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Employing X-ray computed tomography for the non-destructive ice cores analysis

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Применение методики компьютерной томографии для неразрушающего анализа ледниковых кернов

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Summary

Computed tomography (CT) is a nondestructive high-resolution way to investigate the three-dimensional structure of samples (ice, rock, etc.). The results of CT analysis of glacial cores consisting of firn and ice extracted on the Western plateau of the Elbrus Mountain (5100–5150 m a.s.l.) in the summer of 2017 are presented in the article. The core taken from the depth of 20.31–21.87 m and consisting of three sections (average length is 52 cm each) was analyzed. In order to maintain the natural negative temperature of the glacial core, a special cryothermos has been created. It conserved the temperature at the level of –25 °C. Data on the structural features of the samples and the three-dimensional pattern of the ice-firn density were obtained. Correlations between the density and some chemical elements had been established. The CT data made it possible also to determine sizes of ice crystals. Comparison of cross sections of cores with firn and ice thin sections (30 in total) has shown that the crystal structure is best displayed in the ice inter-layers since it is impossible to determine reliably sizes of the firn grains at the given survey resolution. Also, the use of the CT method made it possible to determine inclination of the firn layers within the ice core, which is caused by the inheritance of the slope of the surface microrelief and internal inhomogeneities of the firn thickness. Calculations showed that the angle of inclination of the layers varies from 6 to 9°.

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Ключевые слова: ***данные плотности высокого разрешения, Кавказ, компьютерная томография, криотермос, ледниковые керны.***

Методика компьютерной томографии позволяет получить снимки послойных срезов ледникового керна с помощью рентгеновских лучей. В работе представлен анализ кернов с Западного плато Эльбруса с помощью компьютерного томографа РКТ-180. Для поддержания естественных условий керна был создан специальный криотермос, который препятствует таянию образца и изменению структуры фирна во время съёмки. Исследована внутренняя структура керна, установлены размеры кристаллов в разных слоях, найдены неоднородности и получена трёхмерная картина плотности льда.

Introduction

An importance of gaining information from high altitude ice cores is constantly increasing due to the global warming. In comparison with Greenland and Antarctic regions, which have slower response to the climate changes, mountain glaciers are dramatically retreating and disappearing. A wide range of methods is available for studying ice cores presently: chemistry, isotopes, trace elements etc. A variety of most advanced modern technologies are employed for analyzing the ice cores. The presence of drawbacks, even in most advanced techniques employed over the past decade, leads to attempts to create new analytical methods. For example, the development of the continuous flow analyses (CFA), automated chemical method, permitted to pump samples and reagents continuously through a system of modules interconnected by tubing [1]. Employing the laser ablation mass spectrometry with inductively coupled plasma (LA-ICP-MS) produced detailed information about isotopic and chemical composition in samples [2, 3]. The method allows getting ultra-high-sensitivity chemical analysis at the ppb-level without any special preparation of the sample. The problem, however, remains in most of the methods that is related to the destruction of samples during the analyses. Ice is essentially lost in the process and become unavailable for other types of analyses.

Computed tomography is a well-known technique for obtaining cross-sectional images. In the last decade, this method has become widely recognized and emerged as a leading analytical tool in many areas [4, 5]. Computed tomography system allows obtaining a three-dimensional distribution of the X-ray absorption values in the entire volume of the sample within the limits of the resolution. All X-ray opaque elements give signal through density and chemical composition differences. The morphology and pore size, caverns, cracks, and inclusions are analyzed. The main advantage of the method is the non-invasiveness (using intact ice sections) of the samples [6, 7]. Since ice cores are very precious, the main advantage in using Computed Tomography (CT) is that it does not affect further processing of samples. The analysis cost is relatively low, while the amount of information obtained is substantial. CT method should be used before other analyses. The use of non-destructive CT method does not affect subsequent work with the samples [8].

Initially, the technique of computed tomography was used in medicine and made a revolution in the field as it became possible to obtain information about the internal structure of the human organism without surgery. Almost the same equipment and software for interpretation of the X-ray survey can be used for scientific purposes for sediment or ice core analysis. The first attempts to include CT in the principal methods of ice core studies were made in 1990 when J.M. Barnola and his colleagues used X-Ray tomography for 3D reconstruction of the Vostok firn structure [9]. A related method of micro-tomography is presently in rather high demand [10]. One of its disadvantages is the necessity of laboratory sampling. The general principle of the method is similar to the ordinary CT, however, micro-CT permits studying the inner structure with high resolution (several microns) [11]. Another problem of working with CT is the necessity of maintaining below-zero temperature. Sneed et al. [2] tried to solve this problem by constructing a cryocell that would permit to do laser ablation inside the cryosystem while minimizing the sampling area.

Full-length ice core CT was suggested by Voland et al. [12], however, it required substantial technological support. In the first attempt, the idea was to investigate the structure of ice cores before transportation that lead to applying ice core CT for the first time in the field (Kohnen station, DML, Antarctica). Subsequently, a research group from Alfred Wegener Institute created a laboratory with X-Ray CT system inside a freezer room maintained at −25 °C. The main goal of our study was to further develop the methodology for the full-length ice core CT analysis and to evaluate the adequacy of the CT method for studying ice core stratigraphy. Here we present the first CT results of shallow firn core from Mt. Elbrus A CT system enables analyzing ice cores, determine the porosity, compare the visual stratigraphy and chemistry and to correlate those results to the ice core thin sections. Since high image quality is required to get precise results, a vast amount of measurement data was produced. We discuss the advantages and possible further development of CT analyses for ice core studies.

Data and Methods

The Caucasus mountains are situated between the Black and the Caspian seas in the south of Russia. Ice core drilling took place at Mt. Elbrus, located in the Central Caucasus Mountains. A shallow ice core with a diameter of 9 cm and total length of 24 m was extracted from the Western Elbrus Plateau in the summer of 2017. The drilling site was located at an elevation of 5115 m [13]. Three sections (an average length of 52 cm) extracted from the depth of 20,31–21,87 m were used in this study. In order to perform the CT analysis for the full-length ice core, the freezing temperature had to be maintained during the imaging session. For this purpose, we used a special cryogenic thermos of 100 mm diameter. It consisted of a polyvinyl chloride pipe with sealed ends and double walls. The antifreeze between the walls was cooled at −25 °C. The cryothermos may accommodate 1 m long core sections and maintain a suitable temperature for approximately 5 hours. To test the ability of the CT system to analyze full length ice cores the artificial ice core (diameter 9 cm, length 30 cm) was made. It contained ice, firn, snow, and some mineral particles. Artificial core was made by sequential adding layers of water, snow and firn sam-

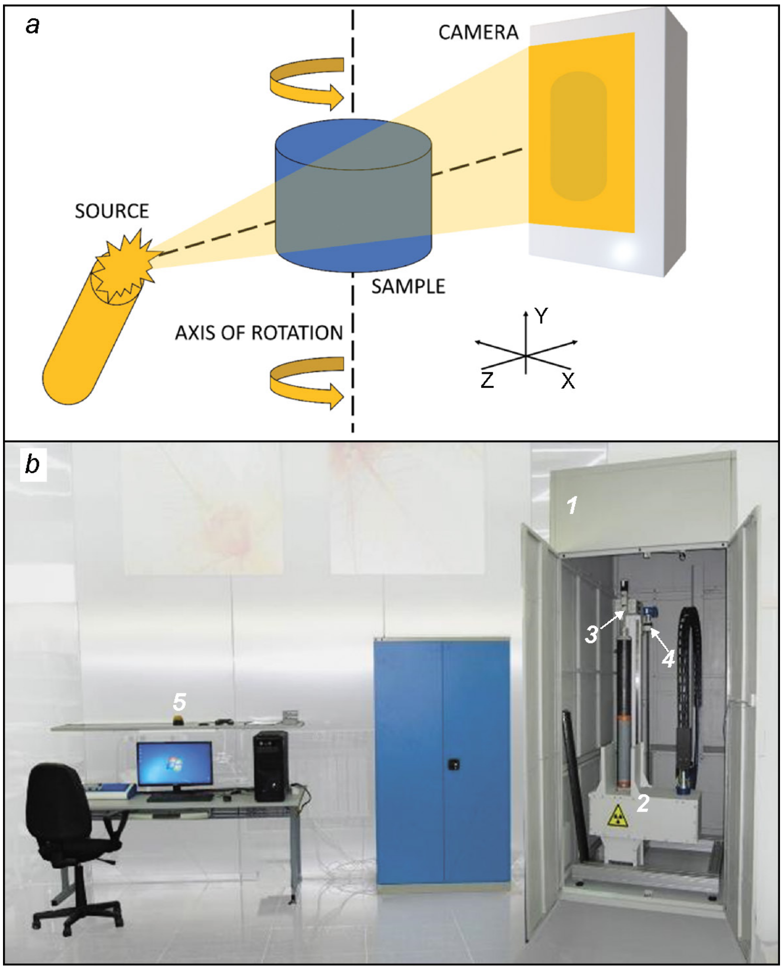

Fig. 1. Computed Tomography system:
a – scheme of the mutual arrangement of the system elements and the sample in a three-dimensional coordinate system with the main system parts: the source of X-rays, the object of study, the detector, fixing the level of radiation absorbed by the object; *b* – photo of a CT-180 tomograph located at the Geological Faculty of Lomonosov Moscow State University. The main parts of a CT scanner are: *1* – a protective cabinet; *2* – a core holder; *3* – a mobile support of an X-ray apparatus; *4* – an X-ray detector moving along the core; *5* – a PC for processing CT data (http://geologika.ru/)

Рис. 1. Система компьютерной томографии:
а – схема взаиморасположения элементов системы и образца при съёмке в трёхмерной системе координат с основными узлами: источник рентгеновских лучей, объект изучения, детектор, фиксирующий уровень излучения, поглощённого объектом; *b* – фотография компьютерного томографа РКТ-180, расположенного на геологическом факультете МГУ имени М.В. Ломоносова (http://geologika.ru/). Основные части томографа: *1* – защитный шкаф; *2* – кернодержатель; *3* – подвижная опора рентгеновского аппарата; *4* – детектор рентгеновского излучения, перемещающийся вдоль керна; *5* – ПК для обработки данных КТ (http://geologika.ru/)

ples collected earlier on Elbrus glacier into the plastic tube. The tube was then frozen (−20 °C). Additionally, layers of mineral particles collected in Elbrus were also added. The CT system consists of the X-Ray source, an object of the study and the detector, that determine the level of radiation absorbed by the object (Fig. 1, *a*) [12].

A separate two-dimensional image corresponding to the intensity of X-Ray radiation after passing through the sample forms the shadow projection. The main principle of computed tomography is to obtain a set of such projections from different angles. This is usually accomplished by its subsequent stepwise rotation. In addition to the x-ray projection of the sample, information about the spatial resolution of the object, the source, and the detector is recorded in contrast with classical X-Ray radiography [4]. The brightness (different gradations of gray) on the X-Ray shadow projection reflects the attenuation of X-Ray radiation, because of the scattering and absorption of the signal passed through the sample. The attenuation depends on the density and the atomic number of the material from which the object is composed. When X-Rays pass through a material, the radiation absorption level can be associated with four types of interaction: photoelectric absorption, Compton scattering, the formation of electron-positron pairs, and coherent Rayleigh scattering. The set of obtained X-ray patterns is then recalculated into a set of density sections that reflect the internal structure of the sample. This operation is called reconstruction. The most common way to display CT densities is to distribute the shades of gray on the graphic slices formed by the system during reconstruction. Lighter shades correspond to a higher density, and darker colors correspond to a lower density. A computed tomograph RCT-180 at the Geological Department of Lomonosov Moscow State University was employed in this study (see Fig. 1, *b*).

This instrument is used for the study of rocks, soils, unconsolidated bottom sediments, biological samples, etc. It allows scanning cores with a length of 1 m and with a diameter of 10 cm. The scanner is characterized by a 150–250 μm spatial resolution, 100 × 100 × 1000 mm active area and 160 kV intensity. The stage rotates for full 360°, in 0.3° steps, with carriage lift 0.2 mm. For the artificial core, the following parameters were used: 1.5–5 mA amperage, 160 kV intensity, and 115 μm spatial resolution. For Elbrus ice cores the parameters of computer tomographer retained the same, except for the spatial resolution (230 μm). The average survey time was approximately 1.5 hours. The data reconstruction was performed using the TomoViewer software, Geologika (Novosibirsk). The data were processed on the Dell Precision T5500 workstation using software products: Data Viewer, CTan (calculations and construction of 3D models of radiopaque components) and CTVol (visualization of volumetric models). Here we also used the results of the chemical and isotopic composition of the Mt. Elbrus ice core. The isotopic composition was measured in Climate and Environmental Research Laboratory, St. Petersburg, while major ions were studied in the Institute for Geosciences and Environmental Research, Grenoble, France [14].

Results

The test core. The artificial ice core contained ice layer, firn and firn with ice lenses to represent the variety of the possible structures in real ice cores (Fig. 2). We also added fine mineral particles from a snow sample collected previously in snow pit in Elbrus as well as some coarse particles collected from

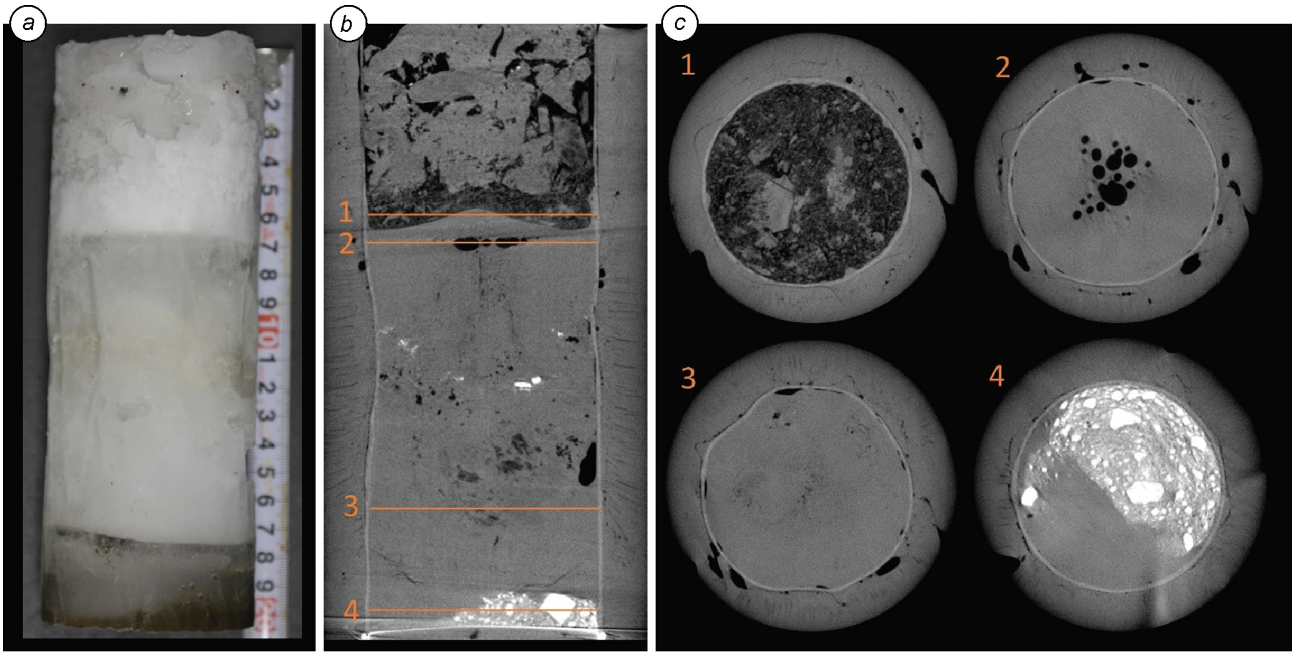

Fig. 2. Artificial ice core:
a – a photo of the ice core with a scale bar (length of the core is 20 cm); *b* – stereological visualization of a longitudinal section of the core with its internal structure; brightness (various shades of gray) reflects the difference in absorption; based on this, areas of ice core with different composition were identified: black color (maximum absorption) – air inclusions, dark gray – firn, gray – ice, white (minimum absorption) – mineral inclusions; *c* – ice core cross sections: 1 – core interlayer with firn (dark gray color); 2 – core interlayer with ice (gray color) and the formation of air bubbles (black color); 3 – core interlayer with ice; 4 – core interlayer with mineral inclusions (white color) and ice. The orange lines indicate the places of the core cross-sections

Рис. 2. Искусственный керн:
а – фотография керна с масштабной линейкой (длина керна 20 см); *b* – стереологическая визуализация продольного среза керна с его внутренним строением; яркость (различные оттенки серого) отражает разницу в поглощающей способности: чёрный цвет (максимальное поглощение) – включения воздуха; тёмно-серый – фирн; серый – лёд; белый цвет (минимальное поглощение) – минеральные включения; *c* – поперечные срезы керна: 1 – прослой керна, вмещающий фирн (тёмно-серый цвет); 2 – прослой керна, состоящий из льда (серый цвет) с образованием пузырьков воздуха (чёрный цвет); 3 – прослой керна, состоящий изо льда; 4 – прослой керна с минеральными включениями (белый цвет) и льдом. Оранжевые линии соответствуют поперечным срезам керна

the rock outcrops near the drilling site. Fig. 2, *b* shows the stereological visualization of the artificial ice core. Main structure heterogeneities were detected. The brightness (different gradations of gray) on the X-ray shadow projection reflects the attenuation of X-ray radiation, due to the effects of scattering and absorption of the signal passed through the sample. The cross sections with contrasting media are shown, namely mineral inclusions, ice, firn and air bubbles (see Fig. 2). The optical density corresponds to the degree of of X-rays attenuation of transparent objects or reflection of light by an opaque object. Optical density can be calculated using the formula $OD = \log_{10}(I_0/I)$, i.e. I_0 – incident optical intensity, I – transmitted optical intensity. The types of objects with the highest values of optical density (270 and more) are the dense mineral inclusions. The average value of the optical density for ice in an artificial core is 120–150. The optical density of air approaches 0. Considering the results of artificial ice core, the inner structure heterogeneities in the natural ice cores can be detected using the same approach.

Shallow core–2017. The Elbrus shallow firn core was dated using well preserved seasonal stable isotopic oscillations. The seasonal amplitude of $\delta^{18}O$ change was 25.2 ‰, with average values being −25 ‰ in winter and −10 ‰ in summer respectively. The ice core covers the period of 2012–2017 with mean annual accumulation rate of 2200 mm w.e. Three sections used in CT analyses correspond to the layers accumulated during the warm season of 2012. A detailed description of section's stratigraphy was made in the cold laboratory. It was revealed that the cores are composed mostly of firn and contain mineral particles. Ice layers and dust horizons can be visually identified. The distribution profiles of the main chemical elements are presented in Fig. 3 together with density characteristics of the core. The density was measured in cold laboratory using discrete sections for every 10 cm. The maximum density of 0.8 g/cm^2 was observed for the layers at the edge of sections 42 and 43. The distribution profiles of Ca^{2+}, Mg^{+}, NH_4^{+}, SO_4^{2-}, and Fe^{2+} were analyzed to find similar patterns.

The values for all ions were elevated in section 41: for Ca the maximum value is 100 ppb, for Mg – 80 ppb, for NH_4 – 800 ppm, for SO_4 – 1850 ppm, for Fe – 60 ppm. Concentrations decreased in section 42: NH_4 = 400 ppm, SO_4 = 600 ppm, Fe = 25 ppm, and the minimum are equal to 10 ppb for Mg. The ion concentrations level off in section 43. This distribution is due to the presence of a large amount of dust in ice in the section 41. A double peak of calcium, corresponding to two dust layers in the ice core is observed also in Fe profile (section 41). When considering the dependence of the presence of chemical inclusions on optical density, it was revealed that the high concentration of Ca indicated the dust layer that can be identified by CT.

In Fig. 4 separate dense horizons are observed, which are reflected in the variations of chemical elements. Pronounced in density is only the dust layer in which concentrations of all chemical elements are rising. However, this may be due to the sampling technique in 10-cm increments, which could cause the chemical signal to be blurred or not manifested at all. The section shown in the red frame in Fig. 4, *a* and *b* has the highest density values. When compared with the gradations of the artificial core, it was suggested that the formations may be separate mineral dust particles. It is possible that individual dust particles in the ice core can be observed using the CT technique. The problem that arises when interpreting data is the separation of noise and the signal from the actual crystals. For their separation, it is necessary to create criteria for the selection of density classes. Under low resolution imaging conditions, it is often difficult to find an increase in the density reflected in CT images corresponding to an increase in the concentration of inclusions. These shortcomings should be considered in future studies.

Another important advantage of this technique is the ability to trace the inclination of the layers in three dimensions (see Fig. 4, *c*), since usually information about the 3D structure of the ice layers is not available. It is possible to clearly trace the firn layers and calculate their angle of inclination relative to the borehole. The firn core from the depth of 2020–2180 cm is composed of firn and the inclination, in this case, is due to the influence of the surface microrelief and possible internal inhomogeneities. The calculations showed, that the angle of inclination of the layers in the studied sections varies from 6 to 9°. With further use of the CT technique for deeper core sections such information may provide insights into basal ice flow disturbances. Such information is important to verify the ice flow and depth age modelling.

Structural features. The method of computed tomography allows determining the internal structure of the core. According to the obtained CT data, it

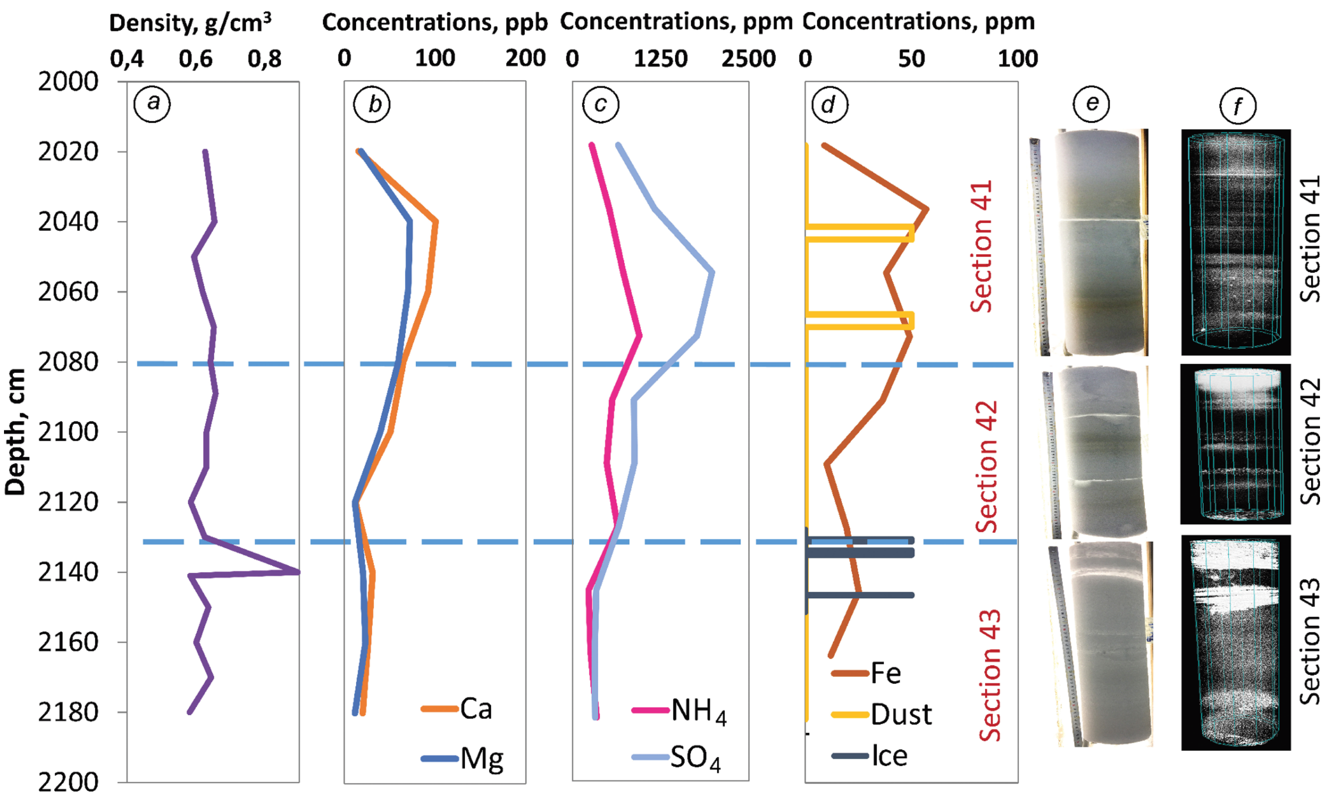

Fig. 3. The distribution profiles of the density, chemical elements, CT data and visual images in three sections of ice cores (sections 41–43) from the depth 20,31–21,87 m.

Dashed blue lines indicate the boundaries between core sections. The distribution profile: *a* – density measured in freezer conditions every 10 cm: *b* – Ca and Mg; *c* – NH_4 and SO_4; *d* – Fe, horizons of mineral dust and ice interlayers; *e* – photographs of sections with visible layers of dust and ice horizons; *f* – stereological visualization of sections with identified internal heterogeneity

Рис. 3. Графики плотности, химических элементов, КТ-данных и фотографии ледниковых кернов в трёх секциях керна (секции 41–43) с глубины 20,31–21,87 м.

Пунктирные голубые линии – границы между секциями кернов. Профили распределения: *a* – плотности, измеренной в условиях морозильной камеры через каждые 10 см: *b* – Ca и Mg; *c* – NH_4 и SO_4; *d* – Fe, горизонтов минеральной пыли и прослоев льда; *e* – фотографии секций керна с видимыми прослоями пыли и ледяных горизонтов; *f* – стереологическая визуализация секций с выявленной внутренней неоднородностью

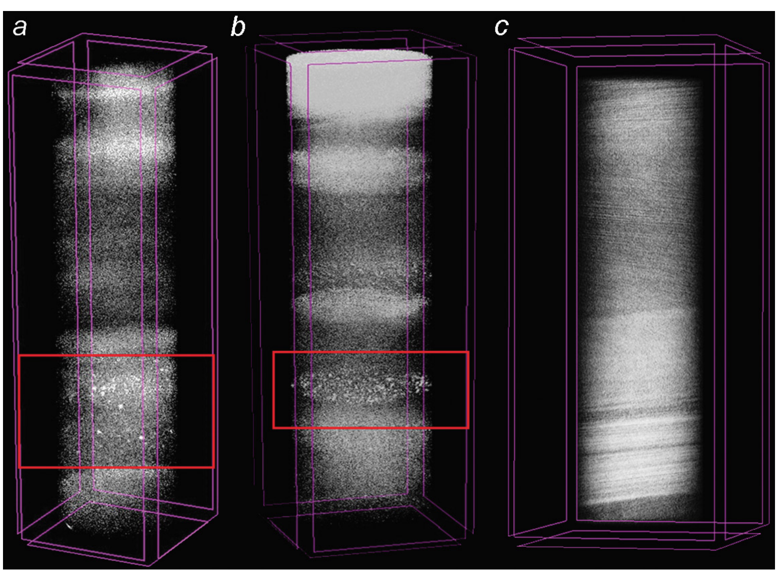

Fig. 4. Stereological visualization of ice cores:
a – lower part of section 41; *b* – section 43 with different absorption of horizons; in the red rectangles – presumably mineral dust particles; *c* – upper part of section 41 with visible slope of the firn layers

Рис. 4. Стереологическая визуализация ледниковых кернов:
a – нижняя часть секции керна 41; *b* – секция 43 с различными по абсорбционной способности горизонтами; в красных прямоугольниках – предположительно минеральная пыль; *c* – верхняя часть секции 41 с видимым наклоном слоёв фирна

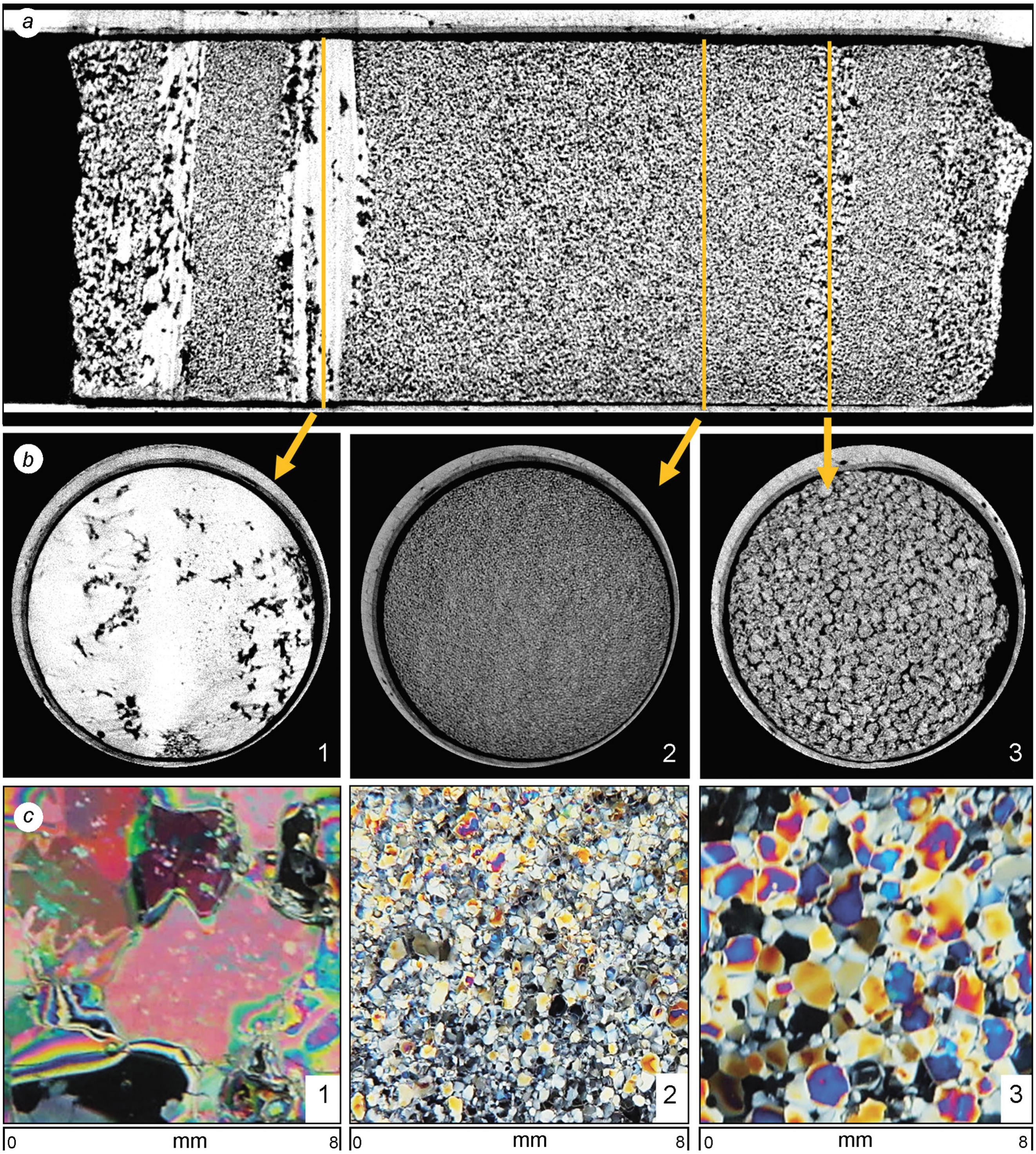

Fig. 5. The inner structure of the ice core:
a – stereological visualization of the section 43; yellow lines with arrows indicate the cross sections; *b* – cross-sections of the core: 1 – ice (white color); 2 – fine-grain firn (dark gray color); 3 – coarse-grain firn (gray color); *c* – thin sections from the same horizons with crystals of: 1 – ice; 2 – fine-grain firn; 3 – coarse-graine firn

Рис. 5. Внутренняя структура ледникового керна:
a – стереологическая визуализация секции 43; жёлтые линии соответствуют поперечным срезам керна; *b* – поперечные срезы керна: 1 – лёд (белый цвет); 2 – мелкозернистый фирн (тёмно-серый цвет); 3 – крупнозернистый фирн (серый цвет); *c* – шлифы кернов из тех же горизонтов с кристаллами: 1 – льда; 2 – мелкозернистого фирна; 3 – крупнозернистого фирна

is possible to identify individual stratigraphic layers, without cutting of the samples. In addition, using the CT technique allows distinguishing the crystals composing the individual density layers according to their size. In order to test whether the resulting image also provides information on the size of the crystals, a comparison was made with the firn and ice flat-surface vertical sections. The sections were made for each horizon, distinguished visually by stratigraphy for all three sections (a total of 30 thin sections). Subsequently, a comparison was made of vertical sections and horizontal core sections using CT. Below we list the most illustrative examples. The horizontal sections of various parts of the core were obtained using the DataViewer program. In this case, mineral inclusions are absent, therefore the densest layer (ice) is displayed in white. The core section 43 consisted of firn with some ice layers up to 4 cm thick (Fig. 5). The ice layers, shown in the first cross section, have a crystal size of up to 6–8 mm. They are characterized by the highest contrast and are easily detectable on the CT images. The second cross section depicts the firn that is representative for most of the core used in this study. As seen in the cross section, the crystals have dimensions on the order of 0,1–0,5 mm. They are not distinguishable on the CT image and are revealed only as noise. The third section displays a coarse-grained firn, the crystal size of which reaches 1 mm. Such firn can be identified by the results of a CT scan. However, there is an ambiguity in the correct interpretation of such horizons associated with a large amount of noise.

Thus, the use of computed tomography allowed identifying heterogeneities in the structure of the ice core. They were not recorded when the core was previously inspected visually. Moreover, in some cases, it was possible to identify patterns of crystals of different sizes in the image. However, only contrasting media with a large difference in the size of crystals are confidently distinguished on the background of fine-grained firn: the ice and coarse-grained firn.

Conclusion

The method of computed tomography was tested on artificial and actual ice cores. The technique has several clear advantages. Compared with microtomography, ordinary CT allows examining samples up to 1 m length. It is non-destructive, meaning that the samples do not need to be cut and melted. In this study, it was possible to solve the problem experienced by the previous researchers employing the CT technique, related to maintaining the proper temperature conditions for the ice. A special cryothermos was designed, that does not require any special investments, as in the case of using a special walk-in freezer laboratory. However, at this point cryothermos is not completely satisfactory and needs to be further improved, since it was not able to maintain the proper conditions for the ice for more than 5 hours. Reliable results were obtained for the test ice core. That sample was characterized by a large contrast in composing media. Density, porosity and structural features of the Elbrus core sections were determined. Correlations with several (Ca, Fe) chemical elements were found. The question of whether it is possible to decipher individual dust particles in large-scale dust horizons still remains open and needs further verification. Information on the crystals size can be obtained using the CT data especially when there is a high contrast with the background medium. It should be noted that the obtained CT data were characterized by a large amount of noise. To identify all structural heterogeneities and clearly separate between inclusions and the noise, it is necessary to carry out the imaging with higher resolution. This will require substantially greater computer storage capacity. In further studies, it is necessary to pay close attention to the choice of spatial resolution, to design a longer-lasting cryothermos for maintaining the proper temperature conditions for the ice cores, and to study a greater number of firn and ice cores to fully explore the possibilities of a CT method.

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Применение методики компьютерной томографии для неразрушающего анализа ледниковых кернов

В настоящее время при изучении ледниковых кернов используется большое число современных методов. Однако многим из них свойственны недостатки, связанные с разрушением и таянием образца при анализе, что препятствует дальнейшему использованию кернов. Компьютерная томография (КТ) – способ получения послойных срезов объекта с помощью рентгеновских лучей. Яркость (различные градации серого) на рентгеновской теневой проекции отражает ослабление рентгеновского излучения за счёт эффектов рассеивания и поглощения сигнала, прошедшего через образец. Ослабление зависит от плотности и атомного числа материала, из которого состоит изучаемый объект. Преимущество метода КТ – его быстрота: затраты непосредственно на съёмку – минимальные, при этом объём получаемой информации – весьма существенный. В нашей работе использовался компьютерный томограф РКТ-180 на геологическом факультете МГУ имени М.В. Ломоносова. Метод компьютерной томографии был протестирован на искусственном и настоящем кернах (ледниковые керны с Западного плато Эльбруса). Авторы решили проблему, связанную с поддержанием температурных условий льда, которая была у предыдущих исследователей КТ-методики. С этой целью был создан специальный криотермос, поддерживающий температуру на уровне −25 °С.

В результате работы с секциями керна Эльбруса получены данные о его плотности, пористости и структурных особенностях. Установлены корреляции с рядом химических элементов, а также явная зависимость оптической плотности по результатам КТ от содержания кальция. Ледниковый керн с глубины 2020–2180 см сложен фирном, наклон которого обусловлен унаследованностью микрорельефа поверхности и возможными внутренними неоднородностями. Расчёты показали, что угол наклона слоёв в изучаемых секциях изменяется от 6 до 9°.

Метод компьютерной томографии позволяет определить внутреннюю структуру керна. Также по данным КТ можно получить сведения о размерах кристаллов. Было проведено сравнение поперечных срезов кернов КТ со шлифами фирна и льда. Шлифы были сделаны для каждого горизонта, различаемого визуально по стратиграфии для всех трёх секций. Общее число шлифов – 30. Далее было проведено сопоставление шлифов и горизонтальных срезов керна по КТ. Установлено, что лучше всего кристаллы отображаются при большом контрасте с фоновой средой. В данном керне фоном служил мелкозернистый фирн, на контрасте с которым в результатах КТ были выделены ледяной горизонт и горизонт с крупнозернистым фирном. Значительный недостаток полученных данных КТ – большое количество шумов. При дальнейших работах необходимо с повышенным вниманием подойти к выбору пространственного разрешения, создать термос с увеличенным временем сохранения температурных условий льда, а также изучить большее число образцов ледниковых кернов для выявления сезонных и годовых закономерностей.

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