**Effect of elevated temperature on the mechanical properties and microstructure
of heavy-weight magnetite concrete with steel fibers**

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23 ABSTRACT

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25 In this study, the results of the physical and mechanical characterization of a high-density
26 structural concrete for radiation shielding subjected to different intensities of thermal
27 shock are analysed. The concrete has been designed using high-density natural
28 aggregates and steel reinforcing fibers, according to structural concrete requirements.
29 The damage caused by high temperatures to concrete has been analyzed by residual
30 physical, mechanical and microstructural characterization after thermal shock exposure.
31 The analysis of the results allows us to determine the behavior and verify the integrity of
32 the shielding concrete over the different temperature ranges.

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35 1 **Introduction**

36 At present, there are about 450 nuclear power reactors in the world and about 50 are
37 under construction. In addition, more than 150 nuclear power reactors have been or are
38 being decommissioned. At the same time, the emergence of new industrial processes
39 and the utilization of new radiotherapeutic treatments has led to continuous research in
40 the development of materials for ionizing radiation shielding.

41 Gamma radiation shielding depends exponentially on the thickness and electron density
42 of the absorber material and the incident photon energy. The use of high-density
43 structural concrete (HDSC) is suitable when there are no limitations in space and weight
44 per plant [1].

45 Several authors have analyzed the factors influencing the radiation shielding for different
46 concrete densities [2–4] and for the incorporation of some typical heavy natural
47 aggregates such as barite, magnetite, goethite, serpentine, hematite, ilmenite or limonite
48 [5–8] . The use of aggregates of a different nature such as galena [9], synthetic
49 aggregates [10], recycled coarse aggregates [11] and steel slags [12] in HDSC can also
50 be found. In general, the influence on radiation shielding of the type of aggregate is
51 greater than its quantity [13]. Magnetite-based concretes present higher attenuation
52 coefficients than conventional structural concretes [14,15] and iron-based concrete
53 shields radiation more efficiently and presents a greater mechanical performance than
54 classic barite-based radiological concretes [16]. However, strength and attenuation are
55 not directly related [17].

56 To achieve high densities, some authors have analyzed nonstructural ferrous additions
57 such as iron punchings [18,19] or electric arc furnace slags [12]. These additions
58 considerably improve concrete attenuation coefficients [20]. On the other hand,
59 increasing the density of concrete increases the production of secondary gamma
60 radiation, which is so dangerous to health [21].

There are studies on the use of high-density concrete for radiotherapy rooms as substitutes for lead and steel plates. These studies [22–25] analyze analytical capture models for installation design, which are conservative estimators for capture calculations, successfully verifying their feasibility.

When concrete is exposed to temperatures between 30 and 120 °C, a loss of free water and a part of the hydration water is observed. At 120 °C, all unbound water is lost. The water loss phenomena reduce the neutron attenuation of the radiation-shielding concrete [26,27]. In the temperature interval of 180-300 °C, the first stages of dehydration of the C-S-H gel begin due to the loss of bound water [28]. A severe loss in strength can be observed within the 400-800 °C range due to the dehydration of C-S-H gel, losing its cementing properties [29] due to the degradation of calcium hydroxide in the cement paste between 400-500 °C [30]. Other authors have reported a loss in neutron shielding capacity of 10 % at 500 °C [31]. Thermal stresses result in micro-cracks due to the different thermal coefficients of the cement paste (shrinkage) and aggregates (expansion), increasing swelling, porosity and modifying the mechanical properties [32–34]. Horszczaruk [35] tested concretes with magnetite and silica fume, verifying an increase in the compressive strength between 300 °C and 450 °C, and a strong reduction from 600 °C, exhibiting a loss of 70% at 800 °C. High temperatures can be reached in this type of installations because of both radiation effects [36] and exceptionally fire exposure [37], so concrete can suffer thermal shock effects.

Variables such as heating rate, peak temperature and time of exposure are the most influential factors on residual mechanical properties [38]. At any elevated temperature, there is a change of mass due to the loss of free, adsorbed and bound water. Some authors recommend the use of coatings on the concrete as protection against the physical damage caused by heat and to prevent induced water evaporation [27].

Spalling [39] is a phenomenon that can occur with moisture higher than 3-4%wt. and temperatures above 370°C in standard structural concretes. This temperature may be much lower for high-density concretes, due to their lower permeability and greater

moisture by volume. Other variables such as the rate of heating, the concrete form or stresses also affect this phenomenon [40].

The addition of steel fibers has been tested previously [41–43]. It has a negative effect on spalling, since it produces more stable propagation of cracks, but also a positive one as the fibers' tensile reinforcement helps to overcome vapor pressure [44]. Other authors attribute an improvement in spalling-prevention behavior due to the creation of discrete bubbles during the mixing process or to the poor interfacial adhesion between steel fibers and concrete [45]. Steel also improves the mechanical behavior and fire resistance for both medium and high-strength concrete [46,47] and for high-density concrete, decreasing the loss of resistance after exposure to high temperatures [48]. Rapid cooling treatments with water also cause thermal shocks that produce losses in residual resistances [49], a fact to be kept in mind when extinguishing fires.

In this study, the behavior of a high-density concrete, designed for radiation shielding, is characterized. The concrete is prepared using magnetite as fine and coarse aggregate and reinforced with steel fibers. It is exposed to temperature gradients ranging between 100°C and 1200°C. The characterization of concrete at such high temperatures responds to the current demand for knowledge about the behaviour under severe thermal conditions, due to exposure to fire or new fourth generation reactors (e.g., very-high-temperature reactors) that can reach core-exit temperatures of about 1000 °C.

The different behavioral temperature ranges are analyzed and compared with the residual compressive strength in order to propose operating temperatures for radiation-shielding concretes and determine the thermal intervals of risks or damage. Moreover, microstructural analysis will enable micro-scale effects to be related to the mechanical behavior of the concrete.

2 Experimental program

The concrete developed was designed by the Fuller method using magnetite aggregates, Portland cement, steel fibers and a superplasticizer additive, with a 0.59 water-cement

ratio. The Portland cement used is CEM I 52.5N according to the European standard, with a Blaine specific surface area of 444.7 m²/kg determined by EN 196-6: 2010. The chemical composition determined by X-Ray fluorescence (XRF) is shown in Table 1.

Table 1. Chemical composition by XRF of the cement.

Cement	CaO	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	SO ₃	K ₂ O	MgO	TiO ₂	C
CEM I 52,5 N/SR	69.6	18.65	3.15	2.66	3.22	0.54	1.17	0.17	0.47

The aggregate used is magnetite >90% purity according to the supplier, with a grading obtained by EN 933 - 1: 2012 (Fig. 1). The fine aggregate (AF) density is 5.16 g/cm³, while the mid (AM) and coarse aggregate (AC) is 4.76 g/cm³, determined according to EN 1097-6: 2014. Iron content is around 43%wt., determined by Energy Dispersive X-ray spectroscopy (EDX). AF fraction was discarded because AM fraction had a lot of fine content, enough to design an adequate arid skeleton as can be seen in Fig. 1

Fig. 1. Magnetite aggregate grading.

In addition, the scanning electron microscope (SEM) allows the morphological distribution of the different phases to be obtained Fig. 2). The micrograph shows the iron oxide phase (phase B) in a whitish color and the aluminosilicate phase in a dark color (phase A). In addition, the chemical composition of the aggregate is shown in Table 2.

Fig. 2. Microstructure of the magnetite aggregate by SEM.

Table 2. Aggregate chemical composition.

Element	Mg	Al	Si	K	Fe
Unit	%wt.	%wt.	%wt.	%wt.	%wt.
Phase A	0.98	12.77	18.45	8.76	7.44
Phase B	-	-	0.63	-	71.09

The steel fibers used (SF) have a short, cylindrical shape and a smooth surface. Fig. 3 shows the fiber surface obtained by SEM. The density of the fiber is 7.8 g/cm^3 , the average diameter and length are of $250 \text{ }\mu\text{m}$ and 12.5 mm respectively. Table 3 shows the chemical composition of the SF obtained by EDX. The results show that the surface is coated by a 70/30 ([Cu]/[Zn]) brass alloy and the fiber core is 100% steel. This type of coating should improve the anticorrosion behavior.

Fig. 3. Steel fiber surface.

Table 3. Chemical composition of fibers.

Element	Fe	Cu	Zn
Unit	%wt.	%wt.	%wt.
Coating	59.33	28.08	12.59
Interior	100	-	-

The mix proportion used is shown in Table 4. The superplasticizer additive (SP) proportion corresponds to 1.3 %wt. of cement.

Table 4. Mix proportions.

Component	Mix proportions [kg/m ³]	Volume [l]
CEM I 52.5N	300	100
Water	176	176
AM	2667	560
AC	1000	210
SP	4	4
Fibers	40	5
TOTAL	4,187	1,000

The mass contribution of the AM and AC is significant, due to its high density. The HDSC has been designed to obtain a slump of 15 cm, determined according to EN 12350 - 2:

2009. The test specimens were unmoulded 24h after their manufacture and were cured for 28 days at a constant temperature and humidity of 20 ± 2 °C and 95% respectively, in a humidity chamber.

2.1. Physical properties

For the physical characterization of the concrete, 12 cylindrical specimens of 100 mm height and 75 mm radius have been used. The water absorption coefficient, open porosity and specific gravity were determined. To analyse the effect of low temperatures after curing, simulating operation cycles, 6 specimens were subjected to 100 °C and 6 to 300 °C for 24 h before testing. The bulk specific gravity, apparent specific gravity and bulk saturated surface dry (SSD) specific gravity were determined by EN 12390-7: 2009 and the water absorption coefficient and open porosity was determined by UNE 83980:2014.

The bulk specific gravity is obtained by the dry weight/apparent volume ratio. The apparent specific gravity is obtained as the ratio between the dry weight and the apparent volume. Bulk SSD specific gravity is the ratio between saturated surface dry mass and the apparent volume.

Open porosity and water absorption coefficient were determined by the volume of open pores and dry weight ratio. High density aggregates with the same open porosity volume show lower absorption coefficients. In this way, the porosity is a more suitable parameter to compare these aggregates with the natural ones.

2.2. Mechanical properties

For the determination of the compressive strength, 5 standard 28-day-aged cubic test specimens of 100 mm were used, tested in a universal servo-hydraulic press, of 2000 kN capacity, at 5 kN/s according to EN 12390 – 3:2009.

For the flexural tests, 10 prismatic test specimens 100x100 mm section and 400 mm length were used, both with and without fibers. The 28-day-aged specimens were

subjected to three-point bending tests, according to EN 12390 - 5: 2009 at 109 N/s loading rate.

To obtain the elastic modulus, 4 standardized 28-day-aged cylindrical specimens of 300 mm in height, 75 mm in radius, capped with sulphur on their upper faces and fitted with 120 mm long and $120 \pm 0.5 \Omega$ strain gauges were used. The test was performed according to EN 12390 - 13: 2014 for the determination of the secant compression elastic modulus, using method B, applying 4 cycles with an initial lower load level of 8.8 kN up to an upper load level of 243 kN with a load/unload speed of 8.8 kN/s.

2.3. Thermal gradients

The characterization of the concrete behavior under thermal gradients was done by means of the ultrasonic pulse velocity test and the residual compressive strength determination. In addition, a visual analysis was carried out. 18 standardized 28-day-aged cubic specimens of 100 mm were dried at 100 °C in an oven until constant weight, in order to avoid spalling, and after that subjected to one of the established temperature gradients (300° C - 500 °C – 700 °C - 1000°C - 1200°C $\pm$ 5°C). The heating process is carried out in a Hobersal HD-230 3.8 kW muffle furnace with digital automatic pyrometer, which starts at room temperature (20 ± 5 °C) and ends when the set point temperature is reached. After 30 min at the maximal temperature, the specimen is tempered inside the oven by natural convection to room temperature. The temperature vs. time curve inside the muffle furnace and the temperature-time curve of concrete structures exposed to fire, according to standard 193 ISO 834 for fire testing, are shown in Fig. 4. The heating rate used induces in the specimen thermal gradients and, consequently, thermal stresses that lead to further damage causing a structural effect, in addition to the damage caused by physico-chemical processes. It is recommended, to heat the specimens sufficiently slowly, typically ≤ 0.5 -1°C/min, for specimens whose maximal transverse dimension is about 100-150 mm, according to RILEM TC 129-MHT [50], in order not to introduce significant thermal gradients. Low heating rates (of the order of 1 °C /min), like those used in other studies [33], prevent the occurrence of internal stresses that may

prematurely damage the material, in our case aggressive heating patterns led to occasional accidents. Similar heating patterns have been used for concrete in other research [29,44,51]. After heating, the specimen is subjected to the ultrasonic pulse test to determine its velocity in direct transmission following EN 12504 - 4: 2006. Immediately afterwards, the test pieces were subjected to a 5 kN/s speed uniaxial compression test according to EN 12390 - 3: 2009.

Fig. 4. Experimental and ISO 834 heating patterns.

2.4. Scanning electron microscopy analysis

Six 1 cm³ cube samples were prepared with their analysis face polished and containing a representative fraction of both the aggregates and the cement matrix. The samples were dried at 105 °C and were analyzed in the SEM before and after exposure to elevated temperatures. The exposure time was scaled to the sample size, and a single specimen was not subjected to all temperatures to avoid thermal stress. The effects produced on the concrete were analyzed by comparison. The samples were not sputtered in order to increase their superficial conductive properties so as to avoid altering the analysis surface and due to the melting temperature of the gold (1064 °C).

The SEM provides high resolution images of the concrete surface, obtaining information about the size, shape and chemical features of its components. Micro-cracks or discontinuities, as well as the voids or different phases, can be seen.

A variable pressure Carl Zeiss EVO MA15 SEM equipped with an Oxford Instruments X-ray detector was used. It can work in low vacuum conditions and uses a lanthanum hexaboride filament as the electron source and secondary and backscattered electron detectors. The microscope is equipped with EDX detector to determine the elemental chemical composition.

3 Results and discussion

In this section, the results obtained are shown and analysed.

3.1. Physical properties

The impact of operating temperature conditions of 100 °C and 300 °C on the physical properties of HDSC, reinforced with steel fibers (SF), is shown in Table 5.

Table 5. Physical properties of the hardened HDSC.

T [°C]	Apparent specific gravity [g/cm ³]	Bulk specific gravity [g/cm ³]	Bulk SSD specific gravity [g/cm ³]	Open porosity [% vol.]	Absorption [% wt.]
HDSC-SF-100	3.88	4.35	3.97	9.82	2.53
HDSC-SF-300	3.75	4.26	3.87	12.15	3.23

At 100 °C, the adsorbed water has already been evaporated and the hydration products remain chemically unchanged [52]. The variation of 3.4 % in density observed at 300 °C is due to the loss of mass caused by the evaporation of the unbound and bound water. The loss of the water affects the microstructure of the cement paste, increasing the volume of voids and thus its porosity. Farage et al. [53] indicates that this increase in paste porosity at 300 °C is, presumably, reversible by saturation in water, since the decomposed C-S-H gel can be rehydrated along with the initially unhydrated grains. The variation in apparent specific gravity is 3%wt. while the porosity varies slightly more than

2% by volume, the absorption varies by 0.7%, due to the low weight contribution of water compared to concrete.

3.2. Mechanical properties at operating temperatures

Table 6 shows the mechanical properties of the HDSC after exposure to 3 different temperatures: 20 °C; 100 °C; 300 °C. The flexural strength corresponds to 14% of the compressive strength at 20 °C. The splitting tensile strength is around 5% of the compressive strength, for each temperature studied. The elastic modulus is slightly lower than the 30.4 GPa obtained by the empirical equation proposed by EHE-08 [54]. This last value is high compared to the typical values obtained for limestone concretes (20-30 GPa), due to the high modulus of the aggregate itself.

Table 6. Mechanical properties of the HDSC.

Mixture	Compressive strength [MPa]	Flexural strength [MPa]	Splitting tensile strength [MPa]	Elastic modulus [GPa]
HDSC-SF-20	45.9	6.61	2.51	37.96
HDSC-SF-100	55.4	-	2.89	41.07
HDSC-SF-300	49.3	-	2.65	38.12

The splitting tensile strength shows a slight variation of about 13% after exposure to 100 °C and about 5% after exposure to 300 °C. The elastic modulus varies by about 8 % at 100 °C and maintains the same value as the initial one at 300 °C. The evolution of the behavior of the compressive strength with temperature is explained below in more detail.

As is shown in the previous chart, all mechanical properties follow the same pattern for the temperatures analysed, reaching a peak at approximately 100 °C, and decreasing later. Li et. Al [55] reported a strong reduction in the mechanical properties of concrete from 400 °C on. These losses are more pronounced in splitting tensile strength and in

flexural strength, due to the appearance of cracks in the cross section, which extend easily.

3.3. Effect of thermal gradients on compressive strength

Fig. 5 shows the behavior of the concrete in terms of residual compressive strength after exposure to the different temperatures considered. Firstly, it is observed that the residual compressive strength decreases with temperature in 3 well differentiated phases: stage 1 (<500 °C: moderate), stage 2 (500-1000 °C: steep and linear) and stage 3 (>1000 °C: moderate asymptotic).

Fig. 5 shows that under 300 °C the temperature does not greatly affect the compressive strength. A slight increase is observed at 100 °C due to internal autoclaving conditions in the cement paste, allowing a flow of water vapor and hydration of unhydrated cement grains [56]. In this range of temperatures (80-150 °C) ettringite is decomposed [32]. At 300 °C, the compressive strength is 90% of the initial compressive strength and this slight loss of compressive strength is prolonged until reaching 500 °C. This loss may be influenced by the behavior of the aggregate under an oxidizing atmosphere: some authors have found a positive thermal expansion in magnetite up to 330 °C, where magnetite oxidizes to γ -hematite (maghemite), which would resist paste shrinkage. In the range from 330 to 900 °C, the γ -hematite oxides to a α -hematite (hematite) structure, showing a linear coefficient of shrinkage [57,58].

The compressive strength at 500 °C is 80% of the initial one (Fig. 5). From this temperature, a linear correlation between the residual compressive strength and the temperature begins. In the range of 500-700 °C, a pronounced loss of strength is observed. This loss may be due to the dissociation of portlandite ($\text{Ca}(\text{OH})_2$) into CO_2 and CaO and its expansive rehydration [37]. Another cause could be the decomposition of the C-S-H gel which decomposes into $\beta\text{-C}_2\text{S}$ at 600-700 °C [59] making the cement paste structurally unsuitable. At 700 °C the compressive strength is 60% of the initial one and from this temperature to 1000 °C, with the C-S-H gel completely

decomposed ($\sim 800^\circ\text{C}$) [35], the loss of strength continues to be highly attenuated, reaching 50% of the initial value at 1000°C . Horszczaruk [35] found similar behavior except for temperatures higher than 700°C and close to 100°C (which was not analysed). Fig. 5 shows the results obtained by this author and those obtained in the present study, along with the trend line obtained in the Q. Ma review [42] for conventional limestone concretes. The behavior found by some authors [60–64] for these conventional concretes is also shown in the figure. The limestone concretes reach residual compressive strengths 20% lower than those obtained with magnetite aggregate.

As can be seen in Fig. 5, from 1000°C , a more attenuated asymptotic tendency can be observed. At 1200°C , a slight increase in residual resistance is observed, which may be due to sinterization between 1000°C and 1200°C , which reduces porosity.

Fig. 5. Effect of temperature on the HDSC compressive strength.

Sinterization of the mineral does not indicate that the concrete improves its mechanical properties at very high temperatures, since sintering has changed the characteristics of

the material [65]. The sintering reaction occurs in magnetite at approximately 1000-1100 °C, greatly increasing the contraction and, depending on the fineness of the aggregate, negatively affecting its oxidation [58], because this coalescence phenomenon causes an increased resistance to oxygen penetration in the reaction interface [66].

The effect of temperature on the mechanical properties of HDSC is evident, but less than for conventional concrete due to the high melting point of the magnetite aggregate (1596 °C).

The final state of the specimens subjected to compressive strength tests after applying thermal gradients can be seen in Fig. 6.

Fig. 6. Specimens subjected to compressive strength test.

With a visual inspection, the evolution of the color tonality of the tested specimens can be established with the increase of the exposure temperature. The factors that generate these color changes are, on the one hand, the progressive elimination of water and the dehydration of the cement paste, and on the other hand the transformations of the aggregate. In the specimens a dark grayish shade was observed with exposure temperatures of 100 °C, a paler gray tone was reached at 300 °C and turning whiter at 500 °C, turning into brown/red at 700 °C and dark brown at 1000 °C, as a consequence of the oxidation of iron ores. At 1000 °C, no fiber signals are found and at 1200 °C, a blackish color is observed, as well as aggregates with a more metallic shine. The outer faces are, in any case, a lighter shade than the inside of the specimen, except in the case of the extreme temperature. Visually, the lack of consistency of the test pieces

subjected to the most extreme temperature is noticeable, crumbling easily at 1200 °C. During the heating process, no visual spalling was detected, as is the case in other studies where the specimens were subjected to high exposure temperatures and spalling on the outer walls [67].

3.4. Effect of thermal gradients on ultrasound pulse velocity

The ultrasonic pulse velocity through the concretes subjected to different temperatures is shown in Fig. 7. The results obtained are adapted to a polynomial fitting curve of degree 3, according to the equation:

$$Y = 4.5 - 0.0047x + 2.7 \cdot 10^{-6}x^2 \quad (2).$$

Temperature affects the ultrasonic pulse velocity in a negative way. Heat affects the physical properties of the concrete, increasing porosity and causing a loss of mass. This generates the appearance of discontinuities that cause a decrease in the ultrasonic pulse velocity.

Fig. 7. Effect of temperature on the HDSC ultrasonic pulse velocity.

A first zone showing a slight reduction in the pulse velocity can be observed up to 300 °C. The ultrasonic velocity at the end of this zone at 300 °C is 90% of the initial one. Increasing the temperature of exposure, a second zone of high variation of pulse velocity is reported. At 700 °C the speed has decreased to 45% of the initial value. Increasing the temperature over 700 °C, a new zone characterized by a more attenuated pulse velocity loss is observed. At 1200 °C the pulse speed is about 40% of the initial value. In the third zone the reduction in porosity, probably due to a sintering process, is the probable cause of the slope loss in the fitting curve. Although ultrasonic wave velocities are related to the modulus of elasticity, since the vibrations introduced in the specimen are consistent with very small strains, a certain relationship can be established between ultrasonic impulse velocity and compressive strength. This relation is typically established in the form $S = a \exp(bV_p)$, where a and b are empirical variables [68]. It can be observed that the trend shown by the ultrasonic pulse velocity at the end is different to the trend observed for compressive strength. The closest fitting curve for both corresponds to a polynomial regression of degree 3. This analogy can be justified by the physical-chemical transformations that affect the mechanical behavior, such as the shrinkage and expansion phenomena in aggregates and paste, and the degradation of the paste also affects the ultrasonic pulse velocity. The correlation obtained between the ultrasonic pulse velocity and the shocking temperature could be used to determine the damage caused to concrete by high temperatures easily and without the need for advanced testing equipment.

3.5. Scanning electron microscopy

For the microstructural analysis, two observation scales are analysed, one at low magnification (250x) and another at higher magnification (1000x). The 250x scale is analyzed to establish the general state of the paste-cement set and the presence of gaps or discontinuities at the interfaces. The 1000x scale enables the visualization of both the phase changes of the minerals and the state of the paste and its formations after the physical-chemical changes.

¡Error! No se encuentra el origen de la referencia.In Fig. 8 the evolution of the concrete subjected to 100 °C and 300 °C can be observed in micrographs at 250x. At 100 °C, good condition of the C-S-H gel is observed. The observed porosity is relatively low. At 300 °C, there were few changes in the porosity level and discontinuities. Micro-cracks can be observed in the paste due to the shrinkage generated by the drying process. The aggregates do not seem to be affected visually. At 500 °C, the level of degradation of the matrix in a shrinkage process generates more severe micro-cracks (**¡Error! No se encuentra el origen de la referencia.**Fig. 9) present in both the paste and the transition zone between paste and aggregate (ITZ). These cracks are responsible for poorer mechanical behavior above these temperatures. At 700 °C, the temperature at which the C-S-H gel decomposes, the effects of heat are visibly more pronounced. The paste shows a lack of cohesion, and the C-S-H gel seems porous, leading to the appearance of wider and larger micro-cracks. In addition, a poor adherence between the paste-aggregate due to the appearance of fissures in the ITZ can be observed (**¡Error! No se encuentra el origen de la referencia.**Fig. 10). These micro-cracks reach 10 µm width and begin to interconnect, both in the paste and in the ITZ. The first micro-cracks in the aggregate begin to appear. At 1000 °C there is visible growth in the interconnection of micro-cracks in the paste but the beginning of changes in the microstructure of the aggregates is also noticeable (Fig. 11). At 1200 °C, in Fig. 12 it can be observed how the C-S-H gel has deteriorated so much that the aggregates have been exposed. There is an evident phase change of the mineral characterized by showing a rhombohedral-hexagonal micro-structure which corresponds to the shape of hematite crystals. There are also micro-structures formed by long tetragonal maghemite ($\gamma\text{-Fe}_2\text{O}_3$) nanocrystals of about 3 µm side. The heating time has not been enough for all the mineral to change phase. The presence of voids derived from the complete degradation of the cement paste is also observed. At this temperature the material becomes more compact because the mineral grains partially melt to give rise to a partial sintering phenomenon.

Fig. 8. 250x HDSC micrographs after exposure to 100 and 300 °C.

Fig. 9. 250x HDSC micrographs after exposure to 100 and 500 °C.

Fig. 10. 250x HDSC micrographs after exposure to 100 and 700 °C.

Fig. 11. 1000x HDSC micrographs after exposure to 100 and 1000 °C.

Fig. 12. 1000x HDSC micrographs after exposure to 100 and 1200 °C.

Conclusions

In this paper, a series of tests have been carried out to determine the properties and behavior of a high-density structural concrete, which has been subjected to elevated temperature gradients from 100 to 1200 °C. From the results obtained, the following conclusions are drawn:

The effect of temperature on the physical properties, for operating temperatures ranging from 100 to 300 °C, is slightly negative and is directly related to the evaporation of bound and unbound water.

Heat negatively affects the residual compressive strength of the concrete, establishing 3 behavioral zones. For temperature ranges between 20 and 500 °C, the strength drops steadily and its magnitude barely varies, while in the range from 500 to 1000 °C, the concrete loses much of its resistance. From 1000 °C, the highly degraded concrete shows an asymptotic compressive resistance loss.

Heat has a negative impact on ultrasonic pulse velocity. The way in which this happens is analogous to that obtained in the case of residual compressive strength, establishing the existence of a direct relationship between the two properties.

The micrographs obtained by SEM show the result of the physical-chemical processes that the concrete underwent in each temperature range studied. The relation between the appearance of cracks in the cement matrix and in the ITZ is established. These cracks provoke the reduction of the mechanical properties of the concrete. In addition, the degree of effect on the aggregate and its transformations are determined. The SEM results provide a better understanding of the changes happening at the micro-scale and how these changes affect the mechanical behavior of the concrete.

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