Chapter 11
Fabrication of Bi2212 Single Crystal Bolometer for Detection of Terahertz Waves

T. Semerci, Y. Demirhan, N. Miyakawa, H.B. Wang, and L. Ozyuzer

Abstract Terahertz (THz) radiation is in powerful region of electromagnetic spectrum because of prosperous application areas yet deficiency still exists about sources and detectors in despite of improvements of the research field in this range. This gap can be filled by focusing on development of THz detectors. Therefore, bolometers were preferred through many detectors due to detection sensitivity above 1 THz. In this study, $Bi_2Sr_2CaCu_2O_{8+\delta}$ (Bi2212) single crystals were used to fabricate THz bolometric detector. Bi2212 single crystals were transferred on sapphire substrate by cleavage process and e-beam lithography and ion beam etching were used to fabricate the microchip clean room facilities. Custom-designed cryogenic cryostat was used for a-b axis electrical and THz response measurements with liquid nitrogen cooled system. After electrical measurements, Bi2212 microchips detected the signals using Stefan-Boltzmann Lamp and response time were calculated. This study have shown with our experimental results that Bi2212 single crystals are potential candidates for THz bolometric detectors.

Keywords Terahertz Sensitive • Detector • Bolometer • Microchip • Bi2212 single crystal • Response Time • High-Tc Superconductor

---

T. Semerci (✉)
Department of Materials Science and Engineering, Izmir Institute of Technology, Urla, Izmir 35430, Turkey
e-mail: tugcesemerci@iyte.edu.tr

Y. Demirhan • L. Ozyuzer
Department of Physics, Izmir Institute of Technology, Urla, Izmir 35430, Turkey

N. Miyakawa
Department of Applied Physics, Tokyo University of Science, Tokyo, Japan

H.B. Wang
National Institute for Materials Science, Tsukuba 3050047, Japan

M.F. Pereira, O. Shulika (eds.), *THz for CBRN and Explosives Detection and Diagnosis*, NATO Science for Peace and Security Series B: Physics and Biophysics,
DOI 10.1007/978-94-024-1093-8_11

11.1 Introduction

By virtue of the fact that THz radiation (0.1–10 THz) has a wide range between microwaves and infrared regions in electromagnetic spectrum, noticeable applications of THz region exist, fundamentally in imaging and spectroscopy research fields [1, 2] Furthermore, THz technology is an effective area to examine the explosive materials in view of that many materials have a special peak at THz range as fingerprints [3]. According to this reason, detectors are candidates to be an important part of THz research fields via improving their sensitivities, but deficiency still exists about sources and detectors in despite of improvements of the research field in this range. This gap can be filled by focusing on the development of THz sources and detectors [4–7]. Many application areas such as imaging, spectroscopy technology will take advantage of filling this gap and developing THz sources [1]. Moreover, detectors have been especially developed to utilize THz radiation range. Recently, many detectors have been produced and developed which are based on different principles and used for distinct application areas but the common important point between these developed detectors is the increasing of the detectivity, sensitivity and decreasing of Noise Equivalent Power (NEP) which are the parameters to determine the performance of the detector.

When comparing the detectors with respect to sensitivities, cryogenic detectors such as bolometers provide more efficiency than room temperatures and also development of NEP for bolometers have been enhanced as diminished NEP by a factor 10^{11} in 70 years. Bolometer is a thermal detector of heat or power, based on measurement of the change of resistance which is a response to incoming THz radiation. Similarly, Hot Electron Bolometer (HEB) consist of two metal layers which are connected to each other with a microbridge. There is a small change of resistance occurred comprised from the incoming THz radiation through microbridge at the near transition region in HEB. This change of resistance is caused from hot electrons on the microbridge and determines the amount of incoming radiation onto the bolometer. When incoming THz radiation power P is detected by the bolometer, temperature (T_B) is increased by the ratio of $\frac{dT_B}{dt} = \frac{p}{c_{th}}$ and incoming radiation is ceased as it backs to normal state by $\tau = \frac{c_{th}(\text{heat capacity})}{c_{th}(\text{thermal conductance})}$ time constant (response time) (τ). Time constant is the remarkable parameter to determine the detectivity and sensitivity of detector which should be small (fast response time) with small heat capacity and high thermal conductance values.

Another notable parameter for getting fast response time from the bolometer is to specify the suitable material to get higher absorbance from incoming radiation with higher detectivity and sensitivity. Material of this chip is determined by the parameter, Temperature Coefficient of Resistance (TCR) $= \frac{1}{R}\frac{dR}{dT}$ which changes with respect to kind of materials. Necessary parameters are to obtaining best result for detectivity and sensitivity for detector with the large TCR and slope of the electrical measurement with small bolometer resistance.

In HEB, there is an antenna structure in which hot electrons escape from superconductor through normal metals by diffusion. Antenna structures have been successfully applied to supply the electromagnetic waves on focusing them onto microbridge by comprehend THz radiations [8, 9]. Length of microbridge is an important parameter for detection in this mechanism and should be small as much as possible to get higher sensitivity for the bolometer depending on the formula, $L_{\max} = 2(D_e \tau_{ee})^{1/2}$, where τ_{ee} is the electron-electron interaction time and D_e is electron diffusion [10].

Researchers have gone towards the THz detectors, which contain superconducting materials and it has been known for 70 years but recently started to be used in technological application areas. High Temperature Superconductors (HTSs) are suitable candidates for the generation of THz radiation because of their layered structure which enables the propagation of electromagnetic wave and have higher energy gap than low temperature superconductors. Therefore, HTSs give great response at the frequency of the THz range. Bi2212 is one of the examples for high energy gap HTSs. Energy gap is wide, from 15 to 40 meV for Bi2212 which refer to wide THz gap [4, 11]. Therefore, Bi2212 is suitable for THz researches due to possession of broad energy gap. Bi2212 crystal has a critical temperature 95 K and layered structure property makes it easy to cleave the crystal during the fabrication of chip. Doping level and critical temperature of Bi2212 superconductor can be changed by adding or removing oxygen with number of Cu-O layer and affect a-b axis resistance-temperature measurement.

There are many detectors fabricated and developed from superconducting materials, especially mostly used superconductors are NbN and Nb based recently because of smaller time constants. Nb-based superconducting tunnel junction using as a THz radiation detector was performed with the range of 1–2 THz with response about 15–20 μs [12]. Later on, superconducting bolometer for THz radiation based on NbN film with log-periodic in antenna was achieved a time constant with approximately 150 ps in the range of 1–5 THz [13]. Additionally, superconducting hot-spot air bridge bolometer based on 50 nm thick Nb film with 15 μm long bridge have been demonstrated and measured by response time 1.2 μs and imaging was performed with this air bridge bolometer [14, 15]. In the year 2013, 4 mm thick NbN film based on planer logarithmic spiral antenna HEB was fabricated in the range of 0.3–3 THz and measured response time as 50 ps [16]. Terahertz detector based on $YBa_2Cu_3O_7$ superconductor microbolometer was demonstrated by Hammer et al. such that responsivity is related to size of antenna structure (1.5 μm × 1.5 μm area structure has nearly 180 V/W and 4 μm × 1 μm area structure has 100 V/W as responsivity) [17].

In this study, bolometric detector was preferred through different kinds of detectors due to detection above 1 THz frequency. As far as we know, fabricated microchip from high temperature superconducting Bi2212 single crystal for THz detector is a first study in the literature. Electrical (a-b axis) and bolometric measurements were done and time constant was calculated for THz bolometer based on Bi2212 single crystal with in this study.

11.2 Fabrication and Experimental Setup

Roughly 700 μm × 400 μm Bi2212 single crystal was glued onto 6 mm^2 sapphire substrate by fast dry epoxy in this study. The crucial part for epoxy is that it should be suitable for variation in temperature during measurements. After the crystal was glued onto substrate, it was cleaved by scotch tape to get the fresh and smooth surface. Before the cleaving process, thickness of the Bi2212 crystal is almost 700–800 nm and this thickness could be minimized until 100–200 nm after cleaving, intended thickness could be obtained by this process. Thickness of crystal is one of the significant parameters to determine the superconductivity property of the crystals [18]. Edge of transferred Bi2212 crystals were cut as pyramidal shapes and masked with aluminum foil to provide the bridge for antenna-like structure. 150–200 nm Au was thermally evaporated on the cleaved, fresh crystal surface immediately to prevent undesirable chemical reactions and any contaminations on the surface [19]. After deposition with Au layer, antenna-like structure were patterned onto crystal surface by conventional e-beam lithography and fabricated using clean room facilities and argon ion beam etching techniques. Finally, gold wires were connected on four paths in order not to cause any contact resistance. The SEM image of final outlook microchip can be seen in Fig. 11.1.

The grey area (600 μm × 1200 μm) indicates the Bi2212 crystal and sphere darker areas represent the silver epoxy in Fig. 11.1. The bright area is the antenna-like structure of microchip which has 100 μm bridge distance and 1200 μm total distance of antenna-like structure. Additionally, the design has 300 μm^2 contact paths and 450 μm distance in between them.

Bi2212 microchips were characterized in our custom-designed cryostat for electrical and bolometric measurements. This cryostat has two main chambers: First, inner part is used for cooling the microchip with liquid nitrogen (77 K) and it has 153 mm diameter, 150 mm height and 3 liters capacity. Second, microchip is vacuumed by outer chamber which has 250 mm height and 200 diameter and provides to keep in low temperatures during characterization until 30 h. Electrical measurement setup to determine the superconducting transition region and operating temperature (middle point of the operating region) of the fabricated microchip can be seen in Fig. 11.2.

According to operating temperature of the microchip, bolometric measurement was done to detect the THz radiation. Stefan Boltzmann Lamp was placed in electrical measurement setup with a distance of 5 cm from the cryostat, to determine the response of the detector. For this detection, microchip was set to the operating temperature by heater in cryostat and pulsed radiation was sent at certain times from Stefan Boltzmann Lamp to the microchip by passing through Polyethylene (PE) window which is transparent in the THz region and focusing on the wave collector. Hitting radiation on the antenna-like structure cause to increase the resistance of microchip which is observed via Labview program. This distinction was induced by increasing the temperature of the microchip during focusing of the

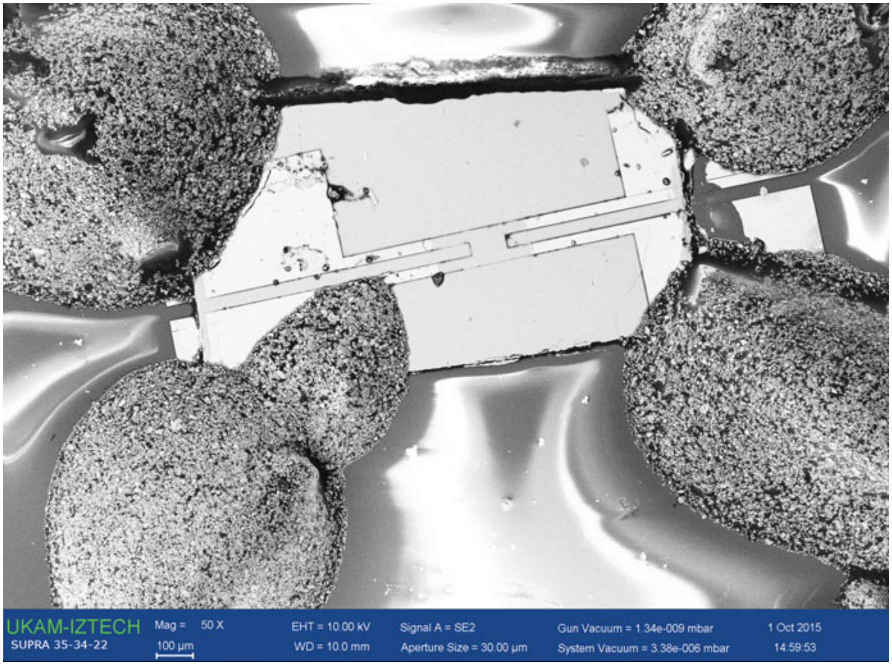

Fig. 11.1 SEM image of antenna-like patterned microchip based on Bi2212 crystal

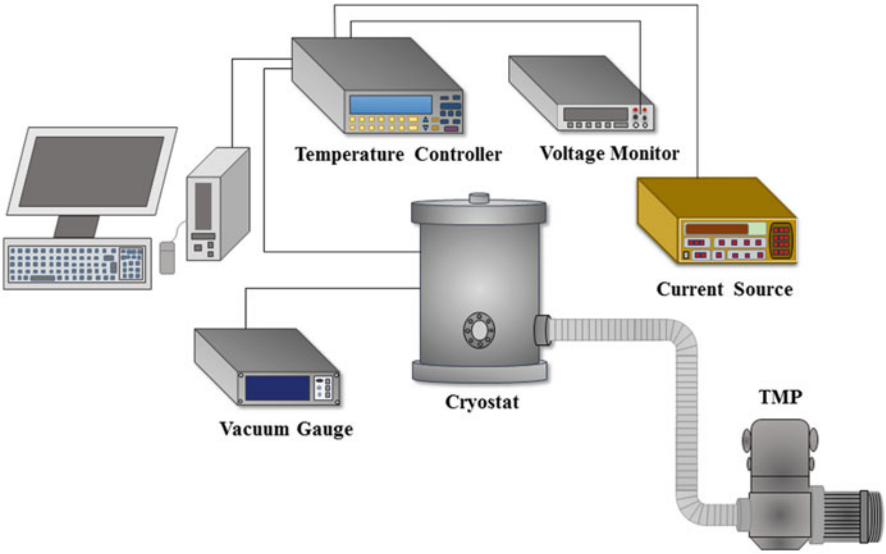

Fig. 11.2 Scheme of the electrical measurement setup

radiation. After measuring the variation of the resistance, time constant was calculated from bolometric measurement results.

11.3 Results and Discussion

Electrical characterization setup was shown in Fig. 11.2 the a-b axis of the microchips by applying constant 10 μA current is measured. It is aimed to observe sharp superconducting transition region because of the fact that this bolometric detector principle is based on the middle point of the superconducting transition region to get the highest temperature factor of the resistance. Electriccal measurements of all fabricated microchips have around the critical temperature as 90 K. Temperature dependence a-b axis resistance also establishes the onset and zero resistance of the microchips. These critical parameters depend on both oxygen level and thickness of the Bi2212 single crystals [20, 21]. The thickness of our crystals were changed between 100 and 500 nm because of the uncontrolled cleavege process. It is uneasy to arrange the thicknesses of the crystals during this process. Watanebe et al. also have shown that there is no contact resistance problem if zero resistance is below 3Ω [20]. All our microchips have been shown no contact resistance during both electrical and bolometric measurements.

Figure 11.3 shows a-b axis temperature resistance result of one of the fabricated microchip which has critical temperature (T_c) 89 K, room temperature resistance (R_{300K}) 0.54 Ω, critical temperature resistance (R_{Tc}) 0.26 Ω and variation of temperature in transition region (ΔT_c) 7.20 K. After obtaining these electrical measurement parameters for each fabricated microchip, operating temperature was selected as a middle point of the superconducting transition region and microchip was kept in that temperature via heater during bolometric measurement which was applied by switching on-off a Stefan Boltzmann Lamp at certain times. After that, response time was calculated for each microchip by indicating the initial resistance point as reference point (almost zero). Response time is the elapse time 1/e of the moment of switch off the signal.

Bolometric measurement of one of the fabricated microchip can be seen from Fig. 11.4, with an operating temperature 84 K which refer to 0.2503 Ω. Stefan Boltzmann Lamp was switched on-off at each 40 s intervals and 1.8 mΩ resistance change was observed and calculated response time value is 1.98 s for this microchip. Consequently, response time of our fabricated microchip was higher than expected values because of the fact that we had the limitation speed of the data transfer during bolometric measurement setup, this delayed data transfer may be solved by using lock-in-amplifier there by getting more exact time constants.

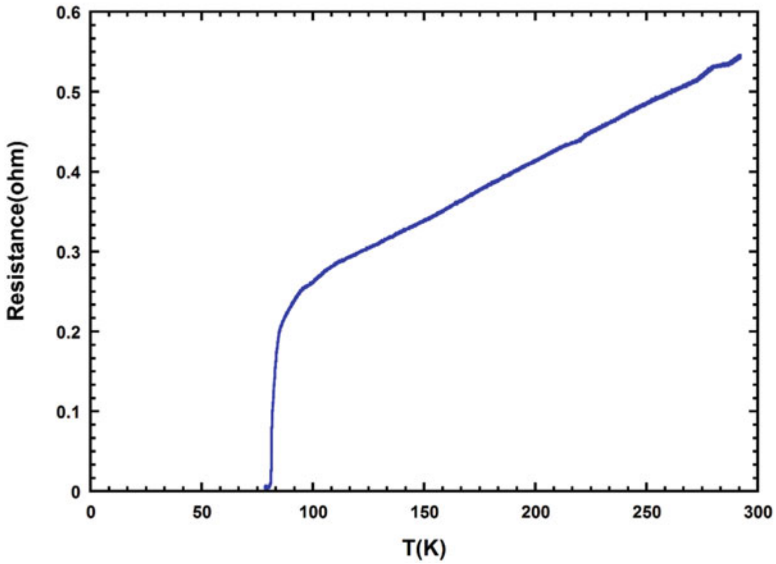

Fig. 11.3 Temperature dependence a-b axis resistance behavior of one of the fabricated Bi2212 microchip

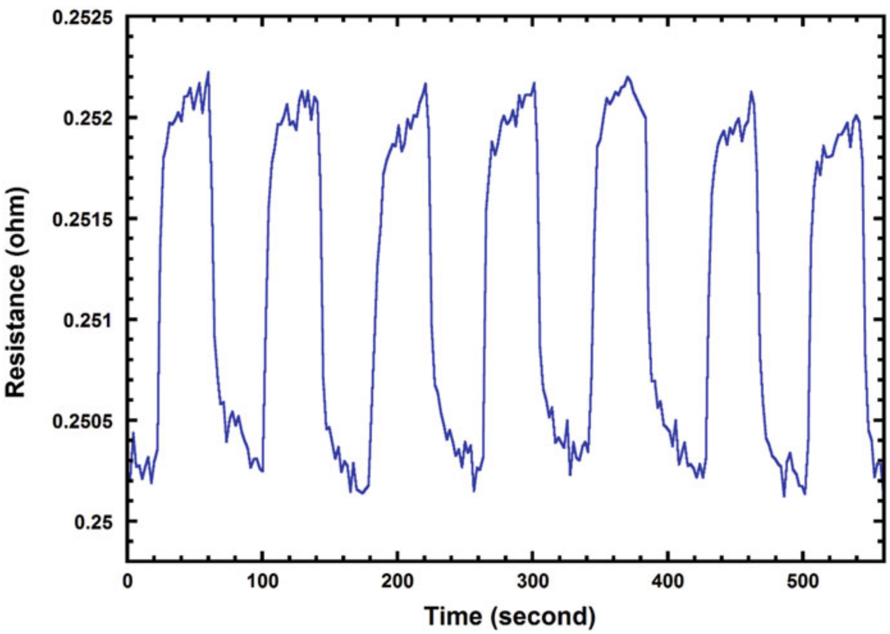

Fig. 11.4 Change of the microbridge resistance during bolometric measurement

11.4 Conclusion

In this study, we have shown that electrical and bolometric measurements of detectors which were fabricated on high quality Bi2212 single crystal in custom-designed and nitrogen cooled system. To the best of our knowledge, there is no HEB from Bi2212 single crystal in literature. Additionally, this study have shown that Bi2212 single crystals are productive candidates for bolometric detectors. Electrical measurement in a-b axis of our microchips has 90 K critical temperatures which depend on thicknesses and oxygen doping levels of the crystals. We have fabricated 100–500 nm Bi2212 thick crystals with oxygen doped range. After electrical measurements, THz radiation was detected by bolometer using Stefan Boltzmann Lamp and it has a response time 1.98 s which is higher than expected value. We concluded that this high value may be caused from speed limitation of our data transfer which might be solved by using lock-in-amplifier.

Acknowledgments This research was partially supported by Ministry of Science, Industry and Technology under project number SANTEZ 1386.STZ.2012-1 which was cooperated with ASELSAN. Additionally, we thank the Applied Quantum Research Center (UKAM) of IZTECH for the research facilities.

References

1. M. Tonouchi, Cutting-edge terahertz technology. Nat. Photon. 1(2), 97–105 (2007)
2. T. Kleine-Ostmann, T. Nagatsuma, A review on terahertz communications research. J. Infrared Millim. Tera. Wav. 32, 143–171 (2011)
3. M.C. Kemp, P.F. Taday, B.E. Cole, J.A. Cluff, A.J. Fitzgerald, W. R. Tribe, Security applications of terahertz technology, Pro. SPIE, 5070, 44–52 (2003)
4. L. Ozyuzer, A.E. Koshelev, C. Kurter, N. Gopalsami, Q. Li, M. Tachiki, U. Welp, Emission of coherent THz radiation from superconductors. Science, 318(5854), 1291–1293 (2007)
5. K. Kadowaki, M. Tsujimoto, K. Yamaki, T. Yamamoto, T. Kashiwagi, H. Minami, M. Tachiki, R.A. Klemm, Evidence for a Dual-Source Mechanism of Terahertz Radiation from Rectangular Mesas of Single Crystalline Bi2Sr2CaCu2O8+δ Intrinsic Josephson Junctions. J. Phys. Soc. Jpn. 79, 023703 (2010)
6. F. Turkoglu, L. Ozyuzer, H. Koseoglu, Y. Demirhan, S. Preu, S. Malzer, Y. Simsek, H.B. Wang, P. Muller, Emission of the THz waves from large area mesas of superconducting Bi2Sr2CaCu2O8+d by the injection of spin polarized current. Physica C Superconductivity 491, 7 (2013)
7. Y. Demirhan, H. Saglam, F. Turkoglu, H. Alaboz, L. Ozyuzer, N. Miyakawa, K. Kadowaki, Area dependence and influence of crystal inhomogeneity on superconducting properties of Bi2212 mesa structures. Vacuum 120, 89–94 (2015)
8. S. Cibella, P. Carelli, M.G. Castellano, V. Foglietti, R. Leoni, M. Ortolani, G. Torrioli, A Superconducting Bolometer Antenna-Coupled to Terahertz Waves. Journal of Low Temperature Physics 154(5–6), 142–149 (2009)
9. R.H. DuHamel, D.E. Isbell, Broadband Logarithmically Periodic Antenna Structures. IRE National Convention Record. 1, 119 (1957)
10. F. Sizov, A. Rogalski, THz Detectors-Review. Progress in Quantum Electronics. 34, 278–347 (2010)

11. L. Ozyuzer, Y. Simsek, H. Koseoglu, F. Turkoglu, C. Kurter, U. Welp, P. Muller, Terahertz wave emission from intrinsic Josephson junctions in high-T-c superconductors. Superconductor Science and Technology. 22(11) (2009)
12. C. Otani, T. Taino, R. Nakano, K. Hoshino, T. Shibuya, H. Myoren, S. Ariyoshi, H. Sato, H. M. Shimizu, S. Takada, K.Kawase. A broad-band THz radiation detector using a Nb-based superconducting tunnel junction. IEEE Trans. on Appl. Supercon. 15, 2 (2005)
13. A.D. Semenov, H. Richter, H.W. Hübers, K.S. LL'in, M. Siegel, Superconducting noise bolometer for terahertz radiation. Appl. Phys. Lett. 87, 173508 (2005)
14. S. Cibella, M. Ortolani, R. Leoni, G. Torrioli, L. Mahler, Ji-Hua Xu, A. Trediccui, H.E. Beere, D.A. Ritchie, Wide dynamic range terahertz detector pixel for active spectroscopic imaging with quantum cascade lasers. Appl. Phys. Lett. 95, 213501 (2009)
15. S. Cibella, M. Ortolani, R. Leoni, G. Torrioli, L. Mahler, Ji-Hua Xu, A. Trediccui, H.E. Beere, D.A. Ritchie, Optical characterization of superconducting hotspot air-bridge bolometer, 35th International Conference on Infrared, Milimeter and Terahertz Wave IEEE. 1–2, (2010)
16. N. Palka, P. Zagrajek, A. Czerwinski, T. Trzcinski, E. Rurka, M. Szustakowski, M. Sypek, Hot Electron Bolometer for detection of fast terahertz pulses from optical parameter oscillator. Milimetre Wave and Terahertz Sensors and Technology V Proc. of SPIE. 8544 (2013)
17. A. Hammar, S. Cherednichenko, S. Bevilacqua, V. Drakinskiy, J. Stake, Terahertz direct detection in YBa2Cu3O7 microbolometers. IEEE Transactions on Terahertz Science and Technology. 1(2), 390–394 (2011)
18. K. Yamaki, M. Tsujimoto, T. Yamamoto, A. Furukawa, T. Kashiwagi, H. Minami, K. Kadowaki, High-power terahertz electromagnetic wave emission from high-Tc superconducting Bi2Sr2CaCu2O8+d mesa structures. Optics Express. 19(4), 3193–3201 (2011)
19. I. Kakeya, K. Fukui, K. Kawamata, T. Yamamoto, K. Kadowaki, Quantum oscillation of the c-axis resistivity due to entrance of pancake vortices into micro-fabricated Bi2Sr2CaCu2O8+d intrinsic Josephson junctions. Physica C. 468, 669–673 (2008)
20. T. Watanabe, T. Fujii, A. Matsuda Anisotropic resistivities of precisely oxygen controlled single-crystal Bi2Sr2CaCu2O8+delta: Systematic study on "spin gap" effect. Physical Review Letters. 79(11), 2113–2116 (1997)
21. L.X. You, A. Yurgens, D. Winkler, C.T. Lin, B. Liang, Superconducting properties of ultrathin Bi2Sr2CaCu2O8+x single crystals. Journal of Applied Physics. 98, 033913 (2005)