

Quick technology for fabrication of BiSrCaCuO mesas and its application for spectroscopy*

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Abstract— The proposed technology of wet etching provides obtaining of stand-alone $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_8$ mesa structures thicker than ones usually being studied, but still allows to get mesas below 1 μm . The time required for the fabrication is much shorter than the standard method of ion milling. The development of technology is aimed to increasing of the copper plating up to 30 microns thick over a thin gold layer to improve heat removal. The measurements of fabricated samples over a wide temperature range from 6 to 77 K were performed. Also experimental study of spectrometer with the oscillator based on the BSCCO mesa structure obtained on the basis of standard ion milling technology was performed. In particular the BSCCO generator can be used to develop compact THz spectrometer with high resolution.

Keywords — *BSCCO mesa, wet etching, phase-diffusion field, THz spectroscopy, molecular gas*

I. INTRODUCTION

At the present time the investigations of subTHz and THz radiation from $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_{8+x}$ (BSCCO) mesas attract a great interest. Therefore the experimental study aimed at the development of technology and the practical use of generators based on high-temperature superconductors mesa structure remain relevant. In addition, the use of generators for spectroscopic tasks requires additional investigation of the interaction of radiation with molecular gas.

II. TECHNOLOGY OF WET ETCHING

Technological studies aimed for the fabrication of a single mesa structure of BSCCO intended for the generation of a subTHz signal have been made. For these experiments we used a lead-doped $\text{Bi}_{1.85}\text{Pb}_{0.35}\text{Sr}_2\text{CaCu}_2\text{O}_{8+x}$ flakes obtained by splitting of a single-crystal BSCCO grown at the Institute of Solid State Physics of RAS by the zone melting method [1]. The flakes were stucked with conductive glue to the silicon substrates coated with gold.

Previous research [2] has allowed to work out the technology of wet etching. The photoresist (PR) mask in the form of circles with various diameters 100-500 μm was put on the surface of flakes by photolithography etching in hydrochloric acid (1:8 water solution). The etching time was about 50 s and yielded stand-alone circular mesas. The PR mask was removed by plasma chemical etching in an oxygen plasma of Oxford Plazmalab 80 Plus facility or dissolved in dimethylformamide without using an ultrasonic bath. The latter method is more preferable because it does not lead to the facility contamination and prevents the destruction of the structure. The maximum time of PR removing was ~ 30 min.

The height of the mesa was measured using a Talysurf 2000 white light interferometer and was roughly 6 μm . It is seen that the etching is anisotropic: the depth of undercut is 40 μm with an etching depth of 6 μm . The anisotropy of the etching rate allows to obtain mesas with almost vertical walls (Fig. 1).

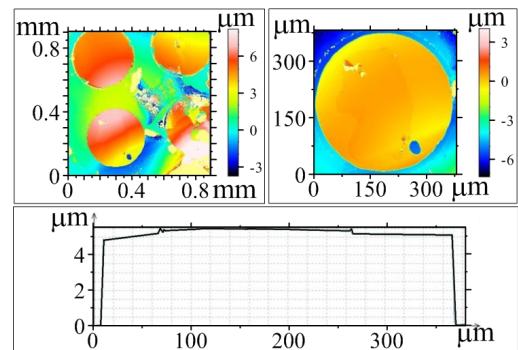


Fig. 1. Talysurf 2000 white light interferometer pictures of stand-alone circular mesas covered with gold on both sides and a single mesa profile with scanning line going through the middle of structure.

The work was supported by RSF (Project 16-19-10478).

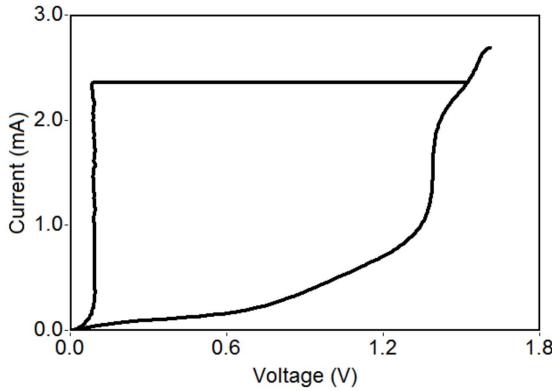


Fig. 2. Current-voltage characteristics for $T = 25$ K excluding resistive contribution.

A conductive glue under the structure was dissolved and a separate mesa was moved. After that the structure was glued by the conductive adhesive to the gold-plated substrate and a second electrical wire was bonded to the mesa surface. This configuration allows measuring the current-voltage characteristics along the c-axis of the BSCCO crystal. In Fig. 2 the characteristic of 16×10^{-4} cm 2 circular mesa is shown for $T = 20$ K. It is seen, that the measured J_c is much less than the expected one. This can be explained by deterioration of the top or bottom layer of the structure. On the other hand, perhaps for some reason only a part of the circular mesa exhibits superconducting properties.

Further development of the technology was aimed at the electrolytic build-up of copper coating over a thin layer of gold. This allows to obtain robust structure with good heat sink. Photo of the sample with a copper layer thickness of about 30 μm is shown in Fig. 3. On the right side of the structure internal splittings are seen, which increases the fragility of the structures. They can lead to damage of the structure during the deposition of a similar copper coating on the back side.

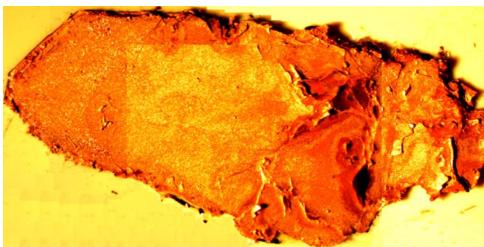


Fig. 3. Sample of BSCCO flake coated with gold and an electrolytically deposited layer of 30 μm copper on a silicon substrate.

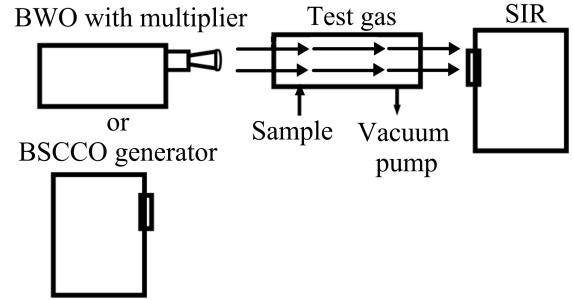


Fig. 4. Block diagram of the experimental setup for gas spectroscopy.

III. SPECTROSCOPIC APPLICATION

The applicability of the BSCCO generator for spectroscopic tasks was investigated in Kotel'nikov Institute of Radio Engineering and Electronics of RAS. The main issue was the influence of the BSCCO signal on the molecular spectral line. It is known [3] that at the resonant frequency the coherent signal induces macroscopic polarization in the molecular gas. The concentration of the investigated components of the gas mixture is determined by the magnitude of this response with a high accuracy [4].

An alternative way is to use a radiation source of phase-diffusion field with the Lorentz profile. It has been predicted [5] and experimentally observed [6] that, such noisy signal induces the macroscopic polarization of comparable magnitude to the polarization of a coherent signal case. In this work, the BSCCO generator having the required characteristics and Lorentz spectrum [7] was used as a source of noisy signal.

The present study was aimed to compare the effect of a coherent signal and a phase-diffusion field on a molecular gas.

The experiment setup is shown in Fig. 4. As a source of coherent signal the backward-wave oscillator (BWO) was used [8]. BWO signal in the range of 120 – 160 GHz was multiplied by the superlattices and reached 450-750 GHz at the 3rd, 4th and 5th harmonics of the BWO signal. To obtain a narrow linewidth of 20 kHz a phase-lock loop system was used.

As a source of phase-diffusion field an oscillator based on the stacks of Bi₂Sr₂CaCu₂O₈ intrinsic Josephson junctions has been used [9]. BSCCO mesastructure has been fabricated from a slightly under-doped BSCCO single crystal, that has been grown using a floating zone technique at National Institute for Materials Science [10]. The working frequency range of BSCCO mesa is between 400-600 GHz, the linewidth has reached 200 MHz.

The noisy signal from BSCCO mesa was measured by the superconducting integrated receiver (SIR) with local oscillator phase locked at 566 GHz and intermediate frequency range 4 - 8 GHz [11-12].

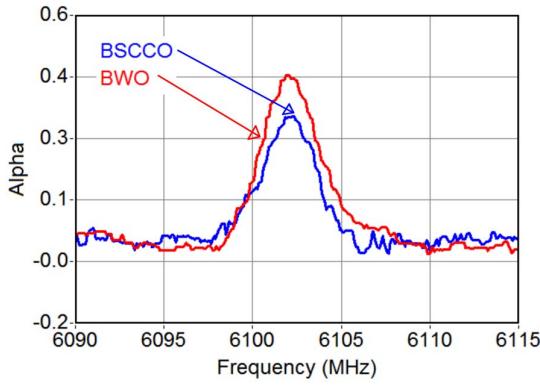


Fig. 5. The ammonia absorption line at a pressure of 0.08 mbar and frequency of 572.5 GHz (intermediate frequency).

The ammonia mixed with water (10% solution) has been studied as a molecular gas. As the results of measurements the absorption line at 572.5 GHz was observed. The ammonia absorption coefficient was calculated as the ratio of the absorbed power to the incident power:

$$\alpha = \Delta P / P_0 \quad (1)$$

The absorption coefficient was obtained for both cases and the values obtained with two different sources were compared, Fig. 5, Fig. 6. The observed deviations of absorption coefficients for the case of coherent signal and phase-diffusion field might be due to the following possible reason: according to the theory, a noise signal in contrast to the coherent signal provides general heating of the system. This reduces the difference in population levels, and, consequently, decreases the absorption coefficient.

IV. CONCLUSIONS

We have reported about current progress in fabrication of thick BSCCO mesa structures for spectroscopic applications. The proposed technology of wet etching allows obtaining stand-alone BSCCO mesa structures with thickness much above 1 μm . The time required for the fabrication is much smaller in comparison with the standard method of ion milling, but the process is still controllable. The stand-alone mesas are better cooled due to the absence of the superconducting substrate with low thermal conductivity. The copper coating over a thin layer of gold allows to obtain robust structure with good heat sink.

We have also proven the possibility of novel noisy THz spectroscopy using BSCCO samples, fabricated in NIMS, Tsukuba, and Superconducting Integrated Receiver, fabricated in IREE, Moscow, by detection of the molecular spectra in the subTHz range. It is shown that the interaction of noisy phase-diffusion signal with gas leads to macroscopic polarization, comparable to the polarization induced by a coherent signal. The performed investigations demonstrate the possibility of

practical application of BSCCO generator in the high-resolution THz spectroscopy.

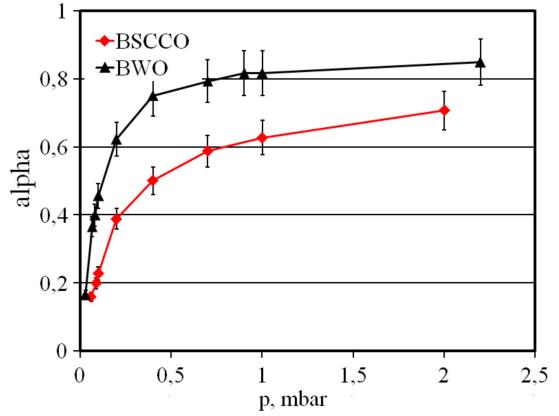


Fig. 6. Absorption α of ammonia at different pressures (BSCCO and BWO).

REFERENCES

- [1] A.B. Kulakov, I.K. Bdikin, S.A. Zver'kov, G.A. Emel'chenko, G. Yang and J.S. Abell, "Phase separation in $(\text{Bi},\text{Pb})_{2.2}\text{Sr}_2\text{CaCu}_2\text{O}_{8+\delta}$ single crystals at an annealing at high oxygen pressure", *Physica C* 371 45, 2002.
- [2] E.A. Vopilkin, A.V. Chiginev, L.S. Revin, A.N. Tropanova, I.Yu. Shuleshova, A.I. Okhapkin, A.D. Shovkun, A.B. Kulakov, A.L. Pankratov, "Quick and reliable technology for fabrication of stand-alone BSCCO mesas", *Supercond. Sci. Technol.* 28, 045006, 2015.
- [3] W.H. Flygare, "Molecular structure and dynamics", School of Chemical Sciences University of Illinois, Urbana [Prentice-Hall, Inc., Englewood Cliffs New Jersey, 1978].
- [4] V.L. Vaks, A.B. Brailovsky, V.V. Khodos, "Millimeter Range Spectrometer with Phase Switching — Novel Method for Reaching of the Top Sensitivity", *Infrared & Millimeter Waves*, 20, P. 883, 1999.
- [5] E.A. Sobakinskaya, A.L. Pankratov, V.L. Vaks, "Dynamics of a quantum two-level system under the action of phase-diffusion field", *Physics Letters A*, v. 376, p. 265, 2012.
- [6] E. Sobakinskaya, V.L. Vaks, N. Kinev, M. Ji, M.Y. Li, H.B. Wang, and V.P. Koshelets, "High-resolution terahertz spectroscopy with a noise radiation source based on high-Tc superconductors", *J. Phys. D: Appl. Phys.*, 50, 035305, 2017.
- [7] S. Guenon, M. Grunzweig, B. Gross, J. Yuan, Z.G. Jiang, Y.Y. Zhong, et al., "Interaction of hot spots and terahertz waves in $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_8$ intrinsic Josephson junction stacks of various geometry", *Phys. Rev. B*, 82, P. 214506, 2010.
- [8] V.L. Vaks, V.V. Khodos, E.V. Spivak, "A nonstationary microwave spectrometer", *Review of Scientific Instruments*, 70(8), p. 3447, 1999.
- [9] L. Mengyue, J. Yuan, N. Kinev, J. Li, B. Gross, S. Guénon, "Linewidth dependence of coherent terahertz emission from $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_8$ intrinsic Josephson junction stacks in the hot-spot regime", *Phys. Rev. B*, 86, P. 060505, 2012.
- [10] H.B. Wang, S. Guénon, J. Yuan, A. Iishi, S. Arisawa, T. Hatano, et al., "Hot spots and waves in $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_8$ intrinsic Josephson junction stacks: a study by low temperature scanning laser microscopy", *Phys. Rev. Lett.* 102, 017006, 2009.
- [11] V.P. Koshelets and S. Shitov, "Integrated superconducting receivers", *Supercond. Sci. Technol.* 13, p. R53, 2000.
- [12] A.L. Pankratov, V.L. Vaks, V.P. Koshelets, "Spectral properties of phase-locked flux flow oscillator", *J. Appl. Phys.* 102, P. 063912, 2007.