ANALYSIS OF THE HEATING EFFECT ON HIGH PERFORMANCE MORTAR THROUGH 2D IMAGES OF X-RAY MICRO-TOMOGRAPHY AND SEM MICROSCOPY

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Résumé

Le document traite de l'application de la technique de micro-tomographie par rayons X pour étudier des éprouvettes entaillé de mortier à haute performance tout d'abord, pour des éprouvettes ayant subit des essais de flexion trois points à chaud à une température de (700 ° C) et en second lieu, après seulement un chauffage des éprouvettes à 500 ° C. Les paramètres de numérisation ont été fixés pour visualiser tout l'échantillon pour une section transversale carrée de 25 mm de côté. Dans ces conditions, une partie de la réelle porosité du matériau, censé être représentatif, était accessible. Des images 2D et 3D reconstruites furent obtenues et analysées. Ainsi, parmi les différents paramètres de la microstructure considérée, l'homogénéité de la porosité ainsi que l'échantillon a été analysée notamment en fonction du paramètre de fraction de surface des pores. Une sensible augmentation dans la porosité des zones entourant le plan de premier ordre a été observée ce résultat est probablement lié à la préparation de l'échantillon. L'impact sur la validité des essais de flexion trois point effectués sur les éprouvettes entaillés a également été examinée.. Enfin, la capacité de cette technique pour évaluer l'effet de la température sur la porosité dans ces conditions particulières a été examinée à la lumière de l'analyse complémentaire des micrographies MEB.

Mots clés *: Micro-tomographie, porosité, éprouvettes entaillés, béton à haute performance, température.*

Abstract

The paper deals with the application of the X-ray micro-tomography technique to study high performance mortar notched test specimens first, after bending tests at room and hot conditions (700°C) and second, after just heating at 500°C. The scanning parameters were set to view all the test specimen transverse square section of 25 mm in side. In these conditions, a part of the material real porosity, supposed to be representative, was accessible. 2D and 3D reconstructed images were obtained and analysed. Hence, among the various microstructure parameters considered, the homogeneity of the porosity along the specimen was especially analysed through the pore area fraction parameter and discussed. A sensible increase in porosity of the areas surrounding the notch plane was observed and this result is probably related to the preparation of the specimen. The impact on the validity of bending tests performed on such notched specimens was also examined. Lastly the ability of this technique to evaluate temperature effect on porosity in these particular conditions was discussed in the light of complementary analysis of SEM micrographs.

***Key words**: Micro-tomography, porosity, notched specimen, high performance concrete, temperature.*

ملخص

هده الورقة تناولت تطبيق تقنية التصوير المقطعي الجزئية الأشعة السينية لدراسة عالية الأداء لهاون حقق لقد انجزة عينات اختبار الأول، بعد الاختبارات الانحناء في غرفة والظروف الساخنة (700 درجة مئوية)، والثانية، بعد مجرد تسخين في 500 درجة مئوية تم تعيين المعلمات المسح الضوئي لعرض كل عينة اختبار المربعة شريحة عرضية من 25 ملم في الجانب في هذه الظروف، وهي جزء من المسامية الحقيقية اجلوهرية، من المفترض أن يكون ممثلا، كان الوصول عينة المربعة شريحة عرضية من 25 ملم داخل الجان وقد تم الحصول على 2 و3 الصور التي أعيد بناؤها وتحليلها. وبالتالي ، أوساط المعلمات البنية المجهرية المختلفة في الاعتبار، تم تحليل تجانس المسامية على و عينة خاصة من خلال المعلمة جزء مساحة المسام ومناقشتها لوحظ وجود ازدياد معقولة داخل المسامية من المناطق المحيطة بها الطائرة من الدرجة الأولى وهذه النتيجة الارجح يرتبط إعداد العينة وتطرق البحث إلى تأثير ذلك على صحة الانحناء الاختبارات التي تجرى على مثل تلك عينات من حقق وأخيرا تمت مناقشة مقدرة هذه التقنية لتقييم تأثير درجة الحرارة على المسامية في هذه الظروف خاص في ضوء التحليل التكميلي

كلمات مفتاحية *: التصوير الشعاعي الطبقي الجزئية، المسامية، اقتنصت العينة، الخرسانة عالية الأداء، درجة الحرارة.*

X-ray microtomography (m-CT) is an uprising non-destructive means more and more used to investigate materials behaviour and especially porous materials. For example, in the context of building materials, this technique has been used to evaluate pore structures and cracking in cement-based materials (concrete, mortar). It has been used, also, to study leaching processes in mortars as well as the role of self-healing agents on partial restoration of concrete after cracking. According to Kim et al. [20], one particular advantage of m-CT is the ability to scan the same sample under different conditions and monitor changes, as demonstrated for concrete at elevated temperatures.

The m-CT technique consists in obtaining a 2D image of an object whatsoever by subjecting it to X-rays while it rotates around a reference axis. The displacement step by step of the x-ray source along the rotating axis allows the acquisition of several images that can be combined by mathematical transformations to obtain a volumetric (3D) image of the object. Hence, one can have a view of the object through any desired plane.

So the reconstruction from the acquired 2D images (slices) gives a virtual image of the object from which a proper operation can reveal what is hidden inside: composition, defects, porosity, different phases or constituents eventually. From this virtual image, quantitative evaluation can be performed in order to isolate defects or some of the constituent and build for instance a mesh aimed at a finite element analysis, for instance.

The literature reports that several studies using the m-CT technique have shown that when cement-based materials are exposed to the high temperatures, the changes in pore structure and density prevail. Kim et al. [20] have observed distinctive changes in the phase in pore volume fraction, material density, and cracking by heating. The effects of fire on concrete include irreversible chemical and physical changes that vary according to temperature. Changes of pore structure and cracking caused by fire induce serious deterioration of the concrete's performance.

The author concluded that m-CT technique appears to be a powerful tool with which to address the physical changes caused by elevated temperatures in cement based materials. Stein et al. [21] have observed an increase in the porosity of thermally loaded concrete specimens and a coarsening of air void sizes. Henry et al. [22] have investigated the effects of heating and water re-curing on cracking in high strength cement paste and concrete by using m-CT, and various image analysis techniques were applied to characterize the changes in the crack structure.

They found that, in the cement paste specimen, heating resulted in straight, discrete radial cracks due to shrinkage caused by dehydration. Cracks in the concrete specimen due to heating generally formed around aggregates due to incapability in thermal expansion between the mortar and aggregates.

Lanzón et al. [23] have showed that low density additions have a strong influence on essential properties of mortars, such as mechanical strength and capillary water absorption.

In a previous study on high performance mortars subjected to high temperatures, strength, stress intensity factor and fracture energy were determined from tests on notched specimens evolving with temperature (Djaknoun [24]). It appeared roughly that these variables first increased from room temperature to 300°C where the maximum was reached and then decreased from 500°C to 700°C where they were notably reduced.

Some SEM micrographs were made and the analysis helped in understanding the evolution with temperature of these mechanical properties. But some questions are raised about the values of the fracture energy that was rather less than attended. One of the most important was, especially in the vicinity of the notch plane: Was the material homogeneous in the notched specimens? Was the material safe of defects unlike large pores visible here and there on the notched specimens' lateral surfaces?

In the present study, we intend to identify changes in the physical characteristics of the internal structure of high performance mortars subjected to high temperatures, and then connect to the mechanical characteristics, especially the resistance to cracking. This is why, an inside vision of the specimen was then necessary and micro-tomography was then the relevant technical investigations to use. In order to reduce the number of temperatures and specimens of the present study, previous investigations results by SEM were accounted [24].

1. MATERIALS AND MODALITIES

1.1. X-ray micro tomography

The measurements have been undertaken on the experimental micro-tomography set-up of Laboratory 3SR. It consists in the specimen with its resin circular base is placed and screwed in the centre of the rotating plate of the set-up. The height displacement course of the moving x-ray source as well as the specimen circular motion is controlled to be right.

1.2. Studied specimens

The studied specimens are prismatic (25x25x160 mm^3) made of high performance mortar including a notch at their mid-length in a transverse plane. They have been used in three-point bending tests at various temperatures in order to determine the tenacity parameter of the material from this previous study three temperatures are retained for micro-tomography: 20, 500 and 700°C. The heating of the specimen followed a strict protocol: heating phase at a rate of 200°C/h until target temperature, constant stage of 2 h to homogenize the temperature in the specimen, mechanical loading if any and then a cooling phase at a rate of 150°C/h.

During the micro-tomography test only the central part of the notched specimens, approximately 25 mm above and bottom the notch plane, was scanned; because we wanted to have a view of the entire specimen in its transverse dimension and study the vicinity of the notch plane. At the end of the three-points bending tests the specimens were broken in two pieces. So the two pieces were first glued with a resin by trying to minimise the resin width all along the fracture surface. The 50 mm of the specimen height is scanned through 1700 slices; that means that the increment of displacement per slice is 0.0294 mm.

One should know and notice that the studied specimens don't have the same thermo-mechanics history. The specimen studied at 20°C has been dried at room temperature in laboratory cure conditions during five months and only underwent mechanical bending test. The second specimen has been only heated at 500°C and cooled and did not underwent mechanical tests. The third specimen has been heated at 700°C and cooled and underwent mechanical test as the first one at ambient temperature. So only the first and the third were glued because the second was entire. Strictly speaking fracture or damage observed on the specimen at 500°C is exclusively due to thermal effects whereas for the specimen at 700 mechanical effects are also present.

2. RESULTS AND DISCUSSION

The study has focused in the central zone of the notched specimens. Figure 1 exhibits a diagram where the three considered zones are displayed. The slice zone located in the vicinity of the notch plane is of special interest.

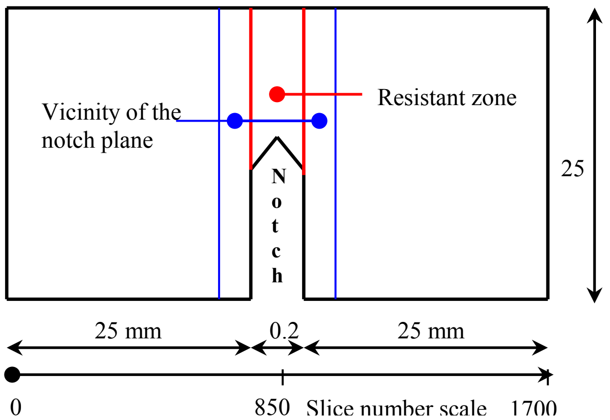


Figure 1 : Schematic front view representation of the central zone of the studied notched specimen with the identification of the different interesting parts.

2.1. 2D and 3D views of the heated specimens

The direct observations of the 2D slices show the presence of two phases appearing black or very dark and the other grey (figure 2). The black phase can be identified as porous areas and the grey phase as the matrix initially composed of cement, sand and silica fume. In the grey phase some dark or clearer spots appear but are difficult to identify. It is not possible to identify the granules of sand on the slices of the specimen at 20 and 500°C. The matrix seems to be continuous and homogeneous. At 20°C the pores are rounded and several of them are big sized.

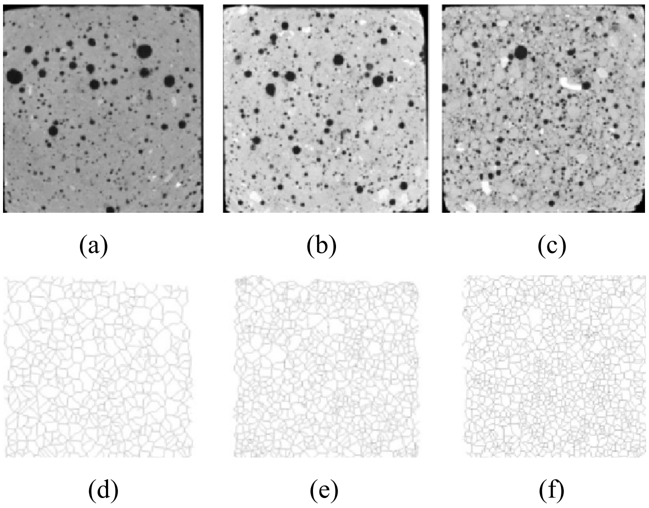


Figure 2 : Typical 2D view of the micro-tomography slices in the current zone, near the vicinity of the notch plane and in the resistant zone for (a) no heated material, (b) material heated at 500°C, and (c) material heated at 700°C.

At 500°C it seems that the number of pores has increased and their size has decreased. But at 700°C, a granular frame covering all the surface of the sample appears. That means that a homogeneous granular phase has appeared since this temperature. The examination of slices located in the vicinity of the notch plane shows presence of defects such as big pores especially near the inner walls of the notch. During the fabrication of the notched specimens, a 0.2 mm width was incorporated in the mould in order to form the notch. The presence of this blade apparently disturbs the concrete mixture by producing bubbles that results in pores after the hardening of the material. In other words, the porosity defects are the consequences of the presence of the blade and the phenomenon can be qualified as a wall effect. The specimen resistance is weakly affected by these defects since these zones of the specimen are weakly stressed during the three-point bending tests.The areas of the resistant section seem to reveal no defect or heterogeneity. The forming blade seems not to influence the homogeneity of the mortar located in its plane. This information is very important since sections located in resistant area bear essentially the load during the three-point bending test. In order to catch a different view from 2D slice, various types of reconstructed 3D images in zones surrounding the notch plane was performed (figure 3(a)).

The 3D view is impressive and visualizes essentially the macro-crack path across the specimen for tested specimens. It seems clearly that in general the crack propagates through the resistant zone of the notched specimen and generates a rupture surface more or less tortuous, not planar but remaining in the notch plane vicinity. In fact, during the propagation, the crack follows the weakest zones in the more stressed areas through or around aggregates and the shortest path is not the weakest. The advantage of the 3D reconstructed images is its ability to visualize 2-D sections through whatever plane and acquire then very relevant information.

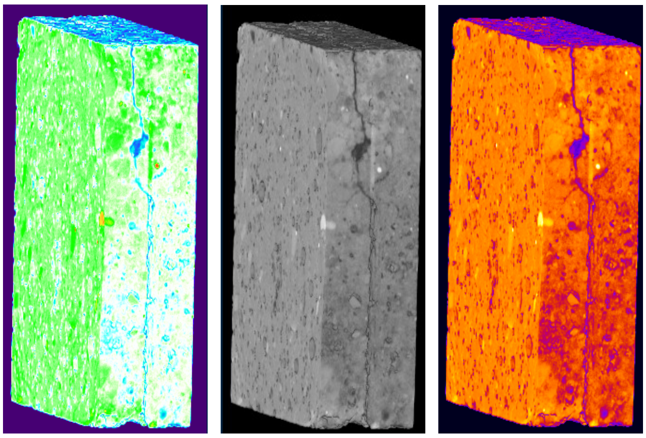


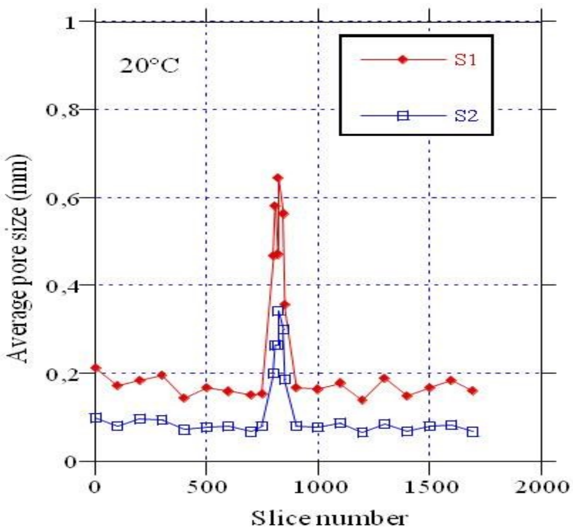
Figure 3 : Typical reconstruction of a 3D view of a portion around the notch plane of the non heated specimen (20°C) in various filter restitutions.

2.2. Determination of the area fraction of the pores

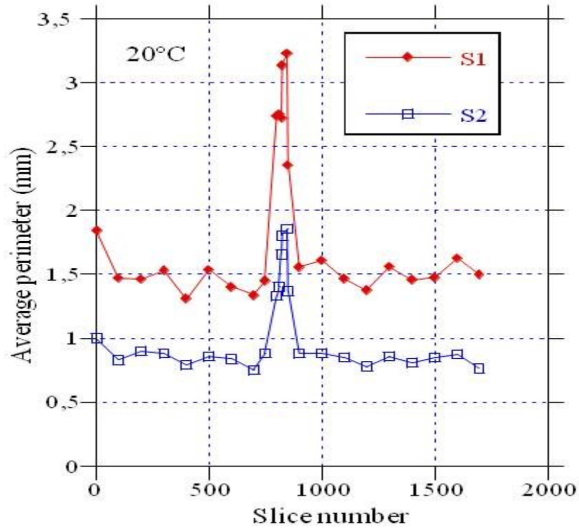
2.2.1. Methods

After the qualitative observations from the images, an attempt has been done to quantify the homogeneity of the material through the porosity parameter. The method is based on a strong assumption which is that all zones appearing black in the slices are assumed to belong to a pore. In these conditions macro-cracks as well as very dense inclusions that also appear black are included in the numbering.

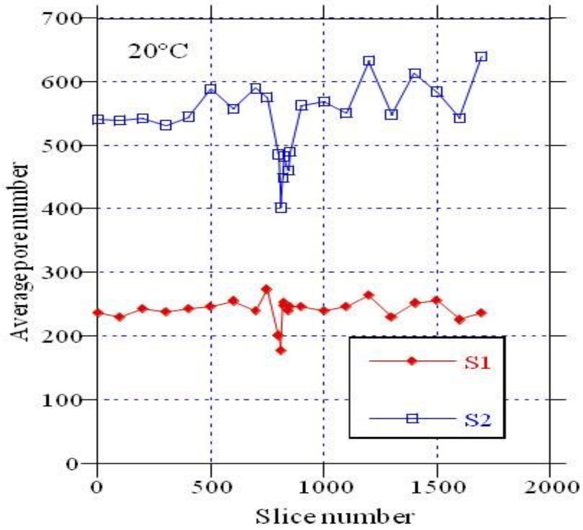
The inclusion of macro cracks is not dramatic or unrealistic as they form a sort of porosity. The inclusion of some very dense particle would lead to false results. But through also the slices that have been carefully examined, no such dense inclusion has been observed. As we said previously the matrix is rather uniformly grey. In other words or assumption in the case of the high mortar studied is rather realistic and acceptable. The method consists then in converting a 8-bit image of a slice into binary black and white image, the pore being black.



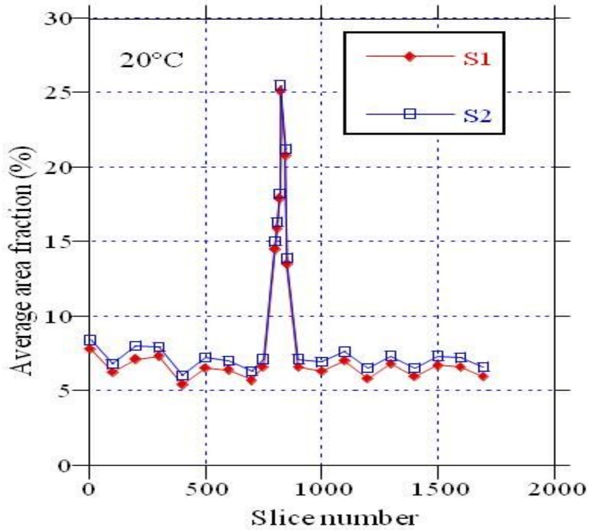
(a) Average pore size



(b) Average pore perimeter



(c) Number of pore enumerated



(d) Average area fraction for the specimen at 20°C

Figure 4 : Influence of the threshold 0.03 mm-Infinity (S1) and 0-Infinity (S2) on the characteristic parameters issue from the image analysis of the micro-tomography slice

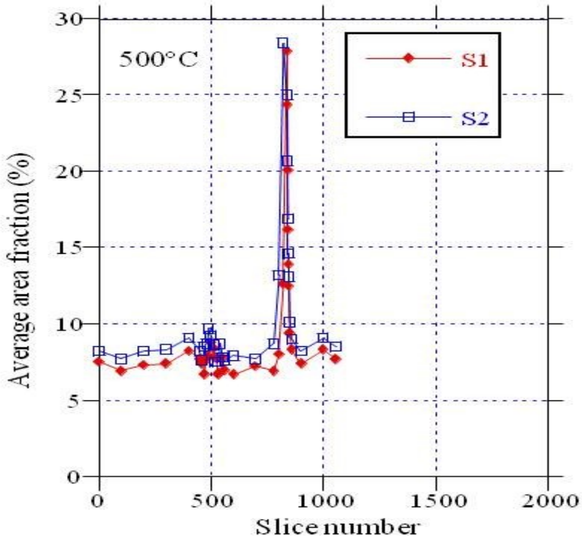
This binary image is then subjected to image analysis with image J in which all the black particles are numbered, measured in area, perimeter and size. A summary of the statistics of the key parameters is then listed: number of particles, average size, average total area, average perimeter, and average area fraction of the particles. In the present case particle should be replaced by pore. The area fraction is the ratio of the total area of the pore to the total area analysed. This area fraction is something like the porosity of the material. One of the questions is what is the relation between the average area fraction and volumetric porosity? However this question is important, in fact in this study we won't focus on it since we just want, first, to know whether this parameter is quantitatively pertinent and, second, to know whether they evolve significantly with increasing temperature.

It is displayed in figure 4 the evolution of the average pore size, the average pore perimeter, the number of pore enumerated and the average area fraction, respectively, with the slice number for a specimen tested at 20°C. A variation of 1 in the slice number corresponds to a distance variation of 0.0297 mm. In fact the slice number corresponds to a distance from a define reference; the notch being located at the mid-distance of the scanned area. As the average total area and the area fraction are redundant information only area fraction has been kept.The area fraction is a global parameter related to the global porosity of the material. The number of pores and the average pore size give information on the mean size of the pore and somewhat "how particles are distributed around this mean value". The average perimeter gives information on the circularity of the pore, a great value means that the pores are big or have very tortuous shapes.

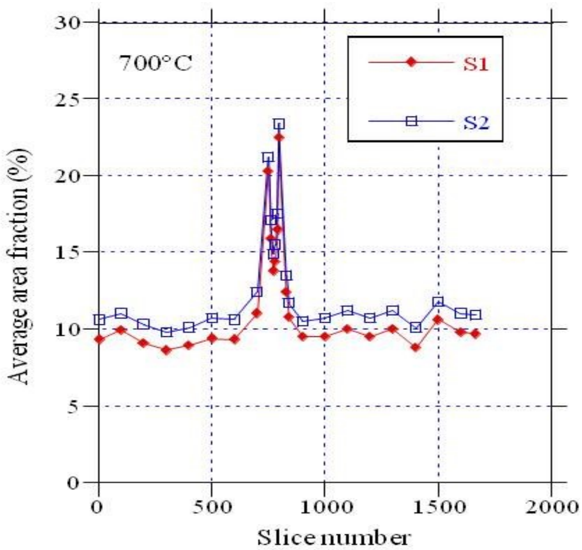
All the figures show a central zone in the vicinity of the notch plane where evolution is very sharp and two zones far from the first one where the parameter oscillates more or less along a quasi constant value. The waves amplitudes are rather small for area fraction particle size and perimeter what traduces spatial constant value, so a certain homogeneity of the material. They are more pronounced for the number of particles but apparently with a minimum effect on the average geometrical parameters. The analysis of the central part is interesting. The analysis of entire slices close to the notch plane shows that all the parameter increases except the number of particle that decreases. The question is why this rise? Is-it related to the effect of the temperature? But in this case, why is observed at 20°C where the specimen was not heated. Is it related to the mechanical loading? But it is also observed at 500°C where the specimen has been just heated. Is the influence of the resin that distorts the measurement? This assumption is probably true. The resin appears rather black and some of the matrix part has been coloured black and has then contributed to increase considerably the area fraction factor. But when one analyses the slice in the resistant part of the notched specimen (i.e. plane of the notch) the value is the same as the one found far from the central zone. Our explanation is that the increase observed in the various parameters around the central part of the specimen is due to the existence of macro-porosity consequence of the wall effect induced both by the mould and the blade during the forming of the specimen. That is why the increase in the various factors is observed at 500°C where the material has just been heated and the resin was not present. At temperatures 20 and 700°C where the resin is present in the central zone, the presence of the resin has just amplified the phenomenon. This is consistent with the evolutions observed.

2.2.2. Influence of the threshold

One of the most important things to do in the image analysis process is to determine the threshold value for the analysis. Particularly you have to choose the particle size you wish to take into account. Initially as one pixel corresponds to a 0.30 mm in distance, we choose to analyse particle with size more than 0.03 mm.



(a) At 500°C

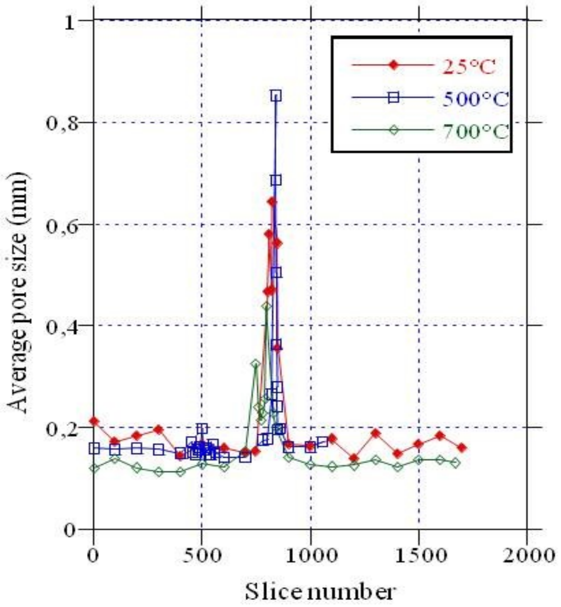


(b) At 700°C

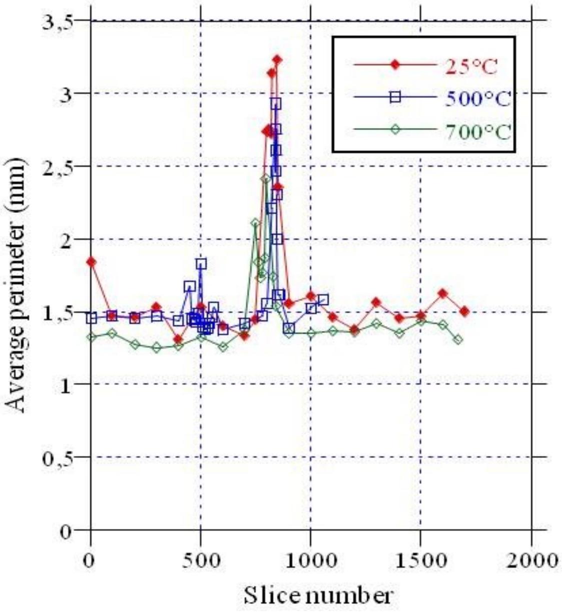
Figure 5 : Influence of the threshold choice on the average area fraction for the specimens heated at (a) 500 and (b) 700°C.

But it appears rapidly that the number of particle is very sensible to that parameter. So the threshold has been set to 0 mm and all the study has been conducted. You can see in figure 5 the evolution of the different parameters with a threshold set to 0.03-infinity mm (S1) or set to 0.-infinity mm (S2).

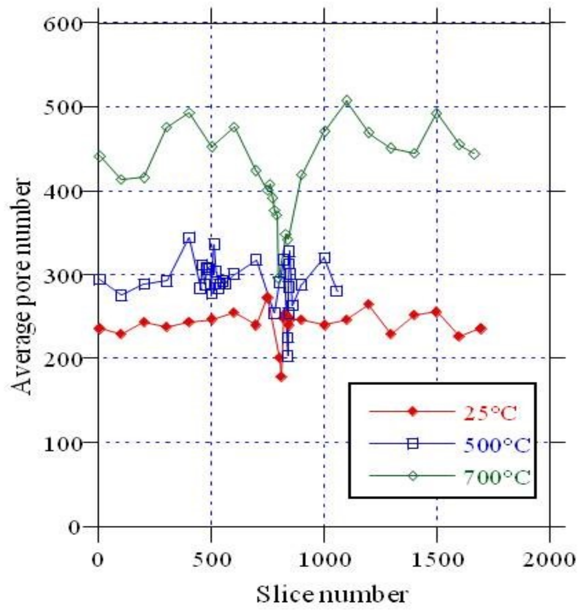
It appears logically that the number of particle enumerated increases whereas the average pore's size and pore perimeter decreases when the threshold changes from S1 to S2. The change is visible for all the parameters but it is particularly visible on the number of pores enumerated. The area fraction is affected but in a lesser level. That's why the S2 threshold has been retained for the next results to be analysed.



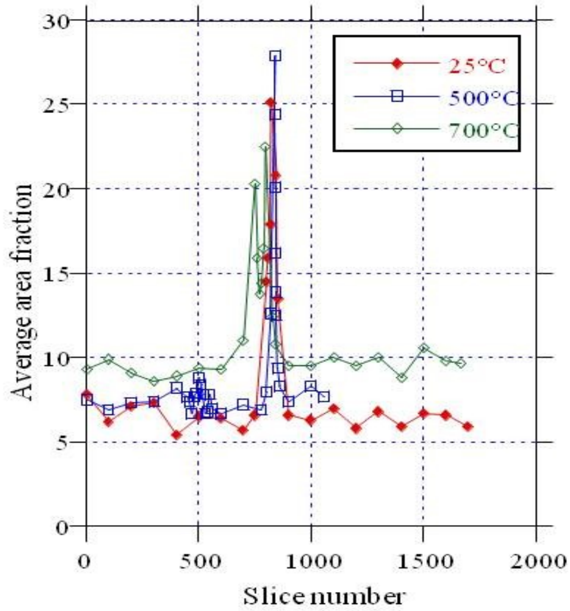
(a) Average pore size



(b) Average pore perimeter



(c) Number of pore enumerated



(d) Average area fraction.

Figure 6 : Influence of the heating temperatures (20, 500 and 700°C) on the characteristic parameters issue from the image analysis of the micro-tomography slice

2.2.3. Influence of the heating temperature

In figure 6 are reported the evolution of the various parameters along the scanned zone for temperatures 20, 500 and 700°C. The number of pores increases whereas the average size and the average perimeter decrease with increasing temperature. And the global area fraction increases with increasing temperature but in a lesser extend than the pore number for instance. In order words these results show that when temperature increases the porosity increases and that the pore distribution also changes; the number of pores increases about their size meanly decreases.

These quantitative results are consistent with the qualitative observation made previously from the direct observation of the images. This is also consistent with some results reported elsewhere (5) on the evolution of porosity, as measured by a mercury porosimetry, as a function of temperature (2). One must be cautious with such a comparison because in this case a partial part of the porosity is accessible. However, our present study focuses on the bulk characteristics hence, making it very interesting.

On the other hand, in the central zone located close to the notch plane, the number of particles decreases while the area fraction, particle size and perimeter increase for all studied temperatures. This means that this zone is more porous than elsewhere and this may be due to the wall effect caused by both the mould and the blade during the forming of the specimen. Direct observations show that the biggest pores are located near the blade and the mould walls. We suppose that these are not cause by the temperature or mechanical loading instead they are specimen preparation defects.

CONCLUSION

The micro-tomography technique has been used to analyse notched specimens of high performance mortar that have been subjected or not to three point bending tests at various elevated temperatures. The results of the tests have been reported in previous papers dealing with the evolution of the material tenacity parameters with increasing temperature. The present study is aimed to examining the specimens through micro-tomography and characterizing their eventual geometrical defects and to evaluating the ability of this technique to detect the evolution of the material porosity with temperature. We conclude that:

- the material is generally homogeneous
- there is a wall effect in the specimen near the notch that induced an increase in the porosity
- the porosity in the resistant zone was the same as the global porosity so the three-point tests are valid and reliable.
- when the heating temperature increases the average particle size and perimeter increase as well as the global area fraction accordingly.

The micro-tomography can reveal the same results as reported in the literature.

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