VAMAS INTERCOMPARISON OF CRITICAL CURRENT MEASUREMENT IN Nb_3Sn WIRES

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Abstract

The VAMAS technical working party in the area of superconducting and cryogenic structural materials has recently carried out the first world-wide intercomparison of critical current, I_c, measurement on multifilamentary Nb_3Sn wires. Three sample wires were supplied from each of EC (European Communities), Japan and USA. The total number of participant labs were 24 (EC 11, Japan 8 and USA 5). There were few restrictions for the I_c measurement at participant labs. The standard deviations of the I_c values reported from these labs varied among test samples, and were 6-21% of averaged I_c's at 12 Tesla.

Introduction

The superconductivity technology may have tremendous impact on important areas of science and technology and should be developed under the concept of the long term project whereby international cooperation would play an essential role. This is the underlying idea in the VAMAS which stands for the Versailles Projects among summit participant countries on Advanced Materials and Standards.[1] The VAMAS Technical Working Party (TWP) on superconducting and cryogenic structural materials consists of representatives of participant countries. The TWP has carried out an intercomparison on the critical current, I_c, measurement in Nb_3Sn multifilamentary wires: I_c is the most important superconducting parameter from the practical point of view.

The purpose of the present intercomparison (round robin) test on I_c is to identify parameters affecting the I_c by accumulating and evaluating the measurement results. The eventual goal of the research is to provide recommendations for the performance of short sample critical-current measurements of Nb_3Sn superconductors.

Participants, Samples and Test Procedure

11 EC, 8 Japanese and 5 US laboratories listed in Table I have participated in the round robin test on the I_c. The distribution of test samples and accumulation of resulting I_c data in EC, Japan and USA were performed by the respective central labs; BCMN(Belgium), NRIM, and NBS.

Multifilamentary Nb_3Sn wires with relatively small current carrying capacity (<500 A at 8 Tesla) were chosen as the test samples which could easily be tested at any participant laboratory. Three sample wires were supplied, one from each of EC, Japan and USA; these samples are labeled disorderly just as sample A, B and C in this paper.

Sample A fabricated by a bronze method has a wire diameter of 0.8 mm and 114 sub-bundles each containing 90 Nb-Ta filaments in a bronze matrix. The Cu stabilizer is located at the center of the wire and separated from the filament region by a Ta barrier. The volume fraction of Cu stabilizer in the wire is much smaller compared to those in other 2 samples.

Sample B has a wire diameter of 1.0 mm and 7 sub-bundles each separated by a Nb barrier from the Cu stabilizer outside. Each sub-bundle contains 721 Nb filaments in a Cu-Sn-Ti alloy matrix. This sample was also fabricated by a bronze process.

Sample C prepared by an internal-Sn diffusion method has a wire diameter of 0.68 mm and 37 sub-bundles each containing 150 Nb filaments. The filament region is separated from the outer layer of Cu by a single Ta barrier.

Specifications and the cross sectional views of these samples are given in Fig 1. Upper critical fields, H_{c2}, for sample A and sample B are enhanced by

Table I. Participant laboratories in I_c round robin test.

Europe (11)
Atominstitut der Oesterreichischen Univ. (Austria)
Inst. Experimental Physik, Oester. Univ. (Austria)
S.C.K./C.E.N (Belgium)
S.N.C.I., C.N.R.S. (France)
Kernforschungszentrum Karlsruhe (FRG)
Siemens AG (FRG)
Vacuumschmelze GmbH (FRG)
E.N.E.A, Centro di Frascati (Italy)
High Field Magnet Lab., U. Nijmegen (Netherlands)
Clarendon Laboratory (UK)
Rutherford Appleton Laboratory (UK)
Japan (8)
Electrotechnical Laboratory
Furukawa Electric Co., Ltd.
Hitachi Ltd.
I.M.R., Tohoku University
I.S.I.R., Osaka University
Japan Atomic Energy Research Institute
Kobe Steel, Ltd.
National Research Institute for Metals
USA (5)
Brookhaven National Laboratory
F.B.N.M.L., Massachusetts Institute of Technology
Lawrence Livermore National Laboratory
National Bureau of Standards
University of Wisconsin

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additions of Ta and Ti, respectively.

Specimen wires from these samples, each several meters long, were distributed to participant labs through relevant central labs. Such wires were cut into parts as specimens at each participant lab and one specimen, wound on a heat treatment holder of drum shape, was collected from participant labs and heat treated at the relevant central labs (central reaction). The reacted specimens were then returned to the participant labs for measurements. In some cases, the participant labs also performed their own heat treatments on additional specimens of the samples (self reaction). The self reaction of samples was not possible at all labs.

Specifications of heat treatment holders were different from lab to lab. At most of the labs stainless steel tubes with a spiral groove on the outer surface were used to have a definite coil pitch. The surfaces of such tubes were usually coated with ceramics or oxidized prior to specimen mounting, in order to avoid reaction of the specimen with the holder. At some labs the same holders were used for both heat treatment and measurement, thereby reducing the possibility to damage the specimen by handling.

Central reaction was carried out in the following manner. For samples A and B, all specimens were heat treated in one vacuum furnace at once. The temperature of the furnaces was well controlled within ± 5 °C with time and in space. For sample C, specimens were individually heat treated in a dynamic vacuum. Both ends of a specimen of internal-Sn processed sample C were extended to a position where the temperature was kept below the melting point of Sn in order to avoid the outflow of molten Sn from the wire. The heat treatment conditions for samples A, B and C were 700 °C for 96hr, 670 °C for 200hr and 700 °C for 48hr, respectively.

More than half of self reactions were done in vacuum. At other labs specimens were encapsulated and heat treated in an argon or a hydrogen atmosphere. The heat treatment temperature was also well regulated in the self reaction.

Apparatuses and Measurement Conditions

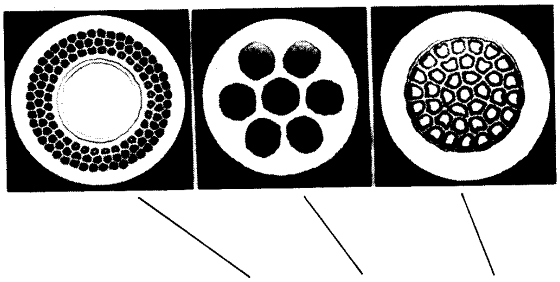
Informations about measurement apparatuses and experimental conditions at each laboratory were described in formatted sheets prepared and distributed prior to the measurements.

At most of participant labs superconducting solenoid magnets were used to generate fields of 8-16.5 Tesla. At some labs copper solenoid magnets or hybrid type magnets were used which can generate fields over 20 Tesla. In any of these magnets rms ripple fields which may cause heat generation in the specimen, were less than 0.1% of the field generated. Field determination was carried out by means of NMR, Hall probe or rotating coil magnetometer, with estimated accuracies of 0.1% to 1%.

At most of the labs transistor type power sources were used to supply electric currents to the specimen. rms ripple noises were less than 0.1% of the full power. The accuracy of current determination was within 0.5% at most of the labs. In order to amplify the voltage signals generated in the specimen, nanovoltmeters were used at most of the labs. At some labs the signals were directly led to high sensitive XY recorders. The typical level of noises observed varied from 0.01 μV to 2 μV, depending on the apparatuses used.

The total length of a specimen, the distance between voltage taps and the distance between a current tap and its nearest voltage tap were much varied among labs. The shortest specimen used in a lab had a total length of about 215 mm and 1.5 turns of winding on the measurement holder. The longest specimen used was 2000 mm long and had 30 turns on the holder.

The variety of materials, e.g., fiberglass reinforced plastic (FRP), stainless steel, alumina, hastelloy and brass were used as the measurement holder. The



	sample A	sample B	sample C
Fabrication Method	Bronze	Bronze	Internal Sn
Wire Diameter (mm)	0.8	1.0	0.68
Structure	NbTa/CuSn	Nb/CuSnTi	Nb/Cu/Sn
Cu/non-Cu	0.22	1.68	0.88
Bronze/Cores	2.8	2.5	3.1
Filament Diam.(μm)	3.6	4.5	2.7
No. Filaments	10,000	5,047	5,550
Heat Treatment	700 C 96h	670 C 200h	700 C 48h

Fig.1 Specifications and cross sectional views of round robin test samples.

Table II. Homogeneity study on sample A at SCK/CEN. Averages and standard deviations of I_c's for fields of 7-10 Tesla.

Field (Tesla)	7	8	9	10
Average (A)	424.5	354.8	296.6	246.4
Std. Dev. (A)	6.0	5.8	5.0	5.8
Std.Dev/Ave(%)	1.5	1.7	1.7	2.4

Table III. Homogeneity study on sample B at Clarendon Lab. Averages and standard deviations of I_c's for fields of 8-14 Tesla.

Field (Tesla)	8	10	12	14
Average (A)	303.8	214.1	150.9	102.3
Std. Dev. (A)	2.5	2.8	1.6	1.0
Std.Dev/Ave(%)	0.8	1.3	1.0	1.0

holders had ring or bar shaped current terminals of copper. Most of holders had a spiral groove on them. The specimen was mounted in a groove on the holder with both ends soldered to current terminals, and fixed using a bond such as grease, varnish, epoxy, and solder. In some cases no bond was used.

The Lorentz force caused by the interaction of the transport current with the applied field has an essential effect on the I_c. This force acts as a tensile or compressive stress to specimen when the spiral specimen generates a central field parallel or antiparallel to the direction of the applied field. At most labs, the direction of both fields were antiparallel. The effect of the field direction on the I_c was examined at several labs.

The I_c was defined at a current where a certain voltage gradient or a certain resistivity appeared along the superconducting specimen. Values of I_c at 5, 10, and 100 μV/m and at integer numbers of magnetic fields were requested to be reported in the present round robin test; I_c values at 10^{-14} and 10^{-13} Ω m were optional.

The relationship between the voltage V, and the transport current, I is empirically expressed as

$$V \propto I^n$$

where the exponent n is nearly constant in the small voltage region. A larger n corresponds to a sharper transition in the specimen. n values were also requested to be reported. In the case where n values were not reported by the participant, they were estimated by using the following relation,

$$n = 1/\log I_c(100\mu V/m)/I_c(10\mu V/m).$$

Homogeneity Study

As the purpose of this test was the intercomparison of results obtained at different labs, it was absolutely important that all the test specimens supplied should have identical superconducting properties. It is difficult, however, to fabricate a sample with homogeneous properties along the whole length of a wire because of the complicated structure of Nb_3Sn multifilamentary composite. The homogeneity in superconducting properties was examined on sample A at SCK/CEN and on sample B at Clarendon Lab.

The homogeneity study at SCK/CEN was performed on 21 specimens of sample A. Specimens were taken from the various parts of a test wire, each wound on a holder and heat treated at Rutherford Lab together with those of central reaction. The I_c measurement was carried out at 4.3 K and at 7-10 Tesla.

115 I_c data defined at a voltage of 10 μV/m for 21 specimens were analyzed at SCK/CEN. For voltages larger than 10μV/m specimens showed a tendency to quenching. 15 specimens quenched under a voltage of 10μV/m. The quench currents were usually lower compared to the true I_c's expected, and were not taken into account in the statistical treatment of the I_c data. For each magnetic field averages and standard deviations of I_c's were calculated according to the 10μV/m voltage and listed in Table II. Standard deviations are 2.4% of average of I_c's at 10 Tesla and decrease to 1.5% at 7 Tesla.

The homogeneity in sample B was examined at Clarendon Lab. 7 specimens were taken from the test wire, each mounted onto a stainless steel holder, and heat treated at Rutherford Lab altogether in a furnace. The I_c measurements were carried out at 4.2 K and at 7-15 Tesla. Averaged I_c's at a voltage of 10 μV/m and standard deviations are summarized in Table III.

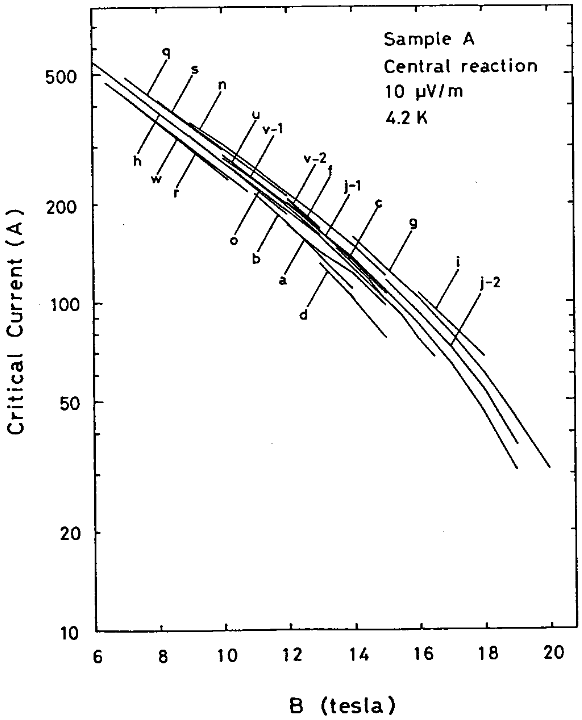


Fig.2 I_c's at 10 μV/m as a function of field for sample A of central reaction; results of participant labs.

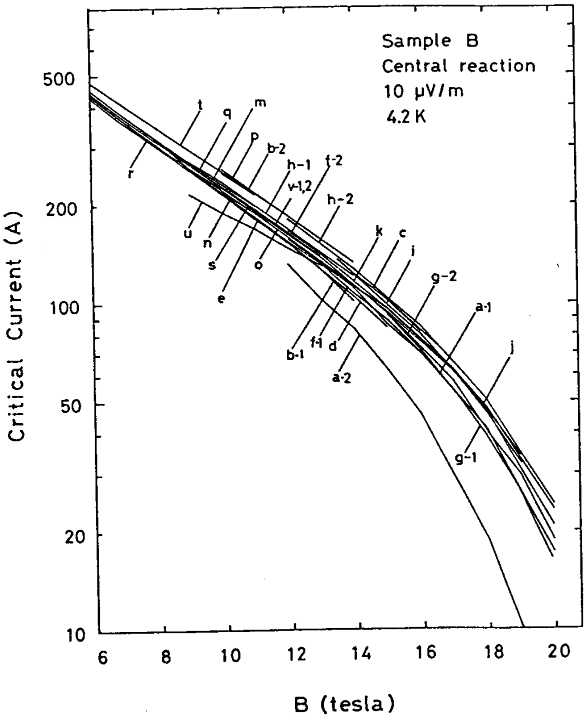


Fig.3 I_c's at 10 μV/m as a function of field for sample B of central reaction ; results of participant labs.

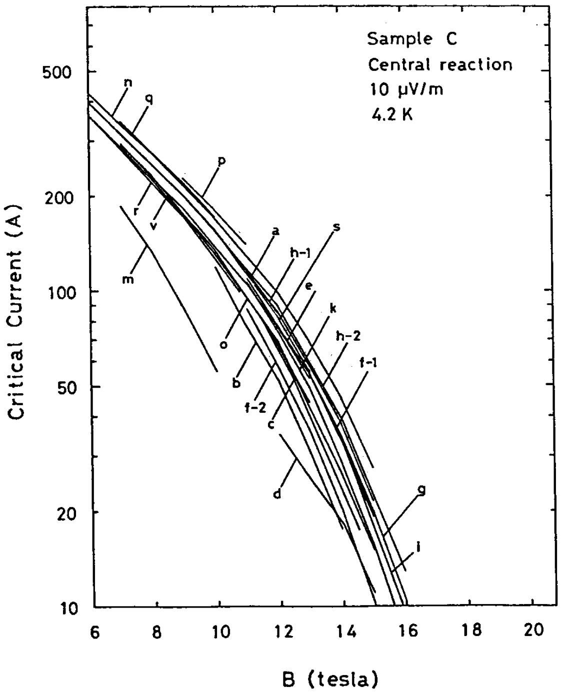


Fig.4 I_c's at 10 μV/m as a function of field for sample C of central reaction ; results of participant labs.

Standard deviations are within 1.3% at all fields.

Based on these homogeneity studies, it may be concluded that the present test samples A and B are rather homogeneous in superconducting properties over the whole length of wire and adequate for the round robin test. The homogeneity study has not yet been carried out on sample C.

Results of the Round Robin Test

In Figs 2, 3 and 4 I_c's at 10 μV/m are shown as a function of applied field for samples A, B and C, respectively. The name of labs is coded like a, b, c in no special order. These results were obtained in the antiparallel self field except for those of labs q and s.

The semilogarithmic plots show that for samples A and B the logs of the I_c decreases almost linearly with increasing field up to about 15 Tesla, and then drops off more rapidly above this field. I_c's in sample C decrease in a similar way but with a steeper slope, and start to drop off above about 12 Tesla. The averaged n values generally decrease with increasing magnetic field and are much more scattered than the I_c's, especially for sample A.

In Tables IV and V are shown averages and standard deviations of I_c data and n values obtained at participant labs, respectively. Four extraordinarily outlying data sets were excluded in the calculations because these data sets were obtained in cases where the specimens stuck to the reaction holders and were likely damaged in removal or for other similar reasons. There was no single magnetic field where all labs reported data on any sample. A future publication comparing all data sets at a single magnetic field, by means of a Kramer extrapolation method, is planned. Coefficients of variation of I_c's at 10 μV/m and 12 Tesla are about 7, 6 and 21 % of the averages for samples A, B and C of central reaction, respectively, and become larger at higher magnetic fields. These values are appreciably larger than those obtained in the homogeneity studies which are 2.4 and 1.3 % for samples A and B, respectively.

Table IV. Averages and standard deviations of critical currents at 10 μV/m for samples A, B and C of central reaction.

Field (Tesla)	sample A				sample B				sample C			
	No.	Ave. (A)	σ (A)	σ /Ave (%)	No.	Ave. (A)	σ (A)	σ /Ave (%)	No.	Ave (A)	σ (A)	σ /Ave (%)
6	1	544.1	0.0	0.0	4	447.4	18.7	4.2	3	393.8	25.7	6.5
7	4	447.9	26.2	5.9	7	372.2	15.4	4.1	7	309.3	24.3	7.9
8	5	388.7	21.8	5.6	8	315.2	13.0	4.1	6	248.6	20.8	8.4
9	7	327.4	23.3	7.1	10	265.3	11.1	4.2	9	197.3	20.6	10.4
10	9	277.1	18.0	6.5	15	229.1	13.1	5.7	11	150.0	18.5	12.3
11	7	234.4	14.3	6.1	13	195.3	12.6	6.5	13	106.9	16.2	15.1
12	11	195.7	13.6	6.9	17	163.6	10.4	6.3	16	72.5	15.2	20.9
13	10	158.0	14.5	9.2	14	134.6	7.2	5.3	13	45.4	10.7	23.6
14	10	130.5	15.5	11.9	16	114.3	7.8	6.8	13	30.0	8.4	28.0
15	10	106.7	12.9	12.1	12	92.2	5.0	5.4	10	16.9	5.0	29.4
16	6	91.9	10.8	11.8	10	75.8	4.8	6.4	7	8.5	2.6	30.5
17	3	71.8	6.3	8.8	4	57.5	2.5	4.4	2	3.4	0.2	6.4
18	4	57.4	9.0	15.7	7	43.7	2.6	8.4	2	2.1	0.7	33.7
19	3	36.8	5.3	14.4	4	30.6	2.6	8.4	1	1.0	0.0	0.0
20	2	26.4	4.4	16.7	5	19.4	2.7	13.9				

No:number of data, σ :standard deviation, σ /Ave:coefficient of variation

In Fig. 5 I_c's at 10 μV/m and at 12 Tesla reported from labs are compared. In cases no I_c values were reported at 12 T, they were estimated by extrapolations not exceeding 2 T. In the figure, $\Delta I_c/\sigma$ denotes the difference in I_c between lab and average normalized to the standard deviation, σ. Labs are arranged in incremental order of mean laboratory value of $\Delta I_c/\sigma$. The arrows attached to some of the data symbols indicate that these data actually lie either above or below the vertical limits of the plot. The maximum to minimum spread in I_c at each lab is almost within 2 times of σ, rather small compared to the total maximum to minimum spread (6 times of σ). These results clearly indicate systematic differences among the labs in the measured I_c of each sample. These differences may be due to the

Table V. Averages and standard deviations of n values for samples A, B and C of central reaction.

Field (Tesla)	sample A				sample B				sample C			
	No.	Ave.	σ	σ /Ave (%)	No.	Ave.	σ	σ /Ave (%)	No.	Ave	σ	σ /Ave (%)
6					4	37.2	2.4	6.5	3	32.2	2.8	8.5
7					6	39.3	5.0	12.8	7	32.8	7.6	23.3
8	4	78.3	33.9	43.4	8	36.8	5.6	15.2	6	32.5	4.9	14.9
9	5	87.8	38.5	43.8	10	34.2	4.5	13.1	9	28.6	3.5	12.1
10	7	58.0	21.4	36.9	13	32.6	5.5	16.8	11	26.7	5.2	19.3
11	6	50.0	23.3	46.7	13	32.3	8.2	25.3	13	26.8	8.8	32.8
12	9	52.7	18.7	35.5	17	30.3	6.6	21.8	16	21.9	7.7	35.0
13	8	43.9	16.5	37.7	15	28.3	7.5	26.7	13	18.7	7.4	39.7
14	7	35.1	14.8	42.3	16	27.8	6.2	22.2	13	14.5	5.1	34.9
15	6	37.3	13.1	35.1	12	28.1	5.1	18.0	10	12.4	3.5	28.5
16	3	33.0	3.6	10.8	10	25.9	4.0	15.4	7	10.5	2.7	25.5
17					4	24.2	5.3	21.9	2	8.8	1.6	17.7
18	1	22	0.0	0.0	7	20.6	4.3	20.9	3	6.3	0.7	10.8
19					4	19.0	3.5	18.2	1	5.2	0.0	0.0
20					5	12.8	2.3	17.8				

No:number of data, σ :standard deviation σ /Ave:coefficient of variation

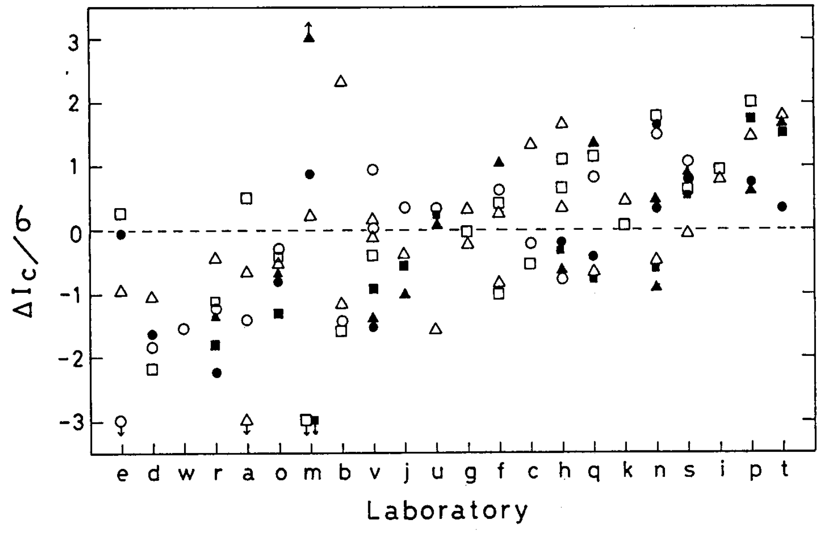


Fig. 5 Comparison of I_c's among participant labs at 12 T. Symbols (○, ●), (△, ▲) and (□, ■) refer to the samples A, B and C (central reaction, self reaction), respectively.

different sample holders, sample handling, the measurement method, and variations in instrument calibration. More detailed papers addressing these possible sources of systematic differences are planned for future publication.

The ratios of $I_c(\uparrow\uparrow)$ to $I_c(\uparrow\downarrow)$ are shown in Fig. 6 for sample B. $I_c(\uparrow\uparrow)$ and $I_c(\uparrow\downarrow)$ denote the I_c's for which the self and the applied fields are in parallel and antiparallel directions, respectively. If the specimen is tightly fixed on the holder, there should be no difference between $I_c(\uparrow\uparrow)$ and $I_c(\uparrow\downarrow)$, the ratio of $I_c(\uparrow\uparrow)$ to $I_c(\uparrow\downarrow)$ being unity. The tightness of the fixing depends on the bonding material and its amount. Epoxy resins such as 'stycast' seem to give better fixing than grease, as is apparently shown in Fig. 6. Some of the curves of the ratio $I_c(\uparrow\uparrow)/I_c(\uparrow\downarrow)$ drop below unity at higher fields in Fig. 6. If a specimen is uniformly deformed by concentric hoop stress, the ratio will approach unity as the field increases. Therefore, nonuniform deformation of specimen may result in the ratio less than unity.

In order to see if the 'self reaction' carried out at each lab was the exact copy of the 'central reaction', data of I_c's for two reactions were compared. This comparison between self and central reactions did not include all of the available data, but rather, it only included data from labs that measured both of their specimens on similar holders. Averaged I_c's of self reacted sample B are nearly the same as those of centrally reacted one. However, averaged I_c's of self reacted sample A are always smaller than those of centrally reacted one, while those of self reacted sample C are smaller at lower fields and larger at higher fields than those of centrally reacted one. These results indicate that I_c's of samples A and C are more sensitive to the heat treatment condition than those of sample B. The reaction time for sample C is the shortest of the three samples and, consequently, the sensitivity of sample C to both reaction time and temperature may be greater than for the other samples. Furthermore, sample C's apparent sensitivity to the reaction conditions may be due to an inadequate confinement of the internal tin at the specimen ends during the heat treatment. The coefficient of variation of I_c of self reacted samples, which may reflect the difference in reaction conditions among labs, correspond to 4-8 % of I_c almost independent of magnetic field and sample. The possibility of sample damage incurred during shipping is also a variable in the comparison between the self and central reactions.

Strain Effect

The tensile strain effect on I_c's were examined at NBS, Osaka Univ. and Rutherford Lab using the round robin test samples. Although the measurement details were different, there was good agreement among three sets of data. As examples, results for 14 Tesla at NBS and those for 15 Tesla at Osaka Univ. are compared in Fig. 7 where I_c's have been normalized to the maximum values. The I_c of Nb_3Sn conductor increases with increasing tensile strain up to 0.2-0.3% where the precompressive strain imposed on the Nb_3Sn compound is mostly released. As can be seen in this figure, the tensile stain sensitivity of I_c is largest for sample C and relatively small for sample B. This may be related partly to the H_{c2} of these samples; sample C has a relatively low H_{c2} of ~ 19 Tesla, while it is ~ 24 Tesla for samples A and B. A more complete comparison of the strain effect data will be the subject of a future paper.

Effects of the thickness of the FRP holder on the I_c were examined at NBS using specimens of samples B and

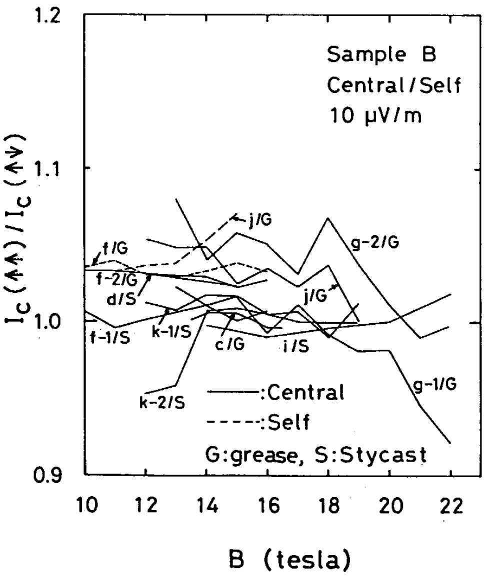


Fig.6 Ratio, $I_c(\uparrow\uparrow)/I_c(\uparrow\downarrow)$ vs. field curves for sample B.

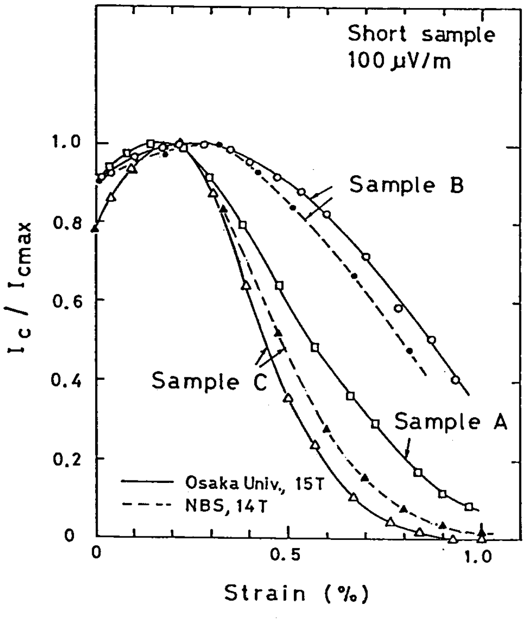


Fig.7 I_c vs. strain curves for samples A, B and C obtained at NBS and Osaka Univ. I_c is normalized to its maximum value.

C. Specimens were first mounted on thick walled FRP holders. After the I_c measurement the holders were bored so as to have thin walls. It was found that I_c's for these bored holders with thin walls were slightly enhanced. The thermal contractions of thick and thin wall holders are different due to the textured structure of FRP, and estimated to be about 0.4% for thick wall and about 0.25% for thin wall on cooling from room temperature to 4.2K. It was concluded that the compressive strain in the specimen resulting from the thermal differential contraction between the FRP holder and the specimen was responsible for the discrepancy in I_c in those specimens mounted on thick and thin wall holders.

The sample holder material and geometry, in conjunction with the specimen bonding method, can have an effect on the strain state of the specimen and this will have a significant effect on the results of the I_c measurements. Thus, this could be a major source of the observed systematic variation in the measured I_c's.

Conclusions

Results of the present round robin test may be summarized as follows.

a) Homogeneity test on the I_c showed the present samples were enough homogeneous along the whole length of wire to be used in the round robin test.

b) Coefficients of variation of I_c's were 7%, 6% and 21% at 12 Tesla for samples A, B and C, respectively. They were largest for sample C which was most sensitive to strain and heat treatment conditions. Coefficient of variation increased at higher fields for all of samples.

c) n values showed larger coefficient of variation than I_c. n values were rather small in case the specimen was soldered on a metallic holder.

d) Strain effects on the I_c of three wires were examined. It was pointed out that the strain in the specimen was a major origin for the scatter in I_c.

e) Materials of the specimen holder and tightness of fixing have significant effects on the I_c through the strain effect.

Several parameters influencing the I_c value of Nb_3Sn wires have been extracted and analyzed through the VAMAS round robin test. However, the present round robin test was performed with few restrictions on the measurement method, and more strict determination of measurement conditions will be required to minimize the scatter in I_c and to furnish a really effective standard method of the I_c measurement.

A more complete report on the present round robin test will be submitted to the steering committee of VAMAS from the Technical Working Party. A number of papers addressing specific aspects of this round robin test are planned for future publication.

Trade names are used in this document to specify the measurement details. In no instance does this identification imply endorsement by the authors or their institutions, nor does it imply that the particular products are necessarily the best available.

The authors wish to thank all the scientists and representatives who have been involved in the present round robin test for their cooperation.

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[2] L.F. Goodrich and S.L. Bray, "Critical-Current Measurements of Nb_3Sn Superconductors: NBS Contribution to the VAMAS Interlaboratory Comparison", submitted for publication in Cryogenics.

Trade names are used in this document to specify the measurement details. In no instance does this identification imply endorsement by the authors or their institutions, nor does it imply that the particular products are necessarily the best available for that purpose.