Available online at www.sciencedirect.com

ScienceDirect

Journal of the European Ceramic Society 34 (2014) 523–531

www.elsevier.com/locate/jeurceramsoc

Compressive fatigue behaviour of refractories with carbonaceous binders

K. Andreev[a,*], M. Boursin[a,b], A. Laurent[a,b], E. Zinngrebe[a], P. Put[a], S. Sinnema[a]

[a] *Tata Steel R&D, PO Box 10000, 1970CA, IJmuiden, The Netherlands*

[b] *Ecole Nationale Supérieure de Céramique Industrielle (ENSCI), Centre Européen de la Céramique (CEC), rue Atlantis 12, 87068 Limoges, France*

Received 25 June 2013; received in revised form 26 July 2013; accepted 6 August 2013
Available online 6 September 2013

Abstract

Furnace linings of magnesia-carbon and micro-porous carbon bricks experience cyclic compressive loads. An experimental programme has been carried out to assess the sensitivity of these materials to compressive fatigue failure. Next to room temperature tests, a number of high temperature tests have been performed. Results of the fatigue tests have been analysed together with the data of the monotonic stress–strain loading and creep tests. Compressive fatigue failure has been seen in both the materials. The less brittle material has shown lower fatigue life. The curve relating the fatigue strains with the amount of cycles has been of classical sigmoid shape with three phases. The strain rates of the secondary (linear) phase have shown good correlation with the number of cycles to failure. The grain–matrix interface has been found to play the critical role in the initiation and propagation of the fatigue cracks.

Keywords: Fatigue behaviour; Compressive behaviour; Creep; Refractories; Quasi-brittle failure

1. Introduction

During the service in the lining of industrial furnaces refractory ceramic bricks are often exposed to compressive cyclic loads.[1,2] The compressive loads result from constrained thermal expansion. The cycles coincide with the production cycles of the furnace or can occur due to the fluctuation in the process parameters. Knowledge of material behaviour in the conditions of subcritical cyclic loading is essential for the furnace life prediction and selection of the materials.

Refractory materials (refractories) of most classes, castables (refractory cement concretes) or shaped bricks, feature the micro-structure of large grains of several millimetres embedded in the matrix of small grains, pores and micro-cracks.[2,3] This makes them similar to the civil engineering concrete. Similar experimental approaches are used for refractories and the civil engineering concrete. Micro-cracks play an important role in the process of crack propagation which lends refractories similarity with toughened structural ceramics. Similarly to those materials refractories demonstrate quasi-brittle failure. Most investigations of thermo-mechanical behaviour of refractories and their structures consider material properties obtained by monotonic loading.[4–7] Some researchers address the creep behaviour.[8–11]

Very little is known about the fatigue resistance for any class of refractory ceramic materials. For ceramic materials in general, the micro-structural influences on the fracture by fatigue are similar to those by overload fracture.[12,13] The fatigue crack propagation is influenced by the extrinsic shielding effects. Those can include inelastic zones surrounding the crack wake (voids, micro-cracks, etc.) or physical contact between the crack surfaces (wedging, bridging, and sliding). Respectively, cycles of loading and unloading can cause fusion of micro-cracks or reduction of the friction between the crack surfaces. For refractory cement castables it was shown that the first phase of the fatigue degradation is due to diffused damage in the specimen.[14] In the following phases, the localisation of damage leads to some unloading, on the one hand, and to formation of the macro-crack, on the another.

Ceramics toughened by shielding mechanisms are more prone to premature fatigue failure than untoughened brittle one, while amorphous glass is essentially immune to the cyclic fatigue.[13] For concrete, contradicting trends have been reported.[15] Some investigations show that stronger and

---

[*] Corresponding author. Tel.: +31 0251 496780; fax: +31 0251 470489.
E-mail address: Kirill.Andreev@tatasteel.com (K. Andreev).

0955-2219/$ – see front matter © 2013 Elsevier Ltd. All rights reserved.
http://dx.doi.org/10.1016/j.jeurceramsoc.2013.08.011

Table 1
Chemical composition and basic physical properties.

	MgO-C	MpC
Density, g/cm^3	2.93	1.71
Open porosity, %	9	17.5
Avrg. pore size, μm	10	<0.5
MgO, %	98	–
CaO, %	1	–
Fe2O3, %	0.5	0.1
SiC, %	0.5	6
Total carbon, %	14	94

stiffer concrete has lower fatigue life.[16] For civil engineering concrete, the majority of investigations reporting fatigue tests deal with flexural set-ups.[15] Compressive fatigue is also investigated.[15,17,18] Compressive tests with stress cycles of 10–70% of the strength register the fatigue failure after some 10 000–100 000 cycles. For the test results linear correlation is demonstrated for the logarithms of secondary creep rate during cyclic loading and cycles to failure.[17]

Our paper presents the work which has been undertaken to estimate the sensitivity of refractory materials with carbonaceous binders to compressive fatigue failure. Magnesia-carbon and micro porous carbon brick qualities used in the linings of steel producing furnaces have been tested. Due to their chemical composition, the bricks of both classes are most often installed in the furnace without mortar. Absence of compressible mortar leads to increased stresses and potentially makes the linings of these materials more susceptible to compressive fatigue failure. Most of the cyclic fatigue results were obtained at room temperature. Several indicative measurements were also done at higher temperatures. The results of cyclic tests were compared with monotonic stress–strain and creep curves. The mechanical investigation was supported by the microstructural analysis.

2. Materials and analysis methods

2.1. Materials

Commercially available grades were used in the investigation (Table 1). Bricks of both grades were shaped by pressing. Bricks of micro porous carbon (MpC) are produced from anthracite and graphite grains. The binding agent is coal tar pitch. To reduce the size of pores special additives (e.g. metallic silicon) were used. The brick is heat treated at temperatures of 1200–1500 °C. During the heat treatment the additives undergo transitions leading to formation of micro-porous microstructure.[1,19,20]

The magnesia-carbon bricks (MgO-C) are made with magnesia and graphite grains. Metallic powder is added for oxidation resistance.[1,20,21] The binding agent is liquid phenolic resin. The bricks are baked at 600 °C.

2.2. Experimental techniques and procedures

The materials were tested as they are installed in the furnace. No additional thermal pre-treatment was done.

The compressive tests were performed in the universal test frame Zwick/Roell Z250. Stress–strain measurements with monotonic and cyclic loading, creep measurements and cyclic fatigue tests of constant amplitude were performed. Preparing for the fatigue tests, the amount of cycles a brick lining can experience during the service was estimated. For converters where MgO-C bricks are employed, the average campaign life features some 5000 production cycles. Including the possibilities of some process variations during one production cycle, the maximum amount of compressive cycles the bricks will face equals 10 000–15 000. However, the progressive wear of bricks in most parts of the lining will reduce the amount of cycles experienced by the given location in the brick. For example, in a lifetime of the converter the wear can reduce the brick thickness by some 800 mm. If we assume that thermo-mechanical failure is restricted mainly to the areas close to the hot face, the volume of the brick 100 mm from the hot face will experience some 1250–1875 cycles, before it is reduced by wear. A more specific estimation is complicated by the fact that the wear intensity can vary in different parts of the lining and during different phases of the campaign. For the blast furnace, where MpC is used, the amount of cycles was estimated from the production regime of some 12 tappings per day and the expected life of 15 years. For the blast furnace the amount of cycles was estimated as 65 000. With possible wear of some 1000 mm, the volume of 100 mm from the hot face will experience some 6500 cycles.

Cylindrical samples with the height of 50 mm were used for the tests. The ratio between the height and diameter was 1.7. Same loading conditions were used for the room and high temperature tests. The cyclic tests were performed with the largest possible amplitude. Constant displacement rate of 0.02 mm/s was used. The cycle duration depended on the amplitude and was approximately 40–70 s. The sampling frequency was 15 times per cycle. The lowest stress during cyclic unloading corresponded to 5.5% of the average failure stress. Regarding the effects of creep and the sensitivity of the results to the loading rates the same displacement rate of 0.02 mm/s was used for monotonic stress–strain measurements and in the creep tests during the loading to reach the defined stress. In monotonic measurements the material stiffness was assessed by the stress–strain curve between 30 and 70% of the compressive strength.

In all tests the sample displacement during the test was measured by the machine cross head travel, corrected for the machine's own displacements. The measurement results were validated by the finger extensometer measurements. The resolutions of the former and latter systems are ±0.5% of the displacement and 0.1 μm, respectively. The measurements were conducted in the direction of the bricks pressing. For high temperature tests the heat-up rate was 5 °C/min. The conditioning at the test temperature was 1 h. The furnace condition accuracy was ±0.5% of the current temperature. To prevent high temperature oxidation of the samples, they were immersed during the high temperature tests in a bed of coke particles. The samples of micro porous carbon were tested at their limit service temperature of 1200 °C.[1] Magnesia-carbon samples were tested at 1400 °C what presents the limit of the equipment capability for this sort of measurements.