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MICROSTRUCTURAL AND TOMOGRAPHIC ANALYSES IN GEOTECHNICAL ASSESSMENT OF SOIL MEDIA

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Abstract: This paper discusses two different approaches to microanalysis of the soil medium. It presents testing results collected by electromicroscopy, which requires special sample preparation, and a non-destructive method, requiring no sample preparation and using a computer-assisted tomograph. The paper presents:

- Applicability of microstructure analyses and chemical microanalysis in the determination of structural damage incurred in the historical time in relation to the effect of contemporary abiotic events;
- Application of a standard medical computer-assisted tomograph in qualitative assessment of structure and observations of mechanisms of failure in the cement/peat soil composite.

Investigations concerning the applicability of computed tomography in soil micromechanics are demonstrative in character and the presented conclusions suggest further directions for research.

Key words: *clay soils, organics soil composition, microstructure, computed tomography*

1. INTRODUCTION

Soil microanalysis by scanning electron microscopy is the only method facilitating direct observations of soil particles much smaller than 2 μm. Microphotographs of structural surface make it possible to determine the shape and dimensions of mineral particles, as well as the degree of dispersion. It is of particular importance in the identification of properties of cohesive soils, e.g., the genetic type, microstructure and expansivity. Cohesive soils comprise primarily the clay and colloid fractions composed of secondary minerals, i.e., clays. In geotechnical engineering, when strengthening the subsoil with the use of binding materials it is essential to determine the degree of crystallinity and crystal decomposition of binding materials and admixtures, as well as chemical changes in the composite determining strength and stability in their operation life.

State-of-the-art electron microscopes are equipped with microanalysers facilitating not only structural, but also chemical analyses of a selected area, pore expansion and identification of their size. Tests are performed on properly prepared quasi-intact samples. Serious problems appear when testing media are sensitive to changes in moisture content, with volume changes (expansive clays) or structural deformations, i.e., destruction of aggregate bridges characteristic of dipping clays [5], [6].

Results of microstructural analyses in combination with chemical and mineralogical analyses are successfully used at present not only for scientific purposes. They are also applied to identify causes and determine the time when failure occurred in objects due to geotechnical causes (landslides, building failure), as well as for verdicts in complicated court cases.

Another highly promising method in the practice and basic research in soil mechanics may be that of identification of properties in soils or soil composites using an X-ray computer-assisted tomograph. An X-ray computer-assisted tomograph (CT-Computed Tomography) is one of the most common diagnostic tools used in medicine. Tomography is a non-destructive and at the same time non-invasive testing method. This means that it does not change chemical or physical properties of samples and for this reason it is not necessary to introduce any measurement devices inside the sample and it does not destroy its structure. Thus sample preparation for analyses does not require laboratory treatment, in this way saving time and reducing costs of analyses.

Computed tomography has revolutionised biomedical imaging and has been applied increasingly often in other fields of sciences [1], including also geotechnics.

In recent years in soil mechanics [4] applied tomographic analysis in the qualitative evaluation of structural changes following glacial till failure. Kumor [7] investigated strength of the organic soil–cement composite based on the results recorded using an X-ray tomograph. Dilatancy, genesis of microfissures, surfaces and geometry of damage were analysed.

This paper presents two complementary approaches to the microanalysis of the soil medium. The study presents results obtained using the electromicroscopic method, requiring special sample preparation, and a non-destructive method requiring no sample preparation applying a computer-assisted tomograph. This paper shows:

- Applicability of microstructure analysis and chemical microanalysis to determine structural incurred in the historical time in relation to the effect of the currently conducted invasive foundation works;
- The use of a standard medical computer-assisted tomograph in qualitative evaluation of structure and mechanisms of damage to the cement/peat soil composite.

Research in the section concerning the application of a computer-assisted tomograph in soil micromechanics is demonstrative in character and the presented conclusions suggest directions for further research.

2. ANALYSES OF MATERIALS AND RESULTS OF MICROANALYSES

Microstructural analyses under a scanning electron microscope (SEM) were conducted for a building structure, in which symptoms of serious failure were detected in the course of monitoring of performed foundation works. Microstructural analyses aimed at the identification of the time and genesis of occurring wall fracturing in relation to the zero state, i.e. prior to the earth works. Analyses were conducted at the Laboratory of Electron Microscopy and Microanalysis, the Institute of Hydrogeology and Engineering Geology, the Faculty of Geology and Hydrology, the Warsaw University.

Tomographic analysis was decided to offer new potential for studies on dilatancy (the shear thickening process) and microstructural relationships in the modification of strength in samples of the cement/peat soil composite. Imaging was performed at the Laboratory of Radiology, the Kujawy-Pomeranian Pulmonology Centre in Bydgoszcz. Analyses were conducted using a 16-row spiral computer-assisted tomograph by Siemens, model Somatom Emotion-16.

2.1. DESCRIPTION AND HISTORY OF THE DAMAGED OBJECT

The building in which damage was observed is located in the city centre, at 2 Gdańska Str. in Bydgoszcz, and it is the second oldest Roman Catholic historic monument in that city. It is of exceptional value for the cultural and historic heritage of Bydgoszcz. It was built in the Gothic-Renaissance style in the years 1582–1602 at the location of the former wooden Holy Spirit chapel. Until 1818 it was a monastery church. In its history it served not only religious functions, as from 1835 it was used as a warehouse. In 1875 it was adapted for use by the fire department. A major repair was performed in the years 1878–1937 and for the last time in the years 1992–1993 (the facade).

A street, Jagiellońska, is located on its southern side, being the route for the main transit traffic of vehicles and tramways across the city. It is an area with the highest concentrations of transport-related air pollution. Elevated contents are recorded for aggressive gaseous compounds (CO, CO_2, SO_2, SO_3, CS_2) and hygroscopic salts (KCl, NaCl). This has a direct effect on the acceleration of material corrosion in objects found in that environment. This environment is chemically aggressive.

Fig. 1. A general view of the damaged object, March 2013

As it is assumed, because of deep excavations conducted since 2005 in the vicinity of the church subsoil, stability was disturbed and dangerous fracturing was observed in that object. After analyses of the structural damage the object was classified as 1° failure [5]. In walls marked concentration zones of cracks of various width and single cracks of 5 to 15 mm in width were observed, running from the foundation and expanding upwards. Problems appeared, such as doors and windows that could not be opened or closed, tilting ceilings, breaking window panes, detached stairs and annexes, etc. (Fig. 1).

Since it was not possible to determine causes for the failure and identify the author of the damage, a series of analyses was performed and observations were taken of the structure, subsoil, hydrogeological conditions and the effect of traffic-related vibrations. Results were variously interpreted and prevented a precise assessment of the moment the cracks and fracturing were formed in the object.

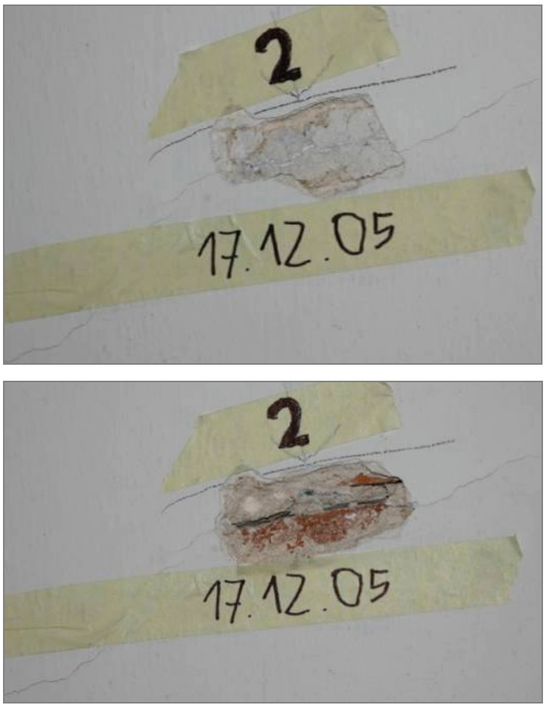

Fig. 2. Phases of sample collection for microanalyses of paint, plaster, fracture filling, material of the southern wall

The ambiguous and complicated history of the object resulted in a situation when the heritage conservator made a decision on the necessity to conduct more in-depth analyses of the cracks and fracturing. The aim was to determine causal connections, which could be readily interpreted and linked directly to the observable effects of object destruction. It was decided to perform microstructural analyses and chemical microanalyses. Samples were collected from the major cracking zones in walls and plaster, as well as successive plaster paint layers (Fig. 2).

2.2. ELECTROMICROSCOPIC ANALYSIS

The reference point for microanalysis was assumed to be the state recorded in undamaged points based on the morphology of cracks and fractures. Samples were collected from cracks and fractures in paints, plaster and bricks for SEM analyses (electron scanning microscopy) and for analyses of the chemical composition (EDS microprobe).

Photographs were taken in the microstructure of surface in plaster samples in the cracks and fractures, as well as samples from intact areas and samples of bricks. Appropriately collected samples with intact structure and retained moisture content were analysed in terms of their chemical composition, focusing on the occurrence of characteristic organic compounds, the chemical background and anthropogenic pollution. Microphotographs below present testing results of samples from the chemical background (Fig. 3), samples of polychrome paint from 1936 (Fig. 4), results of contamination of a crack-fracture filled with titanium paint from walls painted in 1936 (Fig. 5).

- A microphotograph and chemical composition of the intact zone – plaster, chemical background.

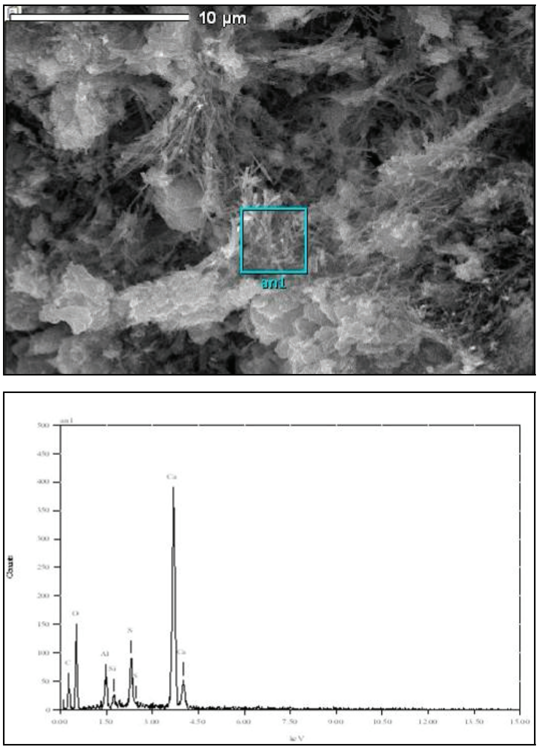

Fig. 3. A microphotograph of plaster surface adjacent to damage site with EDS spectrum of chemical composition

- A microphotograph and chemical composition of damaged plaster in the area of a crack-fracture.

In the quantitative comparisons of EDS spectra and contents of individual elements in Figs. 4 and 5 in relation to results of other analyses (e.g., Fig. 3) the following were found:

- The presence of barium and titanium on plaster,
- An increase in contents of carbon and sulfur, additionally the occurrence of titanium and barium – Fig. 3,
- Occurrence of ettringite.

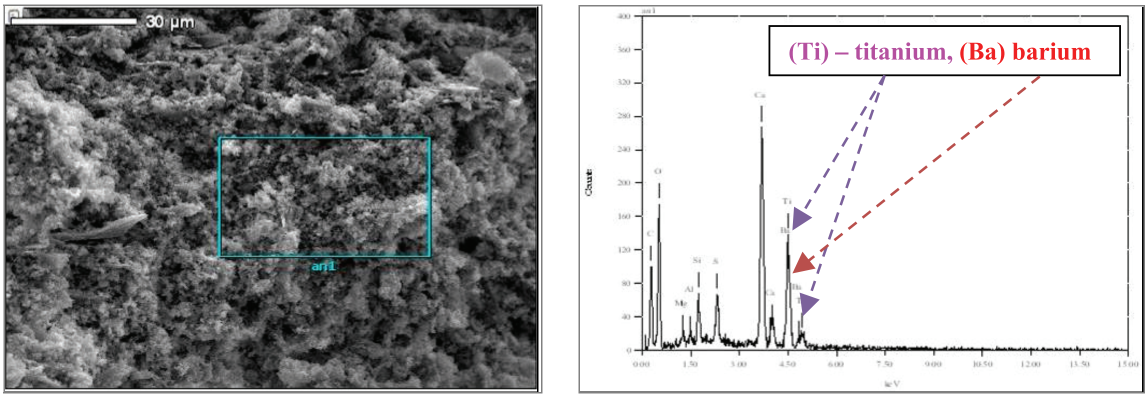

Fig. 4. A microphotograph of surface of damaged plaster with EDS spectrum of chemical composition of contamination with titanium paints (titanium Ti compounds, walls painted in 1936, barium Ba – painted in 1903)

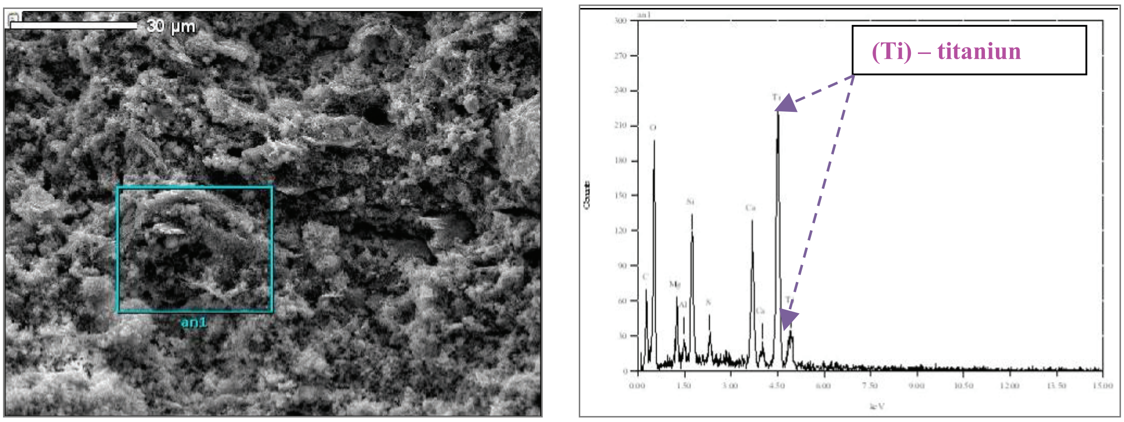

Fig. 5. A microphotograph of plaster damage surface together with EDS spectrum for the chemical composition of contamination of crack-fracture with titanium paints (titanium Ti compounds, walls painted in 1936)

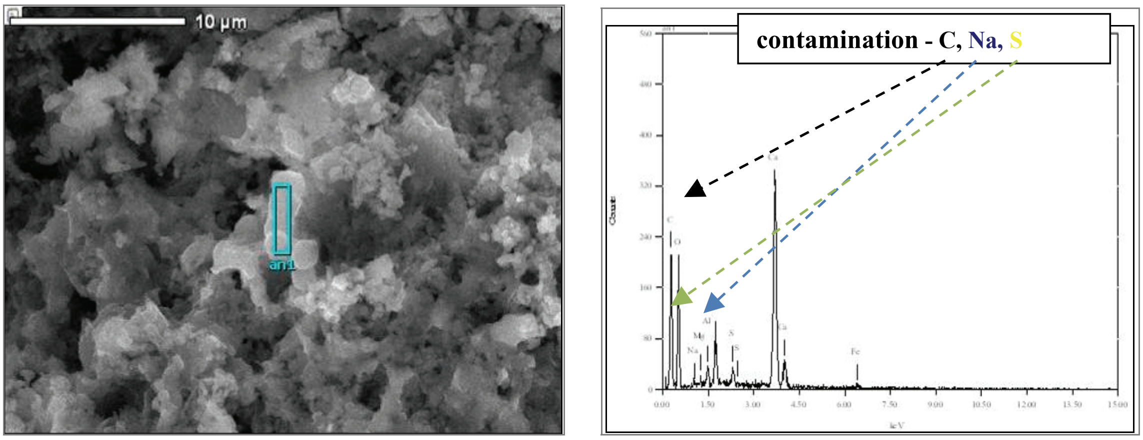

Fig. 6. A microphotograph of surface of damaged external plaster with EDS spectrum of chemical composition of anthropogenic pollutants

- A microphotograph and chemical composition of contamination in crack-fracture.
- A microphotograph and chemical composition of contamination of plaster, external zone.

When analysing the chemical composition, i.e., the occurrence of potassium and sodium, which crystallised on the plaster from air from hygroscopic salt dusts and elevated carbon contents it was found that:

- Plaster damage (cracks) was formed as a result of surface corrosion,
- The result of corrosion – a visible crack, Fig. 2, appeared relatively recently,
- Formed plaster damage is connected with an increase in salt contents.

2.3. DISCUSSION OF MICROANALYSES

Testing results were analysed first of all by comparing contents of individual elements in areas of intact plaster and in relation to contents of elements in damaged areas. The focus was on contents of repair elements, which indicate the occurrence of sedimentation or corrosion processes in plaster, in damaged areas inside cracks-fractures. In view of the fact that samples were collected from walls of the object, the potential occurrence of typical elements deposited from atmospheric air or from applied paints was analysed in cracks of plaster in different periods of operation of the object, including repairs.

Corrosion processes occur at the slowest rate in the air-dry environment, where air contains gases, e.g., CO, CO_2, SO_2, SO_3 and CS_2 as well as salt dusts KCl, NaCl. In historic buildings these compounds attack and destroy materials in wall structures, particularly those with a previously slightly damaged and hygroscopic structure. Table 1 presents the composition of chemical compounds contained in mineral mortars in the object.

The following findings result from the analysis of microphotographs of damaged plaster surface (Fig. 4), as well as EDS spectra for contents of individual elements and the chemical composition of paint pollutants:

- The occurrence of elements contained in paints used on walls, i.e., titanium and barium;
- An increase in the contents of carbon and sulfur in the surface layer of plaster;
- The occurrence of ettringite compounds in mortar.

An increase in carbon contents indicates the deposition of such gases from air as CO and CO_2, which cause leaching and softening of the mortar structure. The occurrence of titanium and barium is connected with the fact that damaged fragments were previously painted (in 1903 and 1936, when paints for outdoor use were based on minerals, barium and titanium). A high sulfur content is caused by its deposition from gases, SO_2 and SO_3, causing sulfur corrosion, resulting in the formation of, e.g., ettringite (Fig. 7).

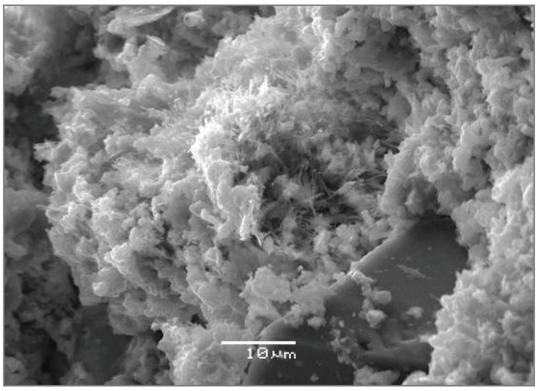

Fig. 7. A marked formation of ettringite in the structure of plaster

Summing up the results of SEM and EDS analyses based on the microanalysis presented in Fig. 4 it may be stated that the analysed crack-fracture is an old case of damage, dating back to over 100 years.

When analysing the chemical composition in the EDS spectrum (Fig. 6) for the crack reported by the owner as new we may see in the damaged plaster the deposition of potassium and sodium, which have salted from air from hygroscopic salt dusts, and an elevated carbon content. It is stated that:

- Plaster damage was caused by surface corrosion;
- The effect of corrosion, i.e., a visible crack-fracture of plaster (Fig. 2) appeared relatively recently;
- Visible damage is connected with salt crystallisation, i.e., an increase in volume of sulfur salt crystals, which in the structure of plaster produce pressure many times greater than that of freezing water and thus burst it (Fig. 8).

Table 1

Percentage contents of individual chemical compounds found in mineral mortars

Mortar (depending on type)	Content of compounds [%]			
	CaO+MgO	MgO	CO_2	SO_3
	>90 to min. 70	to 5	to 12	to 2

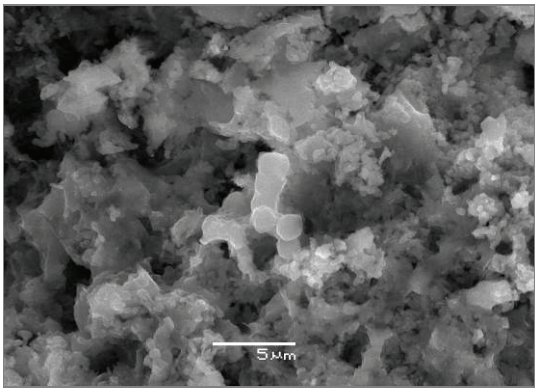

Fig. 8. Visible crystals of NaCl and KCl salts and gypsum in the structure of plaster

Summing up the recorded results of microstructural analyses the crack needs to be classified as new damage, incurred at least 20 to 30 years ago. However, it is only the visual effect of the long-term process of atmospheric corrosion of the plaster material. The process of air-dry corrosion is very slow (i.e., deposition of salt derivatives and aggressive gases in the material). Thus it needs to be remembered that the visual effect of this activity, i.e., the crack, is not formed immediately under similar conditions and develops quasi-statically.

2.4. SUMMARY OF MICROANALYTICAL RESULTS

Results from microstructural analyses in combination with chemical and mineralogical analyses provided positive and convincing results. Chemical microanalyses and analyses of microstructure of plaster materials made it possible to determine the time of damage formation in the historic object. A comprehensive conservation analysis of the history of the object indicates a significant relationship between structural damage and environmental and geotechnical causes. Identification of material properties at the micro level is at present successfully applied not only for scientific purposes. The problem of considerable importance is still connected with an appropriate selection of samples for electromicroscopic analyses and an extensive knowledge of local conditions. Significant contemporary factors accelerating the progressing erosion of materials and degradation are anthropogenic in character (very high atmospheric air pollution, vehicle traffic, etc.), increasing the complexity of conditions and hindering their interpretation.

3. TOMOGRAPHIC ANALYSES OF THE ORGANIC SOIL-BINDER COMPOSITE

The method of identification of soil properties using an X-ray computer-assisted tomograph may prove suitable in soil micromechanics and in geotechnical engineering.

When analysing strength of the organic soil–cement composite in relation to the structure tested using a tomograph we may observe a lack of deformations, high selectivity and easy identification of individual elements in the microstructure [7]. Sample preparation for analyses is generally limited to the collection of material with adequate shapes and dimensions.

Soil composites (cement binder-peat) formed by in situ mixing was subjected to tomographic analysis. The sample was cylindrical in shape with 71 mm in diameter and 140 mm in height [1], [3]. The basic component of the mixtures comprised lowmoor, sedge-reed peat with the H_3/H_4 degree of humification according to the von Post scale, at the average contents of organic substance $I_{\mathrm{om}} = 83.64\%$, natural moisture content $w_n = 323.13–419.72\%$ and mean bulk density $\rho = 1.04$ Mg/m^3. The peat composite was stabilised with Portland ash cement of high strength class CEM II/B-V 32,5R. After 28 days of sample maturation uniaxial compressive strength was determined.

Tomographic analyses, tomograms, were prepared for two example composites with varying shares of cement and organic substance. One sample, K1, had organic substance content $I_{\mathrm{om}} = 20\%$ and the ratio of added cement to the weight of dry soil $m_c/m_s = 1.75$. The other sample, K2, had a natural content of organic substance and $m_c/m_s = 2.25$.

3.1. ANALYSES OF COMPOSITE STRUCTURE USING COMPUTED TOMOGRAPHY (CT)

Samples were lined parallel to the axis of apparatus table travel in accordance with the axis of symmetry of the tested cylinders (the axis of rotation). The thickness of scanned cross-sections for the purpose of analyses was set at 1 mm. Presented 2D images were 512 × 512 pixel. Image analyses were conducted using OSIRIX software for image viewing and processing for medical purposes in the DICOM format.

Figure 9 presents typical samples of composite K1 immediately after determination of uniaxial compres-

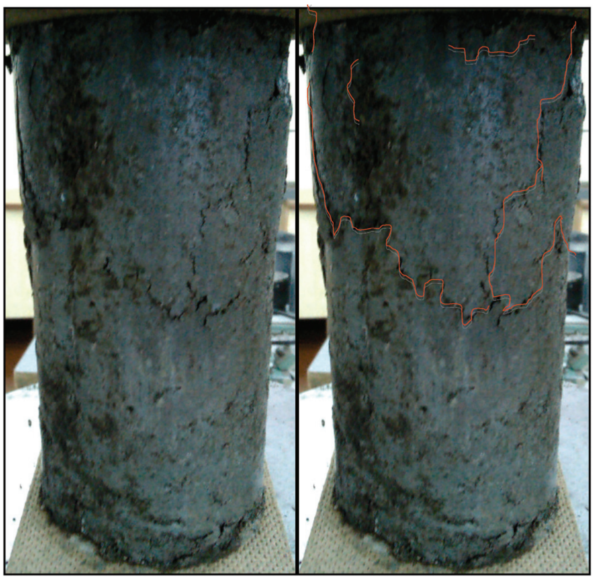

Fig. 9. Destroyed samples of composite K1 immediately after strength testing

sive strength. Formed deformation fractures are marked in red. Based on the macroimages we may identify the range of fractures, as well as the type of deformation formed as a result of damage. We may see the characteristic surface of brittle shear damage – slant surfaces at an angle of 45°.

Figure 10 presents selected structural cross-sections of sample K1. We may observe a heterogeneous structure with a marked random concentration of microaggregates of organic matter in the sample mass. The fracture developed in the weakest zone, the concentration of organic matter, which was tracked downwards starting from the top (a) to approx. mid-height of the sample (i). General observations indicate regularities in the development of sample destruction zones. The location and range of shear surface are dependent on the accumulation of microaggregates of organic particles in the composite. This is particularly evident in Figs. 10 b, c and f, where microfractures

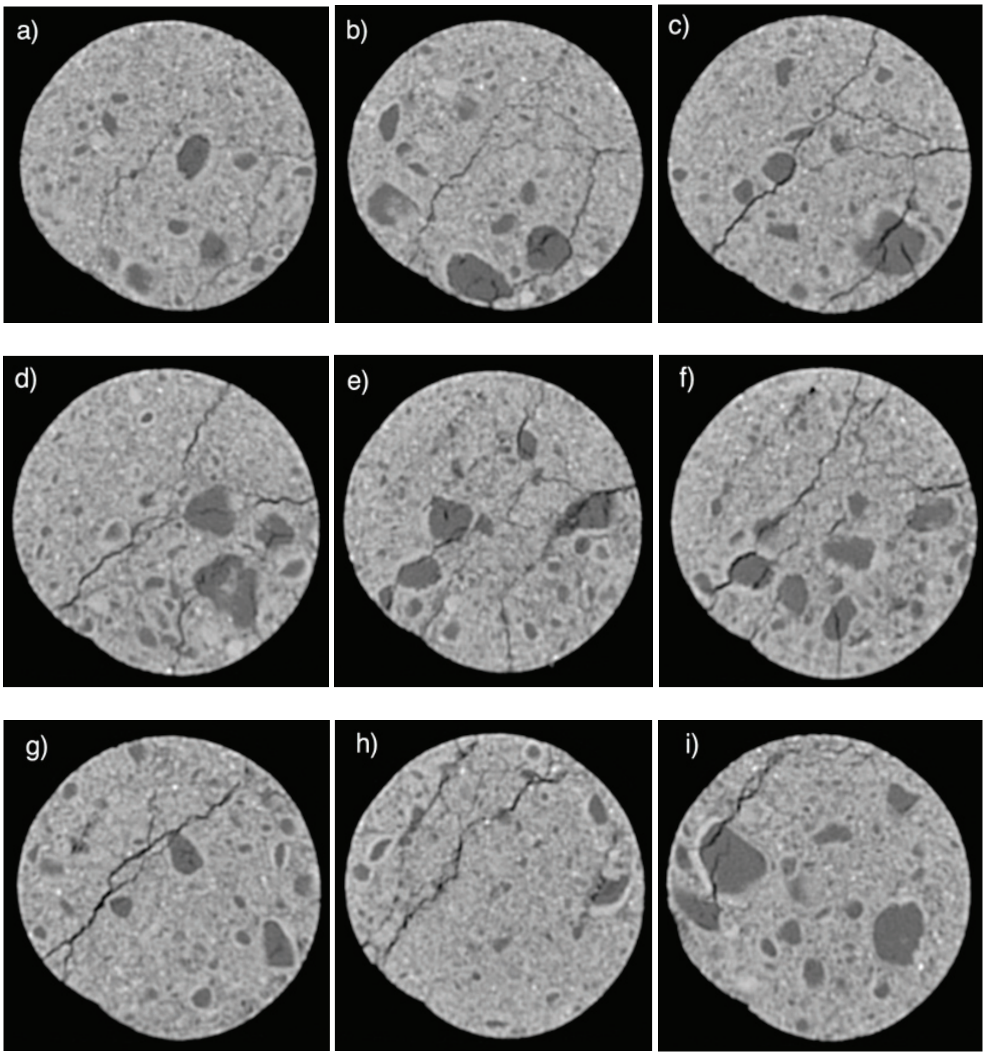

Fig. 10. Analysis of selected cross-sections of composite K1

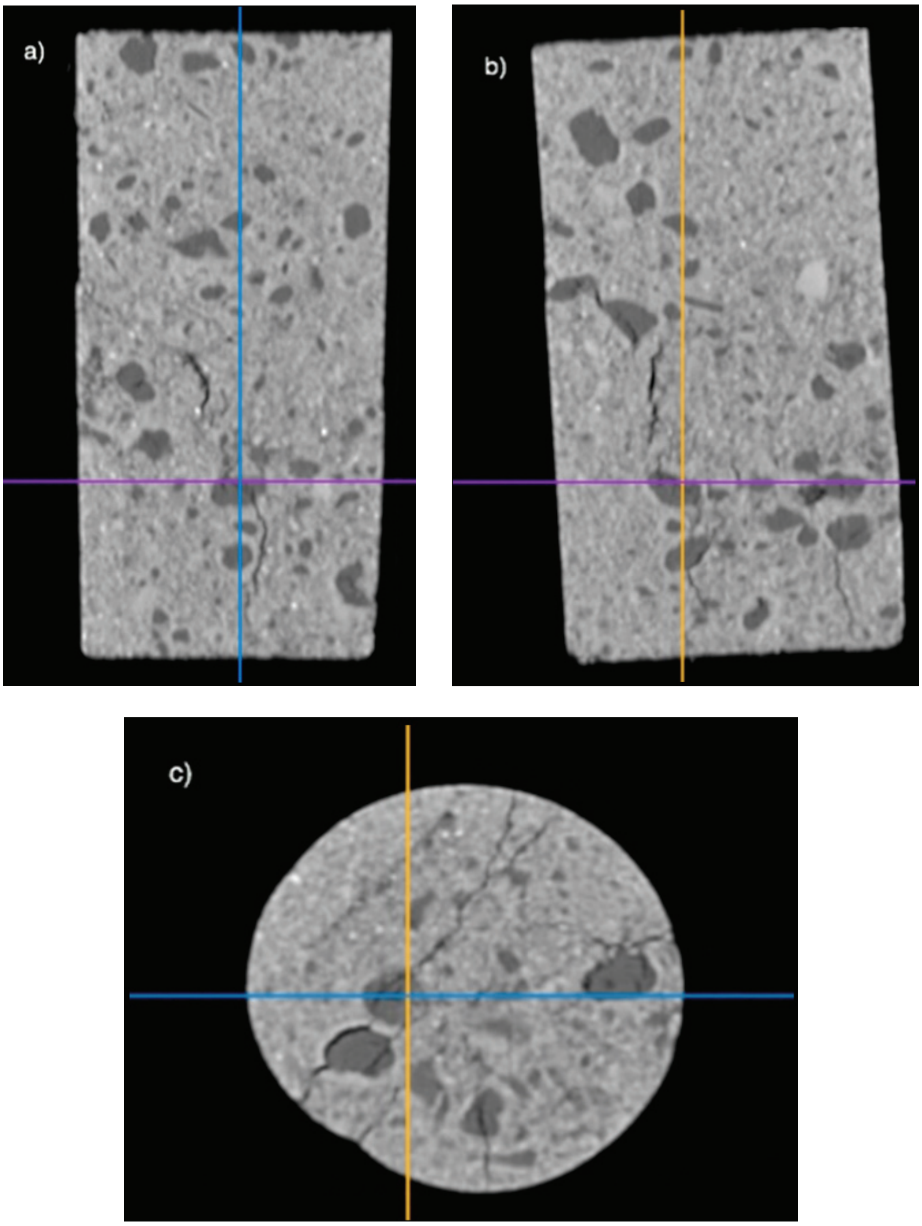

Fig. 11. A spatial image of peat microaggregate: (a), (b) longitudinal sections, (c) a cross-section

are initiated. The shear surfaces unfold the destruction path further through natural weakenings, i.e. organic microaggregates (visible in photographs as darker clusters). This is consistent with the fundamental principle of mechanics, i.e. the principle of least action or Hamilton's principle [8]. This confirms and directly illustrates the significant effect of component homogenisation on the final strength of the composite and justifies the application of CT in its assessment.

Further analysis using appropriate software facilitates precise localisation of a selected point of discontinuity, e.g. organic aggregate in the binder space. An example of deformation discontinuity is presented on mutually perpendicular longitudinal sections (Figs. 11a and b) and the corresponding cross-section (Fig. 11c). A comprehensive analysis makes it possible to track with high accuracy, depending on the set thickness of the scanned section, practically each weakening of the composite as well as the formation and spatial development of stages in mass destruction.

3.2. OTHER POSSIBILITIES TO IDENTIFY SOIL STRUCTURE USING COMPUTED TOMOGRAPHY (CT)

An interesting solution from the engineering point of view is also to use functions connected with Regions of Interest (ROI). In a selected section they facilitate determination of many important parameters

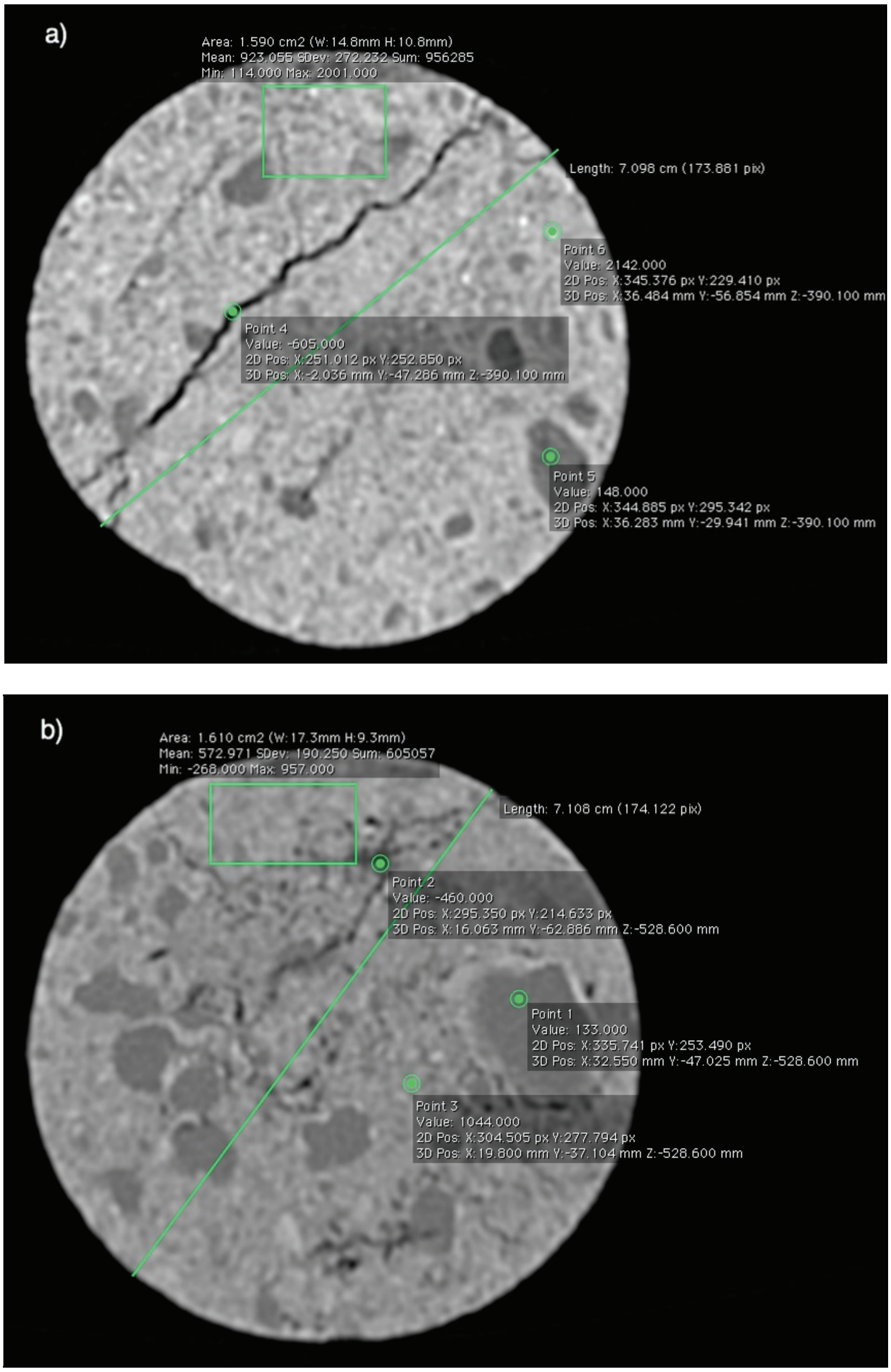

Fig. 12. Application of the ROI function in the analysis of example sections of composites: (a) K1, (b) K2

such as determination of absorption coefficient of a point as well as a marked arbitrary area. It is also possible to magnify the selected area, its dimensioning and many other functions. Below we compare example sections of composites K1 (Fig. 12a) and K2 (Fig. 12 b). Analysis of images indicate a considerably greater density of composite K1 (the greater the value of radiation absorption, the lighter the colour in the grey scale). A markedly greater porosity of composite K2 was observed in relation to K1. Analysis of selected rectangular ROI (denoted as *Area*) shows a mean HU for K1 (approx. +923) to be almost two-fold greater than K2 (approx. +572), as it could have been expected. A markedly greater maximum value of HU (approx. +2001) in composite K2 is connected with the occurrence of quartz sand grains. All pores, air-filled fractures, should have a negative value in the HU scale, e.g., point 4 in Fig. 12 (a) or point 2 in

Fig. 12 (b). Organic substance takes much lower values in analysed sections from +133 for K1 to +148 for K2 in the HU scale.

4. CONCLUDING REMARKS

Results of microstructural analyses taken using SEM in combination with chemical and mineralogical microanalyses, supplemented with knowledge provided by computed tomography, produce a comprehensive method of advanced identification of soil properties. They facilitate the identification of microstructure transformation in the modification of characteristics and mechanical parameters of soil media. At present identification of microscale material properties is successfully applied not only for scientific purposes. Appropriate sampling for electromicroscopic analyses remains a problem of considerable importance. Significant contemporary factors accelerating progressing erosion of materials and degradation of media exposed to external factors are anthropogenic in character (very high atmospheric air pollution, vehicle traffic, etc.), increasing complexity of conditions and hindering their unambiguous interpretation.

Conducted analyses confirm the applicability of medical X-ray computer-assisted tomographs for engineering purposes, including also designing of properties and studies on geotechnical composites. Preliminary CT analyses show that the behaviour of soil composites under load depends on their designed composition and homogeneity of their internal structure (structure and microstructure). Analyses performed using a computer-assisted tomograph provide prospects for improved and natural identification, free from deformation, of the spatial arrangement and interactions of individual components of the composite. This makes it possible to determine many significant characteristics, such as the degree of homogenisation, porosity, microstructure and even density, which have a direct effect on mechanical properties of the stable composite. Tomographic analysis of composites destroyed in the conventional strength tests facilitates the determination of mechanisms of their destruction and their reference to the expected geotechnical parameters.

Analyses presented in this paper were preliminary in character and constitute an introduction to further and more detailed investigations using the presented technology. At this stage the qualitative analysis was presented for the recorded images, particularly focusing on the potential of software and further possible application of a medical X-ray computer-assisted tomograph in analyses and designing of soil composites.

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