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High-temperature mechanical characterisation of an alumina refractory concrete for Blast Furnace main trough PART II. Material behaviour

Evariste Ouedraogo [a,*], Nicolas Prompt [b,1]

[a] *Laboratoire 3S, Domaine Universitaire, B.P. 53, 38041 Grenoble Cedex 09, France*
[b] *TRB, Rue de la Neuville, 62125 Nesles, France*

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Abstract

This paper deals with the experimental mechanical characterisation of a high alumina carbon containing refractory concrete at temperatures ranging from room to high temperature (1500 °C). Uniaxial compression, indirect tensile, creep and cyclic loading–unloading tests have been performed. Scale factor effect, influence of interfaces lubrication, material initial heat treatment as well as oxidising or reducing atmosphere test conditions have been investigated. Some of the material mechanical properties have been found to depend not only on the level of applied temperature but also on its duration revealing then the sensible influence of chemical transformation kinetics within the material. An analysis of the global macroscopic mechanical behaviour of the material through the range of temperature is lastly exposed. The data collected are aimed at identifying damage behaviour constitutive equations for numerical simulation of structures built within this material.

Keywords: Fracture; Strength; Mechanical properties; Refractories; Lifetime

1. Introduction

The numerical modelling is an attractive approach to analyse the behaviour of industrial structures submitted to severe cyclic thermal loading in the aim of lengthening their lifetime. Blast Furnace (BF) main trough is an example of a structure enduring such a complex loading applied to the refractory castable linings. The conception of these traditional structures is based on try-and-error methods that refractory concrete furnishers would like to rationalise. In such a multi-layers structure one can distinguish outer layers playing the role of containing and isolating from inner layers in refractory materials which endure by themselves the cyclic thermal loading. Whereas a thermo-elastic behaviour can be encountered for the modelling of outer layers, inner layers constituted of refractory concrete are severely stressed, by the application of service thermal loading, beyond elastic behaviour. The first level of the present study is the investigation of the material monotonic non-linear behaviour with increasing temperature. Knowledge of the material thermal properties and its mechanical behaviour allows the constitutive equations identification and then the possibility of the structure numerical simulation. A second level of investigation is the determination of the material long term and or cyclic behaviours that can be respectively evidenced by creep and cyclic loading–unloading tests. This second level investigation is rather qualitative and helps in understanding the risks at which the structure is exposed to. In our knowledge, this experimental approach conducted on refractory concrete at high temperature is original.

Refractory concrete are multi-phase containing materials in which several chemical interactions take place during the increase of temperature. These chemical interactions are sensible to the testing atmosphere and to the duration of the application of the temperature. The consequence is that once pre-fired and cooled the material behaves differently from

* Corresponding author. Tel.: +33 476 82 5297; fax: +33 476 82 7043.
E-mail addresses: Evariste.Ouedraogo@hmg.inpg.fr (E. Ouedraogo), nicolasprompt@trbrefractories.com, nicolas.prompt@edf.fr (N. Prompt).
[1] Current address: EDF SEPTEN, 12-14 Avenue Dutrievoz, 69628 Villeurbanne Cedex, France. Tel.: +33 472 827401;
fax: +33 472 82 77 11.

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Table 1
Overview of the tests performed and presented in the present paper

Parameters		Range		
Sample				
Size		40 × 40 × 40	60 × 60 × 60	Ø 40 × *L* 80
Thermal treatment	Without	Y	Y	Y
	Dried 24 h, 110 °C	Y	Y	Y
	Oxidised 4 h, 800 °C	Y	Y	N
	Oxidised 24 h, 800 °C	Y	N	N
Interface	Without	Y	Y	Y
	Grease	Y	Y	Y
	Carbon paper	Y	Y	Y
Thermal conditions				
Heating rate	200 °C/h			
Testing *T*	20–1500 °C			
Testing atmosphere	Air (oxidising)	Y	Y	Y
	Reducing	Y	Y	N
Mechanical conditions	Strain loading rate	$5 \times 10^{-5}\,s^{-1}$		
	Strain unloading rate	$2 \times 10^{-4}\,s^{-1}$		
Comment	Y: yes, N: no			

initial state. The third level of the present study is to investigate at room or elevated temperatures the influence of pre-firing treatment in different chemical conditions on the macroscopic material behaviour through mechanical tests. Lastly, the material mechanical strength measured depends on some experimental parameters such as lubrication or not of the specimen/crushing plate interface. These investigations constitute the last level at which the present study have focused interest.

The present study is aimed at determining experimentally the mechanical behaviour of a high alumina refractory carbon containing castable in order to identify convenient constitutive equations for numerical simulation purposes. To do so, uniaxial compression, indirect tensile, creep and loading–unloading test will be performed at different temperatures. In order to fit close to the real material service conditions, the material will been tested in oxidising or reducing atmosphere for which a specific technique has been developed.[1] Other parameters such as scale factor or influence of lubrication will be also investigated.

2. Experiment

2.1. The material

The material to study is a high carbon containing alumina castable used as a recovering layer of a Blast Furnace main through. It contains 82% Al_2O_3, 12% SiC, 5% SiO_2, 2% TiO_2, 0.5% CaO and 0.1% Fe_2O_3. Aggregates, cement and water are mixed together and poured into moulds of different shapes. The moulds are then vibrated with respect to an industrial strict procedure in order to obtain a quality of material similar to the on site one. Once the different shape specimens are de-moulded some of them are submitted to different types of heat treatments: dried at 110 °C during 24 h, fired at 800 °C during 24 h in oxidising atmosphere, fired at 1100 °C or 1200 °C during 5 h in reducing atmosphere (Tables 1 and 2).

Cubic geometry specimen has been retained for uniaxial compression tests. Two different dimensions, 40 and 60 mm, were considered in order to seek for scale effect. Cylindrical specimen, 40 mm in diameter and 80 in length, is aimed at performing

Table 2
Influence of lubrication conditions on the compression test results, on 40 and 60 mm cubic samples

Thermal treatment conditions	Lubrication	Peak stress (MPa)		Strain at peak stress	
		40 mm	60 mm	40 mm	60 mm
Without	Without	39.8	39.9	1.02×10^{-2}	1.02×10^{-2}
	Grease	33.3	39.5	1.01×10^{-2}	9.43×10^{-3}
	Graphite	27.7	27.2	–	1.02×10^{-2}
Dried 24 h, 110 °C	Without	47.8	43.2	1.08×10^{-2}	9×10^{-3}
	Graphite	34.5	29.4	1.05×10^{-2}	9.76×10^{-3}
Fired 5 h, 1100 °C, reducing atmosphere	Without	77.3	64.3	9.65×10^{-3}	1.15×10^{-2}
	Graphite	62.7	47	1.15×10^{-2}	1.1×10^{-2}

splitting tests also called Brazilian tests. The geometrical and dimensional tolerances are insured by particular strict cautions taken during the moulds fabrication operations. This material contains some corundum particles that are ejected during grinding attempts on the specimen. The consequence is that grinded surfaces are rougher than initial ones. That is why specimens have been tested without any finishing operations after moulding.

2.2. The experimental facility

All the tests presented in the present study have been carried out on an specific high-temperature apparatus designed and developed especially and described in a former paper.[1] This facility is composed of a 400 kN electromechanical testing machine equipped with a 1600 °C furnace capacity. The specificity of this apparatus resides in the fact that a special device located outside the furnace enables the differential displacement measurement of the high and low faces of the specimen for a temperature ranging from room to 1500 °C. The LVDT sensors then measure the specimen height variation leading to a direct strain measurement. The same configuration of the device is used for 40 mm specimen uniaxial compression tests as for the 40 mm diameter specimen Brazilian tests. Configuration needs to be changed in the case of 60 mm specimens' uniaxial tests. A test takes place in four-phase operations: heating at a rate of 200 °C/min up to the target temperature, then maintaining this temperature 3 h 30 min, after which the mechanical loading is applied to the specimen, and lastly the cooling phase at a rate of 150 °C/min. During the heating stage a little constant force is applied to the specimen in order, first to maintain the contact between the specimen and the platens and, most of all, to insure a force regulation that protects the material from the system rising dilatation.[2] Details on the setup operations and the test protocol are described widely in Ref. [1][1].

3. Results and discussion

3.1. Thermo-mechanical behaviour in uniaxial compression

The following results deals with initial state of the material without any heat treatment. Fig. 1 displays a typical monotonic loading response of the material in a stress–strain plane while tested at 800 °C in oxidising atmosphere. The general impression is a bell-shape evolution. In other words the curve is almost linear at the beginning, then becomes more and more non-linear until it reaches a maximum value, called stress peak, after which the stress decreases more or less rapidly. Such a curve is typical of ordinary concrete and can be similarly interpreted. The initial part of the curve more or less linear is generally thought to correspond to the shutting of initially opened cracks induced by the compressive stress. Then the increase of the compressive stress level induces the opening of news cracks or initially present in the compression direction and the resulting macroscopic response is the apparition of non-linearity, which is also supposed to express the occurrence of damage. The raise of

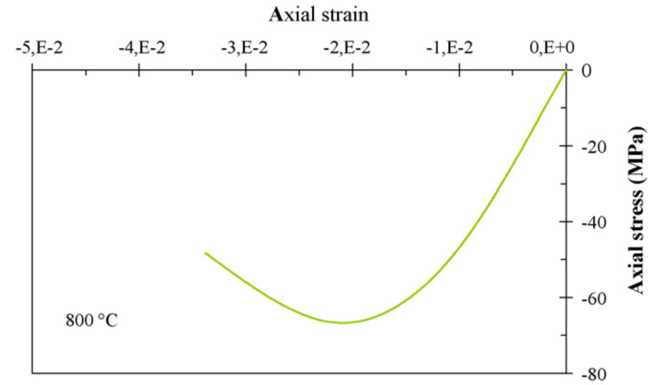

Fig. 1. Stress–strain curve on 40 mm cubic specimen from a compression test performed in oxidising atmosphere, without lubrication.

cracks in number and length in the material weakens the material whose resistance is first limited at a maximum value then decreases sensibly. This description fits the results of the present tests well. For instance, after the peak of stress the material cracking becomes very noisy (one can hear it). The resistance collapse occurs in a very short time characteristic of a quasi-brittle material. Visual observations on the specimen after the peak reveal several cracks and often some macro-cracks in the compressive direction.

The tests have been carried out at different testing conditions and temperatures. Fig. 2 shows a global view of the material uniaxial compression behaviour upon temperature. The following results have been obtained in oxidising atmosphere at the following testing temperatures: 20, 400, 600, 800, 900, 1000, 1050, 1100, 1200, 1300 and 1400 °C. The first remarkable result is the increase of material strength (peak stress) with increasing temperature with a maximum value reached at 900 °C and followed by a deep decrease for higher temperatures. One can see in Fig. 3 the evolution of the mean peak stress versus the testing temperature illustrating then clearly the specificity of the material. The fast decrease of the compression strength also points the change in the material behaviour as temperature increases. Analysis of Fig. 1 shows that the behaviour is quasi-brittle with non-linearity evolution characterised by damage occurrence.

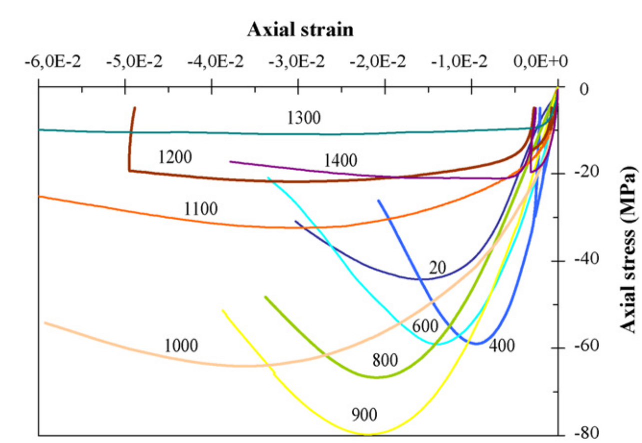

Fig. 2. Evolution of stress–strain curves of tests performed from 20 to 1400 °C in oxidising atmosphere, without lubrication.

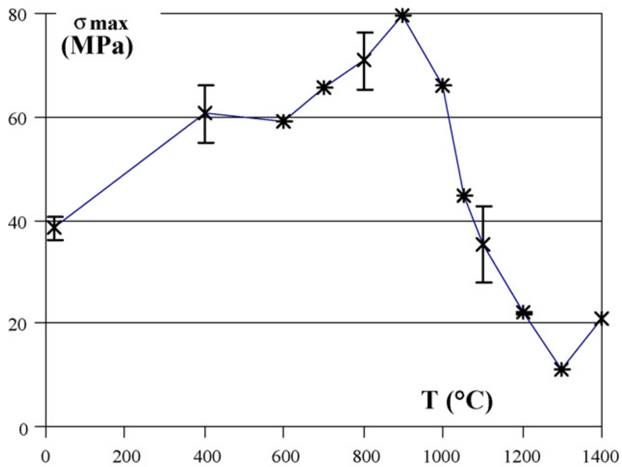

Fig. 3. Evolution of compressive strength versus temperature (oxidising atmosphere, without lubrication).

The second noticeable result is the apparent possibility of three-range temperature classification of the material behaviour. A first group ranging from 20 to 900 °C is characterised by bell-shape evolution which has been already discussed. A second group ranging from 900 to 1100 °C seems to have an intermediate behaviour. The strain at peak stress at these temperatures has sensibly increased what reveals an increase in the material ductility. At least, phenomena of irreversible strain occurring like in plasticity acts there and extend then the material apparent ductility. Lastly, a third group of curves for temperature higher than 1100 °C is characterised by stress–strain flat response evidenced by the existence of a stress threshold. The flat stress–strain response, for a constant displacement rate, is characteristic of viscosity in general and viscoplasticity in the present case. Some plastic strain is probably also present in the total measured strain. The first and the third groups are well described whereas the second one because of its intermediate character is not. Then a relevant way to assess the material behaviour is to distinguish the temperature range where the material is time-dependent ($T \geq 900$ °C) from the range where it is not ($T \leq 900$ °C). This partition of the material behaviour is more convenient in the aim of modelling and will be adopted.

The elastic modulus of the material has been determined by measuring the slope at the beginning of stress–strain curves. Such a measure is not relevant because this slope is influenced by sample/platen's initial contact effects. The value of elastic modulus determined through initial loading stress–strain curve seems to be affected by the contact problem. This difficulty can be avoided by measuring the curve slope at the beginning of the unloading phase. Fig. 4 displays the evolution of the unloading elastic modulus versus testing temperature. The experimental value is about 30 GPa and rather constant through all temperature range. This result is surprising because the variation of material compression strength evidenced previously seems not to be correlated to the Young modulus evolution. One could have waited for a sensible variation of this parameter while the material behaviour evolves from quasi-brittle to viscous. To avoid problems related to static Young modulus measurement some authors determine a dynamic elastic modulus by ultrasonic technique,[3] but in general the resulting value is overestimated.

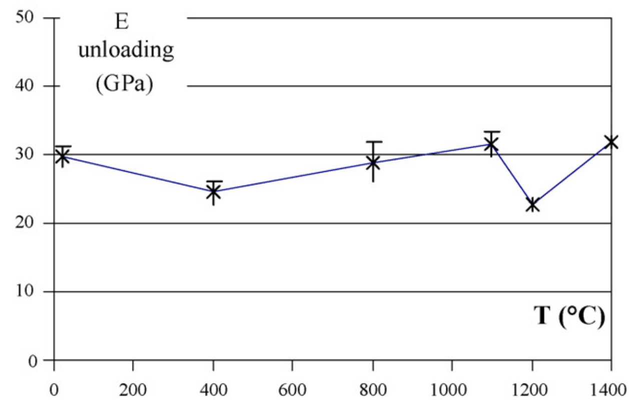

Fig. 4. Evolution of unloading mean elastic modulus versus temperature.

A paper published elsewhere[4] on two refractory concretes constituted with 90% of Al_2O_3 for the first and 70% of Al_2O_3 for the second deals with the elastic modulus determination. These two materials are comparable with the studied material as it constituted of 82% Al_2O_3, 12% SiC. They have found a value of 6.9 GPa for the former material and 5.8 GPa for the latter one. The mean measured value at the loading path of the Young's modulus is of the same order (9 GPa) as those published. Nevertheless, we consider the elastic modulus underestimated due to overestimated measured strains. This is suggested by the elastic modulus measurements realised at the beginning of an unloading phase which are three times higher (for the same temperature) than the previous one. These last values are more relevant for refractory concretes.[5]

3.2. Creep behaviour

The creep tests carried out consist in applying three successive increasing stress levels on the material at different temperatures and recording the postponed occurring deformation. Some tests not displayed here revealed no sensitivity at

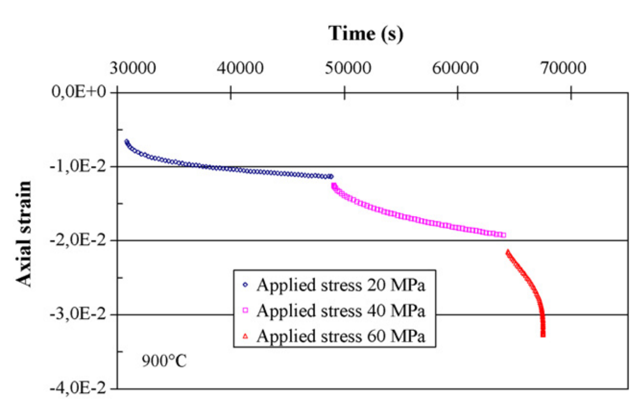

Fig. 5. Exhibition of creep tests curves performed at 900 °C in reducing atmosphere, with graphite lubrication, showing tertiary creep occurrence at 60 MPa loading.

700 °C but a clear sensitivity at and above 800 °C. One can see in Fig. 5 such a test performed at 900 °C in a reducing atmosphere and lubrication conditions. This figure represents the evolution of the axial strain upon time for a specimen submitted to the upgrading three stress levels: 20, 40 and 60 MPa. The three different curves visible on this figure correspond to the strain response of the material to these successive applied stresses. If the response is independent upon time, no creep then occurs and the strain remains constant. If not, primary creep (decreasing slope) and then secondary creep (constant slope) occur. That is the case at 800 °C for 20 and 40 MPa applied stress. The stress in each case has been applied during 4 h. At and above 800 °C, viscosity occurs in the studied material. Another great result is visible in this figure. For 60 MPa stress level, the strain evolution versus time is first linear and then the slope increases infinitely leading to a failure of the material. This is a proof that the material exhibits tertiary creep at high-level compressive stress. Other tests undertaken in the present study showed that this ability to tertiary creep of this refractory concrete is also observed at higher temperatures. This specific mechanical property evidenced by these tests is an important result with implications in the material use in industrial applications. Occurrence of this phenomenon depends probably on the applied strain rate. We assume that the higher the strain rate, the lower the probability of occurrence.

3.3. Cyclic loading–unloading behaviour

Refractory concrete in service conditions is often submitted to cyclic thermal loading inducing cyclic mechanical loading. It is then of the utmost importance to investigate the material cyclic loading response. Figs. 6 and 7 show the material responses at 800 and 900 °C, respectively. Material is first submitted at a first stress-level cycling characterised by an amplitude of 10 MPa. For stress level less than 60% of the material strength, stress cycling evolves in the accommodation of the material. This is characterised by a repetitive same response in the stress–strain curve. The average stress level is then increased to a higher value with a constant cycling amplitude. The tests performed at 800 and 900 °C show that at mean stress level equal to 80–85% of the peak stress, ratchetting phenomenon,[6] characterised by continuous increase of the inelastic deformation developed by the material at each cycle, occurs in the material. It is also characterised by the failure of the material at a stress level lower than the material strength. This phenomenon is dangerous because it inevitably evolves toward the failure of the material. This specific behaviour of the material at intermediate and at high temperatures is a great result revealed by these tests.

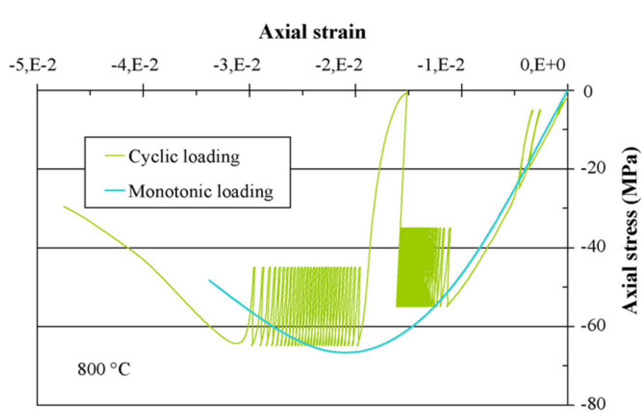

Fig. 6. The refractory concrete exhibits at 800 °C during cyclic loading by a certain stress-level ratchetting phenomenon.

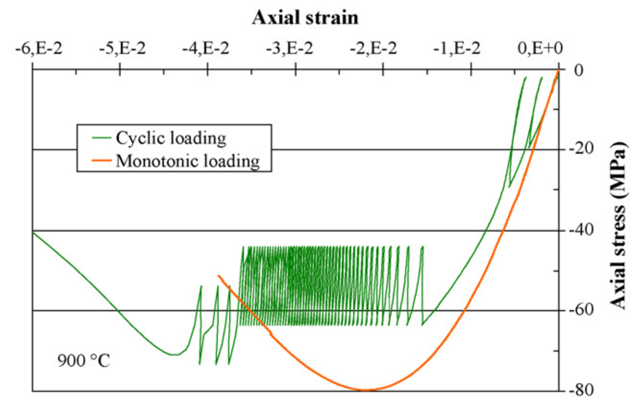

Fig. 7. The refractory concrete exhibits at 900 °C during cyclic loading by a certain stress-level ratchetting phenomenon.

3.4. Indirect tensile behaviour

Brazilian tests or splitting tests consist in crushing a bedded cylindrical specimen between two crushing plates; a schematic illustration is displayed in Fig. 8. Force is then applied in a diameter plane normal to which act tensile stresses. Theoretical studies have derived the stress and strain fields in the sample assuming linear elastic behaviour.[7,8] For instance, for a sample with a D diameter and a length t, the transverse section field of stress is rather complex and given by the following equations:

$$\sigma_{xx} = \frac{2P}{\pi D t} - \frac{2P}{\pi t}\left\{ \frac{x^2(R-y)}{[x^2+(R-y)^2]^2} + \frac{x^2(R+y)}{[x^2+(R+y)^2]^2} \right\} \quad (1)$$

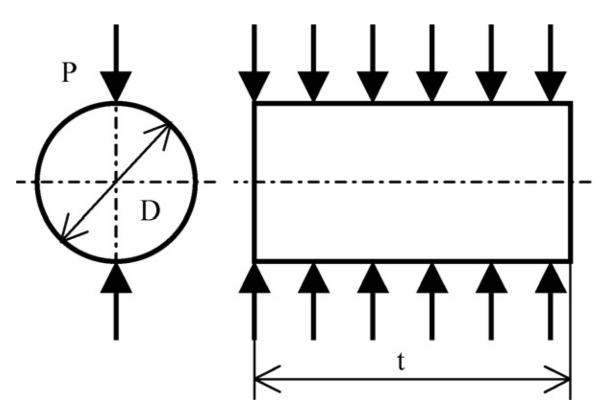

Fig. 8. Illustration of the acting applied load during splitting test.

$$\sigma_{yy} = -\frac{2P}{\pi D t} - \frac{2P}{\pi t}\left\{\frac{(R-y)^3}{[x^2+(R-y)^2]^2} + \frac{(R+y)^3}{[x^2+(R+y)^2]^2}\right\} \quad (2)$$

where P is the applied load, x and y are point coordinates with origin at the centre of the section. From these above equations, the material global mean tensile strength can be deduced:

$$\sigma_T = \frac{2P_{max}}{\pi D t} \quad (3)$$

where P_{max} is the maximum compressive force applied on the specimen during the test. This equation relates the tensile strength to the maximum load applied during the test.

Fig. 9 displays the typical force–displacement curve of a Brazilian test performed at 800 °C. The evolution is similar to compression curve with a maximum force followed by a softening phase. For instance, the maximum force is obtained for a displacement of 0.2 mm. This displacement is surely overestimated because of the inevitable influence of the long contact between the specimen and the platens. This contact plays a very important role in the validity of this test. Splitting tests have been carried at different temperatures.

Viscoplasticity is evidenced at temperatures higher than 900 °C by flat curve evolution, these tests are not representative of indirect tensile stress because the contact at the beginning of the test is plane rather than linear due to the preload applied during the heating stage; the consequence is that failure does not occur in the contact containing diametrical plane. The maximum force applied is no longer representative of the mean tensile stress occurring in the specimen. That is why such a test is valuable at the maximum temperature of 800–900 °C. Fig. 10 visualises safe specimen failure. Calculated maximum tensile stress from Eq. (3), the corresponding maximum compression strength at 20, 400 and 800 °C and their respective ratio are recorded in Table 3. The material seems to be more tensile resistant at 800 °C compared to compression resistance at 20 or 400 °C. This is probably the effect of viscosity occurrence which induces a kind of ductility but it can also be related to an irrelevant failure.

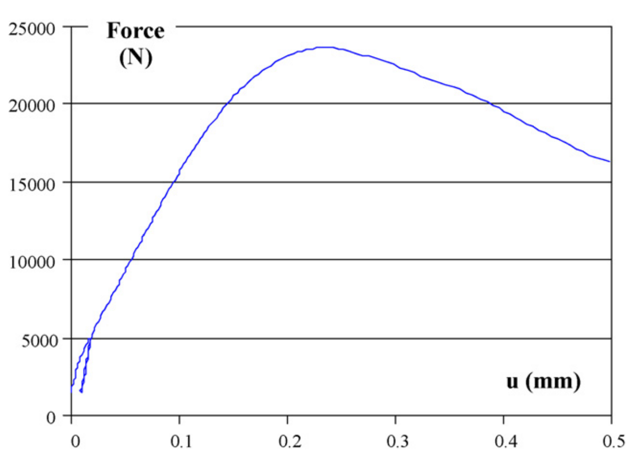

Fig. 9. Typical force–displacement curve of a splitting test at 800 °C.

Fig. 10. Cylindrical sample after a splitting test displaying diametrical plane crack, right failure.

3.5. Influence of the specimen thermal treatment conditions

These tests are aimed at investigating the behaviour of the material in some realistic service conditions. For instance, some part of the BF main through lining layer is exposed to oxidation (external face in contact with air), whereas the main inner part is protected from it. In order to fit as nearly as possible to the service conditions material must be studied in reducing and in oxidising atmospheres. On the other hand, emergency measures or repairing operations can provoke the stop of industrial process so that the heated material can then cool unfortunately. The question is to know whether it is possible to re-use the installation with the in-place material. The purpose of this study is to determine how and how many material behaviour and characteristics changes after different heating processes. Exhausted studies in this subject would be very long. In this paper, some specific experiments have been done and some of the results given hereafter. The first phenomenon met when heating refractory concrete is hydration or loss of water. It has been found that drying the material at 110 °C during 24 h strengthens it sensibly during compression or splitting tests at room temperature. For instance, in splitting test the force is 30–45% higher for the dried material than intact one. Fig. 11 displays the material mean strength measured from tests performed at room temperature on different initial state conditions when considering two sizes of specimens and showing the influence of heat treatment

Table 3
Splitting and compression strength from 20 to 800 °C

	T (°C)		
	20	400	800
σ_{max} splitting (MPa)	2.2	2.5	4.5
σ_{max} compression	35.0	49.6	64.3
Compression/splitting strength	16.2	19.9	14.4

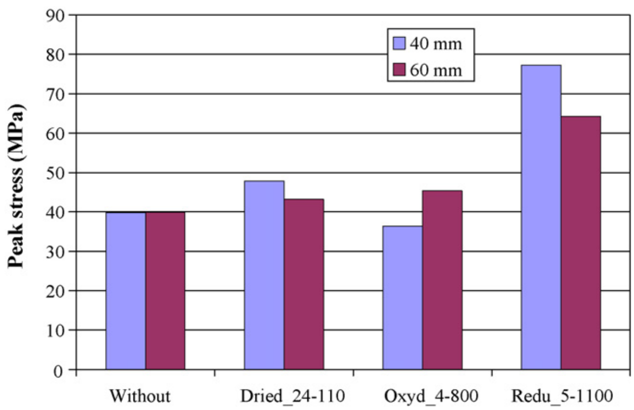

Fig. 11. Influence of the heat treatment on the refractory concrete tested at room temperature.

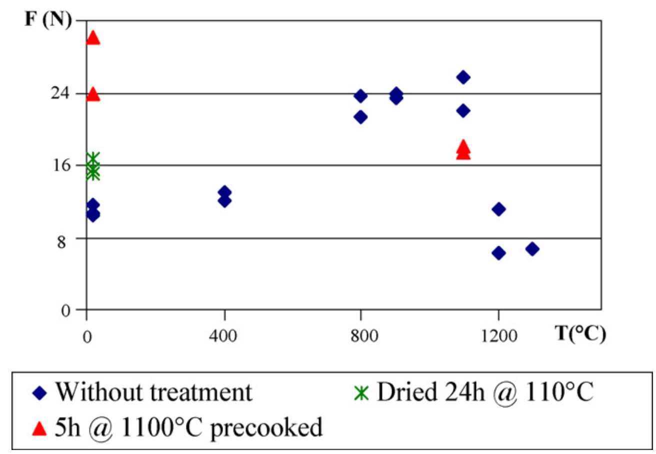

Fig. 13. Synthesis splitting tests curves on different initial state of the material. Temperature mentioned is testing one (the right unit is KN).

on the refractory concrete. The reference is the state of the material without any treatment. Whereas the 40 mm cubic specimen mean strength decreases sensibly when oxidised at 800 °C during 5 h compared to dried state, the resistance of the 60 mm cubic specimen in the same conditions increases slightly. This is probably a diffusion limit layer effect that has been evidenced and will be treated hereafter. This figure also shows that specimens fired in reducing atmosphere at 1100 °C during 5 h improve sensibly their mean strength at room temperature for both sizes. For instance, the 60 mm specimen fired at 1100 °C in reducing atmosphere exhibits a 77.3 MPa compared with the 42 MPa intact specimen strength. The influence of the oxidation duration is visible in Fig. 12. 40 mm specimens oxidised at 800 °C during 5 and 24 h have different resistances: 36.4 MPa for 5 h, 46.4 MPa for 24 h to be compared with the 40 MPa for the intact specimen at ambient temperature (see Fig. 11). But the presence in reducing atmosphere increases the material strength at ambient temperature. It seems however that the material behaviour is different in indirect tension at high temperature. For instance a specimen fired at 1100 °C in reducing atmosphere has been tested at 1100 °C in reducing atmosphere. It appears in Fig. 13 that the fired specimen strength is lower than intact or dried specimens. This result is surprising and should be confirmed by other tests.

3.6. Influence of the test atmosphere and the specimen size

Influence of the test atmosphere is evaluated on tests carried out at high temperature. Reducing conditions have been obtained by a special cylindrical container in stainless steel filled with some coke all around the specimen. The high-density carbon of the coke preferably reacts with the surrounding oxygen and then prevents the specimen from oxidising. Oxidising conditions are tests performed with non-special environment care. These tests have been performed for both conditions at intermediate temperatures because several microstructure changes operate during this temperature range.[9] It can be seen in Fig. 14 that on temperature ranging from 900 to 1200 °C that the material strength is higher in reducing conditions than in oxidising conditions. So, the material is stronger at elevated temperature when it is protected from oxidation. This refractory concrete contains high content carbon that gives it a high resistance to high-temperature solicitation in one hand but exposes it in the other hand to oxidation. Previous study[9] has established that sintering acts in this material at this level of temperature. The sintering phenomena acting within the material at these temperature levels produce stronger links in reducing than in oxidation conditions.

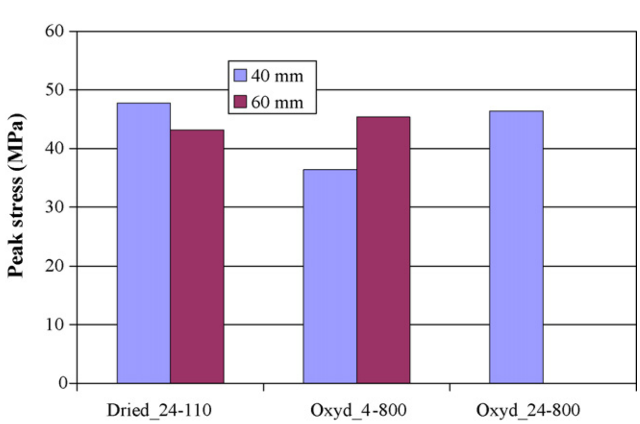

Fig. 12. Evidence of the treatment duration on the material behaviour when preheated at elevated temperature.

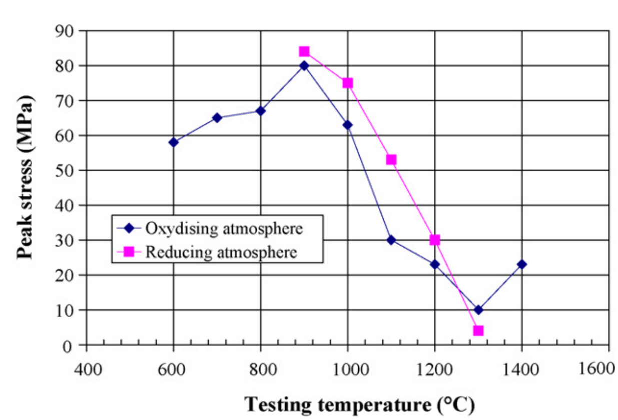

Fig. 14. Impact of the test atmosphere on the strength of the refractory concrete at intermediate to high temperatures.

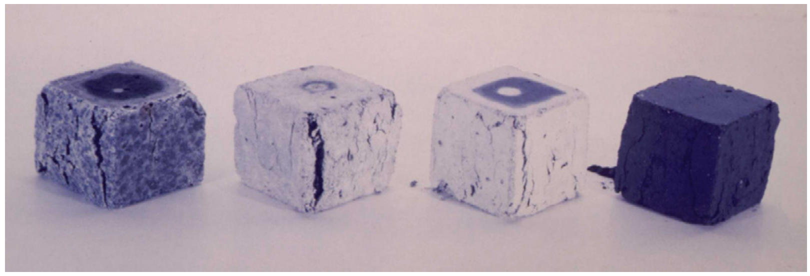

Fig. 15. View of the samples coloured after tests, respectively, at 1500, 1100, 800 and 20 °C (from left to right).

The most spectacular influence of oxidation of the studied material is the specimen colour change. Fig. 15 displays photographs of specimens after bearing test at 1500, 1100, 800, and 20 °C. It appears that initial carbon black colour of the specimen disappears and the specimen whitens with increasing temperature. At high temperature, i.e. 1500 °C, the specimen whiteness decreases due probably to new chemical reactions taking place in the material. On the other hand, tests performed on 40 and 60 mm cubic specimens in oxidising atmosphere at a testing temperature of 1100 °C revealed a great difference in the maximum strength in the same testing conditions: 60 mm specimen's strength was more than 85%. Investigation by cutting the specimen revealed that 40 mm specimen was completely oxidised whereas 60 mm specimen was just oxidised in the surroundings: the core still remains in reduced state. Trying to understand the reasons, some tests have been performed at ambient temperature on 40 mm specimen fired in reducing atmosphere during 5 h at 1100 °C and 60 mm specimen fired in oxidising atmosphere. The oxidised 60 mm specimen had a compression strength (76.2 MPa) comparable to 40 mm reduced specimen (77.3 MPa). The increase of the firing time in the oxidising atmosphere of the 60 mm specimen during tests held at 1100 °C had no effect in its resistance. These investigations suggested that a diffusion limit layer phenomenon was present in the 60 mm specimen and the latest tests confirmed this. 60 mm specimen has enough volume for the oxidised limit layer to develop and stabilize whereas it disappears in the 40 mm specimen, which is then totally oxidised.

The existence of a limit layer in 60 mm samples evidenced by the tests performed in oxidising atmosphere is an interesting result of the present study. Hence, the material behaviour depends not only on the temperature, but also on its state and deepness of oxidation. The study has also shown that this type of material is sensitive to the level of applied temperature and to the time during which it has been applied with a non-linear evolution. In other words, one must take into account chemical kinetics in material behaviour. These different aspects express the complexity of the material behaviour and the a priori difficulty to model it. It seems that an internal variable describing thermal and chemical histories should be taken into account in the constitutive equations for a realistic description.

3.7. Other influent factors

Many tests have been carried out to evaluate the lubrication influence on the material response. In general, when specimen contact faces are lubricated the maximum stress measured during compression test decreases commonly in a rate of 20–30%. One can see in Fig. 16 the evolution of the mean strength of two size specimens when no lubricant, grease or carbon sheet lubricants are used. At ambient temperature it appears clearly that carbon sheet is the more efficient lubricant. Many tests showed that at high-temperature lubrication is efficient in any case in reducing atmosphere. In revenge in oxidising atmosphere the temperature creates so a powerful reactive chemical conditions that no lubricant can resist: grease lubricant is usable up to 540 °C and carbon sheet lubricants up to 900 °C. Lubricated results are theoretically more pertinent, because strain field is more uniform, than non-lubricated tests results. But on the other hand, non-lubricated results are more reproductive and less sensible to scale effect than lubricated ones.

3.8. Characterisation of the material thermo-mechanical behaviour

The compression tests performed in this study show that from room temperature to about 800 °C, the carbon refractory concretes have a non-linear quasi-brittle behaviour characterised by a bell shape of the strain–stress curve and a sharp post-peak rupture. Visual direct observations were difficult to make because of the black colour of the material at room temperature. However, the cracks apparition was noisy and the resulting macro-cracks visible enough. Some quantitative techniques are used to quantify the frequency of cracks acoustic emission. Such testing device was not available so that the evaluation of the emissions

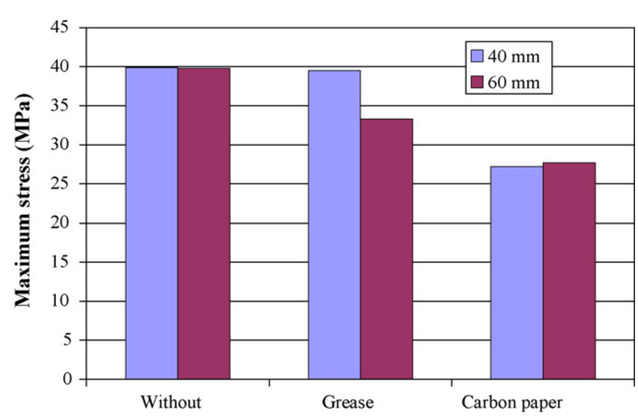

Fig. 16. Example of influence of the lubricant type. Tests have been performed at room temperature.

of the noise induced by cracking and the material fracture were simply qualitative. The brittle nature of the material behaviour is also emphasized by the weak value of the strain measured at peak stress. Unloading and reloading tests did not reveal a great inelastic deformation at this range of temperature. In term of constitutive equations, an elastic model coupled with damage can reasonably describe the material behaviour.[10–14]

For temperatures ranging from 900 to 1000 °C, the material behaviour is non-linear with a bell-shaped stress–strain curve but with more ductility, i.e. the peak strain is bigger. Moreover, unloading and reloading cycles showed that current and postponed inelastic strain were notably present. Constitutive equations gathering plasticity and viscoplasticity coupled with damage are necessary to describe the material behaviour here.

For temperatures higher than 1000 °C, the material stress–strain response becomes flat, which is the sign of essentially viscous behaviour. A viscoplasticity modelling coupled or not with damage should be convenient to describe here the material behaviour.

The viscous behaviour of the material at high temperature explains why the material can endure the great stresses induced by the thermal shock applied to the main trough. The viscous strains induce a stress redistribution, which tends to balance the state of stress in the material. On the other hand, the tertiary creep evidenced by the creep tests is of concern. If the resulting compression stress is high enough, the material can then collapse by creep. The simultaneous or separate occurrence of creep and ratchetting can explain why the material lifetime is necessary limited in service thermal cycling conditions.

4. Conclusion

Mechanical characterisation of a carbon containing alumina refractory concrete has been conducted in a specific high-temperature mechanical testing apparatus that has been first designed and developed. The uniaxial compression and the indirect tensile tests revealed the complexity of such a material.

- The influence of lubrication of the sample/crushing plate interface on the material behaviour is important. But at high temperature, whereas it is effective in reducing atmosphere, the lubricants are generally oxidised and disappear in oxidising atmosphere so that lubrication is no more efficient.
- The investigations on the refractory concrete at ambient and high temperature have revealed the existence of a limit diffusion layer to oxidation when the materials have been heated or tested in oxidising atmosphere. The existence of this limit layer induces a scale effect visible in the responses of the 40 and 60 mm specimens when tested in oxidising conditions. Nevertheless, in reducing atmosphere the scale effect is negligible. That means that firing the material impacts its behaviour at ambient or at high temperature.
- The material compression resistance is temperature-dependent with a maximum value at 900 °C. Creep tests showed an important sensibility of the material to tertiary creep at or above 800 °C under compression solicitation. It has also been established that the materials develop ratchetting effect when it is submitted to cyclic loading–unloading tests since 800 °C.
- Lastly, the studied material has quasi-brittle and damageable non-linear behaviour up to intermediate temperatures and viscous and damageable behaviour at higher temperature. This points out the constitutive equations that should be encountered to predict the material behaviour.

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