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# CHARACTERISATION OF THE BEHAVIOUR OF HIGH PERFORMANCE MORTAR SUBJECTED TO HIGH TEMPERATURES

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## ABSTRACT

An experimental investigation was conducted to evaluate the performance of mortar mixed with silica fume (SF) when exposed to high temperatures. A three-point bending test apparatus was developed to test concrete-like materials at high temperatures. Notched specimens were first heated to various target temperatures from room temperature to 900°C. The maximum peak load occurred at 300°C and decreased sharply at higher temperatures. The experimental results demonstrated a noticeable influence of the temperature on the fracture resistance of the high-performance mortar. SEM micrographs of the heated specimens after the mechanical tests and cooling and TGA/DTA analysis of the dried material matrix facilitated the understanding of the material's macroscopic behaviour.

**Keywords:** high performance mortar, high temperatures, SEM micrographs.

## INTRODUCTION

The properties of concrete are altered significantly by exposure to high temperatures, mostly in terms of reduced stiffness and strength. Part of the temperature effects is due to chemical changes and moisture transport within the cement paste and part are due to damage (micro cracking) from temperature gradients and deformational incompatibilities between aggregate and cement paste. Compressive strength is usually employed as the main parameter for the design of concrete structures. Nevertheless, as in other brittle materials, its fracture is governed by tensile mechanisms. Not only is strength important but so also is the whole behaviour of concrete under tensile stresses, especially its toughness.
Concrete is a composite material where inclusions of different size and shape, the aggregates, are surrounded by a more or less continuous matrix that acts like a binder. This matrix, regarding the observation level could be cement paste or mortar. It includes diverse types of defects (pores, micro-cracks). Like in other brittle materials, the failure mechanism of concrete is closely related to the initiation and propagation of cracks. Coarse aggregates generate weaker zones (interfaces) in which the development of the cracks begins.
Numerous works verified the microstructure and morphological differences of the interfaces with respect to the paste [1]. Furthermore, macro-defects generated during compaction, bleeding, etc. may concentrate around the bigger aggregates and also cracks will appear due to different stiffnesses, shrinkage or thermal expansion coefficients between paste and aggregates. An important case of damage of the structure of concrete appears when it is

exposed to high temperatures. The different dilatation coefficients of the phases of the composite and the changes in water content of the cement paste produce cracks. Initially, the temperature elevation results in the elimination of the water contained in the pore system and the consequent contraction of the paste with crack formation. Temperature rise over 500°C produces an alteration in concrete that can be considered non-reversible, as the loss of chemical bounded water (dehydration of the paste) takes place. The difference between the thermal dilatation coefficients of the aggregates with respect to the paste causes micro-cracking at the interfaces, which increases the size and closeness of the internal cracks. Diverse works have verified the degradation of the microstrure due to the exposure to high temperatures and its effects over the behavior of concrete, the changes being a function of the hearing and cooling cycle [3-5]. Evaluation of the tensile strength shows a great variability, and it is verified that it is more sensitive than the compression strength to the changes produced by the exposure to high temperatures, with the type of aggregate being of great importance. Recent studies on the residual properties in flexure and direct tension of high-strength concretes exposed to high temperature verify the decrease in tensile strength and toughness, the increase in characteristic length, and only minor effects on the fracture energy [6]. The objective of this work is to study the failure behavior of damaged concretes in tension. Temperature was adopted as the damaging tool, comparing the behaviour of concrete series including different strength levels and different component materials.

An effective evaluation of concrete resistance at high temperatures requires a profound knowledge of the phenomena occurring during heat exposure. The phenomena are directly connected to the chemical–physical processes occurring in the components, which are initiated by a temperature increase. The most important processes include the following: chemical reactions, phase transformations, and heat-induced deformations that occur at the microscopic scale in particular phases of concrete components [2,7]. The dense microstructure of HPC seems to be a disadvantage in fire conditions. It was observed that HPC, especially high strength concrete (HSC), is susceptible to spalling or even explosive spalling when subjected to a rapid temperature rise, such as in a fire [3–6]. Nielsen et al. [8] tested about 80 beams of high performance basalt concrete in accordance with the RILEM (Réunion Internationale des Laboratoires et Experts des Materiaux) work of fracture method. The beams were heated at 1°C/min up to reference/target temperature that was kept constant for height hours before cooling the beams back to room temperature; then, three-point bending tests were performed on the beams. The tests showed that the damage introduced by a maximum temperature between 300 and 400°C increased the fracture energy by 50% compared to the reference tests at room temperature. Matesov´ a et al. [4] identified three thermal zones: low (up to~300°C), intermediate (~300–600°C), and high (>600°C).

They showed that in the low thermal zone, the mechanical properties were about the same, or even better than at room temperature. The intermediate thermal zone was characterised by a moderate decrease of mechanical properties, whereas a rapid decrease was observed in the high thermal zone. Explosive spalling due to pressure build-up of volatiles occurred at temperatures higher than 200°C. By using thermal analysis, X-ray diffraction analysis, infrared spectroscopy analysis and mercury porosimetry, Piasta et al. [5] studied the behaviour at high temperature of the following present (that are present in the pastes): $Ca(OH)_2$, CaCO3, C–S–H, non-evaporable water and micropores. From the analysis of the experimental results, temperature ranges for the following changes were determined in the structure of the investigated paste: additional hydration of unhydrated cement grains, recrystallisation and carbonisation of Ca $(OH)_2$, deformation and transformation of C–S–H phases, non-linear changes in the distribution of pore diameters and total porosity.

Most results from the literature focus on the material behaviour at room temperature after concrete was heated to various temperatures and then cooled. These studies essentially evaluated the residual resistance of various concretes [6–8]. Other more ambitious projects have tried to study the concrete behaviour at high temperatures, but the tests were more difficult to perform. In general, target temperatures have been limited to 600°C, which is the reference temperature for fire conditions in a building. For instance, Zhang et al. [11] carried out hot bending test up to 450°C and Khoury et al. [9] conducted hot tests with results up to 600°C.

The present study investigates HPC mortar strength trough three-point bending tests carried out at temperatures up to 900°C on notched specimens; the influence of the specimen notch lengths has been studied. The evolution of the peak load with temperature and notch length were determined and reported. SEM micrographs of specimens after hot testing and cooling were used to help understand the material behaviour.

## MATERIALS TEST MODALITIES

The investigated mortar material was a mixture of calibrated normalised sand, cement, and water mixed with super-plasticiser, and fine particles of silica fume. The maximum particle size of the calibrated sand was 2 mm. The cement was a CEM I 52.5 produced by Vicat, and a liquid super-plasticiser containing polycarboxilate was from Sika. This mortar material had a low porosity with a w/c ratio of 0.25 (Table 1).

The four blade dimensions corresponding to the specimens' notch lengths were: 7.5, 10, 12.5 and 15 mm. The specimens were prismatic with a square section and measured 160 x 25 x 25 mm3, as shown in Fig. 1. The specimens were cured three months long (90 days) in water, put out of the water and dried at ambient temperature three days before the tests. The tests were carried out on a ZWICK Z400 testing machine equipped with a 1600°C temperature heating capacity industrial furnace. The setup was equipped with a special extensometer based on measurements of two LVDTs (Linear Variable Differential Transformer) sensors placed outside the furnace. A differential system, which used alumina rods positioned axially through the furnace, measured the relative displacement between the upper and lower punch; this displacement theoretically corresponded to the specimen height variation in a uniaxial compression test (Fig. 2). The present study specifically examines the application of mechanical loading while the specimen is heated. The following three-phase thermal cycle was applied to the material during the tests: (1) a heating phase at a rate of 3.3°C/min; (2) a rest period at the target temperature; and (3) a cooling phase at a rate of 2.5°C/min. The rest time was divided into two periods: at first, a 2-h period applied in all tests, and used to allow the specimen and the loading apparatus to reach a uniform temperature; during the remaining time mechanical loading was applied. This second phase generally ended with the complete failure of the specimen. The heating rate applied during the test is consistent with RILEM recommendations. A constant 0.015 mm/min displacement rate was applied to the specimen, and the reaction force was measured. The following target temperatures were investigated: 25, 150, 300, 500, and 700°C.Once the specimen had returned to room temperature, it was removed from the furnace, and a SEM analysis was performed.

Table 1 Typical composition of the studied high performance mortar.

| Constituents | Content |
|---|---|
| **Sand (g)** | 1350 |
| **Cement (g)** | 450 |
| **Water (g)** | 112.5 |
| **Superplasticizer (wt.%)** | 1.5 |
| **Silica fume (wt.%)** | 10 |
| **w/c ratio** | 0.25 |

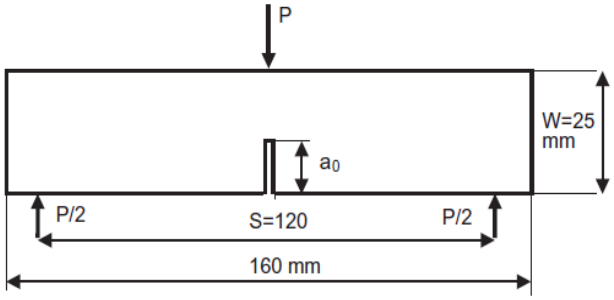


Fig. 1. Geometry of the specimens tested in three-point bending.

Fig. 2. View of a specimen in the testing apparatus after at 500°C.

## RESULTS AND DISCUSSION

### *Global behaviour of the material*

Results were obtained on the fracture load at various temperatures using three-point bending tests. With various notch lengths at the considered target temperatures. The results are characterised by the classic bell-shape evolution, with a peak load followed by a softening branch. A reference curve displayed in Fig. 7 allows a proper description of the material behaviour exhibited in Figs. 3–6 in three stages, as reported elsewhere [12–13]. A first stage consists of quasi linear evolution of the load with increasing deflection, followed by a second stage ranging from the appearance of the first nonlinearities to the peak of load, and, closed lastly by the third stage characterised by a decrease of the applied load. In the first stage, cracks are open but do not extend [14], whereas in the second stage, a fracture process develops, micro-cracks appear and progressive crack growth is observed. In the third stage, called strain softening zone, a rapid crack growth that can lead to instability is observed.
During strain softening, most of the damage occurring in the specimen is localised in a narrow zone as assumed in [15]. This localisation increases, as the load carrying capacity decreases [16,17]. It was observed that energy dissipation occurred through a single major crack [18]. Strain softening is considered as a material characteristic [19].
In the present study, the tests generally ended when the specimen broke into two parts. Figs. 3–6 display the recorded applied load versus deflection curves of the three-point bending test carried out at five different target temperatures on four different notch lengths specimens.

Hence, the maximal values of deflection before fracture of the specimens are comprised between 0.2 and 0.4 mm (Figs. 3–6). A number of observations can be made. Firstly, however the notch length is, the material behaviour is clearly dependent on the target temperature: very sharp with a pronounced peak at low temperatures evolving to more rounded and progressive evolution at higher temperatures. Secondly, the maximal applied load decreases between 25°C and 150°C, then increases and reaches its maximal value at 300°C, and then decreases with increasing temperature. So the examination of the recorded curves reveals two characteristic temperatures: decrease of the bending peak load down to 150°C and increase to its maximal value up to 300°C, however the notch length is. Lastly, the rounded evolution of the curve-deflection curve at 700°C evokes damageable or viscous occurring phenomena in the material.

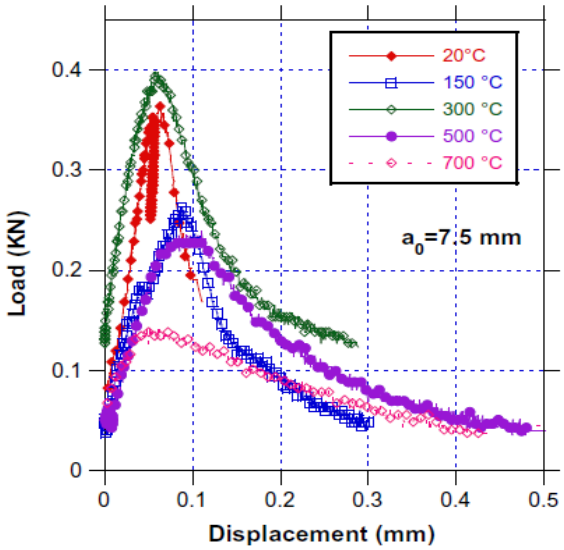


**Fig. 3**. Load–deflection of the specimens with notch length = 7.5 mm for different values of the testing temperature.

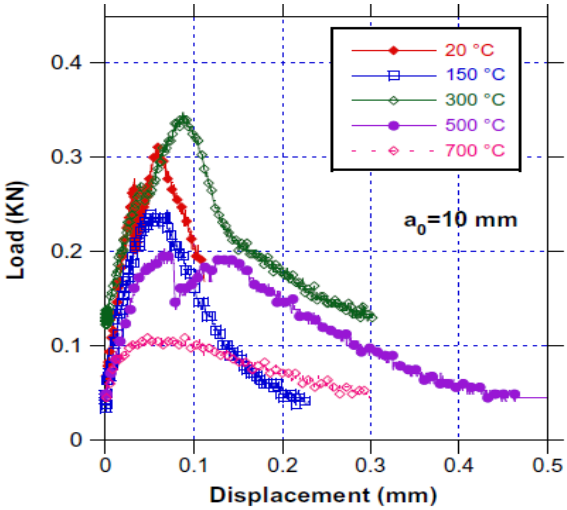


**Fig. 4.** Load–deflection of the specimens with notch length = 10 mm for different values of the testing temperature.

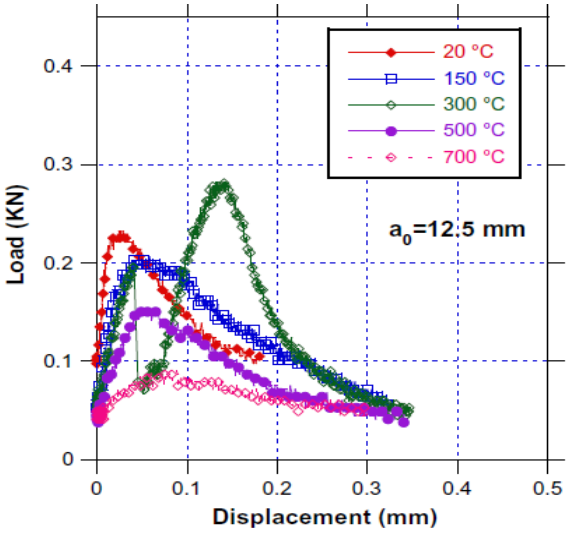


**Fig. 5.** Load–deflection of the specimens with notch length = 12.5 mm for different values of the testing temperature.

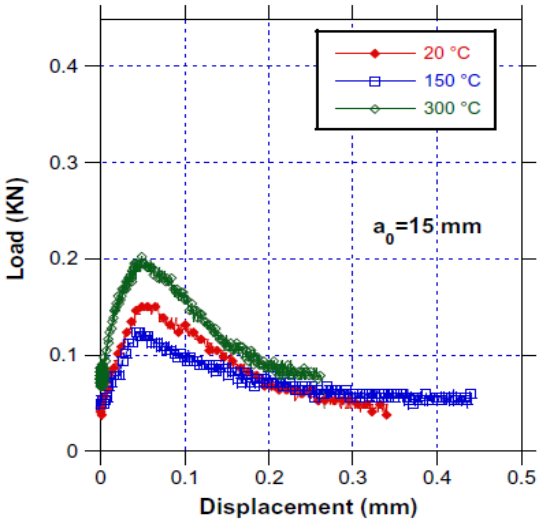


**Fig. 6.** Load–deflection of the specimens with notch length = 15 mm for different values of the testing temperature.

For a global analysis purpose of the obtained results, the maximal bending applied load (peak load) has been plotted versus temperature for various notch lengths (Fig. 8) or versus the notch length for various target temperatures (Fig. 9). The peak load appears to be dependent logically on the specimen notch length: the smaller the notch length the higher the peak load recorded. The decrease of the peak load from room temperature to 150°C as its increase reaching its maximal load at 300°C appear clearly to be common to all the four notch lengths specimens tested. Fig. 9 shows that the peak load decreases with increasing notch length, however the target temperature. The decrease of the peak load at 150°C is significant and interesting. At this temperature drying process by elimination of free and adsorbed water in the material is complete and at this range of temperature, the gypsum constituent is supposed to decompose in the mortar under endothermic reactions [21]. Some authors, for example, Khoury et al. [15] suggested that at the micro-structural scale at this temperature, water fluidity increases, which could induce a decrease in the Van der Waals forces between the slices of calcium silicate hydrate (C–S–H) coat, leading to a decrease in the surface energy of the coat.

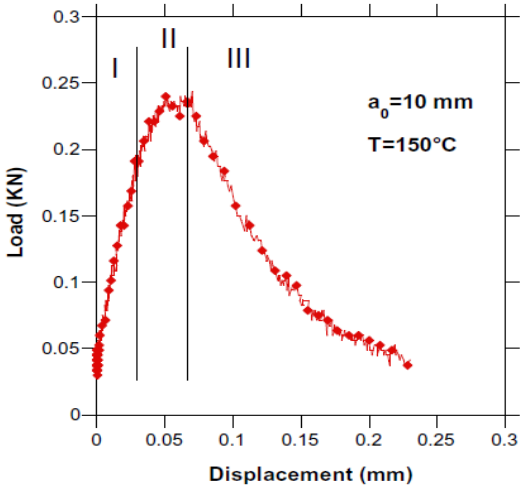


**Fig. 7.** Example of Load-Load Point Deflexion curve illustrating the different stages of the mortar behaviour.

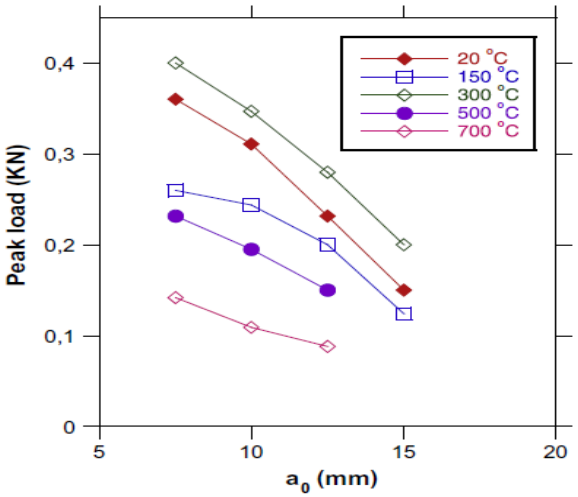


**Fig. 8**. Plots of the peak load as a function of the testing temperature for different values of the notch length.

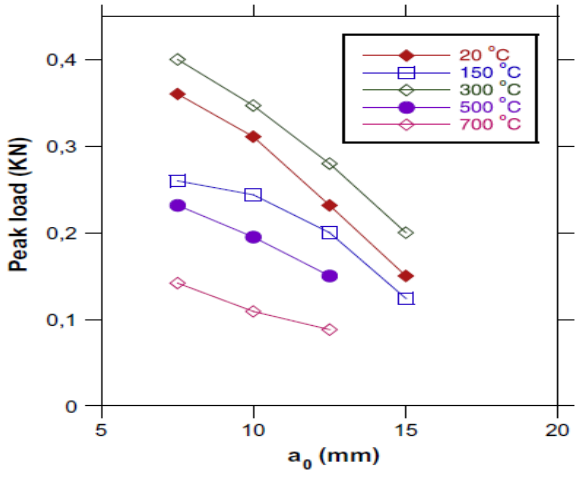


**Fig. 9.** Plots of the peak load as a function of the notch length for different values of the testing temperatures.

### *Analysis of the microstructure*

Concrete and especially cement hydration process is a very complex phenomena. The $C_2S$, $C_3S$, $C_3A$ and $C_4AF$ components initially constituting the Portland cement powder when mixed with water produce essentially Calcium Silicate Hydrate ($CaO–SiO_2– H2O$) named C–S–H in a gel form (50–70%), Portlandite ($Ca(OH)_2$)(25–27%), alumina silicate hydrates, Ettringite ($3CaO– Al_2O_3–3CaSO_4–32H_2O$) and other various hydrated phases.
The CSH phase is principally responsible of the cement resistance so that their appearance and evolution upon time is of the utmost importance, particularly when the material is heated at different increasing temperature. The evolution of these various phases with temperature is important and may explain the material macroscopic behaviour.
The aim of the investigations undertaken in this study was to understand through microstructure analysis the evolution of the material mechanical measured properties. The strategy consists in detecting the presence of C–S–H phase in the material at the various target temperatures. C–S–H phases are characterised by a C/S (CaO/SiO2) molar ratio included between 0.8 and 2.1 with a mean value at 1.7 [22]. The investigated zones were preferably situated near the boarder of the aggregates where the surroundings are probably constituted of the matrix and where C–S–H and Portlandite are supposed to initiate and develop [23].
The micrographs revealed that Ettringite were present at room temperature (Fig. 10a) but no more at 150°C (Fig. 10b) and at higher temperatures. This is consistent with the DTG curve which indicates a phase transformation at about 100°C. Portlandite has been observed at 25, 150, and 300 and even at 500°C (traces). Beyond 500°C, especially at 700°C, Portlandite has dehydrated and is no more present. C–S–H has been found at temperature up to 500°C. At 700°C, C-S-H is present but the ratio C/S of the phase in presence is generally higher than the attended one and tends to a 3 constant value. Fibrillar C–S–H type has been observed exclusively at room temperature [23]. At higher temperature (P150°C) we did not observe fibrillar but bumpy and amorphous morphology for the C–S–H products. Fig. 10b shows the matrix paste at 150°C where Portlandite surrounded by CSH and other phases is visible. The luxuriant fibrilar aspect displayed by the micrographs at room temperature is no more visible. The aspect has become pasty. At 300°C (Fig. 10c), the paste appears very compact with a lesser dense micro-cracks network than at 150°C. The hydrated phases present at 150°C have been found to be present at 300°C. So, the increase of the material strength between 150 and 300°C is not the result of the apparition of a new phase, but seems to be the effect of dehydration of the existing phases. This is consistent with the DTA curve where no new phase occurrence is observed between 150 and 300°C.
However present, it becomes more and more difficult to find C–S–H product since Portlandite decomposition has produced new phases. Hence, the majority of the phases met at 500°C have a C/S ratio of 3. But C–S–H is still present. We have recorded in Fig. 11 and followings the apparition of a new phase with characteristic shell morphology. This phase contains CS elements but also non negligible aluminium. When the temperature reaches 700°C, the cement structure evolves from pasty to granular where the binder seems to have disappeared. The matrix porosity has visibly increased and the lack of binder suggests a visible decrease of the material resistance. At 900°C, the matrix has no consistency and seems to become totally granular.

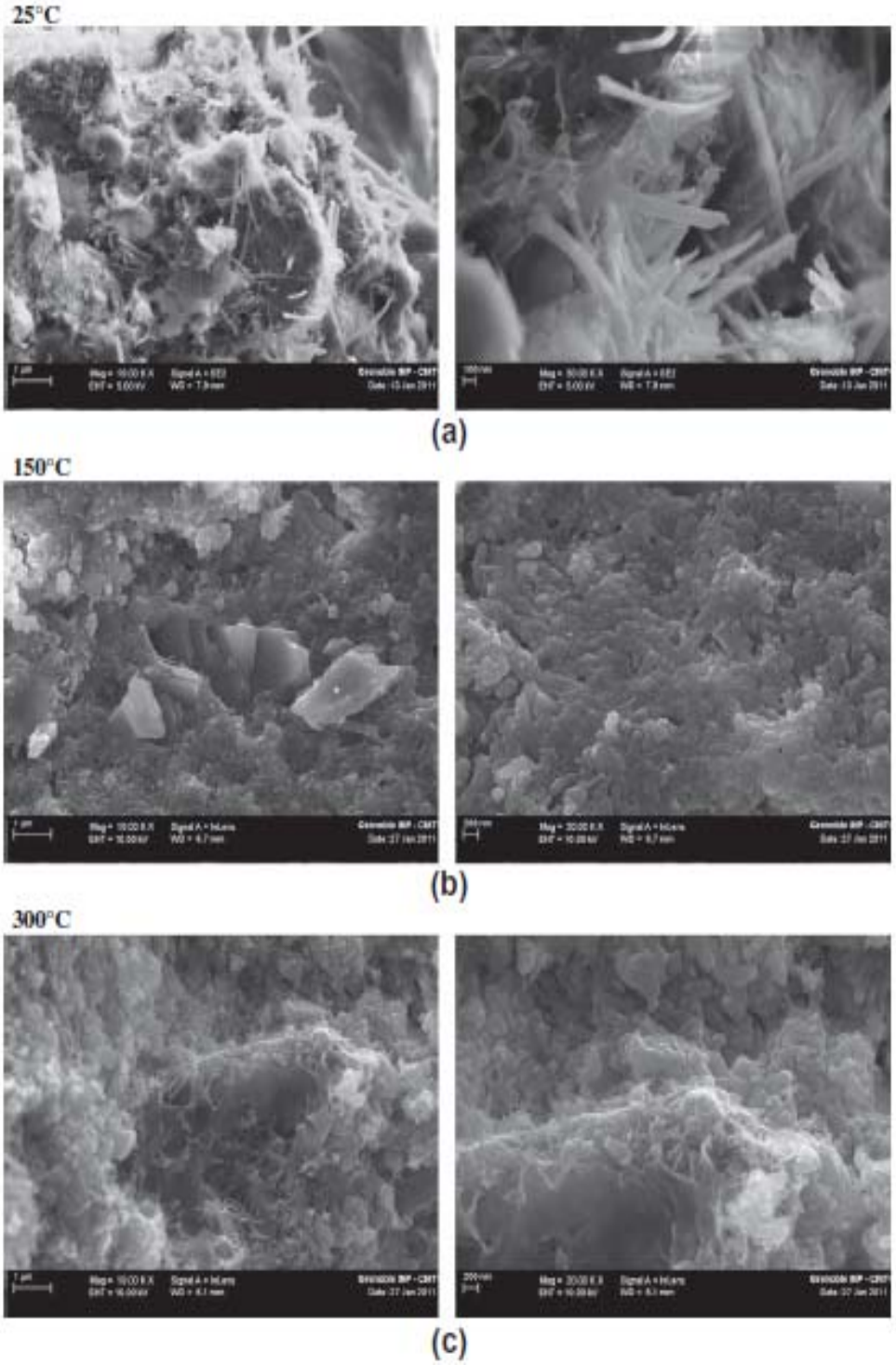


**Fig. 10.** Micrographs of the studied materials at two different scales after heating At (a) _25 °C, (b) _150°C and (c) _300°C.

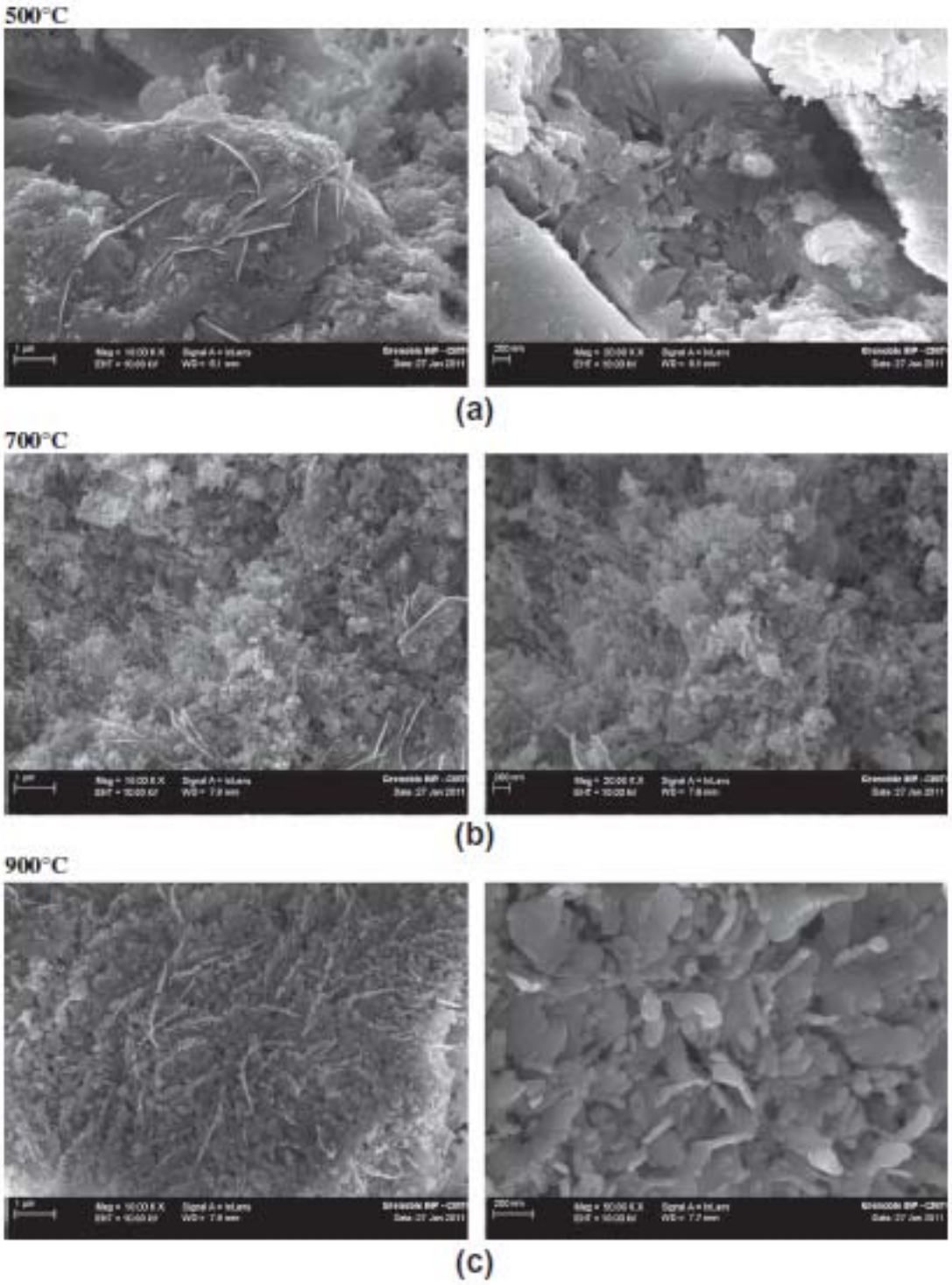


**Fig. 11**. Micrographs of the studied material at two different scales after heating at (a) _500°C, (b)_700°C and (c) _900°C.

## CONCLUSION

The aim of the present work was to investigate high performance concrete mortar behaviour when subjected to increasing target temperatures. A three-point bending test apparatus was developed in alumina material, and tests were conducted by loading various lengths notched specimens that had been heated to various temperatures (25, 150, 300, 500, 700, and 900°C). The following conclusions were reached:

- For all notch lengths; below 500°C, the material exhibited a nonlinear and quasi-brittle behaviour; however, it still retains a remarkable share of the initial strength (65%). At 700°C, the behaviour became nonlinear with pseudo-ductility and notably lesser resistance. The optimal resistance was obtained at 300°C, and the material exhibited no resistance at 900°C.
- A decrease of the material resistance is observed between room temperature and 150°C followed by an increase up to their maximum value at 300°C. This is probably due to not sufficiently drying initially the tested specimens.
- SEM micrographs analysis undertaken on the material after cooling showed a microstructure evolution with increasing temperature that was consistent with the material's macroscopic behaviour expressed by the material strength and tenacity parameters.

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