

CHARACTERISATION OF THERMAL-LOADED CEMENT-BASED COMPOSITES BY COMBINED TIME-LAPSE TOMOGRAPHY AND THE FOUR-POINT BENDING TEST

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ABSTRACT

Quasi-brittle materials like cement-based composites, rocks, and bricks are subjected to a number of environmental loadings throughout the life cycle of buildings. For instance, fluctuation of the ambient temperature (climatic cycles or fire) causing a variety of physical and chemical transitions resulting in structural changes and affecting the mechanical properties. In this work a special mixture containing glass spheres and Portland cement was evaluated by a combination of four-point bending and time-lapse X-ray computed tomography to verify the feasibility of this novel combined method.

The effect of temperature on the behavior of investigated material in terms of sphericity of the present glass spheres and the way of crack propagation under load together with its influence to mechanical fracture parameters was studied. The described methodology was used especially to be able to monitor these changes throughout the loading process, as the characterization of the fracture surface using conventional optical methods is possible only after the complete fracture of the specimen and total damage of used material results in loosening of the matrix and filler to such an extent, that the results of these methods may be very distorted. It has been proven that the developed method can be used to characterize the internal structural changes in building materials and thus contribute to the understanding of the fracture processes during mechanical loading. Up to 600°C the glass spheres stay spherical and the crack is propagating through the interfacial transition zone, while at higher temperatures the glass loses its shape and the newly formed pores cause also cracks within the inclusions. The relationship between compressive strength and the maximum loading temperature was confirmed.

KEYWORDS

Fine-grained cement-based composites, Quasi-brittle material, X-Ray computed tomography, Instrumented four-point bending test, Crack path

INTRODUCTION

Cement-based composites belong to very often used building materials [1, 2]. Structures utilizing these composites were commonly designed assuming normal service temperatures. However, high temperatures carrying out on cement-based composites cause a wide range of physical and chemical processes, which result in changes in the micro-structure of composites and thus affect its mechanical properties [3]. According to the fracture mechanics theory, cement-based composites show quasi-brittle response. Specimens made from these materials have the ability to

carry the load even after the deviance from a linear part of force–displacement diagram until the maximum load and then the decrease of the load follows until the final failure [4, 5]. One of the reasons for this response could be the existence of the interfacial transition zone (ITZ). This well-known zone around aggregate particles of a few micrometres in size has specific features. These are mainly higher porosity and calcium hydroxide contents compared to the bulk matrix [6, 7, 8].

Characterization of quasi-brittle materials concerning their resistance to crack formation and its propagation is within the scope of interest for a long time [9]. Various testing methods based on different experimental configurations and sample geometries have been introduced, but intensive research is still devoted to assessing parameters affecting the reliability and reproducibility of the obtained results. For a closer understanding of the parameters influencing the process of crack evolution, a method based on monitoring of parameters around the crack tip (fracture process zone, FPZ) was developed [10, 11]. In addition, other experimental techniques based on acoustic emission, holographic and infrared interferometry, and X-ray imaging are used to investigate FPZ characteristics for analytical models and numerical simulations purposes. Recently, studies have been published [12, 13], which are focused on the investigation of FPZ in silicate composites and rocks using radiographic observation of the specimens loaded by three-point bending, both by simple 2D transmission radiography and advanced time-lapse computed tomography (4D-CT) [14, 15].

These studies demonstrate that computed tomography (CT) can be used complementary to the mechanical tests thanks to the obtained information about the shape of the fracture process zone and the spatial distribution of cracks within the internal structure [16]. Concluding these results, the appropriate 4D-CT setup in conjunction with optimized in-situ loading allows acquiring several CT measurements within softening of the loaded specimen caused by the evolution of the material damaging, while the detailed 3D model of developed FPZ and cracks is obtained. However, the methodology is highly demanding, both in terms of the CT scanner and loading device. Besides the difficulties arising from the interconnection between the three-point bending test and the 4D-CT, main problems arise from the mechanical contact between the tested specimen and supports. High rigidity of the loading device is essential since its deformation can cause a sudden collapse of the investigated specimen.

Another obstacle directly influencing the quality of the tomographic reconstruction and its resolution is a typical arrangement of the standard three-point bending device: the specimen is oriented horizontally on two supports, while the vertical movement of the third support induces loading. The rotation axis in a typical tomograph for industrial use is vertical. Thus, when the loading device is mounted on the rotation platform, the sample is oriented orthogonally to the rotation axis and during the rotation, which is indispensable for CT data recording, there is a significant variation in the thickness the X-ray radiation penetrates (transmission length). Variable transmission length affects the quality of the tomographic reconstruction. In addition, the dimensions of the loading chamber together with the available radiation power and the desired image resolution greatly limit the possible length of the examined specimen. The development of a new four-point bending device has largely eliminated the problems described. A novel device for four-point bending of the specimens is designed in the way that the sample is oriented vertically, i.e., its longitudinal axis is parallel (ideally identical) to the rotation axis. Hence, the loading force is applied in the horizontal direction. The transmission length variation is significantly lower compared to the specimen oriented orthogonally to the rotation axis.

The paper presents the use of such a four-point bending device for obtaining 4D-CT during loading, extracting information at various stage of the load and studying of the intrinsic structure and fracture properties of a specially designed, fine-grained cement-based composite exposed to thermal stress. This material was chosen with respect to minimization of variable parameters for subsequent numerical simulations. X-ray computed tomography provides various information about the material structure, such as the volume fractions and geometry of constituents, and the formation of cracks (in matrix / inclusions / interfacial transition zone) caused by the thermal

loading. On the other hand, the instrumented loading device provides information about the mechanical behavior of the investigated material. A time-lapse series of computed tomography data were recorded during 4-point bending test applied on the notched specimen (stress concentrator) to provide a comparison of differently thermal-loaded material and its behavior during mechanical loading including visualization of the evolution of the spatial structural changes during loading.

Each investigated specimen was subjected to different loading temperature before the flexural test to investigate its influence on the mechanical properties and cracks evolution. Specimens were manufactured from a special mixture containing glass spheres and Portland cement. This material can be taken as a binary material in terms of present phases and their topology. The possibility to accurately geometrically describe the spheres makes it an ideal material for numerical simulations (not subject of this paper) concerning fracture mechanics parameters. Glass spheres with a diameter of 2 ± 0.2 mm were used as a filler, investigated specimens were subjected to temperature loads up to 100, 200, 400, 600 and 1000 °C.

MATERIALS AND METHODS

X-Ray computed tomography

X-rays, when passing through the material, are attenuated, depending on the elemental composition, density and thickness (transmission length) of the specimen. An intensity image is then observed at the detector. X-ray computed tomography (XCT) employs a set of 2D X-ray images (radiograms, projections) of the object exposed from different directions (angles) around the examined object (typically hundreds to thousands). To obtain a 3D virtual model of the irradiated scene, several methods can be used to process the set of projections, one of the classical being the filtered back projection method. The resultant reconstruction gives us a virtual 3D model of the investigated volume providing the true spatial information about its geometry and internal structure. In the standard industrial cone-beam tomography, the object to be examined is fixed on a rotary stage, which rotates around its axis in a step-wise movement, while the X-ray tube and area imaging detector are static and acquire one projection at each rotation step.

Presented X-ray CT results were obtained by an advanced "on-the-fly" tomography method, where the investigated specimen rotates continuously and one rotation follows the other. This approach makes it possible to obtain a sufficient number of projections in a sufficiently short time without the delays that take place in the step-wise scanning due to the positioning of the precise rotary stage and an appropriate exposition triggering. Furthermore, continuous loading of the specimen eliminates relaxation effects present during intermittent loading, when loading is stopped to perform a revolution of the specimen for one tomographical scan. The relaxation not only presents a problem during the evaluation of the experiment but may also be one of the causes for the sudden formation of macro-crack and failure of the specimen even during initial stages of the experiment. The result is a series of time-dependent tomography datasets. When the rotation time is set carefully with regard to the exposition time, the effect of the movement of the object during the exposition does not cause significant difficulties in the reconstruction.

Tomographic measurements were made using a patented multipurpose modular tomographic device TORATOM (Twinned ORthogonal Adjustable TOMograph) at the Institute of Theoretical and Applied Mechanics of the Czech Academy of Sciences (CAS), Centre Telč, employing the novel device for 4-point bending testing, depicted in Figure 1. TORATOM combines two pairs of imaging lines, each consisting of an X-ray tube and an imaging detector, in an orthogonal arrangement with a shared rotary stage, allowing thus an acceleration of the data acquisition process. The device has fully motorized axes for adjusting the system orthogonality in any position and setting the X-ray tube–specimen–detector distances to set the magnification in a range from about 1.2× to 100× (even more in special cases). The device is designed modularly

and allows the quick exchange of various available types of detectors (flat panels, photon-counting detectors). With respect to the used detector type and its native pixel size, it is possible to change the resolution of the CT reconstruction, i.e. the size of one spatial point of the model (voxel), from about 180 μm to less than 1 μm . Very high and stable resolution is achieved also thanks to an anti-vibration table on which the entire assembly is installed and the use of a high-precision tomographic rotary stage. The TORATOM device is unique in the field of industrial CT even on a global scale, as evidenced by the fact that TORATOM is patented at European level (EP 2835631 B1).

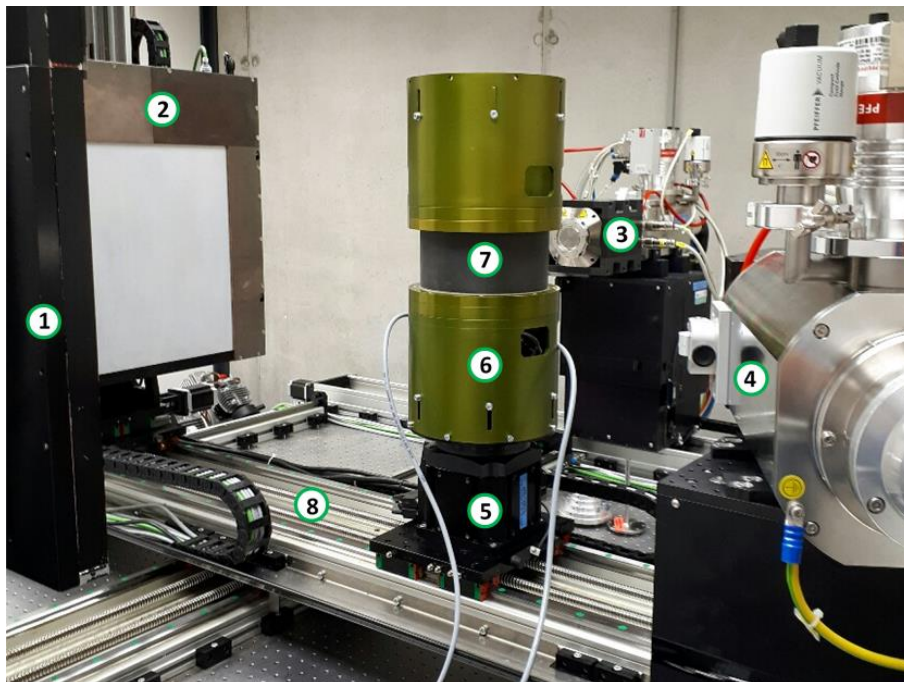


Fig. 1 – TORATOM tomography system employing novel device for 4-point flexural testing: 1) Detector holder, 2) Large area flat panel detector, 3) Nano-focus transmission type X-ray tube, 4) Micro-focus reflective type X-ray tube, 5) High-precision tomographic rotary stage, 6) Device for 4-point flexural testing during CT, 7) Loading chamber accommodating the specimen, 8) Active damped anti-vibration table with high-precision CNC positioning system

Novel device for 4-point flexural testing during 4D computed tomography

Modular device for 4-point flexural testing during 4D computed tomography recently developed at the Institute of Theoretical and Applied Mechanics of the CAS consists of three main components: a pair of motorized loading units with integrated external supports of the four-point bending configuration, a pair of static inner supports of the four-point bending configuration, and a cylindrical supporting frame. During the test, the internal supports are in a fixed position, while the outer movable supports attached to the closed-loop high-precision positioning system perform the loading. The synchronized driving units are equipped with linear encoders and load-cells for measuring the force on both external supports. The frame of the loading chamber accommodating the specimen is made of a 1.95 mm thick carbon-fiber composite that has sufficient stiffness and low X-ray attenuation. Simplified scheme of the device solution in partial section and its orientation in tomographic assembly is shown in Figure 2.

Such design brings advantages against the standard horizontal orientation of the three-point bending arrangement. The specimen length can be extended without the need for increasing the diameter of the loading device, which would necessarily result in a lower achievable resolution

of the obtained radiograms. As a result, it is possible to investigate a wide range of materials, because specimens having representative volume elements can be used. Furthermore, during rotation of a vertically oriented sample, low variability of the X-ray attenuation is achieved leading to an increase in quality of reconstruction. The relative position of internal and external supports can be adjusted independently, which allows the selection of the optimal setup geometry depending on the specimen characteristics (i.e. overall dimensions, the shape of the cross-section, notch geometry, etc.) and material. Input and output wiring is equipped with a pair of slip rings that enable an unlimited number of revolutions of the whole device on the rotational stage of the tomographic scanner. The maximum loading capacity of the device is 1500 N per support, position accuracy and repeatability is better than 10 μm with a minimum increment lower than 1 μm . The device is closed-loop controlled by the proprietary Linux CNC software running on the real-time kernel [16]. As shown in Figure 1 and Figure 2, during the tomographically observed loading, the longitudinal axis of the investigated sample is parallel (nearly identical) to the axis of rotation of the device and the rotational stage.

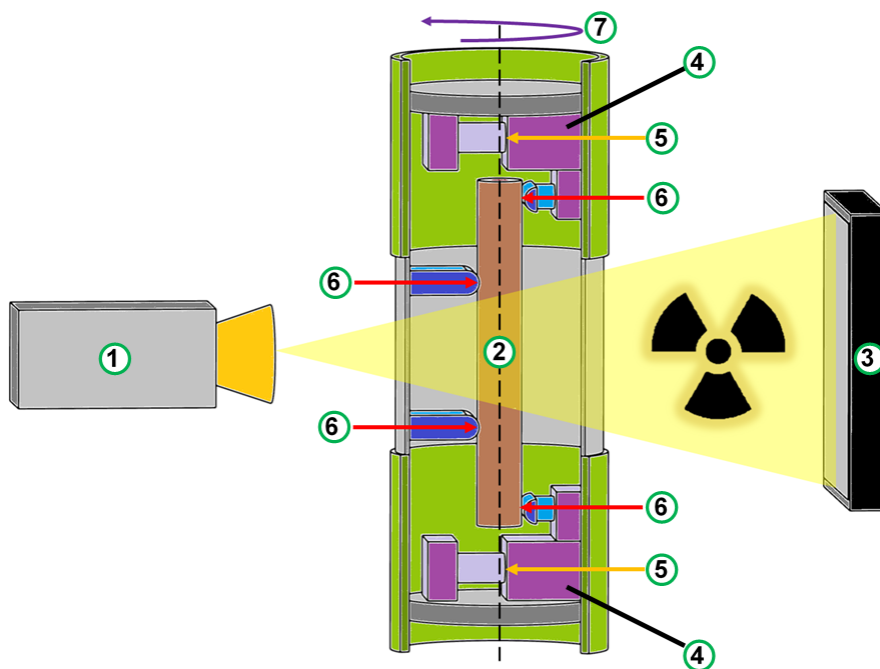


Fig. 2 – Novel device for 4-point flexural testing during 4D computed tomography: 1) X-ray tube, 2) Specimen with the highlighted field of view, 3) Imaging detector, 4) Precise linear drive, 5) Outer supports displacement, 6) Applied force, 7) Rotation of the device during CT

Test specimens and procedure

In total 6 sets, each of 4 test specimens with dimensions of 20 mm \times 40 mm \times 200 mm were prepared from a specially designed, fine-grained cement-based composite. The fresh mixture was made using sodium-potassium glass spheres with a diameter of 2 ± 0.2 mm, Portland cement CEM I 42.5 R (cement factory Mokra, Czech Republic) and water at the weight ratio of 3:1:0.35. After pouring into the prepared silicone molds and compacting, the specimens were left covered with PE foil for one day and then kept in water for 28 days.

Mature samples were dried at the temperature of 100 $^{\circ}\text{C}$ and further kept in ambient temperature and constant humidity in a desiccator. One set of specimens was kept as reference samples (100 $^{\circ}\text{C}$), while the rest sets were exposed to the pre-set temperatures of 200 $^{\circ}\text{C}$, 400 $^{\circ}\text{C}$, 600 $^{\circ}\text{C}$, 800 $^{\circ}\text{C}$, and 1000 $^{\circ}\text{C}$. The range was chosen to capture the process of thermal

degradation with respect to the maximum temperature load provided by the furnace used. For each set, the thermal loading began at 20 °C with an increment of 5 °C / min until the pre-set maximum was reached, which was then maintained for an additional 60 minutes. Temperature loading was delivered using laboratory furnace Clasic 5013 with heating coils on 3 sides. After the specimens were left to cool down to room temperature, they were equipped with an initial notch as a stress concentrator and kept at a constant temperature of 20 °C.

From each set, one specimen was investigated using the X-ray CT with the procedure and results covered in this paper. Other three specimens of each set were used for another investigation [17], which is not described in this paper; however, their compressive strength was measured and used. First X-ray scanning was performed on one specimen from each set after drying and before the temperature load, giving thus 6 CTs. Second X-ray scanning was obtained on each investigated sample after the temperature load, giving thus 6 CTs (even the reference (100 °C) was measured again to observe the potential microstructure changes caused by maturing). The details of the scanning procedure can be found below. The samples subjected to temperatures higher than 200 °C were thermally damaged to the extent that the intended four-point bending experiments were not possible. Thus, only the reference (100 °C) and 200 °C specimens could be subjected to the in situ 4-point bending test in the course of the time-lapse CT, giving thus two sets of 4D CTs. The procedure is in detail described below.

X-ray CT scanning procedure before and after temperature loading

For a qualitative assessment of the damage at the microstructural level of the investigated material caused by the high-temperature loading, standard X-ray tomography measurements were performed with the intact and thermally loaded specimens, i.e. for each specimen a CT was performed before and after the thermal loading procedure. A microfocus X-ray tube (XWT-TCHR-240, X-ray WorX, Germany) operating at 160 kV and the target power of 45 W was used together with the flat panel detector (Dexela 1512NDT, Perkin Elmer, USA) with the active area size of 145.4 × 114.9 mm, a pixel matrix of 1944 × 1536 and a native resolution of 74.8 μm per pixel. The geometry parameters of the tomographic assembly were set to obtain the best possible resolution with respect to the field-of-interest size and the detector area. The *X-ray tube – imaging detector* distance was set to 600 mm and the *X-ray tube – specimen* distance was set to 200 mm providing the geometrical magnification of 3×, which leads to the edge length of the spatial point (voxel) of approx. 25 μm in the resulting 3D reconstruction.

4D CT during the four-point bending measurement

Selected specimens were subjected to four-point bending as a displacement-driven experiment. The distance between the outer supports of the loading device was set to 178.8 mm, the inner supports distance was set to 75 mm. Before the testing procedure, the specimens were pre-loaded by 3 N initial force and the proper geometrical position was checked radiographically. The tests were performed with 0.25 μm/s loading rate up to a displacement value of 1000 μm and 1500 μm in the 100 °C and 200 °C sample, respectively. This loading rate allows on-the-fly imaging mode of the experiment. The ultimate forces of approximately 30 N and 15 N were obtained for 100 °C and 200 °C samples, respectively. Force and displacement data were recorded at the sampling rate of 100 Hz.

For imaging, the same detector and X-ray tube as in the previous case were used. However, the X-ray tube was operated at the accelerating voltage of 180 kV and the target power of 40 W. The geometrical parameters of the tomographic assembly were set to obtain the best possible resolution. The *X-ray tube – imaging detector* distance was set to 480 mm and the *X-ray tube – specimen* distance was set to 200 mm providing the geometrical magnification of 2.4× which leads to the edge length of the spatial point (voxel) of approx. 31.25 μm in the resulting 3D

reconstruction. During the loading under continuous rotation of the loading device on the rotary stage, 17 tomographic datasets of 600 projections were taken. The acquisition time for each projection was 400 ms, giving the time of one rotation 240 s.

Compressive strength value measurement

The remaining parts of the specimens subjected to four-point bending were used for measurement of the compressive strength values. An auxiliary loading mechanism equipped with two square steel plates (top, bottom) was installed in a hydraulic test machine. The specimens were placed between the plates so that the load was applied perpendicularly to the direction of compaction. The specimens were loaded continuously until the failure. The test was carried out in accordance with the principles of standards ČSN EN 196-1 [18] and BS 1881: Part 119 [19]. The compressive strength value was calculated taking the nominal cross-section area of $40 \times 20 \text{ mm}^2$. See selected photo in Figure 3 for illustration.



Fig. 3 – The compression test of specimens: The auxiliary loading device with two auxiliary platens was placed in the hydraulic testing machine with selected specimens prepared for the test

RESULTS

One of the important results is the evidence of the microstructural changes observed with the CT and its correlation with the compressive strength in the temperature loaded samples. X-ray CT proved to be a suitable qualitative tool for this kind of investigations. Some particular samples were placed into the 4-point bending device after the temperature loading and successive CT scans were performed during the loading (4D-CT). The procedure allowed to control and monitor force and displacement over time, giving the possibility of a direct match of the microstructure observed in the individual reconstructed 3D models with the corresponding loading parameters.

From the results of tomographic measurements of specimens before and after the thermal loading, the development of damage caused by the effects of high temperatures is apparent. While the specimen remains compact at $100 \text{ }^\circ\text{C}$ and $200 \text{ }^\circ\text{C}$, heating at $400 \text{ }^\circ\text{C}$ and $600 \text{ }^\circ\text{C}$ already causes the creation of cracks particularly within the interfacial transition zone (ITZ). Upon heating to higher temperatures, the shape of the glass spheres changes and the constituent cohesion deteriorates as the pore size increases and formation of the system of macrocracks causes their interconnection. Examples of the development of damage are shown in Figure 4.

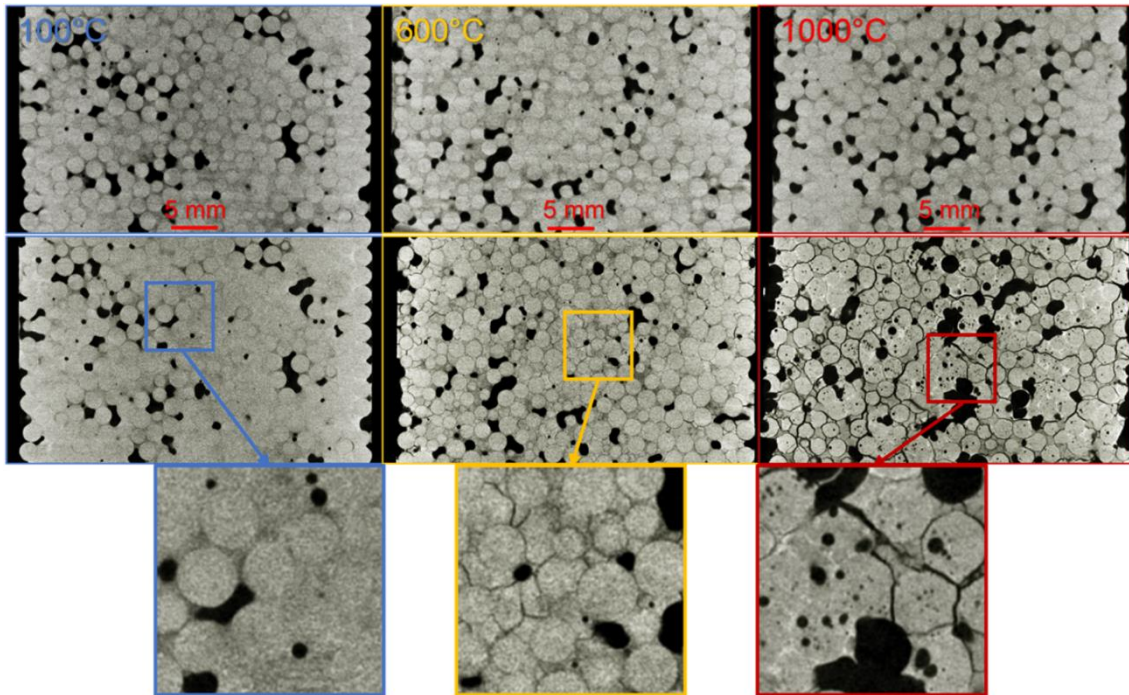


Fig. 4 – Example of visualization of the selected tomographic section for specimens before and after the thermal loading. In the upper part – the selected tomographic sections for specimens in the initial state, in the middle part – identical tomographic sections for specimens after the thermal loading, in the lower part – zoom to microstructure demonstrating progressive damage

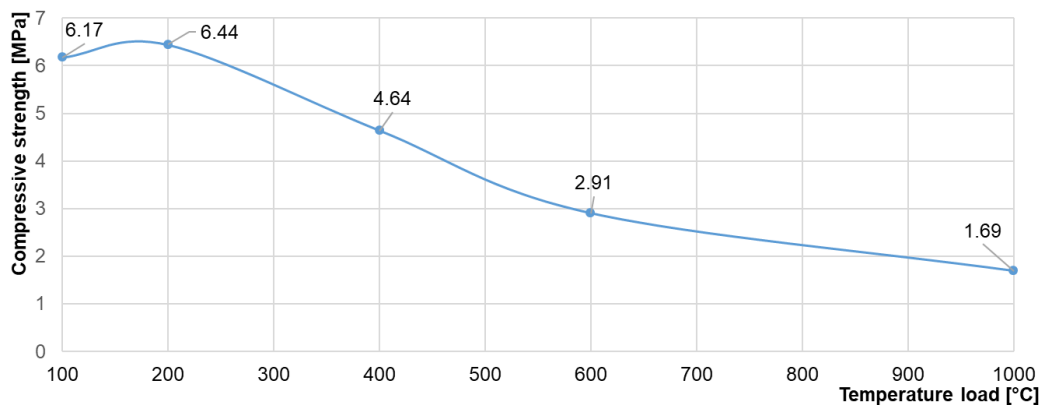


Fig. 5 – The maximum temperature dependence of the mean compressive strength values

Damage observed during tomographic imaging corresponds to the measured compressive strength values shown in Figure 5. The graph depicts the average values of the observed compressive strength measured on all the four manufactured samples in each set (for each temperature), i.e., even for the samples that were not exposed to the CT investigation.

Left part of Figure 6 shows the time dependence of loading force measured during the in-situ four-point bending experiments, while the right part of Figure 6 depicts the force-to-displacement curves. The thin lines in both subfigures show forces measured at individual movable support units. The average values of force are then plotted as the bold lines. The red bold curve shows the average force for the specimen loaded with 100 °C, the green bold curve gives the average force for the specimen loaded with 200 °C. The graph shows, that the resistance of the material to the damaging is decreasing with the temperature increase. The intervals depicted by

blue arrows in the force-time graph define the duration of individual tomographic scans during the loading. From each such tomographic scan, virtual 3D model of the examined specimen was obtained.

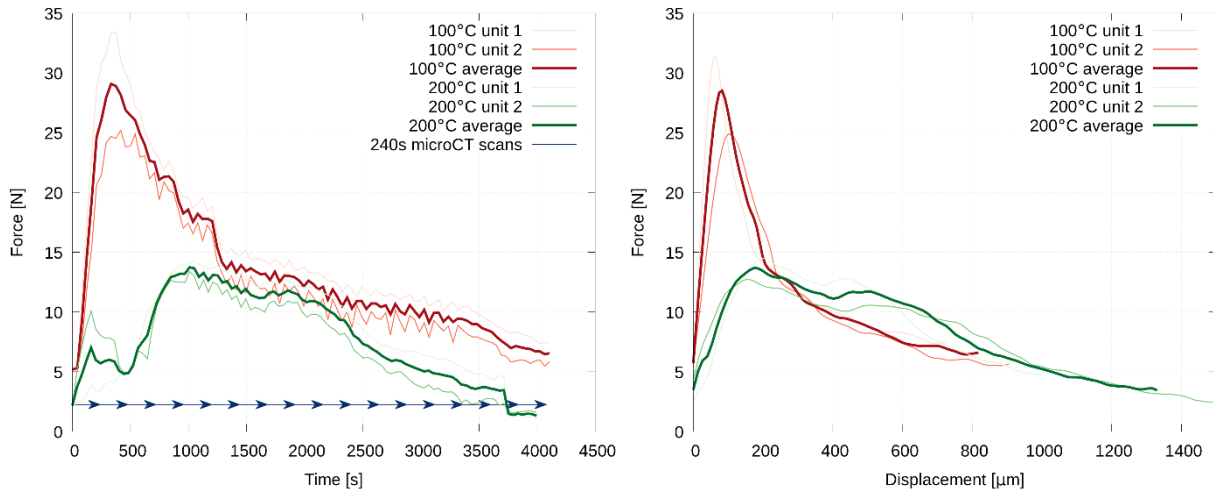


Fig. 6 – Time dependence of the loading forces magnitude obtained by 4-point flexural testing (left), and force-displacement diagram (right)

Based on the resulting 3D images, it is possible to investigate and measure the crack length and opening in individual states within the achieved resolution by the employment of advanced image processing methods including digital image correlation and digital volume correlation. A representative example of the visualization of crack development in 2D (one CT slice) at the beginning of the loading procedure, in the half of the loading procedure and at its end is shown in Figure 7.

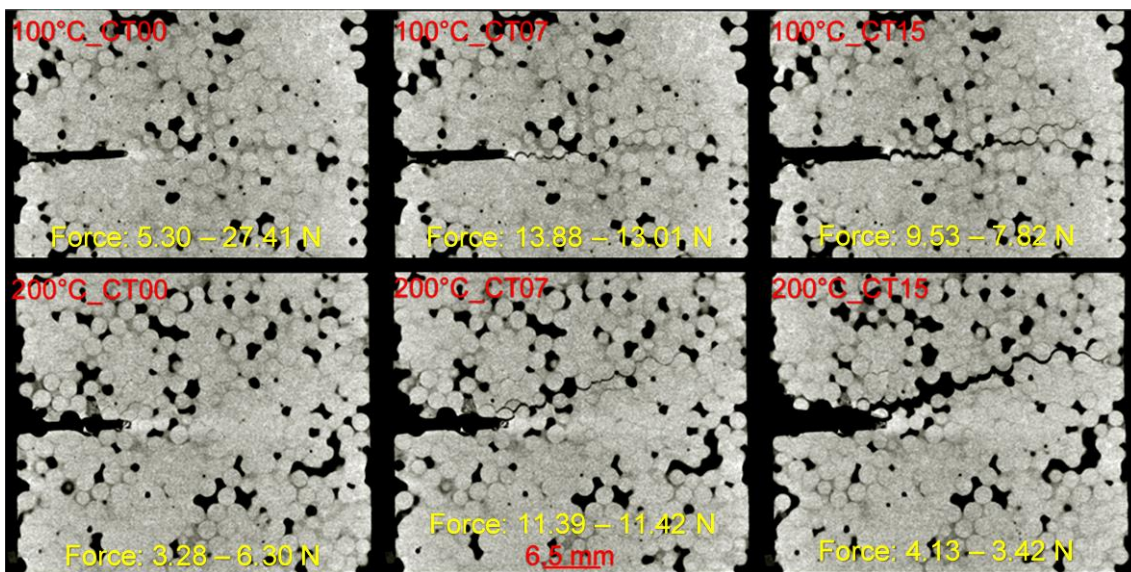


Fig. 7 – Example of visualization of the selected tomographic section showing crack initiation and propagation during loading. In the upper part – the specimen loaded with 100 °C, in the lower part – the specimen loaded with 200 °C

CONCLUSION

The effect of temperature load on the behavior of investigated material in terms of structural changes and the way of crack propagation under load together with its influence to mechanical fracture parameters was studied using a novel method combining four-point bending and time-lapse X-ray computed tomography. It has been proven that the developed method can be used to characterize the internal structural changes in building materials and thus contribute to the understanding of the fracture processes during mechanical loading. The time-dependent alteration in the values of the mechanical parameters together with 3D spatial image information characterizing the damage to the internal structure of the specimens loaded at 100 °C and 200 °C was observed, indicating a decrease in the resistance of the material to the damaging due to exposition to high temperatures.

For specimens loaded with maximum temperatures from 100 °C to 1000 °C, image information provided by standard X-ray computed tomography was evaluated before and after the thermal loading, showing that increasing the temperature leads to formation of cracks within the interfacial transition zone even without action of force. Up to 600°C the glass spheres stay spherical and the crack is propagating through the interfacial transition zone, while at higher temperatures the glass loses its shape and the newly formed pores cause also cracks within the inclusions. The relationship between compressive strength and the maximum loading temperature was confirmed.

ACKNOWLEDGEMENTS

The research was carried out within the framework of the project of the specific university research of the Brno University of Technology No. FAST-J-20-6506 and thanks to the financial support of the Operational Program Research, Development and Education of the Ministry of Education, Youth and Sports within the project No. CZ.02.1.01 / 0.0 / 0.0 / 16_019 / 0000766 (Engineering Applications of Microworld Physics - INAFYM).

Authors express their thanks to Dr. Hana Šimonová, Dr. Patrik Bayer, Ing. Michal Vyhliřdal, Ing. Tomáš Majda, and Mr. Miroslav Friedl, Brno University of Technology, Faculty of Civil Engineering, for the support during the preparation of the specimens and prof. Miloř Drdáký, Institute of Theoretical and Applied Mechanics of the Czech Academy of Sciences, for valuable comments during loading device development.

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