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VAMAS Activities for Standardization of Measurement Methods for Superconducting Materials*

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ABSTRACT

VAMAS research activities are reported on the establishment of standard measurement methods for critical current, ac losses, and critical field in superconducting materials.

KEYWORDS: Superconducting materials, international standardization, critical current, ac losses, critical field.

INTRODUCTION

VAMAS, the Versailles Project on Advanced Materials and Standards, was established via the Versailles economic summit meeting in 1982. Its mission is to foster the development of internationally acceptable standards for various advanced materials, such as superconducting materials, through collaboration on pre-standards measurement research, intercomparison of test results, and consolidation of existing views on priorities for standardization action.

In the area of superconducting materials, a Technical Working Area, TWA6, was formed in 1986 to work on both superconducting and cryogenic structural materials. With respect to superconducting materials, TWA6 covered measurement methods for critical currents in Nb_3Sn, and critical fields and ac losses in NbTi superconductors [1]. In 1993, TWA6 was terminated and TWA16 was established exclusively for superconducting materials, focusing on the characterization and evaluation of critical currents in oxide superconductors: TWA17 deals with cryogenic structural materials.

PRE-STANDARDS RESEARCH

In order to arrive at standard measurement methods acceptable to the international community, interlaboratory measurement comparisons on practical, important, superconducting properties have been carried out in both TWA6 and TWA16.

DC critical current measurement method for Nb_3Sn. Two series of interlaboratory comparisons of critical current measurements on Nb_3Sn multifilamentary conductors [2,3] were carried out with the participation of 26 laboratories from Japan, Europe and USA; Table 1 shows the list of participant laboratories in the two intercomparisons, whereas Table 2 shows the specifications of the test conductors used. In the first intercomparison, data scatter in measured critical current was rather large (coefficients of variation of critical currents ranging from 8 to 30% depending on the test conductors), indicating that the specimen strain can be a significant source of critical current measurement variability. In the second intercomparison, specimens from a single Nb_3Sn conductor (sample B) were measured, wherein measurement techniques were more closely specified. In particular, the measurement mandrel on which a test conductor was mounted after reaction treatment was made to a common specification. In addition, each participant was requested to make a measurement on a specimen of a standard NbTi conductor supplied as a reference material.

Table 1 List of participant laboratories in critical current intercomparisons on Nb_3Sn conductors.

FIRST INTERCOMPARISON	SECOND INTERCOMPARISON
Atominstitut der Oesterreichschen University	Atominstitut der Oesterreichschen University
Brookhaven National Laboratory	CNRS/SNCI
Bureau Central de Mesures Nucleaires	Electrotechnical Laboratory
Clarendon Laboratory	ENEA Centro di Frascati
CNRS/SNCI	Hitachi
Electrotechnical Laboratory	Kernforschungszentrum Karlsruhe
Furukawa Electric	Kobe Steel
Hitachi	National Institute of Standards and Technology
Japan Atomic Energy Research Institute	National Research Institute for Metals
Kernforschungszentrum Karlsruhe	Technische Universitaet Wien
Kobe Steel	Teledyne Wah Chang Albany
Lawrence Livermore National Laboratory	Tohoku University
Massachusetts Institute of Technology	Vacuumschmelze
National Institute of Standards and Technology	
National Research Institute for Metals	
Nijmegen University	
Osaka University	
Rutherford Appleton Laboratory	
Siemens	
SCK/CEN	
Technische Universitaet Wien	
Tohoku University	
University of Wisconsin	
Vacuumschmelze	

Table 2 Specifications of samples used for critical current intercomparisons on Nb_3Sn conductors

	Sample A	Sample B	Sample C
Fabrication method	Bronze	Bronze	Internal tin
Wire diameter (mm)	0.8	1.0	0.68
Structure	NbTa/CuSn	Nb/CuSnTi	Sn/Cu/Nb
Cu to non-Cu ratio	0.22	1.68	0.88
Bronze to filament ratio	2.8	2.5	3.1
Filament diameter (μm)	3.6	4.5	2.7
Number of filaments	6156	5047	5550

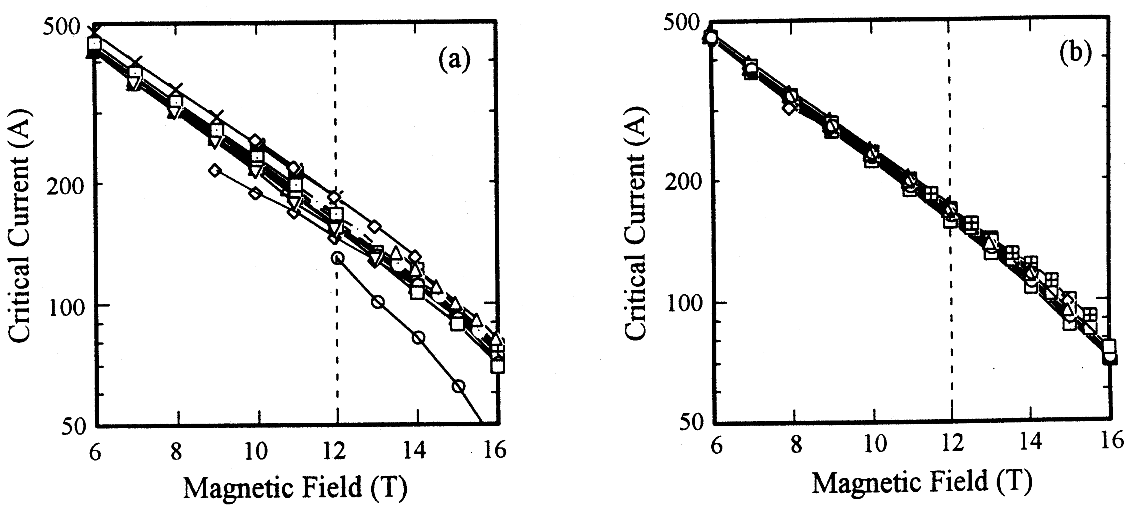

Fig. 1: Summary results of (a) the first and (b) the second interlaboratory comparisons of critical current measurements for the different participating laboratories.

Figure 1 shows the critical current versus magnetic field curves obtained, indicating that all the curves fall in a very narrow range. It turned out that the variability of critical current for the Nb_3Sn sample is comparable to that for the NbTi reference material; the coefficients of variation for critical current at 12 T for sample B were 2.2% in the second intercomparison, while it was 8.0 % in the first intercomparison. These two intercomparisons suggest that the major source for the critical current data scatter is variation in specimen strain and applied field and that specimen handling must be specified precisely in the measurement guideline.

A TWA16 report, including a standard dc critical current measurement method, was published as a supplement volume of Cryogenics [3]. This proposed standard method was later adopted as the base for a corresponding International Electrotechnical Commission (IEC) standard.

AC loss measurement method for NbTi. Two series of interlaboratory comparisons of ac loss measurements on NbTi multifilamentary conductors were carried out with the participation of 22 laboratories from Japan, Europe and USA; Table 3 shows the list of participant laboratories. In the first intercomparison [4], hysteresis, coupling and coil losses were measured using a variety of measurement methods and measuring apparatus. The data scatter among participant laboratories was relatively small for hysteresis losses, whereas it was rather large with respect to coupling and coil losses. In the second intercomparison, measurement methods and the type of measuring apparatus were specified. Namely, two groups were formed that adopted either an ac magnetization method using a pick-up coil or dc magnetization method using VSM or SQUID. Table 4 shows specifications of sample conductors specially manufactured for the test.

The ac magnetization group measured total loss [5]. Data scatter in the hysteresis loss ranged from 6 to 16 %, whereas it was much larger for the coupling loss. It seemed that more precise specifications with respect to test sample geometry and measuring system would be necessary for standardization.

Table 3 List of participant laboratories in ac loss intercomparisons on NbTi conductors.

FIRST INTERCOMPARISON	SECOND INTERCOMPARISON
Battelle Memorial Institute	Atominstitut der Oesterreichschen University
Brookhaven National Laboratory	Battelle Memorial Institute
Central Res. Inst. for Electric Power Industry	Central Res. Inst. for Electric Power Industry
CISE	CISE
Electrotechnical Laboratory	ENEA
Japan Atomic Energy Research Institute	Electrotechnical Laboratory
Kernforschungszentrum Karlsruhe	Hitachi
Kyushu University	Kernforschungszentrum Karlsruhe
National Institute of Standards and Technology	Kobe Steel
National Research Institute for Metals	National Research Institute for Metals
Nihon University	Technische Unversitaet Wien
Tohoku University	Tokai University
Tokai University	Toshiba R&D Center
Toshiba R&D Center	Teledyne Wah Chang Albany
University of Twente	University of Twente
	Vacuumschmelze

Table 4 Specifications of samples used for the second ac loss intercomparison. Diameter of 0.5 mm and twist pitch of 9 mm were common to all of the samples.

Sample Code	H1	H2	H3	H4	I1	I2	I3
Filament diameter (μm)	11.8	3.4	1.3	0.54	1.2	0.54	0.35
Number of filaments	931	6517	56791	336091	56791	336091	866761
Matrix	Cu	Cu	Cu	Cu	CuNi	CuNi	CuNi
Number of bundles	1	7	61	361	61	361	931

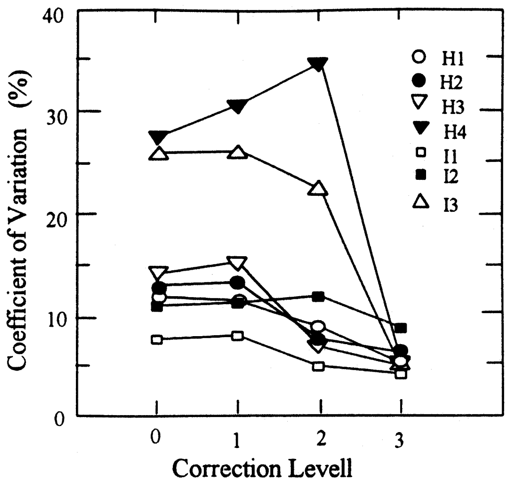

Fig.2 Data scatter in dc magnetization measurement of hysteresis loss (level 0), corrected for temperature (level 1), NbTi superconducting volume (level 2), and proximity effect (level 3).

The dc magnetization group measured hysteresis loss [6]. The data scatter was in the range between 11 and 17%. However, Fig. 2 shows that this can be reduced to approximately 6% after corrections made with respect to proximity effect and NbTi superconducting volume in the sample. It is concluded that standardization of the dc magnetization method may be possible, if error factors involved in the dc magnetization measurement are well controlled.

Critical field measurement method for NbTi. Measurement intercomparison on upper critical field in NbTi [7] was implemented with the participation of 12 laboratories listed in Table 5. NIST-SRM 1457 NbTi [8] wire was used as sample conductor. Measurements were carried out at 4.2 K, and upper critical fields were determined by extrapolating the specimen current to zero. Figure 4 shows upper critical fields thus determined with a specimen current of 100 mA. Data scatter is 0.6%, which is larger than that for measurements of 12 specimens conducted at one site (0.15% at NRIM). This was attributed to the scatter in the magnetic fields used at participant laboratories.

Table 5 List of participant laboratories in critical field intercomparison.

Atominstitut der Oesterreichschen Univ.	Electrotechnical Laboratory
ENEA	Kobe Steel
National Institute of Standards and Technology	National Research Institute for Metals
Technische Universitaet Wien	Tohoku University
University of Twente	Tokai University
Vacuumschmelze	Toshiba

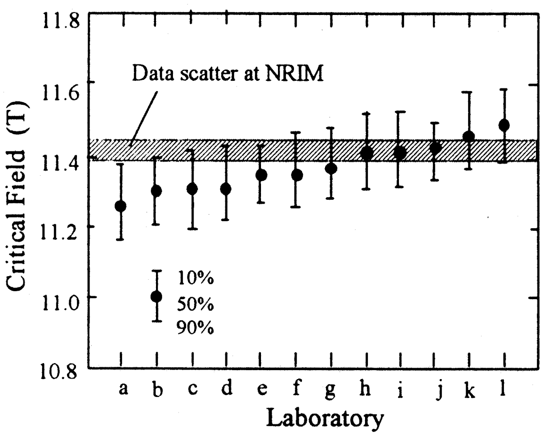

Fig.3: Result of critical field measurement intercomparison. The data symbols indicate the magnetic field at 10, 50, and 90% of the transition.

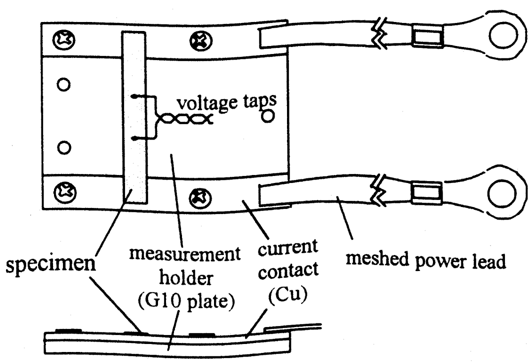

Fig.4: An example of fully instrumented specimen.

Critical current measurement method for Ag-sheathed Bi-oxide superconductors. In each of USA, Europe, and Japan, an independent critical current measurement intercomparion was carried out on short Ag-sheathed Bi-2212 and/or Bi–2223 oxide superconductor tape samples. Samples were either instrumented or free-standing. Fig. 4 shows an example of a fully instrumented specimen which is bonded to an epoxy (G-10) substrate with current and voltage leads attached.

In the USA, six laboratories listed in Table 6 participated in the intercomparison, wherein two kinds of multifilamentary Bi-2223 samples were routed among participant laboratories, either in series or in parallel [9]. Critical current measurements were conducted at 4.2 and 77 K. Figure 5 shows results on the parallel-routed, instrumented specimens. In the parallel route, the variation of the critical current among labs was about 2.4%, although some specimens showed large degradation, probably due to damage caused by specimen routing or handling.

In Europe, the critical current intercomparison was implemented with the participation of 11 laboratories listed in Table 6. Four kinds of Bi-2212 and Bi-2223 tape samples were fully instrumented and routed in series. Substantial data scatter was observed with substantial degradation of the critical current during sample routing. Specimen bonding to the substrate with varnish was effective in minimizing the degradation in critical current during sample routing.

In Japan, the intercomparison was repeated two times, since in the first round [10] with the participation of eight laboratories listed in Table 6 there occurred unacceptable critical current

Table 6 List of participant laboratories in critical current intercomparisons on Bi-oxide superconductors.

US INTERCOMPARISON	Siemens
American Superconductor	Technische Universitaet Wien
Florida State University	University of Milano
Intermagnetics General	University of Twente
National Institute of Standards and Technology	Vacuumschmelze
Oxford Superconducting Technology	
University of Wisconsin	JAPANESE INTERCOMPARISON
	Electrotechnical Laboratory
EUROPEAN INTERCOMPARISON	Iwate University
Atominstitut der Oesterreichschen Univ.	Kobe Steel
CISE	Kyoto University
Clarendon Laboratory	Kyushu University
Forschungszentrum Karlshuhe	National research Institute for Metals
IMGC	Tokai University
Rutherford Appleton Laboratory	Toshiba

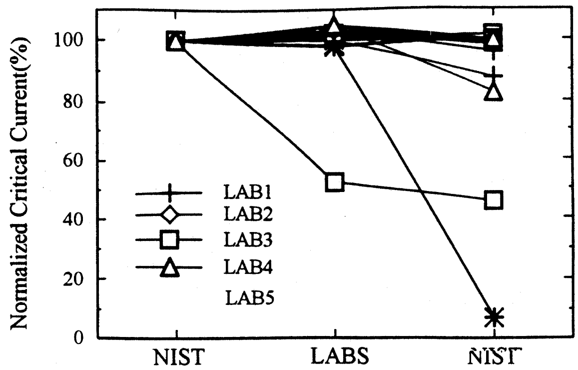

Fig. 5 Result of US critical current measurement intercomparison on parallel-routed samples; critical currents are those for 77 K and zero field.

degradation during sample routing. This was analyzed and attributed to the formation of bubbles in the specimens. In the second round [11] with the participation of six laboratories listed in Table 6, three kinds of Bi-2212 and Bi-2223 multifilamentary samples were parallel-routed. Figure 6 shows measurement results at 4.2 K and 5 T. Although the critical current degradation became smaller than in the first round, a certain level of the degradation was still observed that was serious in some cases. Serious degradation mostly happened in specimens with bubble formation. Specimen routing and handling at each laboratory may be responsible for this degradation, although different measurement details at each of the participant laboratories can also make some difference to the measured critical currents.

Based on these regional intercomparisons, it was concluded that the specimen bonding to the substrate might be very important to the measured critical currents. In Europe and the USA, where specimens were bonded to the substrate, the degradation was relatively small as compared to that found in Japan, where the specimens were mounted on the substrate without using a bonding agent.
This conclusion may be further supported by the reported results of the Chinese domestic interlaboratory comparison of critical current measurements on Bi-oxide superconductor tapes [12]; the degradation of the critical current during sample routing was successfully minimized.

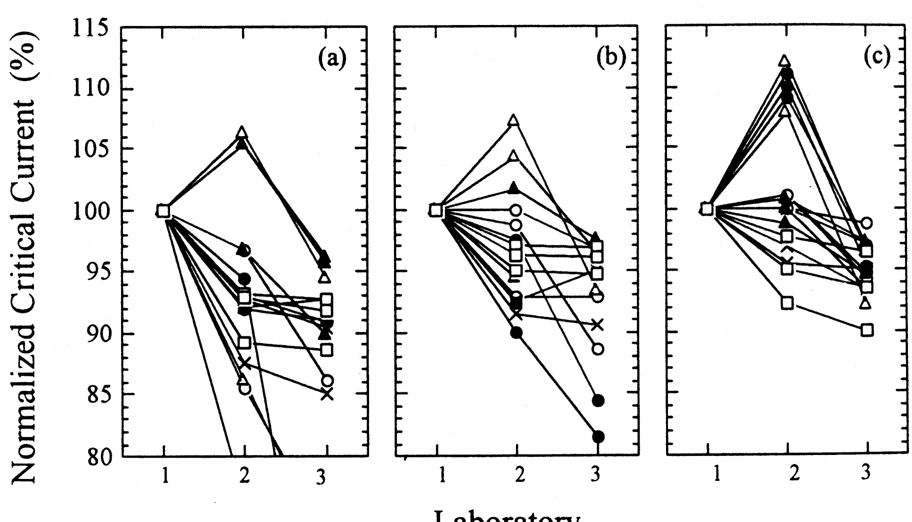

Fig. 6 Results of critical current measurement intercomparison in Japan; on parallel-routed (a) Bi-2223, (b) Bi-2223 and (c) Bi-2212 samples.

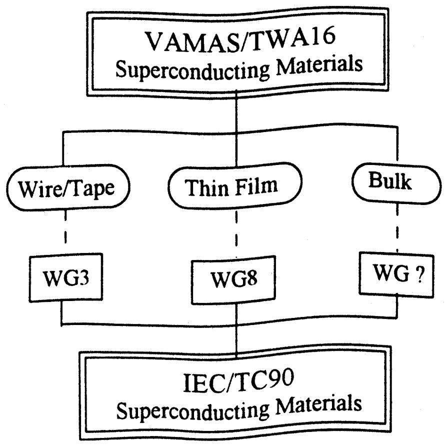

Fig.7: Expansion of TWA16 activities and Liaison with IEC/TC90.

Also based on these intercomparisons, the next step of a worldwide interlaboratory comparison of critical current measurements on a single Bi-oxide superconductor sample is being prepared. The results of this intercomparison, together with those of the above stated intercomparison, will be compiled as a TWA16 report and published in a supplement volume of Cryogenics, as was the case for Nb_3Sn.

FUTURE PLANS

A forthcoming TWA16 report, "A guideline for dc critical-current test method of Ag-sheathed Bi-2212 and Bi-2223 oxide superconductors," will become the basis for a corresponding IEC guideline. In the near future, activities of TWA16 will become more extensive by involving bulk and thin-layer oxide superconductors and will have a stronger link to IEC activities, as shown in Fig. 7. The annual TWA16 meeting will be held in Japan on the occasion of the ISS (International Symposiums on Superconductivity), with the participation of IEC member laboratories.

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