Standardization of the Critical Temperature Measurement by Using an AC Susceptometer

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Abstract

We have studied the standardization of critical temperature measurements by using an ac susceptometer. Wire forms of NbTi, Nb_3Sn, and Bi-2223 superconductors were prepared to measure the temperature dependences of the magnetization. In order to study the optimum ac magnetic field and frequency, various amplitudes from 2 Oe to 10 Oe with frequencies from 11.3 Hz to 1033 Hz were applied to three specimens. Analytical comparison of the magnetic curves with the resistive curves was accomplished to investigate validity of using the new method.

Keywords : critical temperature, composite superconductor, standardization, ac susceptibility

I. Introduction

For determination of the critical temperature, the resistive method and the magnetization measurement method have been generally used. The resistive method is more often used to define the critical temperature because it is simpler than the magnetization measurement method. However, if the specimen is composed of inhomogeneous grains or the spaces between the grains are insulating, the resistive method is not adequate to measure the critical temperature.

Along with the resistive method, the magnetization measurement also provides a significant and useful tool to evaluate the quality of a superconductor. Last year, we studied the dc magnetic method based on a SQUID magnetometer as a standard method [1]. For a dc magnetization measurement, a sample is magnetized with a static dc field H_{dc}. The sample is then moved inside a detection coil that is used to measure variations of the magnetic flux due to the magnetized sample. The output signal from the detection coil is measured by a SQUID. The dc susceptibility χ_{dc} can be described as follows.

$$\chi_{dc} = \frac{M}{H_{dc}} \qquad (1)$$

where M is dc magnetization of the sample.

However, in an ac measurements, the sample is centered within a detection coil while driven with a time-varying field H_{ac} [2]. The ac field produces a time-dependent moment m in the sample and the ac susceptibility χ_{ac} can be given as Eq. (2) where V is volume of the sample.

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$$\chi_{ac} \approx \frac{dm}{V \cdot H_{ac}} \rightarrow \frac{dm}{dH} \quad (2)$$

AC susceptometer is capable of measuring the susceptibity under small ac magnetic fields: It enables us to measure the magnetic susceptibility as a function of the frequency and the amplitude of the applied ac field. AC susceptometer can also separate the real component χ_{ac} from the imaginary component χ'_{ac} of the complex susceptibility.

To standardize the critical temperature measurements, we have studied magnetization-temperature (*m-T*) curves with different amplitudes from 2 Oe to 10 Oe and frequencies from 11.3 Hz to 1033 Hz to study suitable amplitude and frequency. For adequate criterion of the critical temperature, we also studied the peak temperature of the imaginary components and compared the ac magnetization curves with the dc magnetization ones.

II. Sample preparation and experiments

AC susceptibility measurements were performed using a commercial susceptometer (Quantum Design PPMS), which consists of a temperature control unit, drive and detection coil set, and a computer control unit [3]. The drive coil can generate ac fields up to 10 Oe in a frequency range of 10 Hz to 10 kHz at a desired temperature. Before measurements, the ac susceptometer was calibrated with a Palladium standard and a Ge thermometer.

In this study, three specimens of multi-filamentary NbTi and Nb_3Sn wires, and Ag-sheathed Bi-2223 tape were used because they are generally used in many applications. Diameter and length (mass) of NbTi and Nb_3Sn specimen were 0.78mm × 3.72mm (0.013g), 0.77mm × 5.20mm (0.022g), respectively. Dimension (mass) of rectangular Bi-2223 tape were 4.05mm × 0.30 mm × 5.33 mm (0.053g).

The specimens were mounted in a non-magnetic holder and then installed inside the detection coil of the ac susceptometer. After stabilization at the designated temperature, an ac field was applied to the specimen and then the *m-T* curves were measured with increasing temperature.

III. Results and Discussion

Amplitude and frequency dependence of *m-T* curves

Fig. 1 shows result of *m-T* curves of NbTi wire in various ac fields from 2 Oe to 10 Oe at 11.3 Hz. As shown in Fig. 1, the measured magnetic moments at 7.0 K were -6.12×10^{-5} emu at 2 Oe, -1.51×10^{-4} emu at 5 Oe, -2.16×10^{-4} emu at 7 Oe, and -3.03×10^{-4} emu at 10 Oe. In a temperature range from 7 K to 8.5 K, *m* of NbTi changes slowly, but from 8.8 K to 9 K these values change rapidly. If we enlarge the region near 9 K in Fig. 1, the onset temperature from normal state to superconducting state for each amplitude is seen to be 9.03 K and does not appear to depend on the amplitudes of the applied fields.

Results for the frequency dependence of *m-T* curves for NbTi wire are shown in Fig. 2. At amplitude of 10 Oe, the frequencies were varied from 11.3 Hz to 1033 Hz. As shown in Fig. 2, the ac fields at 11.3 Hz and 113 Hz show typical *m-T* curves, but *m-T* curves at 533 Hz and 1033 Hz were shifted toward more negative magnetization. From the *m-T* curves for NbTi specimen, amplitudes of 7-10 Oe with frequencies of 11.3-113 Hz are found to be suitable for the critical temperature measurement.

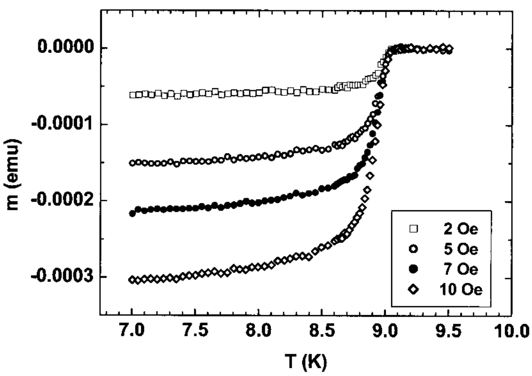


Fig. 1. Amplitude dependence of *m-T* curves for NbTi wire specimen.

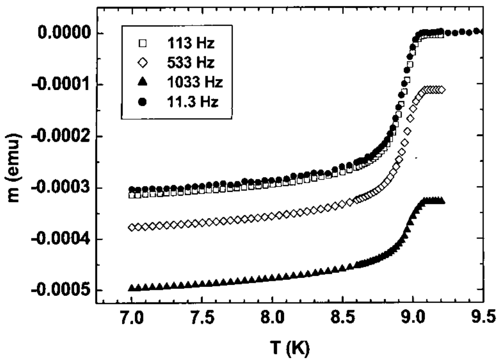


Fig. 2. Frequency dependence of *m-T* curves for NbTi wire specimen.

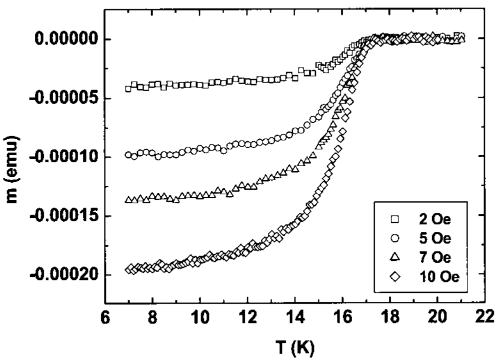


Fig. 3. *m-T* curves for Nb_3Sn wire in the range of 2 Oe to 10 Oe at 11.3 Hz.

Fig. 3 shows *m-T* curves for Nb_3Sn specimen measured with various amplitudes from 2 Oe to 10 Oe at 11.3 Hz. As shown in Fig. 3, the measured moments at 7 K were -4.2×10^{-5} emu at 2 Oe, -1.0×10^{-4} emu at 5 Oe, -1.4×10^{-4} emu at 7 Oe, and -1.9×10^{-4} emu at 10 Oe. In a temperature range from 7 K to 15 K, the *m-T* curves change slowly, but from 16 K to 18 K these values change rapidly. If we enlarge the region near 18 K in Fig. 3, the onset temperature for each amplitude is about 17 K, independent of the amplitude of the applied fields.

The frequency dependences of *m-T* curves for Nb_3Sn wire are shown in Fig. 4. At 10 Oe, frequencies were varied from 11.3 Hz to 1033 Hz. As shown in Fig. 4, the *m-T* curves at 533 Hz and at 1033 Hz do not show clear transitions from the normal to the superconducting state while those at 11.3 Hz, 55 Hz, and 113 Hz show clear transitions near 17 K.

From the *m-T* curves of Nb_3Sn specimen, amplitudes of 7-10 Oe with frequencies of 11.3-113 Hz are also found to be suitable for the critical temperature measurement.

For the Bi-2223 specimen measurements, we used Ag-sheathed tape. We have studied the *m-T* characteristics of Bi-2223 specimen. The amplitude dependence of the *m-T* curves for Bi-2223 tape for amplitudes of 2-10 Oe at frequency of 11.3 Hz are illustrated in Fig. 5.

As shown in Fig. 5, the measured moments at 40 K were -3.6×10^{-4} emu at 2 Oe, -8.5×10^{-4} emu at 5 Oe, -1.1×10^{-3} emu at 7 Oe, and -1.6×10^{-3} emu at 10 Oe. Generally, *m* of Bi-2223 specimen changes slowly

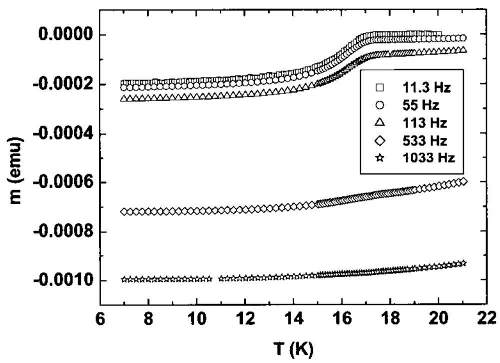


Fig. 4. *m-T* curves measurement for Nb_3Sn wire at a frequency range from 11.3 Hz to 1033 Hz at H=10 Oe.

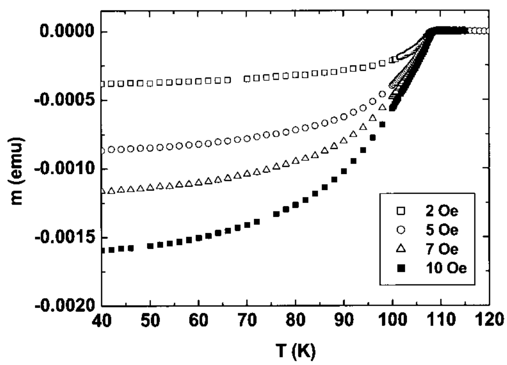


Fig. 5. *m-T* curves for Bi-2223 tape in an amplitude range from 2 Oe to 10 Oe at 11.3 Hz.

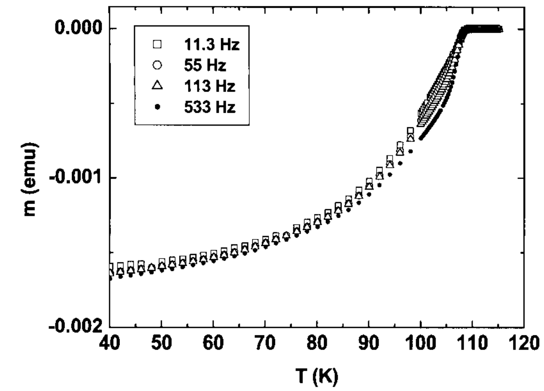


Fig. 6. Field dependence of *m-T* curve of Bi-2223 tape at H_{ac}=10 Oe.

below the onset temperature, but its value changes rapidly at around 110 K.

If we enlarge the region near 105 K in Fig. 5, the onset temperatures from the normal state to the superconducting state are seen to be about 108 K regardless of the amplitude of the applied fields.

Frequency dependences of the *m-T* curve for Bi-2223 specimen are shown in Fig. 6. Unlike the NbTi and Nb_3Sn specimens, Ag-sheathed Bi-2223 tape does not show any significant dependence on the frequency from 11.3 Hz to 533 Hz. We speculate that B-2223 tape consists of Bi-2223 powders while NbTi and Nb_3Sn consist of fine multi-filaments with Cu stabilizer. Thus, high frequency ac loss of Bi-2223 specimen is much smaller than those of NbTi and Nb_3Sn specimens.

In the case of Ag-sheathed Bi-2223 specimen, amplitudes of 5-10 Oe with frequencies of 11.3-533 Hz are found to be suitable for the critical temperature measurements.

For all the specimens, most *m-T* curves did not appear flat below the transition temperature. Thus, the mid-point criterion as in the case of the resistive method is not adequate for the magnetic method. Rather, we found that the onset temperature criterion from the normal to the superconducting state is more reasonable.

Also, we found that ac magnetic field of 5-10 Oe with frequencies of 11.3-113 Hz are suitable for measuring the critical temperature

Peak temperature of the imaginary part for T_c determination

There is some possibility of using the peak temperature of magnetization-temperature curve of imaginary part (*m'-T*) for T_c determination, at which ac loss due to the phase difference is maximum. We studied the peak temperature of the imaginary part for the specimens. Fig. 7 shows each *m'-T* curve for three specimens.

Fig. 7(a) shows *m-T* and *m'-T* curves for NbTi specimen. Among the imaginary parts, *m'-T* curves at 2 Oe and 5 Oe at 11.3 Hz appear noisy and did not show clear peaks. Other *m'-T* curves at 7 Oe and 10 Oe show more clear peaks at 8.94 K while the onset temperature appears to be 9.05 K in the *m-T* curve. This implies that the transition from the normal to the superconducting states begins at 9.05 K, while the maximum ac loss occurs at 8.94 K.

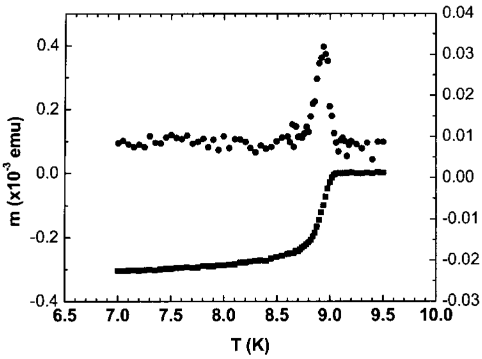
The *m-T* and *m'-T* curves for Nb_3Sn specimen are shown in Fig. 7(b). In the case of Nb_3Sn specimen, the peak in the *m'-T* curve appears broad and is not as sharp as that of NbTi specimen. It might be due to that the superconducting phase of the Nb_3Sn specimen is not homogeneous due to insufficient heat treatment. The peak temperature of the *m'-T* curves is 17.2 K while the onset temperature in the *m-T* curve is 17.4 K.

Fig. 7(c) shows the *m-T* and the *m'-T* curves for the Bi-2223 specimen. Although the *m'-T* curve do not show a clear peak, a peak appeared at 96.0 K. The onset temperature of the real part is 108 K. This means that the superconducting transition begins at 108 K, while maximum ac loss occurs at 96 K.

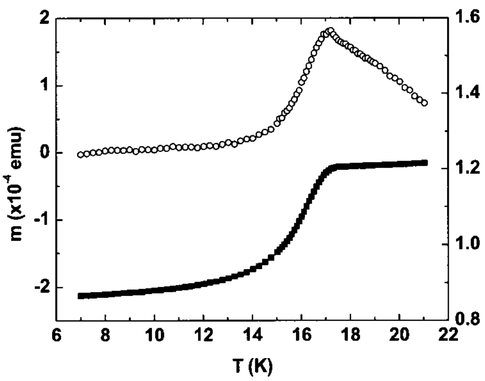
Comparing the peak temperatures in the *m'-T* curves with the onset temperatures in the *m-T* curves, the peak temperatures appeared mostly lower than the corresponding onset temperatures. Thus, we think that the peak temperature is not suitable for being used as the critical temperature in ac susceptibility measurement.

Comparison of dc *m-T* curve with ac *m-T* curve

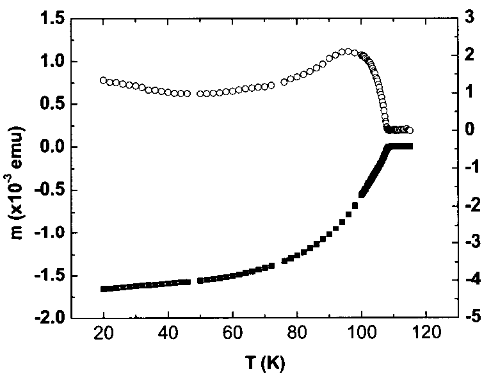
As discussed in the Introduction, ac susceptibility is measured in a time-varying ac field while dc



(a) NbTi specimen



(b) Nb_3Sn specimen



(c) Bi-2223 specimen

Fig. 7. Imaginary and real part of the magnetic moment-temperature curves for three specimens.

susceptibility is measured in a static dc field. Thus, aspects of the ac *m*-*T* curves may be different from those of the dc *m*-*T* curves and eventually may affect the onset temperature.

We compared ac *m*-*T* curves with dc *m*-*T* curves for three specimens with all the ac *m*-*T* curves measured at 10 Oe at a frequency of 11.3 Hz. All the dc *m*-*T* curves were measured by a SQUID magnetometer in a dc field of 10 Oe. Comparisons are made for three specimens as shown in Fig. 8.

When the *m*-*T* curve for NbTi specimen is compared with that for Nb_3Sn and Bi-2223 specimens,

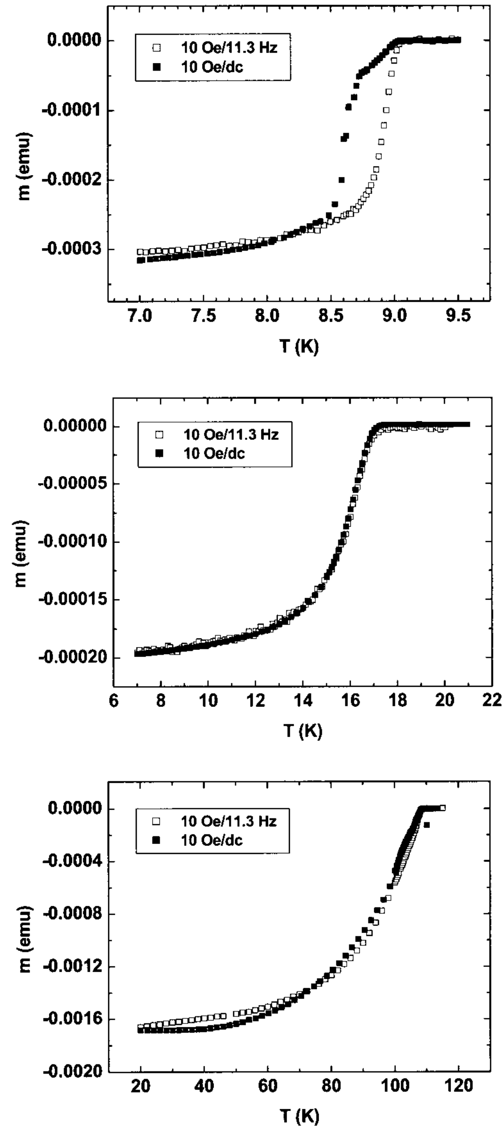


Fig. 8. Comparison of dc with ac *m*-*T* curve for three specimens.

the dc *m-T* curve of NbTi specimen appears somewhat strange. It might be due to that some unknown magnetic materials affect the dc *m-T* curve, considering compositions of NbTi specimen such as of NbTi filaments, Cu stabilizer, and some mechanical supporting material. However, the onset temperatures appear almost the same with the value of 9.05 K for both cases.

For Nb_3Sn specimen, the ac and dc *m-T* curves appear consistent with the respective onset temperatures of 17.3 K and 17.4 K.

Data from the ac curve seem to be consistent with the corresponding ones from the dc curve for Bi-2223 specimen except for a little difference near 100 K. When we enlarged the *m-T* curves near the transition, the onset temperatures appeared to be almost 108 K.

Comparison of *m-T* curves with *R-T* curves

The international standard IEC 61788-10 based on the resistance measurement was published in 2002 [4]. In the standard, the critical temperature is determined as a mid-point temperature of the resistive transition (*R-T* curve) between the normal and the zero resistance state. We compared the *m-T* curves with the corresponding *R-T* curves. As shown in Fig. 9 for Bi-2223 specimen, the *R-T* curve shows a sharp transition near 108 K while the *m-T* curve shows a broad transition.

This implies that Bi-2223 specimen might consist of inhomogeneous grains in the superconducting phase, resulting in percolation of the test current through selective grains in the resistance measurements. Another reason for observation of a broad *m-T* curve might be due to penetration of the applied field. As the temperature of Bi-2223 approaches the transition temperature, the penetration depth becomes longer [5]. As more applied field penetrates into the specimen, diamagnetic signal of the specimen becomes weaker, resulting in a broad *m-T* curve.

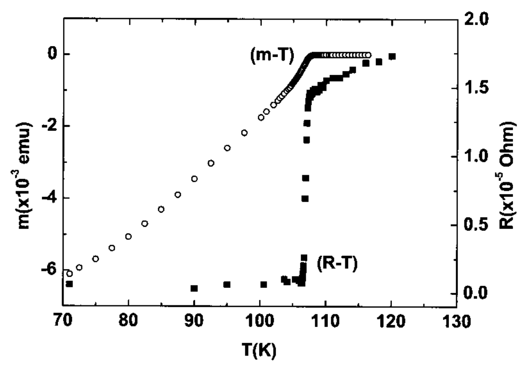


Fig. 9. Comparison of the *m-T* curve with the *R-T* curve for Bi-2223 specimen.

IV. Conclusion

We investigated the magnetic moement-temperature curves by using an ac susceptometer for providing a measurement standard for the critical temperature. Unlike the resistance standard, a mid-point temperature of the *m-T* curve did not appear suitable for determining the critical temperature. We suggest the onset temperature as the critical temperature in the ac susceptibility measurement method. Ac fields with the amplitude of 7-10 Oe and the frequency of 11.3–113 Hz appeared suitable for determining the critical temperature. We think that this method can also be used as a measurement standard for the critical temperature along with the IEC61788-10 standard.

References

[1] K. W. Lee, M. S. Kim, D. H. Kim, and S. G. Lee, "Magnetic T_c measurements of composite superconductors for a standard method", Progress in Superconductivity, Vol. 6, No. 1, 24 (2004).

[2] F. Gomory, "Characterization of high-temperature superconductors by AC susceptibility measurement", Super. Sci. Tech., Vol. 10, 523 (1997).

[3] PPMS manual published by Quantum Design Co.

[4] IEC61788-10 Critical temperature measurement by a resistance method (2002).

[5] Handbook of Superconductivity edited by C. P. Pool, 48-49, (2000).