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Investigation of the thermomechanical properties of industrial refractories: the French programme PROMETHEREF

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Abstract This paper summarises the work that has been done in the framework of the French programme PROMETHEREF. This programme was concerned with the thermomechanical properties at high temperature of two industrial refractories: fused-cast materials for glass melting and alumina castables for steel production. At high temperature, both materials exhibit creep, that has been characterised by tension, compression and bending tests. The microstructural mechanisms of deformation have been investigated and allowed the macroscopic viscoplasticity to be understood. Both types of materials exhibit damage processes that have also been characterised mechanically and microstructurally. The nature and the adhesion of the aggregates have been shown to have a great influence on the mechanical behaviour of the castables, as well as the continuous zirconia skeleton observed in high-zirconia fused-cast refractories by X-rays tomography.

Introduction

PROMETHEREF was a large research programme (1.7M€) that started in January 2003 [1–3]. This programme was supported by the French Ministry of Industry, in the frame of the "Materials and Processes" National Net. The first aim of this federation, constituted of industrial companies and of university research centres, was to create synergies by sharing the results that have been produced by different partners, to facilitate a better efficiency, for understanding the complex thermomechanical processes that affect refractories in service. The mechanical models that have been developed are presently used to simulate the behaviour of the refractories during actual working, and then to facilitate and to improve the development of new refractories and the design of future refractorised industrial structures.

Three industrial partners have been involved in PROMETHEREF, each of them having a particular interest in this study:

- Saint-Gobain CREE, R&D Centre for Saint-Gobain High Performance Materials in Europe (Cavaillon), associated with the SEPR glass furnace builder, provided the specimens and now uses the numerical models, in particular for the simulation of cooling-down sequences for refractory.
- TRB, refractories producer (Nesles), also provided materials, performed mechanical tests and exploited the results to develop new refractory compositions, and now uses the models for the numerical simulation of steel ladles and other applications.
- Electricité de France, Renardières Research Centre (Moret sur Loing), was interested in the mechanical behaviour of refractory linings for circulating fluidised bed boilers.

Five public laboratories used their specific competences in the programme:

- Ecole Nationale Supérieure des Mines de Paris, Materials Centre P.M. Fourt (Evry), investigated the 3D microstructure, performed microstructural numerical simulation of fused-cast refractories and characterised and modelled creep and damage (PhD works of Ludovic Massard [4] and Kamel Madi [5]).

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- Institut National des Sciences Appliquées, MATEIS group (Lyon), studied the mechanical and microstructural effects of damage, micro- and macro-cracking on fused-cast materials (PhD work of Emilie Lataste [6]).
- Ecole Nationale Supérieure de Céramique Industrielle, GEMH laboratory (Limoges), characterised thermal properties and investigated high-temperature elastic properties using an ultrasonic technique (PhD work of Edwige Yeugo-Fogaing [7]).
- Ecole Nationale Supérieure des Techniques Industrielles des Mines, CROMeP laboratory (Albi-Carmaux), performed and analysed tensile and bending tests and microstructural observations on refractory castables (PhD work of Hicham Marzagui [8]).
- Institut National Polytechnique, 3S laboratory (Grenoble), carried out compression and diametral-compression tests on refractory castables, identified the mechanical behaviour constitutive equations and performed numerical simulations (PhD work of Mohsen Roosefid [9]).

Some examples of results obtained in the framework of this programme are given below, successively for fused-cast refractories and for refractory castables.

Fused-cast refractories

Two fused-cast refractories [10, 11], from the alumina–zirconia–silica system, have been selected for this study. One, called AZS, is composed of around 45 wt% of alumina, 35 wt% of zirconia and 20 wt% of glassy phase surrounding the crystalline phases. The other, called HZ (for High Zirconia), is a zirconia-rich material (around 94 wt% zirconia), the spaces between zirconia regions being filled-in by a glassy phase (approximately 6 wt%). The microstructures of these materials are, respectively, represented in Figs. 1 and 2.

3D pictures of these microstructures have been obtained through X-ray tomography using the European Synchrotron Radiation Facility (ESRF) in Grenoble (France). As zirconia is rather an absorbing material, ID19 beamline has been used: an incident energy of 37 keV allows a resolution of about 0.7 μm to be reached on cylindrical specimens of 500 μm diameter. Figure 3a provides an image obtained of the HZ material. The good contrast between the zirconia and the glassy phase allows an easy share of both the phases owing to an image analysis segmentation process. Figure 3b represents the isolated intergranular phase. 3D image analyses prove that both zirconia and glassy phase percolate, even on short distances. Contrary to sintered materials constituted of isolated, individualised grains, the crystalline phase of this fused-cast material is spatially interconnected. We will see later in this paper that this which cannot be obtained using simple 2D imaging, brings important informations about the observed properties of this refractory [12].

Figure 3c represents an automatic meshing [13] of the intergranular phase, which has further been used as the basis for a micro–macro approach to the simulation of mechanical properties, using finite elements [14]. These simulations, that have found other applications [15], are in particular useful to understand and predict the macroscopic behaviour of the material from the behaviours of the elementary constituents, taking into account the actual, original topology of the microstructure of fused-cast refractories.

An ultrasonic technique based on the continuous in situ measurement of ultrasonic wave velocities in materials [16, 17] has been used to monitor the evolution of the elastic modulus versus temperature for both materials. The long

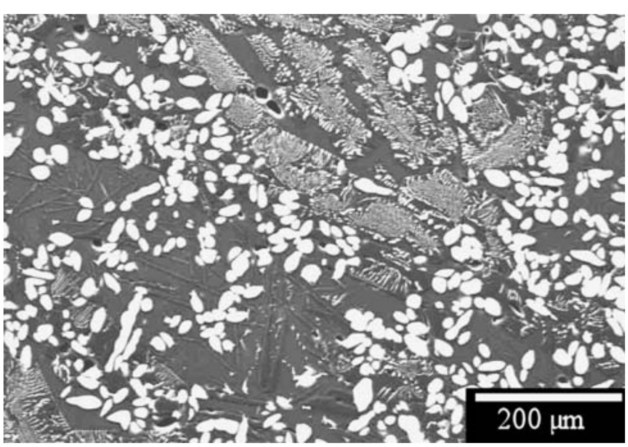

Fig. 1 Microstructure of AZS observed by SEM: zirconia, corundum and eutectic grains are surrounded by the glassy phase

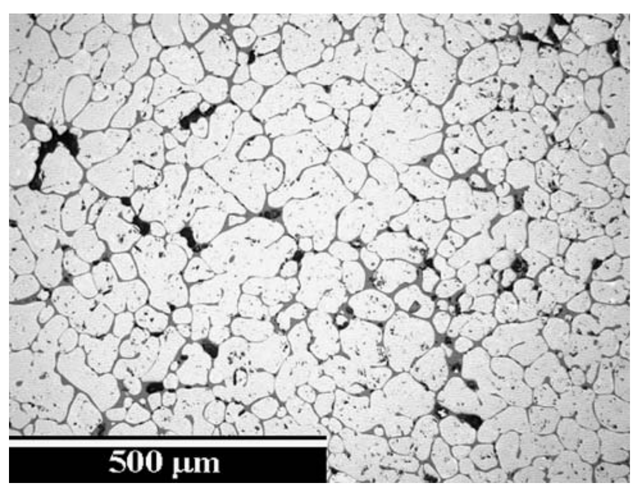

Fig. 2 Microstructure of HZ observed by SEM: saturated dendritic structure of zirconia surrounded by the glassy phase

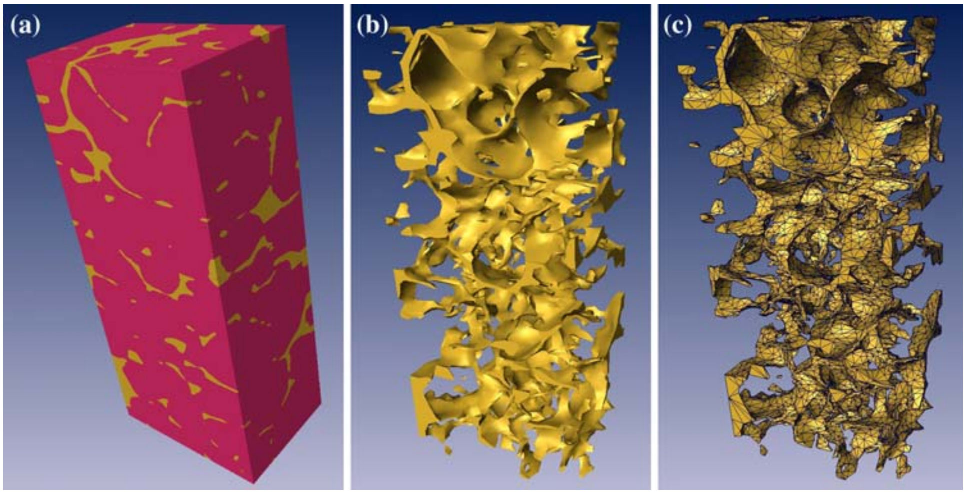

Fig. 3 3D images of the HZ refractory microstructure [5] (**a**) bulk material: zirconia (red) surrounded by the glassy phase (yellow). (**b**) Sole glassy phase. (**c**) Isosurface (interface between both constituents) meshed for FE calculations

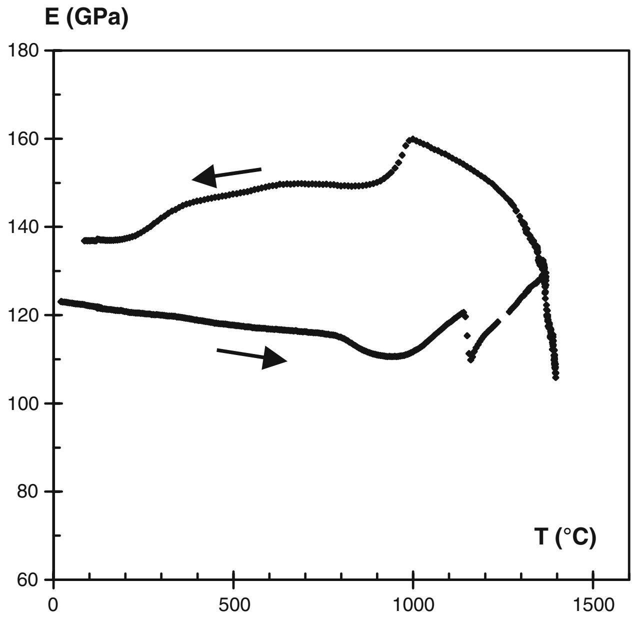

Fig. 4 AZS refractory elastic modulus measured by ultrasonic method during a heating-cooling cycle at 5 °C/min [7]

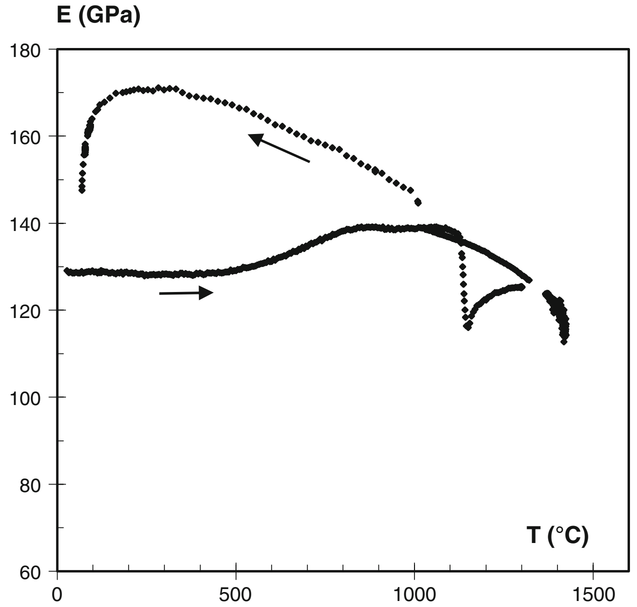

Fig. 5 HZ refractory elastic modulus measured by ultrasonic method during a heating-cooling cycle at 5 °C/min [7]

bar-type specimens have been excited at 140 kHz during a heating-cooling cycle at 5 °C/min. Typical responses obtained for AZS and HZ are illustrated, respectively, in Figs. 4 and 5.

Both materials exhibit a non-linear evolution of the elastic modulus [18]. The hysteretic shapes of the curves reveal that the samples are initially damaged, because of the volume expansion associated with the zirconia transformation and of the thermal expansion mismatch between the different phases of the materials [19, 20]. A confirmation of this assumption has been provided by thermoelastic finite element simulations of the cooling using the previously described mesh (Fig. 3c), which reveals the high stress levels that can be locally reached in the microstructure [5, 14]. The martensitic transformation of zirconia [21–23] (from monoclinic to tetragonal during heating and back during cooling) is clearly observed in both cases. However, this latter effect has opposite consequences on both materials. During heating, the volume reduction (4% for pure zirconia) associated with the transformation may cause decohesions that could lower the Young's modulus. During cooling, an increase of the modulus is observed for the zirconia-rich material. The volume expansion due to the reverse transformation of zirconia allows the closure of the cracks within the microstructure. In the case of AZS material, the higher content of glassy phase enables the healing of decohesions at high temperature, when its viscosity is low enough. Then the viscosity increase of this glassy phase leads to an increase of the Young's modulus at the beginning of

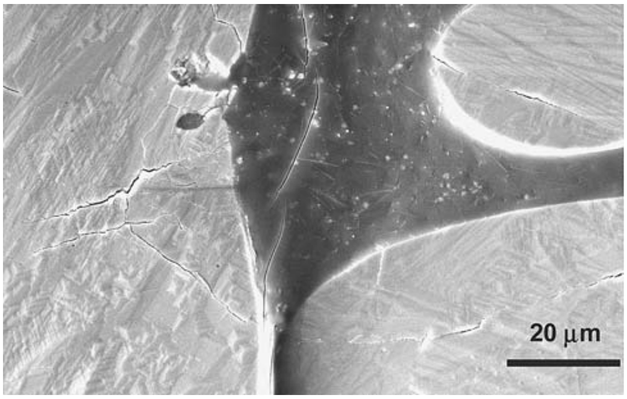

Fig. 6 HZ refractory damage by microcracking observed by SEM after a 1,300 °C heat treatment [6]. Cracks are observed at the interface and within zirconia and glassy phase

cooling. However, when the volume expands due to zirconia transformation, microcracks are created, that lower the modulus. The two materials later exhibit a decrease of modulus at the end of cooling, because of the damage due to thermal expansion mismatches between the different phases [24, 25].

SEM observations confirm the existence of this damage [6]. As illustrated in Fig. 6, it takes the form of microcracks and/or decohesions that affect the zirconia as well as the glassy phase. On the surface of some zirconia grains, intercrystalline planes are revealed, that are able to deviate and complicate the crack path at a very low scale. High-temperature environmental SEM revealed that zirconia grains are strongly damaged by the transformation of zirconia during heating, despite the strain accommodation allowed by the glassy phase. This damage is later healed at higher temperature owing to activated diffusional processes and loss of viscosity of the glass. During cooling, below the glassy transition temperature, internal thermal stresses lead to a diffuse microcracking of the material, that relaxes the internal stresses and then contributes to maintaining the integrity of the refractory.

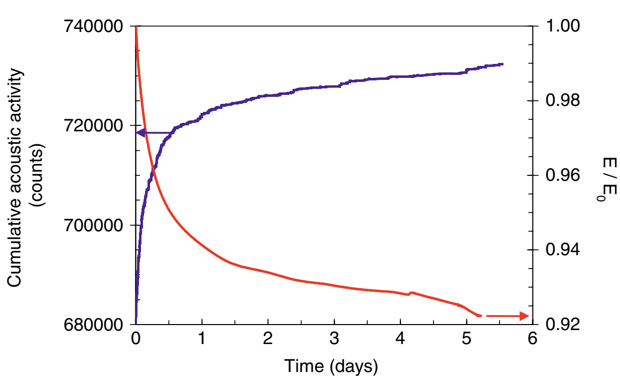

Fig. 7 Acoustic emission and loss of stiffness observed in HZ refractory at room temperature after a 1,000 °C heat treatment [6]. Damage still develops within the refractory

This microcracking continues to develop with time in isothermal conditions at room temperature [6]. Figure 7 illustrates, after a heat treatment at 1,000 °C, the loss in stiffness and the acoustic activity as a function of time. In multiphase ceramic materials constituted of different brittle phases, these associated phenomena are generally attributed to damage by microcracks' extensions caused by internal stresses [26–28]. About 1 week is necessary to reach a steady state. Stress corrosion of the glassy phase by water vapour of the atmosphere probably contributes to this microcracking.

For investigating high-temperature mechanical properties [4, 29], specific experimental devices have been developed. Figure 8 represents a schematic of a tensile jig and of the associated specimen for testing refractories at temperatures up to 1,600 °C. Heating is provided by a furnace that can be opened in three parts, using molybdenum disilicide heating elements. An inversed loading system using two groups of three silicon carbide rods passing through the holes of one specimen shoulder and pushing on the opposite shoulder generates tensile stresses in the central gauge length. The whole specimen is at the test temperature ("hot gripping"). To avoid parasitic bending stresses, the three rods constituting each loading group are supported by hydraulic jacks linked together: this system allows an equal share of the applied load among the three rods. Finally, the average specimen elongation is measured by three extensometers using LVDT-type

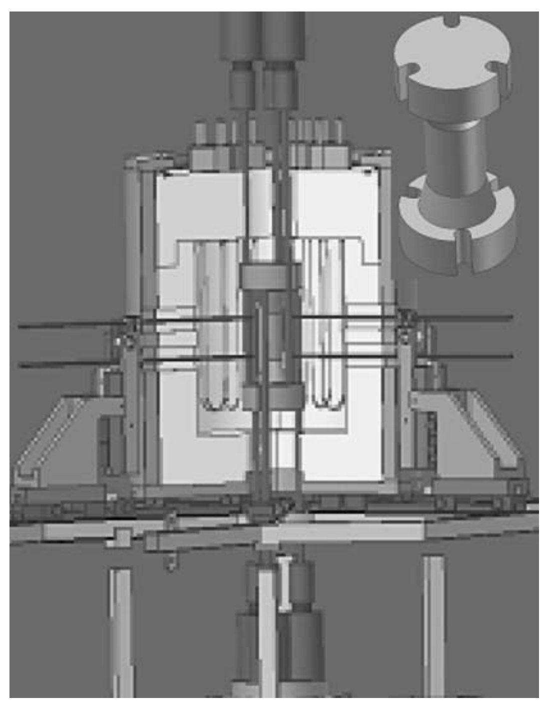

Fig. 8 Schematic of the high-temperature tensile jig and of the associated specimen [4]

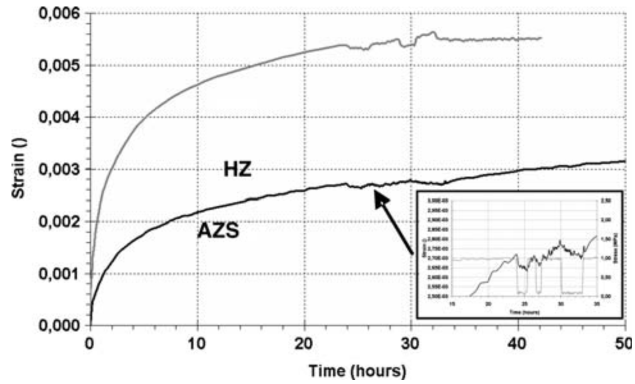

Fig. 9 Typical creep curves (1,450 °C, 1 MPa) of HZ refractory and dip-tests revealing the strain recovery [4]

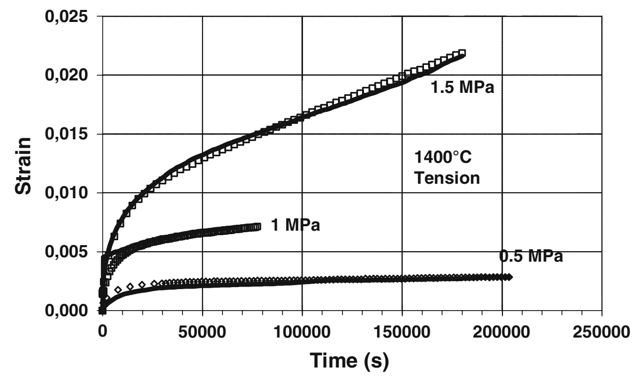

Fig. 10 Tensile behaviour of HZ refractory at 1,400 °C: comparison between experiment and modelling [4]

transducers, located at 120° around the specimen (gauge length 40 mm).

Creep tests have been performed under isothermal conditions, to collect information about the creep type of the materials [4]. Figure 9 represents an example of the increase of the strain versus time for a specimen loaded under 1 MPa at 1,450 °C for both materials. A quasi-steady-state creep is reached after a few tenths of an hour: the creep rates of both materials are then similar. Despite the presence of a glassy phase, since the glass transition temperature is around 780 °C, both materials exhibit low creep rates. The explanation of this remark is probably to be found in the percolation of the crystalline phases previously noted, which leads to a creep resistant crystallised skeleton within the material. This results in a large difference compared with sintered granular materials, in which individualised grains are subjected to grain boundary sliding that considerably increases the creep rate [30, 31].

During creep, partial unloadings of the specimens have been performed to examine, for some minutes, the consequences of a load decrease on the creep rate: this procedure is known as “dip-test”. These unloading sequences are responsible for the drops in strain observed in Fig. 9. A magnification of the deflection observed during these short unloading sequences is also provided in Fig. 9. A negative creep rate is then revealed (the specimen creeps in an opposite direction than that expected with respect to the applied stress), which corresponds to a strain recovery of the material. This observation is typical of a kinematic hardening creep type, which is generally explained by internal stresses developing within the material during creep [32, 33].

Figure 10 illustrates an example of the good agreement observed in tension between experiment and modelling at 1,400 °C. However, the fitting obtained in compression with this simple model is less good, because of three main reasons:

- the experimentally observed hardening is more rapid and more intense in compression,
- the discrepancy is higher,
- average recorded strains are smaller.

The models developed for describing the mechanical behaviour of fused-cast refractories at high temperature are presently used for the simulation of blocks processing. Figure 11 gives an example of calculation result for a 2D simulation of a quarter of a block in its mould (view from upside). Figure 11a illustrates the temperature distribution induced by the heat diffusion from the block (bottom left of the figure) to the mould. The thermal gradient in the block generates thermal stresses (perpendicular to the plane of the figure), that are schematised in Fig. 11b. In this case, the stress concentration located at the edge of the block is, for example, clearly revealed.

Refractory castables

The work carried out on castables focuses on the determination of mechanical behaviour laws and damage and/or cracking criteria, suitable for large panels of refractory concretes, from room temperature to 1,200 °C. Two alumina castables, a Bauxite-based ultra low cement content (Bau-ULCC) and an Andalousite-based low cement content (And-LCC) have been studied. According to the industrial procedure for concrete elaboration, castables have been wetted, cast and vibrated; cured for 1-day at room temperature and finally treated at 110 °C to achieve hydrates' conversion from C_2AH_8 to C_3AH_6 and AH_3.

First, the evolution of crystalline phases has been investigated by X-ray diffraction (XRD) performed at room temperature, after thermal treatments at different temperatures [8]. The results are summarised in Table 1, “x” denoting the presence of referenced phases. Dilatometric and ultrasonic measurements have also been performed during a heating-cooling cycle at 5 °C/min up to 1,500 °C. Figure 12 provides an example of the behaviour observed for And-LCC castable: thermal expansion and elastic modulus are represented together. The dehydration process of the hydrated calcium aluminates [34–36] induces a strong decrease in elastic modulus between 150 and 350 °C. In this temperature range, a slight decrease in thermal expansion is also observed. The increase of the elastic modulus between 900 and 1,200 °C can be attributed to the formation of CA_2 and CAS_2, and also to sintering mechanisms which can occur in this temperature range [36, 37].

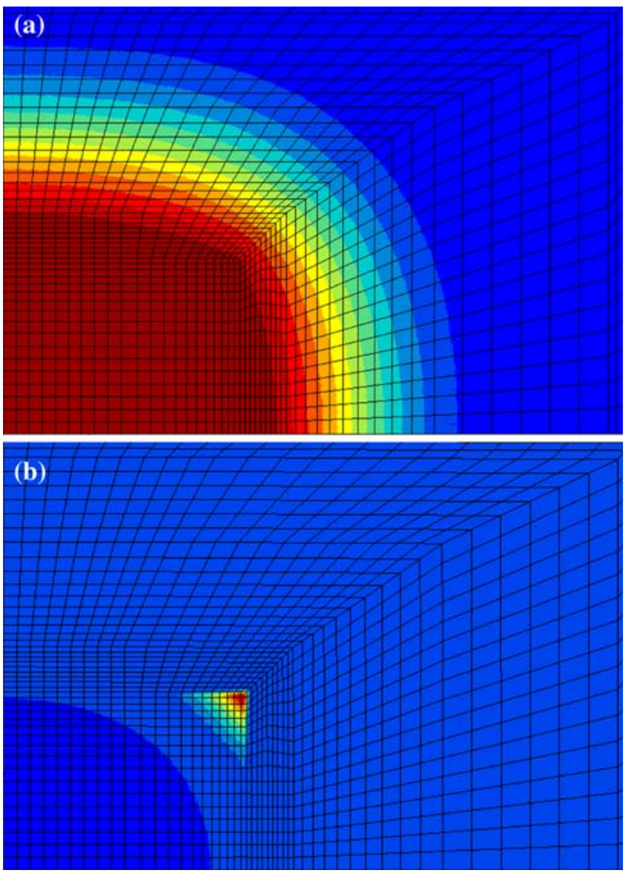

Fig. 11 Numerical simulation results of a fused-cast refractory block processing (**a**) temperature field (**b**) stress field

To investigate microstructural changes occurring in the matrix of the castables from room temperature to 1,300 °C, environmental SEM has been performed [8, 36]. The observed matrix, without aggregates, was prepared after sifting the initial powder through a 200-μm mesh. Figure 13 presents microstructural observations performed at different temperatures. During heating, according to the observations made for elastic modulus and thermal expansion, the material shrinks between 150 and 300 °C. Due to the dehydration of C_3AH_6 and AH_3, this shrinkage generates microcracks in the matrix and the concrete becomes weaker. From about 1,200 °C, Fig. 13 clearly reveals the appearance of a phase having a low viscosity. Moreover, the creation of viscous necks (indicated by the white circles) is also observed, leading to partial crack healing. Because of this partial sintering, during the cooling period, the elastic modulus remains rather high (about 75 GPa between 800 and 1,000 °C), but drops from 750 °C. This decrease in stiffness, associated to a change in thermal expansion, attests to microcracks formation, probably because of thermal expansion mismatches between the different phases.

Tensile tests after thermal treatments at different temperatures confirm the great influence of these microstructural evolutions on the mechanical behaviour: Fig. 14 illustrates the deviations from linearity induced by thermal treatments.

As represented in Fig. 15, according to elastic modulus evolution, tensile strength after thermal treatment exhibits a significant decrease due to microcracking generated by the dehydration of cementitious phases [38]. When the tensile strength is measured at high temperature, results differ slightly from room temperature, especially in the 500–700 °C range (Fig. 15). Up to 700 °C, a quasi-brittle behaviour is observed: the opening of the loading–unloading loops (Fig. 16) and the permanent deformations after unloading remain very limited. In contrast, very large irreversible deformation and extensive cracking are observed at 900 °C, characteristic of a drastic change in the behaviour of the material.

Table 1 Summary of the phases detected by XRD at room temperature after thermal treatments of And-LCC at different temperatures [8]

T (°C)	AH_3	C_3AH_6	CA_2	CA	Anorthite CAS_2	Cristobalite	Andalousite	Mullite	Alumine
110	x	x	x	x			x		x
250			x	x			x		x
500			x	x			x		x
800			x	x			x		x
1,100			x		x	x	x		x
1,300			x		x	x	x	x	x
1,500					x	x		x	x

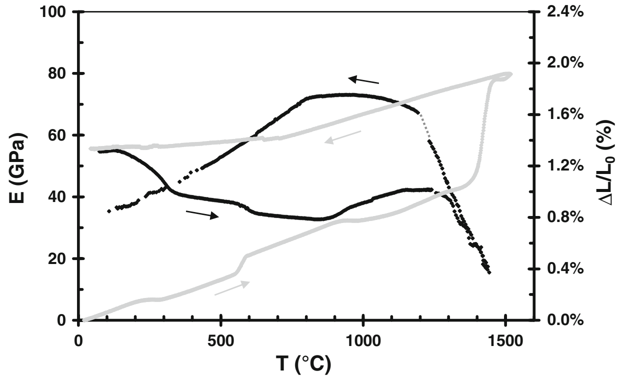

Fig. 12 Thermal expansion (grey line) and elastic modulus (black line) variations of And-LCC castable during the first heating-cooling cycle at 5 °C/min [7]

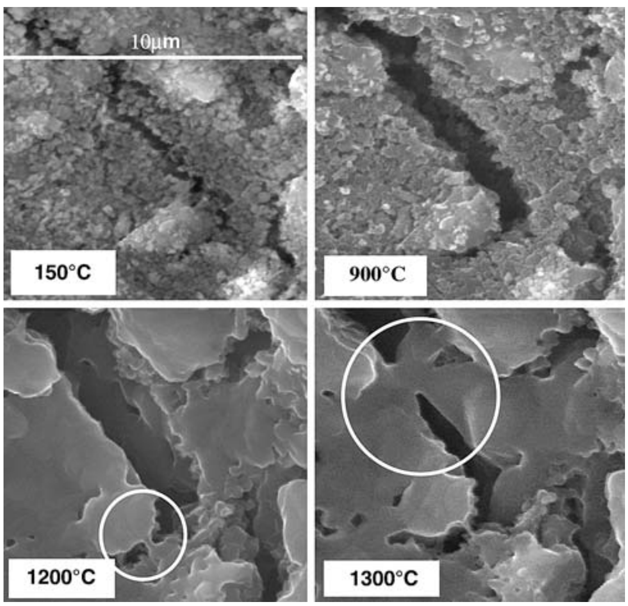

Fig. 13 Microstructural changes (microcracking due to shrinkage and creation of a viscoplastic phase) during heating of the And-LCC matrix observed by environmental SEM [8]

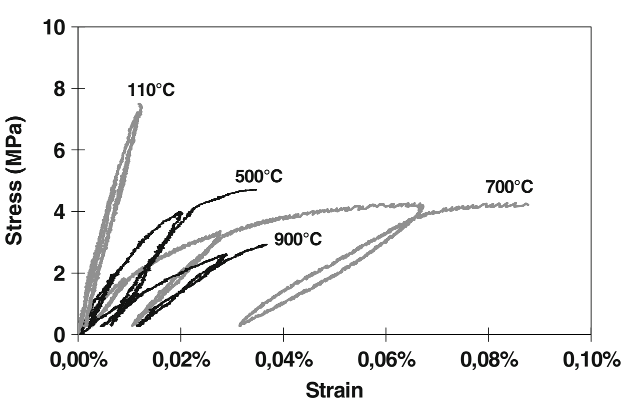

Fig. 14 Room temperature tensile behaviour changes of And-LCC induced by firing at different temperatures [8]

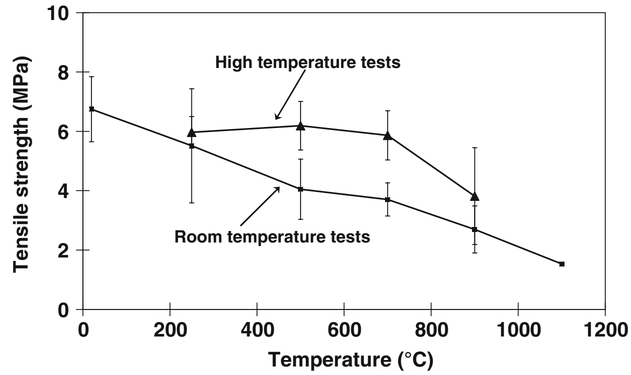

Fig. 15 Room temperature and high-temperature tensile strength of And-LCC castable after thermal treatments [8]

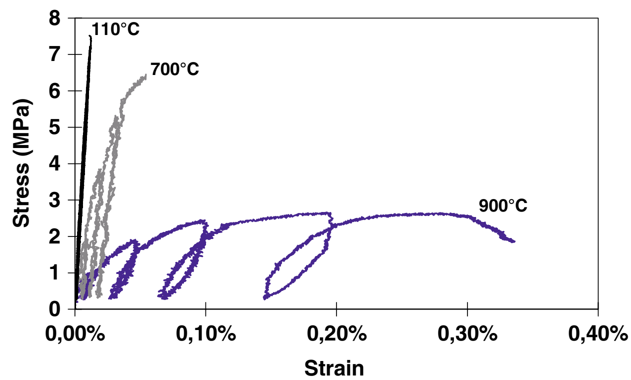

Fig. 16 High-temperature tensile behaviour changes induced by temperature in And-LCC castable [8]

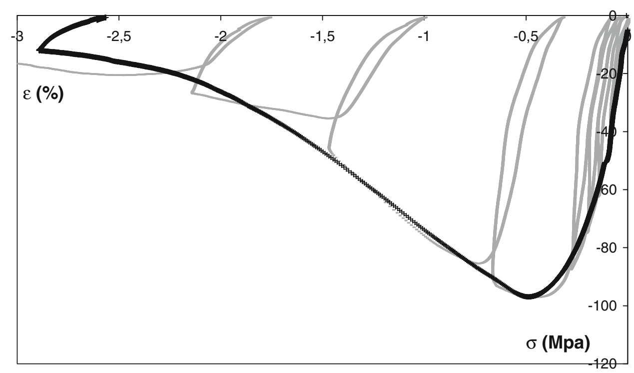

Fig. 17 Room temperature compressive behaviour of And-LCC castable during loading–unloading cycles [9]

As generally observed for concretes or rocks, the mechanical behaviour in compression is very different from that in tension. Figure 17 represents the strain–stress curve recorded in compression at room temperature after a thermal treatment at 110 °C, for a monotonic loading and for loading–unloading sequences [9]. The level of applied stress is about 10 times higher than in tension, and the castable exhibits irreversible deformation because of the

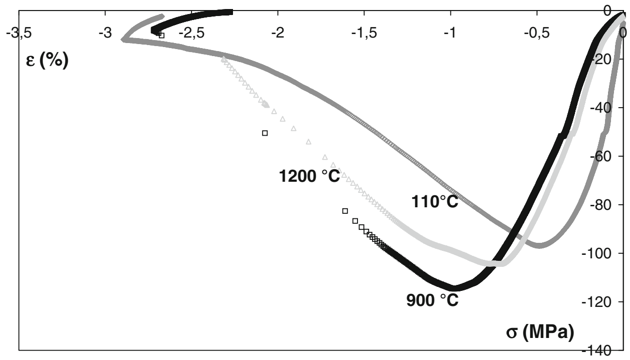

Fig. 18 Room temperature compressive behaviour changes of And-LCC castable induced by firing at different temperatures [9]

damage. As shown in Fig. 18, firing has less influence on the behaviour in compression than in tension: whatever be the thermal treatment temperature, a damaging behaviour is always observed [39]. The strength and the elastic modulus are lightly affected by hydrate conversion and by sintering.

In accordance with the industrial partners, the material responses to different loading conditions and the operational criteria lead to the choice of a model which couples elasticity with damage constitutive equations proposed by Mazars [40–43] to simulate the materials' behaviours. This model is based upon an elastic material behaviour which considers a damage, that is assumed isotropic and that can then be described by a scalar variable D. One limitation of this model is that it is not able to describe non-reversible strains provoked by damage, sometimes exhibited by the materials during loading–unloading tests. However, the used damage variable allowed non-linear evolution of the materials' behaviours subjected to a monotonic loading to be well fitted. Figure 19 illustrates this good agreement in the case of a compression test: the shape of the behaviour curve and the increase in damage are well described by the

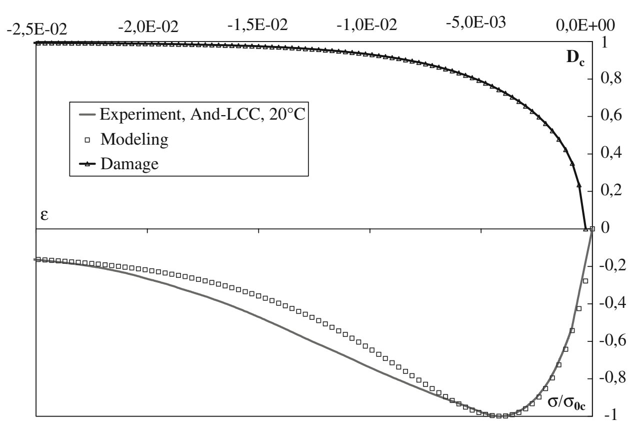

Fig. 19 Room temperature compressive behaviour and damage of And-LCC castable: comparison between experiment and modelling [9]

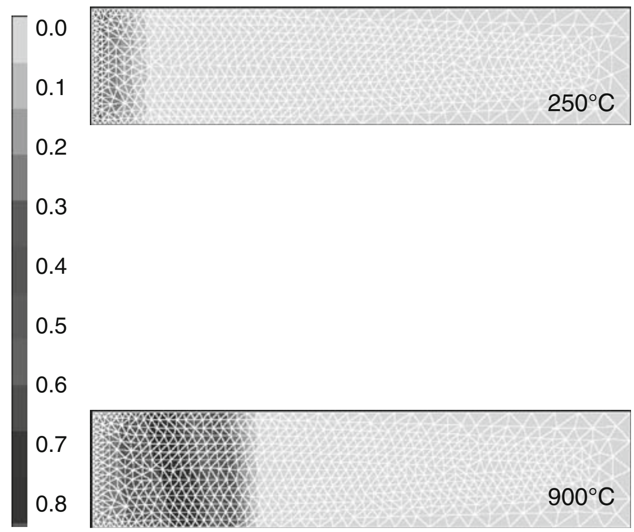

Fig. 20 Evolution of damage field around a steel anchor settlement in the And-LCC refractory concrete from 250 to 900 °C

model. Identification of numerical values of the constitutive equations in compression and tension has been performed owing to compression and 4-point bending tests results, for each test temperature. The mechanical behaviours of both castables were characterised by different values of numerical coefficients. Both the mechanical behaviours experimentally observed were then clearly discriminated by the model.

The developed model was used to simulate numerically, with the CAST3M finite elements code, the thermomechanical behaviour of a refractory concrete disc equipped with a metallic anchor in its centre. A result of such a calculation is illustrated in Fig. 20. The non-local approach used produced more realistic results, because it is supposed to avoid the calculation mesh dependency, but took more calculation time. The results of numerical simulations revealed that damage develops in the refractory concrete in the vicinity of the metallic anchor. It appears near 250 °C and then spreads around when temperature increases. These numerical results exhibited very good agreement with micrographic observations carried out by EMAC after experimental testing. Again, this numerical model discriminates the two refractory castables' behaviours, by predicting two different types of damage expansion. This difference was in good agreement with the experimental observations.

Conclusion

A large three and half-year national programme (PROMETHEREF) has been carried out in France concerning the thermomechanical properties of industrial refractories. It led to the collection of numerous data that improve the knowledge of thermomechanics of refractories, necessary to develop numerical simulations of refractorised industrial structures. The results obtained highlighted the importance of the 3D microstructure topology and of the phase transformation of zirconia in fused-cast materials. The recrystallisation of hydrates in refractory castables also provokes large changes in the microstructure and in the mechanical behaviour. In both cases, a stable diffuse damage through microcracking plays a very important role for maintaining the integrity of the refractories, especially at low temperature.

The collaborative work on the thermomechanical properties of industrial refractories is still continuing with two research programmes which succeed PROMETHEREF: NOREV for fused-cast refractories and DRUIDE for refractory castables.

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