G Model
JECS-10984; No. of Pages 9
ARTICLE IN PRESS
Journal of the European Ceramic Society xxx (2017) xxx–xxx
Contents lists available at www.sciencedirect.com
Journal of the European Ceramic Society
journal homepage: www.elsevier.com/locate/jeurceramsoc

Feature article

Combination of Brazilian test and digital image correlation for mechanical characterization of refractory materials

Y. Belrhiti [a,*], J.C. Dupre [b], O. Pop [c], A. Germaneau [b], P. Doumalin [b], M. Huger [a,*], T. Chotard [a]

[a] Univ. Limoges, CNRS, ENSCI, SPCTS, UMR 7315, F-87000 Limoges, France
[b] Univ. Poitiers, CNRS, PPRIME, UPR 3346, F-86962 Futuroscope Chasseneuil, France
[c] Univ. Limoges, GEMH, EA 3178, F-19300 Egletons, France

ARTICLE INFO

Article history:
Received 19 May 2016
Received in revised form
18 December 2016
Accepted 19 December 2016
Available online xxx

Keywords:
C. Fracture
C. Mechanical properties
D. MgO
E. Refractories

ABSTRACT

Flexibility of refractories is an interesting property to improve thermal shock resistance of refractories. It can often be obtained by promoting a nonlinear mechanical behaviour which reduces brittleness. Among industrial refractory materials developed in this aim, magnesia hercynite is of a particular interest for cement industry. As linings for rotary kilns, magnesia hercynite can be submitted to tensile and compressive stresses. Since tensile strength is usually much lower than compressive one for brittle materials, the mechanical characterization is, in such case, more significant in tension than in compression. To overcome difficulties involved by direct tensile test, an indirect tensile test (Brazilian test) has been applied here and combined to digital image correlation in order to measure kinematic fields on the surface of the sample during loading. This combination has allowed to accurately measure initial elastic properties (Young's modulus and Poisson ratio), to detect crack initiation and to analyze fracture process.

1. Introduction

Thermal shock resistance of refractory materials is among crucial properties that interest refractory researchers and industrials. 'Flexibility' in terms of large strain to rupture is an efficient solution to improve thermal shock resistance [1–3]. This can be obtained by generating an initial network of micro-cracks during material processing. Such dedicated micro-cracked microstructure is usually obtained by a pertinent choice of the constituents of the refractory which can lead to a suitable difference in thermal expansion coefficient between the involved phases [2]. As an outcome, their mechanical behaviour can thus be modified from linear elastic behaviour to a non-linear one, depending on the degree of initial micro-cracking induced within the microstructure of the material after processing.

This specific non-linear behaviour is a key point to enhance level of strain-to-rupture for a better accommodation to high level of strain induced by thermal shock solicitations.

Previous works already demonstrated that magnesia spinel materials constitute an interesting route to promote non-linear behaviour thanks to a network of microcracks generated by thermal expansion mismatch between magnesia matrix and spinel inclusions [2–5]. Besides, the use of such refractory composite can often be preferred to magnesia chromium bricks due health problem involved by Cr^{6+}. Among the different kind of spinel inclusions which can be introduced in the magnesia matrix to promote microcracks, the hercynite natural one ($FeAl_2O_4$ or iron aluminate spinel) has been chosen here.

During thermal shock solicitations in a cement rotary kiln, magnesia hercynite bricks can be subjected to tensile and compressive loads. However, for brittle refractory materials, tensile stresses are much more critical than the compressive ones. It is thus more pertinent to investigate tensile behaviour of those bricks. In the other hand, due to difficulties in sample preparation and in load alignment required for direct tensile test on refractory materials, indirect tests are often preferred. Among these indirect tests, Brazilian test (diametrical compression) is one of the most popular. The principle of this test, initially developed for civil engineering by F. Carneiro [6–9] is to apply a vertical diametrical load on a cylindrical sample. This compressive load induces tensile stresses in the perpendicular direction which leads to the failure in the central part of the sample. The great interest of the Brazilian test is usually to obtain easily elastic modulus, Poisson's ratio, tensile strength or the failure analysis of brittle material on a rather small sample with-

* Corresponding authors.
E-mail addresses: younes.belrhiti@etu.unilim.fr (Y. Belrhiti), marc.huger@unilim.fr (M. Huger).

http://dx.doi.org/10.1016/j.jeurceramsoc.2016.12.032
0955-2219/© 2016 Published by Elsevier Ltd.

out any complexity for the application of the load [6–15]. Since the rupture takes place in the central part of the sample, the obtained strength value is rather insensitive to sample surface defects. On the other hand, Brazilian test is not very well suited for the stress-strain law characterization and not so much work is reported in the area. This is due to the fact that the sample is not submitted to an uniaxial uniform strain field.

Although the Brazilian test has been studied extensively, little attention has been placed on the possibility to obtain more information from a single test. However, last years, the recent studies were dedicated to a global analysis from one test record by using the analytical or numerical approaches coupled with the experimental measurements [13–18]. Wang and Wu [14] propose an analytical and numerical approaches to evaluate elastic modulus, tensile strength and fracture toughness for brittle rocks. He proposes three analytical expressions for the determination of the three material parameters indicated above. This approach is an alternative to experimental measurements of elastic modulus and Poisson's ratio proposed by Hondros by using strain gauges [18]. Jianhong et al. [19] proposes also a simple and convenient test method to measure tensile elastic modulus for different rocks by means the strain gauges. Based on Wang and Wu [14] developpement and numerical simulation by means ABAQUS finite element program, Elghazel et al. [15] propose also experimental and numerical investigations for bioceramics fracture test. Concerning the fracture process Jonsen et al. [20] proposes a method to evaluate fracture energy from diametral compression test for iron powder. The list can be continued with the Guo et al. [12], Jonsen et al. [20] and Jianhong et al. [19] developments to measure the opening mode fracture toughness.

In this context our study deal with the evaluation of the elastic properties (elastic modulus and Poisson's ratio) and the failure process from a single test by using the experimental data obtained from the optical full-fields measurements. Concerning the experimental full-field measurements, digital image correlation seems to be the best to characterize the mechanical behaviour of brittle materials, [1,21–28].

Kourkoulis et al. [29] proposes an experimental approach based on 3D digital image correlation. The study proposes an experimental characterization of the Brazilian test boundary conditions from the displacement and strain distribution measured using digital image correlation. A recent study proposed by Abshirini et al. (2016) [28] states also that the digital image correlation method is a reliable method for exploring the opening mode fracture. Based on the digital image correlation method Abshirini et al. [30] propose an experimentally investigation of stress intensity factors of centrally PMMA cracked Brazilian disc.

In fact, in the last 20 years, digital image correlation (DIC) has shown that it is a valuable non-contact technique for measuring kinematic fields during a mechanical test [21–28]. In the present study, DIC has been combined to Brazilian test. Different aspects have been studied such as crack's initiation and its propagation in order to evaluate energy release rate which allows an indirect characterization of thermal shock resistance. From the evolution of strains on the surface of sample during the first stage of the test, a convenient and a practical method for determination of elastic properties is also proposed.

2. Experimental

2.1. Material

The main purpose of the present paper is to give a new insight about Brazilian test through the combination with digital image correlation. Even if different refractories have been characterized by this route, only results on a magnesia hercynite composite are reported here as a typical example of industrial refractories developed for thermal shock resistance. Samples were provided by RHI through a FIRE (Federation for International Refractory Research and Education) research program dedicated to dense refractories with enhanced flexibility for thermal shock resistance. Developed for cement rotary kilns, the introduction of hercynite ($FeAl_2O_4$) aggregates in a magnesia (MgO) matrix promote the flexibility of the material [31]. In fact, a smaller thermal expansion of hercynite ($8.3\ 10^{-6}\ K^{-1}$ in the 20–1000 °C domain), compared with magnesia matrix ($13.7\ 10^{-6}\ K^{-1}$ in the 20–1000 °C domain) [27] induces an initial network of micro-cracks during the cooling stage after sintering which induces a non-linear stress-strain law in tension and enhance the strain to rupture.

Table 1
Reference properties of magnesia hercynite refractory obtained by ultrasonic measurements.

Microstructure	Density ($g\,cm^{-3}$)	3.01 (+/−0.02)
	Open Porosity (%)	15.11 (+/−0.29)
Elastic properties (ultrasonic measurements)	Young's modulus E (GPa)	39.26 (+/−3.56)
	Poisson's ratio ν	0.160 (+/−0.02)

Fig. 1 provides a backscattered SEM (Scanning Electron Microscopy) micrograph of magnesia hercynite. This analysis has been combined with an energy dispersive spectroscopy to determine the chemical composition of each phase. The picture shows the composite microstructure with the presence of hercynite inclusions surrounded by the magnesia matrix. The results of Energy Dispersive Spectroscopy (EDS) microanalysis are also given in Fig. 1.

2.2. Ultrasonic measurements

In order to obtain reference values for elastic properties of the material, first measurements were managed by ultrasonic waves in transmission mode at room temperature. From longitudinal (V_L) and transversal (V_T) wave velocities, Young's modulus (E), shear modulus G (Pa) and Poisson's ratio (ν) have been evaluated using the following equations:

$$E = \rho \cdot \frac{\left(3 \cdot v_L^2 - 4 \cdot v_T^2\right)}{\left(\frac{v_L^2}{v_T^2} - 1\right)} \quad (1)$$

$$G = \rho . v_T^2 \quad (2)$$

$$\nu = \frac{E}{2 \cdot G} - 1 \quad (3)$$

where ρ is the apparent density.

The elastic properties for magnesia hercynite material obtained using ultrasonic way are resumed in Table 1. Porosity and density are also reported in this table. It is interesting to note here that the obtained value of Young's modulus (about 39 GPa) is rather low in comparison to intrinsic properties of magnesia (312 GPa) and hercynite (248 GPa). Thus, even considering the decrease in Young's modulus which can be attributed to the porosity (about 30–40% in accordance to the 15% of porosity), this low value of Young's modulus for the studied material results mainly from the initial network of microcracks induced in magnesia matrix by the hercynite inclusions.

2.3. Brazilian test setup

Brazilian test is an indirect tensile test which was originally developed to determine the quasi-static tensile strength of concrete materials [7–9]. As it can be seen in Fig. 2, a compression load is applied vertically on the diameter of a rather thin disk (typically 50 mm in diameter and 10 mm in thickness). The final rupture takes

Please cite this article in press as: Y. Belrhiti, et al., Combination of Brazilian test and digital image correlation for mechanical characterization of refractory materials, *J Eur Ceram Soc* (2017), http://dx.doi.org/10.1016/j.jeurceramsoc.2016.12.032