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Broadband measurements of dielectric characteristics of ice near 0 °C temperature

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The paper presents a technique for ultra-wide-band measurements of dielectric permittivity using the microwave and optical ranges of weakly absorbing inhomogeneous media. Measurements of fresh ice near the phase transition point (at $0\,^\circ$C) were performed. The ratio of the extreme values of the probing frequencies used in the experiments is $\sim 10^6$. The recent detection of a new phenomenon has been confirmed — „enlightenment" (an increase in the transmitted radiation power in fresh ice) in the microwave and optical ranges up to tens of percent or more for samples with a thickness of ~ 0.1 m. The greatest effect was observed when the electric field vector was positioned parallel to the base plane of the ice crystals. A mechanism of the phenomenon associated with the occurrence of plasmon resonance on structures appearing in nanometer-thick layers formed during the flow (plastic deformation) of ice is proposed.

Keywords: dielectric constant, fresh ice, phase transition point, ultra-wide-band measurements, microwave and optical range.

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Introduction

Research of electromagnetic characteristics of natural objects of nano- to space-scale plays a key role in deciphering data of their spectroscopy. There is a known number of methods of such research based on considering waves which are passing through the objects or reflected from them [1,2]. These methods have been developed to result in impressive successes when studying the space objects to submicron structures of various materials. For example, traces of life have been searched in the Venus atmosphere along the line of phosphine (PH_3) at the frequency of 266.94 GHz using ground appliances [3]. The Earth surface is actively researched now from space [4,5]. There is also research of microscopic and macroscopic characteristics of media, in particular, water [6], for which at the temperature of $-45\,^\circ$C and under the atmospheric pressure [7] there is confirmation of existence of the Widom line associated with the second critical point.

At the same time, due to complexity of a structure of the objects many cases require improvement of the dielectric research methods and development of special measurement techniques.

This paper objective is a review of the problem of research of an electromagnetic state of fresh ice around its phase transition temperature. The posed problem is associated with a special role of water in existence of the biosphere and with its various applications. It should be noted that physical-chemical properties of water are still understudied as manifested by its 75 anomalies known as of now [8].

The papers [9,10] have shown that within the interval from $-0.5\,^\circ$C to $0\,^\circ$C ice shows an anomaly of the microwave and optical characteristics when electromagnetic radiations pass therethrough. The anomaly consists in substantial reduction of electromagnetic losses in ice when its temperature approaches a value of melting temperature. The anomaly is similar to the effect of „enlightenment" of a medium, which is known in optics, but in this case it is associated with phenomena in a phase transition. Researchers avoid studying such ice as they suggest that liquid inclusions appear in it. However, the inclusions increase a loss factor unlike the anomaly found in [9,10].

The present research has analyzed available and new measurement results which were obtained by various methods in laboratory and natural conditions in the wide range of frequencies from the microwave to the UV ranges. Their comprehensive review has made it possible to obtain more complete information about the object and propose a mechanism of structural features of melting ice which result in the effect of medium enlightenment. Thus, the efficiency of ultra-wide-band measurements has been demonstrated.

1. Research method and results

In terms of its characteristics, fresh ice is similar to dielectric materials and it main electromagnetic characteristic is relative complex dielectric permittivity $\dot{\varepsilon} = \varepsilon' + i\varepsilon''$. Techniques of measurement of its real and imaginary parts are well developed. The present research has used methods of resonance cavity measurements and ice illumination

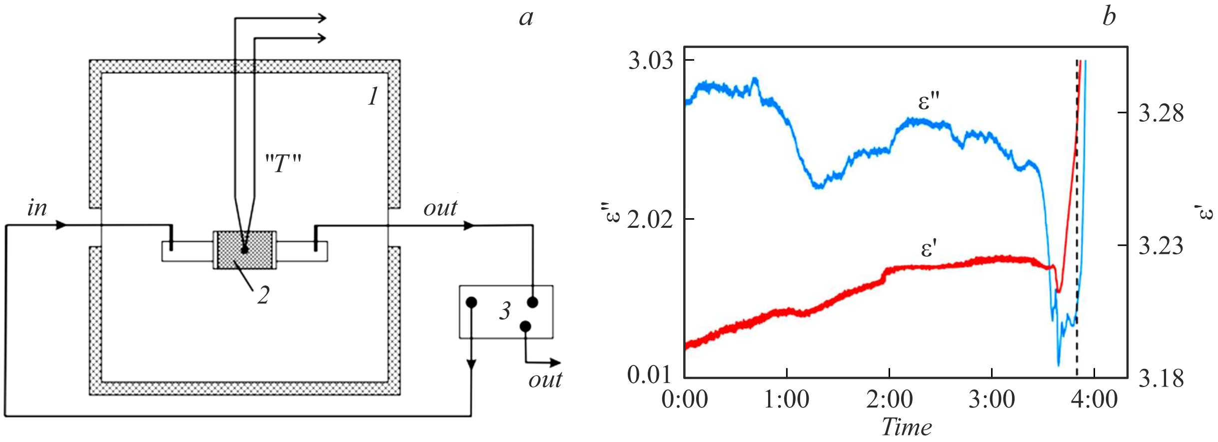

Figure 1. *a* — Diagram of the experimental setup for studying dielectric properties of ice: *1* — climate chamber, *2* — resonant cavity, *3* — scalar analyzer, *T* — temperature sensor; *b* — the dependence of ε' and ε'' of ice when heating it, on time; measurements around 3.4 GHz; the dashed line — start of sample melting at 0 °C.

measurements using monochromatic radiation as well as of radiometric measurements within the frequency band of $\sim 10\,\%$ of the carrier frequency. The used approach was characterized by measuring the samples in the ultra-wide-band range of frequencies with a ratio of extreme wavelengths of $\sim 10^6$. In also included use of broadband noise signals in the microwave range, thereby making it possible to eliminate interference phenomena on heterogeneities of the structures of the studied object.

2. Resonant cavity method

The resonant cavity measurements make it possible to carry out accurate measurements of dielectric permittivity on small-sized samples. They are convenient for temperature measurements of the real ε' and the imaginary ε'' part of relative complex dielectric permittivity of ice. In our experiments, the dielectric characteristics have been measured by means of a rectangular resonant cavity of the H_{101} type with its complete filling with ice. In the research performed, we have used the half-wave resonant cavities at the lowest resonance frequency with eigen frequencies of 2.8, 6.1 and 13 GHz.

Fig. 1, *a* shows the diagram of the experimental setup. In particular, this experiment has used the resonant cavity of theH_{101} type with the internal dimensions $58 \times 26 \times 25$ mm. The resonant cavity with the sample was placed in the Espec climate chamber, wherein the temperature of the pre-cooled sample was increased at a heating rate of about several degrees per hour. With such the heating rate, the difference of temperatures between the sample and its edge will not exceed 0.1 °C. The temperature was measured by means of a thermocouple sensor with absolute accuracy of 0.1 °C. The resonant cavity was connected to the scalar circuit analyzer P2M−18/2 produced by „Micran“. Accuracy of determination of ε'' was 1 %, while ε' — ~ 0.1 %.

Fig. 1, *b* shows the dependences of ε' and ε'' on time when heating the sample from−30 °C, which identify a specific feature within the range of the temperature above −0.5 °C. It is important to note that the measurement results substantially depend on a direction of strength of the electric field in the resonant cavity in relation to a basal plane of ice crystals. So, in the experiments such samples with predominant spatial orientation of the basal planes were specially cut out of lake ice with ultra-fresh water (salt concentration ~ 0.1 kg/m^3, and in ice $\sim 10^{-6}$ kg/kg). As established in glaciology, during formation of lake ice, after the initial stage of cover formation 90 %−100 % of the crystals in the ice cover are oriented with its main optical axis „C“ in perpendicular to an ice-air interface surface and can deviate from the normal within several degrees. The same axis „C“ is perpendicular to the basal plane of the crystals of hexagonal ice (Ih). In accordance with classification of Cherepanov, it corresponds A1-type ice, which is formed in small lakes at the temperature gradients 1−4 K/mm in the sub-ice layer of water [11].

The experiments were carried out with three resonant cavities. For the other two experiments, when filling the resonant cavity with ice the frequencies were around 1.6 and 7.6 GHz. In all the cases, there was observed reduction of ε'' at the temperature within the interval from −0.5 to 0 °C, which was twofold in some cases.

2.1. Experiments of illumination of ice blocks in the microwave range

In this method radiation was passed through the parallelepipedic sample. A signal was formed using a monochromatic radiation oscillator, while for reception of passed radiation a detector receiver was used. By these measurements, the paper [12] has detected the effect of „enlightenment“ for the first time. By the similar method, the power of passing radiation was simultaneously measured

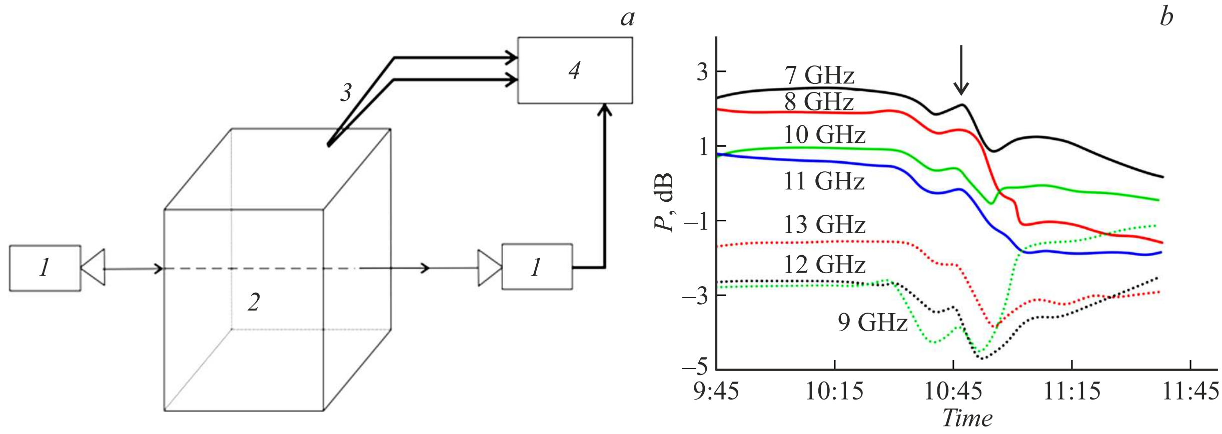

Figure 2. *a* — Diagram of the unit for measuring power (P) in decibels in the microwave range for radiation passing through ice around the phase transition temperature: *1* — scalar circuit analyzer, *2* — sample (ice block), *3* — thermocouple, *4* — data acquisition and recording system; *b* —the dependence of power of radiation passed through the ice block at the various frequencies on time. The arrow marks the effect of „enlightenment".

within the frequency range from 7 to 13 GHz using the circuit scalar analyzer P2M−18/2 produced by „Micran". Fig. 2, *a* shows the diagram of the experimental setup.

The ice samples were extracted from the ice cover of the mesotrophic freshwater lake Arakhley (Zabaykalsky Krai) in February-March. During the experiment, the sample was placed to direct the vector of the electric field of the electromagnetic waves either in parallel or perpendicular to predominant arrangement of the basal planes of the ice crystals.

The results of the performed measurements are shown on Fig. 2, *b* for the case when the basal plane is parallel to the vector of the electrical field. For convenience of comparison of power of passing radiation at the various frequencies, the curves are mutually shifted along the vertical axis. This is due to the fact that owing to close values of power of radiation passing through the ice sample some curves are superposed onto the others. Power variation with „enlightenment" was approximately 1 dB for the block thickness ~ 0.15 m.

2.2. Measurement method using thermal radiations

When using relatively broadband radiations, it is possible to avoid the interference phenomena in the measurements, which distort the results, in order to detect patterns of variation around 0 °C. In case of polarization measurements, it is possible to determine spatial ordering of the ice crystals Ih in the samples due to the effect of „enlightenment". It was previously believed that it was impossible due to weak anisotropy of ε'' in the ice crystals Ih.

The diagram of the measurements as per the proposed method is shown on Fig. 3, *a*. The experiment method was as follows: the radiometric receiver was installed with direction of an antenna axis at the angle of 45° to the horizon. The ice sample in the form of the plane-parallel plate of the thickness of d was installed perpendicular to the antenna axis. The horizontal surface is covered with the metal sheet in order to reflect radiation of cold sky This method includes measuring intrinsic radio-heat radiation of the sample and sky radiation attenuated by the sample. Interference of this radiation is reduced to acceptable values when $d > c/\delta f$, where d — the length of the radiation path in the sample, Δf — the width of the measured-radiation band, whereas c — the light velocity in the medium. For the frequency of 30 GHz and the band of the frequencies of 3 GHz d exceeds 0.1 m. The measurements included use of the radiometer to the frequency of 34 GHz with the band ~ 3 GHz. The intrinsic thermal radiation of the ice sample (its radio brightness temperature T_b) was measured at four linear polarizations, i.e. the horizontal one (HP), the vertical one (VP) and at the angles of +45° and −45°. The radiometer sensitivity was 0.1 K with the time constant of 1 s. The measurements were performed in daytime, with cloudless atmosphere, when the air temperature increased above 0 °C.

Fig. 3, *b* shows the experiment results which exhibit presence of noticeable enlightenment of the sample at the certain polarization, thereby revealing structuring of ice cut out of the fresh ice cover. The areas of reduced radio brightness (A, B, C) are explained by spatially inhomogeneous of sample melting. In the area „A" the maximum reduction of radio brightness temperature is about 25 %. It should be noted that the measurements of T_b for the different polarizations have shown the same value and that the given data correspond to freshly formed ice. For the case when the basal plane of the sample crystals was perpendicular to the surfaces, the value of T_b varied from 12 % for the polarization directed parallel to the basal planes to 9 % for the polarization directed perpendicular to the basal planes of the ice crystals. These values are referred to ice, which was formed in three months prior to the experiment.

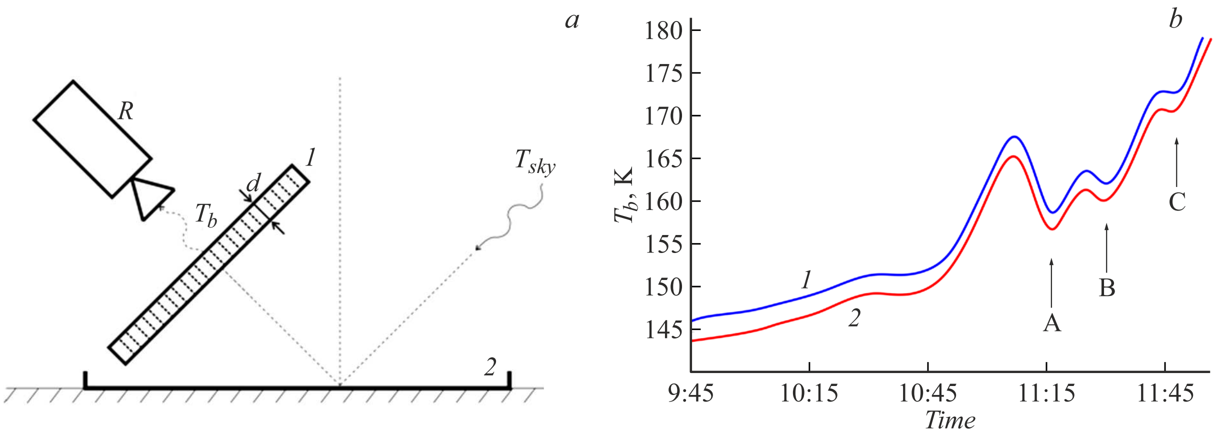

Figure 3. *a* —Diagram of the experimental setup for studying thermal radiation of the ice samples on the background of cold sky: *R* — polarization microwave radiometer, *1* — plane-parallel ice plate with the sizes $1000 \times 400 \times 100$ mm, T_{sky} — radio brightness of the sky, *2* — metal sheet; *b* — results of variation of the radio brightness temperature (T_b) of the ice sample with time. The digit denotes polarization: *1* — horizontal, *2* — vertical. The arrows mark regions of ice enlightenment.

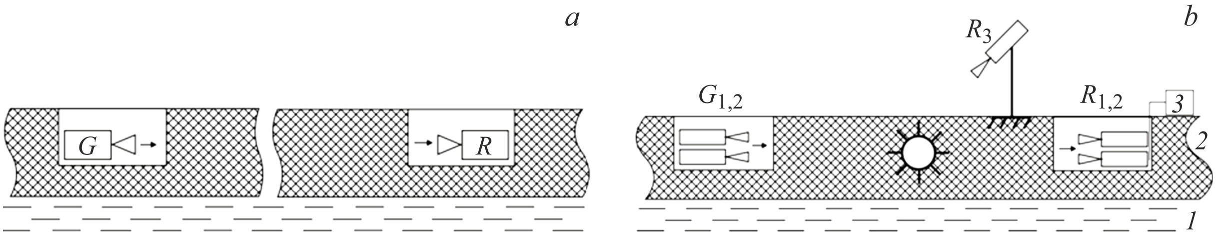

Figure 4. *a* —Diagram of the experiment of radio illumination of the ice cover in the microwave range: *G* — oscillator, *R* — multi-polarization radiometer; *b* —Diagram of the experiment performed to illuminate ice and create pressure in the ice cover as initiated by a powder charge. $G_{1,2}$ — microwave radiation oscillators, $R_{1,2}$ — radiometers, *1* — ice cover, *2* — water, *3* — recording system, *4* — charge detonation area, R_3 — microwave radiometer for observing the ice destruction area.

2.3. Methods of two-position measurements of the ice formations in the natural conditions

These methods are widely applied during radiolocation measurements of, for example, ice glaciers [13]. Besides, it was the paper [13] that noted many anomalies of results of radiolocation of large masses of fresh ice. An option for studying lake ice with daily variations of the temperature has included testing of long measurements for illumination of the ice cover at the 12 linear polarizations in conditions of considerable daily variation of the temperature of surface ice layer of up to 10°C. A diagram of installation of the appliances is shown on Fig. 4, *a*. A large number of the linear polarizations was required to study anisotropy of the ice cover when it is plastically deformed due to temperature stresses.

The present method of measurements includes a capability of affecting the natural object not only with daily variations of the medium temperature, but by other techniques as well, for example, by pulsed local destructions of ice. These experiments were carried out in the paper [14], which created sharp increase in pressure above an ice destruction threshold as provoked by the powder charged (Fig. 4, *b*). The work [14] provides measurements of the power of radiation passing through the ice cover as well as of the radio brightness temperature of the ice cover in an explosion area. With sharp increase in the pressure, there is short-term increase in the power of passing radiation by about 25%. There is also short-term decrease in the radio brightness temperature followed by its increase, which is associated with freezing of liquid water. In the natural conditions, with the daily variations of the temperature, the effect of „enlightenment" was possibly observed in the paper [15], which has found that linear attenuation in ice cores varied from 13 to 0 dB/m.

In all the measurements performed by the given method, in the natural conditions natural fresh ice has exhibited a recorded anomaly, in which there is the observed effect of „enlightenment". It was the most pronounced with higher temperatures of the ice cover and it was almost absent when the temperature of the upper layer of ice was below −20°C.

2.4. Experiments of illumination of ice blocks at the wavelengths of 535 and 370 nm

The paper [10] has done research on transmittance of optical radiation through a block of fresh ice. The experiments for illuminating the ice blocks at the wavelengths of (λ) 535 and 370 nm have been aimed at searching the effect of „enlightenment" in the said ranges. The aim of the experiment has also included detection of influence of arrangement of the basal planes of the ice crystals in relation to a vector of strength of the electric field on the power of passing radiation. The measurements were performed in the visible and ultraviolet ranges. The visible range has used the semiconductor laser for the wavelength of 535 nm with linear polarization. The UV range has used non-polarized light from the LED emitter for the wavelength of 370 nm. Fig. 5 shows the diagram of the unit for measuring power of the passing radiation depending on time.

The measurement thermocouple was placed in the ice block at the depth of 0.01 m.

According to the method used, radiation was passed through the parallelepipedic sample and it was assumed that reflectance by power (R) from its surface slightly changed during the experiment. It follows from the fact that at the wavelengths used, the real part of the refractive index of ice (n) at $0\,^\circ$C is 1.311 for $\lambda = 535$ nm and 1.334 for $\lambda = 370$ nm [16,17], which yields a value of reflectance at the air-ice boundary $R \sim 0.018{-}0.02$ as found from the formula $R = [(n-1)/(n+1)]^2$. If for some reason n changes by 0.001, then the increment ΔR will be 0.0001 or $\sim 0.5\,\%$. This change is possible, if layers of liquid water occur at the boundary. If the lay thickness is of an order of the wavelength, then it is necessary to take into account transmittance coefficient of such a three-layer medium. For water $n = 1.333$ ($\lambda = 535$ nm) and 1.334 ($\lambda = 370$ nm). These values are equal or higher than those for ice, so when the water films occur at the initial stage of melting with their thickness below a quarter of the wavelength and interference appears, the transmission ratio should decrease (i.e. radiation attenuation shall increase) [18]. This specific feature can be used for determining a moment when the liquid starts occurring at the ice surface (when analyzing the influence of the boundary layer on radiation transfer). The imaginary part of the refractive index of water and ice can be omitted as its values is of many orders less than the real part.

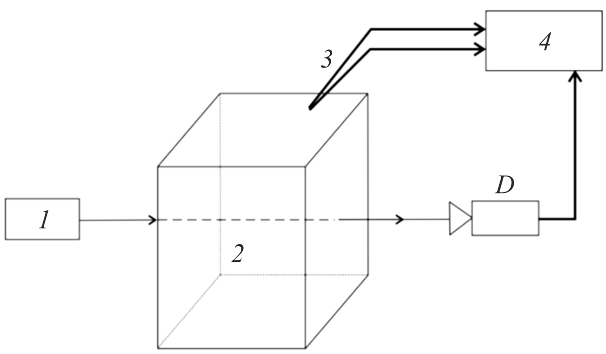

Figure 5. Diagram of the unit for measuring radiation passing through ice in the optical range around the phase transition temperature: *1* — emitter, *2* — sample (ice block), *3* — thermocouple, *4* — data acquisition and recording system, *D* — photodetector.

The ice samples were stored in a freezer at the temperature of $-15\,^\circ$C. The thickness of the samples was 0.10 m. During the experiment, they were placed in the unit and heated at the room they to $0\,^\circ$C. After the ice surface was humid, i.e. after the signs of melting appeared, the experiment was finished. The receiver and source of radiation were at the constant temperature.

The signals from the thermocouple and the photodetector were recorded to the data acquisition system. The signals were registered at the interval of ~ 0.3 s.

Fig. 6, *a* exemplifies study of power of transmittance of visible radiation. It follows from the curve that prior to approaching the phase transition temperature the registered power increases to 25 %. If the vector $\mathbf{E}$ was perpendicular to the basal planes (Fig. 6, *b*), then the effect was substantially lower. The similar dependences were also observed in the UV range

3. Discussion of the results

Based on a specific example of measuring the dielectric characteristics of fresh ice in the microwave and optical ranges, in a dynamic mode when approaching its melting temperature as well as when applying the pulsed load, the effect of „enlightenment" of the medium has been detected. It consisted in reduction of the loss factor (the imaginary part of the relative dielectric permittivity) when the medium starts to flow. It is known that ice flows at any values of mechanical stresses in the medium [19]. The flow is facilitated along the basal planes of the crystal and increases when approaching the phase transition point. The various measurement methods have used electromagnetic measurements within the ultra-wide-band range of the wavelengths — from 0.1 m to hundreds of nanometers, i.e. for extreme frequencies which differ in 10^6 times. All the wavelengths have exhibited a connection of the effect with spatial orientation of the electric field vector and with the predominant position of the basal planes in the sample of ice Ih. It turned out than in case of coincidence of the vector $\mathbf{E}$ and the basal plane of the crystals the linear electromagnetic losses in the medium decreased by dozens of percent.

It was assumed that the phenomenon is associated with occurrence of gliding of ice layers along the basal planes during its heterogeneous heating and creation of shear mechanical stresses. Facilitation of gliding of the ice crystals along the basal planes is well known in cryology. Moreover, the dependence of stress on shear has a descending portion which corresponds to negative differential viscosity of the medium [19]. It should be noted that the electrodynamic characteristics of ice at these conditions have not been studied. At the same time, presence of the descending portion in the dependence of the magnitudes might indicate that there is self-oscillation ordering occurring in it. It is exemplified by the Gunn

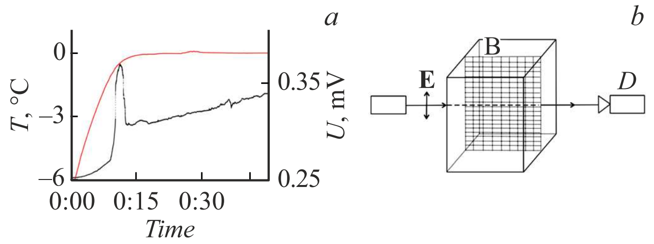

Figure 6. *a* —Ice temperature (an upper line) and power of the linearly-polarized visible radiation passing through the sample, depending on time at the wavelength of 535 nm. Registered power in units of voltage of the photodetector (U); *b* — Diagram of the position of the basal plane (B — one of the planes) and of orientation of the vector of the electric field **E**.

effect in semiconductors, where the dependences of current on voltage exhibit negative differential conductivity. At this, in case of certain geometrical parameters of the layer, there is generation of electromagnetic oscillations.

In case of ice around 0 °C and with occurrence of gliding of the crystal parts, under external impact not just quasi-liquid films appear, but self-organizing conductive structures of nanosizes on it as well. These structures are studied in nanoplasmonics. On the island nanostructures, the variable electric field may be amplified in many times. For example, an effect of gigantic Raman light scattering is known, in which the field can increase in 10^{12} times [20]. An issue of these structures in case of plastic deformation of the ice crystals requires special research.

Conclusions

1. The paper proposes use of the ultra-wide-band measurements of the dielectric characteristics of the weakly absorbing media and their spatial anisotropy when studying a substance structure in a special state, for example, near the phase transition, in the dynamic mode. In the performed study, the ratio of the wavelengths at the range extremes approached 10^6.

2. Based on the example of the measurements of fresh ice when heating it around the phase transition temperature as well as when creating the pulsed pressure, there is the detected effect of reduction of the imaginary part of dielectric permittivity (a kind of „enlightenment" of the medium). The effect was pronounced for radiation, in which the vector of the electric field of the wave coincided with the basal plane of the ice crystals. Based on the microwave data, the effect has been qualitatively pre-explained by association with facilitated mechanical flow of ice along the basal planes of its crystals and occurrence of negative differential viscosity.

3. The measurements in the visible and UV ranges have also confirmed the data (obtained in the microwave range) about occurrence of ice enlightenment and connection of the effect with orientation of the vector of the electric field in relation to the basal plane. Transition to the measurements at the substantially shorter wavelengths has made it possible to draw attention to mesostructures in ice during plastic deformation.

4. Another hypothesis explaining the phenomenon has been posed. It is associated with occurrence of the mesostructures in the form of island films with high electrical conductivity in hyperfine nanometer-thick films. These formations exhibit a resonance phenomenon in surface plasmon oscillation modes. These effects are actively researched now. They are of a broadband nature for irregularly-shaped structures and may result in noticeable change of probing electromagnetic fields, in particular, its scattering, which reach zero frequencies.

5. The result obtained for studying the electromagnetic characteristics of ice requires additional research. They also confirm a promising outlook for research during the ultra-wide-band measurements, especially in case of complex inhomogeneous media, as an additional tool for determining their physical-chemical characteristics.

Conflict of interest

The authors declare that they have no conflict of interest.

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