Single-Channel- and Real-Time Imaging Attenuated Total Reflection Spectrometers for THz range

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Abstract—**Two attenuated total reflection (ATR) spectrometers for study of substances highly-absorbing in the terahertz range have been developed. First system is a home-made single-reflection silicon ATR sampling module with an isolated sample cell to VECTOR-30 and IFS 66v/S Bruker spectrometers. This module was used for the examination of biological objects, including human blood, grounded rat bone, and other tissues. Second system is an imaging terahertz spectrometer using a tunable 100-W terahertz free electron laser as a source and a 160x120 microbolometer focal plane array camera (repetition rate up to 90 frames/s) as a recorder.**

I. Introduction And Background

ABSORPTION coefficients in terahertz for many important substances are very high, and methods based on the transmission spectrometry are unreliable. Our previous experiments [1] on terahertz spectroscopy of bone tissue of intact and senescence-accelerated rats have shown that the errors for the transmission spectroscopy were too large to obtain reliable data. In such cases attenuated total reflection spectroscopy (ATR) can be a good solution. Most of the commercial ATR modules are based on Ge or ZnSe crystals, and the long-wave fraction of spectrum cannot be studied. In this paper we used a home-made single-reflection silicon ATR sampling module to Bruker Fourier spectrometers for the investigation of spectra of a number of substances.

The second, still unsolved in terahertz, problem is spectrally-resolved real-time imaging of films and thin layers. We have designed and fabricated an ATR THz imaging spectrometer, which enables real-time monitoring of transient phenomena.

II. Single-channel ATR module for Fourier spectroscopy

The ATR module with a silicon prism (the internal reflection angle is 45°) and an isolated sample cell enables obtaining spectra of liquids, powders and chemically unstable samples using both VECTOR-30 and IFS 66v/S spectrometers within a spectral range from 1000 to 40 cm^{-1}. Absorption coefficient of the silicon is shown in Fig. 1. Performance characteristics of the module were tested using the substances, whose spectra are known: water, aminoacids, and organic liquids. The module was used for the study of non-biological and biological objects, including human blood and grounded rat bone [2]. As an example we present in Fig. 2 complex refractive index of liquid perfluorodecaline (PFD), one of the components of blood substitutes, as a function of time. It clearly demonstrates instability of PFD on the silicon prism in air.

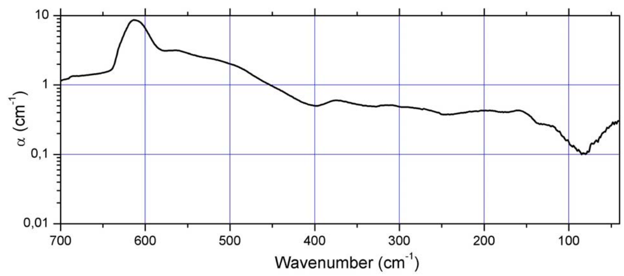

Fig. 1. Absorption coefficient of high-resistivity silicon ($n = 3.42$), measured by comparison of absorbance for two samples of different thickness

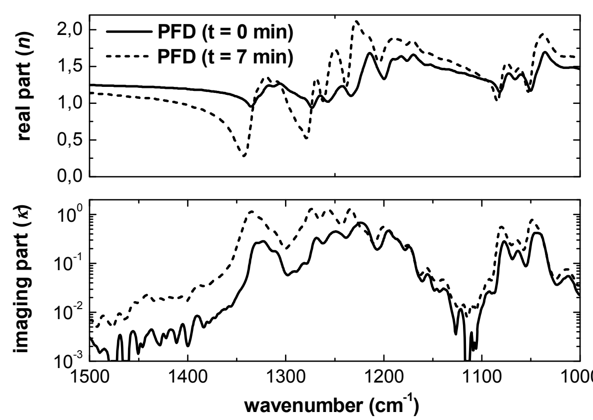

Рис. 2. Time history of perfluorodecaline complex refractive index vs. wavenumber

Data were processed by means of a modified Kramers-Kronig transformation [2] using a program carried over from [3]. Measurements were performed with the non-polarized radiation. Drawbacks of the KK method are the necessity to know a value of real part of the refractive index at the upper frequency bound and large errors of numerical calculations, if the frequency band is narrow. A way to obtain spectra of high precision is to come to a straightforward internal reflection measurement using a monochromatic source.

III. ATR-spectroscopy with a free electron laser

The most intense tunable source of monochromatic

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radiation in MIR and FIR regions is at present Novosibirsk free electron laser (NovoFEL) [4]. First laser (see Fig. 3), tunable within 120 – 240 μm, is operating for the last three year. The second laser (30 – 120 μm) is under commissioning, and the third one is currently assembling.

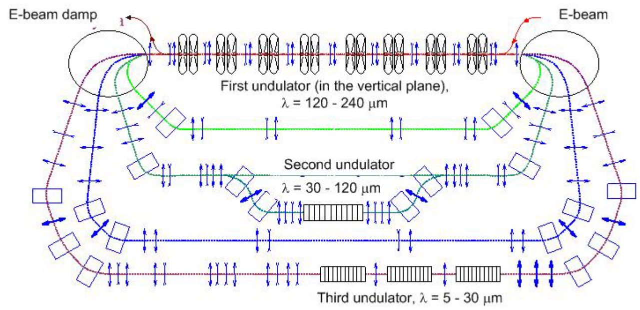

Fig. 3. Schematics of Novosibirsk energy recovery linac and three optical generators

To study the feasibility of ATR spectroscopy with FEL, we have tested operation of a single-channel spectrometer, using *s*- and *p*-polarized radiation at a fixed incident angle of 68°. Data processing included solution of two transcendent Fresnel equations by trial-and-error method. Data obtained with FEL were substantially precise and could be processed with this method, whereas the method did not work in a case of data obtained with Fourier spectrometers. To discover the reason, we have calculated a set of pairs n and κ (Fig. 4), corresponding to known values of the reflection coefficients for water, for two incident angles at $\lambda = 130$ μm. The calculations showed that at any angle switching of polarization does not effect substantially on the trend of $n(\kappa)$ curve, and the determination of the cross-section point cannot be reliable. On the contrary, changing of the incident angle enables reliable determination of the cross-section point.

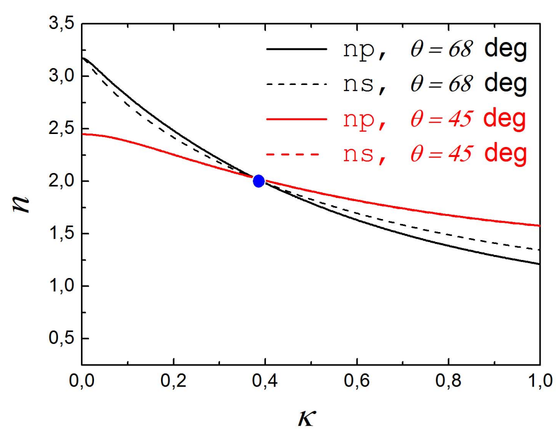

Fig. 4. Curves $n(\kappa)$ corresponding to known reflection coefficients for water. Curves for 45° coincide

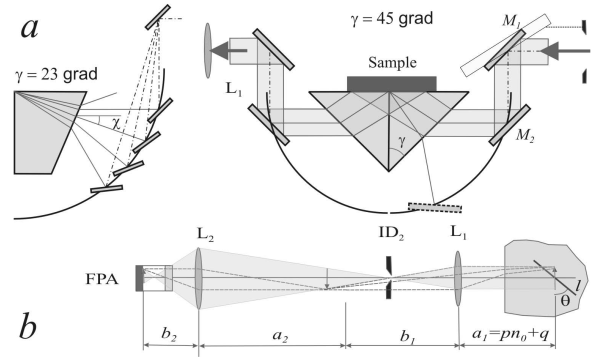

Fig. 5. Design of imaging terahertz ATR-spectrometer with two interchangeable prisms (a) and optical system with a high repetition rate microbolometer focal plane array (b).

Novosibirsk free electron laser beam divergency at the user stations is about $3 \cdot 10^{-3}$ that allows development of an ATR imaging system for study of transient processes. A schematic of a real-time imaging THz ATR spectrometer is shown in Fig. 5. A plane wavefront of monochromatic radiation of Novosibirsk free electron laser, which wavelength can be continuously tuned within a spectral range of 1.2 – 2.4 THz, passes through a computerized system of movable mirrors and ATR silicon prism, with a sample placed on the upper surface, and is imaged with a 90 frames/s, 160x120 microbolometer focal plane array (FPA) [5]. Movable and rotatable mirrors enable the change of the incident angle that is important to ensure correct measurements of complex refractive index of the sample (see. Fig. 4).

Since the penetration length of the evanescent wave is roughly less than a half wavelength, the samples of very limited amount can be investigated. The spectrometer can be applied to spectrally and spatially resolved study of the dynamical processes such as diffusion and chemical reactions.

Acknowledgments

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