Fracture behaviour of magnesia refractory materials in tension with the Brazilian test

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\textbf{ABSTRACT}

In this work, the tensile failure of magnesia, rebound magnesia-chrome and chrome-containing magnesia-spinel refractories under the Brazilian test were investigated. The digital image correlation and acoustic emission were applied simultaneously for ensuring the validity of Brazilian test and studying the fracture process. The brittle refractories fail abruptly while reaching their load peaks because of the unstable crack propagation. However, the chrome-containing magnesia-spinel refractory shows a reduced brittleness due to the pre-existing micro-cracks, which promotes quasi-stable crack propagation evidenced by the nonlinearity in the pre-peak region and the softening in the post-peak region. Besides, the thickness-to-diameter ratio has a great influence on the fracture behaviour, which also shows brittleness dependence. The fracture behaviour of rebound magnesia-chrome refractory varies from brittle to less brittle while the thickness increasing from 10 mm to 50 mm. The quasi-stable crack propagation favors the central crack initiation and ensures the tensile failure under the Brazilian test.

1. Introduction

Refractory materials are widely used as the lining of high-temperature furnaces. Thereinto, magnesia-chrome materials were one dominant variety of basic refractories owing to their high refractoriness and corrosion resistance [1]. Using the Cr\textsubscript{2}O\textsubscript{3} containing refractories in the alkaline atmosphere and at high temperature, the Cr(III) tends to transform into the Cr(VI) if the oxygen activity is large enough, which is harmful for health and environment [2]. This has led to the consideration of replacing the magnesia-chrome refractories by other chrome-free refractories, such as the MgO-MgAl\textsubscript{2}O\textsubscript{4}, MgO-FeAl\textsubscript{2}O\textsubscript{4} etc. [3–5] Especially, the magnesia-spinel refractories serve well in the transition and burning zone of cement rotary kiln, which reduce the application requirement of magnesia-chrome materials. However, in many secondary metallurgy processes and severe environments, for example, waste incinerators, the Cr\textsubscript{2}O\textsubscript{3} containing refractories are still irreparable because of its excellent resistance to aggressive matters and high temperature performance [6,7]. Thus, many research work studied the approaches for inhibiting the leach of toxic Cr(VI) based on its transformation mechanism [8]. It was found that the formation of solid solution and spinel decreased the possible leaching of Cr(VI). It was also proved that the addition of Cr\textsubscript{2}O\textsubscript{3} into the magnesia-MgAl\textsubscript{2}O\textsubscript{4} spinel refractory materials improves its thermo-mechanical strength as well as the corrosion resistance caused by the Al\textsubscript{2}O\textsubscript{3}-Cr\textsubscript{2}O\textsubscript{3} solid solution, and the Cr(VI) formation is limited [9,10].

In service, refractory materials undergo high temperature, thermal shock, mechanical stresses, corrosion and erosion. The chemical corrosion and thermal shock are the predominant factors causing the failure and limiting the campaign life of refractory linings. The corrosion mechanisms of Cr\textsubscript{2}O\textsubscript{3} containing refractories were intensively studied [11,12]. The parameters of mechanical properties and thermal shock resistance of magnesia-chrome refractories as well as of the magnesia-spinel refractory with Cr\textsubscript{2}O\textsubscript{3} addition were also investigated [10,13]. However, the detailed fracture behaviour and thermal shock resistance of these materials are much less understood. As the replacement materials for chrome-containing refractories, the fracture behaviour of magnesia spinel refractories were investigated [14–16]. Previous work demonstrated that the thermal mismatch between magnesia matrix and spinel aggregates induces microcrack network in the structure, which plays an important role in promoting nonlinear fracture behaviour and development of the fracture process zone.

For quasi-brittle materials, the tensile stress is rather critical, and their crack initiation is significantly related with the tensile strength. Tensile tests of quasi-brittle materials could be categorized into direct

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and indirect ones. However, the challenges associated with the direct tensile test (i.e. sample preparation, clamping and alignment) make the indirect tests more favorable. Among these indirect tests, the Brazilian splitting test, which was developed by Carneiro and Akazawa in 1943 [17,18] and standardized by the International Society for Rock Mechanics (ISRM) as well as the American Society for Testing and Materials (ASTM) [19,20], is very commonly used for quasi-brittle materials, such as concrete, granite, refractories and shale etc. The application of the Brazilian test favors the determination of tensile strength, failure process, elastic modulus, Poisson ratio, toughness as well as their impact factors, for instance the geometry parameters, the layer orientation process, elastic modulus, Poisson ratio, toughness as well as their influence on the validity of the Brazilian test [26]. Until now, many researches on ensuring the tensile failure of the Brazilian test and improving the test methods have been performed. Regarding the loading apparatus, some researcher placed cushions between the loading plates and disc to mitigate the load concentration at the loading points [27]. Others proposed to use steel arcs, which contribute to the central crack initiation during Brazilian testing [28]. Whereas, the dimensions of jaws, which should be also related to the specimen geometry, are sensitive especially to their curvatures. The ISRM suggested that the radii of the loading jaws shall be 1.5 times that of the Brazilian disc. Regarding the shape of specimen disc, the flattened Brazilian test as well as the cracked Brazilian test were developed to further study the mode-I fracture of quasi-brittle materials [29,30]. Besides, analytic studies were also extensively performed for better understanding the fracture process and ameliorating the stress distribution of the Brazilian test.

The Brazilian test normally ends with a sudden failure of the disc specimen reaching the peak load. Besides the measurement of the tensile strength, lots of attentions have been paid on the acquisition of more information from a single test. Thus, different Brazilian test strategies and investigation methods have been developed and involved, such as the dynamic Brazilian test, cyclic Brazilian test, numerical simulation, strain gauge technique, acoustic emission (AE) and digital image correlation (DIC) etc. [31–35]. Thereinto, the application of AE and DIC techniques with the Brazilian test is very inspiring due to its non-destructive, non-interfering and entire fracture process real time monitoring. Lavron et al. characterized the Kaiser effect and fracture of limestone by cyclic Brazilian tests together with acoustic emission (AE) [36]. Boulekbache et al. studied the failure mechanism of fiber reinforced concrete under the Brazilian tests using digital image correlation (DIC) [37]. Owing to the application of DIC, the crack initiation was located. In the refractory field, the DIC technique has already used with the Brazilian test for determining the fracture process and elastic properties (Young’s Modulus and Poisson ratio) of magnesia hercynite spinel refractory material [38].

In the present work, the Brazilian test is adopted for investigating the tensile failure of three different magnesia refractories: pure magnesia, magnesia-chrome and chrome-containing magnesia-MA spinel. Although the Brazilian test has been widely studied, less attention has been paid on the influence of material properties (especially brittleness) on the measured results. With the application of DIC and AE techniques, the different mechanical parameters, fracture behaviour and size effect will be studied.

2. Experimental

2.1. Investigation methods

2.1.1. Brazilian test

During the Brazilian test, a disc specimen is loaded in the diametrical direction and fails due to the induced tensile stress normal to the vertical diameter. In fact, the disc suffers both compressive and tensile stress as shown in Fig. 1(a): the compressive stress close to the loading area and tensile stress near the central part, which possibly result in the cracking and cracking of quasi-brittle materials. To ensure the tensile failure under the Brazilian testing, the compressive principal stress should not exceed three times of tensile principal stress presented at the center of tested specimen [24]. Thus, the arc jaws are used as the loading blocks for avoiding the stress concentration caused by the flat plates and the undesired compressive damage. Despite the difference in the Brazilian test procedure and strategy, the suggested and general formula for evaluation of tensile strength $\sigma_t$ is:

$$\sigma_t = \frac{P_{\text{max}}}{\pi DL} \quad (1)$$

where, $P_{\text{max}}$ is the maximum applied load, $D$ and $L$ are the diameter and thickness of cylindrical sample.

The experimental set-up is shown in Fig. 1(b). The loading of disc samples was performed using a displacement-controlled testing machine with a loading rate of 0.05 mm/min, of which the load cell could provide a compressive load up to 100 kN. According to Wu et al., the loading rate has a significant influence on the determination of tensile
strength [29]. It refers that the higher loading rate contributes to a higher measured value, whereas the quasi-static tensile strength could be only achieved at a low loading rate. In addition, the thickness to diameter ratio is another key factor of the Brazilian test. Theoretically speaking, the specimen with a low thickness to diameter ratio is closer to plane stress. For this work, disc samples with a diameter of 50 mm and a thickness of 10 mm were used for investigating the tensile failure of studied refractory materials under the Brazilian test. Besides, there are also researches focused on the comparison of Brazilian test under plane stress and plane strain conditions [39]. Some even recommended that the thickness to diameter ratio should be equal to one [40]. To study the size effect of Brazilian test, magnesium refractory specimens of another two thicknesses were additionally tested, which have thickness of 25 mm and 50 mm, respectively.

2.1.2. Methodology for fracture detecting

The location of crack initiation and fracture pattern are very important for the validity of the Brazilian test and the investigation of material properties under mode I failure with this indirect test. In the present study, the digital image correlation (DIC) and acoustic emission (AE), which are nondestructive testing methods, were simultaneously used throughout the whole Brazilian test for monitoring the fracture process of studied materials. The DIC tracks the homologous points on the specimen surface at different deformation stage and calculates the full-field displacement/strain. The AE detects the so-called stress-wave emission signals, which are generated by the irreversible deformation, initiation and growth of cracks (micro-/macro) etc.

As shown in Fig. 1(b), a monochrome CCD camera with a resolution of 2452 × 2056 pixels² was used to record images of the front surface. Images, which are required for further DIC analysis, were captured during the Brazilian test with a frequency of 1 frame per second. A random speckle pattern was applied at the front surface of the specimen by spraying white dots on a thin black substrate for increasing the contrast and accuracy. For guaranteeing an adequate lighting, two LED light panels were equipped, which provide constant and full-contrast and accuracy. For guaranteeing an adequate lighting, two LED light panels were equipped, which provide constant and full-contrast and accuracy. For guaranteeing an adequate lighting, two LED light panels were equipped, which provide constant and full-contrast and accuracy. For guaranteeing an adequate lighting, two LED light panels were equipped, which provide constant and full-contrast and accuracy. For guaranteeing an adequate lighting, two LED light panels were equipped, which provide constant and full-contrast and accuracy. For guaranteeing an adequate lighting, two LED light panels were equipped, which provide constant and full-contrast and accuracy. For guaranteeing an adequate lighting, two LED light panels were equipped, which provide constant and full-contrast and accuracy. For guaranteeing an adequate lighting, two LED light panels were equipped, which provide constant and full-contrast and accuracy. For guaranteeing an adequate lighting, two LED light panels were equipped, which provide constant and full-contrast and accuracy. For guaranteeing an adequate lighting, two LED light panels were equipped, which provide constant and full-contrast and accuracy. As the evaluation of the obtained images was performed with the computer software MatchID 2D developed by University of Leuven. According to the principle of DIC, a small region (subset) of the specimen surface at the unloaded image is traced to the same region at the loaded image and the distance between the center of two neighboring subsets (step size) is related to the intensity of the homologous points. In the present study, a subset size of 31 × 31 pixels² and a step size of 6 pixels were chosen. On the rear side of the disc specimen, two AE sensors having a frequency range from 50 kHz to 400 kHz were attached to the specimen symmetrically along the horizontal diameter of the disc. The distance between two sensors was 30 mm. Both AE channels were enhanced by a preamplifier with a gain of 40 dB and the minimum threshold of AE signals was 40 dB to remove the environment noise and the signal from the loading machine. The sampling rate of AE signals was 3 MHz. As an advantageous in-situ fracture monitoring method, AE technique can detect the parameters including the AE counts, energy, AE hits, hits duration and amplitude etc. The AE parameters identify information about the internal damage of studied materials.

2.2. Materials

The research interest of this paper is to investigate the tensile failure mechanism of Cr₂O₃-containing magnesia-MA spinel refractory material comparing with pure magnesia refractory as well as the magnesia-chrome refractory. Furthermore, deeper insight concerning fracture of quasi-brittle materials with different brittleness is expected from the Brazilian test results combined with DIC and AE techniques. The refractory materials are commercial products provided by the Ruitai Technology Ltd. and their main chemical compositions are shown in Table 1. Magnesia-chrome refractory, which is rebound, contains approximately 21% of Cr₂O₃ and is marked as MgO-Cr in the present manuscript. Due to the pre-fused magnesia-chrome raw material and high firing temperature, the rebound magnesia-chrome refractory is denser and the ceramic bond is more intense compared with the silicate bonded ones or even the direct bonded ones. The fused magnesia-chrome refractories are composed of the periclase with secondary chromite precipitates, which vary from 1 to 50 μm in size. Besides the secondary intragranular chromite, the intergranular chromite can also be observed (Fig. 2). Compared with fused magnesia refractory (MgO), the Young’s Modulus and cold crushing strength of MgO-Cr are inferior because of the co-existence of chromite in the fused grain and matrix. The CCS shown in Table 1 is collected from the technical data sheet of these commercial products as a reference.

Due to the thermal expansion misfit between magnesia matrix and magnesia aluminate spinel, a microcracking network is generated in the magnesia-MA spinel refractory, which enhances the thermal shock resistance [14]. On the basis of magnesia-MA spinel, chrome is added to produce the magnesia-aluminate-chrome brick, termed as MgO-MACr in this paper. The MgO-MACr refractories are commonly applied in the zinc volatility kiln, roofs of nonferrous metallurgy furnaces and roofs of high temperature furnace for iron making industry attributed to their good thermal shock resistance and corrosion resistance. The content of Cr₂O₃ and Fe₂O₃ in MgO-MACr specimen is lower than that of MgO-Cr as shown in Table 1. According to the microstructure investigation, see in Fig. 2, the periclase containing chromite and magnesia aluminate spinel crystals is observed. The Cr³⁺ ions diffuse and a composite spinel mainly of the composition Mg₃(Cr, Al)₂O₆ precipitates in the periclase crystals along the MA spinel grain boundary. Fig. 3 shows the XRД patterns of three materials. The MgO-Cr consists mainly the composite spinel and periclase. However, the intensity of composite spinel peak decreases and magnesia aluminate spinel phase newly exhibits in the MgO-MACr compared to those in the MgO-Cr.

3. Results and discussion

3.1. Force-displacement curve

At least 4 specimens were tested for each material. Fig. 4 presents the selected force-displacement curves of MgO, MgO-Cr and MgO-MACr. Due to the pre-existing microcracks, the maximum load Pmax of MgO-MACr is only 27% of that of MgO. Meanwhile, there is chromite phase existing in the fused grains of MgO-Cr and its maximum load decreases compared with the MgO. The tensile strengths were calculated for these three materials respectively according to Eq. (1). Due to the small fracture area and high stored elastic energy, it should be noticed that the studied materials under the Brazilian test show unstable crack propagation, especially for MgO and MgO-Cr. While the fracture energy refers as the energy required for stable crack propagation. The integration of the area under the force-displacement curve till the load peak is denoted as the total strain energy W, which contains elastic energy and dissipated energy.

In most cases, the Brazilian test is merely adopted for tensile strength evaluation. However, the force-displacement curve implies more information besides the tensile strength. As seen in Fig. 4, the MgO and MgO-Cr experience sudden rupture of the specimens when the Pmax are reached, which indicates the higher brittleness compared with MgO-MACr. MgO has a much greater Pmax and displacement at Pmax than MgO-Cr. Its failure is more violent with force plunged by 60% with surface spalling near the loading contact (Fig. 5). The first crack propagation in MgO-Cr also causes a straight descending of load to 65% of its Pmax and follows with a short plateau due to further applied compressive load. While releasing the applied load, the separation of two halves of the brittle specimens becomes rather small due to the cohesive stress and friction between the crack faces.

For MgO-MACr shows a significant non-linearity in the pre-peak
region and a smaller stiffness compared with MgO and MgO-Cr. After the maximum load is reached, the crack opening and propagation progressively take place which contribute to a gradual diminishment of load rather than a sudden damage in the post-peak region. With further loading, the load will increase and decrease again erratically at certain point, which is caused by the compression of the disc. This part is not presented in Fig. 4 as it is not mere tensile failure behaviour. Besides, the MgO-MACr samples are loaded until the two halves are totally separated. According to the fracture patterns shown in Fig. 5, the entire failure process generally consists of the tensile stress induced cracking at the central part of the specimen and the compressive stress induced crushing near the loading contact.

Table 1
Main chemical compositions and selected properties of studied materials.

<table>
<thead>
<tr>
<th>Chemical Composition (%)</th>
<th>Bulk Density (g/cm³)</th>
<th>Open Porosity (%)</th>
<th>Young’s Modulus (GPa)</th>
<th>CCS* (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>MgO</td>
<td>95.05</td>
<td>0.35</td>
<td>0.001</td>
<td>1.56</td>
</tr>
<tr>
<td>MgO-Cr</td>
<td>59.65</td>
<td>5.06</td>
<td>21.22</td>
<td>9.86</td>
</tr>
<tr>
<td>MgO-MACr</td>
<td>75.02</td>
<td>11.01</td>
<td>5.70</td>
<td>3.05</td>
</tr>
</tbody>
</table>

Fig. 2. Microstructure of MgO-Cr (a) and MgO-MACr (b).

Fig. 3. XRD patterns of studied materials.

Fig. 4. Force-displacement curves and the mechanical parameters of studied materials.

3.2. Influence of the brittleness on the fracture on refractory materials

Due to the co-existence of compressive and tensile stresses, the fracture process (including the crack initiation and crack propagation) is essential for the validation of tension failure under the Brazilian tests and the investigation of the fracture mechanism of studied materials. However, it is difficult to observe the inherent failure by the naked eye or reveal it merely by the force-displacement curves. In this section, the evolution of strain field obtained by DIC and of AE characteristics is analyzed for providing effective information about the entire failure process of MgO, MgO-Cr and MgO-MACr, which show different brittleness.

As shown in Fig. 6, the force, AE counts, cumulative AE energy
curves as a function of time as well as the strain evolution of MgO, MgO-Cr and MgO-MACr are represented. For brittle refractory specimens (MgO and MgO-Cr), the elastic deformation and damage are less significant at the low force level. While reaching the force peak, the abrupt failure results in a burst of AE counts and a steep rising of the cumulative AE energy. After peak, the AE pulse return to a mild state again. This demonstrates the fracture process of brittle refractories as below: the materials remain intact and energy is accumulated in the early stage, and then the internal energy is released suddenly reaching its tensile strength. The higher the accumulated energy is, the greater is the release intensity. Based on the successively captured digital images, the DIC evaluation is performed and the horizontal strain map at different loading stages are exhibited above the curves. Because of the applied compressive stress along the vertical diameter, the deformation near the loading area is firstly observed. For the brittle MgO refractory, the unstable crack propagation abruptly takes place at load peak. The finale failure of MgO-Cr is also very quick. But its brittleness is reduced compared with MgO, which is justified by its smaller tensile strength. The vertical loading attributes to the horizontal deformation at the central area of the disc surface while approaching the load peak. Although the crack paths are already formed at the maximum load level for both MgO and MgO-Cr, the visible cracks are rather small which is near the center of the specimen and indicated with red arrows.

The MgO-MACr shows the reduced brittleness due to the pre-existing microcracks generated during the cooling process of the manufacture. The pre-peak nonlinearity and post-peak softening are obviously observed in the force-displacement curve. As seen from the Fig. 6, the maximum load of MgO-MACr is much lower and the AE counts amplitude are relative weaker but the release of AE signals lasts for longer time compared with the brittle MgO and MgO-Cr, which is attributed to the further opening of pre-existing microcracks, formation of new microcracks, irreversible deformation and friction before the load peak. As a result, the cumulative AE energy in MgO-MACr refractories is less while reaching the maximum load and the increasing amplitude of AE counts is also less sharp. The evolution of horizontal strain field obtained by DIC calculation demonstrates that there is deformation at the loading area caused by the diametrical loading at early stages. With the load increasing, microcracks appear in the central area due to the compressive load induced tensile stress normal to the vertical diameter. Instead of a single crack initiating at the exact center of specimen, a central zone consisting multiple microcracks is presented in MgO-MACr because of its heterogeneity and reduced brittleness. After the load peak, the cracks further propagate and consume large portion of energy. The macro-crack opening along the crack path is not continuous, as highlighted with red arrows. Once the discrete macro-cracks concatenate, the disc specimen totally fall apart.

According to the mechanical behaviour represented by the force-displacement curves and the fracture behaviour characterized by the DIC and AE of the studied materials, the whole history of damage could be divided into four stages: I) elastic deformation of the testing frame and specimen; II) pre-cracking stage: irreversible deformation and minor microcrack formation with relatively low energy dissipation; III) pre-peak nonlinear stage: more intensive microcracking and significant increase of energy dissipation; IV) fracture or softening: crack propagation. The four stages of the fracture process are easier distinguished for MgO-MACr than MgO and MgO-Cr owing to the quasi-stable crack propagation for less brittle material. Meanwhile, the quasi-stable crack...
propagation is also favourable for the crack initiation from the center based on the DIC observation.

As the DIC can calculate the full-field displacement of the specimen surface, it is used as the strain gauge to investigate the mechanical properties of studied refractory materials in the present paper. The frontal circular surface of the Brazilian test is used as the zone of interest for displacement evaluation by DIC technique. Six lines of interest are adopted for the displacement acquisition as are schematically shown in Fig. 7(a).

The vertical displacement of vertical diameter and the horizontal displacements of five horizontal lines (1 × horizontal diameter, 2 × 1/4 diameter height away from the center, 2 × 1/8 diameter height away from the periphery) are recorded for the whole mechanical test. The relationship between the vertical and horizontal displacements represent the horizontal expansion at different height levels under the squeezing of specimen by the loading jaw, which is shown in Fig. 7(b). The red lines express the evolution of displacement for the central lines, the black dotted lines and black solid lines are the displacement measured at the upper and lower part of the specimen, respectively. For the brittle materials, the failure is instantaneous and the displacement of five horizontal lines almost the same. Thus, only the central displacements are shown for MgO and MgO-Cr. As seen from Fig. 7(b), the horizontal displacement is rather small before the maximum load and remarkably increased at the load peak. Due to the low displacement and numerical uncertainty caused by DIC calculation method, the reliable ratio of the horizontal displacement/vertical displacement is difficult to obtain for materials MgO and MgO-Cr. However, the horizontal displacements of less brittle MgO-MACr slowly increase with the vertical displacement until 0.1 mm, which is the moment reaching 70% P_max. The estimated ratio of the horizontal displacement/vertical displacement approximately equals to 0.15. The horizontal displacements increase significantly further with vertical displacement due to the newly development of microcracks and further crack propagation. The displacement curves around load peak are framed and magnified. A steep increase of displacement is observed as MgO-MACr reaches its tensile failure limit. Meanwhile, the horizontal displacements along the central diameter and 1/4 upper line of interest are higher than the rest, which proves that the stress is not symmetrical and the main microcracking contributed to the failure is firstly formed at the center and upper quarter of the specimen.

3.3. Size dependent fracture analysis

According to the theoretical assumption, the plane stress is favourable for the Brazilian test to achieve the desired tensile failure. Thus, the thin discs or the lower thickness/diameter ratio discs were used in most Brazilian tests. However, it was also studied that the plane-stress and plane-strain condition of a Brazilian test showed similar load-displacement curves based on the finite element modelling [39]. Both ASTM and ISRM allow a relatively wide range of values for the specimen geometry in terms of the thickness to diameter ratio, which has a significant influence on the determined tensile strength by Brazilian tests [41]. In this section, the effect of thickness to diameter ratio on the fracture behaviour of MgO, MgO-Cr and MgO-MACr is studied.

All the specimens have the same diameter of 50 mm, while the thickness varies from the 10 mm, 25 mm to 50 mm. As seen from Fig. 8, the maximum force, the slope of the force-displacement curve (representing elastic stiffness) and total strain energy increase with the thickness to diameter ratio (T/D). If the total strain energy is divided by the fracture area, the variation of the calculated values is small with the T/D ratio. However, the calculated tensile strength decreased for MgO and MgO-Cr. The increase of T/D ratio has less influence on the tensile
strength of MgO-MACr. In addition, the T/D ratio has different influence on the fracture behaviour of refractory materials showing different brittleness. The MgO specimens abruptly rupture at the maximum load due to its high brittleness. The MgO-Cr investigated is the rebound magnesia-chrome refractory, and its tensile strength and accumulated elastic energy before cracking is lower, which results in a lower brittleness compared with MgO. Because of the unstable crack propagation, the MgO-Cr experiences also brittle failure while the fracture area is small. With increasing fracture area, a less brittle fracture behaviour is observed in Fig. 8(a), especially for the specimen with thickness of 50 mm. The MgO-MACr exhibits the lowest brittleness compared with MgO and MgO-Cr, owing to the existence of microcrack network in the structure. At the given specimen geometries, the pre-peak nonlinearity and post-peak softening are very significant for MgO-MACr.

As it is seen from the force-displacement curves, the MgO-Cr is very sensitive to the specimen geometry. With the thickness varying from 10 mm to 50 mm, its fracture behaviour turns from brittle to less brittle. As shown in Fig. 9, a burst of AE counts occurs at the load peak caused by the rupture of the specimen and returns to a mild state showing lower intensity of AE counts. A more detailed observation of the fracture process of MgO-Cr specimens with thickness of 10 mm and 50 mm shows large difference. The thicker specimens of MgO-Cr exhibit a quasi-stable fracture process: AE signals release continuously even at the early stage and the amplitude of AE counts reduces while reaching the peak load compared with MgO-Cr specimens with thickness of 10 mm.

Fig. 10 shows the correlation of the horizontal displacement and vertical displacement measured from the respective diameters at the peak load peak. The surface displacements are evaluated by DIC and the evaluation details are explained in Fig. 7. The MgO-MACr has a higher strain bearing capacity compared with MgO and MgO-Cr. It is obvious that the variation of the geometry impacts the results. With increasing thickness, the strain along the vertical diameter decrease owing to the combined effect of the rising stiffness and maximum load. Meanwhile, a higher horizontal strain is required for reaching the maximum load of the studied refractory materials.

In addition, the horizontal strain maps of each material at the moment of their maximum load is presented in Fig. 11. A comparison study clearly shows that the failure of brittle specimens is instantaneous, which makes it difficult to achieve initiation of the fatal crack in the center. The brittleness of MgO-Cr reduced by increasing the thickness to 50 mm and its crack initiates from the center. For the brittleness reduced MgO-MACr specimens, the microcracks initiate from the central area in the pre-peak region. While reaching the maximum load, great part of the crack path is already paved. The higher strain value and multiple cracks demonstrate its higher energy consuming and strain bearing capacity. The red arrows marked in Fig. 11 indicate the location of first macro-crack observed by naked eyes. The fracture behaviour of MgO-MACr and thicker MgO-Cr specimens proves that the brittleness reduction favors the quasi-stable crack propagation, the non-linearity and the central initiated cracking under the Brazilian test conditions.

4. Conclusions

Brazilian test is one of the most common indirect methods for determining the tensile strength of quasi-brittle materials. This paper reports the application of DIC and AE techniques in-situ monitoring fracture process together with the Brazilian test. The additional benefits of these techniques are to identify the validity of the Brazilian test, the fracture evolution as well as the failure mechanism of the studied materials under tension.

The fracture behaviour of three magnesia-based refractories was investigated. Due to the micro-crack formation induced by the additional chrome ore additive, the material MgO-Cr has a lower tensile strength and brittleness compared with MgO refractory. While reaching the maximum load, the abrupt failure takes place in materials MgO and MgO-Cr, which refers to the brittle fracture caused by the unstable crack propagation. The higher energy cumulation in the material MgO before the load peak also results in a more violent failure. According to the DIC and AE observation, there are very low deformation and AE signals in the pre-peak region. The momentary crack propagation and AE counts burst happen at their maximum loads for these two brittle refractories. MgO-MACr refractory has the lowest tensile strength and brittleness compared with the other two (brittleness ranking: MgO > MgO-Cr > MgO-MACr) due to the pre-existing microcrack
network induced by the thermal mismatch. Although the Brazilian test is not that advantageous for achieving a stable fracture process due to the low fracture area and the relatively high applied loads, the brittleness reduction enables a quasi-stable crack propagation. The crack initiated from the center area of MgO-MACr specimen and continuous energy dissipation before the load peak contribute to the nonlinearity in the pre-peak region. Instead of sudden rupture at load peak, a softening phenomenon follows in the post-peak region of MgO-MACr. MgO-MACr shows a better strain bearing capacity. Based on the experimental characterization, the fracture process could be divided into different stages: elastic specimen deformation, pre-microcracking, pre-peak nonlinearity, and final softening and damage. In addition, the fracture patterns prove the mixed mode of the failure process: crushing near the loading area caused by the compressive stress and cracking in the central area caused by the tension normal to the vertical diameter.

The thickness to diameter ratio of a Brazilian test specimen has different influence on the mechanical properties and the fracture behaviour. The maximum load and total strain energy increase, while the tensile strength decrease with the thickness (except for MgO-MACr with thickness of 25 mm and 50 mm). The tensile strength of MgO-MACr is less influenced by the thickness to diameter ratio as its failure is quasi-stable. Besides, the fracture behaviour of MgO-Cr turns from brittle to less brittle fracture with the thickness increasing from 10 mm to 50 mm. The thick MgO-Cr specimen generates AE signals in the pre-peak region. The main reason for this phenomenon is that the strength of brittle materials showing instable fracture depends on the critical stress intensity factor caused by the critical flaw. The probability that at least one single flaw larger than an arbitrarily fixed size occurs rises with the specimen volume. Therefore, for a given load a larger stress intensity resulting in lower strength is expected for larger volume, and the lower strength contributes to the reduction of brittleness. By comparing all the test specimens, it could be concluded that the quasi-stable crack propagation of brittle reduced refractory materials (MgO-MACr and thick MgO-Cr) favors Mode I failure with central crack initiation under the defined Brazilian testing conditions in the present study.

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