Available online at www.sciencedirect.com

ScienceDirect

Journal of the European Ceramic Society 28 (2008) 2859–2865

www.elsevier.com/locate/jeurceramsoc

High temperature mechanical characterisation of an alumina refractory concrete for Blast Furnace main trough
Part I. General context

Nicolas Prompt[a], Evariste Ouedraogo[b,*]

[a] TRB, Rue de la Neuville, 62152 Nesles, France
[b] Laboratoire 3S-R, Domaine Universitaire, BP 53X, 38041 Grenoble Cedex 9, France

Received 4 June 2005; received in revised form 11 April 2008; accepted 25 April 2008
Available online 30 June 2008

Abstract

Blast Furnace main trough is an industrial structure submitted to severe high temperature cyclic loading applied on its inner lining made of refractory concrete. The attempt to increase the lifetime of such a structure by numerical simulation requires a proper experimental characterisation of all materials involved and particularly of the refractory concrete. The present paper exposes an analysis of the conditions required for an experimental setup in accountancy with the material working conditions. Then, the development of a performing high temperature mechanical testing device aimed at characterising the castable behaviour in its service conditions is introduced. In particular an original extensometer allowing high temperature direct measurement of the specimen height variation has been developed. Lastly, results of an uniaxiale compression test carried out at intermediate temperature are presented and discussed.

Keywords: Mechanical properties; Strength; Al_2O_3; Refractory concrete; Lifetime

1. Introduction

The evolution of metallurgical industry in less and less production sites generates great needs in production rate and productivity. At the same time, processing temperatures are rising, see for example the steel temperature in ladles or the average pig iron temperature and tapping time in Blast Furnace (BF),[1–3] whereas a continuous decrease of specific consumption is reported.[4] This emphasises the R&D efforts of both refractory suppliers and consumers which result in longer life duration of refractory materials involved. The Blast Furnace trough as an industrial structure has a strategic role in the cast iron production which requires its maximum liability.

A good understanding of the scope of the present study requires a flashback on the R&D needs related to refractory castable for the iron and steel industry. To meet the metallurgical industry's requirements, the first refractory developments concerned the installation (flowability, setting time) and low temperature physico-mechanical properties (bulk density, apparent porosity CCS, MOR) of fired products.

The high temperature behaviour of refractory products has been studied as far as static or dynamic corrosion, or thermal shock resistance are concerned. The mechanical testing concerned most of the time the "after firing" properties which reveals the non-reversible changes occurred in the materials after an exposure to high temperature. These tests were supposed to be close to the refractory behaviour in service.

Nevertheless, despite the wide area scanned, those tests did not completely reveal the actual behaviour, as far as they take place after the material firing. From these observations, and considering the development of the numerical methods for engineers, the industry started to consider refractory products high temperature mechanical properties and their influence on the structure behaviour in the 1980s. First attempts have been made towards the industrial structures such as: Blast Furnace stacking,[5] coal gasification vessel,[6] BOF,[7] steel ladles.[8]

A masterpiece of the numerical simulation of these industrial installations should contribute to decrease the refractory specific

---

* Corresponding author. Tel.: +33 476 82 52 97; fax: +33 476 82 70 43.
E-mail address: Evariste.Ouedraogo@hmg.inpg.fr (E. Ouedraogo).

0955-2219/$ – see front matter © 2008 Elsevier Ltd. All rights reserved.
doi:10.1016/j.jeurceramsoc.2008.04.031

consumption ruled by several factors.[9] Some of these factors can be controlled by refractory suppliers at the development stage. BF cast house observations reveal that thermomechanical loadings may lead to structure disorders, such as structures permanent deformations and macro-cracks leading to perforations.

Nevertheless, to the authors knowledge, the thermomechanical behaviour of a BF main trough lining had never been assessed despite the scientific interest of this research topic. Indeed, refractory materials stress–strain curves have hardly been established and published, and most of the characterisation undertaken concerned shaped materials such as high alumina bricks,[10] fireclay or MgO–C bricks.[11] Among the approaches followed, the use of specific tests or procedures should also be underlined, such as creep test,[12] Weibull analysis based on Modulus of Rupture measurement[13] or Young's Modulus measurements by ultrasonic techniques.[14] Another common approach to assess the thermomechanical behaviour of the refractory materials in service is to characterise their resistance to crack propagation using the well known thermal shock resistance parameters defined by Hasselman.[15] Other authors developed a hydraulic testing machine and studied fireclays and carbon bricks refractories at high temperature.[16] They swept a large range temperature behaviour but focussed on the viscoelastic behaviour of the material. However, it appears that each study gives a partial information on the refractory material or the refractory structure behaviour, but none of them offers a global view of both aspects. The target of these series of papers is to present a global approach in which the refractory concrete high temperature behaviour is first investigated and then related to the behaviour of the whole structure.

For this purpose a mechanical testing experimental setup has been designed and typical results of tests carried out on the material at elevated temperature are presented in the present paper. In the other following paper, the materials high temperature mechanical behaviour is investigated and the influence of several testing parameters is discussed. The specification sheet of the relevant model describing the castable is also written down. In a third coming paper, results derived from a BF main trough Finite Element Method (FEM) modelling will be presented. A parametric study will illustrate the benefits induced by this global approach.

2. Description of the BF main trough

The final object of the study being the numerical simulation of a Blast Furnace main trough, it is necessary to sufficiently describe the operation of such an industrial non-common structure. Blast Furnace main trough is a duct through which the pig iron and slag produced by the BF are conveyed respectively to the torpedo car and the slag pits. The separation between slag and pig iron is achieved by settling the overflowing slag being stopped by a skimmer. A typical main trough is 10–20 m long, 2 m deep and 3 m wide. It is composed of several refractory layers embedded in a metallic casing or in structural concrete in few situations. Each layer has its own function, and therefore its properties are adapted to it. From the internal side (fluid-refractory interface) to the external side, one can find (Fig. 1), firstly the working layer which endures the most intense thermal loading, thus is frequently relined. Secondly a safety layer or permanent layer, which supports less intense loads, but is supposed to last 10–30 times longer than the working layer, and to compensate any working lining perforation. Then comes an insulating layer which protects the structure from excessive heat. Lastly the structure, either metallic casing or concrete.

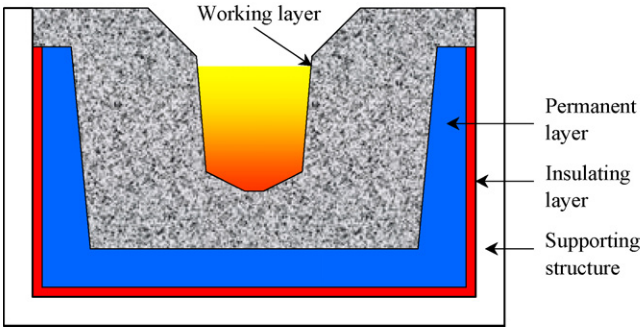

Fig. 1. Typical section of a Blast Furnace (BF) main trough.

Most of Western Europe BF production rate ranges between 2000 and 12,000 pig iron T/day, the slag ratio being around 300 kg/T of pig iron. The pig iron temperature fluctuates around 1500 °C, slag temperature being 50 °C higher. A typical BF main trough life cycle could be described as follows: complete installation, operation and demolition. The main trough demolition occurs several years after the installation. This typical cycle is represented in Fig. 2, where the castable hot side temperature is plotted against time. The operative phase of the main trough is a repetition of operative cycles composed of: a drying and heating phase, the exploitation by itself: alternative fluid tapping (BF is opened) and tap to tap time (BF is plugged), the draining out, and then the working lining repair. The usual castable hot side temperature and liquid level evolution during one operative cycle are presented in Fig. 3.

2.1. Main trough working lining castable

Since the year 1970, Western Europe BF main trough working layers are lined with ULCC [Ultra Low Cement Castable]. This class of products has supplanted the formerly used ramming mix in most of Western Europe BF cast houses. It usually con-

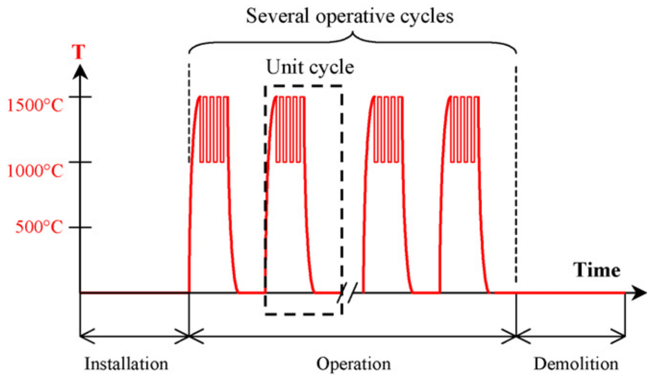

Fig. 2. A typical BF main trough lifetime cycle.

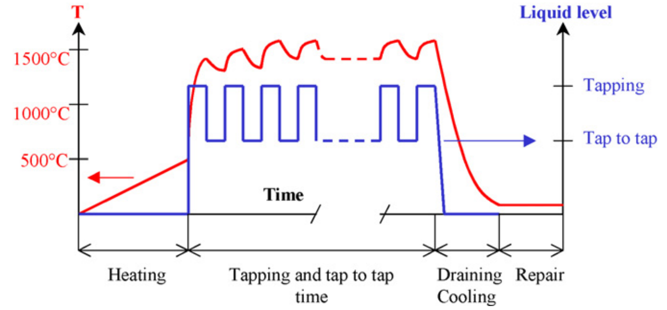

Fig. 3. A schematic typical unit operation cycle of a Blast Furnace main trough.

tains high alumina cement (so that the CaO content is between 0.2% and 1%), Al_2O_3 granulates and fine particles, 10–30% SiC, microsilica and carbon provided in various structures (graphite or carbon black) and additives. Particle size ranges from less than 1 μm to 15 mm according to the targeted application. Microsilica addition helps to control the rheology during installation. SiC enhances the castable resistance to BF slag corrosion, but has a limited resistance to iron, thus its amount is optimised with respect to these constraints.[17] Carbon containing raw materials addition aims at imparting non-wettability by metallurgical fluids.

First products developments have focused on installation techniques (mixing, vibration) and installation properties (flowability, hardening).[18] The second step of the castable development concerned physics–chemical properties, as slag and iron corrosion resistance or carbon oxidation slows down.[19] Theoretical analysis[20] and performances survey[21] have also established that the refractory wear rate depends on the flow condition in the BF main trough. Considering that the mechanical behaviour of the working lining castable, and moreover, of the main trough structure still remains a concern, the thermomechanical behaviour of a typical BF main trough castable has been studied. The castable selected is currently used in several BF cast houses. The main features of this castable are listed in the Table 1.

2.2. Main trough working lining castable operating conditions

Analysing the actual conditions of use of the castable, one can outline the main features of its operative conditions as follows. First of all the applied temperature must range from 20 to 1500 °C.[22] Then it should allow an extensive strain rate range induced by the time variability of loads and the damping due to heat diffusion resistance. On site material bears different environments, in particular oxidising (upper surface) and reducing (surface below the air–liquid interface, and inside the lining) atmospheres. If possible, the investigation of refractory–metal/slag interactions should be possible. And lastly, the device should allow physico-chemical transformations over the whole temperature range. The operative conditions listed above have constituted the starting point of the high temperature mechanical testing device design.

3. Characteristics of the high temperature testing device

As the aim pursued is to study the actual behaviour of the castable in service, it was purposely chosen to seek for the consistency of testing parameters with working conditions. It was also decided to neglect the liquid/refractory interaction influence on the whole structure behaviour, conversely to an other work[23] which focuses on the internal pressure endured by the interface zone in a steel ladle refractory due to slag infiltration in porosity. The purpose is to investigate the macroscopic behaviour of the whole structure, and with that respect, to consider that the local modification of the castable properties does not affect the global structure deformation.

The specificity of the material structure was also considered while defining the main features of the testing device. Finally, the apparatus is constituted of four parts. The load application, the regulation and data saving are performed with a 400 kN electromechanical compressive press ZWICK Z400 E equipped with ZIMT testing software. A PYROX electrical heating furnace with a maximum capacity of 1600 °C is installed. A vertical displacement degree of freedom has been managed in order to position the specimen relatively to the furnace with the aim to ensure temperature homogeneity in the specimen.[24] Temperature regulation is realised by a precise PID controller. Two thermocouples of K type measure the upper and lower face temperatures of the sample, the discrepancy between the two measurements is about 5 °C in general and never exceeds 12 °C. The experiments take place in oxidising or reducing

Table 1
Chemical and physical description of the studied refractory concrete

Chemical analysis (after firing at 750 °C)			
Al_2O_3 (%)	SiC (%)	CaO (%)	Fe_2O_3 (%)
82	12	0.5	0.1
Physical properties after drying 24 h at 110 °C			
Bulk density (kg/m^3)	Open porosity (%)	CCS (MPa)	MOR (MPa)
3250	9	50	8
Other properties			
Main aggregate	Maximum grain size (mm)	Average expansion coefficient (°C^{-1})	Average thermal conductivity (W/m°C)
Brown fused alumina	8	7.5×10^{-6}	4.5

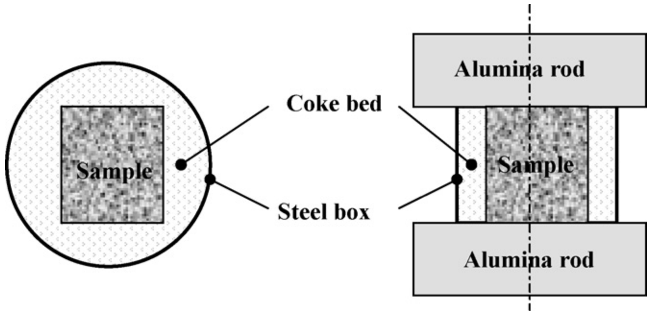

Fig. 4. Schematic representation of reducing atmosphere device for compression tests.

atmospheres. Oxidising atmosphere corresponds to experiments performed in air, while a specific device was developed to perform test under reducing atmosphere. Indeed, stainless steel boxes containing a coke bed around the specimen were built for this purpose (see in Figs. 4 and 5). Lastly, sample strain measurement is obtained by means of four alumina rods which transmit the total sample height variation to two capacitive displacement transducers set outside the electric furnace, supported by an aluminium ring. This class of sensors has been chosen for its low temperature variation sensibility (12 ppm/°C).

4. Operational conditions

4.1. Sample preparation and testing procedure

Refractory concrete samples are formed by casting in stainless steel moulds. After mixing water, cement, aggregates and additives, the concrete obtained is poured into moulds and then vibrated up and down according to an industrial procedure. The determined vibration direction probably creates particles segregation in the concrete that can induce anisotropy in the sample. However the material should have the same behaviour in the other two perpendicular directions for cubic samples. This potential anisotropy problem will not be studied in the present paper.

Fig. 5. View of the reducing atmosphere testing device after compression at low temperature.

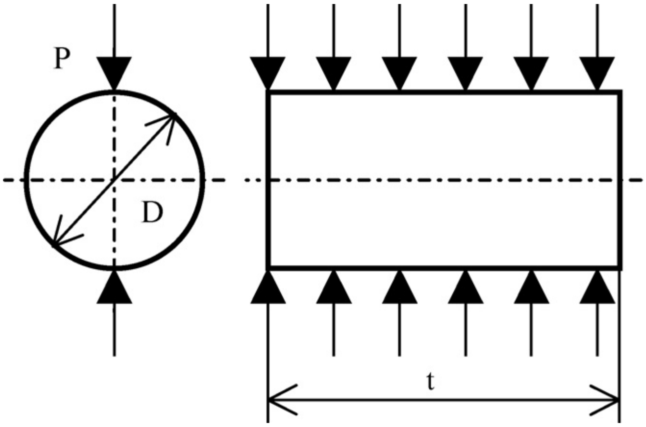

Fig. 6. Geometric and loading main parameters involved in indirect tensile test performed on the testing device.

Cubic geometry specimen has been preferred to cylindrical because grinding operations were excluded. Usually uniaxiale compression tests are performed on cylindrical specimen with a height to diameter ratio of 1.5–2, particularly on ordinary concrete. But when casting a cylinder sample in a steel mould one obtains smooth cylindrical surface and just one smooth flat surface (lower flat surface). The upper surface of the specimen, free during the vibration phase, remains very coarse because of free surface deformation caused by exothermic reaction during the castable setting. In these conditions, the upper face quality is not acceptable in terms of tolerance. Grinding operations on the refractory concrete revealed that the included hard corundum particles instead of being cut are ejected (see Table 1); the resulting surface is then coarser than the initial one. Moreover, these machining operations can initiate damage in the material, which should be avoided. A specific mould has been then designed and reserved to this purpose. Specimens were selected cautiously by examining most of all the parallelism tolerance between the loading surfaces: only 6/100 mm of maximum parallelism tolerance samples were accepted. Cylindrical samples, 40 mm in diameter and 80 mm in length, have been prepared for indirect tensile tests (see in Fig. 6).

Different initial state thermal histories of the material have been considered. Firstly crude state where the material had no preliminary heat treatment. Secondly, dried state where samples have been dried precisely during 24 h at 110 °C. Lastly, fired state in which the material has been fired at different temperatures and various atmospheres, for instance at 800 °C for 24 h and 800 °C in oxidising atmosphere or at 1100 or 1200 °C during 5 h in reducing atmosphere. The last parameter that can be tested is the influence of the sample-pushing rods interface on the material response through the use of different conditions: direct contact, interposition of a graphite sheet or interposition of a high temperature resisting mechanical grease.

4.2. Testing procedure, thermal conditions

A typical thermomechanical testing is carried out through four consecutive stages: 1: heating up to the testing temperature, 2: holding the temperature until the material and experimental

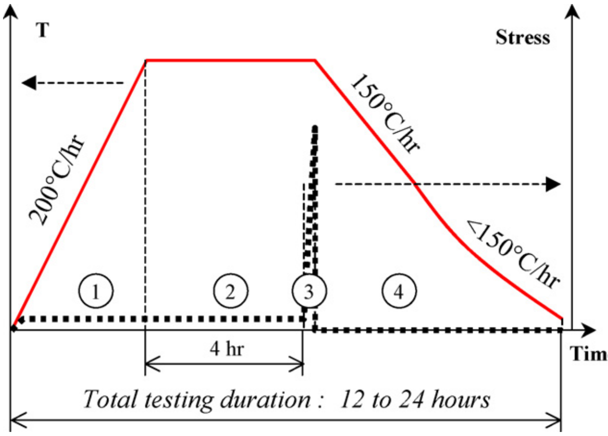

Fig. 7. Typical thermal and mechanical loading conditions during a compression test.

device stabilise from thermal point of view, 3: stress application at the test temperature, regulated strain rate or constant stress for creep, 4: cooling. These stages are illustrated in Fig. 7.

For the sake of data base homogeneity, it is compulsory to observe a homogeneous heating procedure all along the experiment programme. The selection of thermal parameters is discussed below. A 200 °C/h heating rate is applied. This rate has been determined as the best compromise between the thermal shock resistance of the pushing bars, the electric furnace power and the time available for the testing. The holding time is determined in order to stabilize temperature evolution in the sample. Indeed, the parameter depends on the testing temperature which determines the physics–chemical reactions that take place in the material. In practice, a constant holding time of four hours and half is considered. Lastly, the cooling stage has no influence on the mechanical testing as it takes place after the experiment. Nevertheless, when defining cooling rate, one must remember that ceramic rods are very sensitive to decreasing thermal shocks; therefore we have chosen to cool down at 150 °C/h.

4.3. Testing procedure, mechanical conditions

A constant compressive load is applied during the heating and holding time procedure so as to ensure a complete contact between samples and rods. The load intensity should not be too intense in order to minimise creep strain at high temperature, and generally to avoid the activation of microstructure transformation. The Table 2 indicates the compression load applied on the sample during heating and holding time according to test temperature, before mechanical testing. Fig. 8 displays the evolution of temperatures, global displacement of the machine and the local displacement measured by the LVDT sensors. The regulation in force induces an upward motion of the machine loading device in order to absorb the occurring thermal dilatation. One can see in this figure that a certain time is necessary for the sensors to thermally stabilise.

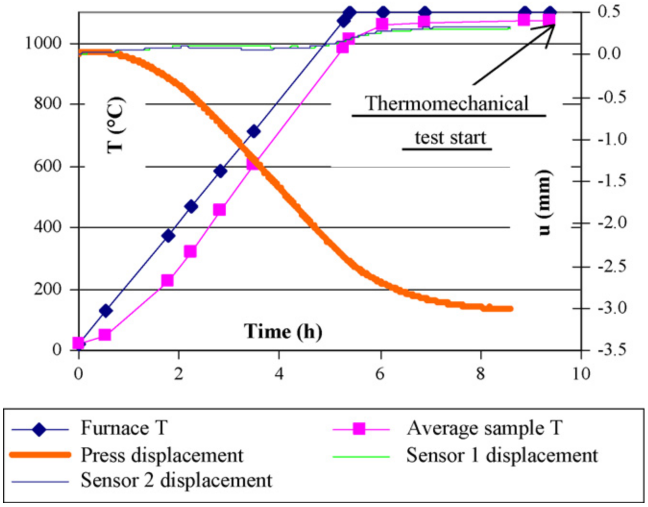

Fig. 8. Typical evolution of local temperatures, local and global displacements illustrated during a test.

5. Typical monotonic compression loading behaviour

A huge tests campaign has been undertaken mostly at a constant standard displacement rate of 0.1 mm/min corresponding to a theoretical strain rate of $5 \times 10^{-5}\,\mathrm{s}^{-1}$. In the present paper a short illustration of the new design mechanical facility will be presented. Fig. 9 displays a curve stress–strain for 40 mm cubic edge specimen without lubrication in oxidising environment at 800 °C testing temperature. The general aspect reminds of a bell-shaped curve characterised by the following two parameters: the maximum stress or peak stress and the corresponding strain that will be called peak strain. Otherwise one can see an uncertain

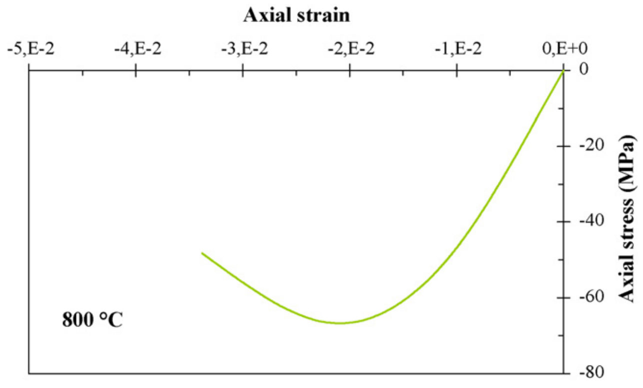

Fig. 9. Example of a compression curve for a test performed at 800 °C, in oxidised atmosphere without lubrication.

Table 2
Preload applied during heating and holding time

Temperature (°C)	From 400 to 900 °C	From 900 to 1200 °C	From 1300 to 1500 °C
Load (MPa)	1.8	1	0.6

Fig. 10. Standard macro-crack network exhibited by a cubic specimen after a compressive test at 20 °C.

Table 3
Compression characteristics at ambient temperature of the studied castable

Characteristic	E (GPa)	$\sigma_{\max}$ (MPa)	$\varepsilon_{\text{peak}}$
Value	9	31	7.1×10^{-3}
S.D.	37%	2.8%	23%

curve beginning followed by a linear to quasi-linear evolution up to 30–70% of the peak stress; then the curve slope decreases non-linearly until the peak stress and then the stress decreases significantly. Peak strain values are about 5×10^{-3} to 3×10^{-2}. During the tests, cracks appear all along the non-linear part of the stress–strain curve, but they are macroscopically visible only after the peak of stress. Cracks are parallel to the compressive loading direction because of transverse extension failure (Fig. 10). The observation of fracture occurring is not possible at high temperature because furnace walls are not equipped with a visualisation window. This could be possible at room temperature but the presence of carbon in the sample blackens it and makes fracture observation quite difficult, in particular for optical micrographs observations. So, investigation of the evolution of the material microstructure was not possible. In revenge at the end of the test after a large deformation, the specimen cracking has been examined. At this stage cracks are clearly visible and somewhat conform to the result published about ordinary concrete[5] for low temperature behaviour of refractory concrete. Table 3 gives the mean values of some of the thermomechanical characteristics of the refractory concrete[25] at room temperature. Despite its ultra low cement content, the refractory castable strength is comparable at ambient temperature to the average ordinary concrete one.

6. Conclusion

In this paper, the necessity of improving the knowledge of the BF main trough refractory castable thermomechanical behaviour has been underlined. The essential features of a mechanical testing device aiming at filling this gap have been defined thanks to an analysis of the actual use conditions of this castable. This analysis allowed us to design and construct a performing testing device dedicated to the study of the thermomechanical behaviour of refractory castable. The specificity of this study is the development of a special extensometer device allowing the direct measurement of the specimen height variation at high temperature. The first results obtained at elevated temperature on a crude material show that the behaviour is qualitatively very close to one of the traditional ordinary concrete. The features of the high temperature behaviour and the influence of several testing parameters are presented in a second article. The data collection will enable the selection of a suitable mechanical model and the identification of its parameters in order to simulate the castable behaviour while incorporated in an industrial structure.

Acknowledgements

Authors acknowledge Mr Laurent Debove for his contribution to the design of the loading device of the experimental setup and TRB Company for financial support.

References

1. Buhr, A., Refractories for steel secondary metallurgy. *CN-Refractories*, 1999, **6**(3), 19–30.
2. Ruther, P. *et al.*, Optimisation of the cast house performance with new types of tap hole clays. In *45th International Colloquium on Refractories*, 2002, pp. 90–93.
3. Lachmund, H., Demands on refractory material for secondary metallurgy. *Refractory Technology: Steel Ladle Lining*, Ijmuiden, June 20–22, 2002.
4. Eschner, A., Management of refractories by the European refractory industry. In *45th International Colloquium on Refractories*, 2002, pp. 16–19.
5. Poirier, J., Conception des creusets de Hauts Fourneaux Choix des matériaux réfractaires et construction, *CESSID: cycle "Hauts Fourneaux"*, November 8–10, 1994.
6. Buyukozturk, O. and Tseng, T.-M., Thermomechanical behaviour of refractory concrete linings. *Journal of the American Ceramic Society*, 1982, **65**(6), 301–307.
7. Chen, E.-S., Lautensleger, R. W. and Brezny, B., Thermomechanical analysis of a 225-ton BOF vessel. *Iron and Steel Engineer*, 1993, 43–51.
8. Belt, D. A., First heat-up of ladle linings. In *Proceedings of the Unified International Technical Conference on Refractories*, 1999, pp. 292–294.
9. Ruther, P. *et al.*, Improvement of the refractory technology for optimised costs in the Blast Furnace cast house operation. In *UNITECR 2003 Congress*, 2003, pp. 400–403.
10. Padget, G. C., Cox, J. A. and Clements, J. F., Stress/strain behaviour of refractory materials at high temperature. *Transactions of the British Ceramic Research Association*, 1969, 63–72.
11. Schacht, C. A., Thermomechanical behaviour of refractories. *Key Engineering Materials*, 1993, **88**, 193–218.
12. Bray, D. J., Smyth, J. R. and Mc Gee, T., Creep of 90+% Al_2O_3 refractory concrete. *American Ceramic Society Bulletin*, 1980, **59**(7), 706–710.
13. Davidge, R. W., Mechanical behaviour of ceramics. *Cambridge Solid State Science Series, Cambridge University Press*, 1979.
14. Nonnet, E., Lequeux, N. and Boch, P., Elastic properties of high alumina cement castables from room temperature to 1600 °C. *Journal of the European Ceramic Society*, 1999(19), 1575–1583.
15. Hasselmann, D. P. H., Unified Theory of thermal shock fracture initiation and crack propagation in brittle ceramics. *Journal of the American Ceramic Society*, 1969, **52**(11), 601–604.
16. Miyamoto, M., Onoye, T. and Narita, K., Deformation and failure behaviour of refractories for the blast furnace at elevated temperature. *Transactions ISJJ*, 1981, **21**.
17. Ioshitomi, J., Harda, M., Eguchi, T. and Mukai, K., Local corrosion of iron trough refractories in the slag–metal interface. In *33th International Colloquium on Refractories*, 1988, pp. 85–89.

18. Chastant, M., Matériaux coulables pour le revêtement réfractaire des rigoles de coulée du haut fourneau. *Revue de Métallurgie-CIT*, Octobre 1986, 719–729.
19. Joly, T., *Modélisation de l'oxydabilité d'un matériau réfractaire carboné*. PhD Thesis. UTC, 1993.
20. He, Q. *et al.*, Fluid flow and refractory wear in a BF trough—a water model using LDV. In *Iron-making Conference*, 2002, pp. 167–176.
21. Prompt, N., *TRB Internal Report*, 2002.
22. Prompt, N., Ouedraogo, E., Joly, T. and Stutz, P., Numerical modelling of the heat conduction in the Blast Furnace trough. In *Proceedings of UNITECR 99*, 1999.
23. Blond, E., Schmitt, N., Hild, F., Blumenfeld, P. and Poirier, J., Thermomechanical stresses in slag-impregnated refractories. In *Proceedings of UNITECR 01*, 2001.
24. Ouedraogo, E. and Tran Van, D., *High Temperature Experimental Characterisation of Materials Formed by HIP, EURO PM 1997*. EPMA, Advanced Materials, Munich, Germany, 1997, pp. 535–542.
25. Prompt, N., *Etude du comportement thermomécanique d'un béton réfractaire—Application à une rigole de haut fourneau*. PhD Thesis. Joseph Fourier University, Grenoble, 2000.