UDK 691:620.179.1
Professional article/Strokovni članek

ISSN 1580-2949
MTAEC9, 47(5)661(2013)

# MICROTOMOGRAPHY IN BUILDING MATERIALS

## MIKROTOMOGRAFIJA GRADBENIH MATERIALOV

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*Prejem rokopisa – received: 2012-05-14; sprejem za objavo – accepted for publication: 2013-03-13*

X-ray computed microtomography (micro CT) is now widely available for a non-destructive evaluation of various building materials. It can provide information on the internal structure of small samples, with the maximum resolution of about 1 μm. For this study, samples of different building materials, such as concrete, mortar, steel, soils and asphalt composites, were selected. Their microstructure and pathology (corrosion) were determined mainly from the applicative point of view.

Keywords: X-ray computed microtomography, micro CT, 3D, imaging, building materials

Rentgenska računalniška mikrotomografija (mikroCT) je postala široko uporabna za nedestruktivne analize različnih gradbenih materialov. Zagotavlja informacije o notranji strukturi manjših vzorcev z največjo ločljivostjo 1 μm. V tej študiji so bili izbrani različni materiali, kot so beton, malta, jeklo, zemljina in asfaltni kompozit. Glavni cilj tega prispevka je oceniti mikrostrukturo in patologijo (korozijo) gradbenega materiala z uporabo rentgenske mikrotomografije, predvsem z vidika uporabe.

Ključne besede: rentgenska računalniška mikrotomografija, mikroCT, 3D, slikovna obdelava, gradbeni material

## 1 INTRODUCTION

Since the end of the 19th century, when Wilhem Conrad Röntgen discovered the existence of X-rays,[1] huge developments have occurred in their application. Computed tomography was introduced in the late 1970s,[2] following the evolution of computer science. CTs were most commonly used in the fields of medicine and industry. In recent years a big step forward, in this field, has been accomplished with the introduction of micro- and nanotomography,[3,4] giving a totally new perspective to material science.

Micro-computed tomography (micro CT) is an X-ray-based imaging technique that can provide 3D views of the internal structures of investigated specimens. As the expression "micro" indicates, this technique makes it possible to achieve a spatial resolution that is better than one micron. The main advantages of tomography are that the specimens normally do not have to be specially prepared and that the method is non-destructive. Thus, the same specimen can be investigated under different conditions (e.g., monitoring the degradation processes during the ageing of a material, or any other changes to the material made under force or temperature loads). Like other methods, microtomography, too, has its limits.[5] In the case of denser materials (with high Z values) and larger specimens, the scanning time quickly increases up to several hours or even several days. In order to increase the scanning speed, it is necessary to sacrifice a certain degree of image quality. The size of the specimens also has an impact on the final resolution, although the size is not a critical parameter and it is possible to achieve good spatial resolution even in the case of large specimens.

## 2 EXPERIMENTAL WORK

### *2.1 X-ray computed micro CT: the principles*

Every tomograph consists of three basic parts; an X-ray source, a sample stage and a detection system. The X-ray source is usually an X-ray tube, in which electrons are accelerated by a known voltage and then they collide with the target (anode). The effect of a rapid braking of an electron as it hits its target is the high-energy electromagnetic radiation (Bremsstrahlung radiation), i.e., the X-rays. The problem is that this kind of radiation contains the whole energy spectrum of photons, up to the accelerating voltage. In any material, the photons of different energies have different absorption coefficients. The effect is known as "the beam hardening" and it makes a homogenous material look denser at the edges than it really is. This problem can be avoided with the use of the synchrotron radiation resulting in the monochromatic radiation with a very narrow energy spectrum. Additionally, the synchrotron radiation flux is many orders of magnitude greater than the radiation emitted by conventional X-ray tubes. In this way the scanning time can be significantly shortened.[6,7] The detector system is another crucial part of each microtomograph. In order to detect the photons that make up X-rays, they have to be converted into visible light using an appropriate scintillator. A scintillator is a special kind of material which emits visible photons when excited by ionizing radiation. Between a scintillator and a CCD detector, there can be an optical system allowing an optical as well as a geometrical magnification. This greatly improves the resolution achieved for larger specimens. The best results and finest resolution can, however, only be obtained for reasonably small specimens. Unlike in the medical or

industrial CTs, the source and detector in a microtomograph are stationary during the scan – the specimen stage rotates around the vertical axis during the scanning process. In this way 2D absorption radiographs of a specimen are taken from every sample-orientation angle. These are then reconstructed in 3D in order to obtain a view of the interior structure of the material.

In its basic form, tomographic reconstructions provide 3D information about X-ray absorption. In this way, each 3D pixel (voxel) represents a piece of the matter with a certain X-ray absorption factor. Due to the different absorption factors of different phases in a specimen, it is easy to detect certain features, such as voids and grains of different compositions. By using a further image analysis, it is possible to determine the values of many different parameters, such as the pore-size distribution and the orientation of features as well as their volumes and areas, all of which are important for a good understanding of material properties and functionality.

### *2.2 X-ray computed micro CT: the building materials*

Microtomography is a very useful technique for the building-material studies. In the case of smaller, specially prepared specimens, the technique is entirely non-destructive. This means that it is possible to study the same specimen at different ageing periods, or, for instance, before and after the temperature or pressure loads have been applied. Very good information can be obtained about how a building material may behave in different exposure environments. For instance, we can monitor a cement hydration process, or the growth of the cracks in concrete under load, 3D crack distribution in steel after a stress-corrosion cracking[8] or a loss of material due to steel corrosion.[9,10] Although there are literally endless possibilities, as with every measuring technique, there are also certain limitations, mainly with regard to the specimen size and the type of specimen material. Clearly, the weight and size of the test specimen must not exceed the nominal table capacity. These limitations vary from system to system, but, roughly, a test specimen can have a weight of up to several kilograms and a size in the order of a few decimeters. Although some tomography systems can achieve very fine resolutions in the case of larger samples, for the best results, smaller samples are much more appropriate. In practice, if a resolution of under a micron is needed, the sample size is usually limited to a few millimeters in diameter. Additionally, the X-ray absorption is an exponential function of the sample thickness. Thus, the scanning time is highly dependent on the sample size.

As already suggested, some types of materials are more appropriate than others for the tomography. In the case of the microtomograph that has been installed at the Slovenian National Building and Civil Engineering Institute (ZAG), the highest acceleration voltage is 150 kV. This voltage is certainly sufficient for investigating the materials such as concrete, stone, mortars, glass, ceramics, wood and polymers. However, there are limitations in the case of the materials such as steel, copper and other denser metals. In order to investigate the materials of this kind, very small samples are needed in order to obtain a strong enough signal from the detector. Otherwise, the scanning time can be unreasonably long and the signal-to-noise ratio can be too low to provide qualitative information.

### *2.3 X-ray computed micro CT: the experimental procedure (image acquisition, processing and analysis)*

In this study, the X-ray energy was set to different values. For the purpose of studying steel corrosion in concrete, using different methods, different concrete specimens with the dimensions of 450 mm × 100 mm × 55 mm with two embedded longitudinal reinforcement steel bars of the B500B quality were prepared. The specimen described in this paper was cyclically exposed to a 3.5 % solution of NaCl for a period of 4 years. The beam energy was set to the highest available value (150 kV, 66 μA), due to the high density of the steel embedded in the concrete. 5000 images were recorded in order to get a good-quality 3D reconstruction (**Figures 1** and **2**). In the case of the tested concrete sample (**Figure 3**), the beam energy was set to a value of 140 kV and the intensity was kept constant at 70 μA. Using a high-precision rotation stage, different projection images were taken at different views, with different exposure times per projection during the 360° rotation. In the case of the concrete sample, 4000 images were taken at different views with an exposure time of 3 seconds per projection. These projections were acquired with a CCD camera, and an optical magnification objective with a nominal power of 0.39 was used. The pixel resolution under these

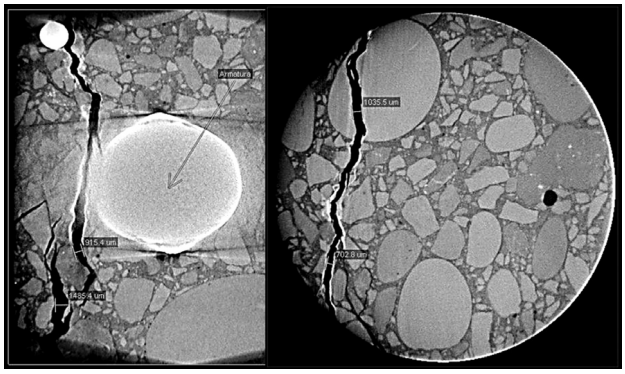


**Figure 1:** 2D slice in the *XY* direction of the steel bar embedded in the concrete (left image). A layer of the secondary corrosion products is visible on the surface of the embedded bar (the bright layer) as well as on the surface of the newly formed crack (left and right image). The cracks in the concrete cover were formed due to the increased volume of the secondary corrosion products.

**Slika 1:** 2D-rezina v *XY*-smeri jeklene armature (levo) v betonu. Na sliki so vidne plasti sekundarnih korozijskih produktov, in sicer tako na površini armature kot tudi v nastali razpoki v betonu (leva in desna slika). Razpoke v betonu so nastale zaradi povečane prostornine sekundarno nastalih korozijskih produktov.

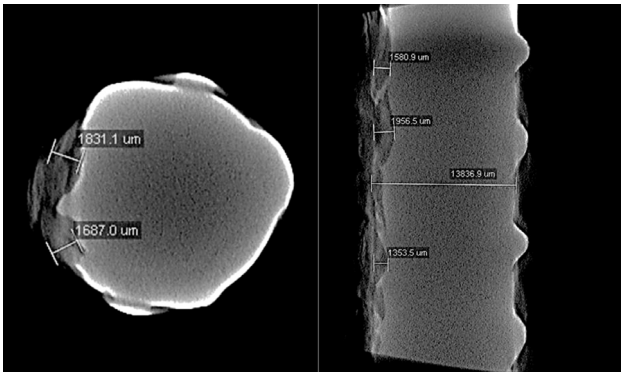


**Figure 2:** Horizontal (left) and vertical (right) cross-sections of the corroded steel bar. Approximately two millimeters of the bar was corroded due to the exposure.

**Slika 2:** Horizontalni (levo) in vertikalni (desno) prerez korodirane jeklene armaturne palice. Približno dva milimetra palice je bilo korodirane zaradi izpostavitve.

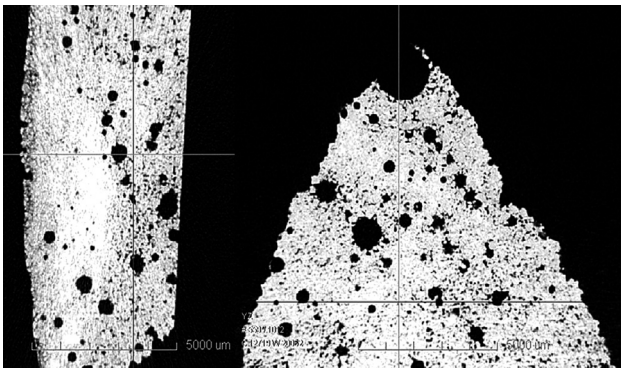


**Figure 4:** Vertical slices of the mortar specimen in the *XY* and *YZ* directions

**Slika 4:** Vertikalni rezini cementne malte z vidnimi zračnimi porami (črna faza) in cementno matrico (svetlejše faze)

conditions was 29 μm. In the case of the mortar sample (**Figure 4**), the beam energy was set to a value of 80 kV. 1600 projections with an exposure time of 4 seconds were taken during the 360° rotation, and a 0.39-times optical magnification objective was used. The pixel resolution under these conditions was 19 μm. In order to investigate the cracks in a humid soil sample (**Figure 4**), the sample was embedded in a 2 mm thick glass capillary. The beam energy was then set to 60 kV and the intensity was kept constant at 166 μA. The sample was rotated during the 360° rotation, and 1000 images with a 25 s exposure time were recorded. With an optical magnification of 20, the final pixel resolution of 1.2 μm was obtained.

## 3 RESULTS AND DISCUSSION

The concrete cover of the embedded steel bar (**Figures 1** and **2**) was 1.0 cm. After four years of exposure, the first cracks became visible on the surface. Using the tomography technique, it was possible to clearly detect and measure the cracks induced by the corrosion of the steel reinforcement. In the vicinity of the bars, the crack width was around 1 mm. The images of the reconstructed slices were somewhat blurred and of a lower quality, very close to the steel, which was a consequence of a very high contrast in the X-ray absorption between the steel bars and the surrounding concrete. Nevertheless, the loss of material on the bars was clearly visible. As expected, only the upper side of the reinforcement, which was closer to the exposed concrete surface, was highly corroded. Up to almost 2 mm of steel was corroded.

The representative slices in the *XY* direction (left) and the *XZ* direction (right) obtained from the concrete specimen are shown in **Figure 3**. As shown in this figure, the background (i.e., the surrounding air) is shown as dark voxels. The same dark voxels in the reconstructed greyscale image also correspond to the

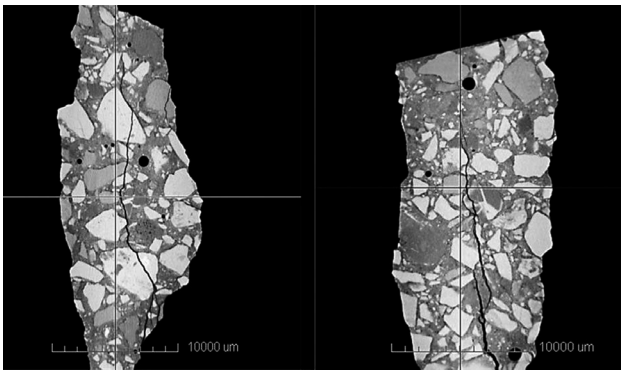


**Figure 3:** Vertical cross-sections of a piece of concrete approximately 3.0 cm in size. The cementitious matrix, at least two types of aggregate grains, air voids and cracks are presented in the image with different greyscale values.

**Slika 3:** Vertikalna prereza približno tri centimetre velikega kosa betona. Na sliki so z različnimi sivinskimi vrednostmi prikazani cementna matrica, vsaj dva tipa agregatnih zrn, zračne pore in razpoke.

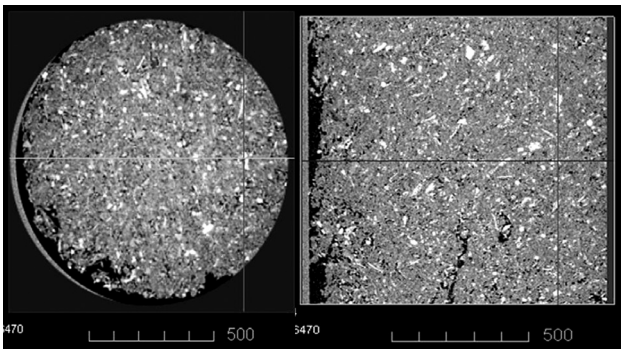


**Figure 5:** Horizontal slice (left) and a vertical slice in the *XZ* direction (right) of the humid-soil specimen in a glass capillary, partly visible on the left image. Different inorganic (brighter) and organic (darker) components are visible in the figure, as well as the cracks and air voids. A snapshot of a 3D representation of the soil structure is given in **Figure 6**.

**Slika 5:** Horizontalna (levo) in vertikalna (desno) rezina v smeri *XZ* mokre zemljine v stekleni kapilari, ki je vidna delno na sliki levo. Na sliki so vidne različne anorganske (svetlejše) in organske (temnejše) komponente, kot tudi razpoke in zračne pore. Izrez iz 3D- predstavitve materiala je podan na **sliki 6**.

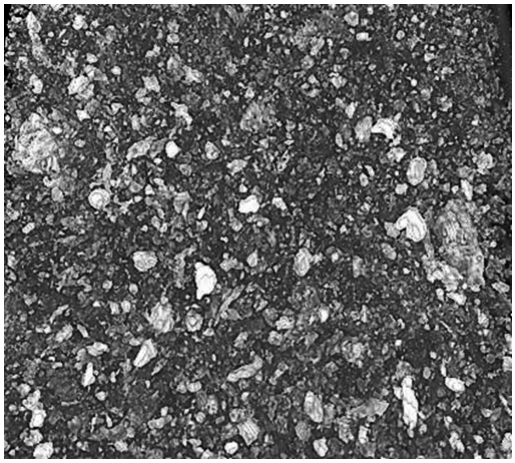

**Figure 6:** Snapshot of a 3D representation of the soil structure
**Slika 6:** Izrez iz 3D-predstavitve vzorca zemljine

low-density phases such as air voids or pores. The brighter voxels denote the high-density phases, e.g., the cementitious matrix and aggregate grains as the phases with the highest density. The representative slices show different distributions of aggregate particles, with the typical diameters in excess of 50 μm. A large crack is visible in the *XY* and *XZ* directions. Thus, in this case the orthoslices show a clear presence of large aggregate particles, the cementitious matrix and air-void porosity. **Figure 4** shows reconstructed slices of the mortar. The brighter voxels denote the high-density phases of sand particles that are smaller than 0.5 mm. The light-to-dark-grey patches indicate the cementitious matrix. The dark voxels in the reconstructed greyscale image correspond to the air voids or pores with the diameters in excess of 1000 μm.

The first images of the soil sample in a glass capillary, only 2 mm thick, were taken with a 20-times optical magnification (**Figures 5** and **6**). To see different inorganic and organic components as well as cracks, further images were taken at a higher optical (40-times) magnification distinguishing the organic matter from the air pores and the fluid in these pores.

## 4 CONCLUSIONS

As can be seen from the results of the experimental study presented in this paper, the micro CT technique is a powerful technique for an in-situ monitoring of the microstructure evolution of different types of building materials. Both heterogenous and homogenous materials can be analyzed using the microtomographic technique, which makes this tool important for a non-destructive three-dimensional characterization. Additional techniques such as scanning electron microscopy can be used to identify individual phases. With the addition of the recently developed nanotomography and with possible phase identification, tomography has become an indispensable tool in the building pathology, as well as in all the individual fields of material science.

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