup>

As shown in Fig. 2b 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. Samples were equipped with a groove in order to apply the splitting load and a starter notch.

Since the thermal shock resistance of these materials has to be improved, the brittleness of these materials has to be reduced and

Table 1

Granulometry and properties of MgO-15% spinel samples compared to pure magnesia samples.

|              |                             | Pure MgO | MgO-15% spinel |
|--------------|-----------------------------|----------|----------------|
|              | Spinel 1–3 mm (%)           | 0        | 15             |
|              | MgO 3–5 mm (%)              | 13.5     | 13.5           |
| Granulometry | MgO 1–3 mm (%)              | 34.0     | 19.0           |
|              | MgO 0–1 mm (%)              | 24.0     | 24.0           |
|              | MgO fines (<0.1 mm) (%)     | 28.5     | 28.5           |
| Properties   | Bulk density $(g  cm^{-3})$ | 2.98     | 2.95           |
|              | Total porosity (%)          | 17.5     | 15.3           |

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