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***Abstract*— A round robin test of residual resistance ratio (RRR) was performed for Nb_3Sn composite superconductors prepared by internal tin method by six institutes with the international standard test method described in IEC 61788-4. It was found that uncertainty mainly resulted from determination of the cryogenic resistance from the intersection of two straight lines drawn to fit the voltage vs. temperature curve around the resistive transition. The measurement clarified that RRR can be measured with expanded uncertainty not larger than 5% with the coverage factor 2 by using this test method.**

***Index Terms*— Nb_3Sn composite wire, copper, residual resistance ratio**

I. Introduction

COPPER or aluminum used as matrix material in Nb-Ti and Nb_3Sn composite superconductors contributes to the stability by working as an electrical shunt in case of too large current and as a good conducting material to carry generated heat to the surrounding coolant. The resistivity of such a material is an important quantity that influences the stability. The residual resistance ratio (RRR), defined as a ratio of the resistance at room temperature to that just above the superconducting transition, is a parameter that represents the quality of the stabilizer. In 2001 the measurement method of RRR of Nb-Ti composite superconductors was standardized by International Electrotechnical Commission (IEC) based on a round robin test (IEC 61788-4 Ed. 1). The measurement method of RRR of Nb_3Sn was also standardized (IEC 61788-11) in 2003 based on the inter-laboratory comparison test [1]. However, the coefficient variation that corresponds to the standard deviation of RRR in Nb_3Sn was about 9% in the worst case, which was much larger than 2.44% in Nb-Ti [2].

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IEC recommends the standard measurement method to characterize superconducting wires, since the common measurement method is useful to compare qualities of different superconducting wires. Hence, the reliability of the standardized measurement method is very important. In 2011 the uncertainty of the measurement is theoretically analyzed and compared with experimental results of the round robin test for Nb-Ti superconductors in IEC 61788-4 Ed. 3. Since each measurement method of RRR is similar, the two measurement methods were unified in IEC 61788-4 Ed. 4 in 2016 [3].

On the other hand, the standard deviation of RRR in ITER type Nb_3Sn strand is also reported to be fairly large [4, 5]. The reason for the relatively larger standard deviation in Nb_3Sn than in Nb-Ti has been argued in the working group (WG4) in the technical committee 90 on superconductivity in IEC. One reason may be inhomogeneity in Nb_3Sn superconductor. In fact the preliminary inter-laboratory comparison test for the same sample showed that the coefficient of variation was very small [3]. Then, WG4 examined a new round robin test to clearly distinguish intrinsic uncertainty in measurement method and inhomogenity of wires. In this paper the result of the round robin test is reported. The uncertainty in the measurement

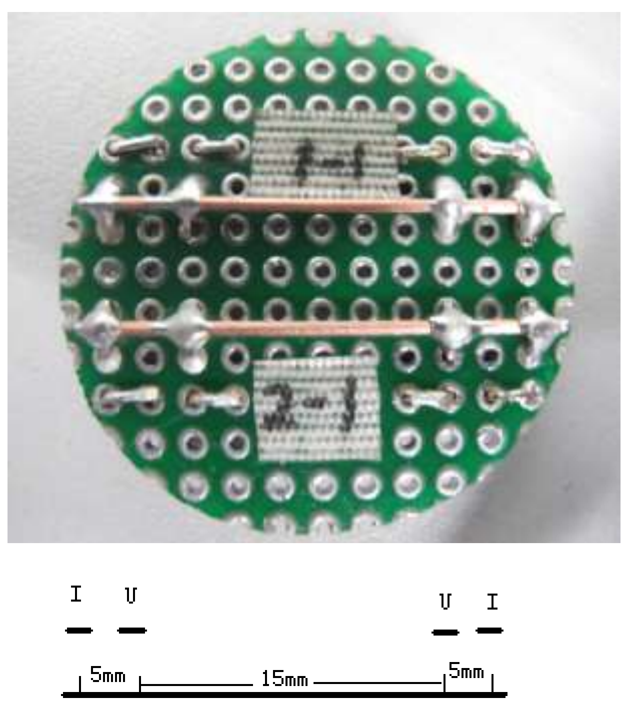

Fig. 1. Photo of mounting of specimens.

method is found to be small enough, while the inhomogeneity of Nb_3Sn wires is fairly large.

II. Experiments

The samples used here are two internal tin-processed Nb_3Sn composite superconductors prepared by Western Superconducting Technologies (WST), China. The diameter was 0.82 mm, the copper to non-copper ratio was 1.0 and the number of filaments was about 3000 for the two wires. The critical current of wires 1 and 2 was 250 and 270 A, respectively, at 4.22 K and 12 T. Three specimens were cut from different positions of each wire.

The four terminal method, defined as a reference method in the standard, was employed for six measurements. A modified method was also used for an additional 7th measurement. Since the electrical resistivity of the stabilizer in Nb_3Sn is sensitive to distortion, the specimens were mounted on base plates so as to be free from applied distortion during measurement or shipping from one institute to the next. The distance between two voltage taps was 15 mm and the distance between the current and voltage taps in each end was 5 mm as shown in Fig. 1. The specimens were mounted on a PCB plate cut in a size that fits into the common liquid helium storage dewar. These specimens were measured for seven times in succession by six institutes for a period of around one year. Finally WST measured RRR for all specimens shipped back to China and confirmed that each RRR value was within the range of observed distribution, suggesting that the specimens had not been damaged during the test.

Six of the seven measurements followed the reference method in IEC 61878-4 Ed. 4 [1]. The resistance at room temperature was measured first, where in this standard, the room temperature is defined as 20° C (293 K), and the room temperature resistance was calibrated using the formula:

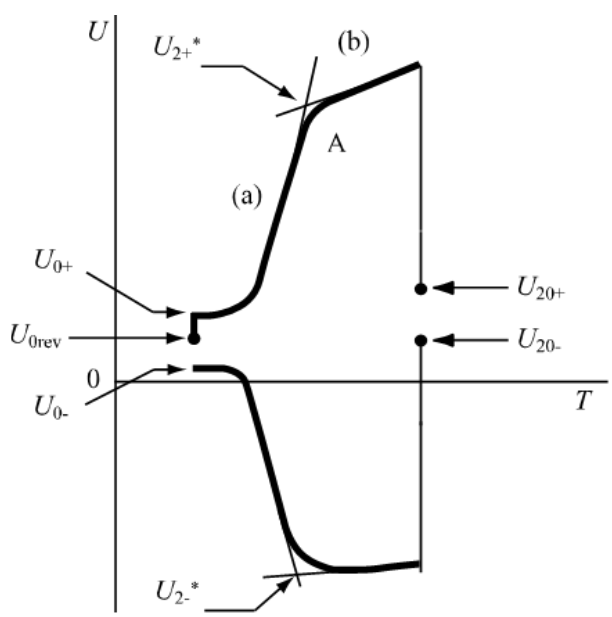

Fig. 2. Voltage vs. temperature curves in two measurements with opposite current directions.

$$R_1 = \frac{R_\mathrm{m}}{[1 + 0.00393(T_\mathrm{m} - 293)]}\ , \qquad (1)$$

where T_m is the temperature in Kelvin at the measurement and R_m is the resistance at that temperature. This simple method has been employed by wire companies, since it makes the measurement simple by skipping a temperature control.

The specimen is slowly immersed in liquid helium bath and cooled to liquid helium temperature over a time period of at least 5 min. Then, the specimen is slowly picked up and placed at sufficiently above the liquid helium surface, and the specimen voltage is measured as a function of temperature. The recommended temperature increase is ranged between 0.1 and 10 K/min. In the superconducting state $U_{0\mathrm{rev}}$ is acquired before the specimen current is applied for the resistance measurement. This voltage may not be zero because of thermoelectric voltage. The specimen current I_2 in the range 0.1 to 10 A/mm^2 is applied so that the specimen voltage above the resistive transition exceeds 10 μV. The initial voltage when the current is applied, U_{0+}, is also acquired. When the specimen is warmed up, the voltage starts to increase sharply, then becomes gradual above the complete transition to the normal state, as illustrated in Fig. 2. The voltage vs. temperature curve is measured sufficiently above the transition but below 25 K. The specimen current is decreased to zero and the corresponding voltage, U_{20+}, is acquired. Then, the specimen is immersed in the liquid helium again, and the same measurement is repeated after the direction of the applied current is reversed. The specimen voltage just after applying current in the superconducting state and that when the current is removed above the transition, U_{0-} and U_{20-}, are acquired. Here we followed the simple reference method in IEC standard that allows us to measure many samples by repetition in a short time. It is also allowed to adopt equipment to control the specimen temperature [3].

The straight lines are drawn in each obtained voltage vs. temperature curve: one is line (a) drawn to fit the sharp resistive transition and the other is line (b) drawn to fit the gradual increase in the fully normal state. The voltage just above the resistive transition is determined at the intersection of these two lines. The corresponding voltages in each measurement are denoted by U_{2+}^* and U_{2-}^*. The corrected voltages are obtained as $U_{2+} = U_{2+}^* - U_{0+}$ and $U_{2-} = U_{2-}^* - U_{0-}$. Then, the average voltage in which the thermoelectric voltage is approximately cancelled is given as

$$\overline{U}_2 = \frac{|U_{2+} - U_{2-}|}{2}. \qquad (2)$$

The cryogenic resistance is determined as

$$R_2 = \frac{\overline{U}_2}{I_2}. \qquad (3)$$

Two conditions must be fulfilled to confirm that the effect of thermoelectric voltage is sufficiently small:

$$\frac{|U_{0+} - U_{0\mathrm{rev}}|}{\bar{U}_2} < 0.01 \tag{4}$$

and

$$\frac{|\Delta_+ - \Delta_-|}{\bar{U}_2} < 0.03, \tag{5}$$

where $\Delta_+ = U_{20+} - U_{0+}$ and $\Delta_- = U_{20-} - U_{0-}$. If these conditions are fulfilled, the residual resistance ratio is obtained as

$$r_{\mathrm{RRR}} = \frac{R_1}{R_2}. \tag{6}$$

A modified method was employed by Yeungnam University in Korea for one measurement (YNU(2)). In this measurement the voltage was recorded as a function of time instead of temperature. This method, which is described as an alternative method in IEC 61788-23*, is considered to be useful when the temperature increase is sufficiently slow and smooth.

III. Results and Discussion

Figure 3 shows the obtained residual resistance ratios of six specimens measured seven times, and measured RRR values of each specimen are listed in Table. I. Table II shows the averaged value (AVE), standard deviation (STD) and coefficient of variation (COV) of RRR for each specimen. COV defined as the value of STD divided by AVE is an important parameter that directly corresponds to the uncertainty of measurement. It is found that the value of RRR is even different between specimens cut from the same wire. This suggests that the Nb_3Sn wire is not homogeneous along the length.

The obtained COV for Nb_3Sn is ranged 1.70 to 3.98% and comparable to that for Nb-Ti, 2.44% [2]. If the result on specimen #2-2 with the largest COV value is disregarded, it is comparable to or even better than that for Nb-Ti.

Now we discuss the origin of uncertainty in the measurement. We apply the same analysis method [2] that has been used for Nb-Ti wires to estimate the correlation between the RRR value and room-temperature and cryogenic-temperature resistances.

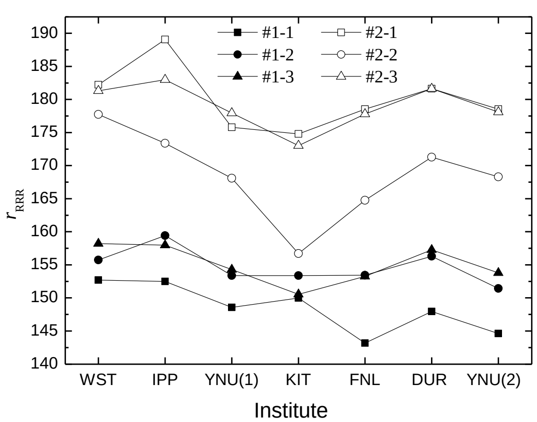

Fig. 3. Residual resistance ratios of six specimens in the order of measured dates.

*IEC 61788-23 Ed. 1, Residual resistance ratio measurement – residual resistance ratio of Nb superconductors. This is now in the stage of Final Draft of International Standard (FDIS).

Table I. Values of RRR for six specimens. The voltage-time curve is used for determination of the cryogenic resistance for the measurement of YNU(2). Although the significant digits of observed RRR value are three, four digit numbers are listed for the analysis of uncertainty.

Institute (Country)	r_{RRR}						Measured Date
	#1-1	#1-2	#1-3	#2-1	#2-2	#2-3	
WST (China)	152.7	155.7	158.2	182.2	177.7	181.3	Jun. 13, 2015
IPP (China)	152.5	159.4	158.0	189.1	173.4	183.0	Nov. 13, 2015
YNU(1) (Korea)	148.6	153.4	154.3	175.8	168.1	177.9	Jun. 14, 2015
KIT (Japan)	150.0	153.4	150.6	174.8	156.7	173.0	Sep. 14, 2015
FNL (USA)	143.2	153.4	153.3	178.6	164.8	177.8	Mar.15, 2015
Durham (UK)	148.0	156.3	157.3	181.6	171.3	181.6	Aug. 15, 2015
YNU(2) (Korea)	144.6	151.4	153.8	178.6	168.3	178.1	May 16. 2016

Table II. Average, standard deviation and coefficient of variation for the six specimens.

	#1-1	#1-2	#1-3	#2-1	#2-2	#2-3
AVE	148	155	155	180	169	179
STD	3.64	2.63	2.86	4.81	6.71	3.35
COV(%)	2.45	1.70	1.84	2.67	3.98	1.87

The correlation can be precisely seen by calculating the correlation coefficient, ρ_k. The coefficient is defined as

$$\rho_k = \frac{\sum_i (R_{ki} - \overline{R_k})(r_{\mathrm{RRR}i} - \overline{r_{\mathrm{RRR}}})}{\{[\sum_i (R_{ki} - \overline{R_k})^2][\sum_i (r_{\mathrm{RRR}i} - \overline{r_{\mathrm{RRR}}})^2]\}^{1/2}}, \tag{7}$$

where $k = 1$ or 2 for the room temperature or cryogenic measurement, respectively. When the magnitude of ρ_k is ranged between 1.0 and 0.7, correlation is strong, and when it is below 0.2, there is almost no correlation.

Table III. Correlation coefficients of RRR with room-temperature and cryogenic resistances.

Correlation coefficient	#1-1	#1-2	#1-3	#2-1	#2-2	#2-3
ρ_1	0.253	0.461	0.705	0.462	0.345	0.045
ρ_2	-0.989	-0.988	-0.980	-0.873	-0.975	-0.967

Table IV. The distribution width of RRR (Δr_{RRR}) is compared with the distribution width of the first term in formula (8).

Specimen	$r_{\mathrm{RRR}}(R_1)$			$r_{\mathrm{RRR}}(R_2)$			Δr_{RRR}
	$a_1[\times 10^{-4}$ S$]$	b_1	$a_1 \Delta R_1$	$a_2[\times 10^{-4}$ S$]$	b_2	$a_2 \Delta R_2$	
#1-1	28.79	-107.5	2.9	-25.92	303.8	10.4	10
#1-2	40.00	-201.4	3.6	-27.21	311.2	7.6	8
#1-3	43.33	-224.3	6.2	-31.33	332.0	6.3	7
#2-1	17.56	25.6	6.3	-37.70	364.4	11.7	14

#2-2	29.16	-96.4	6.7	-31.21	337.0	20.0	21
#2-3	3.34	149.9	0.4	-35.56	351.9	9.2	10

The calculated correlation coefficients of all specimens of Nb_3Sn are shown in Table III. It can be said that the negative correlation is very strong for the cryogenic resistance, while the positive correlation is medium or weak for the room-temperature resistance. That is, too large RRR value can result when the cryogenic resistance is incorrectly underestimated. This feature can be clearly seen in Fig. 4 for specimen #1-1. The straight lines in the figures are represented as

$$r_{\mathrm{RRR}} = a_k R_k + b_k, \tag{8}$$

where a_k and b_k are parameters. These parameters are obtained using the least square minimum method. The obtained parameters for all specimens are listed in Table IV, where the values of $a_k \Delta R_k$ are also compared with Δr_{RRR}, the distribution width of RRR, where ΔR_k is the distribution width of each resistance. The distribution width of each quantity is simply defined as the difference between the observed maximum and minimum values. It can be seen that the distribution width of RRR is mostly determined by the distribution width of cryogenic resistance. This is largely different from the case of measurement of Nb-Ti wires. In the case of Nb-Ti, the resistance of copper just above the resistive transition around 9 K is almost constant. In the case of Nb_3Sn, on the other hand, the resistive transition is completed above 15 K where the resistance of copper changes nonlinearly with temperature. Hence, it is considered that drawing a straight line in the nonlinear region causes a fairly large uncertainty. Nevertheless, the resultant uncertainty of the observed RRR value is mostly below 2.5 %. This means the target uncertainty can be safely set as 5.0 % with coverage factor $k = 2$. As a result, it can be concluded that the reference method described in IEC 61788-4 is reliable to determine RRR of Nb_3Sn composite superconductors.

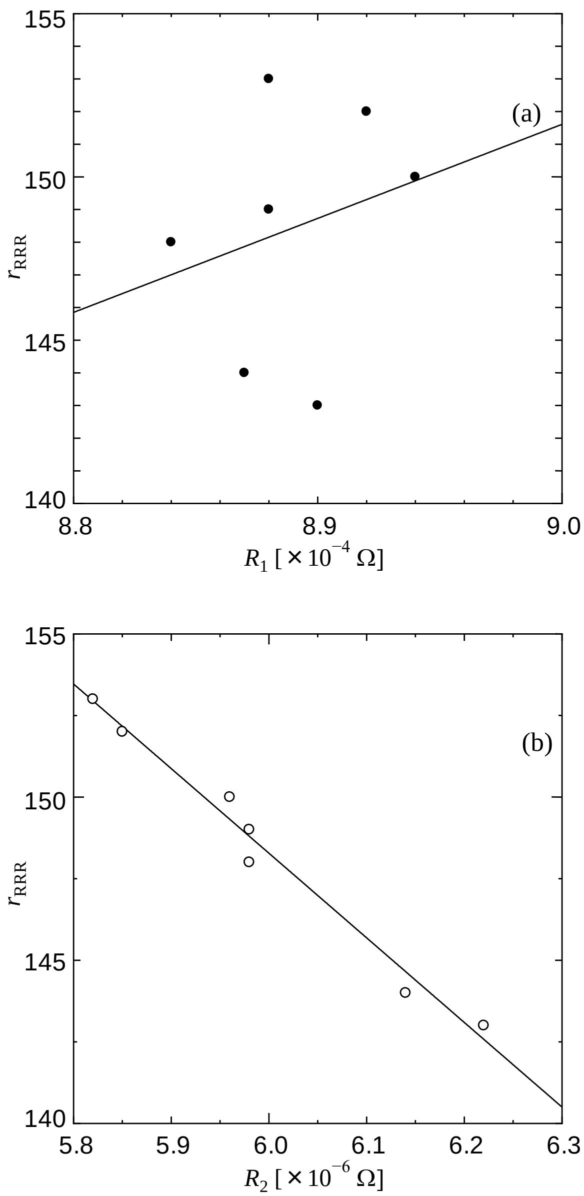

Fig. 4. Residual resistance ratio vs. (a) room-temperature resistance and (b) cryogenic resistance for specimen #1-1.

Here we discuss the reason for similar magnitudes of uncertainties between Nb_3Sn and Nb-Ti in spite of relatively large uncertainty in determination of the cryogenic resistance in Nb_3Sn. The uncertainty in determination of the room-temperature resistance can be assumed to be roughly the same for both Nb_3Sn and Nb-Ti. Then, the only reason we can conjecture for the relatively large uncertainty in Nb-Ti is a possible non-uniformity in specimens measured *in parallel* by participating laboratories. This conjecture may imply that more precise estimation of the uncertainties of the test method requires that the each participating laboratories in the RRT should measure the RRR of the same specimens *in serial order*.

On the other hand, this round robin test clarified that Nb_3Sn composite superconductors are fairly inhomogeneous along their length. This may be caused by the high sensitivity to heat treatment conditions or to random pores or breaks in the diffusion barrier. The large standard deviations of RRR in refs. [4] and [5] will result from the same reason. It is recommended, therefore, to measure several specimens of a given superconductor to evaluate the distribution of the RRR value.

IV. Summary

Round robin test of RRR has been carried out for Nb_3Sn composite superconductors to examine the measurement method described in IEC 61788-4. The obtained uncertainty was as small as that for Nb-Ti composite superconductors, although the main reason for the uncertainty exists in determination of the cryogenic resistance from the intersection of two straight lines. It is concluded that the standard test method in IEC 61788-4 can be used as a reliable method to measure RRR for Nb_3Sn. At the same time it was also found that the RRR value of Nb_3Sn composite superconductor is appreciably inhomogeneous. It is recommended to measure several specimens to check the inhomogeneity.

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