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## Observation and quantification of the fracture process zone for two magnesia refractories with different brittleness

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### ABSTRACT

In this paper, the formation of the fracture process zone (FPZ) of industrially produced magnesia spinel and magnesia refractories was analysed using digital image correlation (DIC). Compared to pure magnesia materials, the magnesia spinel materials exhibited a higher amount of microcracks, causing a larger FPZ. A critical displacement, where the cohesive stress between the crack faces decreases to zero, is determined by analysing the development of the localized zone. Critical displacement determined from the changes of the FPZ width and length is used to determine the onset of macro-cracking and locate the crack tip. The development of the fracture process zone for a magnesia spinel initiates before reaching the maximum load, and the onset of the macro-crack is in the post-peak region. The FPZ size increases until the formation of a macro-crack takes place, but decreases afterwards. For the magnesia refractory, no pronounced FPZ could be detected.

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### 1. Introduction

In many industrial applications, refractory materials sustain considerable stain caused by thermal shock and mechanical loading. To achieve a high strain-to-rupture behaviour, the refractory material needs either a large strain at crack initiation or crack initiation at a smaller strain but a high resistance to crack propagation. By reducing the flaw size and increasing the strength, crack initiation requires a relatively high stress, which increases the associated strain and supports the first case above. However, once a crack initiates, sudden and complete material failure may occur. For the second case, the material may exhibit reduced strength and a similar specific fracture energy, resulting in reduced brittleness [1–3]. Due to pre-existing microcracks, cracks initiate easily, but the development of the process zone enhances the resistance to crack propagation [4]. In this research, a magnesia spinel material and a pure magnesia material are studied to understand the influence of brittleness and microstructure on the fracture process. These materials are typical refractories representing relatively brittle (pure magnesia) and less brittle (magnesia spinel) mechanical behaviour. The microcrack network present in magnesia spinel refractories is formed during the cooling stage of the firing process due to the thermal misfit between the spinel and magnesia. This microcrack

formation was simulated in the work of Fasching et al. [5]. The addition of spinel decreases the strength and increases the thermal shock resistance. The influences of the spinel content and size on the thermomechanical behaviour of magnesia spinel have been previously studied. Examples include the works of Grasset-Bourdel et al. and Aksel et al. [6–8].

Refractories are heterogeneous materials consisting of grains and fines. When containing pre-existing microcracks, refractory materials exhibit pronounced deviations from pure linear elastic mechanical behaviour [3]. These deviations are also reported by Petersson [9] for concrete and similar materials. This author distinguishes between three zones in the loaded material: the elastic zone, the quasi-plastic zone and the process zone. Elastic behaviour is observed far from the crack tip. Within the quasi-plastic zone, the stress-strain relation is nonlinear and the stress increases with the strain. In the process zone, the stress decreases with increasing strain. Fig. 1 shows the stress-strain relationship within the elastic and quasi-plastic zones (Fig. 1(a)) and the stress-strain relationship within the process zone (Fig. 1(b)).

According to Hillerborg et al., the fracture process zone (FPZ) is defined as the region ahead of the traction free crack tip [10]. This region comprises energy consuming processes in the so called frontal process zone and the following process wake (Fig. 2(a)). The frontal process zone is especially composed of distributed microcracks. In the process wake, stress is still transferred between the faces of an already localized crack. Phenomena such as grain bridging and interlocking may be responsible for the load transfer. In this

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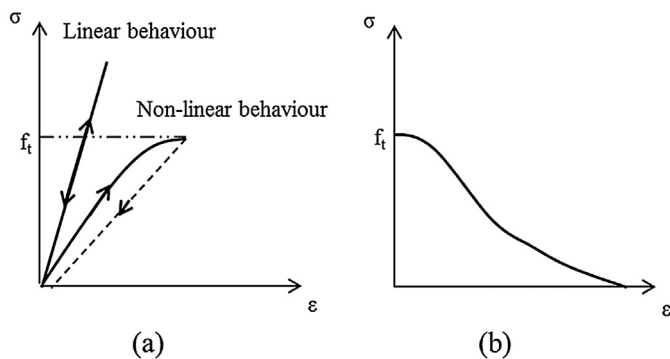


Fig. 1. Illustration of material stress-strain behaviour in elastic and quasi-plastic zones (a) and in process zone (b).

work, the FPZ will be identified and depicted without distinguishing between the frontal process zone and the process wake. The definition of the FPZ as quoted above was first applied for concrete, a material showing several similarities to refractories [10]. The development of the FPZ in quasi-brittle materials, such as concrete [11,12], rock [13] and refractories [2], has a significant influence on the fracture behaviour. Its understanding and characterization is essential for the safe design and application of many different structures [14]. The FPZ development in quasi-brittle material is dependent on the specimen geometry [15,16], the type of loading [17], the microstructure [18] and further material properties, such as the maximum aggregate size [19,20]. Bažant et al. [20] showed that the fully developed FPZ length of concrete can be twelve times the maximum grain size. With the fibre addition in a plain concrete, a material with larger FPZ was observed by Tschegg et al. [18]. For magnesia refractories, the addition of spinel to a pure magnesia product reduces the material brittleness and enables the development of a sizable FPZ [2]. The mechanical behaviour of the FPZ can, for example, be described by a cohesive zone model: when a microcrack localizes and propagates, the stress transferred between its faces begins to decrease and vanishes when the local displacement  $u$  equals or exceeds the ultimate displacement  $u_{ult}$ . The further crack path with  $u$  exceeding  $u_{ult}$  (no stress transferred between the crack faces) is addressed as macro-crack here. The relation representing the decrease in stress with increasing strain is called the strain softening behaviour (Fig. 2(b)) [10]. Here, the initial notch length is  $a_0$  and  $a$  is the traction free macro-crack length (Fig. 2(a)). The transition between microcrack and macro-crack, which is a prerequisite to determine the FPZ length and crack length, is important for the study of fracture process. Several different methods

have been adopted to determine the onset of macro-cracking. Zechner et al. used a tension test to determine the ultimate displacement [21]. This is the most accurate and direct method. However, it is improper to use this method for quasi-brittle materials due to the potential for unstable crack propagation under direct tension. Skarżyński et al. observed a discontinuous crack mouth opening displacement (CMOD) at the onset of the macro-crack propagation in concrete subjected to three-point bending [22]. The CMOD measured for the two refractories examined in this work did not exhibit such a discontinuity. Wu et al. used an empirical strain softening relation to obtain the ultimate displacement for concrete [15].

Until now, various techniques, such as scanning electron microscopy [23], acoustic emission technique [24], computed tomography [12], moiré interferometry [25] and digital image correlation technique [15,21,22], have been utilized to investigate the fracture process. For instance, a sizable FPZ was detected for magnesia spinel refractory material by AE investigation [17]. Meanwhile, different techniques can be combined for the evaluation, i.e. Rouchier et al. [26] used the DIC together with AE technique to investigate the fracture of fibre reinforced mortar. The 2D Digital image correlation technique is a non-contact full-field measurement method that is used to visualize surface displacement by tracking homologous points on the specimen surface at different loading stages. It does not interfere with the development of the FPZ. Due to its non-destructive nature, high availability and simplicity, DIC is widely used to study fracture processes. A random distribution of gray levels, which could be the nature texture of the material surface or paint applied before testing, is required for the DIC evaluation.

In the domain of refractory materials, DIC has been already used in some fracture studies, i.e. for a MgO-based refractory castable [27], for a fiber reinforced refractory castable [28] and for magnesia refractory materials [29]. DIC have been found to be very effective at detecting crack propagation and crack networks. In this study, more attention is paid to the characterization of the fracture process zone development in the specimens. The FPZ size is investigated in this paper, which necessitates the identification of the transition between microcracking and the onset of macro-cracking. The size of the FPZ allows conclusions to be drawn on the crack propagation resistance of the material being examined.

## 2. Materials and investigation method

### 2.1. Materials

To investigate the influence of brittleness and microstructure on the fracture process, two typical refractory materials were selected:

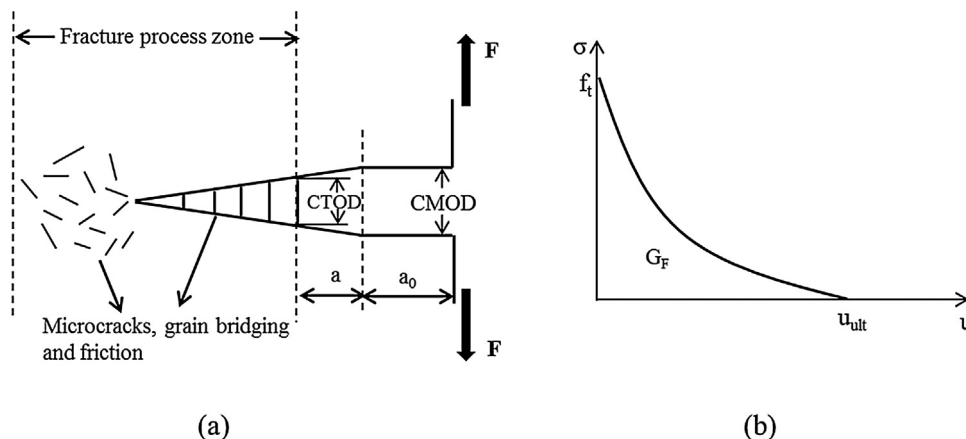


Fig. 2. Schematic representation of the fracture process zone for refractories (a) and of the strain softening behaviour (b).

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