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GRAZING INCIDENCE REFLECTIVITY OF HIGH-T_c SUPERCONDUCTORS: MM WAVE TECHNIQUE OF CONDUCTIVITY MEASUREMENTS

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Non-resonant nondestructive technique has been described for investigation of high-temperature superconductive and relative materials through the measurement of the microwave reflection at grazing incidence. As authors discussed earlier, a reflection coefficient of the electromagnetic wave at grazing incidence can be used to determine an absolute complex conductivity over a wide temperature and frequency range. As of now the experimental measurement setup was realized in millimeter wave range using waveguide phase bridge based approach. The conductivity of $YBa_2Cu_3O_{7-\delta}$ film was measured at temperatures higher than critical. Fig. 5. Ref.: 27 titles.

Key words: grazing incidence reflectivity, microwave conductivity, superconducting films.

Microwave impedance properties study of various materials including high-temperature superconductors (HTS) plays important role in fundamental physics and engineering. Different techniques are used for the mentioned above investigation. A number of the techniques make use of different resonant structures [1]. The other ones are based on measurement of microwave power transmitted through or reflected from HTS sample [1]. The first investigation of conductivity by measurements of signal transmitted through the thin superconducting film in cylindrical waveguide was performed using certain assumptions to obtain complex value by measured real part of the transmission coefficient [2]. The further investigations was performed without any approximations by measuring of the real parts of transmission and reflection coefficients [3, 4]; by measuring complex value of the transmission coefficient [5–7] and by measuring of the complex reflected signal [8]. As a form of the non-resonant method, the technique based on short circuit termination in a coaxial cable was also used [9, 10]. The investigation of complex conductivity by non-resonant method is possible over a wide frequency range and allows obtaining absolute values of imaginary part of complex conductivity (penetration depth) while by the resonant methods it is necessary to use fitting procedure [11]. Moreover another advantage of the method is the possibility of the investigations in the temperature region close to and higher the superconductor critical temperature, where the resonant methods has pure accuracy [12]. This fact makes the method very useful for investigations of fluctuation conductivity (which is mainly studied in DC and there is only few works in microwave region [13–17]) and pseudogap effects (which is one of challenges in physics of unconventional superconductivity) [18]. Unfortunately the investigations of the transmitted signal are applicable only for thin films of thickness less than a field penetration depth. Although the reflection coefficient for a thick conducting plate (which is normal to a longitudinal axis of the waveguide) is close to unity. It has small changes under large variation of the sample conductivity [2, 3, 8]. It is evident from a simple relation for absolute value R of the reflection coefficient:

$$R \cong 1 - \frac{4R_s}{Z_0},$$

where R_s is surface resistance of the sample under test and Z_0 is characteristic impedance of free space. The relation $R_s << Z_0$ is true for all conductors including superconductors.

However situation could be improved by using grazing incident p-polarized wave. This fact was recently discovered in the infrared [19] and millimeter [20] wavebands under free-space and rectangular waveguide conditions accordingly. In this case sensitivity of the reflection coefficient to conductivity changes can be increased by order of values and higher [21]. As it is shown by authors, this fact is connected to decreasing of the p-polarized wave reflection coefficient by the approaching to Brewster angle. The Brewster angle is close to 90 degrees for conducting plate, but it is different for the samples with various conductivity, e. g. for superconductor in normal and superconducting states. As a result, the reflection coefficient sensitivity to conductivity changes rises under grazing incidence angles conditions [22].

The report gives a description of experimental setup, where the idea of grazing incidence reflecti-vity technique of conductivity measurement is realized in millimeter wavelength range using waveguide phase bridge-based approach. The first results of the method application to investigation of HTS materials are presented. $YBa_2Cu_3O_{7-\delta}$ film ($T_c = 92$ K) of 300 nm thickness deposited on 0,3 mm sapphire substrate with CeO_2 buffer layer was studied.

1. The experimental setup. To realize the grazing incidence of the p-polarized wave on a sample in K_a-band, the special rectangular waveguide section with fundamental mode H_{10} (fig. 1) was developed. The section has been realized by oblique short-circuit termination by the measured sample. The temperature sensor is placed on a top of the sample. An angle of incidence was chosen equal to 80 degrees on the basis of the theoretical study [21, 22].

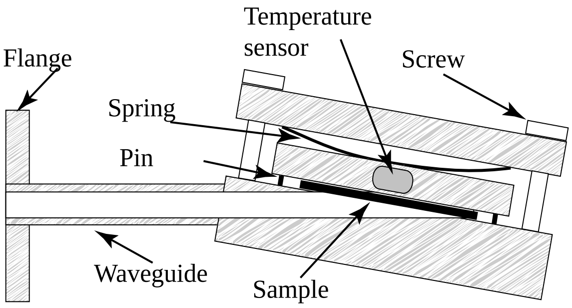

Fig. 1. Waveguide section

The computer-controlled experimental setup (fig. 2) for investigation of temperature dependence of complex reflection coefficient was developed in K_a-band based on phase bridge method [23].

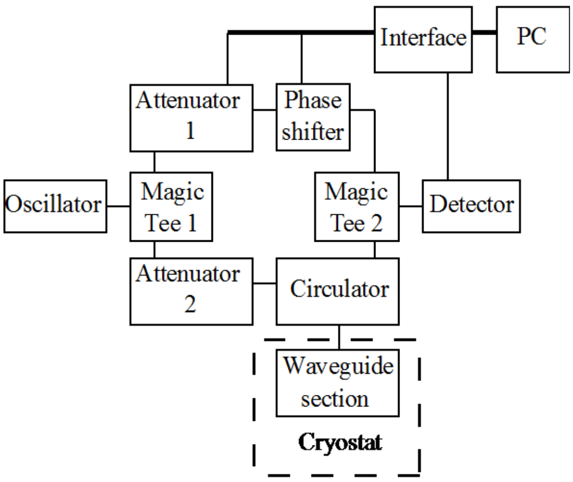

Fig. 2. Schematic diagram of the experimental setup for measurement of complex reflection coefficient

A magic-Tee 1 splits the signal of microwave oscillator in two branches. The first branch, which is a reference, consists of attenuator 1 and phase shifter. The latter devices have been improved to achieve digital control and to obtain data by PC. The second branch, which is a measuring one, includes attenuator 2 and circulator. Attenuator 2 is intended for isolation and a circulator allows decoupling incident and reflected waves in the waveguide section. Sample under test with a waveguide section is placed in the cryostat which allows to perform measurements in a wide temperature range, namely from liquid nitrogen temperature up to room one. Microwave signal from both branches as a result of combining by magic-Tee 2 is converted in DC voltage by detector. Thereafter the signal is amplified and converted to digital by the designed interface and passes into PC for further processing. The temperature at the sample surface was determined by a sensor placed in the copper plate (fig. 1) which is on a top of the sample. The changes of the phase shift and losses were recorded by PC simultaneously with the temperature changes using interface.

The special code was written to control the measurement process. It allows one to detect temperature changes of the sample, change positions of both attenuator1 and a phase shifter in a reference branch of the phase bridge in order to achieve compensation of the bridge output signal throughout minimum of the detected signal. At the same time the code allows determining positions of the attenuator and phase shifter in a reference branch and represents temperature dependencies in real time. The minimum detectable phase shift and attenuation in measuring system were 0.1 degree and 0.03 dB, respectively.

2. Calibration. To obtain absolute values of the complex reflection coefficient the calibration of the setup is needed. It was performed by measuring of samples with known characteristics. The relation of the measured R_m and actual R_a reflection coefficients is determined by [9]:

$$R_m = E_D + \frac{R_a E_R}{1 - R_a E_S},$$

where E_R, E_S and E_D are coefficients specified by imperfections such as losses and reflection in the microwave waveguide transmission line. E_R is the reflection tracking which is connected to the losses and phase shift in the transmission line and E_S is referred to as the source match. The error coefficient E_D is the directivity, which arises from the imperfect nature of the circulator and reflections due to waveguide connections. Three calibration measurement cycles were performed to obtain temperature dependence of the three calibration coefficients by solving system of equations for each temperature point. Bulk copper, titanium and absorber were used as calibration samples. The samples were chosen with different enough but known values of conductivity. Absorber reflection coefficient is close to zero in a whole temperature region, which was proven by low standing wave ratio (SWR) of the waveguide section with such sample (less than 1.1). This means that the absorber measured reflection coefficient is equal to calibration coefficient E_D. The actual reflection coefficient could be obtained by equation:

$$R_a = \frac{R_m - E_D}{E_R + E_S\left(R_m - E_D\right)}.$$

Temperature dependence of microwave losses $L = -20\log R_m$ in measurement branch of the phase bridge for three calibration samples (▲ – absorber, ■ – titanium, ● – copper) and superconducting $YBa_2Cu_3O_{7-\delta}$ film (♦) at frequency 39.6 GHz is shown in fig. 3, a. Changes of the losses at *S*-*N* transition are equal to about 1.5 dB, which is well measurable value (in comparison with normal incidence case [20]).

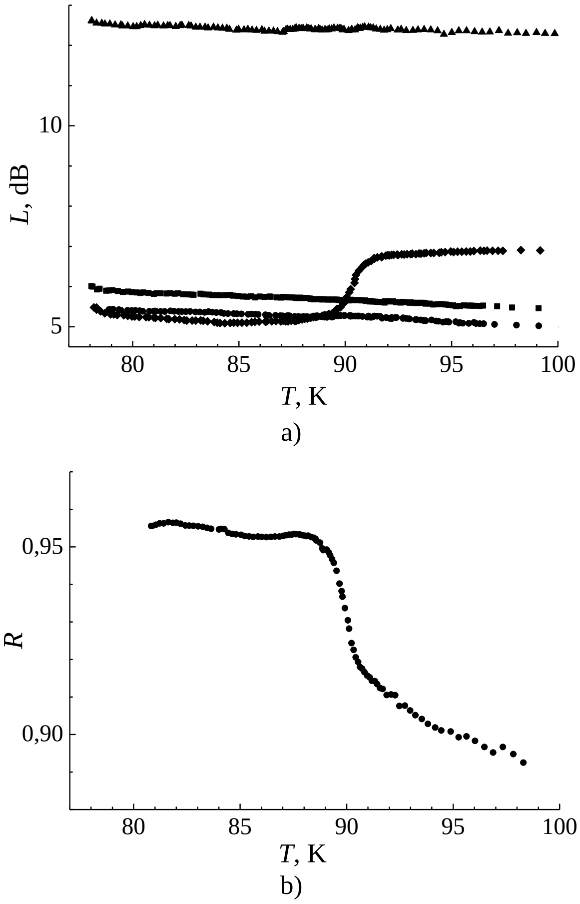

Fig. 3. Losses of $YBa_2Cu_3O_{7-\delta}$ superconducting film and three calibration samples dependence on temperature at frequency 39.6 GHz (a) and temperature dependence of the film reflection coefficient (b)

Knowing the loss of the copper, absorber and titan samples depending on temperature, it is possible to obtain absolute reflection coefficient of the superconducting film – dielectric substrate structure depending on temperature (fig. 3, b) by solving the abovementioned equations.

3. Results and discussion. Complex conductivity (or complex surface impedance) could be obtained by plane wave approach. Full reflection coefficient *r* from multilayer structure could be calculated by using Fresnel equations, Snell's law and dielectric function by [21, 22]:

$$r = \rho_{02} + \frac{\tau_{02} r_{20} \tau_{20} e^{2\psi i}}{1 - r_{20}\rho_{20} e^{2\psi i}},$$

where τ_{02} is the total transmission coefficient through the first (i. e. superconducting) layer taking all the reflections into account; t_{02} and r_{02} are the Fresnel transmission and reflection coefficients for the second interface; ρ_{02} reflection coefficient from the first layer which takes into account the interference which occurs within the film due to the reflection at the last interface; τ_{20} and ρ_{02} are introduced analogously to τ_{02} and ρ_{20}; $\psi = kd_2p$, $k = 2\pi/\lambda$, where λ is the wavelength, d_2 is the substrate thickness and $p = \sqrt{\varepsilon_S}$ are the refractive index of the substrate. Reflection coefficient $R = |r|^2$ could be recalculated from film permittivity ε_f knowing film thickness, substrate thickness and permittivity (which was taken from [24]), and also angle and frequency of incident wave.

Eigen waves in the waveguide including a basic H_{10}-wave are not plane ones but they can be represented as a superposition of the plane waves, so our approximation is hold true at a certain orientation of microwave electric field with regard to plane of the sample under test, i. e. microwave electric field $\vec{E}$ must lie in incident plane of guide wave (see fig. 1). The plane wave approach is more useful than electromagnetic analysis in the waveguide due to its simplicity. The exact electromagnetic analysis is complex at angles, higher than 78 degrees due to a large length of the measurement sample at a waveguide shear comparable to wavelength. Besides the data obtained by plane wave approach and using exact electromagnetic analysis are agreed for thin superconducting films and bulk samples in both normal and superconducting states up to 78 degrees of incidence angles.

To obtain complex conductivity from measured sample reflection coefficient it is necessary to find relation of complex conductivity and reflection coefficient. It is impossible to solve this problem analytically therefore iteration procedure was used.

To check reliability of the measurement approach and calibration procedure the investigation of conductivity of silicon, YBaCuO ceramics at room temperature (which was used only as a test material and does not has as good quality as superconducting film, mentioned above, but is thick) and duralumin samples was performed by two methods. The first one is a given method and the second, i. e. reference, one is a method based on whispering gallery mode sapphire disk resonator with the sample as conducting endplate [25]. The data obtained by both methods conform to each other well within the measurement errors (fig. 4).

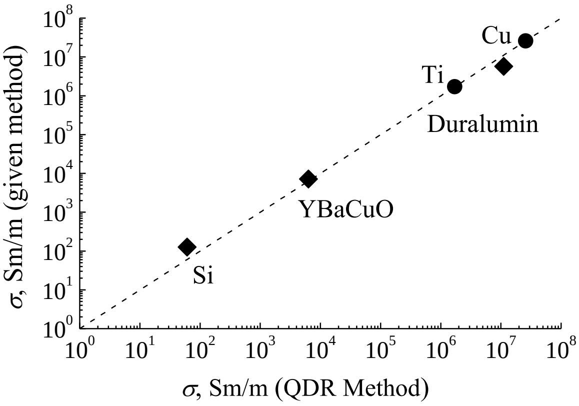

Fig. 4. Conductivity of the bulk test samples (silicon, ceramics, duralumin) and calibration samples (titanium and copper) obtained by given method and method based on whispering gallery mode resonator. All of the samples were at room temperature

The measured dependence of the real part of $YBa_2Cu_3O_{7-\delta}$ film complex conductivity is shown in fig. 5. The calculated dependence of the conductivity on temperature is shown by solid line.

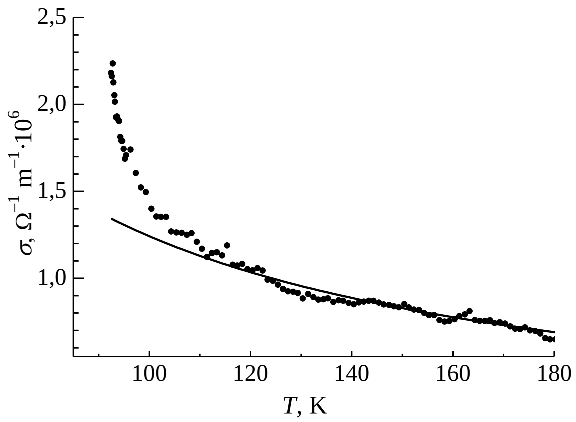

Fig. 5. Experimental temperature dependence of $YBa_2Cu_3O_{7-\delta}$ superconducting film conductivity (points) and calculated results on the basis of linear dependence of the sample resistance in normal state (line)

Here linear dependence of the normal state film resistance on temperature and assumption that normal state conductivity at $T = T_c$ is equal to $1.35 \cdot 10^6$ Sm/m were taken into account. There is a good coincidence of the theoretical and experimental results in temperature region higher than 120 K. Experimental and theoretical conductivities at temperatures 92…120 K differ and experimental values are higher than theoretical ones. This distinction is obviously connected with fluctuation conductivity phenomenon of the superconducting YBCO film [16, 26].

Conclusion. Thus, the possibility to study superconductor impedance properties by measurements of reflection coefficient at grazing incidence angles in rectangular waveguide with a sample inclined at the large angle (more than 80°) in a plane of microwave electric field is shown. The method allows performing study of the superconductors and other materials, e. g. CMR substances [27], conductivity of which changes under different external conditions such as temperature, magnetic field, etc. The technique to obtain such HTS characteristics as complex conductivity or surface impedance at temperature above T_c using measured complex reflection coefficient, calibrated by absolute values of reflection coefficient of the known materials, has been shown. The applicability of the method to studying the microwave properties of $YBa_2Cu_3O_{7-\delta}$ superconducting film in normal state and near T_c was demonstrated. Although only the real part of conductivity is measured in the present work, the measurement of the imaginary part is possible as well. It is worthy to note also, that although a given work was performed in single frequency mode, the grazing incidence reflectivity technique is frequency broadband one in principle. For realization of such a broadband approach, the sweep oscillator or frequency synthesizer is necessary.

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МЕТОД ИССЛЕДОВАНИЯ ПРОВОДИМОСТИ ВЫСОКОТЕМПЕРАТУРНЫХ СВЕРХПРОВОДНИКОВ ПРИ ПОМОЩИ ИЗМЕРЕНИЯ КОЭФФИЦИЕНТА ОТРАЖЕНИЯ ВОЛНЫ ПРИ СКОЛЬЗЯЩИХ УГЛАХ ПАДЕНИЯ В МИЛЛИМЕТРОВОМ ДИАПАЗОНЕ

А. И. Губин, Н. Т. Черпак, А. А. Лавринович

Описан метод неразрушающего микроволнового исследования высокотемпературных сверхпроводников и родственных материалов посредством измерения их коэффициента отражения при скользящих углах падения. Измерения коэффициента отражения при скользящих углах падения электромагнитной волны могут быть использованы для получения абсолютных значений комплексной проводимости образца в широком температурном и частотном диапазонах. Разработана и реализована экспериментальная установка на основе фазового моста в миллиметровом диапазоне длин волн. Измерена проводимость $YBa_2Cu_3O_{7-\delta}$-пленок при температурах выше критической.

Ключевые слова: коэффициент отражения при скользящих углах падения, микроволновая проводимость, сверхпроводящие пленки.

МЕТОД ДОСЛІДЖЕННЯ ПРОВІДНОСТІ ВИСОКОТЕМПЕРАТУРНИХ НАДПРОВІДНИКІВ ЗА ДОПОМОГОЮ ВИМІРЮВАННЯ КОЕФІЦІЄНТА ВІДБИТТЯ ХВИЛІ ПРИ КОВЗНИХ КУТАХ ПАДІННЯ У МІЛІМЕТРОВОМУ ДІАПАЗОНІ

О. І. Губін, М. Т. Черпак, О. А. Лавринович

Описано метод неруйнівного мікрохвильового дослідження високотемпературних надпровідників та споріднених матеріалів за допомогою вимірювання їх коефіцієнта відбиття при ковзних кутах падіння. Вимірювання коефіцієнта відбиття при ковзних кутах падіння електромагнітної хвилі можуть застосовуватись для отримання абсолютних значень комплексної провідності зразка в широкому температурному та частотному діапазонах. Розроблено та реалізовано експериментальну установку на базі фазового мосту в міліметровому діапазоні довжин хвиль. Виміряно провідність $YBa_2Cu_3O_{7-\delta}$-плівок при температурах вищих за критичну.

Ключові слова: коефіцієнт відбиття при ковзних кутах падіння, мікрохвильова провідність, надпровідні плівки.

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