# Standard measurement method of irreversibility field of bulk RE-123 superconductors

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**Abstract.** We propose a standard measurement method of irreversibility field parallel to the $c$-axis of bulk oxide superconductors at 77.3 K. It is proposed that commercial magnetometers such as SQUID magnetometer or VSM are used for the measurement. Since the irreversibility field depends appreciably on the electric field criterion, the size of the specimen and the time starting measurement after setting the external magnetic field are prescribed to fix the electric field criterion. The number of measurement points is proposed to be 5 to 10 within the field range $\pm 5\%$ around the irreversibility field. It is confirmed that the irreversibility field of two (Nd, Eu, Gd)-123 (NEG-123) specimens were successfully measured with proposed standard measurement method. The target COV (Coefficient Of Variation) is 5%.

## 1. Introduction

Irreversibility field is a characteristic magnetic field at which the magnetic behavior of a superconductor changes from irreversible to reversible and vice versa. Since the critical current density is zero in the reversible magnetic region even in superconducting state, it is impossible to apply the superconductor to equipments of practical applications used at magnetic fields above the irreversibility field. Therefore, the irreversibility field is important parameter which determines the upper limit of magnetic field for practical applications. It is necessary to establish the standard measurement procedure of the irreversibility field.

The irreversibility field can be explained by the flux creep phenomenon[1, 2]. We have investigated the irreversibility field of Bi-2223 silver sheathed tapes with various methods including four terminal method, DC magnetization method and so on. It was found that the irreversibility field depends on the electric field criterion to determine the critical current density. The experimental results including such a dependence was well explained by the flux creep-flow model[3].

In the standard measurement of the critical current density of Bi-2223 silver sheathed tapes, $E$-$J$ characteristics are measured by the usual four terminal method, and the critical current density is determined by the electric field criterion. Hence, the standard measurement method of the irreversibility field of Bi-2223 tapes using the standard four terminal method was proposed[4]. That is, commonly used measurement methods are suitable for the standard measurement method.

On the other hand, in the case of RE-123 bulk oxide superconductors, not the four terminal method but the DC magnetization method is widely used for the estimation of the critical current density. Therefore, the DC magnetization method is considered to be suitable for the standard measurement method of the irreversibility field of bulk specimens. The definition of the critical current density depends on the electric field criterion in this case, too. Hence, it is proposed to measure the DC magnetization under a fixed condition to overcome the problem associated with the electric field criterion.

In this report, the standard measurement method of the irreversibility field of RE-123 bulk oxide superconductors is proposed. The irreversibility field is tried to be measured for two NEG-123 specimens with the proposed standard measurement method. The target COV (Coefficient Of Variation) is 5%.

## 2. Estimation method of irreversibility field

### *2.1. Electric field criterion*

Specimen is a RE-123 bulk oxide superconductor prepared by melt growth method. It was cut in a slab shape with the $c$-axis normal to the wide surface as shown in figure 1. The external magnetic field is applied parallel to the $c$-axis. Then, the magnetic moment $m$ is measured as a function of time, and the electric field $E$ is obtained as

$$E = -\frac{\mu_0 G}{2d(l+w)}\frac{\mathrm{d}m}{\mathrm{d}t} = -\frac{\mu_0 G w^2(3l-w)}{24(l+w)}\frac{\mathrm{d}J}{\mathrm{d}t}, \tag{1}$$

where $G$ is a coefficient determined by the geometry of the specimen and the isotropic current density is assumed in the $a$-$b$ plain[5]. Since the relaxation of the magnetic moment due to the flux creep becomes smaller with time, $E$ decreases to a sufficiently small value and $E$ does not appreciably change during the period for the measurement after some waiting time. It should be also noted that $E$ depends on the size of the specimen. Therefore, the time of starting measurement after setting the external magnetic field and the size of the specimen must be prescribed. Typical size is proposed to be $3 \times 2 \times 0.5$ mm$^3$, since this is suitable to commercial magnetometer. Then, the typical electric field is $10^{-9}$–$10^{-10}$ V/m.

### *2.2. Measurement system*

As aforementioned, the DC magnetization method is commonly used for the estimation of the critical current density of RE-123 bulk oxide superconductors, and SQUID magnetometer or VSM (Vibrating Sample Magnetometer) is usually used for the purpose. Hence, it is reasonable to select these systems for the standard measurement

method.

### *2.3. Temperature setting*

Commercial SQUID magnetometer or VSM can set the temperature from 4.2 K to 300 K in typical case. The accuracy of the temperature during the measurement is less than $\pm 0.1$ K. Since practical applications of oxide superconductor at 77.3 K of liquid $N_2$ temperature are supposed, this temperature is considered to be reasonable for the standard measurement. However, the specimen temperature is not equal to the setting temperature just after the setting, since the specimen is placed in vacuum. Therefore, it is necessary to wait about one hour to start the measurement after setting the temperature.

### *2.4. Magnetic field setting*

Magnetic moment measurement is required to start some seconds after the external magnetic field has been set to a constant strength. This is related to the electric field of the specimen mentioned in 2.1. In practical measurement, a period of 10 seconds to 1 minute is suitable for the waiting time.

Here, the example of measurement in an Y-123 bulk specimen at 77.3 K is referred to the explanation of the electric field criterion problem[6]. It was found that $n \simeq 5.5 (E \propto J^n)$ in this specimen around the irreversibility field. Thus, even if $E$ changes by one order of magnitude due to the condition of the measurement, the factor of change of $J$ is estimated as $10^{1/5.5} \simeq 1.5$. This results in the error less than 0.3% in the estimation of the irreversibility field[6]. Therefore, the deviation of one order of magnitude in $E$ at this level is not a serious problem for the estimation of the irreversibility field. That is, it is sufficient to fix the size of the specimen and the waiting time after setting the external magnetic field for the standard measurement of the irreversibility field.

### *2.5. Estimation of critical current density*

The magnetic moment $m$ is measured by SQUID magnetometer or VSM. The critical current density is estimated from the magnetic hysteresis of the magnetic moment $\Delta m$ using the Bean model as[7]:

$$J_{\mathrm{c}} = \frac{6\Delta m}{w^2 d(3l - w)} \tag{2}$$

where $d$, $l$, $w$ are defined in figure 1.

### *2.6. Estimation of irreversibility field*

$10^6$ A/m$^2$ is assumed as the criterion of the critical current density for the determination of the irreversibility field in RE-123 superconductor. For an exact estimation of the irreversibility field, 5 to 10 measurement points seem to be enough within $\pm 5\%$ around the irreversibility field. Therefore, a temporary irreversibility field is firstly measured with a large step of the magnetic field. Then, accurate measurement is performed around the temporary irreversibility field with a small step of the magnetic field. In typical case, the irreversibility field is about several Tesla at 77.3 K. Therefore, the step of the magnetic field is about 0.01–0.05 T.

Measurement conditions such as size and measured points and so on are typical ones for the standard measurement procedure of the irreversibility field. It is necessary to determined an allowable range of conditions based on the results of RRT (round robin test).

## 3. Example of measurement

### *3.1. Specimen*

We performed the measurement of the irreversibility field of RE-123 bulk oxide superconductors. Specimens are two (Nd, Eu, Gd)-123 bulk oxide superconductor (NEG-123), and the critical current density shows peak in increasing magnetic field.

These specimens contain NEG-211 secondary phase for 3 and 7 mol%. The specifications of the specimens are listed in Table 1.

### *3.2. Measurement*

The hysteresis of the magnetic moment $\Delta m$ was measured by Quantum Design MPMS-7 SQUID magnetometer. Since the irreversibility field is larger than 7 T at 77.3 K due to the peak effect of the critical current density, measurement was carried out at 84 K. The waiting time after setting the external magnetic field was 10 seconds.

### *3.3. Results*

The critical current density $J_c$ is estimated from measured $\Delta m$ by (2). Figure 2 shows the magnetic field dependence of the critical current density. The magnetic field at which the critical current density decreases to $10^6$ A/m$^2$ is about 4 T. Therefore, the accurate measurement is performed with the step of the magnetic field 0.05 T around 4 T. Figure 3 shows the result of the $J_c$-$B$ characteristics around 4 T. The solid line in the figure represents the approximate formula which is determined using the second order polynomial function in double logarithm estimated using the least squares approximation with 10 points of measurement data around the irreversibility field. The irreversibility fields of two specimens are estimated as 3.80 T and 4.70 T, respectively. Therefore, the measurement is succeeded with the present method.

Since the simple estimation method is suitable for industrial measurement, it is necessary to consider more simple method for the estimation of the irreversibility field. For example, the irreversibility field of two specimens which are estimated from only 3 points around the irreversibility field and apply the linear least squares approximation are 3.80 T and 4.71 T, respectively. In the case of 5 points, they are 3.81 T and 4.71 T, respectively. Therefore, it is found that the result is not changed appreciably. The validity of these simply estimation methods should be checked through round robin test procedure in the near future.

## 4. Summary

In this report, the standard measurement method of the irreversibility field in RE-123 bulk oxide superconductor is proposed. Commercial SQUID magnetometer or VSM is used for the measurement of the magnetic hysteresis of the magnetic moment. The specimen size and the waiting time after setting the external magnetic field, 10 seconds to 1 minute, are fixed so that the measurements might be done in a fixed range of electric field. The number of the measurement points of $J_c$ is about 10 in the range of $\pm 5\%$ around the irreversibility field to keep the accuracy of the estimation. The irreversibility field is determined by the second order polynomial function in double logarithm and estimated using the least squares approximation. The standard measurement of the irreversibility field is confirmed by two specimens of NEG-123. It is found that the measurement can be performed without problems.

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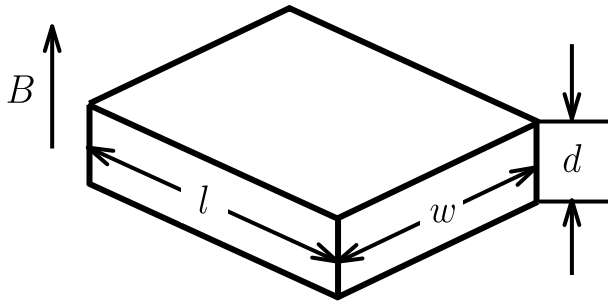


Fig.1 Schematic illustration of slab shape specimen of bulk RE-123 oxide superconductor.

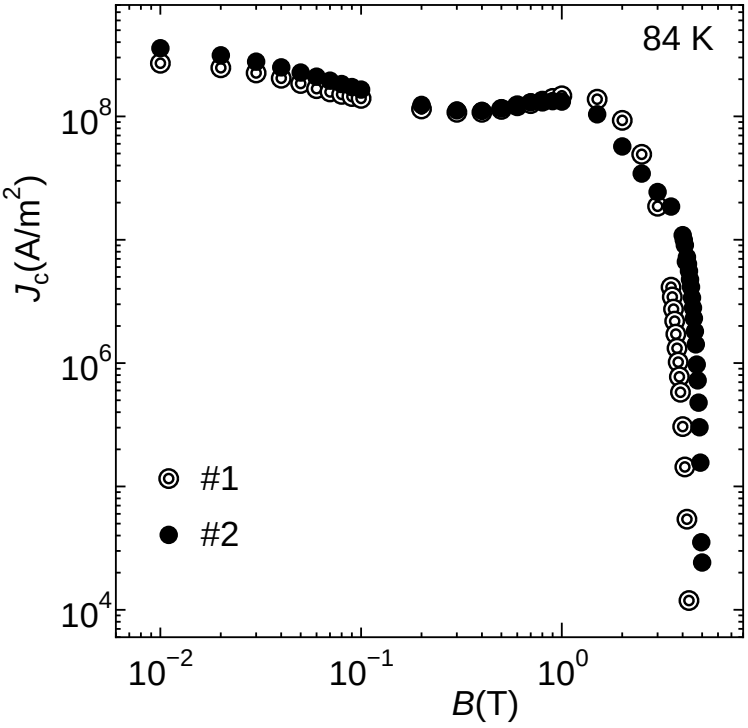


Fig.2 Magnetic field dependence of the critical current density of NEG-123 bulk oxide superconductors with different NEG-211 contents.

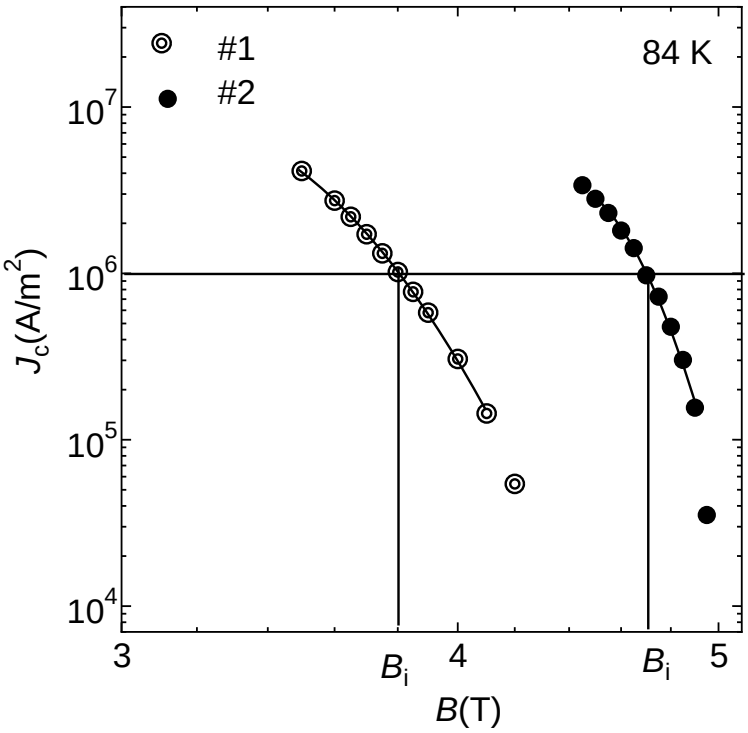


Fig.3 Enlarged figure for determination of irreversibility field.

Table 1 Specifications of specimens

| No. | specimen | size [$mm^3$] | $T_c$ [K] |
|---|---|---|---|
| #1 | NEG-123+NEG-211 3 mol% | $1.73 \times 1.75 \times 0.43$ | 92.5 |
| #2 | NEG-123+NEG-211 7 mol% | $1.92 \times 2.18 \times 0.16$ | 93.3 |