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THE EXPERIENCE OF APPLYING X-RAY COMPUTER TOMOGRAPHY TO THE STUDY OF MICROSTRUCTURE OF FROZEN GROUND AND SOILS

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The structure of frozen ground and soils has been studied by X-ray computer tomography. Significant advantages of the proposed method have been demonstrated. The changes in the structure of ground and soils in the freezing–thawing cycles have been described. Prospects for the use of X-ray computer tomography in the study of cryogenic processes have been identified.

Permafrost, frozen soil micromorphology, mineral skeleton, ice, pores, units, X-ray computer microtomography

INTRODUCTION

Numerous studies of permafrost scientists [*Shumsky, 1955; Konnova, 1957; Konishchev and Faustova, 1966; Konishchev and Rogov, 1977; Zhestkova et al., 1980*] are devoted to the structure of frozen grounds and soils. It is now known that the character of freezing and the resulting structural changes of loose deposits depend on the degree of dispersion, mineral composition, density and water content of the freezing substrate, as well as on the freezing conditions and regime [*Ershov, 1988*]. It is known that, when freezing, loamy soils change their structural characteristics, including the size and shape of structural units. In addition, the volume, shape and orientation of the pore space change. In sands, freezing of the loamy material, given rather high original water content, is accompanied by formation of crevice-like cavities filled with ice and areas with increased density of the intrapedal mass [*Kachinsky, 1927; Sokolov and Shoba, 1982; Gubin, 1993*].

The process of multiple (cyclic) freezing and thawing of soils makes significant changes in the composition and structure of grounds and soils. Over more than half a century of studying such transformations, essential knowledge has been accumulated. Formation of the specific features of microstructure in soil covers and in permafrost soils has been noted by ground, soil and permafrost scientists [*Kosheleva, 1958; Mazurov and Tikhonova, 1964; Konishchev and Rogov, 1977; Parfenova and Yarilova, 1977; Tursina, 1985; Zhangurov et al., 2011; Pastukhov, 2012*]. All the researchers encountered significant difficulties in making the suitable microstructure preparations, which were mainly related to the presence of ice. Until recently, these difficulties were resolved either by studying replicas [*Rogov, 2009*] or examining microsections of the thawed specimens, i.e., without ice [*Gubin and Lupachev, 2012*]. All the approaches and methods applied to the study of the microstructure of frozen soils and deposits have one feature in common – in order to make a preparation to be investigated, the specimen has to be destroyed, i.e., its original condition has to be transformed. Therefore, the researchers of the microstructure of natural objects have long desired to develop nondestructive analysis methods. Such a method emerged only at the turn of the 21st century: this is the method of X-ray computer micro-tomography.

X-ray computer micro-tomography is a nondestructive method of visualization and analysis of the inner structure of specimens using X-ray radiation. The method was developed by the American physicist Allan Cormack and the British engineer and physicist Godfrey Hounsfield, who were awarded a Nobel Prize in 1972 for this invention. Originally, computer tomographs were used for medical purposes only. Later, as devices with higher resolution emerged (computer micro-tomographs), computer tomography began to be used for solution of physical, chemical, biological, and geological problems.

The appearance of tomographs extended the abilities of scientists to study frozen rocks and soils. At the same time, literature reviews have shown that computer micro-tomography studies of frozen and freezing rocks and soils are still quite rare [*Taina et al., 2008; Torrance et al., 2008; Shi Jie Chen et al., 2014*].

The objective of this study is to present the first results of X-ray computer micro-tomographic investigations of the cryogenic elements of the structure of soil and rock specimens exposed to one-time or multiple freezing. The goal of the study consisted in developing a method of micro-tomographic study of soils and rocks in a frozen state, in order to elucidate the possibilities of this method. In addition, the authors have tried to demonstrate the regularities in the changes in the micro-structure of the soil and rock specimens under conditions of freezing–thawing by employing the method of X-ray computer micro-tomography.

OBJECTS AND METHODS OF THE STUDY

As objects of study, both model soils and the material of genetic horizons of different loamy soils of the European part of Russia were chosen. As model objects, porous ice, quartz sand of the 0.25–0.05 mm fraction and a mixture of kaolin clay 10 % quartz sand were selected. In natural objects (soils), specimens taken from the BT horizon of the virgin sod-podzol soil of the Moscow region (Zelenograd base of the Dokuchayev Soil Institute), of the A1 humus horizon of the virgin chernozem (Streletskaya steppe of the Central-chernozem reserve), of the Bca horizon of the virgin solonetz soil (Dzhanibeksky base of the Institute of Forest Studies of RAS) (Table 1).

The soil specimens were prepared as follows: to level the original differences in the micro structure of the soil mass, the soil specimens were ground with a rubber-tipped pestle and sieved with a sieve with a mesh size 250 μm. Then the ground mass of the specimen was put into plastic cylinders 12 mm high and with the internal diameter of 8 mm (these dimensions were chosen due to the technical capabilities of the micro-tomograph). The specimens were saturated with water in a capillary way and, to ensure homogenous freezing simulating natural freezing, placed into a plastic foam holder to rule out lateral freezing. The procedure of one-time freezing and thawing was conducted with an Espec SH-241 temperature humidity chamber. Each cycle consisted of a freezing phase (10 hours at the temperature −20 °C) and a thawing phase (10 hours at the temperature +20 °C).

X-ray scanning of the frozen specimens was conducted with a Bruker SkyScan-1172 micro-CT scanner. The scanning was carried out with the detail resolution up to 2.5 μm/pixels. The tomograms were processed and analyzed with specialized software for the purpose of two-dimensional and three-dimensional image analysis by Bruker. The voluminous image was reconstructed with the Recon software, the 3D model was built using the CTvox program. The micro-tomograph used was selected not only for its X-ray imaging parameters but also for the fact that it had a cooling table with a possibility of keeping the scanned specimen at the temperature of −15 °C as a standard option.

RESULTS AND DISCUSSION

The main task of any microscopic study is to validate the images produced for compliance of the actual object details and the image details. For computer tomographs, this task is solved by measuring and computer processing of the attenuation difference of X-ray radiation by the details of the object under study differing for their density. It was discovered in the first tomographic studies of frozen soil [*Torrance et al., 2008*] that ice and pores in the specimens almost equally dissipated X-ray radiation and therefore could be poorly distinguished on the tomograms. To solve this problem, Russian researchers used the method of saturating of water with heavy isotopes [*Davletshina et al., 2014; Nadeev et al., 2014*]. However, it was technically difficult to apply this method to the study of soils and frozen rocks in the field conditions; therefore, the authors of the paper chose the way of improving the quality of the images of the specimens in question.

For two-component media (for example, porous ice), visual identification of individual elements of the structure presents no methodological difficulties.

Table 1. **The proportion (%) of different fractions in soil specimens**

Soil	Horizon (depth, cm)	Fraction size, μm						Kachinsky scale
		1000–250	250–50	50–10	10–5	5–1	<1	
Quartz sand	–	–	99	1	–	–	–	Medium-grained sand
Prosyanovsky kaolin	–	–	–	4.2	5.0	17.6	73.2	Heavy clay
Sod-podzol	BT (50–60)	0.01	1.59	52.51	17.06	22.92	5.91	Heavy loam
Chernozem	A1 (10–15)	0.00	2.74	41.04	17.84	33.27	5.11	Heavy loam
Solonetz	Bca (8–15)	0.82	24.56	29.98	12.41	24.56	7.46	Light clay

Figure 1 shows the 3D structure of a porous ice specimen obtained by 1D freezing of gas-saturated water. Such an image allows rather precise evaluation of the inclusions of gas, their volume and shape, which is problematic for other methods. Hence, the method of X-ray computer micro-tomography seems to be very promising for the study of the structure of rather loose cryogenic formations.

For frozen soils, the notion of porosity is usually defined as space among the mineral particles of the skeleton filled with ice, water and gas. While revealing particles of the mineral skeleton presents no difficulty, it is quite complicated to diagnose components in the space of pores. This problem was studied in the example of the frozen specimens of quartz of 0.10–0.25 mm fraction with incomplete (W = 15 %) and complete filling of pores with water.

As seen from Fig. 2, in the specimens with incomplete water saturation, the space among the skeleton particles (3) is filled with ice (1) and voids (2). These components may be also distinguished by their morphology – wetting of particles with water results in the fact that ice occupies all the contacts among the particles ("cuffs"), and the gas volumes are round in shape.

It is to be noted that analysis of the structure of the specimens with incomplete filling of pores in the tomograph indicates the presence of needle ice-cement (Fig. 2, *b*), which almost cannot be determined by conventional methods due to its instability, as it quickly melts irrespective of the method of surface exposure. This ice is formed due to ablation of evaporated water, and in the soils with low ice content, it may prevail compared to the other types of needle ice-cement [*Zhestkova et al., 1980; Rogov, 2009*].

The use of a micro-tomograph in studying cryogenic textures proved to be effective: ice schlieren forming laminar-lenticular structure can be easily distinguished in the specimen of the frozen loam specimen (Fig. 3, *a*). The tomograph capabilities allow the cryogenic structure to be discerned in any plane, including the horizontal plane parallel to the freezing front (Fig. 3, *b*). Studying the cryogenic structure in such an aspect has not yet become a common feature among the permafrost scientists but it has far-reaching prospects.

More detailed analysis of the cryogenic structure of the specimens allows confirmation with real imagery of many cryogenic processes which have been previously determined only indirectly. Thus, in the kaolin specimen with the mass fraction of 10 % quartz of fraction 0.10–0.25 mm, migration of water towards the freezing front is well visualized, as well as soil precipitation in the dehydration zone and formation of ice lenses under particles of sand, resulting in their movement to the freezing surface (Fig. 4).

To study the dynamics of the structure of soils and rocks in cyclic freezing and thawing, the speci-

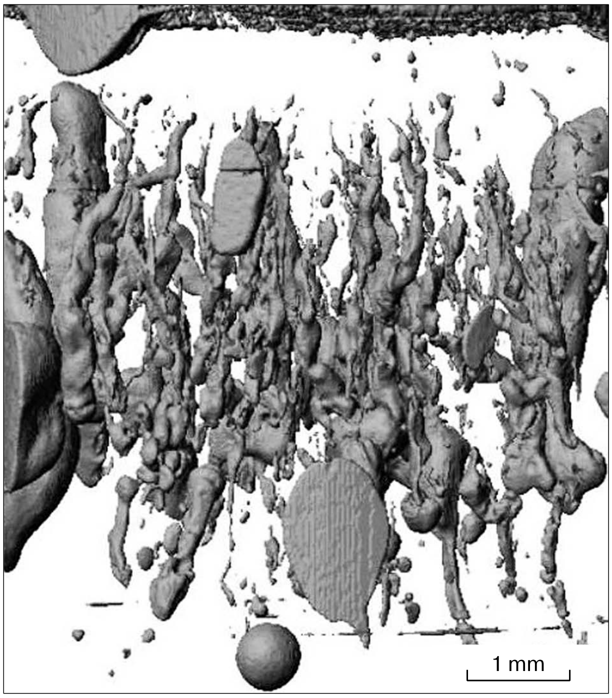

Fig. 1. Distribution of pores (grey figures) in an ice specimen.

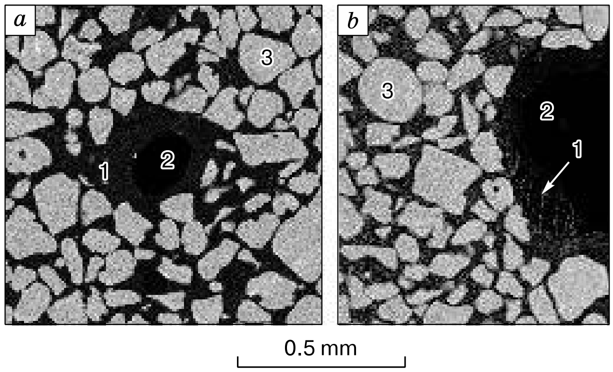

Fig. 2. The micro-structure of a quartz specimen with incomplete filling of pores (*a*) and needle ice-cement in a quartz specimen with incomplete filling of pores (*b*).

1 – ice; 2 – voids; 3 – sand grains.

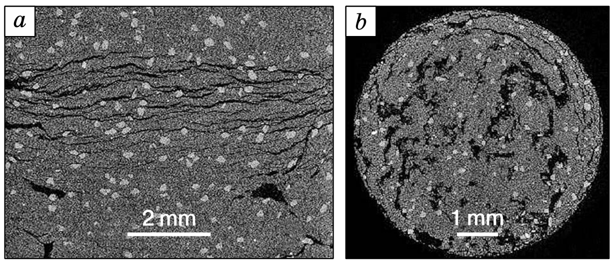

Fig. 3. Cryogenic structures in a loam specimen.

a – vertical cross-section; *b* – horizontal cross-section; black – ice schlieren.

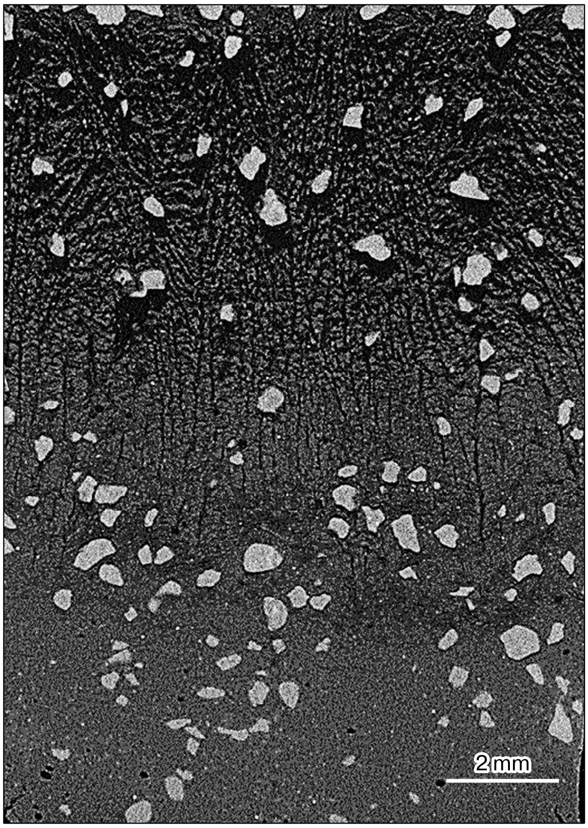

Fig. 4. The vertical cross-section of a model mixture of 90 % kaolinite and 10 % sand in a frozen state after one-time freezing.

Black – ice inclusions; light – quartz particles; light-gray background – kaolin.

mens prepared as described above were scanned with a micro-CT after the first freezing and after the fivefold, tenfold and twentyfold freezing cycles. At the end of the experiment, the information obtained was analyzed to ascertain the transformations of the micro-structure of the specimens under study.

A mixture of kaolin and sand was chosen as a simple model of soil with the presence of the crude and clayey fractions. Analysis of the micro-structure of such a model has shown that, as the number of the freezing–thawing cycles grows, differentiation of particles takes place in the specimen, which is expressed in the transport of the particles to the contacts with ice schlieren and in the ring-shaped sorting of the particles. Similar phenomena have already been noted for frozen soils and cover loams [*Konishchev and Faustova, 1966; Konishchev and Rogov, 1977; Tursina, 1985*], and their existence is now confirmed by the method of computer micro-tomography (Fig. 5).

The experiments with soil specimens were conducted under a more comprehensive plan: changes in the micro-structure were observed in the specimens, beginning with the non-frozen state, the first freezing phase was considered, followed by the other phases, up to the twentieth freezing–thawing cycle. After the first freezing, large isometric aggregates from 130 to 250 μm in diameter, cut by fractures 20–30 μm thick (Fig. 6) emerged in the specimen of the humus horizon of chernozem. In the mass of aggregates, there were pores of a round shape with indistinct edges 120–250 μm in diameter, filled with ice containing fine clayey material. As the number of the freezing-thawing cycles grew, the density and width of the fractures in the soil mass increased, and the aggregates acquired the shape of plates with distinct edges. After 20 freezing–thawing cycles, large pores were formed (up to 500 μm) with ice free from clayey particles.

In the specimen of the BT horizon of the sod-podzol soil, the first freezing also essentially changed the character of the soil structure (Fig. 7). Originally the homogeneously disperse mass became restructured into the aggregate mass – round and oval ag-

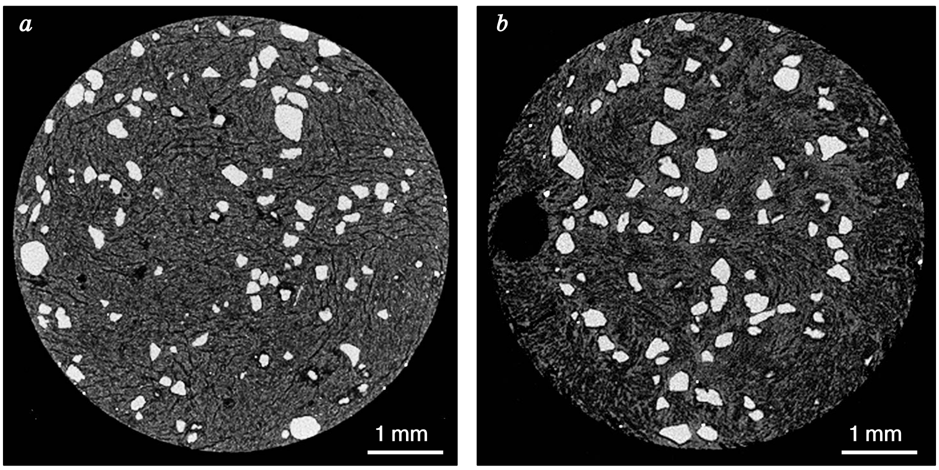

Fig. 5. Distribution of sand grains in a specimen of a model mixture of kaolinite and sand:

a – after the first freezing; *b* – after 20 freezing–thawing cycles.

gregates sized 150–200 μm in diameter; part of the aggregates had the shapes of plates. The aggregates were divided by thin 20–30 μm branches of ice. As the experiment continued, the number of the plate-shaped aggregates grew in the specimen edges (the edge effect of freezing of the cylindrical specimen seems to have manifested itself). Large pores had indistinct edges and contained fine-grained material inside. After 20 freezing–thawing cycles, the depth of the ice stratum increased, especially in the central part; the pores became more distinct, and freezing of the sand particles into the cores was observed.

In the solonetz specimen, significant morphological changes have not been revealed after one-time freezing and after the freezing–thawing cycles (Fig. 8). The general patttern of the micro-structure of the unfrozen state was preserved; only few fractures filled with ice were formed mostly in the mass of the fine-grained material, filling the space among the micro-aggregates sized 200–250 μm in diameter. We can suppose that, under conditions of intense salination, migration of water and ice formation are weakened, just as their impact on the changes in the micro-structure.

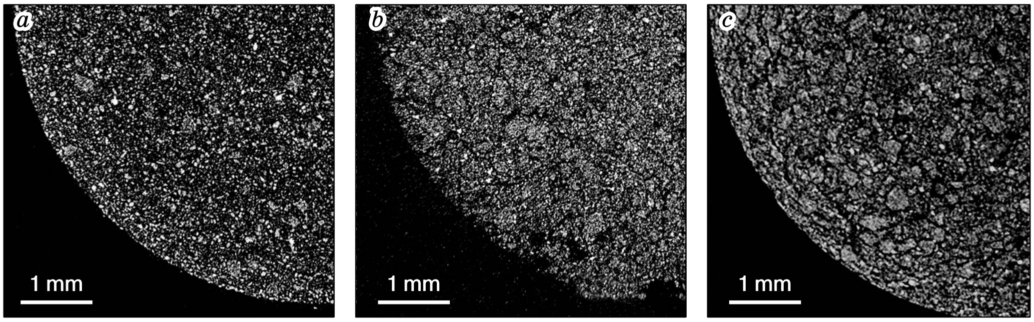

Fig. 6. The micro-structure of the specimens of the humus horizon of chernozem:
a – before freezing; *b* – after the first freezing; *c* – after 20 freezing cycles.

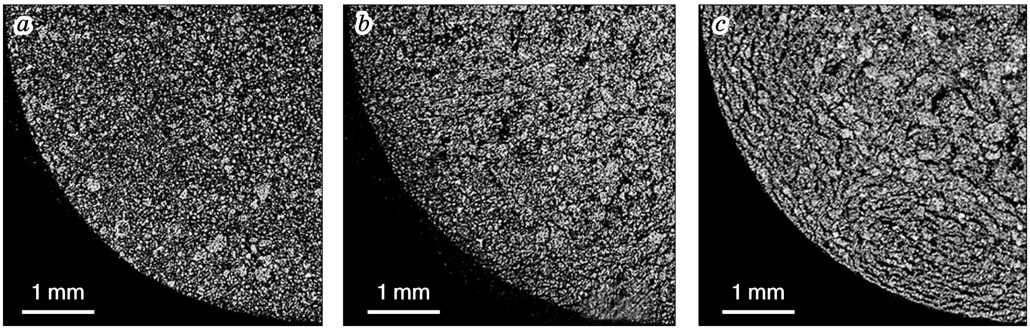

Fig. 7. The micro-structure of a sod-podzol specimen, the texture BT horizon:
a – before freezing; *b* – after the first freezing; *c* – after 20 freezing–thawing cycles.

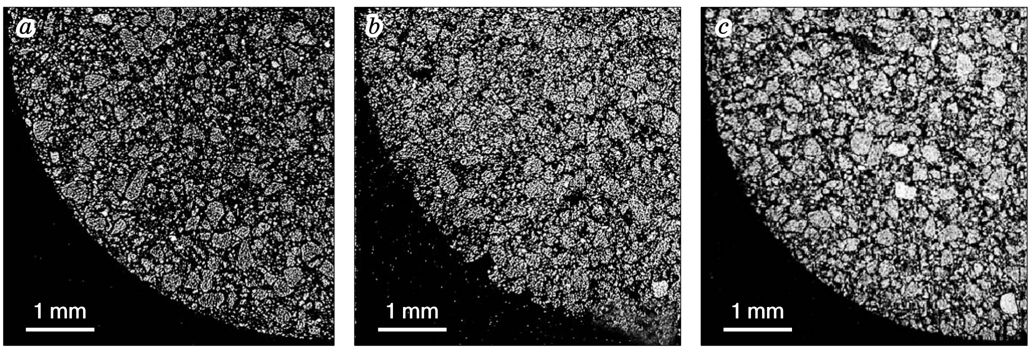

Fig. 8. The micro-structure of a solonetz specimen:
a – before freezing; *b* – after the first freezing; *c* – after 20 freezing cycles.

CONCLUSION

The experience of using X-ray computer micro-tomography in studying frozen soils and rocks has demonstrated this method to be rather effective for the study of the micro-structure of objects in different states, including moist and frozen objects. Snow, firn, frozen soils and fine-grained deposits may be such objects. The main advantage of the X-ray computer micro-tomography method consists in the possibility of studying a specimen without mechanical impairment and hence without impairing the structure and properties of the objects under study. In addition, it enables researchers to carry out direct observations of different cryogenic processes in time. Using this method, one can distinguish ice, air and the hard matrix and monitor changes in their configurations in the course of various processes, including formation of ice and ice schlieren in the freezing soil and in rocks, during interaction of ice with the structural components of the hard matrix.

At the same time, the experience of using the Bruker SkyScan 1172 micro-CT scanner has shown that a computer tomograph is required for studying frozen soils and rocks, with a possibility of studying specimens of larger sizes and with a high resolution.

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