EXPERIMENTAL STUDY OF TILE GROUT MATERIAL BEHAVIOR

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Summary: *Study provides preliminary results of experimental study of tile grout material behavior. Experiments were performed with the use of microCT, three point bending test and methods for chemical analysis. It was proven that material behave very elastic and the suitability of the combination of used methods.*

Keywords: *grout, three point bending test, microtomography, X-ray diffraction, FTIR*

1 Introduction

White tower of Hradec Králové is a ten floor renaissance building with gothic features.[1] During repairs of its sandstone envelope conservators found a special elastic behavior of the material used as a tile grout between the sandstone blocks. In 2010 a detailed assessment of stone and brick structures of outside envelope was done. The result (Kopecká 2010 in Nosek 2010 [2]) showed that most of the joints are made of very fine grained sealant, containing black cement with addition of polymer dispersion.

The purpose of this research is determination of the tile grout type and assessment of its characteristics. Results will be used as referential for measuring of small samples utilizing newly developed Innovated measuring line for identifying the physical characteristics of the historic stone and wood (described by Major et al.[3]) in the future.

2 Materials and methods

2.1 Specimen and its preparation

Experiments were performed with a specimen of the tile grout with dimensions 22 x 24 x 40 mm. Specimen was taken at the gallery on the south facade of the tower, where wind erosion, abrasion and typical loss of the original relief structures are particularly reflected. Place of collection of the sample is shown in figure 1(a), (b), the sample itself in figure 1(c). The whole specimen was scanned by X-ray microtomography.

Figure 1: Specimen - (a) Place of collection, (b) gaps appearance, (c) sample.

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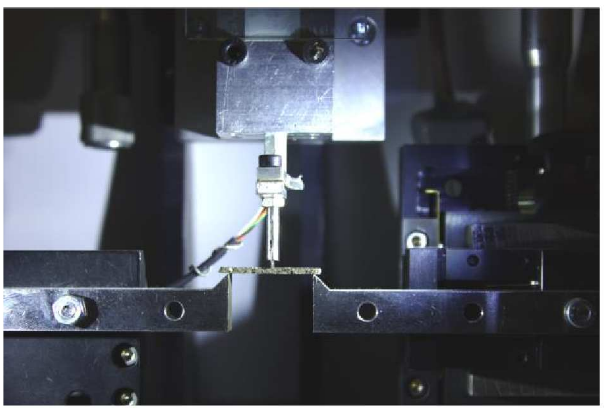
For mechanical tests, four samples were cropped in different distance from the surface to take into account different composition of the material due to weathering and climatic influences. During the preparation of the sample for Fourier transform infrared spectrometry, hardening of the sample by cyklododecane was used before making the cut. In order to analyze the average chemical composition by X-ray powder diffraction, about $4\,\mathrm{g}$ of sample were ball-milled.

2.2 Mechanical analysis

Three point bending test was carried out employing custom developed experimental setup for micromechanical testing. Detail of the setup with the sample is depicted in figure 2(a). Micrometric screws allow repeatability of the measurements in range of micron. Based on real time controlling software module both displacement and load driven experiments could be performed. Set of samples obtained from different level of the bulk material was tested in displacement controlled mode. Span of the supports was set to $20\,\mathrm{mm}$, cross section area of the sample was approximately 1.5×2.6 mm. Maximal deflection of the samples was set to $500\,\mu\mathrm{m}$. Euler-Bernoulli beam theory was used for calculation of elastic properties of the samples. Graph of the load test is plotted in figure 2(b).



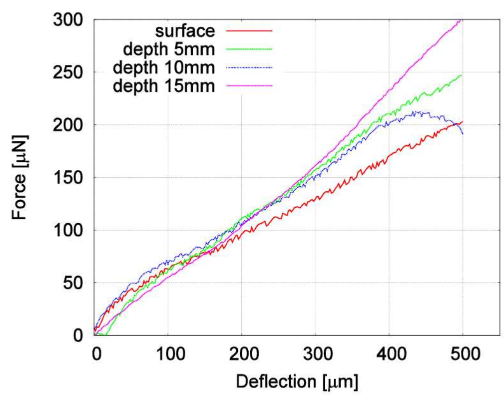


Figure 2: Three point bending test - (a) experimental setup, (b) graph of force versus displacement.

2.3 Chemical analysis

Chemical and mineralogical analyses were performed by powerful analytical techniques Fourier transform infrared spectrometry (FTIR) and X-ray powder diffraction (XRPD). XRPD was carried out in a D8 Advance instrument from Bruker equipped with a LynxEye 1-D silicon strip detector. A Bragg-Brentano $\theta - \theta$configuration was used with CuKα radiation ($40\,\mathrm{kV}$ and $40\,\mathrm{mA}$). A virtual step scan of $0.01\,^\circ$ (2θ with $0.4\,\mathrm{s/step}$ counting time, was employed. A FTIR iZ10 spectrometer from Nicolet was used in an attenuated total reflectance (ATR) mode with diamond crystal to obtain average IR spectra. Chemical maps were obtained using FTIR (Nicolet iN10) in the reflection collection mode with MCT (mercury cadmium telluride) detector cooled with liquid nitrogen. A representative area of the sample was chosen and around 4200 spectra were collected. Step size between each spot was fixed to $100.0\,\mu\mathrm{m}$.

2.4 X-ray microtomography

Tile grout specimen was tomographically analyzed using in-house developed system for X-ray microtomography (see figure 3(a)). X-ray tube XWT-240-SE was operated at target voltage $120\,\mathrm{kV}$ and target current $180\,\mu\mathrm{A}$. Flat panel detector Perkin Elmer XRD 1622 AP 14 with active area 400x$400\,\mathrm{mm}$ and pixel pitch $200\,\mu\mathrm{m}$ (2048 x 2048 pix) served as X-ray imager. Radiograms with acquisition time 2x1 sec were acquired during 800 projections with angular step $0.45\,^\circ$ for computed tomography (CT) reconstruction purpose. Beam hardening correction was applied on all radiograms. CT reconstruction was done using cone beam reconstruction algorithm implemented in the Volex 6 software. In a reconstructed volume, one voxel represents a cube with edge length $25.2\,\mu\mathrm{m}$ due to projective magnification (given by the relation between distances tube-sample/tube-detector). The reconstructed 3D microstructure of the tile grout is imaged in figure 3(b).

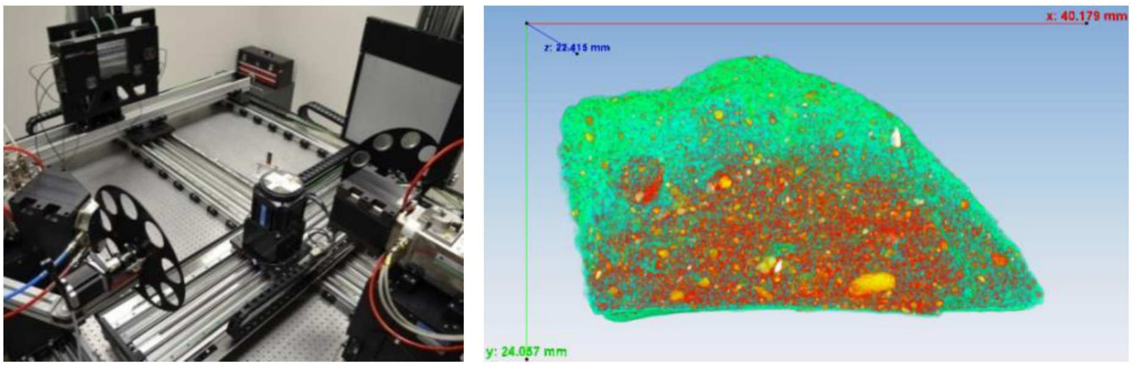


Figure 3: X-ray microtomography - (a) experimental setup, (b) 3D visualisation

Visualization of tomographic volume data in figure 3(b) shows different composition of the material caused by weathering. In the depth (top) grout was protected against climatic influences, but on the surface (bottom) occurs the loss of light small particles and large ones are therefore very visible and more represented.

3 Results

From three point bending test modulus of elasticity was calculated. Summary of results is presented in table 1. Values increases with respect to the distance from the surface.

Table 1: Modulus of elasticity

Surface [GPa]	Depth $5\,\mathrm{mm}$ [GPa]	Depth $10\,\mathrm{mm}$ [GPa]	Depth $15\,\mathrm{mm}$ [GPa]
8.00 ± 3.80	8.89 ± 3.71	9.15 ± 3.48	10.22 ± 3.51

Against expectations that the material will behave similarly such as concrete (MOE around $30\,\mathrm{GPa}$), elastic modulus values of this material is closest to the values commonly obtained for spruce wood (MOE around $9.1\,\mathrm{GPa}$). This tile grout is comparatively soft and flexible.

Obtained XRPD mineralogical qualitative phase analysis confirmed the presence of quartz (SiO_2), calcite ($CaCO_3$), orthoclase ($KAlSi_3O_8$), albite ($NaAlSi_3O_8$), kaolinite ($Al_2Si_2O_5(OH)_4$) and gypsum ($CaSO_4 \cdot 2H_2O$). Results from FTIR are in good agreement with XRPD measurements, but one organic phase was also detected, best fit was found for poly(acrylic acid)-co-(2-ethylhexyl acrylate)-co-styren.

Obtained chemical maps of the sample are depicted in figure 4(a). The distribution of quartz, calcite and the organic phase is well distinguished. Big grains in the pointing mortar are formed by quartz. Calcite is present around these grains as the final product of carbonation reaction. Organic phase as well as calcite is well distributed in areas between aggregates. The occurrence of the specific analyte in maps is depicted by different color: in red are represented the areas with the highest concentration and in blue the absence of each phase.

In figure 4(b) comparative microtomography slices are captured in the same scale. Local color distribution of materials corresponds to results of chemical analysis. Moreover microtomography has in this case much better resolution and contrast.

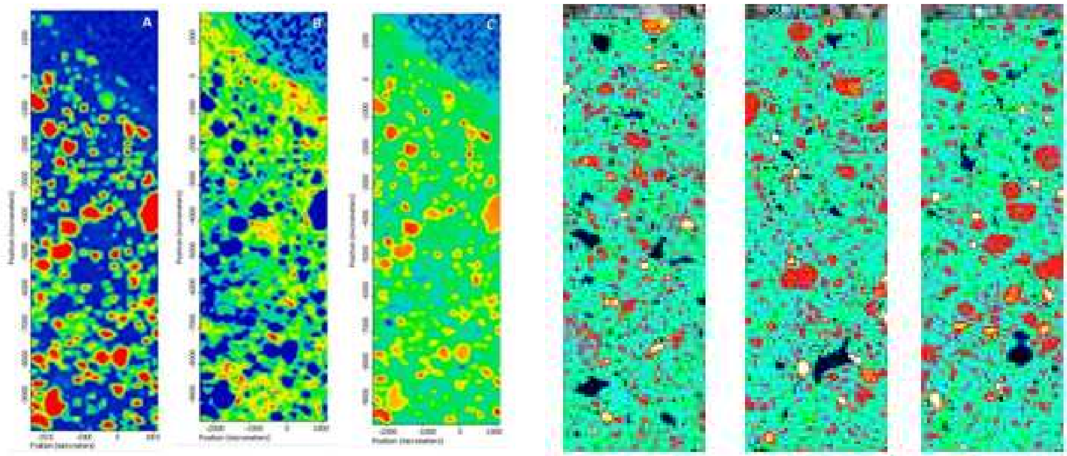


Figure 4: Maps - (a) Chemical maps: A - quartz, B - organic phase, C - distribution of matrix around quartz grains; (b) Material maps of the sample acquired by microCT.

4 Conclusions

Mechanical tests have shown unexpected elasticity of the studied material. Chemical analyses confirmed the presence of many minerals and one organic phase. Poly[(acrylic acid)-co-(2-ethylhexyl acrylate)-co-styrene] was found to have best fit. The exact percentage of individual components, especially sealants and synthetic polymers, which are responsible for elastic behaviour of the grout, will be the subject of further research.

It was proven by microCT that a structure of the grout differs according to the depth from the surface. These differences are caused by weathering. Image data obtained by the tomographic reconstruction corresponds to results obtained by chemical analyses.

5 Acknowledgment

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