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Compaction and shear failure of refractory mortars – effects of porosity and binder hardening

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ABSTRACT

With the aim to correlate the global properties of refractory mortars with the micro-mechanical processes, a series of uni-axial compression and shear tests was conducted. The test program was developed with the view that the shear grain slip and cracks are frequent failure mechanism under compressive loads. The micro-structural changes during compression were monitored by X-ray micro focus computed tomography. Discrete element modelling was used to highlight the effects of individual factors of influence. Mortars with a water glass binder of different maturity were tested. In compression the mortars demonstrated cracking and pore closure. Shear tests showed that the failure process consists of multiple local failure events. The combined effects of the porosity and immature binder promote increased tendency for crack branching and arrest. This results in low shear strength and high compressibility. Cohesion and interlocking between the grains prevents crack branching and increases the stiffness and the strength.

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1. Introduction

Refractory mortars are disordered ceramic materials [1–3]. Their micro structure features grains of different sizes, pores and the binder. The mortars serve in joints of masonry linings of high temperature installations. In service, the lining is exposed to complex thermo-mechanical loads [4,5]. Studies of mechanical properties of refractory mortars have predominantly concentrated on the acquiring of global material properties in tension [6], shear [6,7] and compression [1,8,9]. Analysis of the failure process has been done only for sintered mortars after compression at high temperatures [1]. SEM micrographs demonstrated inter-particle slips and shear cracks.

For disordered materials the cohesion and friction between the grains determines the failure both in compression and in shear [10]. The failure processes in both modes are closely related. On microstructural level the compressive failure, e.g. in porous rocks and civil engineering mortar, can be either by the formation of shear bands, or by pore collapse or by a combination of these mecha-

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http://dx.doi.org/10.1016/j.jeurceramsoc.2016.08.040 0955-2219/© 2016 Published by Elsevier Ltd. nisms [11,12]. The compressive failure mode is determined both by the material properties and by the ruling stress state. Lower grain cohesion and multi-axial compression increase the probability of the failure by pore collapse (material crushing) [12–14]. Stiffer materials and lower lateral constraints favour the failure by cracking. The collapse of the pores is enabled by the damage zone developing around the pore. In materials with extensive porosity the damage zones may overlap. In general, the porosity is shown to be reversely proportional to the stiffness of the civil engineering mortars [15–17], the shear and compressive strength, and the onset stress of the pore collapse in sandstone [18]. In mortars, the binder determines the friction and bond between the grains [1,19]. The changes of the binder are known to influence the strength and stiffness of the mortar.

The aim of this paper is to analyse the failure mechanisms of refractory mortars and to highlight the influences of the binder maturity and the pore size distribution. The changes of the mortar's micro structure during progressive compressive loading have been visualized using non-destructive X-ray micro focus computed tomography (micro CT). Direct shear tests have been performed to quantify the cohesion between the grains and to get a better understanding of the mechanisms of shear slip. The latter is expected to be relevant both for compression and shear failure. Modelling according to the Discrete Element Method (DEM) has been performed to further correlate the failure process with the global properties and

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the micro structure. The tests have been carried out on mortar with water glass binders of different maturity. This material has been chosen as it shows high variability of mechanical properties during mortar hardening [1,20]. As a reference, mortar featuring no binder has also been tested. This paper further elaborates and extends the discussion on failure mechanisms of refractory mortars started in [1].

2. Materials and experimental procedures

2.1. Materials

A water glass mortar (WG) and mortar featuring no binder (NB) were prepared and tested. The composition and preparation method was identical to that reported in [1]. Alumina grains were used in both mortars. The composition of the grains was 99.7% of Al₂O₃ and 0.3% of Na₂O. The grains had bi-modular grain size distribution. The most frequent sizes for small and large grains were 2,5 and 70 μ m, respectively. The total amount of water used to prepare both mortars was 17 wt.%. The water added with the water glass solution was included in the total water balance. A water glass solution of 24 wt.% in the amount of 8% was used.

2.2. Experimental and analysis procedures

Mortars were prepared in a standard rotary blade mixer. Samples for the shear and compression tests were formed using plexi-glass parts. Prepared samples were kept in (uncontrolled) indoor conditions for certain amount of days before testing. Further in the paper, the age of the test specimens is indicated by a number of drying days followed by "dd", e.g. 2dd. Shear and compression tests were performed at room temperature.

Uni-axial compression tests were performed on samples that featured flat layers of mortar. Samples of two types were tested. Samples of the first type were as described in [1]. They featured three layers of mortar with a diameter of 50 mm and a height/diameter ratio of 0,06. These dimensions are representative for the thickness of a mortar joint in refractory lining, which is generally between 1 and 6 mm. Samples of the second type were compressed in combination with the micro focus X-ray computed tomography (micro CT). Micro CT allows a non-destructive visualization of the material's inner structure, based on the attenuation of X-rays. The principles of micro CT analysis are given in [21–24]. The geometry of the samples was chosen regarding the abilities of the micro CT equipment available at KU Leuven's Materials Engineering Department. The samples featured two layers of mortar and three disks of plexi-glass. The discs had a diameter of 9 mm and a height of 2 mm. The height/diameter ratio of one mortar layer was 0,22. A special sample holder was used that allowed keeping the sample under load when the sample is transferred from the mechanical testing frame to the micro CT scanner. The test procedure and the design of the sample holder are described in detail in [23]. Tests were performed with constant displacement rate of 0,12 mm/min. The compressive strains after every loading step were measured from the micro CT images by calculating the sample height changes at 4 locations. The stresses were calculated by dividing the measured force by the original cross-section of the sample.

X-ray tomography was performed using a Micro CT Skyscan 1172 system. The following set-up was used: source voltage 100 kV, computer frame 2000×1048 , filter Cu-Al, rotation step 0,4 deg, frame averaging 3. The image pixel size was 8,36 μ m. The image analysis was done using the software AxioVision 4.8. For calculating the porosity and the analysis of the pore size distribution a simplified semi-3D analysis (multiple stacked slices) was done as it



Fig. 1. Compressive stress-strain curves of the samples featuring mortar joints with the height/diameter ratio of 0,06.

allowed for the needed observations. Only the pores larger than the pixel size were accounted for. The diameter of a pore was obtained from the area of the pore assuming it to be a circle. The pore size distribution was analysed using 11 2D cross sections orthogonal to the direction of compression. The sections were located at equal distances from each other. The distance was obtained by dividing the current thickness of the sample by 12. On one cross-section, the density of pores of a given size was obtained by counting the pores and dividing this number by the surface of the cross-section. For a set of cross-sections, the average pore densities and respective standard deviations were obtained.

The shear samples consisted of two prismatic plexi-glass parts with aligned cylindrical channels filled with mortar (Fig. 6a insert). The prisms had a height, width and thickness of 30, 25 and 10 mm, resp. The channels were drilled along the thickness axis. The diameter of the channel in one part was 9 mm and in the other it was 8 mm. This guaranteed that the mortar cross-section loaded in shear had a constant diameter of 8 mm. The shear developed at the interface of two parts due to a shear displacement of one of the parts. The loading was applied with a constant displacement rate of 0,2 mm/min in the test frame described in [23], and no lateral forces were applied during testing. The displacement was measured from the cross-head travel. From the shear load displacement curves the global strength and specific fracture energy were calculated. The former was the maximal stress registered during the sample loading. The specific fracture energy (Gf) was obtained by calculating the area under the force-displacement curves and dividing it by the sheared cross-section. At least 30 samples were tested in shear for each material.

Shear failure was modelled using a basic DEM model. The modelling is performed using the software PFC2D 4.00 of Itasca Consulting Group Inc. [25]. Only qualitative likeness was expected between the DEM modelling results and the lab tests. The modelling was done following the basic principals of DEM modelling of cohesive granular materials discussed e.g. in [16,26,27].

3. Results

3.1. Compression tests and CT-scan

Obtained stress-strain curves are presented in Fig. 1 for the regular tests, and in Fig. 2 for the samples which were tested with micro CT. In all samples, the compressive loading produced a consistent stress increase. No strain softening is seen. The stress-strain curves of NB 2dd and WG 14dd are almost linear. The curve obtained for WG 2dd shows stiffening of the material with progressive loading (strain hardening). The response of the WG 14dd is much stiffer than that of the WG 2dd. E.g. at the same strain of some 12% the

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