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Standardization of Critical Temperature Measurement based on IEC International Standard

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Abstract—**For disscminating a ney IEC international standard of critical trmperature of NbTi,Nb_3Sn and Bi-2223 Composite Suterconductors, we develpted a measuring system and studied standardization of test method. The measuring system consisted of cryogenic reservoir, base plate, thermometer, voltmeter and current source. Various specimens of the Nbti, Nb_3Sn and Bi-2223 composite superconductors were tested using this system for measuring their critical temperatures. After measuring the resistance-temperature relation, the data were compensated with thermoelectric voltages for NbTi Nb_3Sn specimens. NbTi specimens showed 9.2 K ~ 9.5 K of transition temperature and Nb_3Sn specimen showed about 18 K. Bi-2223 specimens showed 104 K ~ 107 K of transition temperature.**
Keywords: critical temperature, composite superconductor, resistance measurement, standardization.

1. Introduction

Critical temperature is the most important parameter for characterizing superconducting properties since the higher critical temperature is the lower the cooling power consumption for practical application. There are various methods for measuring the critical temperature including resistive, magnetic and thermal methods. The magnetic methods require expensive and complicate SQUID magnetometer or VSM(Vibrating Sample Magnetometer). The thermal methods are also complicate for measuring their specific heat and the accuracy of measurement is not so good as the resistive and magnetic methods. However, the resistive method is simpler and more practical than other methods.

IEC(International Electrotechnical Commission), one of the international standardization organization, published a new international standard concerning the test method of critical temperature of NbTi , Nb_3Sn and Bi system oxide composite superconductors in June 2002. The standard named "IEC international standard 61788-10" describes preparation of specimen, temperature control and voltage measurement, measurement procedure, and etc.

At present, many research institutes and universities are developing superconducting system for application of superconductors in Korea. In order to disseminate the new IEC international standard 61788-10, a measurement system was fabricated and test method for critical temperature measurement of various composite superconductors was studied at KRISS.

2. Definition of Critical Temperature

In the IEC61788-10 standard[1] , the critical temperature(T_c) is determined as the mid-point of resistive transition from normal state to superconducting state with a minimum of dc transport current and at no applied magnetic field strength except for geomagnetic field. Fig. 1 shows schematically a curve of resistance versus temperature for a composite superconductor, which was cited from the IEC standard. Determination method for critical temperature is following. A tangential line to the part of the curve in the normal tate region is drawn. The value of the temperature at the intersection of the transition curve and a line with 50% of the height of the tangential line is T_c.

The standard also defines a transition width(ΔT_c). First, two lines with height equaling 10% and 90% of the tangential line are drawn. The temperature at the respective intersections with the transition curve, denoted $Tc_{0.1}$ and $Tc_{0.9}$, respectively, are determined as shown in Fig. 1. The transition width, ΔT_c is then defined as $T_{c0.9} - T_{c0.1}$.

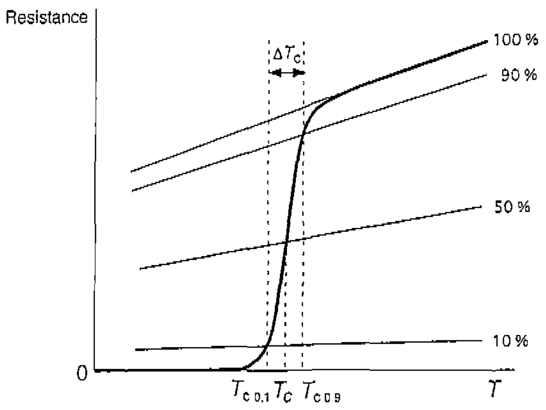


Fig.1. Determination of critical temperature from a R-T curve [1].

The resistance-temperature(R-T) curve shall be measured at least up to 10% or 20% above T_c and for both positive and negative currents. From each R-T curve, each critical temperature corresponding both polarity, T_{c+} and T_{c-}, shall be measured. The critical temperature, T_c, shall then be determined as $T_c=(T_{c+} + T_{c-})/2$. $Tc_{0.1}$ and $Tc_{0.9}$ shall also be determined with the same procedure.

3. Fabrication of Measurement System

To satisfy requirement of the IEC 61788-10 international standard, we designed and fabricated a R-T measurement system as shown in Fig. 2. Temperature control being recommended in IEC61788-10 standard is a modified adiabatic method in which cryostat holds the specimen at a fixed distance above the cryogenic liquid for obtaining a temperature. For temperature control, the specimen is immersed into the cryogenic liquid and then gradually warmed by raising the specimen above the liquid level.

The system consisted of cryogenic reservoir, base plate for mounting a linear specimen, thermometer, voltmeter and stable current source.

The cryogenic reserviorr(MVE Cryogenics E4)keeps 10 liter liquid helium for about 5 hours or liquid nitrogen for about 20 hours. The current source(HP 6642 A) supplies a constant current of 0.05 A to 5 A with 0.18%-accuracy and 0.01%-stability. The voltmeter(Keithley 181) measures the produced voltage from the specimen with 0.01%-accuracy and 10 nV-sensitivity. The thermometer consists of a temperature indicator(Lakeshore 218) and a calibrated germanium sensor(LakeshoreGR-200A). Measurement range of the thermometer is 1.4 K to 475 K and the accuracy is ±0.005 K below 10 K and ±0.09 K at 100 K, respectively.

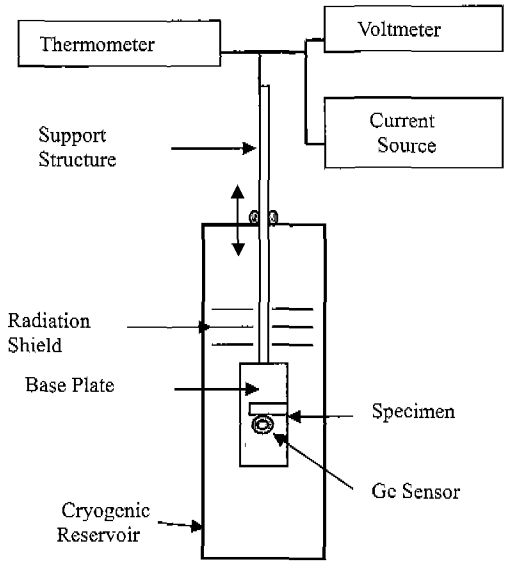


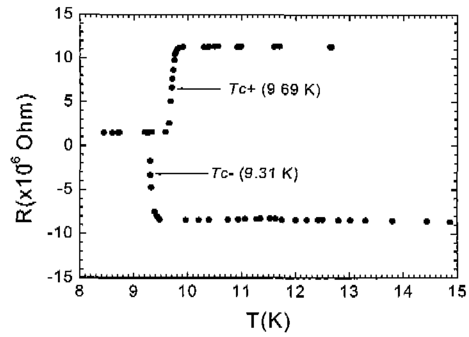
Fig. 2. Experimental setup for measurements of critical temperature[2].

A specimen of NbTi, Nb_3Sn, or Bi-2223 wire is mounted on the base plate as shown in Fig. 2. For measurement below 20 K, radiation shield of three folds was attached to the support structure. The more details are shown in the reference[2]. The specimen was soldered with current contacts near each end of the specimen and a pair of voltage contacts between the current contacts of the specimen. After wiring, the specimen was mounted on the base plate with crycon grease(Lakeshore). For thermal equilibrium, the specimen mounted to the base plate was wrapped with overlapping aluminum foils.

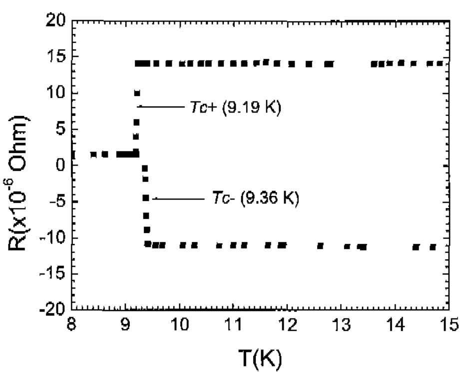
4. . Measurements and Discussion

After mounting the specimen, the base plate was slowly lowered into the cryogenic liquid and cooled to liquid temperature for about 5 minutes. When the specimen was in the superconducting state, a test current with a positive polarity was applied and thermoelectric voltage, U_{0+}, was measured for compensating temperature gradient between cryogenic and room temperatures. And then, a positive R-T curve was measuring while the specimen was gradually warming. The same procedure but negative polarity current was performed and measurement values were obtained for negative U_{0-} and R-T curve.

The R-T curves of two NbTi specimens for determination of the critical temperature are shown in Fig. 3. In the Fig. 3(a), the resistance of 100% value at transition is +11.27 μΩ for the positive R-T curve while the resistance of 100% value for the negative R-T curve



(a) NbTi-A specimen



(b)NbTi-B specimen

Fig. 3. R-T curves of two NbTi specimens for the positive and negative polarity measurements.

is −8.24 μΩ. The difference between positive and negative curves is due to the thermoelectric voltage. The thermoelectric voltage is caused from large temperature difference between room temperature of voltmeter and cryogenic temperature of specimen. In the NbTi-A specimen, U_{0+} and U_{0-} were same value of +1.53 μV and nearly constant during measurement. For obtaining T_{c+} and T_{c-}, we compensated the measured values of both R-T curves with thermoelectric voltage(U_{0+} and U_{0-}). T_{c+} and T_{c-} are 9.69 K and 9.31 K, respectively, as shown in Fig 3(a) and the critical temperature of the NbTi-A specimen is determined to 9.50 K. From the positive and negative curves, the transition width is also determined to 0.12 K.

In Fig. 3(b), the NbTi-B specimen shows a sharp resistive transition. The U_{0+} and U_{0-} were about +1.58 μV. We compensated the measured data of both R-T curves with the thermoelectric volatage as same procedure as done in the NbTi-A specimen. NbTi-A specimen.

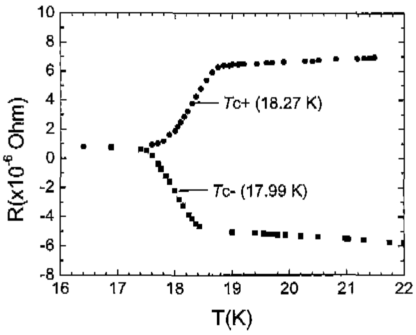


Fig. 4. R-T curves of a Nb_3Sn specimen for the positive and negative polarity measurements.

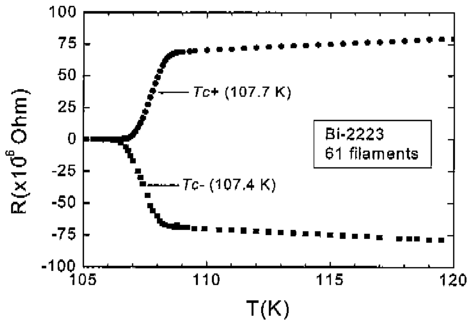
In the case of the NbTi-B specimen, T_{c+} and T_{c-} are 9.19 K and 9.36 K as shown in Fig. 3(b) and the critical temperature is determined to 9.27 K with transition width of 0.04 K.

The R-T curves of a Nb_3Sn specimen are shown in Fig. 4. The resistive transition from normal state to superconducting state begins at about 18.8 K and ends at about 17.4 K. Dissimilarly as in NbTi specimens, the Nb_3Sn specimen exhibits some slope of the R-T curves after end of the superconducting transition. The thermoelectric voltages are in the range of +1.62 μV for the positive and negative measurements. After compensating the thermoelectric voltage, T_{c+} and T_{c-} are calculated to 18.27 K and 17.99 K, respectively. The critical temperature of the Nb_3Sn specimen is determined to 18.13 K.

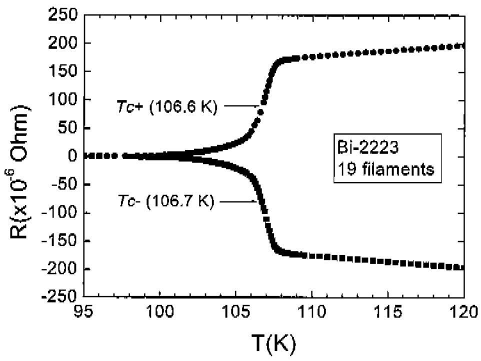
Fig. 5 shows R-T curves of the two Bi-2223 specimens for determination of the critical temperature. In the case of Bi-2223 specimens, the critical temperatures are higher than 100 K and the resistance values just before superconducting transition for both polarity are almost same while NbTi and Nb_3Sn showed different values. The thermoelectric voltages(U_{0+} and U_{0-}) of the Bi-2223 specimens are +0.2 μV ~ +0.3 μV. These values are negligiable if we compare to several decade μV of voltage at transition.

The Bi2223-A specimen shows typical R-T characteristics of Bi-2223 phase while the Bi2223-B specimen shows broad transition. In the case of the Bi2223-A specimen, T_{c+} and T_{c-} are 107.7 K and 107.4 K as shown in Fig. 5(a) and the critical temperature is determined to 107.5 K with transition width of 1.2 K.

In the case of Bi2223-B, the R-T curves show broad superconducting transition. The resistive transition from normal state to superconducting state begins at about 108 K and ends at about 98 K. Although the critical temperature was calculated to 106.6 K according to the



(a) Bi2223-A specimen



(b) Bi2223-B specimen

Fig. 5. R-T curves of two Bi-2223 specimens for the positive and negative polarity measurements.

mid-point of IEC 61788-10 standard, the value is not reasonable. We should think over application of other criterion definitions.

The IEC 61788-10 standard recommends three other criteria if specimen shows a broad or multi-stage transition. Three criteria are resistivity criterion of $3x10^{-9}$ Ωm, modified resistivity criterion of $1x10^{-8}$ x(1/RRR), and 10%-transition criterion. (The RRR is residual resistance ratio of specimen.)

If we apply the resistivity criterion to Bi2223-B specimen, T_c is 106.4 K. RRR of the Bi2223-B specimen is calculated to 3.1. When the modified resistivity criterion is applied, T_c is also nearly 106.4 K. Finally, if the 10%-transition criterion is applied, T_c becomes 104.2 K.

We think that the 10%-transition criterion for the Bi2223-B is more practical than other two criteria because the specimen shows a broad resistive transition from 98 K to 108 K. Comparison of applications of

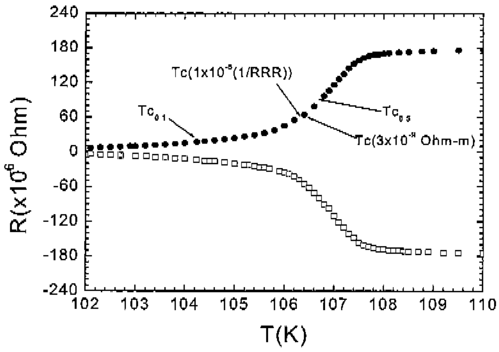


Fig. 6. Comparison of T_cs calculated from alternative criteria.

several T_cs criteria for critical temperature of the Bi2223-B is shown in Fig. 6.

In Table 1, measurement results of various specimens are summarized. As shown in Table 1, the NbTi specimens exhibit 9.2 K ~ 9.5 K and the Nb_3Sn specimen shows 18 K as their critical temperatures. The Bi-2223 specimens show above 100 K. The thermoelectric voltages are in the range of +1.5 μV for NbTi and Nb_3Sn, and +0.2 μV for Bi-2223.

TABLE I Measurement results of the specimens.

Specimen	T_{c+} (K)	T_{c-} (K)	T_c (K)	ΔT_c (K)	U_0 (μV)
NbTi-A	9.69	9.31	9.50	0.12	+1.53
NbTi-B	9.19	9.36	9.27	0.04	+1.58
Nb_3Sn	18.27	17.99	18.13	0.70	+1.62
Bi2223-A	107.7	107.4	107.5	1.2	+0.2
Bi2223-B	104.2	104.2	104.2	-	+0.3

5. CONCLUSION

To disseminate the IEC 61788-10 international standard, we designed and fabricated a simple R-T measurement system for testing NbTi, Nb_3Sn and Bi-2223 composite superconductors. The system provided a good experimental environment for measuring R-T curves of various composite superconductors.

We characterized various specimens of NbTi, Nb_3Sn and Bi-2223 composite superconducting wires for determining their critical temperatures. NbTi specimens showed 9.2 K ~ 9.5 K of transition temperature and Nb_3Sn specimen showed about 18 K. Bi-2223 specimens showed 104 K ~ 107 K of transition temperature.

The thermoelectric voltages are approximately +1.6 μV for NbTi and Nb_3Sn specimens and +0.2 μV for Bi-2223 specimens. It is important that the measured data should be compensated with thermoelectric voltage for NbTi or Nb_3Sn superconductors.

The fabrication of measuring system and research result will be used to characterize the superconducting properties of composite superconductors. Besides, this study could lead to establishment of the standardization of the critical temperature measurement in Korean superconductivity society.

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