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Influence of the thermal history on the mechanical properties of two alumina based castables

M. Ghassemi Kakroudi [a,b], E. Yeugo-Fogaing [c], M. Huger [b,*], C. Gault [b], T. Chotard [b,*]

[a] *Department of Ceramic Engineering, University of Tabriz, Islamic Republic of Iran*
[b] *Groupe d'Etude des Matériaux Hétérogènes (GEMH), ENSCI, Limoges, France*
[c] *Matériaux à Finalités Spécifiques, EA 3834, Institut des Sciences de l'Ingénieur de Toulon et du Var, Av. G. Pompidou, BP56, 83162 La Valette du Var Cedex, France*

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Abstract

Monolithic refractories are of importance due to their increasing applications in the metallurgical industries. In a wide range of industrial applications, refractory castables are subjected to demanding requirements, and their properties strongly depend on their thermal history.

This work is devoted to the study of the evolution of the mechanical properties of two refractory castables related to different thermal treatments corresponding to their conditions of use. The studied materials are two alumina refractory castables: an ultra low cement content bauxite based one (Bau-ULCC) and a low cement content andalusite based one (And-LCC).

Samples of both refractories have been fired at different temperatures (110, 250, 500, 700, 900 and 1100 °C) in order to simulate several conditions of use. During these firing stages, microstructural evolutions have been monitored by Young's modulus ultrasonic measurements. Then, the tensile loading behaviour of each sample has been determined both at room temperature and for a specific temperature (800 °C), chosen according to thermal history ultrasonic measurements.

Keywords: Refractories; Thermal history; Mechanical properties; Tensile behaviour

1. Introduction

The share of monolithic materials in the whole refractories market is growing worldwide. In general, wherever fired refractory bricks are used, they can be advantageously replaced by monolithics, in terms of production cost, installation efficiency, safety, material consumption, etc.[1]

Refractory castables are subjected to demanding loads, especially from a thermomechanical point of view and can be degraded by a combination of several mechanisms, mainly thermal shock, abrasion, corrosion and mechanical impact. The response of these materials is influenced by many factors such as their chemical composition, their microstructure as well as phase transformations which occur at high temperature during firing process, and/or in service.[2]

The physical properties of a refractory concrete are highly temperature dependent. This is primarily caused by the complex hydration and dehydration reactions of calcium aluminate cement.[3] The installation sequence for monolithics containing calcium aluminate cement involves several steps such as mixing, placing, drying out, curing and finally using in service. Each of these steps has influence on the hydration–dehydration processes of calcium aluminate cement (CAC).[4]

Previous studies have already been performed in the field of the high temperature behaviour of refractory castables.[2,5] This paper deals with thermal history and mechanical properties of two alumina castables and with results of an experimental approach developed to characterise the microstructural transfor-

* Corresponding authors.
E-mail addresses: marc.huger@unilim.fr (M. Huger), thierry.chotard@unilim.fr (T. Chotard).

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Table 1
Chemical analysis and characterisation data of the two refractories.

Castable type	And-LCC	Bau-ULCC
Aggregate type	Andalusite	Bauxite
Al_2O_3 (wt%)	58	85
SiO_2 (wt%)	37.5	10
CaO (wt%)	2.3	1.1
Fe_2O_3 (wt%)	0.9	1
Max. aggregate size (mm)	5	5
Water requirement (wt%)	4.5–5.5	4.2–5.2
Open porosity (vol.%)	6	10
Apparent density (kg/m^3)	2600	2970

mations and damage processes, which occur in such materials during the first heating.

2. Materials and experimental procedures

2.1. Materials and sample preparation

Two commercial castables are considered. The first one is a low cement andalusite castable (And-LCC) made of andalusite aggregates, fumed silica, alumina and of a calcium alumina cement. The second is an ultra low cement bauxite castable (Bau-ULCC) made of bauxite aggregates, fumed silica, alumina and of the same calcium–alumina cement. Both materials are characterised by the same fumed silica content (~10 wt%). In Bau-ULCC, the alumina content is two times higher than in And-LCC. Table 1 shows the chemical compositions of the castables supplied by TRB company.

The high difference between the silica contents of the two materials is mainly due to the high silica content in the andalusite aggregates compared to the bauxite ones. For both castables, the maximum aggregate size is about 5 mm. The materials were cured for 24 h at 110 °C (as-cured state). Fig. 1 shows pictures of polished sections of the as-cured materials. After machining, some samples have been fired at 250, 500, 700, 900 and 1100 °C in order to simulate a variety of thermal histories before characterisation. These temperature levels have been chosen due to the temperature range encountered by refractory castables in the considered industrial applications. Firing thermal cycles are characterised by 5 °C/min heating and cooling rates and by a 5 h isothermal dwell at the maximum firing temperature.

2.2. Ultrasonic measurements

An ultrasonic technique based on a continuous in situ measurement of the velocity of longitudinal long bar mode waves in the material has been used to monitor the evolution of the elastic modulus versus temperature on both materials.[6,7] Fig. 2 is a schematic representation of the ultrasonic device. The ultrasonic pulse is transmitted from the transducer to the sample through a wave-guide. The measurement of the time τ between two successive echoes within the sample makes it possible to calculate the wave velocity and then to obtain the value of the Young's modulus by $E_{US} = \rho(2L/\tau)^2$, where L and ρ are sample length and density, respectively.

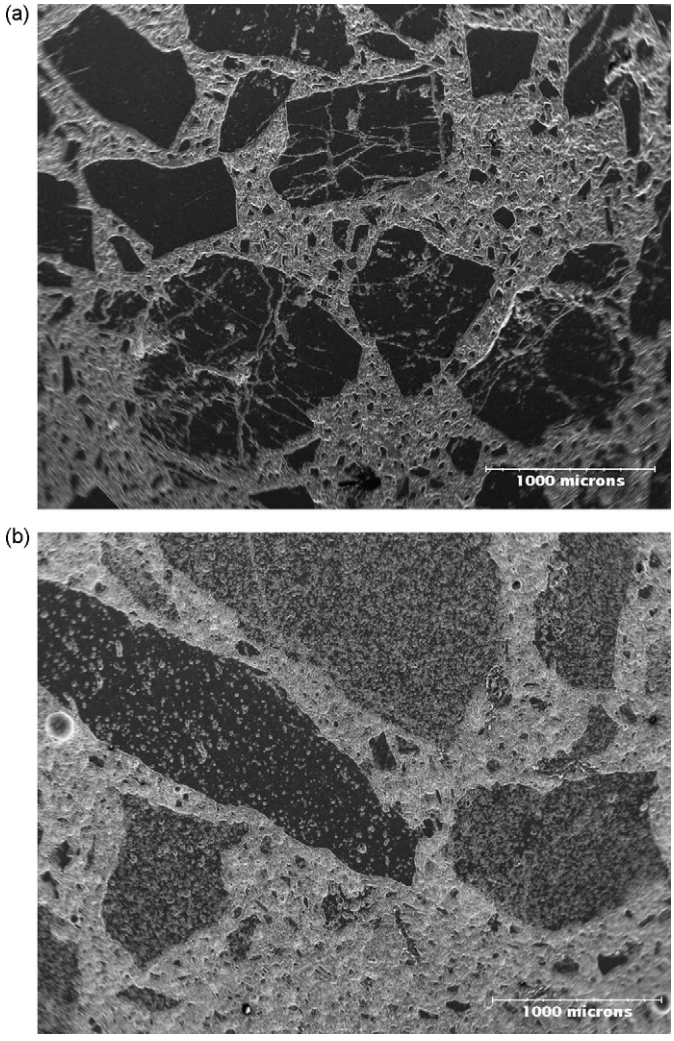

Fig. 1. Microstructure of as-cured refractories: (a) And-LCC and (b) Bau-ULCC.

2.3. Tensile test

Tensile tests have been performed with an INSTRON 8862 electro-mechanical universal testing machine at room temperature. Fig. 3 represents a schematic of the tensile test device. The variation of length is measured by two extensometers equipped

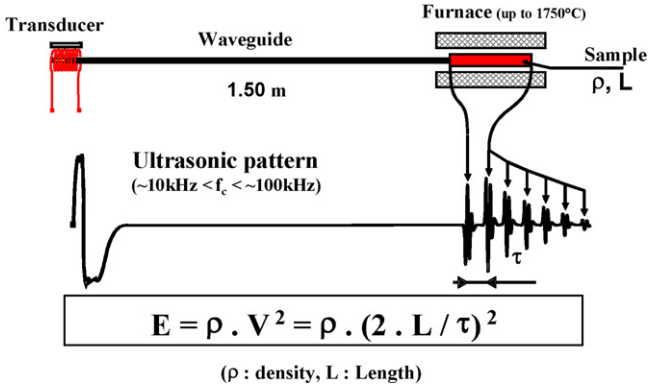

Fig. 2. Experimental setup used for Young's modulus measurement at high temperature by long bar mode ultrasonic pulse technique.

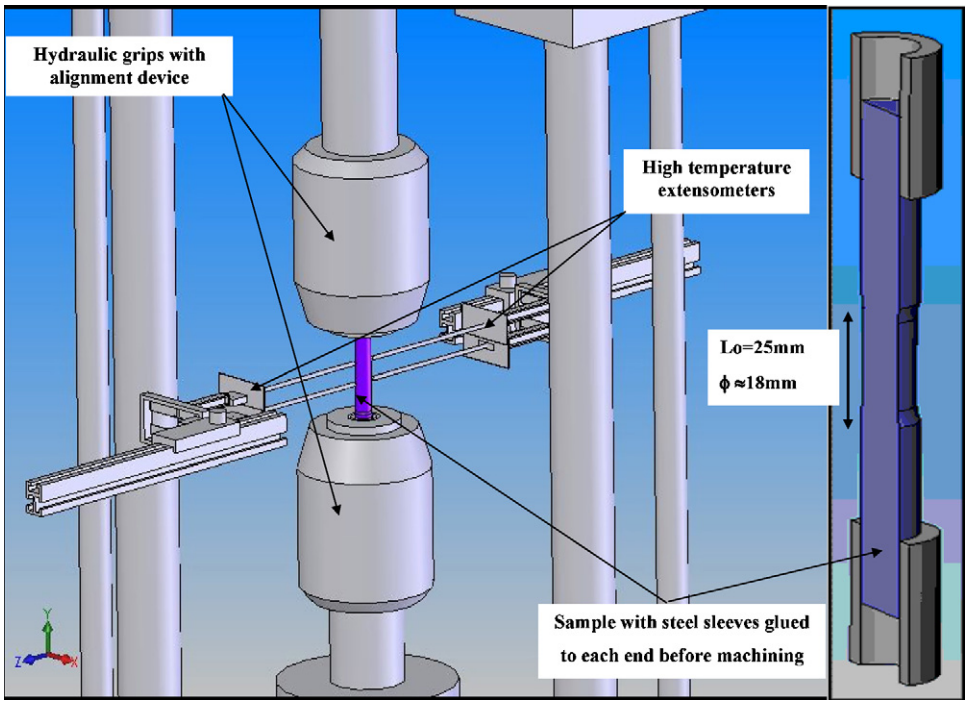

Fig. 3. Scheme of the tensile test device.

by silicon carbide rods which are placed on two opposite faces of the specimen. The extensometer gauge length is 25 mm.

The low values of the strain at rupture exhibited by these materials correspond to very low variations of the gauge length (3–5 μm), which required a good control of the thermal stability of the extensometers for accurate measurement. The refractory samples are constituted of a cylindrical rod (18 mm in diameter) glued to two metallic parts. The geometry is precisely adjusted thanks to a final cylindrical machining step of the global assembly. The tensile tests are carried out until rupture with a constant displacement velocity of 0.04 mm/min, with intermediate unloading at several levels of stress. To accurately determine the Young's modulus from tensile test results (E_T), the early slope of the first loading step of the stress–strain curve (between 0 and 0.6 MPa) has been evaluated. Fig. 4 illustrates an example of stress–strain curve obtained on as-cured And-LCC and the method to determine the Young's modulus.

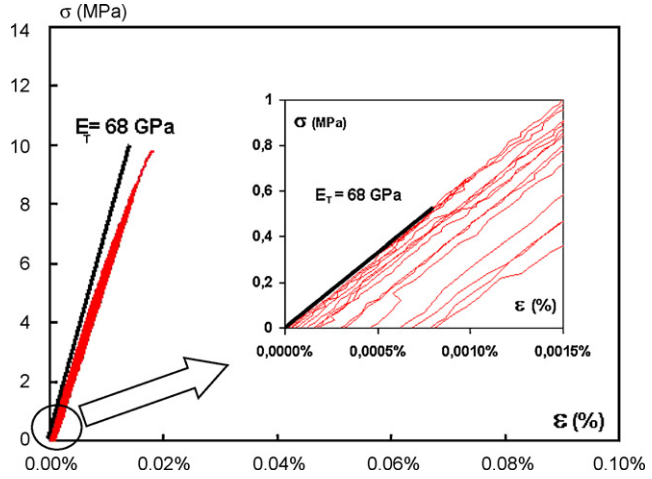

Fig. 4. Stress–strain tensile curves of as-cured And-LCC at room temperature. Magnification of the initial part of the curves that illustrates the determination of the Young's modulus E_T.

3. Results and discussion

3.1. Young's modulus values at room temperature

Table 2 presents the results of the Young's modulus measured at room temperature by the two techniques (ultrasonic measurement and tensile test) on both castables. The reported values from tensile tests correspond to an average of at least five tested samples. The two methods exhibit a good agreement within the experimental error.

In each case, a dispersion of about 6% is observed from one sample to another. In addition, the bauxite base castable, despite

Table 2
Young's modulus at room temperature for the two castables in the as-cured state.

Measurement method	And-LCC		Bau-ULCC	
	Ultrasonic (E_{us})	Tensile (E_T)	Ultrasonic (E_{us})	Tensile (E_T)
Young's modulus at room temperature (GPa)	62–70	65–75	95–100	91–100

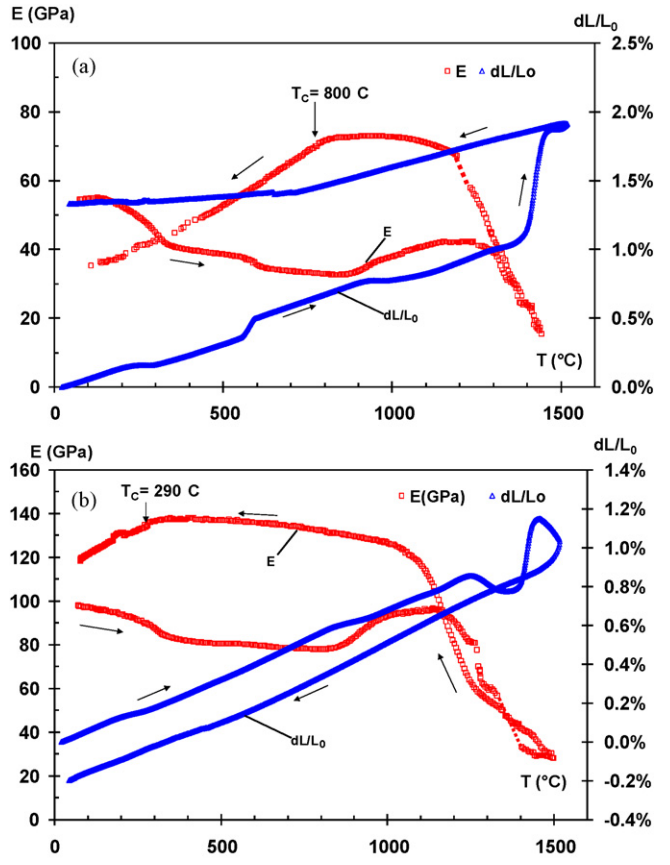

Fig. 5. Variation of the Young's modulus measured by the ultrasonic technique (E_{us}) and thermal expansion for the as-cured castables: (a) And-LCC and (b) Bau-ULCC.

its higher porosity, is stiffer than the andalusite one because of the higher intrinsic elastic properties of its aggregates.

3.2. Young's modulus evolution and thermal expansion during a first thermal cycle up to 1500 °C

Due to their compositions (natural aggregates and hydrated cementitious phases), several microstructural evolutions can take place during a first heating at high temperature. Young's modulus evolution coupled to dilatometric measurements were carried out (Fig. 5) in order to investigate these changes.When heating, between 150 and 350 °C, a strong decrease of E occurs due to the dehydration of calcium aluminates hydrates from cement. In this temperature range, a slight decrease in thermal expansion is also observed.After this stage, E slowly decreases as in a stable material up to 900 °C. Nevertheless, a slight inflection can be observed on the evolution of the Young's modulus for And-LCC around 600 °C, most probably related to the presence of quartz as impurity in andalusite aggregates (Fig. 5a). In the same temperature range, the two materials exhibit a quite linear thermal expansion up to around 900 °C where a low shrinkage is observed. This could be related to CA crystallisation which is accompanied by a small expansion which can involve a decrease of porosity in the matrix, as well as a beginning of sintering.This can explain also the increase of the modulus between 900 °C and approximately 1200 °C, enhanced by the formation of CA_2 and CAS_2 expansive phases. No particular thermal expansion effect is noted in this temperature range.

The important reduction of the Young's modulus occurring above 1200 °C for the two materials illustrates the beginning of their visco-plastic behaviour, closely related to the low viscosity of glassy phases in this temperature range. For Bau-ULCC, a shrinkage is observed between 1250 and 1400 °C (Fig. 5b). This is most probably related to sintering in the matrix. For And-LCC, the mullite formation, resulting in a strong expansion can hide this shrinkage on the dilatometric curve, due to its magnitude.

Above 1400 °C, the visco-plastic behaviour of the two materials involves a strong attenuation of the ultrasonic waves, making difficult Young's modulus measurements. Both materials exhibit a sharp thermal expansion up to 1450 °C, greater for And-LCC than for Bau-ULCC, because of the higher content of mullite formed from andalusite decomposition. The shrinkage after 1450 °C in Bau-ULCC corresponds to sintering.

For the cooling period, in both materials, Young's modulus firstly increases because of crack healing and sintering processes which have occurred at high temperature. Below 1000 °C, the materials behave like a stiff elastic ceramic, with a regular increase of the Young's modulus versus decreasing temperature. But at a given temperature T_C (different for the two castables), a

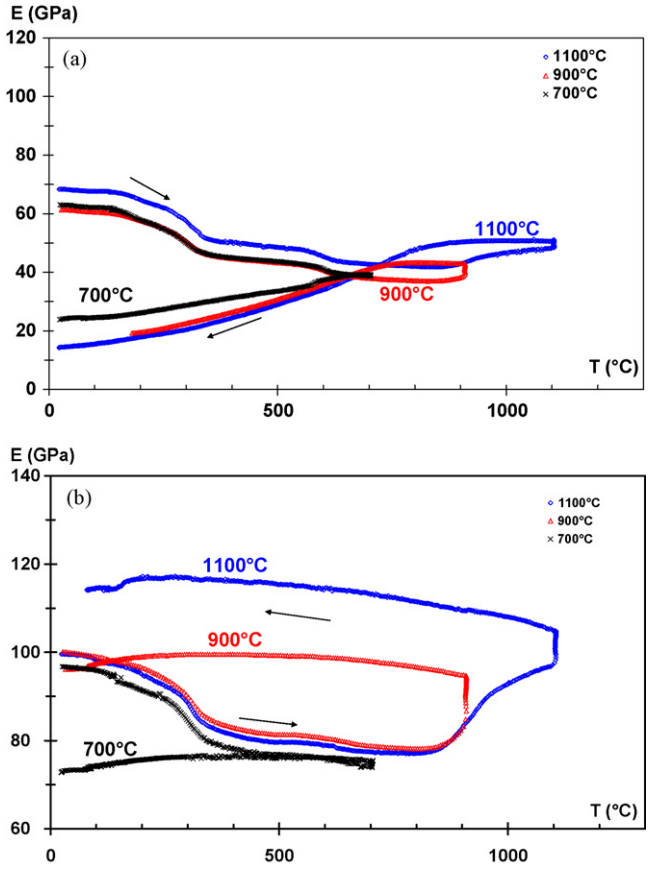

Fig. 6. Evolution of the Young's modulus measured by the ultrasonic technique (E_{us}) for both as-cured castables during different first thermal cycles: (a) And-LCC and (b) Bau-ULCC.

sudden decrease of E occurs down to room temperature. This has been attributed to damage due to thermal expansion mismatches between the different phases within the materials, in particular between aggregates and matrix. However, this phenomenon is much less marked in the case of Bau-ULCC (Fig. 5b). In the And-LCC case, the Young's modulus at the end of the thermal cycle is always lower than the one measured at the beginning where the material is in its hydrated state. Such a mechanism is also responsible of the inflexion at low temperature of the dilatometric cooling curves.[8,9]

3.3. Young's modulus evolution during thermal cycles at lower temperatures

Fig. 6 shows the evolution of the Young's modulus versus temperature for both as-cured castables during three thermal cycles carried out at different temperatures: 700, 900 and 1100 °C with a 5 h dwell.

For the two materials, the heating parts of the curves, up to the maximum temperature, are remarkably reproducible with the same parts of the curves of Fig. 5. Therefore the interpretations of the variations of Young's modulus during the increase of temperature in these ranges are confirmed.

In the case of And-LCC, similarly to the cycle carried at 1500 °C, the final value of the Young's modulus after thermal cycles is always lower than the one taken at the beginning. At 700 °C, the material can be assumed as only dehydrated and the healing of microcracks or other sintering processes have not occurred, even after 5 h at this temperature That is why after the dwell, one can observe the regular decreasing of E most probably due to crack opening in the damaged dehydrated material. For higher temperatures and especially for 1100 °C, it seems that a partial recover, especially during the temperature dwell, of the elastic properties has occurred. Indeed, at this temperature, it is most probable that a part of the recovering mechanisms previously quoted (crack healing, crystallisation of CA, sintering) has taken place. It must be noted that this temperature is too low for the formation of mullite. During cooling, a critical temperature exists below which a significant drop of E is observed, as for the material sintered at high temperature (Fig. 5a). This effect is related to the damage due to thermal expansion mismatches existing between phases within the material, as illustrated in micrographs of Fig. 7c.

In the case of the Bau-ULCC, the behaviour during the thermal cycle at 700 °C is similar to the one of And-LCC and is also attributed to the microstructure subsequent to dehydration. An increase of Young's modulus (higher than in And-LCC) is observed during the dwells at 900 and 1100 °C. This can be attributed to healing of cracks favoured by crystallisation–densification of CA and low viscosity glassy phases. Indeed, the presence of some impurities within the bauxite aggregates (TiO_2, Fe_2O_3, K_2O), involves the formation of vitreous phases promoting sintering processes at high temperature. It clearly appears that the Bau-ULCC castable is less damaged after cooling than the And-LCC one. The damage that occurs towards 300–250 °C during cooling is smaller and has been mainly identified as microcracks in the matrix (Fig. 7b and d).

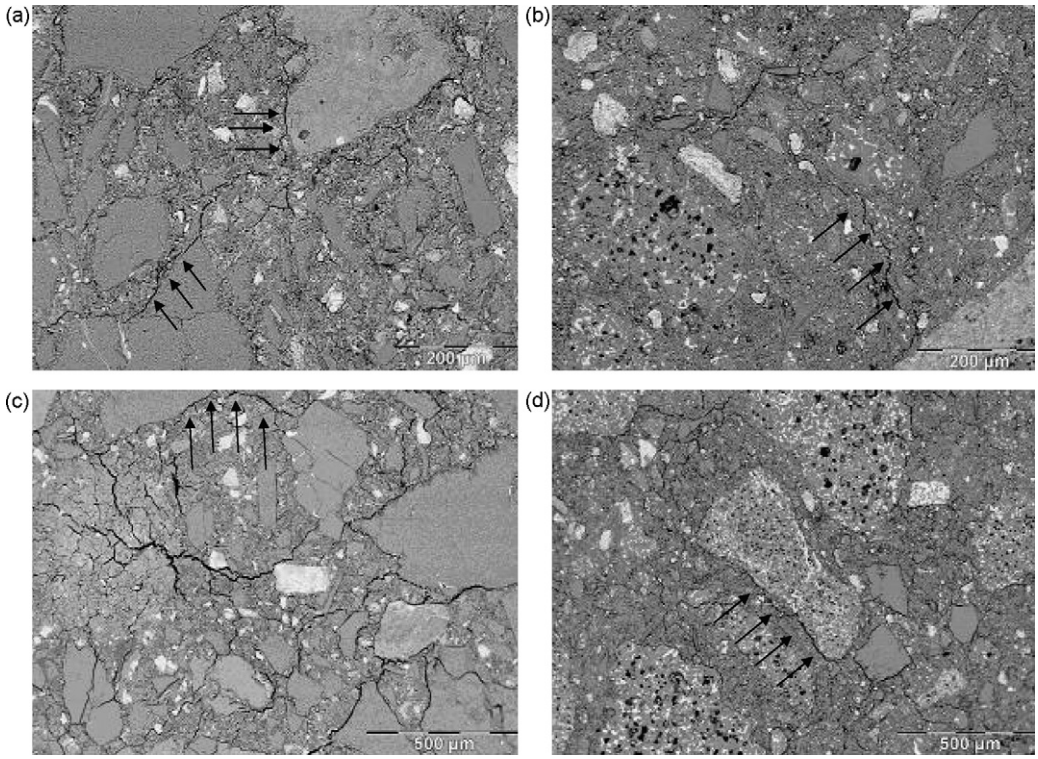

Fig. 7. Identification of damage in the two castables after thermal treatments: (a) And-LCC at 700 °C; (b) Bau-ULCC at 700 °C; (c) And-LCC at 900 °C; and (d) Bau-ULCC at 900 °C.

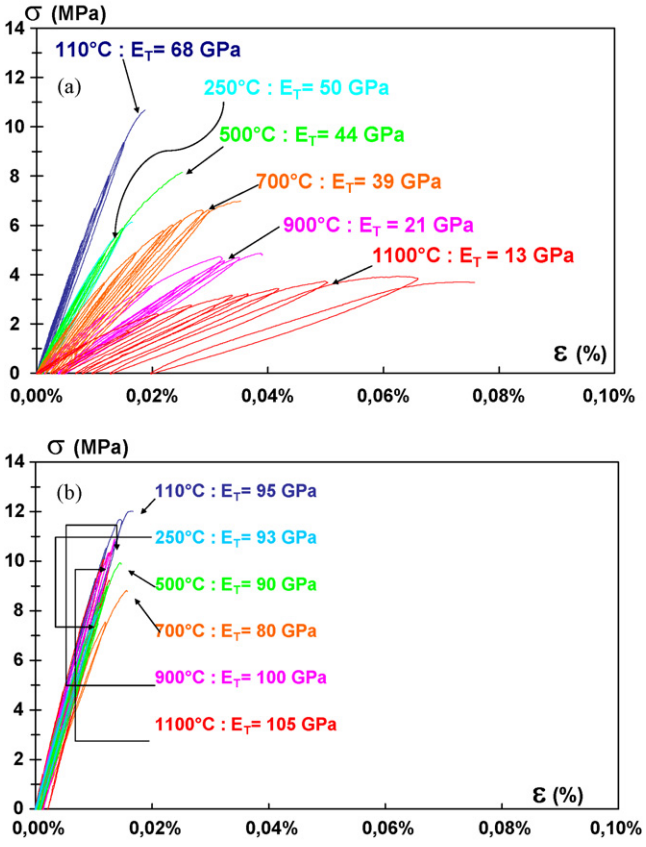

Fig. 8. Tensile behaviour at room temperature of both castables treated at different temperatures: (a) And-LCC and (b) Bau-ULCC.

3.4. Mechanical behaviour in tension after thermal treatment

Elasticity measurement highlights that the elastic properties of the materials at room temperature are strongly affected by the thermal history. The interest of performing tensile tests is to supplement these data by the determination of tensile stress–strain behaviour up to rupture, including the non-linear character linked to damage or anelastic effects in the material. Such tests were carried out at room temperature on samples which had been previously treated at various temperatures (110, 250, 500, 700, 900, 1100 °C, 5 h dwell) in order to investigate the influence of the thermal history on the mechanical behaviour of the two castables. These results are presented in Fig. 8a and b.

At the beginning of loading, the two materials present an elastic linear behaviour. The initial slope, determined between 0 and 0.6 MPa, gives the Young's modulus E_T. Table 3 compares the results of Young's modulus measured by the two different techniques (ultrasonic pulse echo and tensile tests) after treatments at 700, 900 and 1100. Again, these results enhance the good agreement between dynamic and static methods for determination of elastic modulus.

Beyond the elastic domain, the two materials exhibit a non-linear behaviour. This non-linear domain is very limited in the case of Bau-ULCC; it is very extended in the case of And-LCC. So these tensile results confirm:

Table 3
Comparison of the Young's moduli obtained at room temperature by two different techniques (ultrasonic and tensile test) for both castables previously treated at different temperatures.

Temperature of treatment (°C)	And-LCC		Bau-ULCC	
	E_{us} (GPa)	E_T (GPa)	E_{us} (GPa)	E_T (GPa)
110	62–70	65–75	95–100	91–100
250	–	49–53	–	91–96
500	–	46–50	–	86–91
700	21–25	36–43	73–75	75–89
900	14–17	18–24	94–96	95–104
1100	10–15	8–15	110–115	96–121

- the high stiffness of the bauxite based castable;
- the damaged microstructure of And-LCC castable after thermal treatments which lowers the Young's modulus and induces a highly non-linear tensile behaviour.

In the case of Bau-ULCC castable, the evolution of tensile behaviour is very limited. As found by ultrasonic measurements, the Young's modulus increases at room temperature after thermal treatments above 700 °C.

3.5. Source of damage in the And-LCC castable

The And-LCC castable presents, at the same time, a very significant reduction in Young's modulus during cooling and a non-linear behaviour in tension after thermal treatment. In heterogeneous materials, these two points often find their origin in the existence of thermal expansion mismatch between the constitutive phases.[8] Thus, a study has been carried out on the dilatometric properties of the aggregates compared to those of the matrix (granulometry lower than 200 μm) for both castables.[10] Samples of matrix were prepared by casting and samples of aggregates were prepared by pressing.

In the case of Bau-ULCC, the thermal expansion coefficient value between 900 °C and 450 °C of components of this castable are $9.0 \times 10^{-6}\,K^{-1}$ for the bauxite aggregates and $7.6 \times 10^{-6}\,K^{-1}$ for the matrix. These close values explain the limited damage observed by ultrasonic measurement during cooling.

Concerning And-LCC, Table 4 reports the average thermal expansion coefficients obtained in the range 900–450 °C for a polycrystal made of andalusite aggregates and for the matrix. The values, about $7.6 \times 10^{-6}\,K^{-1}$ for andalusite aggregates and about $7.8 \times 10^{-6}\,K^{-1}$ for the matrix, are quite close. This surprising result (obtained here on sintered polycrystalline

Table 4
Thermal expansion coefficients (α) of andalusite aggregates and matrix of the And-LCC castable between 900 and 450 °C ($10^{-6}\,K^{-1}$).

Matrix	Andalusite aggregates			
	Polycrystal	Single crystal		
		$\vec{a}$	$\vec{b}$	$\vec{c}$
7.6	7.8	12.9	9.6	3.1

andalusite aggregates) suggests that the origin of the damage observed in And-LCC could not be attributed to thermal expansion mismatch between these two parts. In contrast to bauxite aggregates, which are roughly polycrystalline (therefore isotropic particles), andalusite aggregates are highly anisotropic orthorhombic single crystals.[11] Indeed, a number of studies have recently underlined the strong anisotropy in the thermal expansion behaviour of such aggregates.[12,13] The results of these works, summarised in Table 4, highlight that, even if the thermal expansion coefficient of polycrystalline andalusite is very close to that of the matrix, very strong thermal expansion mismatch exist because of the anisotropy of single crystals. This important result explains the origin of the strong damage observed during cooling in the andalusite based castables.

3.6. Influence of thermal history

Tensile tests performed at high temperature also illustrate the strong dependence of the mechanical properties of the materials on their thermal history. In this part, the tensile behaviour has been studied at a fixed temperature of 800 °C, but corresponding to various sequences of heat treatment including two cycles up to 1200 °C.

At first, the choice of measurement conditions has been made by considering the curves $E = f(T)$ obtained from ultrasonic measurements during 20–1200 °C thermal cycles for each material (Fig. 9). Once more, the two materials exhibit a strong difference after the first heating step: And-LCC behaves like a damaged material with reproducible hysteretic $E = f(T)$ loops corresponding to crack closure–opening mechanisms (Fig. 9a); but the variation of Young's modulus of Bau-ULCC is almost regular and reversible as in a dense stable ceramic (Fig. 9b).

From these curves three points A, B, C have been chosen for tensile measurements. At point A, the materials have been dehydrated and, as presented before, the E value is rather low (about 41 GPa for And-LCC and 74 GPa for Bau-ULCC) compared to that of the initial state. At point B, the materials have been heated up to 1200 °C and cooled down to 800 °C, so the E value has been raised up to 55 GPa for And-LCC and 116 GPa for Bau-ULCC, because of stiffening mechanisms at high temperature as previously explained. At point C, the materials have been cooled down to room temperature and heated again up to 800 °C. For And-LCC, thermal expansion mismatch between andalusite aggregates and matrix leads to a highly damaged state then, to a very low value of E (26 GPa). In contrast, the curve corresponding to the second cycle is quite reversible for Bau-ULCC and the value of Young's modulus is approximately the same as at point B. This suggests that the tensile behaviour at 800 °C will be very different between the two materials. Fig. 10 shows results of tensile tests results performed at 800 °C for the three considered points: A, B and C. Additionally loading–unloading cycles have been regularly made in order to point out visco-plastic effects.

The And-LCC castable (Fig. 10a) exhibits very different tensile behaviours according to the thermal histories. The three curves exhibit a very limited linear domain. Therefore, Young's moduli derived from tensile tests are slightly lower than those found by ultrasonic measurements. The aspect of the curve at point A is very different compared to the tensile curves at room temperature (Fig. 8a): high non-linearity; increasing remanent strain after unloading, increasing area of the successive unloading–loading loops; high value of ultimate strain (about 0.07%). This is consistent with the damaged state of the dehydrated material with, additionally, a visco-plastic contribution of glassy phases at this temperature. After the first heating at 1200 °C (point B), the material is more brittle with rather poor strength and ultimate strain. Then, the tensile curve at point C shows, as predicted from ultrasonic measurements, a weak stiffness (20 GPa) but a recovering of the ability to deformation with a significant strain to rupture (about 0.04%), though a low strength (~4 MPa).

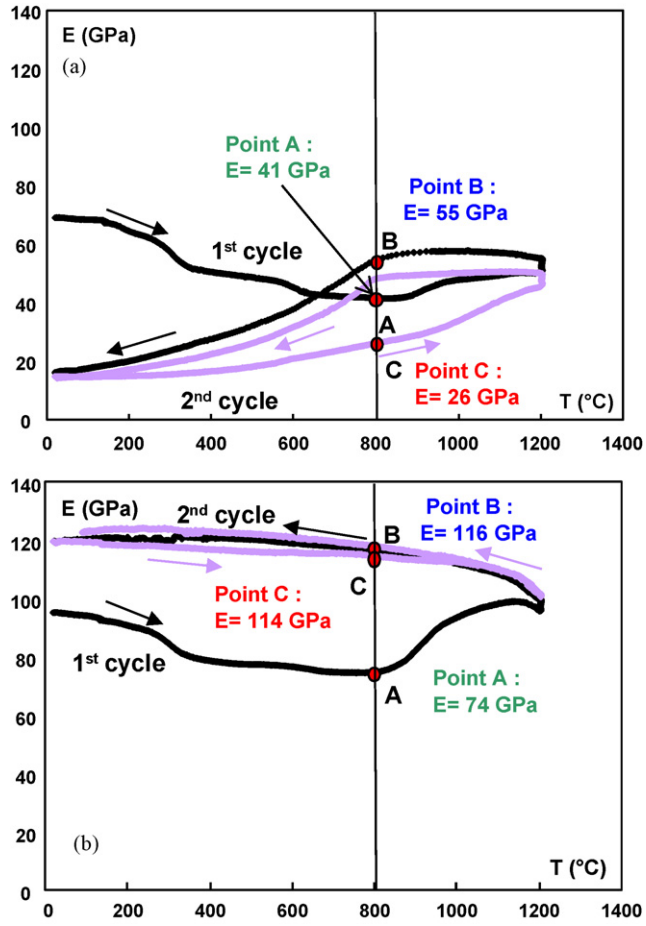

Fig. 9. Variation of Young's modulus of the castables during two successive thermal cycles up to 1200 °C (as-cured initial state): (a) And-LCC and (b) Bau-ULCC.

In a same manner, Fig. 10b shows the results of tensile tests corresponding to these three points in the case of Bau-ULCC. At point A, the curve has a similar non-linear and visco-plastic aspect as for And-LCC. It is also due to damage in the dehydrated state and the influence of glassy phases. As predicted from ultrasonic measurements, the material shows approximately the same quasi brittle behaviour (with limited remanent strains when unloading) in points B and C, close to the behaviour at room temperature (Fig. 8b).

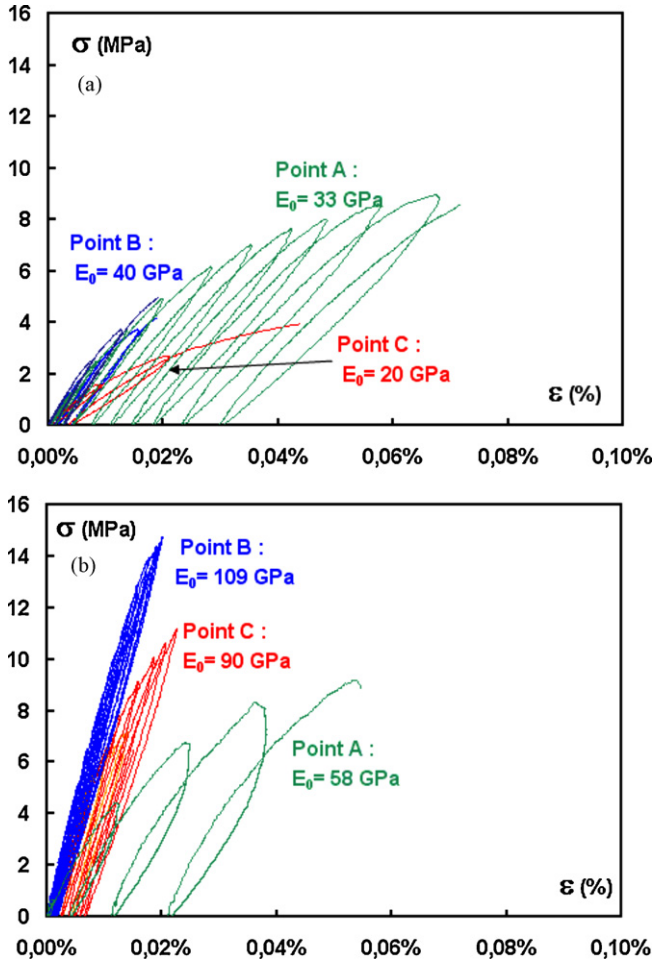

Fig. 10. Mechanical behaviour in tension for the castables subjected to different thermal histories: (a) And-LCC and (b) Bau-ULCC.

4. Conclusion

Difference of thermal expansion between the constituents of refractory materials can lead during heating and cooling, to a large microcrack network within the microstructure, which deeply affects the mechanical properties: stiffness, stress–strain curves, parameters at rupture. Considering the ultrasonic results, these microstructure effects induce typical hysteretic behaviours on elastic properties at high temperature. For a similar firing temperature, the extent of the non-linear domain, measured in tension at room temperature, for the And-LCC castable is higher than that for the Bau-ULCC one because of damage enhancement. In the case of the andalusite castable, temperature cycling reduces the maximum strength, but strongly increases the compliance and the strain to rupture, thanks to microstructure effects related to andalusite aggregate properties. The single-crystal character of those aggregates involves a very strong anisotropy of thermal expansion, which leads to large thermal expansion mismatch with the cementitious matrix. Through a good knowledge and control of such microstructural mechanisms, one can take advantage of the resulting enhancement of compliance and strain to rupture for applications with thermal shock conditions.

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