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Magnetic Relaxation Measurements of $Bi_2Sr_2CaCu_2O_x$ Irradiated with 1 MeV Electrons using Microwave Absorption

by

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Abstract

An apparatus has been developed to detect the microwave absorption near the surface of materials within the range of 1 MeV electrons. Firstly, the temperature dependence of the dielectric constant in KH_2PO_4 and of the magnetic permeability in $BiSrCaCu_2O_x$ has been measured to evaluate the reliability and the limitation of the microwave absorption apparatus. Secondly, this apparatus has been used to determine the pinning potential of $Bi_2Sr_2CaCu_2O_x$ single crystals irradiated with and without 1 MeV electrons flux of 2.0×10^{20} e/m²s at room temperature through their magnetic relaxation. The magnetic relaxation measurements with use of microwave absorption method were performed at $T = 79$ K under magnetic fields, $H = 0$-0.13 T. The pinning potential of unirradiated and irradiated $Bi_2Sr_2CaCu_2O_x$ was determined to be 0.8-5.6 eV and 3.8-11 eV, respectively, depending on magnetic field. It is concluded that lattice defects induced by 1 MeV electron irradiation enhance the pinning potential of $Bi_2Sr_2CaCu_2O_x$, and that they provide effective pinning centers.

Keywords : High T_c superconductor, Microwave absorption, Pinning potential, Pinning center, Electron irradiation

1. Introduction

High-T_c superconductors, such as $Bi_2Sr_2CaCu_2O_x$, have been expected to be used for magnetic coils because of their no electric resistance in the superconducting state even at the liquid nitrogen temperature. The critical current density, J_c, of high-T_c superconductors is one of the critical factors for their application to the magnetic coils, and it generally decreases with increasing the applied magnetic field. Phenomenologically, magnetic fluxes in superconductors are held at pinning centers with pinning potential and creep through their

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release from the pinning centers, decreasing J_c. It is well known that lattice defects, such as point defects, impurities, dislocations and grain boundaries, act as pinning centers and that the defects themselves and their distributions strongly influence J_c. Several groups have investigated the influence of radiation induced defects on J_c[1-5]. Though they have reported that J_c is increased by defects induced by irradiation, they have not cleared what kind of defects are effective pinning centers.

Irradiation is a useful method for introducing electronic and crystal defects artificially. Especially, electron irradiation produces simple point defects, vacancies and interstitials, providing advantages for getting the fundamental information about the interaction between defects and magnetic fluxes. However, the defects induced by 1 MeV electron irradiation distribute within the range of electrons in materials; or the vicinity of the surface of irradiated samples.

The interaction between defects and magnetic fluxes is expressed in terms of the pinning potential. The pinning potential is generally obtained from conventional magnetic relaxation measurements, such as dc magnetization, ac susceptibility and electrical resistivity[6,7]. The conventional measurements can get the pinning potential for bulk samples. However, they are not sensitive to the surface properties of samples and provide inaccurate information about the pinning potential of electron irradiated samples.

The microwave absorption method, on the other hand, gives the information from only the surface of samples because microwave penetrates within a few μm from the surface of sample due to skin effect, and it is one of the best ways to get the pinning potential in the vicinity of the sample surface. Moreover, the microwave absorption method has an advantage of its fast response with time resolution of less than one second, giving more precise values of the pinning potential than those obtained from the conventional methods. In addition, powdered samples can be applicable to measure the pinning potential through the microwave absorption method. In spite of its advantages, however, the magnetic relaxation measurements by use of the microwave absorption method have not been performed because of its technical difficulties in getting the signal from samples.

The purposes of the present study are to develop a microwave absorption apparatus for measuring the magnetic relaxation in the vicinity of the surface of samples and to evaluate the reliability and the limitation of the apparatus. Further purpose is to investigate the effects of 1 MeV electron irradiation on the pinning potential of $Bi_2Sr_2CaCu_2O_x$ under various applied magnetic fields by using the apparatus.

2. Microwave absorption method

When a microwave in air impinges on a sample whose magnetic permeability and dielectric constants are μ and ε, respectively, it is absorbed and reflected at the interface between air and the sample. The absorbance of microwave power involves interactions between microwave and electrons and its power R is expressed as follows:

$$R = \frac{\mu''}{\mu_0} \int h^2 dv + \frac{\varepsilon''}{\varepsilon_0} \int e^2 dv, \qquad (1)$$

where μ_0 and ε_0 are the magnetic permeability and the dielectric constant in vacuum, respectively, and μ'' and ε'' are the imaginary parts of μ and ε, respectively. Furthermore, h and e are magnetic and electric field of microwave, respectively, and v is the volume of a sample. When μ'' and/or ε'' change, the microwave absorption changes in accordance with eq.(1), that is, the microwave absorption is proportional to μ'' and ε''. No microwave
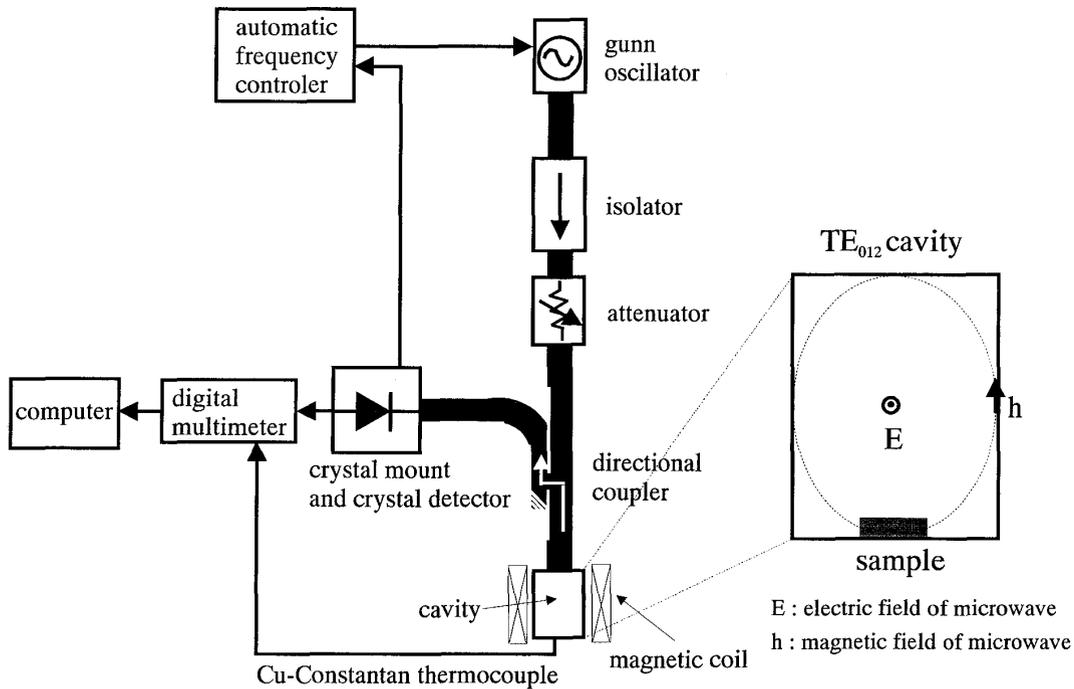
Fig. 1 A block diagram of microwave absorption apparatus developed in the present work. The microwave absorption apparatus consists of a gunn oscillator, an attenuator, a wave guide, a cavity, a directional coupler and a diode detector. A sample is located in the TE_{012} cavity where microwave magnetic field is the maximum and shown in the enlarged figure.

absorption occurs if $\mu'' = \varepsilon'' = 0$. Therefore, no microwave absorption occurs in superconductors, because the magnetic permeability and the dielectric constant of superconductors are zero in superconducting states. When a magnetic field is applied to a superconductor, on the other hand, some magnetic fluxes will be pinned by pinning centers where normal regions are induced. Therefore, the microwave absorption will be induced.

We have developed a microwave absorption apparatus whose block diagram is shown in **Fig. 1**. The heavy lines in **Fig. 1** show microwave circuits. The microwave absorption apparatus consists of a gunn oscillator, an isolator, an attenuator, a directional coupler, a cavity and a diode detector. The gunn oscillator generates 50 mW of microwave power. The frequency of microwave is locked by automatic frequency controller (AFC) around 8.9 GHz (X-band). The isolator is the device which isolates the gunn oscillator from the reflection microwave. The attenuator reduces the microwave power. The sample is placed in the cavity with the TE_{102} mode, where the magnetic field of microwave is the maximum. The TE_{102} mode cavity has higher Q values than any other mode cavities and detects the signal from the sample with high sensitivity. Since the size of the cavity at the X-band is larger than that at the K-band[6], a larger size of samples can be measured than that used at the K-band. This means that the microwave absorption apparatus at the X-band is powerful in case where it is difficult to detect the signal from small size of samples. The diode detector detects the microwave power reflected from the sample. A liquid N_2 bath is used to cool the sample from room temperature to 77 K. The temperature of samples is measured by a Cu-Constantan thermocouple attached to the outside of the cavity. The values of detected microwave power and temperature are recorded by using a personal computer through a digital multi-meter with a GP-IB (General Purpose Interface Bus) interface.

3. Experimental

In order to confirm the accuracy of microwave absorption apparatus, the Curie temperature (123 K) of potassium dihydrogen phosphate (KH$_2$PO$_4$), at which it changes from the low temperature ferroelectric state to high temperature paraelectric state, has been measured. A single crystal of KH$_2$PO$_4$, which was provided by Katayama Chemical Industries Co. LTD, was used as a sample for the measurements. The size of the sample was 3.9×3.7×1.8mm^3.

A poly crystal of BiSrCaCu$_2$O$_x$ was used as a sample to confirm the capability of measuring the change in magnetic permeability from the normal to the superconducting states by microwave absorption. The sample was prepared by mixing high purity powders of Bi$_2$O$_3$, SrCO$_3$, CaO and CuO. The mixtures were ground, pressed and fired at 1133 K for 220 hours in air and cooled to 723 K at the rate of 100 K/min, then quenched into air at room temperature. The size of the sample was 10.2×3.6×1.9mm^3. The critical temperature T_c was confirmed to be 110 K by electrical resistivity measurements.

Bi$_2$Sr$_2$CaCu$_2$O$_x$ single crystals of 7.5×5×1mm^3 were used as samples for magnetic relaxation measurements and its critical temperature T_c was confirmed to be 89 K. The sample was supplied by Japan Fine Ceramic Center. Irradiation with a 1 MeV electron flux of 2.0×10^{20} e/m^{2}s was performed at room temperature in a high voltage electron microscope (HVEM) in the Research Laboratory of HVEM, Kyushu University. Magnetic relaxation of Bi$_2$Sr$_2$CaCu$_2$O$_x$ was measured through microwave absorption at 79 K with an applied magnetic field range from zero to 0.13 T. The magnetic fields were applied parallel or perpendicular to the c-axis of the sample.

Magnetic relaxation measurements through microwave absorption were performed as follows: the sample was cooled below T_c and was kept at a temperature under an applied magnetic field. The microwave absorption was measured as a function of time after the applied magnetic field was taken away from the sample.

4. Results and discussion

4.1 Temperature dependence of microwave absorption in potassium dihydrogen phosphate

The temperature dependence of microwave absorption in potassium dihydrogen phosphate (KH$_2$PO$_4$) was measured to confirm the accuracy of the microwave absorption apparatus. The microwave absorption is expected to change as a function of temperature, reflecting the temperature dependence of its dielectric constant.

Figure 2 shows the temperature dependence of the microwave absorption in KH$_2$PO$_4$. The measurement was performed with increasing temperature after KH$_2$PO$_4$ was cooled to 77 K. The microwave absorption increases with increasing temperature, reaches the maximum value at 123 K and then decreases. The temperature dependence of the microwave absorption coincides with that obtained by M. Horioka and R. Abe[7].

4.2 Temperature dependence of microwave absorption in BiSrCaCu$_2$O$_x$

The temperature dependence of microwave absorption in BiSrCaCu$_2$O$_x$ was measured. The microwave absorption drastically changes at T_c and diminishes below T_c, reflecting the temperature dependence of magnetic permeability of BiSrCaCu$_2$O$_x$. **Figure 3** shows the temperature dependence of microwave absorption in BiSrCaCu$_2$O$_x$. The microwave absorp-
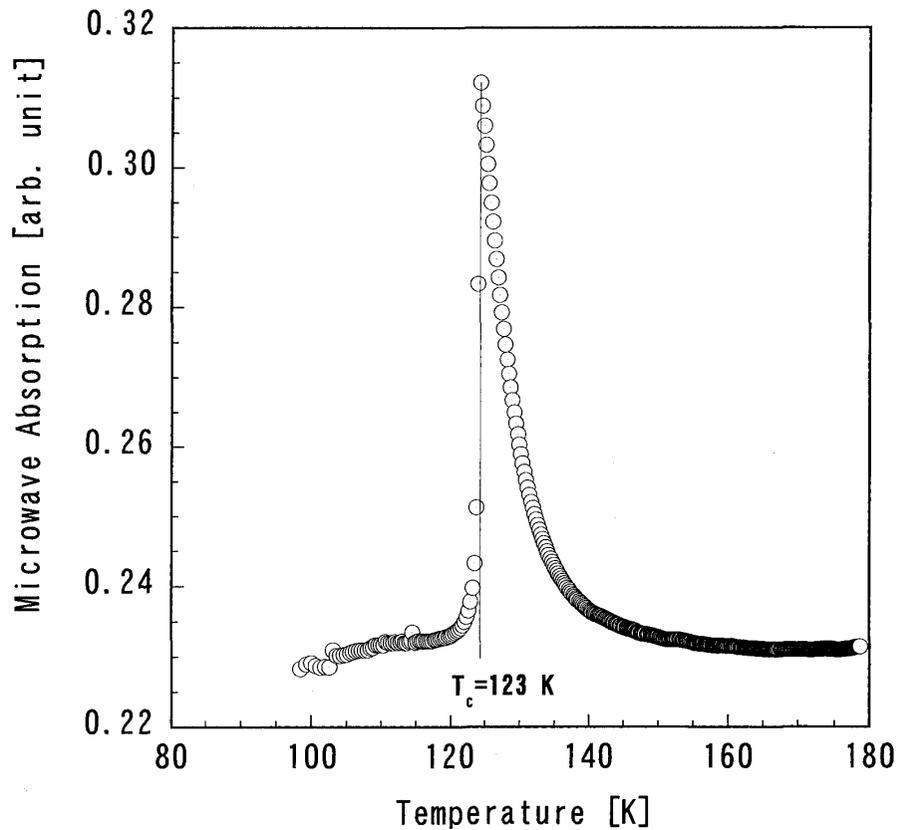
Fig. 2 The temperature dependence of microwave absorption in potassium dihydrogen phosphate(KH_2PO_4). The change of microwave absorption corresponds to that of dielectric constant in KH_2PO_4.
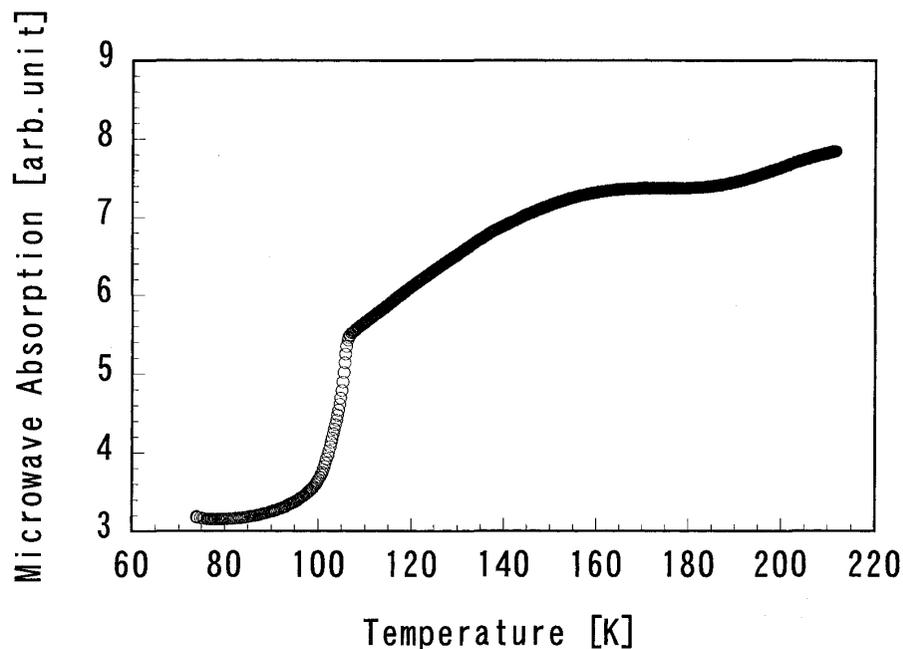
Fig. 3 The temperature dependence of microwave absorption in $BiSrCaCu_2O_x$. The change of microwave absorption corresponds to that of the magnetic permeability in $BiSrCaCu_2O_x$. The sharp bend in the line indicates T_c.

tion gradually increases from 110 K with increasing temperature, and its temperature dependence shows a sharp bend at T_c. The T_c is determined to be 110 K from the microwave absorption method and corresponds to that from electrical resistivity measurements[6].

It has been confirmed that the microwave absorption apparatus here developed has enough sensitivity to measure the change of dielectric constant of KH_2PO_4 and of magnetic permeability of $BiSrCaCu_2O_x$.

4.3   Magnetic relaxation in $Bi_2Sr_2CaCu_2O_x$

Anderson and Kim[8] proposed a theoretical formula for the process of magnetic relaxation based on a thermally activated flux creep model. According to their model, the time dependence of superconducting current J can be expressed as follows:

$$\frac{dJ}{dt} = -\frac{2Ba_f\nu_0}{\mu_0 d^2}\exp\left[-\frac{U_0}{kT}\left(1-\frac{J}{J_{c0}}\right)\right], \tag{2}$$

where U_0 is the pinning potential, B the magnetic density of superconductor, a_f the length between magnetic fluxes, ν_0 the frequency of magnetic flux, k the Boltzmann constant and J_{c0} the critical current density. If $t=0$ and $J=J_{c0}$, the solution of eq.(2) is expressed by

$$\frac{J}{J_{c0}} = 1 - \frac{kT}{U_0}\ln\left(\frac{2Ba_f\nu_0 U_0 t}{\mu_0 d^2 J_{c0}kT}+1\right) \tag{3}$$

$$\equiv 1 - \frac{kT}{U_0}\ln\left(\frac{t}{t_0}+1\right), \tag{3'}$$

where t_0 is the time when a magnetic flux jumps from original pinning center to the nearest neighbor one. Equation (3)' can be approximated by the following relation after long time duration of the magnetic relaxation where $t \gg t_0$:

$$J = J_{c0}\left(1-\frac{kT}{U_0}\ln(t/t_0)\right). \tag{4}$$

According to the Bean model[9] the absorbance of microwave power R is proportional to the magnetization (M) and therefore to the superconducting current J. On the basis of the Bean model, therefore, eq.(4) can be converted to the following equation:

$$R(t) = R_0\left(1-\frac{kT}{U_0}\ln(t/t_0)\right). \tag{5}$$

Equation (5) indicates that the microwave absorption power decays logarithmically with time and the slope of the relation between $R(t)/R_0$ and $\ln(t/t_0)$ gives the value of kT/U_0.

The microwave absorption in unirradiated and irradiated $Bi_2Sr_2CaCu_2O_x$ under various applied magnetic fields decays logarithmically with time, which indicates that the process of magnetic relaxation follows the thermally activated flux creep model.

Figures 4 and 5, respectively, show the microwave absorption power R normalized by R_0, which is the microwave absorption at $t=0$, as a function of time for $Bi_2Sr_2CaCu_2O_x$ irradiated without and with 1 MeV electrons. The slope of the lines increases with increasing applied magnetic field, indicating that the pinning potential decreases with increasing applied magnetic field. The slope of lines of the irradiated samples for each applied magnetic field is higher compared with that of unirradiated samples, indicating the pinning potential of irradiated sample to be higher than that of unirradiated one.

Figure 6 shows the relation between pinning potential and applied magnetic field for unirradiated and 1 MeV electron irradiated $Bi_2Sr_2CaCu_2O_x$. The values of pinning potential for unirradiated sample are 0.8-5.6 eV, depending on applied magnetic field. These values are larger compared with the results obtained from the conventional measurements[10]. The microwave absorption measurements give information only from the surface of sample due
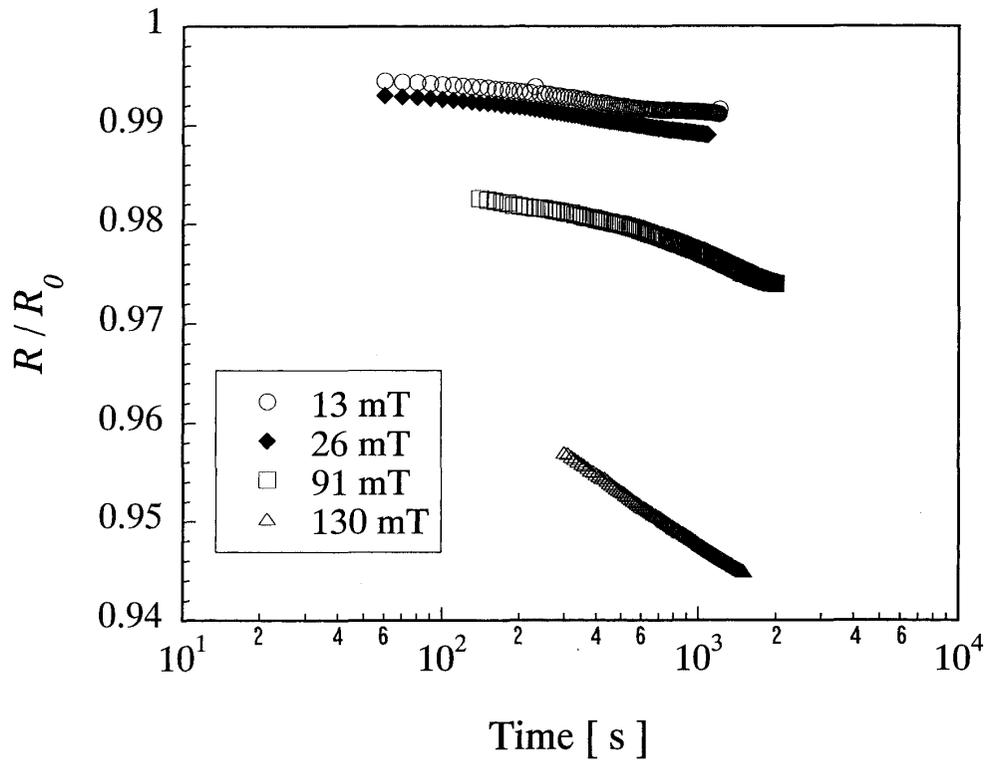
Fig. 4 The time dependence of microwave absorption in $Bi_2Sr_2CaCu_2O_x$ under various applied magnetic fields. The microwave absorption power R is normalized by R_0 which is the microwave absorption at $t=0$.
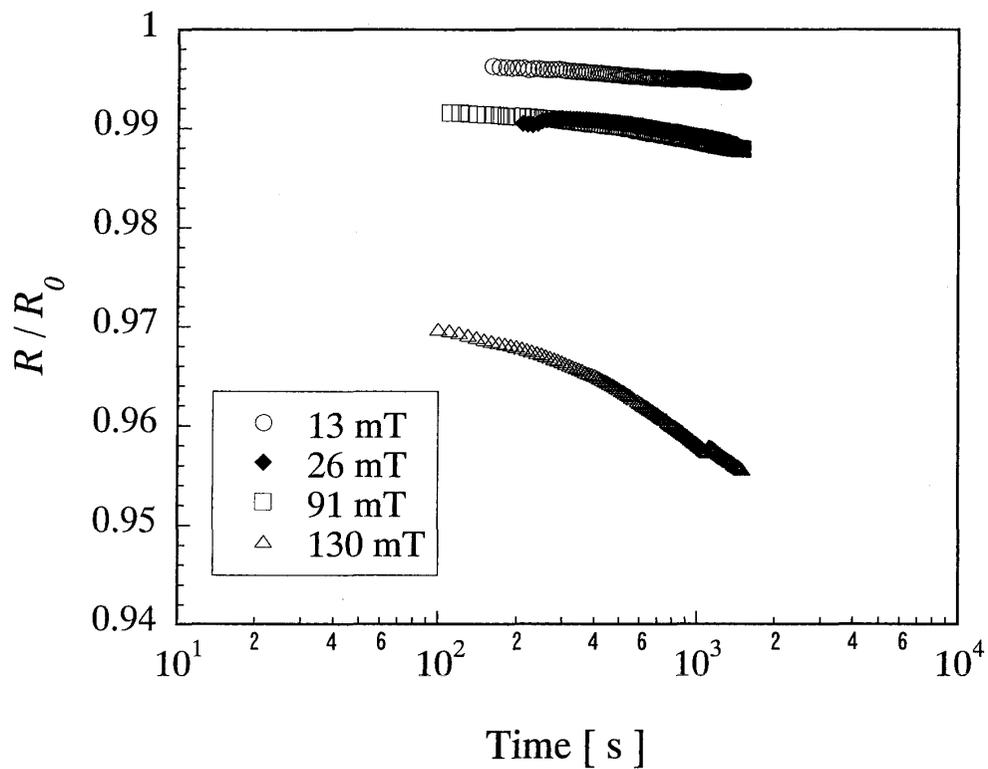
Fig. 5 The time dependence of microwave absorption in $Bi_2Sr_2CaCu_2O_x$ irradiated with 1 MeV electrons flux of 2.0×10^{20} e/m²s under various applied magnetic fields. The microwave absorption power R is normalized by R_0 which is the microwave absorption at $t=0$.
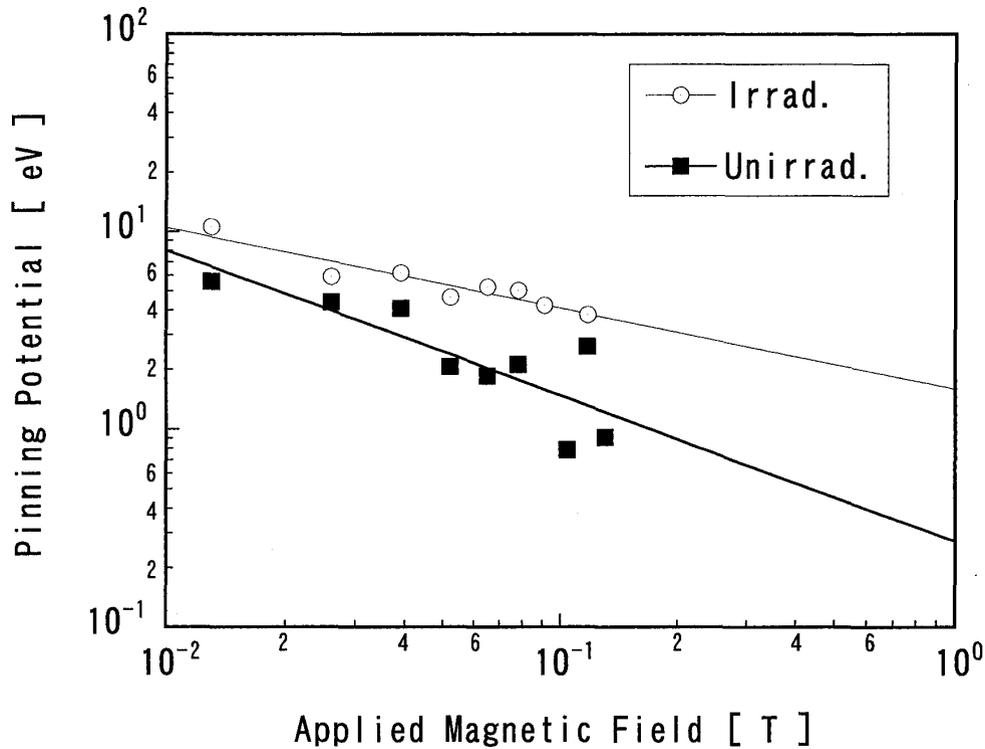
Fig. 6 The magnetic field dependence of the pinning potential of unirradiated and 1 MeV electron irradiated $Bi_2Sr_2CaCu_2O_x$.

to the skin effect. The pinning potential at the surface might be larger than that of bulk. The values of pinning potential for 1 MeV electron irradiated sample are 3.8-11 eV under magnetic fields 0-0.13 T. Those results suggest that 1 MeV electrons irradiation enhances the pinning potential. One MeV electrons produce simple point defects, such as vacancies and interstitials. The point defects induced by 1 MeV electrons are most probable pinning centers and act as good pinning centers in $Bi_2Sr_2CaCu_2O_x$.

5. Conclusions

A microwave absorption apparatus was developed to measure the magnetic relaxation in $Bi_2Sr_2CaCu_2O_x$. The magnetic relaxation of magnetic fluxes in superconducting $Bi_2Sr_2CaCu_2O_x$ with or without 1 MeV electron irradiation was measured through the microwave absorption. The pinning potential estimated under applied magnetic fields 0-0.13 T was 0.8-5.6 eV for the unirradiated sample and was 3.8-11 eV for the irradiated sample, depending on the applied magnetic field. The pinning potential was increased by 1 MeV electron irradiation. The point defects induced by electron irradiation are most probable effective pinning centers in $Bi_2Sr_2CaCu_2O_x$.

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