FIBRE REINFORCED REFRACTORY CONCRETE AS A TOOLING MATERIAL FOR ORGANIC MATRIX COMPOSITE PROCESSING ROUTES

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Abstract

Refractory concretes are characterised by a low tensile strength and a brittle or quasi-brittle failure. Short fibre addition is widely used to improve the mechanical behaviour of brittle materials such as concretes. The fibre effects mainly depend on the fibre material, the fibre shape and the fibre volume fraction. Besides, both the testing and firing temperatures influence the thermomechanical behaviour of reinforced refractory concretes.

The purpose of this study is to quantify and to understand the effect of a steel and/or mineral fibre reinforcement on the thermomechanical properties of a SiC based refractory concrete. Four point bending tests are carried out in order to study the thermomechanical behaviour of the composite. Three types of strengthening are considered: steel fibres, glass fibres and a mix of the two types of fibre.

Results show that fibres have benefit effects on both the pre-peak and post-peak mechanical behaviour of the refractory concrete. They are discussed by considering the material microstructural evolutions. The sensitivity to size effects is also discussed.

1 Introduction

The increase of the use of high performance organic matrix composites induces new challenges in the field of toolings. Problematic to be considered for toolings deal with the decrease of recurrent costs and of delivery time, with the increase of the tooling size and of the tooling life duration and with the environmental impact. These challenges are particularly visible in the aeronautic domain [1]. Starting from that considerations of the experience acquired in the development of toolings for the forming of metallic alloys using the superplastic forming process, Institut Clément Ader has started some studies to develop a new tooling solution, based on a Metallic Fibre Reinforced Refractory Concrete and for shaping large parts of organic matrix composites. Such a development needs to characterize and to analyse the thermomechanical behaviour of a metallic fibre reinforced SiC based refratory concrete and to consider its size effect sensitivity. The considered temperature range is the 20°C-400°C one.

Refractory concretes are unshaped materials mainly used is the metallurgy industries, in thermal power plants and in industrial furnaces. They are made of aggregates with a size ranging between 200μm and 10mm. Aggregates are linked by fine particles of an alumina rich cement [2]. Concretes are characterized by lower mechanical performances in tension, compared to compression, and by a brittle of quasi-brittle rupture mode. Their reinforcement is considered since the 1960teens in the field of civil engineering. Today, many fibre types are considered to monitor the behaviour and the characteristics of fibre reinforced concretes [3,4].

Reinforcement effects are mainly linked to the fibre material type, to the fibre shape and to the fibre volume fraction. Short fibres generally allow enhancing the strain capability of concretes by modifying microcrack initiation and propagation and by delaying macrocrack initiation. Short fibres allow enhancing the reinforced concrete toughness too. Multiple reinforcement mechanisms can be coupled by reinforcing concretes with multiple fibre types, developing hybrid reinforcements.

For refractory concretes, the firing and testing temperature highly influence their thermomechanical behaviour. Various studies have shown such an influence by establishing links between microstructural evolutions in such materials and their thermomechanical behaviour, on the one hand for unreinforced materials [5] and in the other hand for reinforced materials [6]. Works have been conducted to quantify and understand the temperature effects on the microstructural and mechanical properties of refractory concretes reinforced with metallic fibres [7] and with mineral fibres [8]. They dealed with refractory concretes with a high alumina content and their behaviour at temperatures higher than 500°C was particularly considered, mainly for tooling applications. In such fields, high mechanical properties and durability are necessary. [9]. Such toolings are often used in temperature fields where microstructural changes take place in refractory concretes with large effects on their mechanical behaviour. Understanding the effects of a fibre reinforcement is particularly helpful to be able to formulate well-adapted reinforced refractory concretes. The main objective of the present work deals with the development of a high performance refractory concrete for applications in the 20°C-400°C range, particularly in toolings for shaping organic matrix composites.

2 Materials and experiments

2.1 Materials and thermomechanical behaviour

A commercial grade of a low cement content and SiC based refractory concrete is considered. The maximum aggregate size is of 3mm. During the processing route, 7 wt.% of water are added to adapt the mix rheology and to allow the development of hydraulic bonds in the cementitious matrix. Two fibre types are taken into account. They deal on the one hand with stainless steel hooked fibres (AISI310 grade) and on the other hand on alkali-resistant glass fibres (AR glass grade). Fibre properties are detailed in table 1. Table 2 contains some details on the eight reinforced refractory concrete grades.

Properties	**Length [mm]**	**Diameter [mm]**	**Density (g/cm^3)**	**Young's modulus [GPa]**	**Tensile strength [MPa]**	**CTE [K^{-1}]**
Stainless steel fibres (AISI310)	25	0.38	7.37	190	>1400	18.5 10^{-6}
Glass fibres (AR glass)	12	0.011	2.68	775	2200	6.0 10^{-6}

Table 1. Fibre properties.

		Grade							
	Fibre type	NF	HF05	HF10	HF15	HF20	AR05	AR10	AR05-HF10
Volume fraction [%]	AISI310 Steel	0	0.5	1	1.5	2	0	0	1
	AR Glass	0	0	0	0	0	0.5	1	0.5

Table 2. Details on the eight considered grades of fibre reinforced refractory concretes.

Samples are elaborated following a conventional processing route. The initial mix of the refractory concrete constituents is mixed at the dry state with a planetary mixer. Water and fibres are then added and the mixing time is of 3 min. Samples are then elaborated by casting under vibrations. They are then maintained at room temperature during 48 hours in the moulds. Samples are then demoulded and dried at 110°C during 24 hours. Samples are finally machined in order to obtain their parallelepipedic shape (160x35x40mm^3).

Samples are then fired to stabilize the material microstructure before its characterisation. Thermal cycles have the following characteristics: heating/cooling rate 100°C/hour, isothermal dwell duration of 1 hour, isothermal dwell temperature of 250°C or of 400°C.

The materials mechanical behaviour has been characterized in 4 points bending. Tests have been performed on a servo-hydraulic MTS 810 testing machine, equipped with a 1600°C radiative furnace. Distances between lower and upper loading points are respectively 125mm and 45mm. Tests are monitored at a constant hydraulic actuator displacement speed of 0,02mm/min. A deflection measurement is performed at the middle point of the lower surface by the way of an LVDT sensor coupled to the sample with an alumina rod. To minimize the effects of compression phenomena under the contact points of the bending tests [10], behaviour curves are only plotted for stress levels greater than 2 MPa.
Three samples have been tested for each grade and for each (firing temperature, testing temperature) couple. The following designation has been retained to identify the firing and the testing conditions of each sample: FxTy for a sample fired at x°C and tested at y°C (for example F400T250 sample fired at 400°C and tested at 250°C).

2.2 Size effect sensitivity

The size effect sensitivity of a the SiC based materials of the study has been characterised by the way of 3 points bending tests performed on unnotched beams. Five sample sizes have been considered for the unreinforced refractory concrete and six for the reinforced one. The sample height and the distance between lower loading points follow a homothetic evolution between each size and the sample width is kept constant and equal to 100mm (Figure 1 and Table 3). For the reinforced grade, the volume fraction of hooked end AISI310 fibres is of 1.5vol%. Samples have been fired at 250°C.

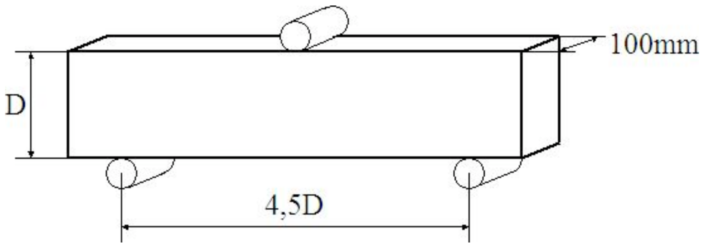

Figure 1. Three points bending geometry of samples considered to study the size effect sensitivity.

		D1	D2	D3	D4	D5	D6
Number of tests	**NF**	12	9	6	4	2	0
	HF15	12	7	6	4	2	2
Width	**mm**	100	100	100	100	100	100
Height (D)	**mm**	12,50	25	50	100	200	400
Distance between loading points	**mm**	56,25	112,50	225	450	900	1800

Table 3. Unnotched sample size for 3 points bending tests (size effect sensitivity).

Tests have been performed on a 100kN tension/torsion hydraulic machine for the D1 to D4 samples sizes and on a 150kN platform for D5 and D6 sample sizes. The same loading contacts have been considered for all sample sizes (D1 to D6). Tests have been performed at a constant hydraulic actuator speed 0.08mm.s^{-1}.

3 Results

Firstly, figure 2a gives the terms that have been retained to describe the mechanical behaviour and properties in bending of the studied materials. These terms are classically considered to describe the behaviour of fibre reinforced concrete for civil engineering, for example by Naaman et al [11].

The mechanical behaviour of a refractory concrete can generally be divided in two domains. Before macrocracking, the behaviour is mainly linear elastic (domain AB), even if diffuse damage already exists in the material. After point B, the behaviour moves to a non-linear one, due to the progressive increase of the diffuse damage level. At point C, initiation of one or of several macrocracks occurs. For unreinforced materials, such a macrocrack propagates rapidly and leads to a brittle or quasi-brittle rupture. For reinforced materials, a loading transfer can occur from the matrix to the fibres and can lead to a more or less softening behaviour. In some cases a hardening behaviour is observed and stress levels can increase after the stress initiation level, up to a maximal stress level that corresponds to the rupture stress of the material.

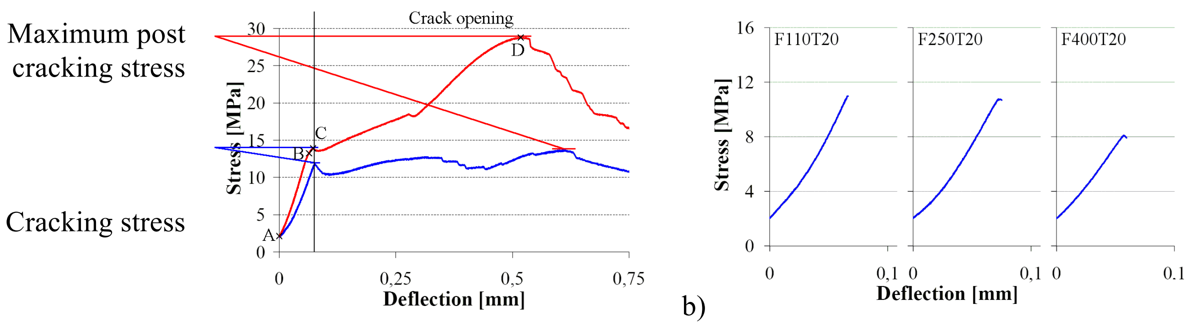

Figure 2. Four points bending behaviour curves (stress vs. deflection): (a) for HF05 (blue) and HF20 (red) grades dried at 110°C and tested at room temperature, (b) for the NF HF20 grade (unreinforced material) fired at different temperature and tested at room temperature.

3.1 Unreinforced concrete.

Figure 1(b) shows the typical room temperature mechanical behaviour of the unreinforced concrete for the three considered firing temperatures. First, the unreinforced concrete rupture is brittle and becomes quasi-brittle after firing. Secondly, the cracking stress decreases as the firing temperature increases. Thus, the cracking stress of samples fired at 250°C and tested at room temperature is 88% higher than for samples fired at 110°C and 82% higher than for samples fired at 400°C. The cracking stress of samples tested at the firing temperature is 92% higher than for samples tested at room temperature for 250°C and 118% higher for 400°C.

3.2 Steel fibre reinforced concrete.

Figures 3 and 4 show typical behaviour curves of steel fibre reinforced concretes in bending. It allows observing the changes due to the firing and testing temperatures and to the fibre volume fraction. As shown in previous studies, for all testing conditions, a fibre volume fraction increase leads to an increase of the maximum post cracking stress. Firing reduces both the crack stress and the maximum post cracking stress and increases the nonlinear behaviour of the composite. [5]

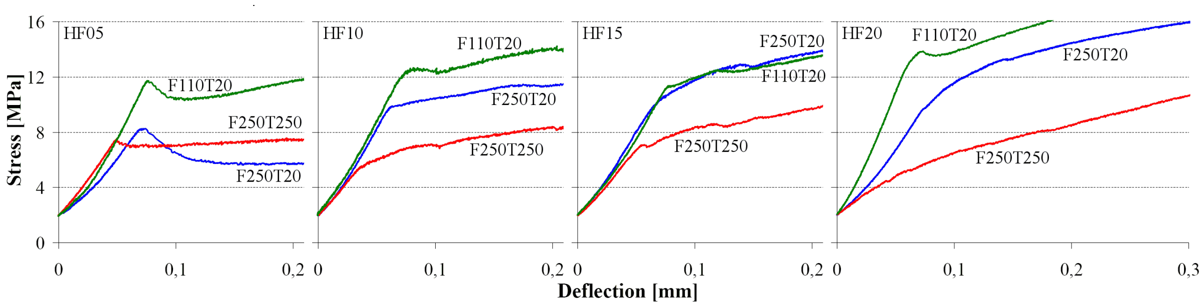

Figure 3. Stress-deflection curves in four points bending of steel fibre reinforced concretes fired at 110°C and tested at 20°C (green), fired at 250°C and tested at room temperature (blue), fired at 250°C and tested at 250°C (red) for the HF05, HF10, HF15 and HF20 grades

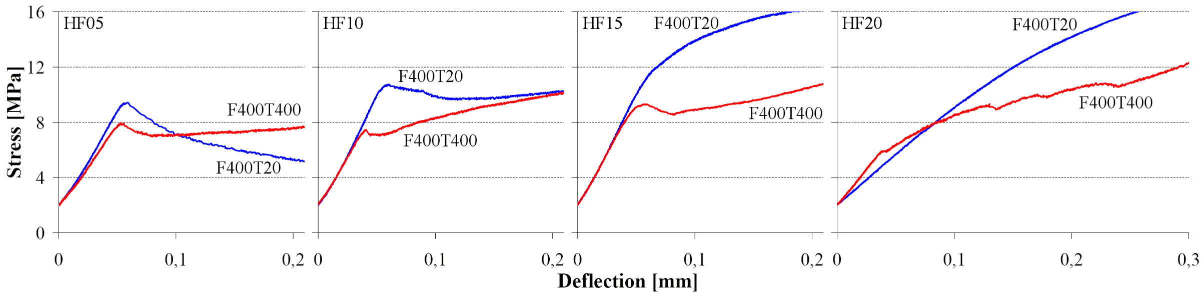

Figure 4. Stress-deflection curves in four points bending of steel fibre reinforced concretes fired at 400°C and tested at room temperature (blue) and fired at 400°C and tested at 400°C (red) for the HF05, HF10, HF15 and HF20 grades

The mechanical behaviour of the composite at room temperature changes both with the volume fraction and the firing temperature. For the fired HF05 samples, a most important stress decrease is observed after cracking. A similar behaviour characterizes the HF10 material after firing at 400°C. This decrease is partially reduced by testing samples at the firing temperature. The fired HF20 samples, as the H15 ones fired at 400°C, are characterized by a nonlinear behaviour and by a multicracking damage process. Indeed, multiple macrocracks are observed on samples at the end of the bending tests.

Tested at the firing temperature, the cracking stress drops and the pre-peak non-linear behaviour is reduced compared to the behaviour of fired samples tested at room temperature.

3.3 Glass fibre reinforced concrete.

The glass fibre reinforced concrete is characterized by a quasi-brittle failure and a softening behaviour after cracking (Figure 5). The composite softening behaviour increases with the glass fibre volume fraction.

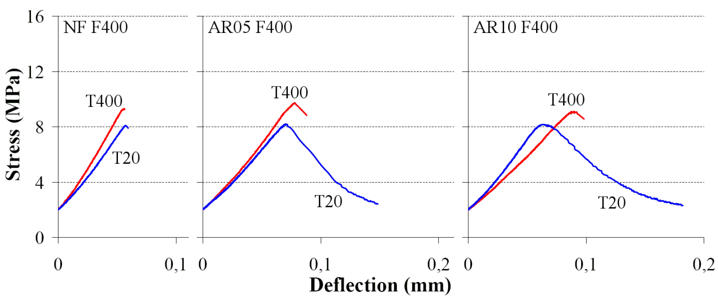

Figure 5. Stress-deflection curves in four points bending for NF, AR05 and AR10 grades fired at 400°C and tested at room temperature.

Besides, reheating the material at its firing temperature reduces the post cracking fibre effect and leads to an increase of the cracking stress. If the non-linear behaviour of the glass fibre reinforced concrete reduces when tested at the firing temperature, it still increases when the glass fibre volume fraction increases.

3.4 Hybrid reinforced concrete.

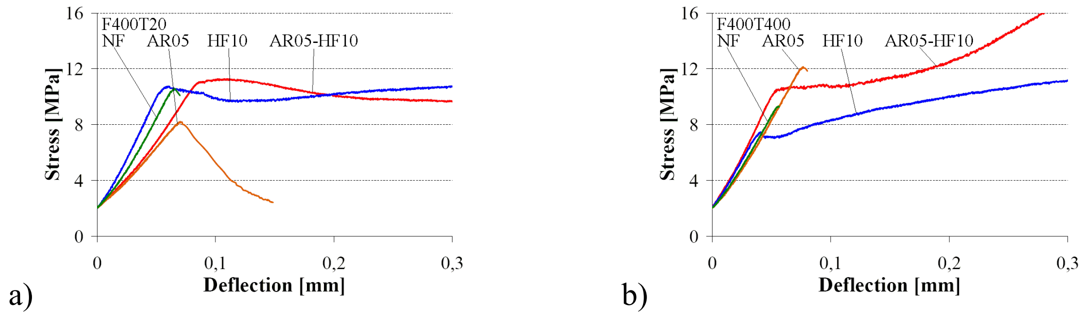

Figure 6. Stress-deflection curves in four points bending of NF (green), HF10 (blue), AR05 (orange) and AR05-HF10 (red) grades fired at 400°C and tested at room temperature (a) and at 400°C (b)

Figure 6 allows observing the typical mechanical behaviour of different samples fired at 400°C and tested both at room (a) and at the firing temperature (b). The hybrid reinforced concrete combines the steel and glass fibres strengthening mechanisms. Thus, after cracking, no stress decrease is observed in the AR05-HF10 sample behaviour, compared to the HF10 one tested at the firing temperature If the post crack stress of AR05-HF10 samples is close to the HF10 one, the cracking stress is 1,65 times higher.

3.5 Size effect sensitivity

For the two sample series NF and HF15, results of the size effect sensitivity study are detailed in table 4. Evolutions of the maximal stress as a function of the sample size and behaviour curves are given in figures 7 and 8.

			D1	D2	D3	D4	D5	D6
HF15	**Mean value**	**MPa**	22,23	20,36	15,60	14,90	18,60	15,85
	Std deviation	**MPa**	1,76	1,65	1,88	0,83	0,71	0,64
NF	**Mean value**	**MPa**	20,17	19,39	10,55	10,18	7,15	
	Std deviation	**MPa**	1,72	1,81	0,27	0,35	0,49	

Table 4. Results of 3 points bending tests for unnotched samples of HF15 and NF grades fired 250°C and tested at 20°C.

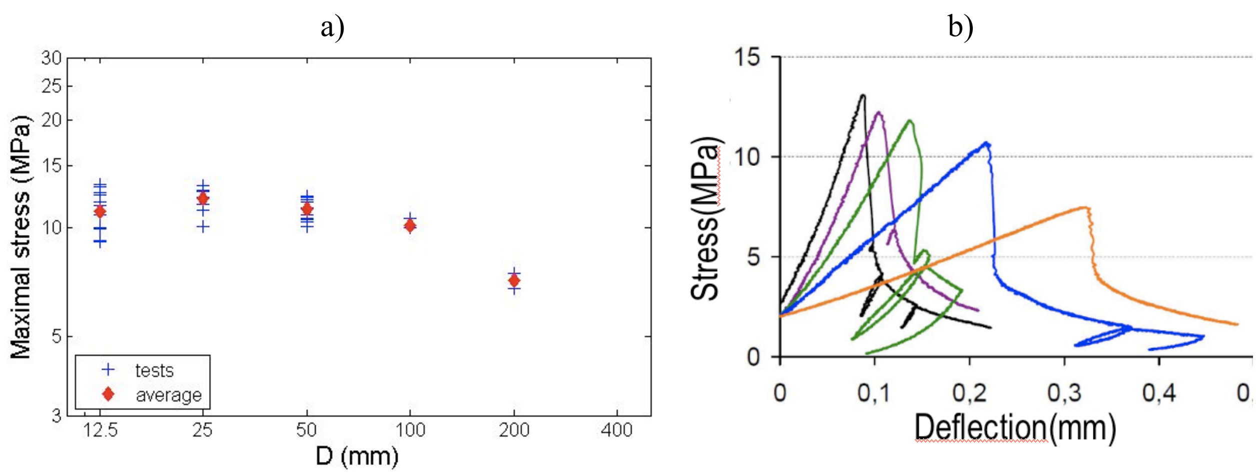

Figure 7. Results of size effect sensitivity results for an unreinforced refractory concrete tested in 3 points bending, (a) Maximal stress (b) Behaviour curves.

The unreinforced concrete exhibits a high size sensitivity effect since the maximal stress level for D5 beams is only equal to 35% of the maximal stress level of D1 beams. This decrease is confirmed by behaviour curves obtained from the 5 beam sizes (figure 7(b)). These graph show that the material behaviour still remains brittle, in the considered size range.

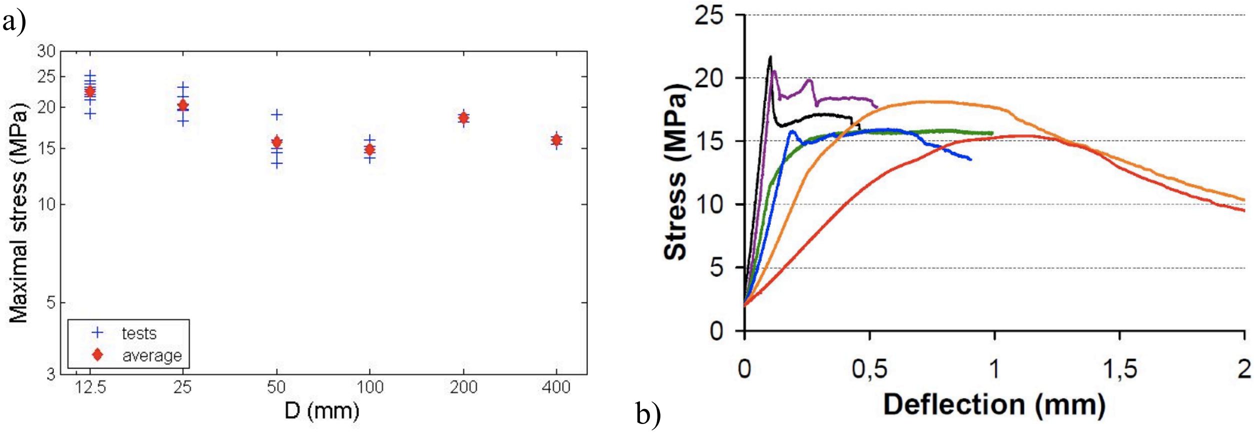

Figure 8. Results of size effect sensitivity results for a metallic fibre reinforced refractory concrete tested in 3 points bending (Vf=1,5%), (a) Maximal stress (b) Behaviour curves.

Results of figure 8 show that the metallic fibre reinforced concrete developed and characterised in the present work is not really sensible to size effect, in the considered beam size range. This constitutes an important and interesting result particularly when considering the design and the sizing of structures (for example toolings for shaping organic matrix composites) based on this material. Behaviour curves of figure 8b show that a change occurs for the largest sizes (i.e. above the D4 size). This change deal on the one hand on the pre-peak behaviour and on the other hand with the post-peak behaviour too. The origin of such a change still remains to be identified.

4 Discussion.

4.1 Cracking stress – Peak stress.

The effect of temperature on the mechanical behaviour of the unreinforced refractory concrete arises from diffuse damage. Indeed, thermally induced damage increases microcracking in bending and leads to a quasi-brittle failure. Such a damage is mainly due to dehydration phenomena which take place in the 110°C-300°C temperature range and to the thermal expansion mismatch of the concrete constituents. [5]

Reheating the material partially reduces the thermally induced damage and closes some microcracks. Thus the cracking stress of NF and AR grades increases when tested at their firing temperature. Besides, the width of the nonlinear behaviour between points B and C ('Figure 1(a)') decreases because of the decrease of diffuse damage into the concrete.

Figure 9 allows observing the effect of steel fibre additions on the cracking stress. Introducing steel fibres leads to an increase of the thermally induced damage and to a decrease of the cracking stress. The nonlinear behaviour increases. For a 1% to 1.5% volume fraction of steel fibres, the crack stress increases. When heating the composite, the expansion of steel fibres generates cracks into the concrete matrix, at the fibre periphery. This damage area around fibre grows as the temperature rises. The fibre induced damage results in a non-linear behaviour for high fibre volume fractions and in the inability to determine the crack stress because of the development of multiple macrocracks. When increasing the fibre content, fibres are close enough to have their damage zone to overlap each other's. Thus, there is a critical steel fibre volume fraction and this critical value decreases as the firing temperature increases. For this study, the critical steel fibre volume fraction is in the 1.5vol% to 2vol% range for a firing temperature of 250°C and in the 1vol% to 1,5vol% for a firing temperature of 400°C.

Reheating the reinforced material reduces the thermally induced damage but, as shown in Figure 9(b), the cracking stress decreases. Due to the fibre expansion, internal stresses are developed in the material and leads to a decrease of the microcrack propagation. Samples tested at the firing temperature show a less nonlinear behaviour.

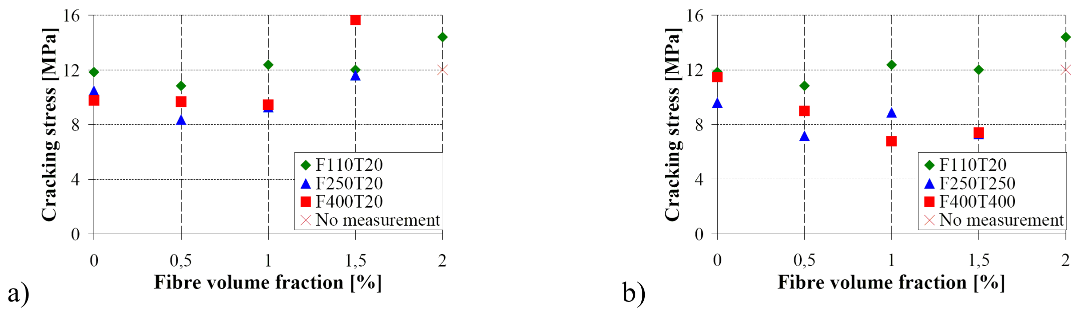

Figure 9. Cracking stress in four points bending of steel fibre reinforced concretes fired at 110°C (green points), 250°C (blue points) and 400°C (red points) and tested at room temperature (a), and at the firing temperature (b)

4.2 Post-peak behaviour

4.2 1 Metallic fibre reinforced refractory concrete

Temperature exposure induces changes in the concrete microstructure and induces diffuse damage. Besides, previous studies have shown that debonding takes place at the steel fibre/concrete interfaces, when the composite is fired. Fibre pull-out being the main source of energy dissipation after cracking (point C (‘Figure 1(a)’), the fibre/matrix debonding leads to a decrease of the maximum post cracking stress [12]. Nevertheless, the hooked end geometry allows fibres bearing the load.

The fibre/matrix debonding is a three steps process:

1- During heating, fibres and matrix expand. Fibres having the highest expansion coefficient, the matrix at the periphery of the fibres is stressed and could be damaged.

2- During cooling, the large contraction of fibres leads to a debonding at fibre/matrix interfaces and to a gap between the fibres and the concrete. Near to interfaces, a matrix layer is damaged by microcracks that are mainly perpendicular to interfaces. The microcrack length increases when the firing temperature increases.

3- During reheating, fibres expand again and the contact with the matrix is restored. Finally when temperature reaches the firing one, a fretting pressure has developed at the fibre/matrix interfaces.

The last step mainly explains differences observed when comparing Figure 10(a)’ and Figure 10(b). Thus the fibre/matrix cohesion recovery leads to an increase of the maximum post cracking stress.

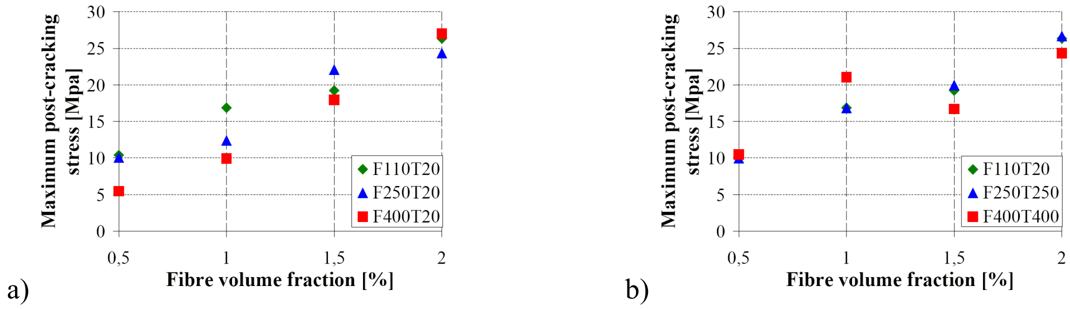

Figure 10. Maximum post cracking stress s in four points bending of steel fibre reinforced concretes fired at 110°C (green), 250°C (blue) and 400°C (red) and tested at room temperature (a), and at the firing temperature

4.2.2 Glass fibre reinforced concrete.

Previous studies have shown the effect of a mineral strengthening on the thermomechanical behaviour of an andalusite based refractory concrete [8]. A significant effect of a glass fibre introduction was particularly observed on the pre-cracking behaviour of this highly damaged concrete after firing. Short fibres interact with the existing microcracks that have been induced by firing and influence their propagation. Thus, the macrocrack initiation phenomenon is delayed. Besides, the fibres slow down the macrocrack propagation and reduce the concrete brittleness.

Because reheating the concrete partially reduces the damage, the glass fibre effect is less marked when the sample is tested at the firing temperature.

4.2.3 Hybrid reinforced concrete.

The purpose of hybrid strengthening is to combine the benefit effects of different fibre strengthening. Results show that such a combination can be obtained for example by mixing steel and glass fibres.

5 Conclusion

Results of this study allows to obtain a better understanding of the metallic fibre reinforcement effects on the mechanical behaviour of a SiC based refractory concrete, up to 400°C. Two fibre types have been considered: short stainless steel (AISI310) fibres and short AR glass fibres. Each fibre reinforcement has a specific effect on the refractory concrete behaviour. In such a material reinforcement effects depends on the fibre type, on the fibre volume fraction, on the material thermal history and on the considered testing temperature. Combining metallic and glass fibres reinforcements allows cumulating the positive effects of each fibre type. Results have been discussed by considering bending behaviour curves and the microstructural changes that take place in such heterogeneous materials. Furthermore, the study of the size effect sensitivity has shown that the unreinforced refractory concrete is characterised by an important size effect sensitivity whereas the metallic fibre reinforced refractory concrete does not exhibit any size effect sensitivity, for the considered sample sizes. Those are important and interesting results for these materials that have shown their ability to be used as tooling materials for shaping organic matrix samples particularly because of their non-brittle behaviour combined with a low CTE.

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